UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

NORIT AMERICAS, INC.
Petitioner,

v.

CALGON CARBON CORPORATION
Patent Owner.

Case: Unassigned
U.S. Patent No. 12,478,948
Issue Date: June 3, 2025

Title: "PERFLUOROALKYL AND POLYFLUOROALKYLSORBENT MATERIALS AND METHODS OF USE"

DECLARATION OF INGRID HSIEH-YEE, PH.D.

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I, Ingrid Hsieh-Yee, declare as follows:

I. INTRODUCTION AND QUALIFICATIONS

1. I am over the age of eighteen and otherwise competent to make this declaration.

2. I have been retained as an independent expert on behalf of Norit Americas, Inc. ("NORIT") for proceedings before the United States Patent and Trademark Office ("PTO") regarding the authenticity and public availability of certain prior art references in relation to U.S. Patent No. 12,478,948.

3. I am being compensated for my time in connection with this Post-Grant-Review at my current consulting rate. I am also being reimbursed for any reasonable expenses associated with my work and testimony in this investigation. My compensation is not contingent on the results of my study, the substance of my opinions, or the outcome of this matter.

A. Qualifications and Professional Experience

4. My complete qualifications and professional experience are described in my academic curriculum vitae (**Appx. A**). The following is a brief summary of my relevant qualifications and professional experience.

5. I was a professor in the Department of Library and Information Science at the Catholic University of America for 32 years and was conferred the title of Professor Emerita in December 2022. I have experience working in an academic

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library, a medical library, and a legislative library. I hold a Ph.D. and a Masters in Library and Information Studies from the University of Wisconsin-Madison.

6. I am an expert on library cataloging and classification and have published two editions on this subject, *Organizing Audiovisual and Electronic Resources for Access: A Cataloging Guide* (2000 and 2006). I taught a variety of courses, including Cataloging and Classification, Advanced Cataloging and Classification, Organization of Internet Resources, Organization of Information, Metadata, Digital Content Creation and Management, Internet Searches and Web Design, Information Literacy Instruction, Advanced Information Retrieval and Analysis Strategies, and The Information Professions in Society. I am familiar with metadata schema design and implementation. In my teaching, I covered the design and implementation of metadata in databases, search engines, digital repositories, digital libraries, and digital archives. I also covered how information organization affects the discovery and access to digital resources on the Internet. My research interests cover cataloging and classification, information organization, metadata, information retrieval, information architecture, digital collections, scholarly communication, social media, user interaction with information systems, and others.

II. BACKGROUND OF LIBRARY CATALOGING

A. Machine-Readable Cataloging ("MARC")

7. I am very familiar with a library cataloging encoding standard known

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as the "Machine-Readable Cataloging" standard, also known as "MARC," which became the national standard for sharing bibliographic data in the United States by 1971 and the international standard by 1973. MARC is the primary communications protocol for the transfer and storage of bibliographic metadata in libraries. Experts in my field reasonably rely upon MARC records when forming their opinions.

8. A MARC record consists of several fields, each of which contains specific data about the work. Each field is identified by a standardized, unique, three-digit code corresponding to the type of data that follows. **Appx. B** is a true and correct copy of Parts VII to X of "Understanding MARC Bibliographic: Machine-Readable Cataloging" (https://www.loc.gov/marc/umb/um07to10.html), a brief description and tutorial published by the Library of Congress in 2009 that explains commonly used MARC fields. For example, the personal author of a work is recorded in Field 100, the title is recorded in Field 245, publisher information is recorded in Field 260, the physical volume and characteristics of a publication are recorded in Field 300, and topical subjects are recorded in Fields 650. More details of MARC21 format for Bibliographic Data are available on the Library of Congress website at https://www.loc.gov/marc/bibliographic/.

9. I am very familiar with library cataloging and processing practices and the relationships between OCLC (Online Computer Library Center) and library catalogs, and how libraries create and share their records to help the public discover

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and access information.

10. OCLC is the largest bibliographic network of the world, and its WorldCat database contains more than 609 million records (as of October 2025) from thousands of member institutions (many of which are libraries of some type) in more than 100 countries. OCLC was founded in 1967 to promote and support library cooperation. According to the third article of the "Amended Articles of Incorporation of OCLC [Online Computer Library Center], Inc.," OCLC was created "to establish, maintain, and operate a computerized library network and to promote the evolution of library use, of libraries themselves, and of librarianship, and to provide processes and products for the benefit of library users and libraries, including such objectives as increasing availability of library resources to individual library patrons and reducing the rate of rise of library per-unit costs, all for the fundamental public purpose of furthering ease of access to and use of the ever-expanding body of worldwide scientific, literary, and educational knowledge and information" (source: https://www.oclc.org/content/dam/oclc/membership/articles-of-incorporation.pdf).

11. OCLC members can contribute original cataloging records in MARC to the system or derive cataloging records from existing records, an activity referred to as "copy cataloging." When an OCLC participating institution acquires a work, it can create an original MARC record for the work in OCLC's WorldShare system (a system for catalogers to create and share MARC records), and the system will

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automatically generate a code for the date of record creation in the yymmdd format, and the creating library's OCLC symbol is recorded in subfield "a" of the 040 field. Once the MARC record is in WorldShare, it becomes available to other OCLC members for adoption to their local online catalogs (*i.e.*, copy cataloging).

12. After a MARC record is created in WorldShare, the record is put into the OCLC WorldCat database. The record also becomes searchable and viewable on WorldCat.org, a free web portal for users to explore more than 10,000 libraries worldwide. Records in WorldCat.org, however, are not presented in MARC fields. Instead, the data elements are labeled to help users interpret the records. Thus, the information stored in MARC records in the OCLC WorldCat database is available to the interested public free of charge through the user-friendly WorldCat.org web portal.

13. In addition, records in the OCLC WorldCat database, which is "the world's most comprehensive database of information about library collections" (https://www.oclc.org/en/worldcat.html), are discoverable on OCLC FirstSearch, a subscription-based search platform for users to explore proprietary databases. WorldCat database records on FirstSearch contain more details than records on WorldCat.org (the free web porta), such as the date of entry data that indicate when records were added to the OCLC WorldCat database.

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B. Library Online Catalogs

14. Library online catalogs gained acceptance in the early 1980s and many libraries migrated their systems to the World Wide Web in the mid-1990s. Library online catalogs are based on MARC records that represent their collections in order to help the public understand what materials are publicly accessible in those libraries. Most libraries with online catalogs have made their catalogs freely available on the Web. These online catalogs offer user-friendly search interfaces. Strong user interest in keyword searches and the popularity of Google have led to the "googlization" of library search systems. As a result, many library catalogs nowadays provide a single search box for users to conduct keyword searches, with additional support for searches by author, title, subject terms, and other data elements such as ISBN (International Standard Book Number). Library catalogs these days also offer features for users to narrow their search results by language, year, format, and other elements. Many libraries display MARC records on their online catalogs with labels for the data elements to help the public interpret MARC records. Many libraries also offer the option to display MARC records in MARC fields.

15. Libraries create MARC records for works they acquire, including books, serials, motion pictures, and publications in other formats. Monograph cataloging is fairly common in libraries, and most libraries make a newly cataloged monograph available to the public soon after the cataloging work is completed,

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usually within a week. Libraries can create original cataloging records or use an existing record in the OCLC WorldCat database to create a copy cataloging record. As soon as the cataloging record is completed, it is added to the library's online catalog for users. If the record is an original record, it is also entered into the OCLC system. If it is a copy cataloging record, the library's holding symbol is attached to the existing original record in the OCLC system to facilitate searching and interlibrary loan.

16. I am personally familiar with many online catalogs, databases, digital repositories, and search engines. In preparing for this declaration, I used the following authoritative information systems to search for records:

- Google Scholar (https://scholar.google.com/)
- Internet Archive (https://archive.org/)
- JSTOR.org (https://www.jstor.org/)
- OCLC FirstSearch (https://firstsearch.oclc.org/; subscription required for access)
- Online catalog of the Catholic University of America (https://libraries.catholic.edu/)
- Online catalog of the Johns Hopkins University Libraries (https://www.library.jhu.edu/)
- Online catalog of the Library of Congress (https://catalog.loc.gov/)

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- Online catalog of the National Library of Medicine (https://catalog.nlm.nih.gov/discovery/search?vid=01NLM_INST:01NLM_INST&lang=en)
- Online catalog of the North Carolina State University Libraries (https://catalog.lib.ncsu.edu/catalog/)
- Online catalog of the University of Colorado Denver, Auraria Library (https://library.auraria.edu/)
- Online catalog of the University of New Hampshire Library (https://library.unh.edu/)
- Online catalog of the University of Wisconsin-Madison General Library System (https://www.library.wisc.edu/)
- Public Records System of the US Copyright Office (https://publicrecords.copyright.gov/)
- PubMed (https://pubmed.ncbi.nlm.nih.gov/)
- WorldCat.org, the free web portal (https://search.worldcat.org/)

C. Scope of this Declaration

17. I have been asked to offer my opinions on the public availability dates of the following documents:

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Exhibit	Prior Art Document
1004	Kempisty, D. M., Xing, Y., & Racz, L. (Eds.). (2018). *Perfluoroalkyl Substances in the Environment: Theory, Practice, and Innovation*, Taylor & Francis Group, Environmental and Occupational Health Series, 2018. ("Kempisty")
1005	Mueller, R., & Yingling, V. (2018, March). Remediation Technologies and Methods for Per-and Polyfluoroalkyl Substances (PFAS). ITRC Fact Sheet. 12 pages. ("Mueller")
1006	McDougall, G. J. (1991, April). The physical nature and manufacture of activated carbon, *Journal of the Southern African Institute of Mining and Metallurgy*, 91(4), 109-120. ("McDougall")
1007	Mimna, R. (2017, January 30). "Granular Activated Carbon: A Proven Solution for PFC Removal," Calgon Carbon Presentation, 30 Jan. 2017, https://www.calgoncarbon.com/app/uploads/PFC-Webinar-Presentation-1-30-17.pdf. ("Mimna")
1009	Appleman, T. D., Higgins, C. P., Quiñones, O., Vanderford, B. J., Kolstad, C., Zeigler-Holady, J. C., & Dickenson, E. R. (2014, March). Treatment of poly-and perfluoroalkyl substances in US full-scale water treatment systems, *Water Research*, 51, 246-255. ("Appleman")
1011	Cannon, F. S., Snoeyink, V. L., Lee, R. G., Dagois, G., & DeWolfe, J. R. (1993, March). Effect of Calcium in field-spent GACs on pore development during regeneration, *Journal-American Water Works Association*, *85*(3), 76-89. ("Cannon")
1013	McNamara, J. D., Franco, R., Mimna, R., & Zappa, L. (2018, January). Comparison of activated carbons for removal of perfluorinated compounds from drinking water, *Journal-American Water Works Association*, 110(1), E2-E14. ("McNamara")
1014	Calgon Carbon. (2015). Reactivation Services Frequently Asked Questions. ("RS FAQ")

18. I am informed by counsel and understand that a given reference is "published" upon a satisfactory showing that such a document has been disseminated or otherwise made publicly available to the extent that persons interested and ordinarily skilled in the subject matter or art could locate it exercising reasonable diligence and obtain the document. I have also been informed by counsel and

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understand that materials available in a library constitute "printed publications" if they are catalogued and indexed according to general library practices and protocols that make the references available and accessible to members of the interested public.

19. I have been informed by counsel that Patent '948 claims April 3, 2019, as its earliest priority date. I have assessed public accessibility of the documents referenced below as of this claimed priority date.

20. I have been informed by counsel that the subject matter of this proceeding generally relates to removal of perfluoroalkyl substances in the environment.

21. I have been informed by counsel that a "person of ordinary skill in the art at the time of the invention" (POSA) is a hypothetical person who is presumed to be familiar with the relevant field and its literature at the time of the inventions. I have been informed by counsel that this hypothetical person is also a person of ordinary creativity, capable of understanding the scientific principles and literature applicable to the pertinent field.

22. With respect to the 948 Patent, a person of ordinary skill in the art (POSA) by April 3, 2019, would have a working knowledge of the principles of water purification and sorbents. A POSA would have gained knowledge of these materials and processes through a mixture of education and work experience, such as by (i) having a Bachelor's degree in environmental engineering, civil engineering,

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mechanical engineering, chemical engineering, or equivalent degree, coupled with at least three years of experience; (ii) by obtaining a Master's degree or Doctorate in environmental engineering, civil engineering, mechanical engineering, chemical engineering, or equivalent degree, but having less experience; or (iii) by having no formal degree, but having experience in the relevant field of at least six years.

23. It is my opinion that such a person would have been actively engaged in learning about the field, possibly through formal instruction, field experience, and bibliographic resources. By at least April 3, 2019, such a person would have had access to a vast array of print and electronic resources, including at least the documents referenced below.

D. Evidence Considered in Forming My Opinion

24. In the preparation of this declaration, I have reviewed **EXs-1004**, **1005**, **1006**, **1007**, **1009**, **1011**, **1013**, and **1014** referenced in paragraph 17 above, and any other documents I reference herein. Each of these is a type of material that experts in my field would reasonably rely upon when forming their opinions. I have provided a further description of materials I considered in **Appx. C**.

III. Authenticity and Public Availability of Kempisty (EX-1004)

A. Authentication

25. **EX-1004** is a true and correct copy of partial content of *Perfluoroalkyl Substances in the Environment: Theory, Practice, and Innovation*, ("Kempisty"),

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edited by D. M. Kempisty, Y. Xing and L. Racz and published by CRC Press in 2018, that I received from counsel. This copy is presented as **EX-1004** in this declaration.

26. Page 1 of **EX-1004** is the front cover that shows the title, subtitle, editors, publisher, and the series that includes Kempisty. Page 2 is title page that contains the same information as the cover and indicates CRC Press is an imprint of the Taylor & Francis Group. Page 3 is the copyright page that shows this book has a "2019" copyright date and Taylor & Francis Group, LLC holds the copyright. This page also shows the URLs of the publishers and the Library of Congress Cataloging-in-Publication Data that shows the ISBN of the hardback copy is "9781498764186" and the ISBN of the ebook is "9780429487125." Pages 4 to 6 are the table of contents that shows Kempisty has 21 chapters in four sections, an author index, and a subject index. Pages 7 and 8 (internal pages xi and xii) are the Foreword. Pages 9 to 11 (internal pages xix to xxi) are the Introduction. Pages 12 to 34 (internal pages 13 to 44) are the text of Chapter 1 "Fluorosurfactants in Firefighting Foams Past and Present" by Stephen H. Korzeniowski, Robert C. Buck, David M. Kempisty, and Martial Pabon; pages 45 to 65 (internal pages 303 to 323) are the text of Chapter 13 "Reactivation of Spent Activated Carbon Used for PFAS Adsorption" by John Matthis and Stephanie Carr; and page 66 to 92 (Internal pages 325 to 351) are the text of Chapter 14 "Ion Exchange for PFAS Removal" by Steven Woodard, Michael G. Nichelsen, and Marilyn M. Sinnett.

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27. To authenticate **EX-1004**, I located a preview copy of Kempisty from the publisher Taylor & Francis at https://api.taylorfrancis.com/content/books/mono/download?identifierName=doi&identifierValue=10.1201/9780429487125&type=googlepdf. This preview file is presented as **Appx. 1004A** in this declaration. I have closely compared **EX-1004** with **Appx. 1004A** and found that the front matter pages and Chapter 1 in **EX-1004** and their corresponding pages in **Appx. 1004A** are the same, confirming the counsel copy of Kempisty is the same as the one from the publisher. The publisher's preview file does not include Chapter 13 and Chapter 14, but the Chapter 13 and Chapter 14 included in **EX-1004** appear to be authentic because these chapters are not missing any intermediate pages and do not contain marks of alteration.

28. **EX-1004** is in a condition that raises no suspicion about its authenticity. Specifically, the front matter and Chapter 1 of **EX-1004** are the same as their corresponding pages in the publisher's preview file (**Appx. 1004A**). In addition, Chapter 13 and Chapter 14 of **EX-1004** are not missing any intermediate pages, and show no visible alterations to those pages. Based on my review and analysis, it is my opinion that **EX-1004** is authentic.

B. CRC Press Webpages on Internet Archive

29. Internet Archive is a digital library that archives scholarly materials, cultural artifacts, and other web resources on the Internet and provides free public

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access to its collections. I have used Internet Archive in the past and am familiar with their Wayback Machine and their practice of assigning an extended URL to an archived resource in the format of http://www.archive.org/web/[Year in yyyy][Month in mm][Day in dd][Time code in hh:mm:ss]/[Archived URL] to show the point of time the archived file was captured.

30. Because Internet Archive usually does not archive web resources the moment they appear on the Internet, and a web resource must be available on the Internet before it can be archived, the archive date of a file captured by the Internet Archive means that the archived file was available on the Internet before, and definitely no later than, the archive date.

31. To determine the public availability date of Kempisty, I searched Internet Archive for information. **Appx. 1004B** is a true and correct copy of the CRC Press webpage for books on Environmental Science. I personally located, identified, and obtained this archived copy. This is the type of materials experts in my field would reasonably rely upon when forming their opinions.

32. Page 1 of **Appx. 1004B** is a screen capture of a Wayback Machine calendar that shows this webpage (https://www.crcpress.com/environmental-science) was archived 41 times between 2014 and 2024, and this particular copy was archived on "AUG 26 2018" and available at https://web.archive.org/web/20180826202517/https://www.crcpress.com/environme

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ntal-science. Pages 2 to 6 are the PDF of this archived copy, and page 5 shows Kempisty is listed with the book cover, title, editors, and a publication date of "July 30, 2018." **Appx. 1004B** informs my opinion that CRC Press published Kempisty on July 30, 2018, and this book was available for public discovery and purchase no later than August 26, 2018.

C. Library Records for Kempisty

33. To determine the public availability of Kempisty at libraries, I searched WorldCat.org, the free web portal, and learned that the Library of Congress (LC) was one of the holding libraries. **Appx. 1004C** is a true and correct copy of the MARC record obtained from LC, an OCLC FirstSearch record, and records from the University of New Hampshire Library. I personally located, identified, and obtained these records. They are the type of records experts in my field would reasonably rely upon when forming their opinions.

34. Pages 1 to 4 of **Appx. 1004C** show the LC MARC record for Kempisty. The first six digits of Field 008 show the record was created on "180319" (*i.e.*, March 19, 2018). Field 955 records local cataloging activities, and shows "1 copy rec'd., to CIP ver." on "2023-05-25" (meaning one copy was received and sent to the Cataloging-in-Publication Program for record verification on May 25, 2023). Subfields "a" and "c" of Field 040 show "DLC" (the OCLC library symbol for the Library of Congress) created and entered the record into the OCLC WorldCat

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database. Fields 008, 955, and 040 inform my opinion that LC created a pre-publication record, known as CIP (Cataloging-in-Publication) record), on March 19, 2018, and entered the record into the OCLC WorldCat database to share it with the OCLC community. After the book was published, LC received a copy and sent it to the CIP Program on May 25, 2023 to verify the pre-publication record.

35. Field 010 shows the LC control number ("2018011967"). Fields 020 show the ISBNs of the hardcopy and ebook of this title. Field 245 shows the title, subtitle, and editors, Field 264 shows the publisher, Field 300 shows the physical description, Field 490 shows the series title, and Field 504 shows a bibliographical note for references and indexes. The editors are recorded in Fields 700 to help users discover this book. The subjects of this book are represented in Field 050 by a Library of Congress Classification (LCC) number ("TD427.D4"), in Field 082 by a Dewey Decimal Classification (DDC) number of "628.5/2," and in Fields 650 by two Library of Congress subject headings, with "Surface active agents $x Environmental aspects" ("$x" indicates a topical subheading), and "Fluorocarbons $x Environmental aspects."

36. Pages 5 to 7 of **Appx. 1004C** are the OCLC FirstSearch record that shows 16 libraries hold this book and have used this record to facilitate the discovery of this book for their users. The title, editors, publisher, year of publication, description, series, note, the 13-digit ISBNs for the hardcopy and ebook, LCC, DDC,

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and Library of Congress subject headings (the first two terms in the Descriptor field) are the same as those presented in the LC MARC record. The Entry field, the Database field, and the Accession No field show this record was entered into the OCLC WorldCat database on "20180319" (*i.e.*, March 19, 2018), and its OCLC record number is 1012206334. This OCLC FirstSearch record confirms that LC created and entered the pre-publication record for Kempisty on March 19, 2018, into the OCLC WorldCat database, making this record available on that date for the OCLC community for copy cataloging.

37. Pages 8 and 9 of **Appx. 1004C** are the bibliographic record for Kempisty from the University of New Hampshire Library. The bibliographic record shows this book is available at the Kingsbury Library with a call number of "TD427.D4 P3945 2019." The title, editors, Library of Congress subject headings, series title, publisher, publication year, physical description, and 13-digit ISBNs are the same as the information presented in the LC MARC record.

38. Page 10 is the MARC record, and Field 008 and Field 040 show the University of New Hampshire Library based their copy cataloging record on the pre-publication record created by LC on March 19, 2018. As a result, other than data specific to the local library, this MARC record is nearly identical to the LC record, meaning the record enables users to discover Kempisty by the Library of Congress control number (Field 010), ISBNs (Fields 020), title (Field 245), and editors (Fields

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700). Users can also conduct subject searches by subject headings (Fields 650) and LCC number (Field 945) to discover this book. Field 945 shows a date of "3-11-19" (*i.e.*, March 11, 2019) for ILL LOAN (meaning interlibrary loan). This date means that Kempisty was available for interlibrary loan at the University of New Hampshire Library no later than March 11, 2019.

D. Library Records for the eBook version of Kempisty

39. To determine the public availability of Kempisty in libraries, I searched WorldCat.org, the free web portal, and found that most libraries held the eBook version of this book ("Kempisty eBook"). **Appx. 1004D** is a true and correct copy of the publisher webpage for this eBook, available at https://www.taylorfrancis.com/books/edit/10.1201/9780429487125/perfluoroalkyl-substances-environment-yun-xing-leeann-racz-david-kempisty. Page 1 is a screen capture of the top of this webpage that shows the title, subtitle, editors, edition, "5 August 2018" as the publication date of this eBook, publisher location, publisher, DOI (digital object identifier), total number of pages, and ISBN of the eBook ("9780429487125"). The right side of the page instructs visitors to this webpage on how to access the ebook and the print version. Pages 2 to 8 are the PDF of this webpage for Kempisty. These pages show the abstract, key features, and individual chapters for users to access, if they are affiliated with institutions that subscribe to the Taylor & Francis eBook collection. Page 3 shows the title, authors, and pages of

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Chapter 1 ("32 pages"), page 5 shows the title, authors, and pages of Chapter 13 ("22 pages"), and page 6 shows the title, authors, and pages of Chapter 14 ("28 pages"). These bibliographic details are the same as the information presented in **EX-1004**, confirming this eBook has the same content as the print version.

40. **Appx. 1004E** is a true and correct copy of three OCLC FirstSearch records for the Kempisty eBook. I personally located, identified, and obtained these records from OCLC FirstSearch, a search system for users to explore proprietary databases, including the OCLC WorldCat database. These records are the type of records experts in my field would reasonably rely upon when forming their opinions.

41. Pages 1 to 6 of **Appx. 1004E** show 102 libraries hold the Kempisty eBook and use this record to facilitate the discovery and access. The title, subtitle, editors, publisher location, publication year ("2018") and the Description field ("1 online resource") show this record is for the Kempisty eBook. The Access field contains a link to the ebook on the EBSCOhost platform (see page 7 for a screen capture of this ebook on EBSCOhost), and the "Materials specified" field includes a link to Taylor & Francis webpage at https://www.taylorfrancis.com/books/e/9780429487125, which leads to the ebook webpage presented as **Appx. 1004D** above (see page 8 for a screen capture of the top of this webpage). The Date of Entry field, the Database field, and the Accession No field show this record was entered into the OCLC WorldCat database on 20180821

(*i.e.*, August 21, 2018) and its OCLC number is "1049150067." I have checked the OCLC WorldShare system and learned that the NetLibrary of Massachusetts created this record. This OCLC FirstSearch record informs my opinion that the NetLibrary created a record for the Kempisty eBook on the EBSCOhost platform and entered the record into the OCLC WorldCat database on August 21, 2018, making this record available for OCLC members whose libraries provide access to the Kempisty eBook on EBSCOhost to use it for copy cataloging.

42. Pages 9 to 13 show 33 libraries hold the Kempisty eBook and use this record to facilitate the discovery and access. The title, subtitle, editors, publication year ("2018") and the Description field ("1 online resource") show this record is for the Kempisty eBook. The Access field contains a link to the ebook in ProQuest Ebook Central on the EBSCOhost platform (see page 14 for a screen capture of an eBook record on ProQuest Ebook Central). The Date of Entry field, the Database field, and the Accession No field show this record was entered into the OCLC WorldCat database on 20180825 (*i.e.*, August 25, 2018) and its OCLC number is "1049914424." I have checked the OCLC WorldShare system and learned that ProQuest Ebook Library was the creator of this record. This OCLC FirstSearch record informs my opinion that ProQuest Ebook Library created a record for the eBook of Kempisty on the ProQuest Ebook Central platform and entered the record into the OCLC WorldCat database on August 25, 2018, making this record available

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for OCLC members whose libraries provide access to the eBook on ProQuest Ebook Central to use it for copy cataloging.

43. Pages 15 to 18 show eight libraries hold the Kempisty eBook and use this record to facilitate the discovery and access. The title, subtitle, editors, publication year ("2018") and the Description field ("1 online resource") show this record is for the Kempisty eBook. Several ISBNs are included in this record ("9780429945021; 0429945027; 9781498764186; 1498764185; 9781138747777; 1138747777 Other: 10.1201/9780429487125). The Access field contains a link to the Kempisty eBook at https://nls.ldls.org.uk/welcome.html?ark:/81055/vdc_100066339082.0x000001. I have checked the OCLC WorldShare system and learned that the National Library of Scotland created this record. The National Library of Scotland's record for this ebook showed that this record is for a "Legal Deposit" eBook that "can only be viewed on National Library premises" (see page 19 for a screen capture of the top of the library record). The Date of Entry field, the Database field, and the Accession No field show this record was entered into the OCLC WorldCat database on 20180908 (*i.e.*, September 8, 2018) and its OCLC number is "1053862345." This OCLC FirstSearch record informs my opinion that the National Library of Scotland created a record for the *Kempisty* eBook, which is a legal deposit copy at their library. The Library entered their record into the OCLC WorldCat database on September 8, 2018, making

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this record available for OCLC members for copy cataloging. Libraries whose *Kempisty* eBook matches the ISBNs in this record would be able to use the record for copy cataloging.

44. Taken together, the three OCLC FirstSearch records inform my opinion that the Kempisty eBook is available on several platforms, and at least three records were created for the Kempisty eBook and entered into the OCLC WorldCat database in August and September of 2018 to help libraries create copy cataloging records to facilitate discovery and access to the ebook. It is therefore my conclusion that the Kempisty eBook became discoverable and accessible to users affiliated with ebook subscribing institutions on August 21, 2018, August 25, 2018, and September 8, 2018.

E. Records of the University of Colorado at Denver

45. **Appx. 1004F** is a true and correct copy of the bibliographic and MARC records for the Kempisty eBook. I personally located, identified, and obtained these records from the online catalog of the Auraria Library of the University of Colorado at Denver. They are the type of records experts in my field would reasonably rely upon when forming their opinions.

46. Pages 1 to 3 of **Appx. 1004F** are the bibliographic record. The title, subtitle, editors, publisher, publication year ("[2018]"), description, series title, contents, subjects, and ISBN for the ebook ("9780429487125") match the

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information presented on the publisher's webpage for this ebook (**Appx. 1004D**), confirming this record represents the Kempisty eBook.

47. Pages 4 and 5 are the MARC record. The first six digits of Field 008 show the record was created on "180928" (*i.e.*, September 28, 2018). Subfield "a" and "c" of Field 040 show "FlBoTFG" created the record and entered it into the OCLC WorldCat database. According to the MARC Code List for Organizations (https://www.loc.gov/marc/organizations/org-search.php), "FlBoTFG" is the code for Taylor & Francis Group, LLC. Field 005 is a machine-generated field that automatically records the last time this library modified this MARC record, and it shows the Auraria Library last modified the record on "20180928140608" (*i.e.*, September 28, 2018, at 2 p.m., 6 minutes and 8 seconds). Fields 008, 040, and 005 inform my opinion that the Auraria Library used the record created by Taylor & Francis on September 28, 2018, for copy cataloging and last modified their record on the same day, making this record available to their library users on that date.

48. Field 020 shows the ISBN for the Kempisty eBook. Field 245 shows the title. Field 250 shows the edition statement. Fields 264 show the ebook publication date and the copyright date. Field 300 shows this work is an online resource that has a total of 520 online pages, includes 143 illustrations, and is available as PDF. Fields 490 and 830 show the series title is recorded as an access point. Field 505 is a contents note. Field 520 is a summary. Field 530 shows this

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work is also available in print. Field 567 informs users of access issues. Subjects of this ebook are represented in Field 050 by a Library of Congress Classification (LCC) number of "TD427.D4" that represents "Detergents. Surfactants" (Library of Congress Classification Schedule T at https://www.loc.gov/aba/publications/FreeLCC/LCC_T2024TEXT.pdf); and by two Library of Congress subject headings in Fields 650, with "Surface active agents" and "Fluorocarbons" as the main topics, each followed by "Environmental aspects" as a topical subheading encoded in subfield x ("|x"). These subject representations enable users to conduct subject searches to discover this ebook.

49. This MARC record (**Appx. 1004F**) shows the Kempisty eBook became discoverable and accessible at the Auraria Library on September 28, 2018. As of that date, interested users could discover this ebook by the ISBN, title, series title, and editors. They could also conduct subject searches by the LCC number and the subject headings to discover this work.

F. Usage Records

50. Actual usage of a publication is reflected by other publications that make reference to it. I have reviewed citations to Kempisty on Google Scholar and found one citation to Kempisty that was first published online in January 2019. This citation is: Fábelová, L., Loffredo, C. A., Klánová, J., Hilscherová, K., Horvat, M., Tihányi, J., ... & Trnovec, T. (2019, April). Environmental ototoxicants, a potential

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new class of chemical stressors. Environmental research, 171, 378-394. The publication history on ScienceDirect (https://www.sciencedirect.com/science/article/pii/S0013935119300477) shows the manuscript was "Received 22 May 2018, Revised 24 January 2019, Accepted 25 January 2019, Available online 26 January 2019, Version of Record 1 February 2019." This citation shows Kempisty was used by other researchers before the April 3, 2019, priority date.

G. Summary of My Opinion on Kempisty

51. **EX-1004** contains partial content of Kempisty and is authentic because it is in a condition that raises no suspicion about its authenticity. Specifically, the front matter and Chapter 1 in this copy are the same as their counterparts in the publisher's preview file (**Appx. 1004A**). In addition, Chapter 13 and Chapter 14 in **EX-1004** are not missing any intermediate pages, and there are no visible alterations to the document.

52. Archived copy of the CRC Press webpage for books on Environmental Science (**Appx. 1004B**) shows that Kempisty was discoverable and available for online purchase on the Internet no later than August 26, 2018. As mentioned earlier, Internet Archive usually does not archive a web resource as soon as it appears on the Internet, meaning the archive date tends to lag behind the actual appearance of a web resource on the Internet. **Appx. 1004B** shows that by August 26, 2018, CRC Press

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had listed Kempisty as a published work on their website. It is therefore my opinion that this book was available for user purchase by that date.

53. The Library of Congress created a pre-publication record for the Kempisty book on March 19, 2018, and entered their record into the OCLC WorldCat database, making it available to OCLC members for copy cataloging. The University of New Hampshire Library used the LC record for copy cataloging, and their record shows a March 11, 2019 interlibrary loan date, indicating Kempisty was available at this library by that date (**Appx. 1004C**).

54. The publisher's webpage for the Kempisty eBook (**Appx. 1004D**) shows the ebook was published on August 5, 2018. Three OCLC FirstSearch records for the Kempisty eBook (**Appx. 1004E**) show that these records were created on August 21 and 25, and September 8, 2018, and available in the OCLC WorldCat database. In August and September of 2018 these records made the Kempisty eBook discoverable by OCLC members, subscribers to OCLC FirstSearch, and the general public that can access to WorldCat.org, the free web portal. Users affiliated with institutions that subscribed to the Kempisty eBook would be able to access the ebook on appropriate platforms free of charge.

55. Records for the Kempisty eBook from the Auraria Library of the University of Colorado at Denver (**Appx. 1004F**) show the library based their copy cataloging record on a record created by Taylor & Francis that published Kempisty

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and the library last revised their record on September 28, 2018, making this ebook discoverable and accessible from the record for their library users on that date.

56. Kempisty is used by researchers of an article by Fábelová et al. that was first published online in January 2019, and published in print in April 2019. The publication history of this article shows the article was "Accepted 25 January 2019, Available online 26 January 2019."

57. Based on the records and documents I have reviewed, it is my opinion that *Perfluoroalkyl Substances in the Environment: Theory, Practice, and Innovation* that was edited by Kempisty, Xing, and Racz and published by CRC Press as a book on July 30, 2018, became publicly discoverable and accessible from the publisher website by August 26, 2018. The print version was publicly available at the University of New Hampshire Library no later than March 11, 2019. The Kempisty eBook was published on August 5, 2018, and became discoverable and accessible on August 26, 2018, on the publisher website. In addition, it became publicly discoverable at libraries in August and September of 2018. Specifically, the Kempisty eBook became discoverable and accessible by users of the Auraria Library of the University of Colorado at Denver no later than September 28, 2018. Citation history shows an article published online in January 2019 cited to Kempisty.

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IV. AUTHENTICITY AND PUBLIC AVAILABILITY OF MUELLER (EX-1005)

A. Authentication

58. **EX-1005** is a true and correct copy of "Remediation Technologies and Methods for Per- and Polyfluoroalkyl Substances (PFAS)," ("Mueller"), by Robert Mueller and Virginia Yingling. This document is a fact sheet of the Interstate Technology & Regulatory Council (ITRC) that I received from counsel. This copy is presented as **EX-1005** in this declaration.

59. Page 1 of **EX-1005** shows the title, a logo of ITRC, and a note on the right side stating "ITRC has developed a series of fact sheets that summarize the latest science and emerging technologies related to PFAS," and highlights the purpose of this remediation fact sheet. **EX-1005** has a total of 12 pages, including a color figure, and section 4 explains "the references cited in this fact sheet, and the other ITRC PFAS fact sheets, are included in one combined list" available on the ITRC web site. Page 12 shows this fact sheet has a "March 2018" publication date.

60. To authenticate **EX-1005**, I personally located, identified, and obtained a copy of Mueller archived by Internet Archive on December 18, 2018, available at https://web.archive.org/web/20181218193617/https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf. This copy is presented as **Appx. 1005A** in this declaration. I have closely compared **EX-1005** with **Appx. 1005A** and found them to be identical. The only difference is that page 1 of

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Appx. 1005A is a screen capture of the Wayback Machine calendar that shows this document was archived six times between "18 Dec 2018" and "8 Jul 2024" and this particular copy was archived on "DEC 18 2018" and available at the URL listed above.

61. **EX-1005** is in a condition that raises no suspicion about its authenticity. Specifically, this copy is not missing any intermediate pages, and there are no visible alterations to the document. Furthermore, this copy is the same as the copy archived by Internet Archive (**Appx. 1005A**). Based on my review and analysis, it is my opinion that **EX-1005** is authentic.

B. Availability of PFAS Fact Sheets

62. Internet Archive is a digital library that archives scholarly materials, cultural artifacts, and other web resources on the Internet. It provides the public with free access to its collections. I have used the Internet Archive in the past and am familiar with their practice of assigning an extended URL to an archived web resource to show the point of time the archived file was captured.

63. Internet Archive aims to archive resources posted on the Internet to inform our understanding of web resources that have been available on the Internet. Internet Archive has been archiving cached web pages since May 1995, and its Wayback Machine reached 1 trillion webpages archived in October 2025 (Internet Archive Blogs, posted on July 1, 2025, at

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https://blog.archive.org/2025/07/01/wayback-machine-to-hit-once-in-a-generation-milestone-this-october-one-trillion-web-pages-archived/). However, Internet Archive is not able to archive every web file ever posted on the Internet because the volume of web files is huge. When the Internet Archive has an archived copy of a web resource, we can conclude that that particular web resource was available on the Internet no later than the archive date.

64. Internet Archive usually does not archive web resources the moment they appear on the Internet, and a web resource must be available on the Internet first before it can be archived. The archive date of a file captured by the Internet Archive therefore is typically later than the date when the file first became publicly accessible on the Internet. In other words, the archive date means that the archived file was available on the Internet before, and definitely no later than, the archive date. For example, if a file was first archived by the Internet Archive on June 1, 2001, that archive date informs us that the file was available on the Internet for users to discover no later than June 1, 2001. By that date, users could use search engines such as Google to conduct keyword searches or title searches to discover the file on the Internet. In addition, interested users could search the URL of a file or an organization on the Wayback Machine, a service of the Internet Archive, to determine if the Wayback Machine has archived a specific file, or web resources from a specific organization.

65. To determine when Mueller became publicly available on the Internet,

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I searched Internet Archive for records. ITRC announcements, announcements of other organizations, and usage records of Mueller inform my opinion that Mueller became publicly available on March 20, 2018. As evidence, I have personally located, identified, and obtained **Appxs. 1005B** to **1005S**. They are the types of materials experts in my field would reasonably rely upon when forming their opinions.

66. I have obtained ITRC PFAS webpage archived by Internet Archive in 2017 and 2018 to document the history of Mueller. **Appx. 1005B** is a true and correct copy of the ITRC PFAS webpage archived by Internet Archive on November 17, 2017, available at https://web.archive.org/web/20171117143016/https:/pfas-1.itrcweb.org/. I used screen captures to produce this appendix. Page 2 shows Interstate Technology and Regulatory Council (ITRC) "has developed six fact sheets to summarize the latest science and emerging technologies for per- and polyfluoroalkyl substances (PFAS)" and page 3 shows the title of Fact Sheet #6 is "Remediation Technologies and Methods." The bottom of page 3 shows "November 2017" as the publication date of this webpage. The link for "Fact sheets" leads to an archived PFAS Fact Sheets webpage, which is presented as **Appx. 1005C** in this declaration. I used screen captures to produce this appendix.

67. Page 1 of **Appx. 1005C** shows the PFAS Fact Sheets webpage was archived 253 times between "17 Nov 2017" and "1 Jul 2026" and this particular copy was archived on "NOV 17 2017," available at

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https://web.archive.org/web/20171117143547/http://pfas-1.itrcweb.org/fact-sheets. Page 1 states that this page "includes in the links for the ITRC PFAS fact sheets. The fact sheets are available as PDF files." Page 2 shows links to the first three fact sheets, and indicates the last three fact sheets ("Remediation Technologies and Methods" included) are "to be published December 2017."

68. The availability of the first three fact sheets was shared on Facebook in November 2017. **Appx. 1005D** is a true and correct copy of the post of Johnsie Lang on "November 20, 2017," on the Facebook of the North Carolina Stop GenX and Nafion in Water, available at https://www.facebook.com/groups/353457201741568/permalink/384656191955002/?rdid=dvFBIZQkBc3VNEQV#. This post shows "ITRC has developed six fact sheets to summarize the latest science and emerging technologies for per- and polyfluoroalkyl substances (PFAS)" and the first three fact sheets "were released last week. Here is the Naming Conventions and Physical and Chemical Properties Sheet, which contains some useful information." This post includes a link ("http://pfas-1.itrcweb.org/.../pfas_fact_sheet_naming...") to this fact sheet.

69. Taken together, **Appx. 1005B**, **Appx. 1005C**, and **Appx. 1005D** inform my opinion that ITRC announced on November 17, 2017, the development of six fact sheets on PFAS and the availability of the first three fact sheets on their website, and the news was shared on Facebook on November 20, 2017.

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70. The PFAS Fact Sheets webpage later announced a later availability date for the last three fact sheets. **Appx. 1005E** is a true and correct copy of the PFAS Fact Sheets webpage archived by Internet Archive on "JAN 19 2018," available at https://web.archive.org/web/20180119214445/http:/pfas-1.itrcweb.org/fact-sheets/. I used screen captures to produce this appendix. Page 2 shows the first three fact sheets are available online, and the last three fact sheets are "to be published February 2018."

C. ITRC Release of Mueller

71. On March 20, 2018, ITRC announced the availability of three new fact sheets. **Appx. 1005F** is a true and correct copy of the ITRC Factbook announcement on "March 20, 2018," available at https://www.facebook.com/profile/100063488651502/search/?q=ITRC%20thrilled%20to%20release%20three%20new%20PFAS. The post announces that "ITRC is thrilled to release three new PFAS fact sheets!" and this Facebook post shows "Remediation Technologies and Methods" is one of the fact sheets made publicly available on that date. This announcement is corroborated by several documents.

72. **Appx. 1005G** is a true and correct copy of the webpage of "Interstate Technology & Regulatory Council (ITRC) News" archived by Internet Archive on "JUL 19 2019," available at http://web.archive.org/web/20190719230817/http://www.itrcweb.org/About/News.

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The Wayback Machine calendar shows the news webpage was archived 48 times between "19 Jun 2013" and "19 Sep 2024" and the detailed calendar shows the news webpage was not archived in 2018 at all, so this archived copy of July 19, 2019 is the first available archived copy after the three new fact sheets became publicly available on March 20, 2018. Pages 2 to 8 are the PDF of this archived copy and page 4 shows a news entry of "3/20/2018 – ITRC is thrilled to release three new PFAS fact sheets! (/web/20190719230817/http://www.itrcweb.org/About/three-new-PFAS-fact-sheets)". This link leads to an ITRC announcement on the Internet.

73. **Appx. 1005H** is a true and correct copy of the archived ITRC news announcement on March 20, 2018. Page 1 shows this announcement was archived three times between August 2019 and April 2021, and this particular copy was archived on August 25, 2019, available at http://web.archive.org/web/20190825090656/https://itrcweb.org/About/three-new-PFAS-fact-sheets. Page 1 shows "ITRC is thrilled to release three new PFAS fact sheets" and the date of this news entry is "March 20, 2018." This archived webpage shows ITRC released three new PFAS fact sheets, including "Remediation Technologies and Methods," on March 20, 2018. Page 2 is the PDF of this archived webpage that shows users can "Click HERE (https://web.archive.org/web/20190825090656/https://pfas-1.itrcweb.org/fact-sheets/) to access the fact sheets."

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74. Taken together, archived copies of the ITRC Facebook posting (**Appx. 1005F**), ITRC News webpage (**Appx. 1005G**), and the ITRC news announcement on their website (**Appx. 1005H**) inform my opinion that ITRC announced the public release of "Remediation Technologies and Methods" on March 20, 2018, making this document publicly available on their website on that date.

D. Public Awareness of Mueller in March and April 2018

75. The availability of Mueller on March 20, 2018, was publicized by a number of non-ITRC publications. **Appx. 1005I** is a true and correct copy of a MERIT Lab blog post that I personally located, identified, and obtained. Page 1 shows this blog post is available at https://www.meritlabs.com/blog/2018/3/21/just-the-facts-pfas-fact-sheets-published-by-itrc, with a title of "March 21, 2018. Just The Facts! PFAS Fact Sheets Published by ITRC." Pages 2 to 4 are the PDF of this webpage that shows ITRC "just published three new fact sheets on Per- and Polyfluoroalkyl Substances (PFAS) along with one updated fact sheet … These four PFAS updates from ITRC are the most current guidance available and will provide environmental professionals with detailed information on PFAS." The four fact sheets reported in this post include "Remediation Technologies and Methods (https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf)". The "3_15_18.pdf" in the filename indicates Mueller was published on March 15, 2018.

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The blog post also describes what this Remediation fact sheet covers: "The fact sheet on Remediation Technologies and Methods (https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf) explores the treatment techniques for solid and liquid matrices. Solid remediation treatment covered include capping, excavation and disposal, sorption and stabilization, and thermal techniques. The liquid techniques discussed include sorption (e.g., granular activated carbon), precipitation/flocculation/coagulation, redox manipulation, and membrane filtration." **Appx. 1005I** informs my opinion that this MERIT Lab blog post publicized the availability of Mueller and described its coverage on March 21, 2018, indicating the author of the post made use of the Mueller fact sheet.

76. The ITRC's March 2018 release of Mueller was also publicized on Facebook. **Appx. 1005J** is a true and correct copy of a post on the North Carolina Stop GenX and Nafion in Water Facebook. I personally located, identified, and obtained this post at https://www.facebook.com/groups/353457201741568/permalink/433432190410735/?rdid=QyM5tAdVl2DQ1qi6#. I used screen captures to produce this appendix. Page 1 shows the post by Johnsie Lang on March 22, 2018, that announces that ITRC "released three new PFAS sheets" ("Mediation Technologies and Methods" included). The post includes a link to the newly released fact sheets at https://pfas-1.itrcweb.org/.../pfas_fact_sheet_fate_and... In 2026 this link no longer works and

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leads to a "Not found, error 404" message on the PFAS website.

77. Another Facebook post also publicized the availability of Mueller in 2018. **Appx. 1005K** is a true and correct copy of a post of "March 31, 2018" on the Clean Cape Fear Facebook. I personally located, identified, and obtained this post from https://www.facebook.com/CleanCapeFear/posts/pfbid02iCEmLpNueUjCGQn6D1BC1Bk3kRdu3pMsRCHSP9mSFpAw5NPKhK7PjX73ci8afXabl?rdid=vKLImBdZoRUzEvSw#. This post reports that ITRC "released three new PFAS fact sheets this month." This post included a link ("https://pfas-1.itrcweb.org/.../pfas_fact_sheet_fate_and...") to the fact sheets. This link leads to a "Not found, error 404" message on the PFAS website in 2026. This post of March 31, 2018, also included a link to the PFAS Fact Sheets webpage.

78. In addition, the availability of Mueller was announced in the "April 1, 2018" issue of TechDirect of the US Environmental Protection Agency. **Appx. 1005L** is a true and correct copy of this online issue. I personally located, identified, and obtained this copy from https://clu-in.org/download/newsltrs/techdirect/td042018.pdf. Page 1 shows the purpose of TechDirect "is to identify new technical, policy and guidance resources related to the assessment and remediation of contaminated soil, sediments and groundwater." The "New Documents and Web Resources" section on page 5 shows "ITRC Releases

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Three PFAS Fact Sheets" ("Remediation Technologies and Methods" included) and explains that "ITRC has developed this series of fact sheets to summarize the latest science and emerging technologies for per- and polyfluoroalkyl substances (PFAS)." It also shows users can view or download the fact sheets at https://pfas-1.itrcweb.org/fact-sheets/.

79. Taken together, the MERIT Lab post (**Appx. 1005I**), two Facebook posts (**Appx. 1005J** and **Appx. 1005K**) and the TechDirect post of the US Environmental Protection Agency (**Appx. 1005L**) inform my opinion that ITRC released Mueller and two other fact sheets on March 20, 2018, and the news was publicized on March 21, 22, 31, and April 1 of 2018 on the Internet by people and organizations interested in the remediation technologies and methods for PFAS. Authors of these posts include links to the three new fact sheets to facilitate access. Although these links no longer work in 2026, in March and April of 2018 readers of the MERIT Lab post, Facebook posts and the TechDirect post would have been able to follow the links to access the newly released fact sheets, including Mueller, at that time, because it is highly unlikely that these authors would provide non-functioning links to frustrate readers.

E. Usage of Mueller

80. Usage of Mueller is reflected by the MERIT Lab post (**Appx. 1005I**), and two other documents. **Appx. 1005M** is a true and correct copy of the Record of

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Meeting of the Groundwater Issues Subcommittee of the Texas Groundwater Protection Committee that I personally located, identified, and obtained from https://tgpc.texas.gov/gwissues/minutes/TGPC_GWIminutes21Mar2018.pdf. Page 1 shows the document title and "March 21, 2018" as the meeting date. Page 6 shows "Deba Dutta (DSHS) referred members to the ITRC PFAS Fact Sheets webpage that was in the handout package. Three new fact sheets had recently been released," including "Remediation Technologies and Methods." **Appx. 1005M** shows that at this meeting of March 21, 2018, members of the Subcommittee were informed of the availability of Mueller and two other fact sheets, and the ITRC PFAS Fact Sheet webpage was included in the handout package.

81. In addition, Mueller was cited by a 2018 article: Steele, M., Griffith, C., & Duran, C. (2018). Monthly Variations in Perfluorinated Compound Concentrations in Groundwater. Toxics, 6(3), 56. https://doi.org/10.3390/toxics6030056. **Appx. 1005N** is a true and correct copy of this article and Mueller is cited as Reference 55: Interstate Technology Regulatory Council. Remediation Technologies and Methods for Per- and Polyfluoroalkyl Substances (PFAS). Available online: https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf (accessed on 22 March 2018). The "3_15_18.pdf" mark in the filename indicates Mueller was published on March 15, 2018. This reference shows the authors accessed Mueller on March 22, 2018, two

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days after ITRC announced the release of Mueller.

82. Taken together, the MERIT Lab post (**Appx. 1005I**), the Record of Meeting of the Groundwater Issues Subcommittee of Texas (**Appx. 1005M**), and the Steele citation (**Appx. 1005N**) inform my opinion that ITRC made Mueller publicly available on March 20, 2018, and interested users made use of this fact sheet on March 21 and 22 of 2018.

F. Mueller on Internet Archive

83. My research on Internet Archive found Mueller was listed as available on the PFAS Fact Sheets webpages archived on August 19, 2018 and March 5, 2020. As evidence, I have personally located, identified, and obtained **Appxs. 1005O** to **1005S** from Internet Archive.

84. **Appx. 1005O** is a true and correct copy of the ITRC PFAS Fact Sheets webpage archived on August 19, 2018, available at https://web.archive.org/web/20180819033439/https://pfas-1.itrcweb.org/fact-sheets/. I used screen captures to produce this appendix. Pages 3 and 4 show "Remediation Technologies and Methods" was "published 3-15-18." The link for "Remediation Technologies and Methods" leads to a copy of Mueller archived in December 2018.

85. **Appx. 1005P** is a true and correct copy of the archived Mueller. Page 1 is a Wayback Machine calendar that shows this document was archived six times

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between "18 Dec 2018" and "8 Jul 2024" and this particular copy was archived on December 18, 2018, available at https://web.archive.org/web/20181218193617/https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf. The filename indicates this document has a "3_15_18" publication date. This archived copy shows Mueller has a total of 12 pages, and the end of page 12 shows a "March 2018" publication date on the right side. This archived copy is the same as **Appx. 1005A** that I discovered earlier.

86. As noted earlier, the archive date of a web resource archived by Internet Archive means the original web resource is available on the Internet no later than the archive date, because a web resource must be on the Internet before it can be archived. This means interested users can use search engines such as Google to search for the original web resource on the Internet before or on the archive date. For example, although the PFAS Fact Sheets webpage archived on August 19, 2018 (**Appx. 1005O**) leads to a copy of Mueller archived in December 2018 (**Appx. 1005P**), if users accessed the PFAS Fact Sheets webpage on the Internet before or on August 19, 2018, they would be able to follow the link for "Remediation Technologies and Methods" to access Mueller. Interested users in 2018 also had the option to search for the archived copy on Internet Archive.

87. Internet Archive archived Mueller six times (**Appx. 1005P**), so I

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reviewed the archiving history of this fact sheet to determine how long Mueller was available on Internet Archive. **Appx. 1005Q** is a true and correct copy of records I reviewed. I personally located, identified, and obtained these web resources. Page 1 is a detailed Wayback Machine calendar for "https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf" (the URL of Mueller) that shows this document was archived six times from 2018 to 2024, but my research found that the links after February 16, 2020, lead to a message of "Not found, error 404" for each archive date. Page 2 of **Appx. 1005Q** demonstrates the PFAS system response to the link for a copy archived on August 26, 2020 (https://web.archive.org/web/20200826110120/https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf).

88. To determine how long Mueller was available for public access, I reviewed archived copies of the PFAS Fact Sheets webpage and found that a copy archived on March 5, 2020, showed Mueller was available. I used screen captures to produce this archived webpage. Page 3 of **Appx. 1005Q** is a Wayback Machine calendar that shows this particular copy was archived on "MAR 05 2020," available at https://web.archive.org/web/20200305013935/https://pfas-1.itrcweb.org/fact-sheets/. Page 5 shows "Remediation Technologies and Methods" was "published 3-15-18" and available. The link for this fact sheet leads to a copy of Mueller archived on February 16, 2020, available at

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https://web.archive.org/web/20200216215831/https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf. This archived copy is presented as **Appx. 1005R** in this declaration. I have closely compared **EX-1005**, **Appx. 1005Q** (the copy archived on December 18, 2018), **Appx. 1005R**, and found them to be the same.

89. Since the links for archived copies of Mueller no longer work from August 26, 2020 on (see **Appx. 1005Q**), I reviewed archived copies of the PFAS Fact Sheets webpages on Internet Archive. **Appx. 1005S** is a true and correct copy of the PFAS Fact Sheets webpage. I used screen captures to produce this appendix. Page 1 shows this particular copy was archived on "APR 17 2020," available https://web.archive.org/web/20200417054249/https://pfas-1.itrcweb.org/fact-sheets/. The entry for "Remediation Technologies and Methods" on page 3 includes a note that "the fact sheet has been withdrawn. The user is directed to the guidance document section 12 for updated information about Treatment Technologies."

90. Taken together, **Appxs. 1005O** to **1005S** inform my opinion that "Remediation Technologies and Methods" was "published 3-15-18" and the earliest archived copy was archived on December 18, 2018. Internet Archive archived this fact sheet six times between December 2018 and July 2024, but only the December 2018 and February 2020 archived copies are available on Internet Archive, and ITRC announced on the PFAS Fact Sheets webpage archived on April 17, 2020, that this

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fact sheet was withdrawn.

G. Summary of My Opinion on Mueller

91. **EX-1005** is authentic because it contains no signs to raise suspicion about its authenticity. Furthermore, **EX-1005** is the same as an archived copy on Internet Archive in December 2018 (**Appx. 1005A**). Based on my review and analysis, it is my opinion that **EX-1005** is authentic.

92. On November 17, 2017, ITRC announced the development of six PFAS fact sheets, indicating three fact sheets were available, and the other three, including Mueller, would become available in December 2017 (**Appx. 1005C**). In a later post, ITRC moved the availability date for Mueller to February 2018 (**Appx. 1005E**).

93. On March 20, 2018, ITRC announced the release of three new fact sheets, including Mueller, on their Facebook (**Appx. 1005F**). This announcement is corroborated by their news record (**Appx. 1005G**) and their announcement on the Internet (**Appx. 1005H**). The ITRC release of three new PFAS fact sheets on March 20, 2018, was publicized by a MERIT Lab post (**Appx. 1005I**), two Facebook posts (**Appxs. 1005J** and **1005K**), and a post on TechDirect of the US Environmental Protection Agency (**Appx. 1005L**). **Appxs. 1005 I** to **1005L** inform my opinion that after ITRC released Mueller on March 20, 2018, interested users publicized this new development on March 21, 22, 31, and April 1 of 2018, and readers of those posts in March up through April 1, 2018 would have been able to follow the links provided

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to access Mueller on the PFAS Fact Sheets webpage on the Internet.

94. Usage of Mueller is reflected by the MERIT Lab post (**Appx. 1005I**), a Record of Meeting (**Appx. 1005M**), and a published article (**Appx. 1005N**). These usage records inform my opinion that after ITRC released Mueller on March 20, 2018, interested users made use of this fact sheet on March 21 and 22 of 2018.

95. My review of the archive history of Mueller found two archived copies are available on Internet Archive, the first copy was archived in December 2018 and the second in February 2020 (**Appxs. 1005P** and **1005R**). I also discovered that ITRC withdrew Mueller in April 2020 (**Appx. 1005S**).

96. Based on the records and documents I have reviewed and analyzed, it is my opinion that Mueller was published on March 15, 2018, and became publicly accessible on March 20, 2018, when ITRC announced its release. Several posts on the Internet publicized the ITRC release of three new fact sheets (Mueller included) on March 21, 22, 31, and April 1 of 2018; and Mueller was used by interested users on March 21 and 22 of 2018.

V. AUTHENTICITY AND PUBLIC AVAILABILITY OF MCDOUGALL (EX-1006)

A. Authentication

97. **EX-1006** is a true and correct copy of "The physical nature and manufacture of activated carbon," ("McDougall"), by G. J. McDougall, *Journal of the Southern African Institute of Mining and Metallurgy*, 91(4), 109-120, that I

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received from counsel. This copy is presented as **EX-1006** in this declaration.

98. Page 1 of **EX-1006** shows the title, author, and synopsis. The top of the page shows this article appears in "J. S. Afr. Inst. Min. Metall., vol. 91, no. 4. Apr. 1991, pp. 109-120." The bottom of the page shows this article appears in the "April 1991" issue of *Journal of the South African Institute of Mining and Metallurgy*. It also shows this article has a "1991" copyright date and the journal holds the copyright. A note indicates the paper was received by the journal on "25th October, 1989" and the journal's ISSN (International Standard Serial Number) is "0038-223X." **EX-1006** shows McDougall has a total of 12 pages, including 11 figures, two tables, six equations, and 20 references.

99. To authenticate **EX-1006**, I obtained a copy of McDougall from the publisher at https://journals.co.za/doi/pdf/10.10520/AJA0038223X_2042. This publisher copy is presented as **Appx. 1006A** in this declaration. I have closely compared **EX-1006** with this copy and found them to be identical.

100. To show McDougall is available at libraries, I obtained a library copy for this article from the University of Wisconsin-Madison General Library System. This copy is presented as **Appx. 1006B** in this declaration. Pages 3 to 14 of this appendix show the full text of McDougall. I have closely compared **EX-1006** with this library copy and found them to have the same content for McDougall.

101. **EX-1006** is in a condition that raises no suspicion about its authenticity.

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Specifically, this copy is not missing any intermediate pages, and there are no visible alterations to the document. Furthermore, this copy is the same as the copy from the journal publisher (**Appx. 1006A**) and a library copy (**Appx. 1006B**). Based on my review and analysis, it is my opinion that **EX-1006** is authentic.

B. Date Stamps on the Copy from the University of Wisconsin-Madison Libraries

102. The library copy of McDougall (**Appx. 1006B**) includes the front matter of the journal issue containing McDougall. Page 1 is the cover that identifies the journal and shows this issue as vol. 91, no. 4 with an issue date of "April 1991". The cover carries two library date stamps. The first stamp shows "Received JUL 24 1991 Univ. Wis. Library" that indicates the university library system received this issue on July 24, 1991. The second date stamp shows "Kurt F. Wendt Library, College of Engineering JUL 25 1991 UW-Madison, WI 53706" that indicates the library of the College of Engineering received this issue on July 25, 1991. A customary library practice is to put newly received issue through the serial check-in process, then promptly process it for public access, usually within a week after serial check in. These two date stamps inform my opinion that the university library system received the issue on July 24, 1991, processed it, and made it available at the library of the College of Engineering on July 25, 1991, meaning McDougall became publicly accessible at the library of the College of Engineering on July 25, 1991.

103. Page 2 is the table of contents of vol. 91, no. 4 that shows the journal's

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title, ISSN, "April 1991" issue date, and page 109 as the starting page for McDougall.

C. Records of the University of Wisconsin-Madison Libraries

104. **Appx. 1006C** is a true and correct copy of the bibliographic and MARC records for the journal containing McDougall. I personally located, identified, and obtained these records from the online catalog of the University of Wisconsin-Madison Libraries. These are the type of record experts in my field would reasonably rely upon when forming their opinions.

105. Pages 2 to 4 present the bibliographic record that shows the journal title, library holdings (print issues from volume 57 (1956) to volume 94 (1994)), variant titles, format, language, corporate contributor, publisher, beginning date of the journal, ISSN, and indexing and abstracting services that index this journal. The title, ISSN, corporate contributor and publisher match the information presented in the library copy of McDougall (**Appx. 1006B**), confirming this record represents the journal containing McDougall.

106. Pages 5 to 7 are the MARC record. The first six digits of Field 008 show the record was created on "760825" (*i.e.*, August 25, 1076), and the code of "c19569999" following the record creation code indicates this publication is a continuing resource that began publication in 1956 and has no pre-determined conclusion date. Subfields "a" and "c" of Field 040 show "UDI" created the original record and entered it into the OCLC WorldCat database for other OCLC members to

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use. According to the Directory of OCLC Members (https://www.oclc.org/en/contacts/libraries.html), "UDI" is the OCLC symbol for the library of the US Department of the Interior, Natural Resources. Fields 008 and 040 inform my opinion that the University of Wisconsin-Madison library system used the record created by the UDI library for copy cataloging.

107. Field 022 shows the ISSN, Field 245 shows the journal title, Field 246 show three variant titles, and Field 260 shows the publisher and journal beginning year. Field 362 shows the journal began with vol. 57, no. 1 in August 1956. Field 510 show this journal is fully indexed by Nuclear science abstract and Engineering Index Month, and selectively indexed by 16 other indexing and abstracting services. These services make articles published in this journal easier to discover. Field 710 shows the parent organization of the journal is included in the record to help users discover this journal.

108. The bibliographic and MARC record show the journal began publication with vol. 57 in 1956, and the University of Wisconsin-Madison library system holds vol. 57 to vol. 94 (1956 to 1994) of this journal, and used the original serial record created by the library of the US Department of Interior, Natural Resource in 1976 to create their copy cataloging record. The MARC record (**Appx. 1006C**) made this journal discoverable in the online catalog of this library system. The date stamps on the library copy of McDougall (**Appx. 1006B**) show the issue

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containing this article was publicly accessible on July 25, 1991, meaning this journal was discoverable in the library system no later than July 1991. Interested users would be able to discover it by the ISSN, title, variant titles, and the parent organization of this journal.

D. Usage Records

109. Actual usage of a publication is reflected by the publications that make reference to it. I have reviewed the citation history of McDougall on Google Scholar to compile a list of citations before 2008. **Appx. 1006D** presents five citation and shows the earliest citation was published in April 1992.

E. Summary of My Opinion on McDougall

110. **EX-1006** is authentic because it contains no signs to raise suspicion about its authenticity. Furthermore, **EX-1006** has the same content for McDougall as the copy from the journal publisher (**Appx. 1006A**) and a library copy (**Appx. 1006B**). These three copies show McDougall is included in a journal issue that has an issue date of April 1991.

111. The date stamps on the library copy (**Appx. 1006B**) show the university library system received the issue containing McDougall on July 24, 1991, and the library of the College of Engineering received this issue on July 25, 1991, making this issue publicly accessible at the library. The bibliographic and MARC records for the journal containing McDougall from the University of Wisconsin-Madison

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Library System (**Appx. 1006C**) show the library system holds the print version of volumes published from 1956 to 1994, and used an original serial record created in 1976 to create their copy cataloging record, making this journal discoverable in their library catalog. The date stamps on **Appx 1006B** show the journal was discoverable by at least July 1991 in this library system. The MARC record also shows this journal is fully indexed by two indexing and abstracting services, and partially indexed by 16 other indexing and services, which make the articles of this journal easier to discover by users. Citation records (**Appx. 1006D**) shows the earliest citation appeared in April 1992.

112. Based on the documents and records I have reviewed and analyzed, it is my opinion that McDougall became publicly accessible on July 25, 1991 at the library of the College of Engineering of the University of Wisconsin-Madison.

VI. AUTHENTICITY AND PUBLIC AVAILABILITY OF MIMNA (EX-1007)

A. Authentication

113. **EX-1007** is a true and correct copy of "Granular Activated Carbon: A Proven Solution for PFC Removal," ("Mimna"), by Richard Mimna of Calgon Carbon, that I received from counsel. This document is the presentation of a webinar and is presented as **EX-1007** in this declaration.

114. Page 1 is Slide 1 of the presentation that shows the title, authors, a date of "January 30, 2017," and a "2017" copyright date with Calgon Carbon Corporation

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as the copyright holder. **EX-1007** has a total of 35 slides, including figures and charts. The final slide shows the file is available at www.calgoncarbon.com/PFOA.

115. To authenticate **EX-1007**, I searched Internet Archive for records and located an archived copy of Mimna. This copy is presented as **Appx. 1007A** in this declaration. As evidence, I used a screen capture, included as page 1 of **Appx. 1007A**, to show this PDF document was archived once by Internet Archive on "SEP 18 2017" and available at https://web.archive.org/web/20170918144158/http://www.calgoncarbon.com/wp-content/uploads/2016/02/PFC-Webinar-Presentation-1-30-17.pdf. I have closely compared **EX-1007** with this archived copy and found them to be the same.

116. **EX-1007** is in a condition that raises no suspicion about its authenticity. Specifically, there are no visible alterations to the document. Furthermore, this copy is the same as the copy archived by Internet Archive (**Appx. 1007A**). Based on my review and analysis, it is my opinion that **EX-1007** is authentic.

B. Internet Archive Records

117. **Appx. 1007A** shows Mimna was archived once on September 18, 2017. This archive date means that interested users would be able to discover and access this presentation on this date.

118. Since the final slide of **EX-1007** shows this presentation file can be found at www.calgoncarbon.com/PFOA, I searched this URL in Wayback Machine

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for archived copies. **Appx. 1007B** is a true and correct archived copy of this webpage that I personally located, identified, and obtained. This is the type of record experts in my field would reasonably rely upon when forming their opinions.

119. Page 1 of **Appx. 1007B** is a screen capture of a Wayback Machine calendar that shows the PFOA webpage at http://www.calgoncarbon.com/PFOA/ was archived 31 times between 2016 and 2024, and this particular copy was archived on "FEB 22 2017" and available at https://web.archive.org/web/20170222172255/http://www.calgoncarbon.com/PFOA/. Pages 2 to 4 are the PDF of this archived copy, and page 4 includes a link for "Webinar: Granular Activated Carbon – A Proven Solution for PFC Removal" that leads to the archived copy of Mimna at https://web.archive.org/web/20170918144158/http://www.calgoncarbon.com/wp-content/uploads/2016/02/PFC-Webinar-Presentation-1-30-17.pdf (presented as **Appx. 1007A** above). As evidence, the Wayback Machine calendar of the archived copy of Mimna is included as page 5 of **Appx. 1007B**.

120. **Appx. 1007B** shows that it is fairly straightforward for interested users to discover Mimna on Internet Archive in 2017. As of February 22, 2017, interested users could begin their search on Internet Archive with the PFOA webpage on the Calgon Carbon website and follow the link for the webinar title to locate and access Mimna. If the link to Mimna in the archived PFOA webpage did not work because

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Mimna itself was not archived until September 2017, interested users could easily use a search engine such as Google to access the PFOA webpage on the Internet and follow the link to Mimna to access the file on the Internet.

C. Summary of My Opinion on Mimna

121. **EX-1007** is authentic because it contains no signs to raise suspicion about its authenticity. Furthermore, **EX-1007** is the same as the copy archived by Internet Archive (**Appx. 1007A**). These two copies show Mimna has a "January 30, 2017" presentation date.

122. The PFOA webpage on the Calgon Carbon website (**Appx. 1007B**) shows interested users could discover Mimna on February 22, 2017, and **Appx. 1007A** shows Mimna was publicly discoverable and accessible on September 18, 2017. Taken together, these two records inform my opinion that Mimna was publicly discoverable and accessible on the Internet as early as February 22, 2017, and an archived copy of Mimna became publicly accessible on Internet Archive on September 18, 2017

VII. PUBLIC AVAILABILITY OF APPLEMAN (EX-1009)

123. **EX-1009** is a true and correct copy of "Treatment of poly-and perfluoroalkyl substances in US full-scale water treatment systems," ("Appleman"), by Appleman et al., *Water research*, 51, 246-255, that I received from counsel. This copy is presented as **EX-1009** in this declaration.

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124. Page 1 shows the title, authors, eight keywords, and abstract, and indicates this article appears in "Water Research 51 (2014) 246-255" and is available on ScienceDirect and the journal homepage at www.elsevier.com/locate/watres. The article history on this page shows the revised manuscript was accepted on "27 October 2013" and became available online on "7 November 2013." The bottom of this page shows "0043-1354" as the ISSN (International Standard Serial Number) of the journal *Water Research*, "http://dx.doi.org/10.1016/j.watres.2013.10.067" as the article's digital object identifier, "2013" as the copyright date of this article, and Elsevier as the copyright holder. **EX-1009** shows Appleman has a total of 10 pages, including two figures, two tables, and 52 references.

A. Copy and Records of the National Library of Medicine

125. To determine the public availability of Appleman, I searched WorldCat.org, the free web portal to library collections, and learned that the National Library of Medicine ("NLM") held the journal containing Appleman. I then obtained a copy of this article from NLM via a document vendor. This library copy is presented as **Appx. 1009A** in this declaration.

126. Page 1 of **Appx. 1009A** is the issue cover that shows *Water Research* is a journal of the International Water Association published by Elsevier and the journal's ISSN is 0043-1354. This page identifies the issue as "15 March 2014" and a library label shows this issue is "Water research. V. 51 (Mar. 15 2014)" that is in

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the General Collection of the library with a call number of "W1 WA692A" and the library received this issue on "2014-02-25." This date means the journal issue containing Appleman was received by NLM on February 25, 2014. A customary library processing practice is to process a newly received issue promptly after it has gone through the serial check-in process, usually within a week. It is therefore my opinion that, after processing, vol. 51 would become publicly accessible by March 4, 2014.

127. Pages 4 to 6 are the table of contents of vol. 51 and page 5 shows Appleman begins on page 246. Pages 7 to 16 are the full text of Appleman. I have closely compared **EX-1009** with the NLM copy (**Appx. 1009A**) and found them to have the same content for Appleman.

128. As evidence that the journal containing Appleman is discoverable at NLM, I obtained NLM's records for this journal. **Appx. 1009B** is a true and correct copy of the NLM records for *Water Research*. I personally located, identified, and obtained these records. They are the type of record experts in my field would reasonably rely upon when forming their opinions.

129. Pages 1 and 2 are the bibliographic record that shows the journal title, corporate creator, publisher, country of publication, related titles, language, frequency, ISBNs, medical subject headings, and notes. The title, corporate creator, ISBN match the information presented in **EX-1009A**, confirming this serial record

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represents the journal containing Appleman. The record shows full text of this journal is available online in "Elsevier ScienceDirect Journals Complete" and the Location Items field shows NLM holds vol 1 to vol. 147 of the print journal.

130. Page 4 is the MARC record, and the first six digits of Field 008 show the record was created on "670620" (*i.e.*, June 20, 1067). Subfields "a" and "c" of Field 040 show DNLM created the record and entered it into the OCLC WorldCat database. According to the MARC Code List for Organizations (https://www.loc.gov/marc/organizations/org-search.php), DNLM is the code for the US National Library of Medicine. Fields 008 and 040 inform my opinion that the National Library of Medicine created this original serial record in June 1967, making the journal discoverable in the library since then.

131. Field 022 shows the ISSNs for the print and electronic versions of the journal. Field 245 shows the title, Field 246 shows a variant title, Field 260 shows the publisher, Field 362 shows the journal began with volume 1 in January 1967, and Field 310 shows the publication frequency since 2002 has been "Twenty no. a year." Field 500 notes the parent organization of this journal. Field 510 show MEDLINE and PubMed have indexed this journal since January 2001, and Index Medicus indexes this journal as well. Field 710 shows the International Association on Water Pollution Research is included to help users discover this journal.

132. The subjects of this journal are represented in Field 060 by a National

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Library of Medicine Classification Number of "W1" that represents "Serials. Periodicals" in General Medicine (National Library of Medicine Classification Schedule W, https://classification.nlm.nih.gov/schedules/w); and in Field 650 by a medical subject heading, "Water Pollution," and in Field 655 that indicates this publication is a "Periodical."

133. The NLM MARC record has made *Water Research* discoverable in their catalog since 1967. Interested users could discover this journal by ISSNs, title, variant title, and parent organization of the journal. They could also search for this journal by a subject term such as "Water Pollution" and browse for the journal by the W1 classification number.

B. Copyright Registration Record

134. **Appx. 1009C** is a true and correct copy of the copyright registration record for "no. 51, 15 March 2014" of *Water Research*. I personally located, identified, and obtained this record from the Public Records System of the US Copyright Office at https://publicrecords.copyright.gov/detailed-record/voyager_27205734. This is the type of record experts in my field would reasonably rely upon when forming their opinions.

135. The title, copyright claimant, ISSN, and publication frequency match the information presented in the NLM copy of Appleman (**Appx. 1009A**) and the NLM records (**Appx. 1009B**). The "Issues Registered" field shows no. 51 that has a

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"15 March 2014" issue date was published on "2014-02-10" (*i.e.*, February 10, 2014) and registered with the Copyright Office on "2014-10-29" (*i.e.*, October 29, 2014). This record informs my opinion that the journal volume containing Appleman was published on February 10, 2014.

C. PubMed Metadata Record

136. Since the NLM records (**Appx. 1009B**) indicate *Water Research* has been indexed by PubMed, I obtained the PubMed record for Appleman to determine when it was indexed. PubMed is an authoritative system that contains "more than 40 million citations for biomedical literature from MEDLINE, life science journals, and online books" and PubMed metadata records may "include links to full text content from PubMed Central and publisher web sites" (source: https://pubmed.ncbi.nlm.nih.gov).

137. **Appx. 1009D** is a true and correct copy of the PubMed metadata record for Appleman that I personally located, identified, and obtained from https://pubmed.ncbi.nlm.nih.gov/24275109/. This is the type of record experts in my field would reasonably rely upon when forming their opinions. I saved the metadata record in PDF and added the PubMed format of the record to show the record creation date and other data by data fields.

138. Page 1 of **Appx. 1009D** shows the title, authors, and abstract of Appleman, and identifies this article with a note of "Water Res. 2014 Mar 15:51:246-

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55. Doi: 10.1016/j.watres.2013.10.067. Epub 2013 Nov 7" that indicates this article was published online on November 7, 2013, and included in vol. 51 (March 15, 2014) of the print journal later. The top of the record shows a full text link to "Elsevier Full-Text Article" that leads to Elsevier's webpage for this article at https://www.sciencedirect.com/science/article/abs/pii/S0043135413008932?via%3Dihub where users can access the full text if their institutions subscribe to this journal. The metadata record shows "24275109" as the article's PMID (PubMed identifier). The title, authors, publication source, abstract, and keywords in this PubMed metadata record match the information contained in **EX-1009**, confirming this metadata record represents Appleman.

139. Pages 3 to 7 are the PubMed metadata record saved in the PubMed format to show the data elements in specific fields. I use a copy of the "MEDLINE/PubMed Data Element (Field) Descriptions" (https://www.nlm.nih.gov/bsd/mms/medlineelements.html) archived by Internet Archive on January 27, 2013 (available at https://web.archive.org/web/20130127130628/https://www.nlm.nih.gov/bsd/mms/medlineelements.html) to interpret this PubMed metadata record with data fields.

140. The "PMID" field on page 3 shows "24275109" as the PubMed identifier for the metadata record for Appleman. The "STAT" field shows the status of the record is "MEDLINE" meaning this record is a true MEDLINE record. The

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DCOM (Date Completed) field shows the index record was last processed on "20140929" (*i.e.*, September 29, 2014).

141. The IS field for "Linking" shows the ISSN for the print journal is "0043-1354." Fields VI (volume), DP (date of publication), Ti (title), PG (pagination), and LID (Location Identifier) show Appleman was published from pages 246 to 55 in vol. 51 that has an issue date of "2014 Mar 15" and its digital identifier is "10.1016/j.watres.2013.10.067." The first FAU (full author) field shows "Appleman, Timothy D" as the first author. The JT (journal title) field shows Appleman appears in "Water research." The EDAT field on page 6 shows an "Entrez Date" that is the date the record was added to PubMed, and it shows the Appleman record was entered into PubMed on 2013/11/28 (*i.e.*, November 28, 2013), making this article discoverable in PubMed on that date. The MHDA field (MeSH Date) shows the date when medical subject headings are added to a PubMed record, and the field on page 6 shows ten medical subject headings were added to the Appleman record on 2014/09/30 (*i.e.*, September 30, 2014).

142. This PubMed metadata record (**Appx. 1009D**) informs my opinion that Appleman was published online on November 7, 2013, and became discoverable in PubMed on November 28, 2013. As of that date, interested users could discover this record by authors, title, ISSN, journal title, abstract, and author keywords, and follow the link in the record to access the full text of Appleman on the publisher's website.

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After medical subject headings were added to this metadata record on September 30, 2014, interested users could use these subject terms to discover Appleman.

143. As evidence that PubMed was in operation when the PubMed metadata record for Appleman was entered into PubMed on November 28, 2013, I searched Internet Archive for records of PubMed. **Appx. 1009E** is a true and correct copy of the PubMed webpage archived by Internet Archive. I personally located, identified, and obtained this archived copy. This is the type of record experts in my field would reasonably rely upon when forming their opinions.

144. Page 1 of **Appx. 1009E** is a screen capture of a Wayback Machine calendar that shows this webpage of PubMed was archived 81,840 times between 1997 and 2026, and this particular copy was archived on "OCT 10 2013" and available at https://web.archive.org/web/20131010054145/http://www.ncbi.nlm.nih.gov/pubmed. Pages 2 and 3 are the PDF of this archived copy. **Appx. 1009E** shows PubMed was in operation in October 2013, and users could use it to discover the metadata record for Appleman and use it to access the full text of this article when the metadata record for Appleman was added to PubMed on November 28, 2013.

D. Internet Archive record

145. **Appx. 1009F** is a true and correct copy of the webpage for vol. 51 of *Water Research* archived by Internet Archive. I personally located, identified, and

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obtained. This is the type of record experts in my field would reasonably rely upon when forming their opinions.

146. Page 1 is a screen capture of a Wayback Machine calendar that shows this webpage was archived seven times between "22 Jan 2014" and "20 JUL 2015" and this particular copy was archived on "APR 06 2014" and available at https://web.archive.org/web/20140406113630/http://www.sciencedirect.com/science/journal/00431354/51. Pages 2 to 9 are the PDF of this archived copy, and page 8 shows Appleman, as entry #26, was available for online purchase on April 6, 2014.

E. Usage Records

147. The usage of a publication is reflected by other publications that make reference to it. I have reviewed the citation history of Appleman on Google Scholar to compile a list of early citations. **Appx. 1009G** is a list of five citations published in 2015 that shows the earliest citation appeared in April 2015.

F. Summary of My Opinion on Appleman

148. The date stamp on the copy of Appleman from the National Library of Medicine (**Appx. 1009A**) shows **EX-1009** was received on February 25, 2014, and, after processing, became publicly available at this library by March 4, 2014. NLM records (**Appx. 1009B**) show this journal was discoverable in the catalog of the library in 1967. The copyright registration record for vol. 51 of *Water Research* containing Appleman shows the issue was published on February 10, 2014. PubMed

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metadata record for Appleman (**Appx. 1009D**) shows that after this article was published online on November 7, 2013, the metadata record for Appleman was entered into PubMed on November 28, 2013, making it discoverable. As of that date, interested users could discover this article by its title, authors, abstract, author keywords and digital object identifier. The PubMed metadata record was enriched with ten medical subject headings on September 30, 2014, making it possible for interested users to use medical subject terms to discover Appleman. The archived webpage for vol. 51 of Water Research (**Appx. 1009F**) shows Appleman was available for online purchase on April 6, 2014. Citation records (**Appx. 1009G**) show the earliest citation appeared in April 2015.

149. Based on the documents and records I have reviewed and analyzed, it is my opinion that Appleman was published online on November 7, 2013, and appeared in a print issue that has a March 2014 issue date. This article became publicly discoverable on PubMed on November 28, 2013. It became publicly accessible at the National Library of Medicine on March 4, 2014. In addition, the article became available for online purchase from the journal website on April 6, 2014.

VIII. AUTHENTICITY AND PUBLIC AVAILABILITY OF CANNON (EX-1011)

A. Authentication

150. **EX-1011** is a true and correct copy of "Effect of Calcium in field-spent

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GACs on pore development during regeneration," "Cannon," by Cannon et al., *Journal American Water Works Association*, *85*(3), 76-89, that I received from counsel. This copy is presented as **EX-1011** in this declaration.

151. Page 1 shows the title, authors, abstract, a note that Cannon is published in "85#3 Journal – American Water Works Association, March 1993," and a "1993" copyright date for this article with the American Water Works Association as the copyright holder. **EX-1011** shows Cannon has a total of 14 pages, including 16 figures, three tables, and 49 references.

152. To authenticate **EX-1011**, I searched WorldCat.org, the free web portal, and learned that the Catholic University of America Libraries held the journal containing Cannon. I then searched the online catalog of this library system, located the record for Cannon, and followed the link to obtain a copy of this article from JSTOR. This JSTOR copy is presented as **Appx. 1011A** in this declaration. Page 1 of **Appx. 1011A** is a cover sheet accompanying the downloaded copy that shows the title, authors, publisher ("Wiley"), and URL (https://www.jstor.rg/stable/41294338), and indicates Cannon is published as pages 76-89 in "*Journal AWWA*, March 1993, Vol. 85, No. 3" that has a theme of "Drinking Water and Health: Balancing Risks" and an issue date of "March 1993." These details match the information presented on the publisher webpage for vol. 85, issue 3 of *Journal AWWA* at https://awwa.onlinelibrary.wiley.com/toc/15518833/1993/85/3. This Wiley webpage

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is presented as **Appx. 1011B**. I have closely compared **EX-1011** with **Appx. 1011A** and found them to have the same content for Cannon. The differences are that **EX-1011** includes a note that indicates the journal issue contains Cannon, a copyright date and copyright holder.

153. **EX-1011** is in a condition that raises no suspicion about its authenticity. Specifically, this copy is not missing any intermediate pages, and there are no visible alterations to the document. Furthermore, this copy has the same content as the publisher copy archived by JSTOR (**Appx. 1011A**), and the publisher webpage for vol. 85, issue 3 (**Appx. 1011B**) confirms Cannon is included in this issue. Based on my review and analysis, it is my opinion that **EX-1011** is authentic.

B. Record of the Catholic University of America Libraries

154. **Appx. 1011C** is a true and correct copy of the index record for Cannon. I personally located, identified, and obtained this record at https://wrlc-cu.primo.exlibrisgroup.com/permalink/01WRLC_CAA/11ramo0/cdi_crossref_primary_10_1002_j_1551_8833_1993_tb05959_x from the online catalog of the Catholic University of America Libraries. This is the type of record experts in my field would reasonably rely upon when forming their opinions.

155. Pages 1 to 3 are the index record for Cannon. Page 1 shows a brief record of Cannon, including the article title, authors, and source of publication. The View Online area shows the full text of this article is available in "JSTOR Archival

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Journals and Primary Sources Collection." The notes also show the coverage of the archival collection for this journal. Record details for Cannon include the title, creators, journal issue ("Journal – American Water Works Association, 1993-03, Vol. 85(3), p. 76-89"), subjects, keywords, abstract, publisher, language, source ("JSTOR Archival Journals and Primary Sources Collection," ISSN for the print journal ("0003-150X"), ISSN for the electronic journal ("1551-8833"), digital object identifier ("10.1002/j.1551-8833.1993.tb05959.x"), and "JAWWAS" as the CODEN (a six-character alphanumeric code that uniquely identifies a journal). The Links area shows links for users to "Get PDF" or "Read Online."

156. The "Read Online" link leads to the JSTOR record for Cannon. A screen capture of the top of this record is included as page 4 in **Appx. 1011C**. This screen capture shows the archived article is from the publisher Wiley. It also shows the title, authors, journal issue and pages, a URL for the archived article (https://www.jstor.org/stable/41294338), a link for download, and a display area for interested users to read this article online.

157. In the early 1990s university and colleges were running out of space for their print journal collections, so JSTOR (short for Journal Storage) was launched to digitize journal backfiles to help libraries save physical space. JSTOR Archival Journal Collections serve as a "trusted source for curated, high-quality, full-run journal collections" and all journals "include the full archival run" (JSTOR overview,

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https://about.jstor.org/products/journals-and-primary-sources/).

158. As evidence that JSTOR's archive collection of *Journal AWWA* includes a full run, I obtained the journal landing page at https://www.jstor.org/journal/jamewatworass. **Appx. 1011D** is a true and correct copy of the journal page that shows the archive collection covers "1914-2020 (Vol. 1, No. 1 – Vol. 112, No. 12)." Page 2 shows the publisher, ISSNs of the print and electronic versions of the journal, subjects, and "Arts & Sciences X Collection" as the archival collection that includes this journal.

159. To determine when JSTOR began providing access to the archival collection of *Journal AWWA*, I reviewed the JSTOR Global Arts & Sciences X collection title list at https://www.jstor.org/kbart/collections/asx?fileFormat=xlsx and learned that the release date for this archival collection was "2012-05-18" (*i.e.*, May 18, 2012). I copied the record for *Journal AWWA* from this list (a spreadsheet) to produce a PDF. This PDF record is included as pages 4 to 8 in **Appx. 1011D**. The archive release date is on page 8. **Appx. 1011D** informs my opinion that the archival collection of *Journal AWWA* includes issues from 1914 to 2020, and JSTOR released this collection on May 18, 2012, meaning interested users would be able to access vol. 85, issue 3 (March 1993) that contains Cannon in this archival collection on May 18, 2012.

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C. Copyright Registration Record

160. **Appx. 1011E** is a true and correct copy of the copyright registration record for vol. 85, no. 3 of *Journal American Water Works Association*. I personally located, identified, and obtained this record at https://publicrecords.copyright.gov/detailed-record/voyager_19968847 from the Public Records System of the US Copyright Office. This is the type of record experts in my field would reasonably rely upon when forming their opinions.

161. The title, ISSN, and serial publication year ("1993") show this record is for issues of *Journal American Water Works Association* published in 1993. The Issues Registered field shows "v. 85, no. 3, Mar93" has a publication date of "1993-03-08" (*i.e.*, March 8, 1993), and a copyright registration date of "1993-03-16" (*i.e.*, March 16, 1993).

D. Usage Records

162. The usage of a publication is reflected by other publications that make reference to it. I have reviewed the citation history of Cannon on Google Scholar to compile a list of citations. **Appx. 1011F** presents a list of five citations published from 1995 to 1997 and shows the earliest citation appeared in 1995.

E. Summary of My Opinion on Cannon

163. **EX-1011** is authentic because it contains no signs to raise suspicion about its authenticity. Furthermore, **EX-1011** has the same content for Cannon as the publisher copy archived by JSTOR (**Appx. 1011A**), and the publisher webpage for

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vol. 85, issue 3 (**Appx. 1011B**) confirms Cannon was published in this issue in March 1993.

164. The index record for Cannon from the Catholic University of America (**Appx. 1011C**) shows that after locating this record, interested users can follow the links to obtain a PDF of the full text of Cannon or read the article on the JSTOR platform. JSTOR records (**Appx. 1011D**) show the archival collection for Journal AWWA includes a full run of the journal minus the "moving wall" period (i.e., the embargo period of five years), and that JSTOR released the archival collection on May 18, 2012, meaning interested users would be able to access this archival collection on that date, including vol. 85, no. 3 that contains Cannon.

165. Copyright registration record for the journal issue containing Cannon (**Appx. 1011E**) shows the issue has a publication date of March 8, 1993. Citation records (**Appx. 1011F**) show at least five citations were published from 1995 to 1997, and the earliest citation appeared in 1995.

166. Based on the documents and records I have reviewed and analyzed, it is my opinion that Cannon became publicly discoverable and accessible soon after March 8, 1993 when the journal issue containing it was published. Citation history shows actual usage took place no later than 1995; and JSTOR archival collection of *Journal AWWA* became available on May 18, 2012 to users of libraries that subscribed to this journal. As of May 18, 2012, vol. 85, no. 3 (March 1993) containing

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Cannon became available on JSTOR in the archival collection for *Journal AWWA*.

IX. AUTHENTICITY AND PUBLIC AVAILABILITY OF MCNAMARA (EX-1013)

A. Authentication

167. **EX-1013** is a true and correct copy of "Comparison of activated carbons for removal of perfluorinated compounds from drinking water," ("McNamara"), by McNamara et al., *Journal American Water Works Association*, 110(1), E2-E14, that I received from counsel. This copy is presented as **EX-1013** in this declaration.

168. Page 1 of **EX-1013** shows the title, authors, abstract, and four keywords, and indicates this document is a peer reviewed article published in "January 2018. 110:1" of *Journal AWWA* and the pages are numbered E2 to E14. **EX-1013** has a total of 13 pages, including seven tables, four color figures, three equations, 20 endnotes, and 29 references.

169. To authenticate **EX-1013**, I searched the online catalog of the Catholic University of America for McNamara and found a link to an archived copy in JSTOR. This archived copy is presented as **Appx. 1013A** in this declaration. Page 1 of **Appx. 1013A** is a cover sheet accompanying the downloaded copy that shows the title, authors, publisher ("Wiley"), URL (https://www.jstor.rg/stable/10.2307/26653669), and indicates McNamara is published as pages E2-E14 in "*Journal AWWA*, January 2018, Vol. 110, No. 1" that has an issue date of "January 2018." These details match

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the information presented on the publisher webpage for Vol. 110, Issue 1 of *Journal AWWA* at https://awwa.onlinelibrary.wiley.com/toc/15518833/2018/110/1. This Wiley webpage is presented as **Appx. 1013B**. I have closely compared **EX-1013** with **Appx. 1013A** and found them to have the same content for McNamara.

170. **EX-1013** is in a condition that raises no suspicion about its authenticity. Specifically, this copy is not missing any intermediate pages, and there are no visible alterations to the document. Furthermore, this copy has the same content as the publisher copy archived by JSTOR (**Appx. 1013A**), and the publisher webpage for Vol. 110, Issue 1 (**Appx. 1013B**) confirms McNamara is a peer reviewed article included in this issue. Based on my review and analysis, it is my opinion that **EX-1013** is authentic.

B. Record of the Catholic University of America Libraries

171. **Appx. 1013C** is a true and correct copy of the index record for McNamara. I personally located, identified, and obtained this record at https://wrlc-cu.primo.exlibrisgroup.com/permalink/01WRLC_CAA/11ramo0/cdi_proquest_miscellaneous_2718337756 from the online catalog of the Catholic University of America Libraries. This is the type of record experts in my field would reasonably rely upon when forming their opinions.

172. Pages 1 and 2 are the index record for McNamara. Page 1 shows a brief record for McNamara, including the article title, authors, and source of publication.

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The View Online area shows the full text of this article is available in "JSTOR Archival Journals and Primary Sources Collection." The notes also show the coverage of the archival collection for this journal. Record details for McNamara include the title, creators, journal issue ("Journal – American Water Works Association, 2018-01, Vol.110(1), p.E2-E14"), subjects, keywords, abstract, publisher, language, source ("JSTOR Archival Journals and Primary Sources Collection," ISSN for the print journal ("0003-150X"), ISSN for the electronic journal ("1551-8833"), digital object identifier ("10.5942/jawwa.2018.110.0003").

173. The link to view full text in "JSTOR Archival Journals and Primary Sources Collection" leads to the JSTOR record for McNamara. A screen capture of the top of this record is included as page 3 in **Appx. 1013C**. This screen capture shows the archived article is from the publisher Wiley. It also shows the title, authors, journal issue and pagination, a URL for the archived article (https://www.jstor.org/stable/26653669), a link for download, and a display area for interested users to read this article online.

C. Copyright Registration Record

174. **Appx. 1013D** is a true and correct copy of the copyright registration record for vol. 110, no. 1 of *Journal American Water Works Association*. I personally located, identified, and obtained this record at https://publicrecords.copyright.gov/detailed-record/voyager_30108947 from the

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Public Records System of the US Copyright Office. This is the type of record experts in my field would reasonably rely upon when forming their opinions.

175. The title, ISSN, and serial publication year ("2018") show this record is for issues of *Journal American Water Works Association* published in 2018. The Issues Registered field shows vol. 110, no. 1, January 2018 has a publication date of "2018-01-15" (*i.e.*, January 15, 2018), and a copyright registration date of "2018-03-05" (*i.e.*, March 5, 2018).

D. Records of the Johns Hopkins University Libraries

176. To determine the public availability date of McNamara at libraries, I searched WorldCat.org for libraries holding the journal containing this article and learned that the Johns Hopkins University Libraries held the print and online versions of *Journal AWWA*. I then obtained records from this library system. **Appx. 1013E** is a true and correct copy of the bibliographic and MARC records for *Journal AWWA* that I personally located, identified, and obtained. They are the type of records experts in my field would reasonably rely upon when forming their opinions.

177. Pages 1 to 3 are the bibliographic record that shows the print journal is stored offsite with a call number of "TD 201 .A51." Record details include the journal title, creator, subject, MESH subjects, genre, other title, related title, publisher, date, frequency, LC number, ISSN ("0003-150X"), and general notes.

178. Pages 4 and 5 are the MARC record. The first six digits of Field 008

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show the record was created on "860207" (*i.e.*, February 7, 1986), and the code of "c19489999" following the record creation date indicates this publication is a continuing resource that began publication in 1948 and has no pre-determined conclusion date. Subfields "a" and "c" of Field 040 show DLC created the original serial record and entered it into the OCLC WorldCat database. According to the Directory of OCLC Members (https://www.oclc.org/en/contacts/libraries.html), DLC is the OCLC symbol for the Library of Congress. Field 005 is a machine generated field that automatically records the date and time of the latest update to this record, and it shows the latest update date was "20180119150900" (*i.e.*, January 19, 2018, at 3 p.m., 9 minutes, 0 second). Field 946 is a note with a date of "1/19/2018" (*i.e.*, January 19, 2018) that indicates "Cancel print; online coming via our Wiley package" on that date, meaning the library system canceled the print journal and would provide future access to this journal via their online subscription with Wiley. The date of this note matches the last update date in Field 005. Fields 008, 040, 005 and 946 inform my opinion that the Johns Hopkins University library system used the record created by the Library of Congress to create their copy cataloging record, and last updated their copy cataloging record on January 19, 2018 when they canceled the print version and switched to the electronic version of the journal.

179. Field 022 shows the ISSN of the print journal, Field 245 shows the title, Field 246 shows a variant title, and Field 260 shows the publisher since 1948. Field

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362 shows the journal began with vol. 40, no. 1 in January 1948, and Field 310 shows the publication frequency is "Monthly." Field 500 explains that the publisher information has changed since 1985. Field 710 shows the American Water Works Association is included to help users discover this journal.

180. The subject of this print journal is represented in Field 050 by a Library of Congress Classification number of "TD201" that represents English periodicals for the subject of "Water supply for domestic and industrial purposes" (Library of Congress Classification Schedule T, https://www.loc.gov/aba/publications/FreeLCC/LCC_T2019TEXT.pdf); and in Field 650 by a Library of Congress subject heading with "Water-supply engineering" as the main heading, followed by "Periodicals" as a form heading encoded in subfield "v". These subject representations enable users to conduct subject searches to discover this journal.

181. The MARC record (**Appx. 1013E**) shows the Johns Hopkins University library system created a copy cataloging record for the print version of *Journal AWWA* and made this journal discoverable in their library catalog. Users could search for this journal by ISSNs, title, variant title, and the parent organization of the journal. On January 19, 2018, the library system canceled the print subscription and provided access to this journal via their Wiley online package from then on. This change to online access is reflected in the library record for McNamara (**Appx. 1013F**) that I

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obtained from the online catalog of the library system at https://catalyst.library.jhu.edu/permalink/01JHU_INST/62los8/cdi_proquest_miscellaneous_2718337756.

182. Page 1 shows the title, authors, abstract, source of publication for McNamara and indicates the library holds the print and online versions of *Journal – American Water Works Association*. The "View Online" area shows full text is available from JSTOR Archival Journals and Primary Sources Collection (the entire run of the journal from 1914, except for the most recent six years), Wiley Online Library Database Model 2026 (coverage from 1997), and Wiley Online Library Journals (coverage from 1997). The record also shows older print issues are stored offsite. Record details include the title, authors, source of publication, subject keywords, abstract, publisher, ISSNs and the digital object identifier for McNamara. Interested users can access the full text by following the links to "Get PDF" or "Read Online."

183. Taken together, the journal records (**Appx. 1013E**) and the record for McNamara (**Appx. 1013F**) inform my opinion that the Johns Hopkins University library system subscribed to the print version of *Journal American Water Works Association*, and changed their subscription format from print to online on January 19, 2018. From that date on, interested users would be able to access full text of this journal from JSTOR and Wiley online services.

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184. Electronic journal publishers provide access to new issues based on a set schedule of a journal or a continuous rolling model, making new articles available as soon as they are ready. Since the publication frequency of *Journal AWWA* is monthly, it is my opinion that the publisher Wiley would make vol. 110, no. 1 (January 2018) available to users in January 2018, and definitely no later than February 2018. This means that McNamara became publicly discoverable and accessible no later than February 2018 at the Johns Hopkins University Libraries.

E. Summary of My Opinion on McNamara

185. **EX-1013** is authentic because it contains no signs to raise suspicion about its authenticity. Furthermore, **EX-1013** has the same content for McNamara as the publisher copy archived by JSTOR (**Appx. 1013A**), and the publisher webpage for vol. 110, issue 1 (**Appx. 1013B**) confirms McNamara was published in this issue in January 2018.

186. Copyright registration record for the journal issue containing McNamara (**Appx. 1013C**) shows the issue has a publication date of January 15, 2018. The journal records from the Johns Hopkins University Libraries (**Appx. 1013D**) show the library holdings began with vol. 40, no. 1 (January 1948) and the library system changed their subscription to this journal from print to electronic on January 19, 2018, meaning access to this journal since that date has been online. Since electronic journal publishers provide access to new issues on an established schedule

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of a journal or on a rolling model such as daily update, it is my opinion that after the journal issue containing McNamara was published on January 15, 2018, interested users at the Johns Hopkins University Libraries would be able to access this issue (and McNamara contained therein) online no later than February 2018.

187. Based on the documents and records I have reviewed and analyzed, it is my opinion that users of the Johns Hopkins University Libraries would be able to discover and access McNamara online from Wiley no later than February 2018.

X. AUTHENTICITY AND PUBLIC AVAILABILITY OF RS FAQ (EX-1014)

A. Authentication

188. **EX-1014** is a true and correct copy of "Reactivation Services Frequently Asked Questions," ("RS FAQ"), from Carlgon Carbon that I received from counsel. This document addresses seven questions: How is spent carbon reactivated? How are the emissions from the reactivation process treated? Will reactivated carbon work for my application? What is the advantage of reactivating spent carbon? Is reactivated carbon suitable for use in drinking water? If spent carbon is returned, can it be replaced with virgin carbon? Can hazardous spent carbons be returned for reactivation? The bottom of page 2 shows the contact information of Carlgon Carbon and their website address (www.calgoncarbon.com). This copy is presented as **EX-1014** in this declaration.

189. To authenticate **EX-1014**, I searched Internet Archive for copies of this

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document. **Appx. 1014A** is a true and correct copy of RS FAQ. I personally located, identified, and obtained this copy from Internet Archive. This is the type of material experts in my field would reasonably rely upon when forming their opinions.

190. Page 1 of **Appx. 1014A** is a screen capture of a Wayback Machine calendar that shows this webpage (http://www.calgoncarbon.com/wp-content/uploads/2015/02/Reactivation_Services_FAQ.pdf) was archived once by Internet Archive on "APR 19 2016" and available at https://web.archive.org/web/20160419140003/http://www.calgoncarbon.com/wp-content/uploads/2015/02/Reactivation_Services_FAQ.pdf. Pages 2 and 3 are the PDF of this archived copy that shows this document addresses seven questions. I have closely compared **EX-1014** with this archived copy and found them to be the same.

191. **EX-1014** is in a condition that raises no suspicion about its authenticity. Specifically, this text flows clearly, and there are no visible alterations to the document. Furthermore, this copy is the same as the copy archived by Internet Archive (**Appx. 1014A**). Based on my review and analysis, it is my opinion that **EX-1014** is authentic.

B. Discovery of RS FAQ

192. The archive date of **Appx. 1014A** shows that an archived copy of RS FAQ became publicly discoverable and accessible on Internet Archive on April 19, 2016. The discovery of this archived copy of RS FAQ is straightforward. **Appx.**

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1014B and **Appx. 1014C** are true and correct copies of Calgon Carbon webpages that I consulted to locate RS FAQ. These webpages are the type of material experts in my field would reasonably rely upon when forming their opinions.

193. Page 1 of **Appx. 1014B** is a screen capture of a Wayback Machine calendar that shows the Calgon Carbon website landing page at http://www.calgoncarbon.com has been archived 1,001 times between 1997 and 2026, and this particular copy was archived on "JUL 05 2015" and available at https://web.archive.org/web/20150705234254/http://www.calgoncarbon.com/. Pages 2 to 5 are the PDF of this archived copy that shows the services, products, expertise, and news about the company.

194. Page 5 is a screen capture of the Products menu, archived on July 5, 2015, that shows links to product subject areas, and the second link on the menu is "Reactivated Services." This link leads to the webpage for Reactivation Services. Page 7 is a screen capture of an archived webpage for Reactivation Services. It shows the webpage at http://www.calgoncarbon.com/reactivation-services/ was archived 65 times between "5 Jul 2015" and "21 May 2026" and available at https://web.archive.org/web/20150705031533/http://www.calgoncarbon.com/reactivation-services/.

195. **Appx. 1014C** is a true and correct copy of the webpage for Reactivation Services archived by Internet Archive on July 5, 2015. Page 1 is a screen capture of

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a Wayback Machine calendar. Pages 2 to 4 are the PDF of this archived webpage that describes the features and benefits of Calgon Carbon Reactivation Services and the application of such services in six areas. In addition, this webpage presents links to resources related to reactivation services, including "Reactivation Services Frequently Asked Questions." This link leads to an archived copy of RS FAQ. The screen capture presented as page 5 of **Appx. 1014C** shows this document was archived once by Internet Archive on "APR 19 2016." This archived RS FAQ is the same as the file presented as **Appx. 1014A** above.

196. Taken together, **Appx. 1014B** and **Appx. 1014C** show that the Calgon Carbon website and their webpage for Reactivation Services were on the Internet by July 5, 2015. By July 2015 interested users could use search engines such as Google or Bing to locate these web resources on the Internet. Since the archived webpage for Reactivation Services included a link to RS FAQ, it is my opinion that Calgon Carbon intended to make RS FAQ available to interested users on the Internet no later than July 2015. This means that when interested users followed the link to RS FAQ on the Reactivation Services webpage on the Internet in July 2015, they would be able to access RS FAQ.

197. As for discovery of RS FAQ on Internet Archive, interested users would be able to find archived copies of the Calgon Carbon website and their webpage for Reactivation Services on July 5, 2015, but they might not be able to find

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the archived copy of RS FAQ through the FAQ link in the archived webpage for Reactivation Services, because RS FAQ was not archived by Internet Archive until April 19, 2016. Nevertheless, interested users in July 2015 had an option to find RS FAQ on the Internet, as described above.

C. Summary of My Opinion on RS FAQ

198. **EX-1014** is authentic because it contains no signs to raise suspicion about its authenticity. Furthermore, **EX-1014** is identical to an archived copy on Internet Archive (**Appx. 1014A**).

199. RS FAQ was archived by Internet Archive (**Appx. 1014A**) once on April 19, 2016, meaning interested users would be able to discover and access this archived copy of RS FAQ no later than April 19, 2016.

200. The public availability of RS FAQ on the Internet was, in fact, much earlier. A web file has to appear on the Internet before it can be archived. Copies of the Calgon Carbon website and their webpage for Reactivation Services archived on July 5, 2015 (**Appxs. 1014B** and **1014C**) mean that these web resources were available on the Internet by that date and interested users could use search engines like Google to discover these web resources. Since the Reactivation Services webpage archived on July 5, 2015 included a link to RS FAQ, interested users would be able to visit the Reactivation Services webpage on the company's website on the Internet and follow the RS FAQ link to access this document.

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201. Based on the documents and records I have reviewed and analyzed, it is my opinion that "Reactivation Services Frequently Asked Questions" became publicly available for user discovery and access on July 5, 2015, through the Calgon Carbon webpage for Reactivation Services. An archived copy of this document became available for public access on Internet Archive on April 19, 2016.

XI. SUMMARY OF FINDINGS

202. The public availability dates of the prior art documents discussed in this declaration are presented in the table below:

Prior Art Document	Earliest Public Availability Date
EX-1004 Kempisty, D. M., Xing, Y., & Racz, L. (Eds.). (2018). *Perfluoroalkyl Substances in the Environment: Theory, Practice, and Innovation*, Taylor & Francis Group, Environmental and Occupational Health Series, 2018.	August 26, 2018
EX-1005 Mueller, R., & Yingling, V. (2018, March). Remediation technologies and methods for per-and polyfluoroalkyl substances (PFAS). 12 pages.	March 20, 2018
EX-1006 McDougall, G. J. (1991, April). The physical nature and manufacture of activated carbon, *Journal of the Southern African Institute of Mining and Metallurgy*, 91(4), 109-120.	July 25, 1991
EX-1007 Mimna, R. (January 30, 2017). "Granular Activated Carbon: A Proven Solution for PFC Removal," Calgon Carbon Presentation, 30 Jan. 2017, https://www.calgoncarbon.com/app/uploads/PFC-Webinar-Presentation-1-30-17.pdf.	February 22, 2017
EX-1009 Appleman, T. D., Higgins, C. P., Quiñones, O., Vanderford, B. J., Kolstad, C., Zeigler-Holady, J. C., & Dickenson, E. R.	March 4, 2014

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(2014, March). Treatment of poly-and perfluoroalkyl substances in US full-scale water treatment systems, *Water Research*, 51, 246-255.	
EX-1011 Cannon, F. S., Snoeyink, V. L., Lee, R. G., Dagois, G., & DeWolfe, J. R. (1993, March). Effect of Calcium in field-spent GACs on pore development during regeneration, *Journal-American Water Works Association*, *85*(3), 76-89.	By 1995; archived copy available on May 18, 2012
EX-1013 McNamara, J. D., Franco, R., Mimna, R., & Zappa, L. (2018, January). Comparison of activated carbons for removal of perfluorinated compounds from drinking water, *Journal-American Water Works Association*, 110(1), E2-E14.	February 2018
EX-1014 Calgon Carbon. (2015). Reactivation Services Frequently Asked Questions. Counsel copy.	July 5, 2015

XII. Conclusion

203. In signing this Declaration, I recognize that the Declaration will be filed as evidence in a contested case before the Patent Trial and Appeal Board of the United States Patent and Trademark Office. I also recognize that I may be subject to cross-examination in this proceeding. If required, I will appear for cross-examination at the appropriate time.

204. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. § 1001.

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Dated: 8-21-2026

Respectfully submitted,

Ingrid Hsieh-Yee

Ingrid Hsieh-Yee, Ph.D.
Annapolis, Maryland

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Appendix A.

Ingrid Hsieh-Yee

Professor Emerita
Dept. of Library and Information Science
Catholic University of America
Washington, D.C. 20064
E-mail: hsiehyee@cua.edu

Education

Ph.D. 1990. Library and Information Studies, University of Wisconsin-Madison
Minors: Sociology and Psychology

M.A. 1984. Library and Information Studies, University of Wisconsin-Madison.

M.A. 1983. Comparative Literature, University of Wisconsin-Madison.

B.A. 1980. Foreign Languages and Literature, National Taiwan University.

Work Experience

Professor, School/Dept. of Library and Information Science, Catholic University of America, 1990-2022 (Assistant Professor, 1990-1996; Associate Professor, 1997-2004; Professor 2004-2022; Professor Emerita 2022)

Co-Chair, Dept. of Library and Information Science, Catholic University of America, June 2015-August 2016.

Acting Dean, School of Library and Information Science, Catholic University of America, January 2010-June 2012.

Cataloger, Dept. of Legislative Reference Library, Annapolis, Maryland, 1989-1990.

Lecturer, School of Library and Information Studies, University of Wisconsin-Madison, 1988.

Teaching Assistant, School of Library and Information Studies, University of Wisconsin-Madison, 1986-1988.

Cataloger, Health Sciences Library, University of Wisconsin-Madison, 1984-1986.

Areas of Teaching and Research Interests

Information Organization and Access; Metadata; Cataloging & Classification; Information Architecture; Information Retrieval; Digital Archives, Repositories & Libraries; Social Media;

Scholarly Communication; Information Behavior; Health Informatics; Human Computer Interaction; Usability Studies

Grants & Honors

Cultural Heritage Information Management Project. IMLS grant. Amount: $498,741. Period: Aug. 2012 to July 2015. Co-PI with Dr. Youngok Choi.

D.C. Health Information Technology (HIT4): Building Capacity & Providing Access in Our Nation's Capital. Dept. of Labor H2B Training Grant. Grant amount: $4,175,500. Grant period: Nov. 2011 to Dec. 2015. Partner with the Metropolitan School of Professional Studies of the Catholic University of America, Children's National Medical Center, D.C. Department of Employment Services, Holy Cross Hospital, Howard University, Center for Urban Progress, Providence Hospital, and Sibley Memorial Hospital.

Capital Health Careers Project. Department of Labor Healthcare Sector and Other High Growth and Emerging Industries Grant. Grant amount: $4,953,999. Grant period: March 2010 – February 2013. Awarded to a group of healthcare organizations and educational institutions in Washington, D.C. Providence Health Foundation of Providence Hospital (Lead institution). Part of the grant supported the development of a Master's degree program in Information Technology with a concentration in Health Information Technology offered by the School of Library and Information Science.

The Washington D.C. School Librarians Project. IMLS grant. Grant amount: $412,660. Grant period: Aug. 2007 – June 2011. The School partnered with the District of Columbia Public Schools (DCPS) and the District of Columbia Library Association to educate and mentor school media specialists for the DCPS system. PI, Jan. 2010 to June 2011.

SIG Member of the Year, American Society for Information Science and Technology (2009).

Most Outstanding Paper of *OCLC Systems & Services* (2001).

ALISE Research Grant (2001).

Most Outstanding Paper of *OCLC Systems & Services* (2000).

Research Grant from ERIC (1999-2000).

Best Research Paper Award; Association for Library and Information Science Education (1998).

Research Grants, Catholic University of America. 1991, 1992, 1993, 1996, 1998, 1999, 2004, 2005, 2006, 2007, 2013-14.

Cooperative Faculty Research Grant, Consortium of Universities in the Washington Metropolitan Area (1993-1994).

Cooperative Research Grant, Council on Library Resources (1993-1994).

Journal of the American Society for Information Science Best Paper Award (1993).

ASIS/ISI Information Science Doctoral Dissertation Scholarship (1989).

HEA Title IIB Fellowship (Dept. of Education) (1989)

Chinese-American Librarians Association Scholarship (1987).

Beta Phi Mu (1985).

Vilas Fellowship, University of Wisconsin-Madison. 1984

Publications

Kimura, M., & Hsieh-Yee, I. (2026?). Relationships Recording in Authority and Bibliographic Data: What is Recorded and How Much? Revised manuscript submitted to *Cataloging & Classification Quarterly* in July 2026.

Hsieh-Yee, I. (2021). Can We Trust Social Media? *Internet Reference Services Quarterly*, 1-15. Published online: 28 July, 2021. 10.1080/10875301.2021.1947433.

Bailey, T., & Hsieh-Yee, I. (2020). Combating the Sharing of False Information: History, Framework & Literacy Strategies, *Internet Reference Services Quarterly*, 24 (1-2), 9-30.

Choi, Y., and Hsieh-Yee, I. (2010). Finding Images in an OPAC: Analysis of User Queries, Subject Headings, and Description Notes. *Canadian Journal of Information and Library Science,* 34(3): 271 – 295.

Hsieh-Yee, I. (2008). Educating Cataloging Professionals in a Changing Information Environment. *Journal of Education for Library and Information Science,* 46(2): 93-106.

Vellucci, S. L., Hsieh-Yee, I., and Moen, W.E. (2007). The Metadata Education and Research Information Commons (MERIC): A Collaborative Teaching and Research Initiative. *Education for Information,* 25(3&4): 169-178.

NISO Framework for Guidelines for Building Good Digital Collections. 3rd ed. Baltimore, MD: National Information Standards Organization, 2007. Also available online: http://www.niso.org/framework/framework3.pdf (NISO Working Group members: Priscilla Caplan (chair), Grace Agnew, Murtha Baca, Tony Gill, Carl Fleischhauer, Ingrid Hsieh-Yee, Jill Koelling, and Christie Stephenson.)

Choi, Y., Hsieh-Yee, I., and Kules, B. (2007). Retrieval Effectiveness of TOC and LCSH. *Proceedings of the Joint Conference on Digital Libraries*, pp. 233-234.

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Mitchell, Vanessa, and Ingrid Hsieh-Yee. (2007). Converting Ulrich's Subject Headings to FAST Headings: A Feasibility Study. *Cataloging & Classification Quarterly,* 45(1): 59-85.

Hsieh-Yee, I., Tang, R., and Zhang, S. (2007). User Perceptions of a Federated Search System. *IEEE Technical Committee on Digital Libraries Bulletin,* Summer 3(2) (URL = http://www.ieee-tcdl.org).

Tang, R., Hsieh-Yee, I., and Zhang, S. (2007). User Perceptions of MetaLib Combined Search: An Investigation of How Users Make Sense of Federated Searching." *Internet Reference Services Quarterly,* 12(12): 211-236.

Hsieh-Yee, I., Tang, R., and Zhang, S. (2006). User Perceptions of a Federated Search System. *Proceedings of the Joint Conference on Digital Libraries*, *June 11-15, 2006, Chapel Hill*, p. 338.

Hsieh-Yee, I. (2006). *Organizing Audiovisual and Electronic Resources for Access: A Cataloging Guide*. 2nd ed. Westport, Conn.: Libraries Unlimited.

NISO A Framework of Guidance for Building Good Digital Collections. 2nd ed. Bethesda, MD: National Information Standards Organization, 2004. Framework Advisory Group: Grace Agnew, Liz Bishoff, Priscilla Caplan (Chair), Rebecca Gunther and Ingrid Hsieh-Yee.

Hsieh-Yee, I. (2004). Cataloging and Metadata Education in North American LIS Programs. *Library Resources & Technical Services,* 48(1): 59-68.

Hsieh-Yee, I. (2004). Cataloging and Metadata Education. In Gary E. Gorman (Ed.), *International Yearbook of Library and Information Management 2003: Metadata Applications and Management*, (pp.204-234). London: Facet Publishing.

Yee, P. L., Hsieh-Yee, I., Pierce, G.R., Grome, R., and Schantz, L. (2004). Self-Evaluative Intrusive Thoughts Impede Successful Searching on the Internet. *Computers in Human Behavior,* 20(1): 85-101.

Hsieh-Yee, I. (2003). Cataloging and Metadata Education: A Proposal for Preparing Cataloging Professionals of the 21st Century. A report submitted to the ALCTS-Education Task Force in response to Action Item 5.1 of the *Bibliographic control of Web Resources: A*

Library of Congress Action Plan. Approved by the Association for Library Collections and Technical Services. Web version available since April 2003 at http://lcweb.loc.gov/catdir/bibcontrol/CatalogingandMetadataEducation.pdf.

Hsieh-Yee, I. (2002). Cataloging and Metadata Education: Asserting a Central Role in Information Organization. *Cataloging & Classification Quarterly* 34(½): 203-222.

Hsieh-Yee, I., and Smith, M. (2001). The CORC Experience: Survey of Founding Libraries, Part I. *OCLC Systems & Services,* 17: 133-140. (Received "The Most Outstanding Paper of OCLC Systems & Services in 2001" award.)

Hsieh-Yee, I., and Smith, M. (2001). The CORC Experience: Survey of Founding Libraries, Part II, Automated Tools and Usage. *OCLC Systems & Services,* 17: 166-177. (Received "The Most Outstanding Paper of OCLC Systems & Services in 2001" award.)

Hsieh-Yee, I. (2001). ERIC User Services: Changes and Evaluation for the Future. *Government Information Quarterly,* 18: 31-42.

Hsieh-Yee, Ingrid. (2001). Research on Web Search Behavior. *Library and Information Science Research,* 23: 167-185.

Logan, E., and Hsieh-Yee, I. (2001). Library and Information Science Education in the Nineties. *Annual Review of Information Science and Technology,* 35: 425-477.

Hsieh-Yee, I. (Ed.) (2001). *Library and Information Science Research*, 23 (2). A special issue in honor of the retirement of Douglas L. Zweizig.

Hsieh-Yee, I. (2000). *ERIC User Services: Evaluation in a Decentralized Environment*. Washington, D.C.: Dept. of Education.

Hsieh-Yee, Ingrid. (2000). *Organizing Audiovisual and Electronic Resources for Access: A Cataloging Guide*. Littleton, CO: Libraries Unlimited.

Hsieh-Yee, I. (2000). Organizing Internet Resources: Teaching Cataloging Standards and Beyond. *OCLC Systems & Services,* 16: 130-143. (Received "The Most Outstanding Paper of OCLC Systems & Services in 2000" award.)

Hsieh-Yee, I. (1998). The Retrieval Power of Selected Search Engines: How Well Do They Address General Reference Questions and Subject Questions? *Reference Librarian,* 60: 27-47.

Hsieh-Yee, I. (1998). Search Tactics of Web Users in Searching for Texts, Graphics, Known Items and Subjects: A Search Simulation Study. *Reference Librarian,* 60: 61-85. (Received the 1997 Best ALISE Research Paper Award.)

Hsieh-Yee, I. (1997). Access to OCLC and Internet Resources: LIS Educators' Views and Teaching Practices. *RQ,* 36: 569-86.

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Hsieh-Yee, I. (1996). Modifying Cataloging Practice and OCLC Infrastructure for Effective Organization of Internet Resources. In *Proceedings of the OCLC Internet Cataloging Colloquium*. [Online]. Available: http://www.oclc.org/oclc/man/colloq/hsieh.htm

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Hsieh-Yee, I. (1995). Ten entries in James S. C. Hu (Ed.), *Encyclopedia of Library & Information Science*, 913, 1028-29, 1036, 1037, 1145-46, 1514, 1575, 1763-64, 2216-27, 2378-79. Taipei, Taiwan: Sino-American Publishing. (Topics include "Advanced Technology/Libraries," "Information Ethics," "Instruction on Cataloging and Classification," "Instruction on Reference Services.")

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Presentations

Bailey, T. & Hsieh-Yee, I. (February 2020). The Phenomena of Sharing Misinformation and the Need for Information Literacy. Presented at the 2020 Bridging the Spectrum Symposium, Washington, D.C., February 14, 2020.

Hsieh-Yee, I. and Fragan-Fly, J. (May 2018) Trends, Design & Strategies for Digital Scholarship Services. Presented at the 2018 Maryland/Delaware Library Association Conference, Cambridge, MD.

Hsieh-Yee, I. (February, 2018) Research Data Management: What It Takes to Succeed. Presented at the 10th Bridging the Spectrum Symposium, Washington, D.C.

Hsieh-Yee, I. (February, 2017) *Research Data Management: New Competencies and Opportunities for Information Professionals*. Presented at the 9th Bridging the Spectrum Symposium, Washington, D.C.

Hsieh-Yee, I. and Lawton, P. (February, 2017) *Enhancing Catholic Portal Searches with User Terms and LCSH*. Presented at the 9th Bridging the Spectrum Symposium, Washington, D.C.

Hsieh-Yee, I. (2016, October) *Visualizing Data for Information*. Presented at the 2016 Virginia Library Association Conference, Hot Springs, VA.

Hsieh-Yee, I. (2016, August) *Religious Materials Toolbox for Archivists: Solutions to Problems Facing the Profession*. Presented at Archives * Records 2016, Atlanta, GA.

Hsieh-Yee, I. and Lawton, P. (2016, March) *Enhancing Retrieval of Catholic Materials with LCSH Knowledge Structure*. Presented at the 2016 Catholic Library Association Conference, San Diego, CA.

Fagan-Fry, J. and Hsieh-Yee, I. (2016, February) *Approaches to Digital Scholarship at Top Universities around the World: Scholarly Publishing in the Digital Age.* Presented at the 8th Bridging the Spectrum Symposium, Washington, D.C.

Hsieh-Yee and Fagan-Fry, J. (2016, January) *Innovative Services for Digital Scholarship at Top 100 Research Libraries of the World*. Poster presented at the 2016 Annual Conference of the Association for Library and Information Science Education, Boston, Mass.

Hsieh-Yee, I. and Lawton, P. (2015, June). *Crowdsourcing terms for CRRA portal themes.* Poster presented at the third CRRA symposium and annual meeting, Bringing the created toward the Creator: Liturgical art and design since Vatican II. Catholic Theological Union, Chicago, Illinois.

Hsieh-Yee, I. and Lawton, P. (2015, February). *Crowdsourcing terms for thematic exploration in the Catholic Portal.* Poster presented at the 7th Annual Bridging the Spectrum Symposium, Washington, D.C.

Hsieh-Yee, I., James, R., and Fagan-Fry, J. (2015, February). *Support for digital scholarship at top university libraries of the world*. Poster presented at the 7th Annual Bridging the Spectrum Symposium, Washington, D.C.

Hsieh-Yee, I., Zhang, S., Lin, K., and Cherry, S. (2015, February). *Thus said the end users: Summon experience and support for research workflows*. Poster presented at the 7th Annual Bridging the Spectrum Symposium, Washington, D.C.

Yontz, E., Hsieh-Yee, I., & Houston, S. (2015, February). *Healthy Heroes Summer Reading Club: Developing healthy youth at public libraries.* 11th Annual Jean Mills Health Symposium, Greenville, North Carolina.

Yontz, E., Hsieh-Yee, I., and Houston, S. (2015, January). *Healthy youth and libraries: A pilot study.* Association for Library & Information Science Education (ALISE) Annual Conference, Chicago, Illinois.

Hsieh-Yee, I. (2014, May). *Linking CRRA resources to portal themes via authority files*. Presented at the Catholic Research Resources Alliance 2014 Membership Meeting, Marquette, WI.

Hsieh-Yee, I. (2014, April). *Enhancing subject access to CRRA resources*. Presented at the 2014 Catholic Library Association Conference, Pittsburgh, PA.

Hsieh-Yee, I. (2014, January). *Health Information Technology Program: Educational entrepreneurship in action*. Presented at the 2014 annual Conference of the Association for Library and Information Science Education, Philadelphia, PA

Hsieh-Yee, I., Zhang, S., Lin, K., and Cherry, S. (2014, January). *Discovering information through Summon: An analysis of user search strategies and search success*. Paper presented at the 6th Bridging the Spectrum Symposium, Washington, D.C.

Hsieh-Yee, I. (2012, December). *National Digital Stewardship Alliance and SLIS at CUA: An Educational Partnership*. Paper presented at Best Practices Exchange: Acquiring, Preserving, and Providing Access to Government Information in the Digital Era, Annapolis, MD

Choi, Y. and Hsieh-Yee, I. (2010, January). *Finding Images in an OPAC: Analysis of User Queries, Subject Headings, and Description Notes*. Paper presented at 2nd Annual Bridging the Spectrum Symposium, Catholic University of America, Washington, D.C.

Hsieh-Yee, I. and Coogan, J. (2010, January). *Google Scholar vs. Academic Search Premier: What Libraries and Searchers Need to Know*. Paper presented at 2nd Annual Bridging the Spectrum Symposium, Catholic University of America, Washington, D.C.

Hsieh-Yee, I. (2009, November). *Information Science Education: An LIS School's Perspective*. Paper presented at Annual Meeting of the American Society for Information Science and Technology, Vancouver, British Columbia, Canada.

Hsieh-Yee, I., Menard, E., Ya-Ning Chen, A., Shu-Jiun Chen, S., Kalfatovic, M. R., Wisser. K. M. (2009, November). *Information Organization in Libraries, Archives and Museums: Converging Practices and Collaboration Opportunities*. Presented at Annual Meeting of the American Society for Information Science and Technology, Vancouver, British Columbia, Canada. (Organizer and moderator of this panel.)

Hsieh-Yee, I. and Coogan, J. (2009, July). *Catching up to Google Scholar: The Retrieval Power of Academic Search Premier and Google Scholar*. Poster presented at American Library Association Conference, Chicago, Illinois.

Hsieh-Yee, I., with the CUA Scholarly Communications Project Team. (2009, January). *Digital Scholarship@CUA: Developing an Institutional Repository for CUA*. Poster presented at 1st Annual Bridging the Spectrum Symposium, Catholic University of America, Washington, D.C.

Wise, M., Cylke, K., and Hsieh-Yee, I. **(**2009, January). *Digital Talking Books: Meeting the Needs of the Blind and the Handicapped*. Paper presented at the Bridging the Spectrum Symposium, Catholic University of America, Washington, D.C.

Hsieh-Yee, I. (2009, January). *User Expectations of MERIC*. Presented at the Information Organization Competencies for the 21st Century Discussion Session of the 2009 Conference of the Association for Library and Information Science Education, Denver, Colorado.

Choi, Y., and Hsieh-Yee, I. (2008,November). *Subject Access for Images in an OPAC.* Annual Meeting of the American Society for Information Science and Technology, Columbus, Ohio. (Also co-organized a panel on Retrieving and Using Visual Resources: Challenges and Opportunities for Research and Education.)

Hsieh-Yee, I. (2008, June). *Educating Cataloging Professionals in a Changing Information Environment*. National Taiwan University, Taipei, Taiwan.

Vellucci, S. L., Moen, W.E., Hsieh-Yee, I., Marson, B., and Wisser, K. (2008, January) *Building a Metadata Education and Research Community through MERIC (Metadata Education and Research Information Commons): Demo and Stakeholder Input*. A panel presented at the 2008 Conference of the Association for Library and Information Science Education, Philadelphia, Pennsylvania.

Hsieh-Yee, I., Choi,Y. and Kules, B. (2007, October). *Searching for Books and Images in OPAC: Effects of LCSH, TOC and Subject Domains.* A poster presented at the American Society for Information Science and Technology Annual Meeting, Milwaukee, Wisconsin.

Hsieh-Yee, I. and Coogan, J. (2007, August) *Google Scholar vs. Academic Search Premier: A Comparative Analysis.* Presented to the Faculty and Staff of the University of the District of Columbia.

Hsieh-Yee, I. and Coogan, J. (2007, June). *Google Scholar vs. Academic Search Premier: A Comparative Analysis*. Presented to the Washington Research Library Consortium Community, Catholic University of America, Washington, D.C.

Hsieh-Yee, I., Choi, Y., and Kules, B.. (2007, June). *What Users Need for Subject Access: Table of Contents or Subject Headings?* A poster presented at the 2007 American Library Association Annual Conference, Washington, D.C., June 2007.

Choi, Y., Hsieh-Yee, I., and Kules, B. (2007, June). *Retrieval Effectiveness of TOC and LCSH.* A paper presented at the Joint Conference on Digital Libraries 2007, Vancouver, Canada.

Vellucci, S. L., Hsieh-Yee, I., and Moen, W.E. (2007, May). *If We Build It, Will They Come? Building a Community of Practice for Metadata Stakeholders.* A poster presented at the Rutgers University Research Day, Bridgeton, New Jersey.

Hsieh-Yee, I. (2007, May). *Federated Searching: User Experience & Perceptions.* International Conference on Information Organization & Retrieval, National Taiwan University, Taipei, Taiwan.

Hsieh-Yee, I. (2007, May). *Search Performance of Google Scholar and Academic Search Premier.* International Conference on Information Organization & Retrieval, National Taiwan University, Taipei, Taiwan.

Hsieh-Yee, I. (2007, May) *MERIC: Building a Digital Commons for Metadata Education & Research.* International Conference on Information Organization & Retrieval, National Taiwan University, Taipei, Taiwan.

Hsieh-Yee, I., and Coogan, J. (2007, March/April). *A Comparative Analysis of Google Scholar and Academic Search Premier.* Poster presented at the Association of College & Research Libraries 13th National Conference, Baltimore, Maryland.

Vellucci, S. L. and Hsieh-Yee, I. (2007, March/April) *They Didn't Teach Me That in Library School! Building a Digital Teaching Commons to Enhance Metadata Teaching, Learning and Research*. On-site presentation and Webcast by Elluminate. A contributed paper presented at the Association of College & Research Libraries 13th National Conference, Baltimore, Maryland. The acceptance rate for contributed paper was 20%. This paper was one of 10 conference papers chosen for live webcast during the conference.

Moen, W., Hsieh-Yee, I. and Vellucci, S.L. (2007, January) *A DSpace Foundation for a Teaching & Research Commons: The Metadata Education and Research Information Commons.* A poster session presented at the Open Repositories Conference 2007, San Antonio, Texas.

Tang, R., Hsieh-Yee, I., and Zhang, S. (2006, November) *User Perception of MetaLib Combined Search*. Paper presented at the Annual Meeting of the American Society for Information Science and Technology, Austin, Texas, Nov. 2006.

Hsieh-Yee, I. (2006, November). *Federated Searching: User Perceptions, System Design, and Library Instructions.* Paper presented at the Annual Meeting of the American Society for Information Science and Technology, Austin, Texas. (Panel organizer, moderator, presenter).

Hsieh-Yee, I. (2006, November). *Building a Digital Teaching Commons to Enhance Teaching and Learning: The MERIC Experience and Challenges.* Paper presented at the Annual Meeting of the American Society for Information Science and Technology, Austin, Texas. (Panel organizer, moderator, presenter)

Hsieh-Yee, I. (2006, September). *Search Performance of Google Scholar and Academic Search Premier.* Paper presented at the ERIC Publishers Meeting, Washington, D.C.

Hsieh-Yee, I., Zhang, S., and Rong Tang, R. (2006, June). *User Perceptions of a Federated Search System*. Poster presented at Joint Conference on Digital Libraries, Chapel Hill, North Carolina.

Hsieh-Yee, I. and Zhang, S. (2006, June). *Preparing Users for Federated Search: Implications of a MetaLib User Perceptions Study.* Paper presented at the 2006 Ex Libris User Groups of North America Conference, Knoxville, Tennessee.

Hsieh-Yee, I. (2006, January). *MERIC Organizations and Navigation*. Paper presented at the 2006 ALISE Annual Conference, San Antonio, Texas.

Hsieh-Yee, I. (2006, January). *Metadata and Cataloging Education: Recommended Competencies.* Paper presented at the 2006 ALISE Annual Conference, San Antonio, Texas.

Hsieh-Yee, I. (2005, November). *Digital Library Evaluation: Progress & Next Steps.* Presentation at the Annual Meeting of the American Society for Information Science & Technology, Charlotte, North Carolina.

Hsieh-Yee, I. (2005, August). *Providing Access to Digital Content: Issues for DL Managers.* Presentation at MDK12 Digital Library Steering Committee Meeting, Columbia, Maryland.

Hsieh-Yee, I. (2005, April). *Enhancing Teaching and Learning: The Role of School Library Media Specialists.* Presentation at Meeting of the Baltimore County Public School System School Media Specialists, Baltimore, Maryland.

Hsieh-Yee, I. (2005, January). *Subject Access and Users: Insights & Inspirations from Marcia J. Bates.* Paper presented at the Historical Perspectives SIG, 2005 Conference of the Association for Library and Information Science Education, Boston, Massachusetts.

Hsieh-Yee, I. (2005, January). *Electronic Resource Management: Practice, Employer Expectations, & CE Interests.* Paper presented at Technical Services Education SIG, 2005 Conference of the Association for Library and Information Science Education, Boston, Massachusetts.

Hsieh-Yee, I. (2004, October). *Library Professionals for the Digital Age: Competencies & Preparation.* Paper presented at Bibliographic Access Management Team meeting, Library of Congress, Washington, D.C.

Hsieh-Yee, I. (2004, January). *Cataloging and metadata expertise for the digital era.* Presented at Preparing 21st Century Cataloging and Metadata Professionals: A Workshop for Educators and Trainers, San Diego and sponsored by ALCTS, ALISE, LC, and OCLC.

Hsieh-Yee, I. (2004, January). *Educating catalogers for the digital era.* Paper presented at the Technical Services SIG, 2004 Conference of the Association for Library and Information Science Education, San Diego.

Hsieh-Yee, I. (2003, July). *Cataloging Education for the 21st Century.* A presentation at the Library of Congress, Washington, D.C.

Hsieh-Yee, I. (2002, January) *Metadata Education and Research Priorities: A Delphi Study of Metadata Experts.* Presentation at the 2002 Conference of the Association for Library and Information Science Education, New Orleans.

Hsieh-Yee, I. (2001, November). *A Delphi Study of Metadata: Preliminary Findings.* Poster session at the 2001 Annual Meeting of the American Society for Information Science & Technology, Washington, D.C.

Hsieh-Yee, I. (2001, June). *Resources on Asian American Children: Analysis of Retrieval by Search Engines and WorldCat.* Presentation at the National Conference on Asian Pacific American Librarians, San Francisco.

Hsieh-Yee, I. (2001, January). *Delphi Study on Metadata: Project Design.* Presentation at Research Awards Session, Association for Library & Information Science Education, Washington, D.C.

Hsieh-Yee, I. (2000, May). *Web Search Behavior Research: Progress and Implications.* Presentation at the Symposium on Evaluating Library and Information Science Research, University of Wisconsin-Madison, Madison, Wisconsin.

Hsieh-Yee, I. (2000, March). *ERIC User Services: Evaluation in a Decentralized Environment.* Presentation at the National ERIC Joint Directors/Technical Meeting, Arlington, Virginia.

Hsieh-Yee, I. (2000, January). *Enhancing Learning with Web Technology.* Presentation at Faculty Conversations, Catholic University of America, Washington, D.C.

Hsieh-Yee, I. (2000, January). *From Surrogates to Objects: CUA's Approaches to Organizing Electronic Resources.* Paper presentation at the Annual Conference of the Association for Library and Information Science Education, San Antonio, Texas.

Yee, P., and Hsieh-Yee, I. (1997, November). *Individual Differences in Search Behavior on the WWW*. A poster session presented at the 38th Annual Meeting of the Psychonomic Society, Philadelphia, Pennsylvania.

Hsieh-Yee, I. (1997, April). *Research + Marketing + Preparation = Job!* Presented at the "Workshop on Resume and Interview Techniques," Special Libraries Association, Student Chapter, Catholic University of America, Washington, D.C.

Hsieh-Yee, I. (1997, February). *Creating CyberCatalogers: Education and Training.* Presentation at ALA's Midwinter Meeting, Washington, D.C.

Hsieh-Yee, I. (1997, February). *Search Tactics of Web Users in Searching for Texts, Graphics, Known Items and Subjects: A Search Simulation Study.* Presented at the Conference of the Association for Library and Information Science Education, Washington, D.C.

Hsieh-Yee, Ingrid. "Beginning Your Special Library/Information Center Career." Presented at SLA's "Career Day," Jan. 11, 1997, Catholic University of America.

Hsieh-Yee, I. (1996, September). *The Roles of Library and Information Scientists in Managing Electronic Information.* Presentation at Hamilton College, Clinton, New York.

Hsieh-Yee, I. (1996, May). *The Future of Cataloging as a Profession.* Presented at "The Cataloging Forum, Library of Congress, Washington, D.C.

Hsieh-Yee, I. (1994, October). *The Impact of the Internet on OPACs.* Presented at the Third Workshop on User Interfaces for OPACs, Library of Congress, Washington, D.C.

Reports

Hsieh-Yee, I., with Knowledge Management Competencies and Performance Action Group of the Federal Knowledge Management Initiative. “From Knowledge Management Competencies to Improved Organizational Performance.” April 9, 2009.

Hsieh-Yee, I., with Knowledge Practices Action Group of the Federal Knowledge Management Initiative. “KM Practice in Government Agencies: Findings and Recommendations.” April 9, 2009.

Hsieh-Yee, I. “Delphi Study on Metadata.” 2001. Three quarterly reports submitted to the Association for Library and Information Science Education.

Hsieh-Yee, I. “College Students' Information Channels: Patterns of Use and Possible Factors in Channel Selection.” 1995. Submitted to the Catholic University of America.

Hsieh-Yee, I. "The Information-Seeking Patterns of Scholars and Their Use of an Online Information System." 1994. Submitted to the Council on Library Resources.

Book Reviews

Review of *The Measurement and Evaluation of Library Services*, by Sharon L. Baker and F. Wilfrid Lancaster. *Information Processing and Management* 30 (1994): 450-52.

Review of *Subject Access to Films and Videos, by Sheila S. Intner and William E. Studwell; and Cataloging Unpublished Nonprint Materials*, by Verna Urbanski with Bao Chu Chang and Bernard L. Karon. *Information Processing and Management* 30 (1994): 449-50.

Review of *Automated Information Retrieval in Libraries: A Management Handbook*, by Vicki Anders. *Journal of Library and Information Science* 19 (1993): 98-100.

Review of *Full Text Databases*, by Carol Tenopir and Jung Soon Ro. *Information Processing and Management* 28 (1992): 667-68.

Review of *Descriptive Cataloging for the AACR2R And USMARC: A How-to-Do It Workbook*, by Larry Millsap and Terry Ellen Ferl. *Information Processing and Management* 28 (1992): 809-11.

Review of *MARC Manual: Understanding and Using MARC Records*, by Deborah J. Byrne. *Information Processing and Management* 28 (1992): 537-38.

Service

Professional Associations and Societies

- Library of Congress. RDA Training Program for the Profession. Co-authored with Tim Carlton. 2013-2014.
- 2014 Digital Preservation Outreach & Education Survey. Contributed to the design of the survey, 2014.
- National Digital Stewardship Alliance. Outreach Committee. 2011-2014.
- National Digital Stewardship Residency Program. Advisory Group, 2012-2013.
- FEDLINK Health Information Technology Advisory Council, 2011-2015.
- 2012 Joint Conference on Digital Libraries. Program Planning Committee, Pre-Conference Proposals Review Committee, 2012
- Catholic Research Resources Alliance. Five-Year Strategic Plan Task Force, 2011-2012
- Institute of Museum and Library Services. Grant reviewer. 2004, 2005, 2010.

- Association for Library and Information Science Education.
  * ALISE Bodan Wynar Research Paper Award Committee, 2015, 2016, 2017
  * ALISE Eugene Garfield Dissertation Award Competition, Jury, 2013, 2014
  * ALISE Research Grant Competition Committee. Chair, 2012
  * Pratt-Severn Faculty Innovation Award. Chair, 2009, 2010

* ALISE Doctoral Poster Jury, 2012
* "Information Organization Competencies for the 21st Century" Discussion session leader. 2009 Conference of the Association for Library and Information Science Education.
* Assisted Technical Services SIG Convener in organizing a program, ""Building a Metadata Education and Research Community through MERIC (Metadata Education and Research Information Commons): Demo and Stakeholder Input" for the 2008 ALISE conference.
* Association for Library Collections and Technical Services/Association for Library and Information Science Education (ALCTS/ALISE) Metadata Education and Research Information Center (MERIC) Advisory Board, Co-Chair (with Sherry Vellucci), 2005-2007. Chair, 2008-2009 (leading the effort to build MERIC, a repository and collaborative space for metadata educators, practitioners, and researchers)
* Technical Services SIG, Convener, 2004-2005. Organized a program on "Electronic Resources Management: Current Practices, Employer Expectations, and Teaching Strategies" for the 2005 conference in Boston, Massachusetts.
* Technical Services SIG, Convener, 2003-2004. Organized a program on "Organizing Information with Metadata: Desired Competencies and Teaching Innovations" for the 2004 conference.
* Technical Services SIG, Convener, 1999-2000. Organized a program on "Teaching the Organization of Electronic Resources" for the 2000 conference.
* Curriculum SIG, Co-convener (with Sibyl Moses), 1996-97. Organized a program on "Government Information Policy" for the 1997 conference.

- American Society for Information Science & Technology.
  * Reviewer, Conference program panel submissions and poster submissions, 2005, 2006, 2007, 2009, 2011, 2012, 2013, 2014, 2015, 2016, 2017
  * Nomination Committee, 2009-2011
  * Information Science Education Special Interest Group. American Society for Information Science and Technology. Chair-Elect, 2007-2008. Chair 2008-2009.
  * Committee on Information Science Education. 1999-2006.
  * Committee on Information Science Education. Organizing Committee for an orientation program for students at ASIS annual meetings, 1999-2001
  * Committee on Information Science Education. Sub-committee on Student Welfare (focusing on issues related to master's education), 1998-2001
  * SIG ED. Organizing Committee for the "Seminar on Research and Career Development" for junior researchers. 1995-96 (chair), 1997-2001
  * ISI Doctoral Dissertation Proposal Scholarship Jury, 1997; 2001, 2002
  * Pratt-Severn Best Student Research Paper Award Jury. Chair. 1997
  * 1998 Midyear Meeting (referee of contributed papers), 1997
  * Organizer and moderator of the ASIS Doctoral Forum and the Doctoral Research Seminar 1994-1995
  * SIG Human Computer Interaction. Chair-Elect, Chair, 1993-1995
  * Doctoral Forum Award Jury, 1995
  * Best Student Paper Award Jury, 1995

- American Library Association.
  * Committee on Accreditation, External Review Panelist, 2009- (site visiting team 2013-2014; site visiting team 2016-2017)
  * Association for Library Collections and Technical Services Task Force on Competencies and Education for a Career in Cataloging, member, 2008-2009
  * Facilitator for "What They Don't Teach in Library School: Competencies, Education and Employer Expectations for a Career in Cataloging," an Association for Library Collections and Technical Services Preconference, June 22, 2007 in Washington, D.C. Also a local liaison for bringing this program to the Catholic University of America.
  * Facilitator for a discussion on "Effect of Electronic Resources on Technical Services" at ALA's Midwinter Meeting held in Feb. 1997 in Washington, D.C.
  * International Relations Committee, Subcommittee Task Force for IFLA and China, 1994-1997

- Virginia Association of School Librarians. Scholarships and Awards Committee. 2010-2012

- Federal Knowledge Management Initiative, Knowledge Management Practices Action Group. Member. 2009 (leading the effort to build a knowledge management repository)
- Federal Knowledge Management Initiative, Knowledge Management Competencies & Learning Action Group. Member. 2009 (developing an action plan for helping government knowledge workers and government agencies to develop knowledge management competencies)
- National Center for Education Statistics. Technical Review Panel. 2008.
- External evaluator for a case of promotion to full professorship. University of Tennessee. 2008.
- National Information Standards Organization (NISO). Advisory Board, Revision of "IMLS Framework of Guidance for Building Good Digital Collections," 2004, 2007.
- Library of Congress, Bibliographic Control of Web Resources: A Library of Congress Action Plan. Principal Investigator of Action Item 5.1, focusing on cataloging and metadata education for students and new librarians, 2002-2003. (worked with the Association for Library Collections and Technical Services, Education Task Force)

- Chinese American Librarians Association
  * Chinese American Librarians Association Outstanding Library Leadership Award in Memory of Dr. Margaret Chang Fung, Award Committee, 2016-2017
  * Achievement Award Jury, 2000-2001
  * CALA Goal 2000 Task Force, 1997
  * Scholarship Committee, 1995, 1996-1997 (chair)
  * Board of Directors, 1994-1997
  * Publication Committee, 1993-1995
  * International Relations Committee, 1993-1996

- Sailor[SM] Assessment Advisory Group (An impact study of Sailor, Maryland's Public Information Network), 1995

- Editorial boards
  Journal of Library & Information Science. Editorial Board, 2012-
  Chinese American Librarians Association, *Occasional Papers Series*. Editorial Board, 2009-2016.
  Library Quarterly. Editorial Board, 2003-2008
  Bulletin of the Medical Library Association, 1994-97
  Newsletter editor for the Chinese American Librarians Association, 1989-92

- Referee for the following journals
  Information Processing and Management
  Journal of Digital Information
  Journal of Education for Library and Information Science
  Journal of Information Science
  Journal of Library & Information Science
  Journal of Library Metadata
  Journal of the American Society for Information Science & Technology
  Library and Information Science Research
  Library Quarterly

- Expert reviewer, "Digital Library" course, Evaluation module, University of North Carolina, Chapel Hill, 2007-2008.
- Expert reviewer, "Information Organization" course, University of Michigan, Ann Arbor. 2007.

Catholic University of America

- Academic Senate Committee on Appointments and Promotions, 2005-2008
- Academic Senate Committee on Committees and Rules, 2009-2012, 2019-2022
- Academic Senate representative (for School of Arts & Sciences), 2017-2020
- Academic Senate representative (for School of Library & Information Science) 2003-2012
- Academic Senate Library Committee, Interim Chair (2007), Member, 2008-2012
- Academic Senate Committee on Computing, 1995-2003

- President's Administrative Council, 2010-2012
- Deans' Council, 2010-2012
- Academic Leadership Group, 2010-2012
- Graduate Board, 2010-2012

- School of Arts & Sciences, Committee on Appointments and Promotions, 2015-2019
- School of Arts & Sciences, Academic Council, 2015-2016.
- School of Arts & Sciences, Ordinary Professor Group, 2013-
- Doctoral Dissertation Defense Committee, Chair, Dept. of Psychology, 2016, 2017, 2018, 2019, 2020, 2021, 2022
- Doctoral Dissertation Defense Committee, Chair, Dept. of Education, 2014, 2015, 2017, 2018, 2019
- Doctoral Dissertation Defense Committee, Chair, National Catholic School of Social Services, 2019, 2020, 2021
- Doctoral Dissertation Defense Committee, Chair, School of Nursing, 2006, 2008
- CUA Scholarly Communication Project Team, Member (2007), Chair, 2008-2009
- Dean Search Committee, 1992-1994, 1998-1999, 2002-2003, 2006-2007
- Fulbright Review Panel, 2006
- CUA Service Learning Advisory Board, 2001-2002
- CUA Faculty Conversations on Enhancing Teaching and Learning through Technology, Planning Group, 1999-2001
- CUA Initiative on Technology and Teaching, 1998-2001

Dept. of Library and Information Science

- Admissions Committee, 2007-2009, Chair 2010-2012, Member 2013-2015, Member 2018-2022
- Appointments and Promotions Committee, 1991-2022
- Student advisement, 1990-2022
- 4+1 Bachelor to Master program, Program Advisor, 2014-2022
- LIS Minor, Advisor, 2017-2022
- Curriculum Committee, 2020-21 (Member), 2021-2022 (Chair)
- Scholarship & Awards Committee, 2016-2018, 2020-21
- Comprehensive examination editor, 2020-21
- Symposium, Colloquium, Lecture Series Committee, May 2018-May 2020 (Chair)
- Symposium and Colloquium Committee, fall 2016-May 2018
- Bridging the Spectrum Symposium session moderator, 2018, 2019, 2020, 2021
- Organization of Information Core Course Outcomes Assessment, 2019, 2020
- The Information Professions in Society Core Course Outcomes Assessment, 2019, 2020
- Community Services Librarianship, Course of Study Review and Revision, 2020
- Organization of Information, Course of Study Review and Revision, 2019
- Organization of Information, Competency and Course Offerings Review, 2018-2019
- Accreditation presentation, Chair, June 2015-August 2016
- Interim Co-Chair, June 2015-August 2016.
- Blended/OWL Learning Committee, spring 2016-2018
- Technology Committee, fall 2016-2017
- Comprehensive examination editor, 2016-2017, reader (every year since 1990)

- LIS Advisory Board, 2015-2016 (chair); fall 2016- May 2018 (member)
- Committee on Planning and Assessment, 2015-2016 (chair)
- Senior Faculty Committee, 2014-2016.
- Accreditation Steering Committee, 2014-2016 (Chair, 2015-2016)
- Accreditation Students Standard Committee, co-chair, 2014-2016
- Accreditation Mission, Goals, and Objectives Standard Committee, co-chair, 2014-2016
- Accreditation Curriculum Standard, member 2014-2-16
- Accreditation Administration and Finance Standard, member 2014-2016
- Cultural Heritage Information Management Project (IMLS-funded), Co-PI, 2012-2015
- Cultural Heritage Information Management Forum (scheduled for June 2015), Co-Organizer, 2013-2015
- Health Information Technology Interim Review Committee, 2015 (chair)
- Health Sciences Librarianship Advisory Group, 2015- (chair)
- Comprehensive examination editors, 2013-2014, 2016-2017
- National Digital Stewardship Alliance liaison, 2011-2014
- Advisory Board, Chair 2010-2012
- Academic Honesty Committee, Chair, 2008-2012
- Blended Learning Committee, 2010-2012
- Colloquium Committee, 2010-2012
- Comprehensive Examination Administration, 2010-2012
- Cultural Heritage Information Management Advisory Committee, 2010-2012 (chair), 2013-
- Curriculum Committee, 1991-2003, 2007-2009, Chair 2010-2012, member 2013-
- Curriculum Subcommittee on Comprehensive Examination, Chair 2009-2012
- Health Information Technology Advisory Board, Chair 2010-2012. Member 2013-
- Health Sciences Advisory Committee, 2009, Chair 2010-2012. Member 2013-
- HIT Expert Forum, Chair 2012. Member 2013-2015
- Health Information Technology Student Group Advisor, 2011-2012
- State Council for Higher Education of Virginia, SLIS Representative, 2010-2012
- Symposium Planning Committee, 2010-2012
- Website Management Team, Chair, 2010-2012
- Urban School Librarianship Project (IMLS-Funded), PI, 2007-2011 (chair, 2010-11)
- Failing Grades Committee, 1995-1997 (chair), 2000-2001 (chair), 2004-2005 (chair), 2007 (chair)-2011
- Faculty Search Committee, 1994-1998, 2002-2004, 2006 (chair), Fall 2007-2009, Chair fall 2009-2012
- Recruitment Committee, Chair 2010-2012
- Strategic Planning Committee, Chair 2010-2012
- Technology Committee, 2010-2012
- Accreditation Advisory Committee, 2007-2009
- Accreditation Coordinating Committee, 2007-2009
- Accreditation Steering Committee, 2007-2009
- SLIS Advisory Group, 2007-2009

- Accreditation Curriculum Standard Committee, Co-chair, 2007-2009
- Accreditation Faculty Standard Committee, Co-chair, 2007-2009
- LSC 551 Information Organization Review Team, Co-chair, 2008-2009, 2015-2016.
- Curriculum Subcommittee on Portfolios, 2009
- LSC 555 Information Systems in Libraries and Information Centers Review Team, contributor, 2008-2009
- Redesign of LSC 730 Use and Users of Libraries and Information. 2009-
- Development of a metadata institute that was taught as LSC 715 Organization of Internet Resources in 2008. The institute is being revised and will be offered in 2010 under a new course title.
- Development of lesson plans, assignments, and evaluation rubrics for LSC 606, Cataloging and Classification, for the School's NCATE accreditation. 2008
- Howard and Mathilde Rovelstad Scholarship Committee, Chair, 2004-2007
- Assistant Dean Search Committee, Chair, Fall 2007
- Liaison to the Association for Library Collections and Technical Services to bring its preconference program, Cataloging Education and Employer Expectations, to CUA during the 2007 American Library Association Annual Meeting in Washington, D.C.
- Organizer of the colloquium presentation and reception for Tamar Sadeh of Ex Libris on PRIMO June 2007
- Practicum review and design (work with potential supervisors, such as the American Indian Museum internship description revision) 2006-
- Comprehensive examinations (edits, proctoring, and grading), 1990-
- SLIS Web site redesign: Comments and suggestions. Fall 2007
- Conducted surveys of current students and alumni in preparation for the 2005 re-accreditation, 2004-2005
- Technology Committee, 1992-1999 (chair, 1996-1998), 2002-2003 (member)
- Colloquia Committee 1997-1999, 2002-2003.
- Advisor of the CUA Student Chapter of the American Society for Information Science and Technology, 2002-2003
- Visiting Professor Search Committee, 1999, 2000, 2001
- Leader, Participation in the CORC experiment, 1999-2000
- Advisor of the Special Libraries Association Student Chapter, 1993-1999; the group was recognized for outstanding leadership by SLA in 1999.
- COA planning Committee, Task Force on Electronic Presentation of SLIS Reports (team leader) 1997-1998
- COA Planning Committee, Subcommittee on Technology 1996-1998
- NLM practicum coordinator, 1997-1998
- Computer Literacy Workshops: Assisted with the development and evaluation of the workshops, 1996-1998
- Leader, Participation in the InterCat project, 1995-1997

Appendix B.

MARC 21 Reference Materials

Part VII: A Summary of Commonly Used MARC 21 Fields
Part VIII: A List of Other Fields Often Seen in MARC Records
Part IX: The Leader
Part X: Field 008 for Books

Part VII:

A Summary of Commonly Used MARC 21 Fields

This is a summary of the MARC 21 tags used most frequently by libraries in entering their own bibliographic records. For full listings of all MARC 21 tags, indicators, and subfield codes, see *MARC 21 Format for Bibliographic Data*.

In the explanations on these pages:

Tags -- The tags (3-digit numbers) are followed by the names of the fields they represent. In this summary, and in the *MARC 21 Format for Bibliographic Data*, if a tag can appear more than once in one bibliographic record, it is labeled repeatable (R). If it can only be used once, it is labeled non-repeatable (NR). For example, a catalog record can have several subjects, so the tags for subject added entries (6XX) are labeled repeatable (R).

Indicators -- The use of indicators is explained in fields where they are used. Indicators are one-digit numbers. Beginning with the 010 field, in every field -- following the tag -- are two character positions, one for Indicator 1 and one for Indicator 2. The indicators are not actually defined in all fields, however. And it is possible that a 2nd indicator will be used, while the 1st indicator remains undefined (or vice versa). When an indicator is undefined, the character position will be represented by the character # (for blank space).

Subfield codes -- All the data in each field (beginning with the 010 field) is divided into subfields, each of which is preceded by a delimiter-subfield code combination. The most common subfield codes used with each tag are shown. Each subfield code is preceded by the character $, signifying a delimiter. The name of the subfield follows the code.

In general, every field MUST have a subfield 'a' (**$a**). One exception that is often seen is in Field 020 (ISBN), when the ISBN information (subfield **$a**) is unavailable but the price (subfield **$c**) is known. Some subfields are repeatable. In this summary, repeatability is noted for only the more common repeatable subfields.

Examples: Examples follow the explanation for each field. For clarity, one space has been placed between the tag and the first indicator, one space has been placed between the second indicator and the first delimiter- subfield code, and one space has been inserted between the delimiter-subfield code and the subfield data.

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010 Library of Congress Control Number -- (LCCN)
(NR, or Not Repeatable
Indicators undefined.
Subfield used most often:
$a -- Library of Congress control number

```
Example:    010 ##  $a ###86000988#
```

020 International Standard Book Number -- (ISBN)
(R, or Repeatable)
Indicators undefined.
Subfields used most often:
$a -- International Standard Book Number
$c -- Terms of availability (often a price)
$z -- Cancelled/invalid ISBN (R)

```
Example:    020 ##  $a 0877547637
```

040 Cataloging source -- (NR)
Indicators undefined.
Subfields used most often:
$a -- Original cataloging agency
$c -- Transcribing agency
$d -- Modifying agency (R)

```
Example:    040 ## $a DLC
                   $c DLC
                   $d gwhs
```

100 Main entry -- Personal name -- (primary author)
(NR; there can be only one main entry)
Indicator 1: Type of personal name entry element
0 -- Forename
1 -- Surname (this is the most common form)
3 -- Family name
Indicator 2 undefined.
Indicator 2 became obsolete in 1990. Older records may display 0 or 1
Subfields used most often:
$a -- Personal name
$b -- Numeration

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$c -- Titles and other words associated with a name (R)
$q -- Fuller form of name
$d -- Dates associated with a name (generally, year of birth)

```
Example:    100 1# $a Gregory, Ruth W.
                   $q (Ruth Wilhelme),
                   $d 1910–
```

130 Main entry -- Uniform title -- (NR)

Indicator 1: Nonfiling characters

0-9 -- Number of nonfiling characters present (for initial articles, including spaces)

Indicator 2 undefined.

Indicator 2 became obsolete in 1990. (See 100 above.)

Subfields used most often:

$a -- Uniform title
$p -- Name of part/section of a work (R)
$l -- Language of a work
$s -- Version
$f -- Date of a work

```
Example:    130 0# $a Bible.
                   $p O.T.
                   $p Psalms.
```

240 Uniform title (NR)

Indicator 1: Uniform title printed or displayed

0 -- Not printed or displayed
1 -- Printed or displayed (most common)

Indicator 2: Nonfiling characters

0-9 -- Number of nonfiling characters present (for initial articles, including spaces)

Subfields used most often:

$a -- Uniform title
$l -- Language of a work
$f -- Date of a work

```
Example:    240 10 $a Ile mystérieuse.
                   $l English.
                   $f 1978
```


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245 Title Statement (NR)

Indicator 1: Title added entry

(Should the title be indexed as a title added entry?)

0 -- No title added entry

(indicates a title main entry; i.e. no author is given)

1 -- Title added entry

(the proper indicator when an author given in 1XX; the most common situation)

Indicator 2: Nonfiling characters

0-9 -- Number of nonfiling characters present, including spaces; usually set at zero, except when the title begins with an article; e.g., for *The robe*, the second indicator would be set to 4. The letters *T*, *h*, *e*, and the space following them are then ignored in alphabetizing titles. The record will be automatically filed under "*r*" -- for *Robe*.

Subfields used most often:

$a -- Title proper

$h -- Medium (often used for non-book media)

$p -- Name of part/section of a work (R)

$b -- Reminder of title (subtitles, etc.)

$c -- Remainder of title page transcription/Statement of responsibility

```
Example:    245 14 $a The DNA story :
                   $b a documentary history of gene
                      cloning /
                   $c James D. Watson, John Tooze.
```

246 Varying form of title (R)

Indicator 1: Note/title added entry controller

1 -- Note, title added entry

3 -- No note, title added entry

Indicator 2: Type of title

-- No information provided

0 -- Portion of title

1 -- Parallel title

4 -- Cover title

8 -- Spine title

Subfield used most often:

$a -- Title proper

```
Example:    246 3# $a Four corners power review
```

250 Edition statement (NR)

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0114

Indicators undefined.
Subfield used most often:
$a -- Edition statement

```
Example:    250 ## $a 6th ed.
```

260 Publication, distribution, etc. (Imprint) (R)
Indicator 1: Sequence of publishing statements
-- No information provided
Indicator 2: Undefined
Subfields used most often:
$a -- Place of publication, distribution, etc. (R)
$b -- Name of publisher, distributor, etc. (R)
$c -- Date of publication, distribution, etc. (R)

```
Example:    260 ## $a New York :
                   $b Chelsea House,
                   $c 1986.
```

300 Physical description (R)
Indicators undefined.
Subfields used most often:
$a -- Extent (number of pages) (R)
$b -- Other physical details (usually illustration information)
$c -- Dimensions (cm.) (R)
$e -- Accompanying material (for example, "teacher's guide" or "manual")

```
Example:    300 ## $a 139 p. :
                   $b ill. ;
                   $c 24 cm.
```

440 Series statement / Added entry--Title
This field was made obsolete in 2008 to simplify the series statement. See 490 and 830.

490 Series statement (No added entry is traced from field) (R)
Indicator 1: Specifies whether series is traced (whether an 8XX tag is also present)
0 -- Series not traced
1 -- Series traced (8XX is in record)
Indicator 2 undefined.
Subfield used most often:

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$a -- Series statement (R)
$v -- Volume number (R)

```
Example:    490 1# $a Colonial American craftsmen
```

500 General note (R)
Indicators undefined.
Subfield used most often:
$a -- General note (Used when no specialized note field has been defined for the information. Examples: Notes regarding the index; the source of the title; variations in title; descriptions of the nature, form, or scope of the item.)

```
Example:    500 ##  $a Includes index.
```

504 Bibliography, etc. note (R)
Indicators undefined.
Subfield used most often:
$a -- Bibliography, etc. note

```
Example:    504 ## $a Includes bibliographical
                      references.
```

505 Formatted contents note (R)
Indicator 1: Type of contents note
0 -- Complete contents
1 -- Incomplete contents (used with multivolume set when some volumes are not yet published)
2 -- Partial contents
Indicator 2: Level of content designation
-- Basic
Subfield used most often:
$a -- Formatted contents note

```
Example:    505 0# $a Pride and prejudice -- Emma
                      -- Northanger Abbey.
```

520 Summary, etc. note (R)
Indicator 1: Display constant controller
-- Summary

EX-1040
Norit Americas, Inc. v. Calgon Carbon Corporation
0116

1 -- Review
2 -- Scope and content
3 -- Abstract

Indicator 2 undefined

Subfields used most often

$a -- Summary, abstract, or annotation
$b -- Expansion of summary note

```
Example:    520 ## $a This basic guide to parliamentary
                     procedure tells how to conduct
                     and participate in a meeting
                     properly.
```

600 Subject added entry -- Personal name (R)

Indicator 1: Type of personal name entry element

0 -- Forename
1 -- Surname (this is the most common form)
3 -- Family name

Indicator 2: Subject heading system/thesaurus (identifies the specific list or file which was used)

0 -- Library of Congress Subject Headings
1 -- LC subject headings for children's literature
2 -- Medical Subject Headings
3 -- National Agricultural Library subject authority file
4 -- Source not specified
5 -- Canadian Subject Headings
6 -- Répertoire de vedettes-matière
7 -- Source specified in subfield $2

(Note regarding Sears subject headings: The MARC 21 format does not provide an assigned indicator for Sears subject headings. Therefore, an indicator of 7 is used, and the MARC defined code "sears" is placed in subfield $2.)

Subfields used most often:

$a -- Personal name (surname and forename)
$b -- Numeration
$c -- Titles and other words associated with a name (R)
$q -- Fuller form of name
$d -- Dates associated with a name (generally, year of birth)
$t -- Title of a work
$v -- Form subdivision (R)
$x -- General subdivision (R)
$y -- Chronological subdivision (R)
$z -- Geographic subdivision (R)
$2 -- Source of heading or term (used with 2nd indicator of 7)

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```
Example:    600 10 $a Shakespeare, William,
                   $d 1564-1616
                   $x Comedies
                   $x Stage history.
```

```
Example:    600 10 $a Shakespeare, William,
                   $d 1564-1616
                   $x Knowledge
                   $z Rome
                   $v Congresses.
```

Notice that subfields $v, $x, and $z in the 600 field are repeatable. Subfields $v, $x, $y, and $z do not have to be in alphabetical order. They will be in the order prescribed by the instructions given by the subject heading system.

610 Subject added entry -- Corporate name (R)

*Indicator 1:*Type of corporate name entry element

0 -- Inverted name (not used with AACR2)

1 -- Jurisdiction name

2 -- Name in direct order

Indicator 2: Subject heading system/thesaurus.

See indicator 2 under 600

Subfields used most often:

$a -- Corporate name or jurisdiction name as entry element

$b -- Subordinate unit (R)

$v -- Form subdivision (R)

$x -- General subdivision (R)

$y -- Chronological subdivision (R)

$z -- Geographic subdivision (R)

$2 -- Source of heading or term (used with 2nd indicator of 7)

```
Example:    610 10 $a United States.
                   $b Army Air Forces
                   $v Biography.
```

650 Subject added entry -- Topical term (Most subject headings fit here.) (R)

Indicator 1: Level of subject

-- No information provided

Indicator 2: Subject heading system/thesaurus

(identifies the specific list or file which was used)

0 -- Library of Congress Subject Headings

1 -- LC subject headings for children's literature

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2 -- Medical Subject Headings
3 -- National Agricultural Library subject authority file
4 -- Source not specified
5 -- Canadian Subject Headings
6 -- Répertoire de vedettes-matière
7 -- Source specified in subfield $2

Note regarding Sears subject headings: The MARC 21 format does not provide an assigned indicator for Sears subject headings. Therefore, an indicator of 7 is used, and the MARC defined code "sears" is placed in subfield $2.)

Subfields used most often:
$a -- Topical term
$v -- Form subdivision (R)
$x -- General subdivision (R)
$y -- Chronological subdivision (R)
$z -- Geographic subdivision (R)
$2 -- Source of heading or term used with 2nd indicator of 7)

```
Example:    650 #0 $a Theater
                   $z United States
                   $v Biography
                   $v Dictionaries.
```

Notice that subfields $v, $x, and $z in the 650 field are repeatable. Subfields $v, $x, $y, and $z do not have to be in alphabetical order. They will be in the order prescribed by the instructions given by the subject heading system.

651 Subject added entry -- Geographic name (R)
Indicator 1: undefined.
Indicator 2: Subject heading system/thesaurus.
See indicator 2 under 600
Subfields used most often:
$a -- Geographic name
$v -- Form subdivision (R)
$x -- General subdivision (R)
$y -- Chronological subdivision (R)
$z -- Geographic subdivision (R)
$2 -- Source of heading or term (used with 2nd indicator of 7)

```
Example:    651 #0 $a United States
                   $x History
                   $v Chronology.
```


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Notice that subfields $v, $x, and $z in the 651 field are repeatable. Subfields $v, $x, $y, and $z do not have to be in alphabetical order. They will be in the order prescribed by the instructions given by the subject heading system.

700 Added entry -- Personal name (R)

Indicator 1: Type of personal name entry element

0 -- Forename

1 -- Surname (this is the most common form)

3 -- Family name

Indicator 2: Type of added entry

-- No information provided (most common; co-authors, editors, etc.)

2 -- Analytical entry (The values for Indicator 2 changed in 1994 with Format Integration, and older records may display additional values. An analytical entry involves an author/title of an item contained in a work.)

Subfields used most often:

$a -- Personal name

$b -- Numeration

$c -- Titles and other words associated with a name (R)

$q -- Fuller form of name

$d -- Dates associated with a name (generally, year of birth)

$e -- Relator term (such as ill.) (R)

$4 -- Relator code (R)

```
Example:    700 1# $a Baldridge, Letitia.
```

710 Added entry -- Corporate name (R)

Indicator 1: Type of corporate name entry element

0 -- Inverted name (not used with AACR2)

1 -- Jurisdiction name

2 -- Name in direct order

Indicator 2: Type of added entry.

See Indicator 2 under 700

-- No information provided

2 -- Analytical entry

Subfields used most often:

$a -- Corporate name or jurisdiction name as entry element

$b -- Subordinate unit (R)

```
Example:    710 2# $a Sunburst Communications (Firm)
```

740 Added entry -- Uncontrolled related/analytical title (R)

Indicator 1: Nonfiling characters

0-9 -- Number of nonfiling characters present (for initial articles, including

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spaces)

*Indicator 2:*Type of added entry. See Indicator 2 under 700

-- No information provided

2 -- Analytical entry

(This field was redefined in 1994 with Format Integration. Prior to 1994, the field was also used for variant titles, such as a different wording on a spine title. In records created since Format Integration, those variant tiles appear in a 246 field.)

Subfield used most often:

$a -- Title

```
Example:    740 02 $a Uncle Vanya.
```

800 Series added entry -- Personal name (R)

Indicator 1: Type of personal name entry element

0 -- Forename

1 -- Surname

3 -- Family name

Indicator 2 undefined.

Subfields used most often:

$a -- Personal name

$b -- Numeration

$c -- Titles and other words associated with a name (R)

$q -- Fuller form of name

$d -- Dates associated with a name (generally, year of birth)

$t -- Title of a work (the series)

$v -- Volume number

```
Example:    800 1# $a Fisher, Leonard Everett.
                   $t Colonial American craftsmen.
```

830 Series added entry -- Uniform title (R)

Indicator 1 undefined.

Indicator 2: Nonfiling characters

0-9 -- Number of nonfiling characters present (for initial articles, including spaces)

Subfield used most often:

$a -- Uniform title

$v -- Volume number

```
Example:    830 #0 $a Railroads of America (Macmillan)
```


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Part VIII:

A List of Other Fields Often Seen in MARC Records

001	Control number
003	Control number identifier
005	Date and time of latest transaction
006	Fixed-length data elements -- additional material characteristics
007	Physical description fixed field
008	Fixed length data elements (See Part X)
022	International Standard Serial Number (ISSN)
037	Source of acquisition
041	Language code
043	Geographic area code
050	Library of Congress call number
060	National Library of Medicine call number
082	Dewey Decimal classification number (the one recommended by the Library of Congress; locally-assigned call numbers may appear elsewhere)
110	Main entry -- Corporate name (less frequent under AACR2 rules)
256	Computer file characteristics
263	Projected publication date (indicates a CIP -- Cataloging in Publication -- record)
306	Playing time
508	Creation/production credits note
510	Citation/references note (review sources)
511	Participant or performer note
521	Target audience note (first indicator: 0 = reading grade level, 1 = interest age level, 2 = interest grade level, 3 = special audience characteristics, 4 = motivation interest level)
530	Additional physical form available note
538	System details note
586	Awards note
656	Index term -- Occupation
730	Added entry -- Uniform title
852	Location
856	Electronic location and access
9XX	Reserved for local use. (They are used by vendors, systems, or individual libraries to exchange additional data)

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Part IX:

The Leader

There are 24 positions in the Leader, numbered from 00 to 23. For fuller explanation, see the *MARC 21 Format for Bibliographic Data*.

00-04 Record length (calculated by the computer for each record)
05 Record status
- a = increase in encoding level
- c = corrected or revised
- d = deleted
- n = new
- p = increase in encoding from prepublication (previous CIP)

06 Type of record
- a = language material
- c = printed music
- d = manuscript music
- e = cartographic material
- f = manuscript cartographic material
- g = projected medium
- i = nonmusical sound recording
- j = musical sound recording
- k = 2-dimensional nonprojectable graphic
- m = computer file
- o = kit
- p = mixed materials
- r = 3-dimensional artifact or naturally occurring object
- t = manuscript language material

07 Bibliographic level
- a = monographic component part
- b = serial component part
- c = collection
- d = subunit
- i = integrating resource
- m = monograph/item
- s = serial

08 Type of control
- # = no specified type
- a = archival

09 Character coding scheme
- # = MARC-8
- a = UCS/Unicode

10 Indicator count (always "2")
11 Subfield code count (always "2")
12-16 Base address of data (calculated by the computer for each record)
17 Encoding level
- # = full level

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1 = full level, material not examined
2 = less-than-full level, material not examined
3 = abbreviated level
4 = core level
5 = partial (preliminary) level
7 = minimal level
8 = prepublication level (CIP)
u = unknown
z = not applicable

18 Descriptive cataloging form
# = non-ISBD
a = AACR2
i = ISBD
u = unknown

19 Multipart resource record level
# = Not specified or not applicable
a = Set
b = Part with independent title
c = Part with dependent title

20 Length of the length-of-field portion (always "4")
21 Length of the starting-character-position portion (always "5")
22 Length of the implementation-defined portion (always "0")
23 Undefined (always "0")

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Part X:

Field 008 for Books

Field 008 is used for Fixed Length Data Elements ("Fixed Field Codes"). There are 40 character positions in field 008, numbered from 00-39. Undefined positions must contain either a blank (#) or a fill character (|). Positions 00-17 and 35-39 are defined the same way for all media.

The information shown here for positions 18-34 applies only to books. For explanation of all the positions below and for positions 18-34 for other media, see the *MARC 21 Format for Bibliographic Data*.

Note that field 008 has no indicators or subfield codes.

00-05 Date entered on file (YYMMDD),
where Y=year, M=month, and D=day

06 Type of date/publication status:
b = no dates given; B.C. date involved
e = detailed date
s = single known date/probable date
m = multiple dates
r = reprint/reissue date (Date 1) and original date (Date 2)

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n = dates unknown
q = questionable date
t = publication date and copyright date
| = no attempt to code

07-10 Date 1/beginning date of publication

11-14 Date 2/ending date of publication

Date fields contain the year(s) of publication. The type of date(s) in these elements are specified in fixed field element 06: Type of date/publication status. (For further details, see the field 008 description in the *MARC 21 Format for Bibliographic Data*.)

15-17 Place of publication, production, or execution

For example:
pk# = Pakistan
cau = California (US)

(For a full list of codes used in these positions, see the *MARC Code List for Countries*.)

18-21 Illustrations (up to 4 codes):

# = no illustrations
a = illustrations
b = maps
c = portraits
d = charts
e = plans
f = plates
g = music
h = facsimiles
i = coats of arms
j = genealogical tables
k = forms
l = samples
m = phonodisc, phonowire, etc.
o = photographs
p = illuminations
| = no attempt to code

22 Target audience:

# = unknown or not specified
a = preschool
b = primary
c = pre-adolescent
d = adolescent
e = adult
f = specialized
g = general
j = juvenile
| = no attempt to code

23 Form of item:

# = none of the following
a = microfilm

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b = microfiche
c = microopaque
d = large print
f = braille
r = regular print reproduction
s = electronic
| = no attempt to code

24-27 Nature of contents (up to 4):
# = no specified nature of contents
a = abstracts/summaries
b = bibliographies (is one or contains one)
c = catalogs
d = dictionaries
e = encyclopedias
f = handbooks
g = legal articles
i = indexes
j = patent document
k = discographies
l = legislation
m = theses
n = surveys of literature
o = reviews
p = programmed texts
q = filmographies
r = directories
s = statistics
t = technical reports
u = standards/specifications
v = legal cases and notes
w = law reports and digests
z = treaties
| = no attempt to code

28 Government publication:
# = not a government publication
i = international intergovernmental
f = federal/national
a = autonomous or semi-autonomous component
s = state, provincial, territorial, dependent, etc.
m = multistate
c = multilocal
l = local
z = other type of government publication
o = government publication -- level undetermined
u = unknown if item is government publication
| = no attempt to code

29 Conference publication:
0 = not a conference publication
1 = conference publication

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| = no attempt to code

30 Festschrift:
0 = not a festschrift
1 = festschrift
| = no attempt to code

31 Index:
0 = no index
1 = index present
| = no attempt to code

32 Undefined (since 1990) (Earlier records may contain the values 0 or 1)
= Undefined
| = no attempt to code

33 Literary form:
0 = not fiction (not further specified)
1 = fiction (not further specified)
c = comic strips
d = dramas
e = essays
f = novels
h = humor, satires, etc.
i = letters
j = short stories
m = mixed forms
p = poetry
s = speeches
u = unknown
| = no attempt to code

34 Biography:
# = no biographical material
a = autobiography
b = individual biography
c = collective biography
d = contains biographical information
| = no attempt to code

35-37 Language:
A three-letter code. For example: eng fre ger spa rus ita

(For a full list of codes used in these positions, see the *MARC Code List for Languages*.)

38 Modified record:
# = not modified
x = missing characters (because of characters unavailable in MARC character set)
s = shortened
d = "dashed-on" information omitted
r = completely romanized/printed cards in script
o = completely romanized/printed cards romanized
| = no attempt to code

39 Cataloging source:

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# = national bibliographic agency
c = cooperative cataloging program
d = other sources
u = unknown
| = no attempt to code

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Appendix C. Materials Considered

Exhibit/ Appendix	Description
Ex. 1004	Kempisty, D. M., Xing, Y., & Racz, L. (Eds.). (2018). *Perfluoroalkyl Substances in the Environment: Theory, Practice, and Innovation*, Taylor & Francis Group, Environmental and Occupational Health Series, 2018. ("Kempisty")
Appx. 1004A	Preview of Kempisty, available from the publisher at https://api.taylorfrancis.com/content/books/mono/download?identifierName=doi&identifierValue=10.1201/9780429487125&type=googlepdf.
Appx. 1004B	Publisher webpage for Environmental Science Titles, archived by Internet Archive on August 26, 2018, available at https://web.archive.org/web/20180826202517/https://www.crcpress.com/environmental-science.
Appx. 1004C	Library of Congress MARC record for the print version of Kempisty, available at https://lccn.loc.gov/2018011967 (select "MARC record"); OCLC FirstSearch record for the print version of Kempisty (subscription required for access); and the bibliographic and MARC records for the print version of Kempisty, available at https://unh.primo.exlibrisgroup.com/permalink/01USNH_UNH/1gs8dob/alma991014401959805221 from the online catalog of the University of New Hampshire Library.
Appx. 1004D	Publisher webpage for the eBook version of Kempisty, available at https://www.taylorfrancis.com/books/edit/10.1201/9780429487125/perfluoroalkyl-substances-environment-yun-xing-leeann-racz-david-kempisty.
Appx. 1004E	OCLC FirstSearch records for the eBook version of Kempisty (subscription required for access), including OCLC records 1049150067, 1049914424, 1053862345.
Appx. 1004F	Bibliographic and MARC records for the eBook version of Kempisty, available at https://skyline.ucdenver.edu:443/record=b4238690 from the online catalog of the University of Colorado Denver, Auraria Library.
Ex. 1005	Mueller, R., & Yingling, V. (2018, March). Remediation Technologies and Methods for per-and polyfluoroalkyl substances (PFAS). ITRC Fact Sheet. 12 pages. ("Mueller")
Appx. 1005A	Copy of Mueller, archived by Internet Archive on December 18, 2018, available at https://web.archive.org/web/20200417054249/https://pfas-1.itrcweb.org/fact-sheets/

Appx. 1005B	ITRC PFAS webpage archived by Internet Archive on November 17, 2017, available at https://web.archive.org/web/20171117143016/https:/pfas-1.itrcweb.org/.
Appx. 1005C	PFAS Fact Sheets webpage archived on "NOV 17 2017," available at https://web.archive.org/web/20171117143547/http://pfas-1.itrcweb.org/fact-sheets.
Appx. 1005D	The post of Johnsie Lang, dated "November 20, 2017," on the Facebook of the North Carolina Stop GenX and Nafion in Water, available at https://www.facebook.com/groups/353457201741568/permalink/384656191955002/?rdid=dvFBIZQkBc3VNEQV#.
Appx. 1005E	The PFAS Fact Sheets webpage archived by Internet Archive on "JAN 19 2018," available at https://web.archive.org/web/20180119214445/http:/pfas-1.itrcweb.org/fact-sheets/.
Appx. 1005F	ITRC Factbook announcement on "March 20, 2018" of the release of three new PFAS fact sheets, available at https://www.facebook.com/profile/100063488651502/search/?q=ITRC%20thrilled%20to%20release%20three%20new%20PFAS.
Appx. 1005G	The webpage of "Interstate Technology & Regulatory Council (ITRC) News" archived by Internet Archive on July 19, 2019, available at http://web.archive.org/web/20190719230817/http://www.itrcweb.org/About/News.
Appx. 1005H	ITRC announcement of the release of three new PFAS fact sheets on the PFAS website, archived by Internet Archive on August 25, 2019, available at http://web.archive.org/web/20190825090656/https://itrcweb.org/About/three-new-PFAS-fact-sheets.
Appx. 1005I	MERIT Lab blog post of March 21, 2018, available at https://www.meritlabs.com/blog/2018/3/21/just-the-facts-pfas-fact-sheets-published-by-itrc.
Appx. 1005J	A post by Johnsie Lang on March 21, 2018, on the North Carolina Stop GenX and Nafion in Water Facebook, available at https://www.facebook.com/groups/353457201741568/permalink/433432190410735/?rdid=QyM5tAdVl2DQ1qi6#.
Appx. 1005K	A "March 31, 2018" post on the Clean Cape Fear Facebook about the ITRC release of three new PFAS fact sheets, available at https://www.facebook.com/CleanCapeFear/posts/pfbid02iCEmLpNueUjCGQn6D1BC1Bk3kRdu3pMsRCHSP9mSFpAw5NPKhK7PjX73ci8afXabl?rdid=vKLImBdZoRUzEvSw#.

Appx. 1005L	April 1, 2018 issue of TechDirect of the US Environmental Protection Agency that publicized the release of Mueller, available at from https://clu-in.org/download/newsltrs/techdirect/td042018.pdf.
Appx. 1005M	The Record of Meeting of the Groundwater Issues Subcommittee of the Texas Groundwater Protection Committee that I personally located, identified, and obtained from https://tgpc.texas.gov/gwissues/minutes/TGPC_GWIminutes21Mar2018.pdf.
Appx. 1005N	Steele, M., Griffith, C., & Duran, C. (2018). Monthly Variations in Perfluorinated Compound Concentrations in Groundwater. *Toxics*, *6*(3), 56. https://doi.org/10.3390/toxics6030056.
Appx. 1005O	ITRC PFAS Fact Sheets webpage archived by Internet Archive on August 19, 2018, available at https://web.archive.org/web/20180819033439/https://pfas-1.itrcweb.org/fact-sheets/.
Appx. 1005P	Copy of Mueller, archived by Internet Archive on December 18, 2018, available at https://web.archive.org/web/20181218193617/https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf.
Appx. 1005Q	PFAS Fact Sheets webpage archived by Internet Archive on March 5, 2020, available at https://web.archive.org/web/20200305013935/https://pfas-1.itrcweb.org/fact-sheets/.
Appx. 1005R	Copy of Mueller, archived by Internet Archive on February 16, 2020, available at https://web.archive.org/web/20200216215831/https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf.
Appx. 1005S	PFAS Fact Sheets webpage, archived by Internet Archive on April 17, 2020, available at https://web.archive.org/web/20200417054249/https://pfas-1.itrcweb.org/fact-sheets/.
Ex. 1006	McDougall, G. J. (1991, April). The physical nature and manufacture of activated carbon. *Journal of the Southern African Institute of Mining and Metallurgy*, *91*(4), 109-120. ("McDougall")
App. 1006A	Copy of McDougall, available at https://journals.co.za/doi/pdf/10.10520/AJA0038223X_2042 from the publisher's website.

App. 1006B	Library copy of McDougall, obtained from the University of Wisconsin-Madison General Library System.
App. 1006C	Bibliographic and MARC records for *Journal of the Southern African Institute of Mining and Metallurgy* that contains McDougall, available at https://search.library.wisc.edu/catalog/999543521702121 from the online catalog of the University of Wisconsin-Madison General Library system.
App. 1006D	Citations to McDougall, obtained from Google Scholar.
Ex. 1007	Mimna, R. (January 30, 2017). "Granular Activated Carbon: A Proven Solution for PFC Removal," Calgon Carbon Presentation, 30 Jan. 2017, https://www.calgoncarbon.com/app/uploads/PFC-Webinar-Presentation-1-30-17.pdf. ("Mimna")
Appx. 1007A	Copy of Mimna archived by Internet Archive on September 18, 2017, available at https://web.archive.org/web/20170918144158/http://www.calgoncarbon.com/wp-content/uploads/2016/02/PFC-Webinar-Presentation-1-30-17.pdf.
Appx. 1007B	Web page for "Perfluorinated Compounds (PFCs), Perfluorooctane Sulfonate (PFOS) and Perfluorooctanoic Acid (PFOA)," archived by Internet Archive on February 22, 2017, available at https://web.archive.org/web/20170222172255/http://www.calgoncarbon.com/PFOA/, and a screen capture of the top of Mimna archived by Internet Archive on September 18, 2017, available at https://web.archive.org/web/20170918144158/http://www.calgoncarbon.com/wp-content/uploads/2016/02/PFC-Webinar-Presentation-1-30-17.pdf
Ex. 1009	Appleman, T. D., Higgins, C. P., Quiñones, O., Vanderford, B. J., Kolstad, C., Zeigler-Holady, J. C., & Dickenson, E. R. (2014, March). Treatment of poly-and perfluoroalkyl substances in US full-scale water treatment systems, *Water Research*, 51, 246-255. ("Appleman")
Appx. 1009A	Library copy of Appleman, obtained from the National Library of Medicine.
Appx. 1009B	Bibliographic and MARC records for *Water Research*, available at https://catalog.nlm.nih.gov/permalink/01NLM_INST/7b9jl0/alma99381403406676 from the online catalog of the National Library of Medicine.
Appx. 1009C	Copyright registration record for vol. 51 of *Water Research*, available at https://publicrecords.copyright.gov/detailed-record/voyager_27205734 from the Public Records System of the US Copyright Office.
Appx. 1009D	PubMed record for Appleman, available at https://pubmed.ncbi.nlm.nih.gov/24275109/.

Appx. 1009E	PubMed webpage archived by Internet Archive on October 10, 2013, available at https://web.archive.org/web/20131010054145/http://www.ncbi.nlm.nih.gov/pubmed.
Appx. 1009F	Webpage for vol. 51 of *Water Research*, archived by Internet Archive on April 6, 2014, available at https://web.archive.org/web/20140406113630/http://www.sciencedirect.com/science/journal/00431354/51.
Appx. 1009G	Citation records, obtained from Google Scholar.
EX-1011	Cannon, F. S., Snoeyink, V. L., Lee, R. G., Dagois, G., & DeWolfe, J. R. (1993, March). Effect of Calcium in field-spent GACs on pore development during regeneration, *Journal-American Water Works Association*, *85*(3), 76-89. ("Cannon")
Appx. 1011A	Copy of Cannon, obtained from JSTOR at https://www.jstor.org/stable/41294338.
Appx. 1011B	Wiley webpage for vol. 85, issue 3 of *Journal AWWA*, available at https://awwa.onlinelibrary.wiley.com/toc/15518833/1993/85/3.
Appx. 1011C	Bibliographic record for Cannon, available at https://wrlc-cu.primo.exlibrisgroup.com/permalink/01WRLC_CAA/11ramo0/cdi_crossref_primary_10_1002_j_1551_8833_1993_tb05959_x from the online catalog of the Catholic University of America Libraries, and the top of the JSTOR record for Cannon.
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Perfluoroalkyl Substances in the Environment

Theory, Practice, and Innovation



Edited by
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Foreword

Addressing the issues that poly- and perfluoroalkyl substances (PFAS) raise will be an enduring challenge for our generation of environmental engineers and chemists. Other classes of trace organic chemicals, such as pharmaceuticals and personal care products, may raise concerns wherever and whenever wastewater impacts our aquatic environment; however, to quote Calvin Coolidge, "Nothing in this world can take the place of persistence." In this manner, PFAS seem to have cornered the market.

It is not just the extreme persistence of their multiple carbon-fluorine bonds that have captured our attention. The unique surfactant behavior of many PFAS, particularly those associated with the use of aqueous film-forming foams (AFFFs), which are the focus of this book, is leading to many complications as we consider the environmental fate and transport of these otherwise relatively mobile substances. Moreover, as much as one might want to simplify the PFAS world into just perfluorooctanoate (PFOA) and perfluorooctane sulfonate (PFOS), the chemistry, and everything that goes along with it, is much more complex than I think many of us imagined when we first started to study these chemicals. It is not just about the anions—there are neutrals and cations too! Indeed, developing multiple lines of analytical evidence to demonstrate that one has accurately accounted for the diverse chemistry of PFAS present at AFFF-impacted sites is a current focus of many research groups. Though I am a reluctant analytical chemist, I seem to be more often suggesting that we simply embrace the complexity of the PFAS chemistry (as best we can) in the environment if we want any hope of making sense of the complicated web of precursors and products.

Of course, it is not just the chemistry of the PFAS themselves and what happens to them in the environment that is so complex: the multitude of uses of PFAS has also led to considerable confusion, or at least complexity, related to sources of exposure. Folks can get confused pretty quickly when you explain that the chemicals that they have used to keep stains off of their carpets and clothing are kissing cousins of those that were used to put out fuel fires and are now contaminating their drinking water supply. Never mind the fact that we are really only starting to scratch the surface with respect to understanding their toxicity. Nevertheless, the exposures that are happening and have happened are real, and the public is left wanting at least an explanation, if not a solution, to this complicated problem.

This leads me to why *this* book and why *now*. On the whole, this collection of information is just what is needed at this point in time. In November 2016 the US Environmental Protection Agency issued its long-anticipated health advisories for PFOS and PFOA in US drinking water supplies. Since then, there has been an onslaught of media coverage of the issue, with local press outlets reporting on every new groundwater plume. As a result, many nonacademicians are having to quickly come up to speed on a variety of topics and issues specific to PFAS. For those who have not been directly engaged with the scientific discussions on PFAS for the past 15 years, this could seem a very daunting task. What this book provides is

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information not just on the complicated chemistry and nomenclature, but also on the toxicology, regulatory approaches, analytical chemistry, and perhaps most importantly, the diverse ways in which environmental engineers are trying to remediate PFAS. Though it is downright impossible to capture every PFAS issue, particularly in this rapidly expanding research environment, Drs. Kempisty, Racz, and Xing have included a broad array of perspectives and cover an even broader array of PFAS topics. At the very minimum, this book will serve as an excellent gateway to the wild world of PFAS. Whether you are a federal, state, or local government official being thrust into the PFAS world, a student wanting to learn what all the fuss is about, or simply a member of the public trying to understand just why these chemicals are so difficult to treat, I would encourage you to engage with this book as much as you can. Take notes. Look up references. Follow your passion.

Good luck!

Christopher Higgins
Department of Civil and Environmental Engineering
Colorado School of Mines, Golden, Colorado

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Editors

David M. Kempisty is a bioenvironmental engineer in the US Air Force with more than 20 years of active duty experience. He is currently serving in the grade of lieutenant colonel. In his present assignment, Dave is the chief of medical chemical, biological, radiological, and nuclear operations in the Surgeon's Directorate at the North American Aerospace Defense Command and US Northern Command at Peterson Air Force Base, Colorado. Previously, Dave was an assistant professor and the director for the Graduate Environmental Engineering and Science Program in the Department of Systems Engineering and Management at the Air Force Institute of Technology (AFIT). In this capacity, he and his students conducted research into perfluorinated alkyl substances (PFAS), to include both toxicological and remedial aspects of PFAS. He's been recognized for this research with the 2017 US Environmental Protection Agency's Office of Research and Development bronze medal for water infrastructure and technologies research. Previous to AFIT, Dave earned his PhD from the University of Colorado–Boulder in civil engineering. His dissertation involved investigated improved PFAS adsorption to granular activated carbon. Dave is a licensed professional engineer in the state of Michigan. He earned his BS degree from Michigan Technological University and his MS degree from AFIT. In addition to research and policy associated with PFAS, other areas of interest include environmental health issues, sustainability initiatives, and improving defense support to civil authorities after natural and man-made disasters. Dave is active in multiple professional societies, has led and presented at international and domestic conferences, and has authored many peer-reviewed and trade journal articles.

Yun Xing is a postdoctoral research scholar at the Air Force Institute of Technology (AFIT). She earned a BS in biochemical engineering from Tianjin University, China, and a PhD in bioengineering from the Georgia Institute of Technology. Her previous work experiences include cancer nanotechnology research at Stanford University and research on polyfluorinated alkyl substance remediation and isomer cytotoxicity at AFIT. Her areas of interest include bionanotechnology, toxicity of emerging materials, pollutants of emerging concern, and characterization of the effects and fate of biocontaminants in the environment. She has authored 19 peer-reviewed journal articles and 5 book chapters with more than 1500 citations. She is a member of several professional associations and has served as a reviewer for a number of journals, including the *Analyst*, *ACS Nano*, and *Applied and Environmental Microbiology.*

LeeAnn Racz is a bioenvironmental engineer in the US Air Force, having served at bases across the globe. She currently serves as commander of the 1st Special Operations Aerospace Medicine Squadron at Hurlburt Field, Florida. Previous assignments have included chief of consultative services at the US Air Force School of Aerospace Medicine, as well as assistant professor of environmental engineering and director of the Graduate Environmental Engineering and Science Program in the

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Department of Systems Engineering and Management at the Air Force Institute of Technology. She presently holds the rank of lieutenant colonel. She is a licensed professional engineer, certified industrial hygienist, and board-certified environmental engineer. She earned a BS in environmental engineering from California Polytechnic State University, San Luis Obispo; an MS in biological and agricultural engineering from the University of Idaho; and a PhD in civil and environmental engineering from the University of Utah. Her areas of interest include characterizing the fate of chemical warfare agents and pollutants of emerging concern in the natural and engineered environments, as well as environmental health issues, and using biological reactors to treat industrial waste. She has authored dozens of refereed journal articles, conference proceedings, magazine articles, and presentations, and edited four handbooks. She is a member of several professional associations and honor societies and has received numerous prestigious teaching and research awards.

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Contributors

Abinash Agrawal
Wright-State University
Dayton, Ohio

David F. Alden
Tersus Environmental
Wake Forest, North Carolina

Janet K. Anderson
Integral Consulting Inc.
San Antonio, Texas

R. Hunter Anderson
Air Force Civil Engineer Center
Joint Base San Antonio–Lackland
San Antonio, Texas

John Archibald
Tersus Environmental
Grimsby, Ontario, Canada

John Berry
Emerging Compounds Treatment Technologies, Inc.
Bedford, New Hampshire

Gary M. Birk
Tersus Environmental
Wake Forest, North Carolina

Miranda Brannon
US Air Force
Office of the Surgeon General
Falls Church, Virginia

Joseph M. Braun
Department of Epidemiology
Brown University
Providence, Rhode Island

Carl E. Brown
Environment and Climate Change Canada
Ottawa, Ontario, Canada

Robert C. Buck
The Chemours Company
Wilmington, Delaware

Justin Burgess
Environment and Climate Change Canada
Ottawa, Ontario, Canada

Gabriel Cantu
US Air Force School of Aerospace Medicine
Wright-Patterson Air Force Base
Dayton, Ohio

Stephanie Carr
Calgon Carbon Corporation - A Kuraray Company
Moon Township, Pennsylvania
Retired

Bharat Chandramouli
SGS AXYS
Sidney, British Columbia, Canada

Michelle Crimi
Clarkson University
Potsdam, New York

Kavitha Dasu
Battelle
Norwell, Massachusetts

Eric R. V. Dickenson
Southern Nevada Water Authority
Henderson, Nevada

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Sean M. Dyson
Air Force Institute of Technology
Wright-Patterson Air Force Base
Dayton, Ohio

Thomas M. Holsen
Clarkson University
Potsdam, New York

David M. Kempisty
US Northern Command
Peterson Air Force Base
Colorado Springs, Colorado

David Klein
Department of Pathology and Laboratory Medicine
Brown University
Providence, Rhode Island

Stephen H. Korzeniowski
BeachEdge Consulting, LLC
Media, Pennsylvania

G. Cornell Long
Air Force Civil Engineer Center
Joint Base San Antonio–Lackland
San Antonio, Texas

John Matthis
Calgon Carbon Corporation - A Kuraray Company
Moon Township, Pennsylvania

Marc A. Mills
US Environmental Protection Agency
Cincinnati, Ohio

Brandon Newman
Amec Foster Wheeler Environment & Infrastructure, Inc.
Portland, Maine

Michael G. Nickelsen
Emerging Compounds Treatment Technologies, Inc.
Rochester, New York

Blossom N. Nzeribe
Clarkson University
Potsdam, New York

Martial Pabon
Dynax Corporation
Pound Ridge, New York

Ronald C. Porter
Noblis
San Antonio, Texas

Penelope Rice
Food and Drug Administration
College Park, Maryland

Christopher T. Schmidt
US Air Force
Little Rock Air Force Base
Little Rock, Arkansas

Marilyn M. Sinnett
Emerging Compounds Treatment Technologies, Inc.
Portland, Maine

Richard J. Stewart
Ziltek Pty Ltd.
Thebarton, South Australia, Australia

Gunnar Stratton
Clarkson University
Potsdam, New York

John E. Stubbs
Air Force Institute of Technology
Wright-Patterson Air Force Base
Dayton, Ohio

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Selma M. Thagard
Clarkson University
Potsdam, New York

Edgard M. Verdugo
Southern Nevada Water Authority
Henderson, Nevada

Steve Via
American Water Works Association
Washington, DC

Konstantin Volchek
Environment and Climate Change Canada
Ottawa, Ontario, Canada

Andrew J. Wagner
US Air Force
Joint Base Andrews
Camp Springs, Maryland

Bo Wang
Wright-State University
Dayton, Ohio

Steven Woodard
Emerging Compounds Treatment Technologies, Inc.
Portland, Maine

Yun Xing
Air Force Institute of Technology
Dayton, Ohio

Yuan Yao
Environment and Climate Change Canada
Ottawa, Ontario, Canada

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Introduction

Per- and polyfluorinated alkyl substances (PFAS), often referred to as per- (and poly-) fluorinated compounds, have been used for years, providing desirable properties to many everyday products we use. From nonstick cookware to stain-resistant coatings on clothing and upholstery and even in our personal care products, perfluorinated compounds are common in our everyday environment. Particularly in the aviation and firefighting communities, perfluorinated chemicals have been incorporated into the agents used to fight hydrocarbon-based fires. A pivotal event highlighting the need for the best firefighting agent possible was a shipboard fire aboard the USS *Forrestal* in 1967. In the end, 134 sailors lost their lives, countless more were injured, and the loss of aircraft and ship damage was substantial. Lack of standardization, inadequate firefighting procedures, and a lack of capabilities for the firefighting operations were identified as major lessons learned from the incident. Improvements to the firefighting agents were made as a result of the tragic fire, and the use of aqueous film-forming foam (AFFF) containing perfluorinated compounds gained popularity over similar protein-based foams. AFFF use has continued, and although effective in extinguishing Class B hydrocarbon-based fires, perfluorinated compounds also have negative aspects associated with them; most notably, exposure has been linked to adverse health effects in humans. Other concerns include the long biological half-life in humans (on the order of years) and their persistence in the environment for periods of years and tens of years after application. Soluble in water, PFAS are now being identified in drinking water sources. Research continues to determine adverse health effect contributions due to various exposure routes, but nonetheless, a remedy for drinking water sources is needed.

This book discusses the various challenges of PFAS in our environment today. First, the diverse number of per- and polyfluorinated compounds is vast. In order to establish some kind of boundary condition for the scope of this book, discussion is primarily centered around PFAS contained in the firefighting and aviation community. Some discussion is involved outside of this scope, but it is limited. Section I, "Introduction and Analysis," provides background of the issues we face today. Chapter 1 sheds light on the history, nomenclature, and chemistry associated with the compounds in the PFAS suite. Discussion on the different types of firefighting foams and the military specification requirements is also covered in this chapter.

There are analytical challenges with reaching statistically reproducible detection limits. In addition to the detection of the trace concentrations, the ubiquitous presence of potentially cross-contaminating Teflon and other fluorinated compounds in an analytical laboratory make the baseline analytical work center requirements more stringent than those for other more common environmental contaminants. Chapters 2 and 3 provide an overview of the various detection methods available to determine PFAS concentrations in various environmental media.

Regulatory perspectives, discussed in Section II, are another dynamic landscape, and navigating them presents another challenge. The US Environmental Protection

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Agency released a health advisory in 2016 stating that water with a combined perfluorooctane sulfonate (PFOS) and perfluorooctanoate (PFOA) concentration under 70 parts per trillion would result in no adverse health effects. The language went on to state that the advisory was not to be used for regulatory purposes, but with the lack of a maximum contaminant level for any perfluorinated substance, the health advisory has become a de facto regulatory limit. Further complicating things, individual states have established different allowable limits of PFAS to finished drinking water. Although not yet passed, New Jersey's Drinking Water Quality Institutes has recommended setting a limit of 14 parts per trillion for PFOA and 13 parts per trillion for another perfluorinated substance, the nine-carbon perfluorononanoic acid. Different concentrations and different regulatory language (maximum contaminant limits, health advisories, and summed concentrations) complicate remedial goals, drinking water standards, and risk communication language for agencies working in multiple states, such as often occurs in the aviation and defense communities. Chapters 4 through 7 explore these regulatory perspectives and give practical recommendations for operating with these changing requirements.

Section III discusses toxicological profiles, which are still being developed and advanced. Chapter 8 is a comprehensive review of the human health risks associated with exposure to PFAS. Toxicity with early life exposures is discussed further in Chapter 9. Isomers are another suite of PFAS that deserve toxicological investigation; depending on the manufacturing process, up to 30% of the finished PFAS product can be in an isomer form. The toxicological profile for these isomers is different than that of their straight-chained counterparts and is discussed in Chapter 10.

Determining the true toxicity, reacting to the changing regulatory landscape, and being aware of analytical and sampling challenges all present challenges to the community responsible for developing a PFAS management strategy. Perhaps the most challenging aspect of dealing with legacy PFAS contamination is the remediation of impacted soil and water. Remedial treatment objectives influenced by changing regulatory standards and advisories can make identifying efficient and effective remediation methods difficult. A considerable portion of this book, Section IV, discusses methods employed with varying degrees of success. The remediation of soil media and short-chain PFAS compounds are covered in Chapters 17 and 21. Conventional approaches for treating PFAS-impacted water, such as activated carbon and its reactivation of spent media, are discussed in Chapters 11 and 13. More novel approaches employing redox processes are covered in Chapter 12. Additionally, combinations of the treatment technologies, such as an advanced oxidization process with activated carbon and ion exchange and activated carbon polishing, are covered in Chapters 14 and 16.

Although perfluorinated compounds are a daunting challenge across multiple fronts, there is a plethora of ongoing work to resolve these challenges. This book explores the challenges across the topical areas of regulation and management, toxicology, environmental remediation, and analytical sampling and analysis. The text also provides insight into the great amount of work progressing to address the challenges. We hope the readers find this text helpful in understanding the complexities

to a greater level. Our goal for this text is to have the analytical chemist and others practicing in the environmental engineering field appreciate PFAS regulatory issues and to increase understanding of PFAS toxicology. At the same time, an equal motivation for assembling this effort is to leave readers with an optimistic view of the meritorious efforts underway and the promising advances being seen across the PFAS spectrum.

David M. Kempisty
Yun Xing
LeeAnn Racz

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Section 1

Introduction and Analysis



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1 Fluorosurfactants in Firefighting Foams
Past and Present

Stephen H. Korzeniowski, Robert C. Buck, David M. Kempisty, and Martial Pabon

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1.1 INTRODUCTION

Fluorosurfactants are a unique class of surfactants whose properties originate from the substitution of hydrogen with fluorine along the carbon backbone that makes up the hydrophobic part of a surfactant (Taylor 1999; Kissa 2001; Pabon and Corpart 2002; Buck et al. 2012). Fluorinated surfactants have been commercially available since the 1950s. The most common commercial fluorosurfactants contain a perfluoroalkyl moiety, $F(CF_2)_n^-$, bound to a spacer group that is connected to a cationic, anionic, amphoteric, or nonionic functional group (Figure 1.1).

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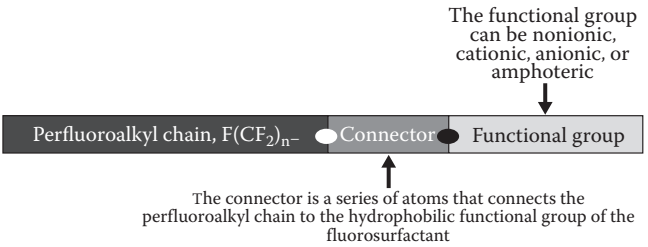


FIGURE 1.1 Fluorosurfactant schematic.

The carbon-fluorine bond is very strong. The atomic volume and ionic radius of fluorine shield carbon from a chemical attack. Thus, the perfluoroalkyl moiety is very rigid compared with the analogous hydrocarbon equivalent. The rigidity, ionic character, and large molecular volume of the perfluoroalkyl moiety drive the unique adsorption and aggregation behavior of fluorosurfactants. Moreover, the perfluoroalkyl functional moiety has very low surface energy and is both hydrophobic and oleophobic. Therefore, in both aqueous and hydrocarbon solutions, fluorosurfactants have a driving force to move to the air–liquid interface. The high chemical and thermal stability, surface tension lowering, and efficacy at low concentration (e.g., 100 parts per million [ppm]) have led to the widespread use of fluorosurfactants where traditional hydrocarbon or silicone surfactants cannot achieve the required performance. In parallel to a high number of other uses, these and other properties have made fluorosurfactants useful and valuable in aqueous film-forming foam (AFFF), which is used to extinguish high hazard hydrocarbon and polar solvent fires.

1.2 FLUOROSURFACTANT CHEMISTRY

The two primary commercial production processes employed to synthesize the perfluoroalkyl moiety that is used to make fluorosurfactants are electrochemical fluorination (ECF) and the fluorotelomer process (Figure 1.2) (Taylor 1999; Kissa 2001; Pabon and Corpart 2002; Buck et al. 2011, 2012). Among the first commercially available fluorosurfactants were perfluoroalkyl sulfonates (PFSAs) (e.g., perfluorooctane sulfonate [PFOS], $C_8F_{17}SO_3^-$) and perfluoroalkyl carboxylic acids (PFCAs) (e.g., perfluorooctanoic acid [PFOA], $C_7F_{15}COOH$), manufactured using the ECF process (Taylor 1999; Kissa 2001; Pabon and Corpart 2002; Buck et al. 2012). Subsequently, fluorosurfactants derived from the fluorotelomer process were introduced some time later (10–15 years after ECF products). A wide array of fluorosurfactants containing typical surfactant terminal functionalities (e.g., anionic, cationic, amphoteric, and nonionic) have been made and used (Taylor 1999; Pabon and Corpart 2002; Buck et al. 2012).

1.2.1 Electrochemical Fluorination (ECF) Process

A general synthetic scheme for synthesis of ECF-based products is shown in Figure 1.2(a). ECF accomplishes the substitution of hydrogen with fluorine by electrolysis of

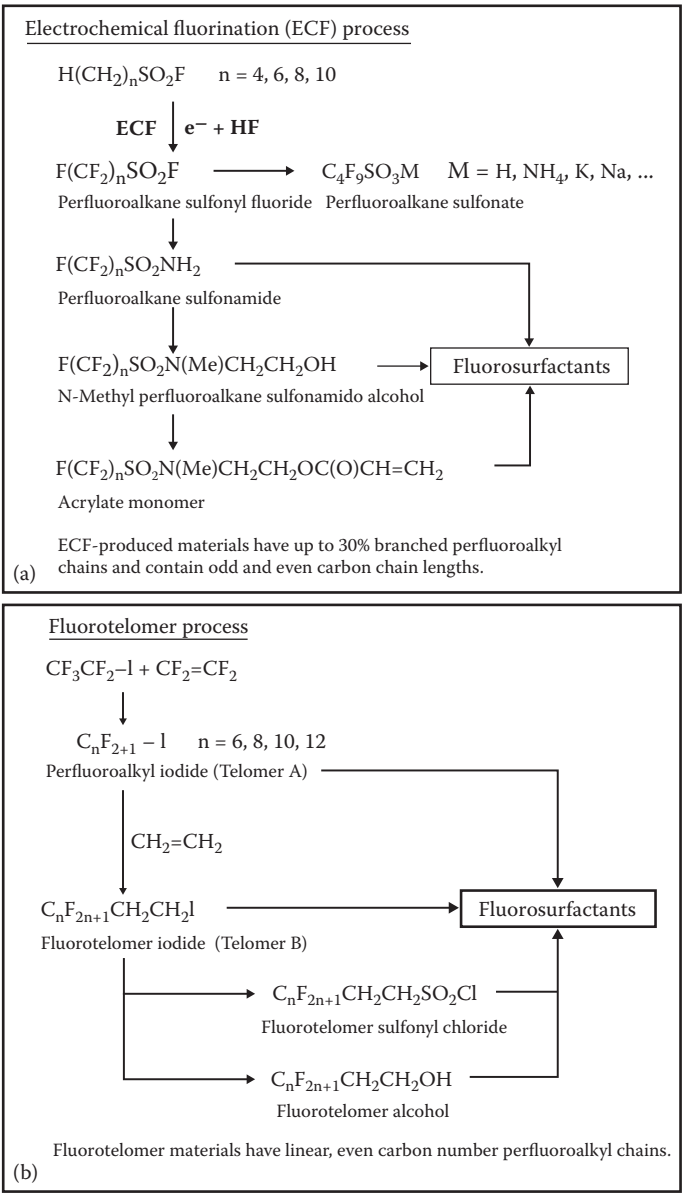


FIGURE 1.2 The two primary commercial processes to synthesize the perfluoroalkyl moiety: (a) the ECF process and (b) the telomerization process.

alkane sulfonyl chlorides or fluorides of the type $H(CH_2)_{n+1}SO_2X$, where X is Cl or F in a hydrogen fluoride (HF) solution (Alsmeyer et al. 1994). ECF is a free radical process yielding perfluoroalkane sulfonyl fluoride molecules with both linear and branched perfluoroalkyl chains and chain length homologues. For example, an eight-carbon starting material (e.g., octane sulfonyl fluoride) would yield a perfluoroalkane

sulfonyl fluoride mixture composed of roughly 80% linear and 20% branched perfluoroalkyl chains and odd and even three- to eight-carbon-chain-length homologues. It is also worth noting that the electrolysis of alkane sulfonyl fluoride may also yield a homologous series of perfluorocarboxylic acids as impurities in percentage amounts. The perfluoroalkane sulfonyl fluoride is then reacted to create raw materials that can be used to make fluorosurfactants. Note that the "connector" in the ECF chemistry is a sulfonamidoalkyl group, $–SO_2N(R)CH_2CH_2–$, as shown in Figure 1.2(a). The historic production using ECF manufactured long-chain perfluorohexanesulfonyl (C6), perfluorooctane sulfonyl (C8), and perfluorodecane sulfonyl (C10) products, with the majority being C8, of which PFOS is the most well-known product. When 3M, the major global ECF manufacturer, withdrew from PFOS, PFOA, and related chemistry in 2000–2002, it transitioned to short-chain, C4, perfluorobutane sulfonyl chemistry. The reader is reminded that the short-chain (C4, perfluorobutane sulfonyl) ECF-based fluorosurfactants are not used in AFFF because to date, no AFFF formulation containing them has met required firefighting specifications.

1.2.2 Fluorotelomer Process

The second route most widely practiced for the synthesis of a perfluoroalkyl moiety is the fluorotelomer process (Taylor 1999; Pabon and Corpart 2002; Buck 2011, 2012). In the fluorotelomer process, tetrafluoroethylene (TFE) is oligomerized with perfluoroethyl iodide (PFEI) (CF_3CF_2I), yielding a mixture of exclusively linear, even-carbon-number perfluoroalkyl iodides that are commonly called Telomer A. The fluorotelomer chain length could range up to n = 20, but was primarily n = 6, 8, 10, and 12, with 6 and 8 predominant in commercial products. Ethylene is then inserted into Telomer A to make Telomer B, perfluoroalkyl ethyl iodide raw material, which may then be reacted to make fluorotelomer alcohol raw material and/or acrylate/methacrylate monomers. These raw materials or other types are then used to manufacture fluorosurfactants, as shown in Figure 1.2(b). Historic fluorotelomer-based fluorosurfactants contained mainly n = 6 (C6) and n = 8 (C8) perfluoroalkyl moieties ($C_nF_{2n+1}–$), with the majority being C6. As the major global fluorotelomer manufacturers have fulfilled their commitment to no longer make long-chain C8 chemistry (USEPA 2010), the short-chain C6 fluorotelomer-based fluorosurfactants are presently made and used.

1.3 LONG CHAIN AND SHORT CHAIN: TERMINOLOGY AND STRUCTURES

The historic use of fluorosurfactants, including their use in firefighting foams, has partially contributed to the widespread presence of PFOS and PFOA, aka long-chain perfluoroalkyl substances, in the environment. The discovery of environmental contamination began with the work by Moody and Field (1999) and continues today (Anderson et al. 2016). In 2000, 3M announced that it would exit from C8 manufacture, including PFOS and PFOA and all commercial products related to them (3M 2000). In 2006, eight major fluorochemical global manufacturers committed to exiting the manufacture and use of PFOA, longer-chain homologues, and

related substances by the end of 2015 (USEPA 2010). Concurrent with these actions, the Stockholm Convention concluded that PFOS and PFOA meet the criteria to be classified as persistent, bioaccumulative, and toxic (PBT) substances (Stockholm Convention 2009; Matthies et al. 2016).

The PBT properties of the long-chain perfluoroalkyl substances, PFOS and PFOA, and the related long-chain raw materials and commercial products were the driving force behind manufacturers ceasing manufacture of these long-chain chemistries and transitioning to short-chain chemistry. To continue to deliver the unique and valuable properties of fluorosurfactants in uses where they are necessary for performance and to eliminate long-chain products, manufacturers have developed additional short-chain alternatives with fewer fluorinated carbons. The data on these alternatives are admittedly less than the data on their long-chain counterparts, but still exist in a considerable measure. Available data on short-chain fluorotelomer-based raw materials, products, and degradation products show that the short-chain alternatives have a more favorable environmental and human health profile (Environ 2014; Environ 2016; Buck 2015). Numerous short-chain products have been approved by regulators, and significant data are being required before the commercialization of new short-chain products. Therefore, today, short-chain ECF-based fluorosurfactants contain the perfluorobutane sulfonyl moiety, $C_4F_9SO_2$–, while fluorotelomer-based fluorosurfactants contain the perfluorohexylethyl moiety, $C_6F_{13}CH_2CH_2$–. Readers should note that there is a clear and distinct difference in what is considered long chain and short chain based on the origin chemistry and its connection to the corresponding perfluoroalkyl acid (PFAA) (Figure 1.3). Within the perfluoroalkane sulfonate (PFSA) family, all substances with a carbon chain length greater than or equal to six are considered long chain. In contrast, within the perfluorocarboxylic acid (PFCA) family, all substances with a carbon chain length greater than or equal to eight are considered long chain. This comes about because of the significant difference in bioaccumulation and toxicity properties between perfluorohexane sulfonate (PFHxS) and perfluorohexanoic acid (PFHxA), both called C6s, but with dramatically different properties (Gannon et al. 2011).

Long-chain		**Short-chain**
	Perfluoroalkane sulfonate PFSA $C_nF_{2n+1}SO_3(H)$	
n ≥ 6 PFDS, PFOS, PFHxS		n ≤ 5 PFBS
	Perfluoroalkyl carboxylate PFCA $C_{x-1}F_{2x-1}COOH$	
x ≥ 8 PFDA, PFNA, PFOA		x ≤ 7 PFHxA, PFPeA, PFBA

FIGURE 1.3 Long-chain versus short-chain PFAAs. (Adapted from Organisation for Economic Co-operation and Development, Portal on per and poly fluorinated chemicals, 2017. http://www.oecd.org/chemicalsafety/portal-perfluorinated-chemicals/aboutpfass/.)

Foam, fluorosurfactants, and firefighting have been inextricably linked for the past 50 years following the invention of AFFF in the 1960s. With the unique film-forming and surface tension properties provided by fluorosurfactants (Kissa 2001; Pabon and Corpart 2002), the category of AFFF was created approximately 50 years ago. This invention and subsequent application of fluorosurfactants for use in Class B foam concentrates forever changed the capability of firefighters worldwide to extinguish and rapidly control large-scale hydrocarbon liquid and polar solvent fires. This introductory chapter provides an overview of fluorosurfactant use in AFFF foams and concentrates. Subsequent chapters discuss challenges arising with the historic use of AFFF and the discovery of per- and poly-fluoroalkyl substances, PFAS, in the environment. Chapters explore issues associated with the remediation of impacted environmental media, particularly soil and water. The current regulatory landscape is also surveyed. The lack of published regulatory limits, and instead the presence of advisory limits, drives new decision logic regarding remedial options. Sampling and analysis considerations ensuring the integrity of analytical data at the low parts per trillion (ppt) range are discussed. Also, the ongoing development of the toxicological picture of PFAS is discussed in detail, as are efforts to understand the ecological impact of PFAS in the environment. The reader is also directed to a summary documenting the history and issues facing the use of AFFF prepared by the Fire Protection Association of Australia (FPAA 2017).

1.4 FIREFIGHTING FOAM BEFORE FLUOROSURFACTANTS

The need for and use of firefighting foams followed the introduction and wide-scale use of liquid hydrocarbons and later polar solvent fuels. Historically, water was used as the primary firefighting agent. However, water is more dense than liquid hydrocarbons and sinks, rendering it an ineffective bottom layer below a burning hydrocarbon surface. The ability to dilute or "lighten" water with air in concert with a foaming surfactant created a firefighting foam that floated on the burning liquid and prevented flammable vapor evolution. This higher-surface-area aqueous foam also provided a heat sink for the absorption of radiant heat and thus helped cool the burning surface and surrounding areas.

The early history of foam development was documented by Ratzer (1956). The earliest efforts to extinguish hydrocarbon liquid and/or petroleum fires were accomplished with aqueous mixtures of sodium bicarbonate, saponin (a glycoside with foaming characteristics), and acidic aluminum sulfate to extinguish a naphtha fire (Johnson 1877; Ratzer 1956). Concurrently, foam mechanically created from a solution of ammonium soap with dissolved borax (or ammonium sulfate) in a vessel that was connected to a gas cylinder containing either ammonia, nitrogen, or carbon dioxide under pressure was also developed (Gates 1903). The gas was used to force the liquid out of the vessel into another container. Additional cylinder gas was used to further enhance the foam generated in this mechanical process. By 1912, this equipment was broadly used in England (Ratzer 1956). Efforts ensued to find suitable materials to make mechanical foams more viable and effective. In 1937, water-soluble protein products were created by digestion of hoof or horn meal that were superior for mechanical foam generation (Weissenborn 1939). Consequently, other

groups developed protein-based air foam concentrates that provided favorable firefighting characteristics (Friedrich 1940; Katzer 1943; Ratzer 1944).

1.5 FLUOROSURFACTANTS IN AFFF

The invention of fluorosurfactants provided revolutionary new technology to advance firefighting foam. The initial discovery and innovation were catalyzed because the US Navy was simultaneously spraying a protein foam and a Class A powder (also called "dry chemical") to fight Class B fires aboard ships. The Class A powder was helping to control the fire but was also partially breaking down the foam blanket, like any solid powder can do on classical aqueous foams. In 1963 (Gipe and Peterson 1972), it was shown that the addition of fluorosurfactants to a protein foam allowed that foam not to be significantly affected by a simultaneous projection of a Class A powder. After that first discovery, work continued on film-forming fluoroprotein (FFFP) and AFFF, where the foam was "lightened" further using fluorosurfactants. Incorporating fluorosurfactants dramatically lowered the foam's aqueous surface tension, delivering unprecedented rapid wetting and spreading and establishing a new standard for effectively extinguishing hydrocarbon fuel fires. The full-scale implementation of that discovery was accelerated by the July 1967 USS *Forrestal* tragedy, during which more efficient firefighting foams could have limited the propagation of the jet fuel fire that took place on the flight deck. Additionally, specific fluorosurfactants were also used to generate foam (e.g., amphoteric fluorosurfactants having a positive and a negative charge on their hydrophilic part).

The first work on AFFF containing fluorosurfactants was carried out by the Naval Research Laboratory and 3M in the early 1960s and led to the development of 3M's "Lightwater" AFFF products based on ECF chemistry (Gipe and Peterson 1972). As discussed above, there are two principal chemical processes for the synthesis of the perfluoroalkyl moiety to make fluorosurfactants used in AFFF: ECF and fluorotelomerization. Figure 1.4 provides structural examples of ECF-based and fluorotelomer-based fluorosurfactants that have been developed and used by the firefighting industry over four decades (Pabon and Corpart 2002; Buck et al. 2012; Place and Field 2012; Backe et al. 2013; Barzen-Hanson et al. 2017). Figure 1.4 shows structures of various fluorosurfactants, to include different functional types (e.g., betaine, sulfonate, carboxylate, and n-oxide), historic versus present-day products, and long-chain and short-chain fluorosurfactants.

1.5.1 ECF-Based Fluorosurfactants Used in AFFF

ECF-based fluorosurfactants used in AFFF are shown in Figure 1.4. As mentioned earlier, the ECF process was the first implemented to produce fluorosurfactants. The ECF process is also called the Simons process (Simons 1949). The two types of key fluorinated intermediates used to produce the ECF fluorosurfactants of Figure 1.4 are represented in Figure 1.5. They are obtained by the full fluorination of their corresponsing hydrocarbon acid chloride and sulfochloride homologues. The full fluorination of alkyl acid chlorides also generates by-products that consist of fluorinated alkyl chains terminated by a fluorinated furan and pyran group (Banks et al. 1994).

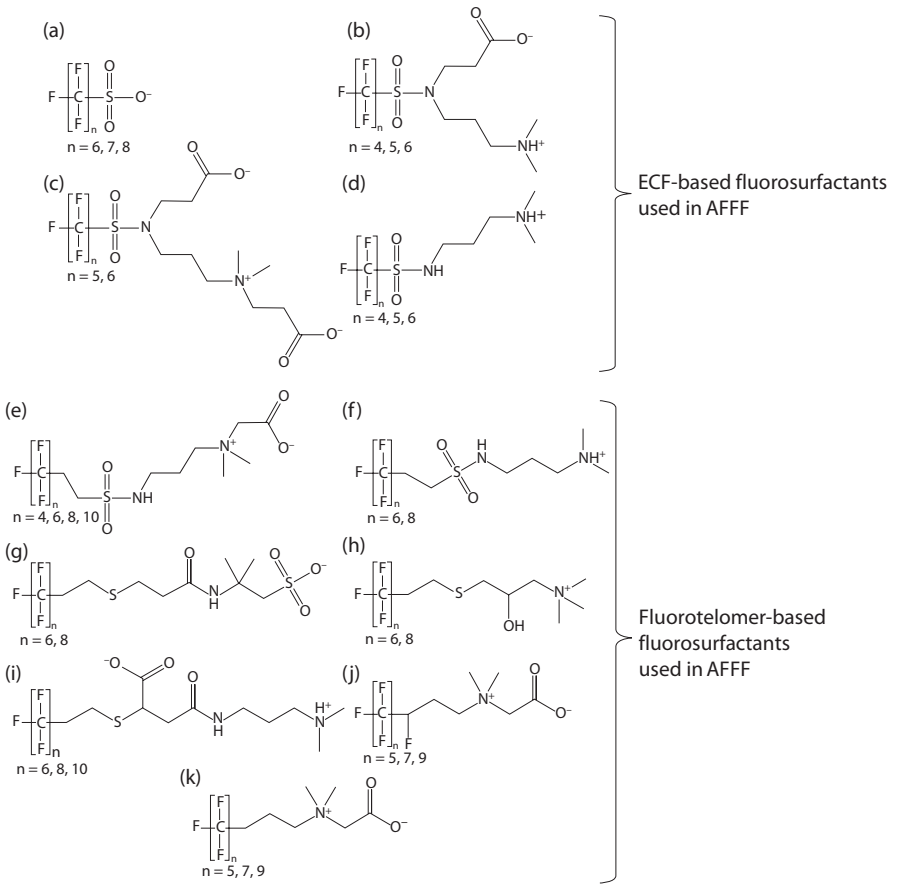


FIGURE 1.4 (a–d) ECF-based and (e–k) fluorotelomer-based fluorosurfactants used in AFFF. (Adapted from Place, B.J., and Field, J.A., *Environmental Science & Technology*, 46, 7120–7127, 2012.)

	Key intermediates	
Before electrofluorination:	$C_nH_{2n+1}SO_2Cl$	$CnH_{2n+1}COCl$
	↓	↓
After electrofluorination:	$\mathbf{C_nF_{2n+1}SO_2F}$	$\mathbf{C_nF_{2n+1}COF}$
Products	n = 8 PFOS	n = 7 PFOA
	n = 6 PFHxS	

FIGURE 1.5 Key intermediates used for the production of the ECF fluorosurfactants presented in Figure 1.4.

These by-products were mainly used as fluids for the electronic industry. Increasing the number of carbon of the alkyl chain of the starting acid chloride (n value on Figure 1.4) increases the fraction of by-products that are produced.

Four major types of surfactants are used in ECF-based firefighting foams. Perfluoralkane sulfonates and perfluorocarboxylates are anionic surfactants, perfluoroalkane sulfamido amines are cationic surfactants, and perfluoroalkane sulfonamide amino carboxylates are amphoteric surfactants. Most firefighting foams are formulated with a mixture of anionic and amphoteric fluorosurfactants. Interestingly, typical amphoteric ECF fluorosurfactants, such as the one presented in Figure 1.4 (perfluoroalkane sulfonamido carboxylate), have their positive and negative charges on two distinct groups of the tertiary amine. For ECF-based fluorosurfactants, the amphoteric structure is designed in such a way that the isoelectric point of the surfactant is at the typical pH of the firefighting foam concentrate (in the range of pH 7–8.5). Cationic surfactants can be added as a minor fraction of the fluorosurfactant mixture for specific formulations.

The ECF process induces the formation of both linear and branched perfluoroalkyl moieties. Fluorotelomers are only linear for all processes starting with PFEI. In terms of performance in firefighting foams, branched fluorosurfactants are not as efficient as equivalent linear fluorosurfactants (Taylor 1999; Kissa 2001). This implies that, on average, to obtain a similar performance level, a firefighting foam based on an ECF-produced surfactant requires a higher fluorine content than a fluorotelomer-based firefighting foam. Historically, ECF-based fluorosurfactant firefighting foams used mainly long-chain perfluorooctane (C8) and perfluorohexane (C6) sulfonyl surfactants. In the early 2000s, attempts had been made to use short-chain ECF-based perfluorobutane sulfonyl fluorosurfactants. However, these did not meet the AFFF performance requirements stipulated in military specifications.

1.5.2 Fluorotelomer-Based Fluorosurfactant Used in AFFF

Fluorotelomer-based surfactants used in AFFF are shown in Figure 1.4 and are nominally derived from three structural families: (1) a thiol (–S–) in the spacer (e.g., $–CH_2CH_2–\mathbf{S}–CH_2–$), (2) a sulfonyl ($–SO_2–$) in the spacer (e.g., $–CH_2CH_2–\mathbf{SO_2}–N–$), or (3) an alkyl spacer (e.g., $–CH_2CH_2CH_2–$ or $–CFHCH_2CH_2–$) between the perfluoroalkyl ($C_nF_{2n+1}–$) moiety and the surfactant functionality.

Figure 1.4 shows that the fluorotelomer-based surfactants used in firefighting foams are either amphoteric (fluorotelomer thioamido sulfonates or fluorotelomer sulfamido betaines, fluorotelomer betaines), cationic (fluorotelomer sulfamido amines), or anionic (fluorotelomer sulfonates).

Historically, as of the early 1970s, fluorotelomer-based surfactants used in firefighting foams contained perfluoroalkyl moieties, $F(CF_2)_n–$, with n = 6 or a mixture of different perfluoroalkyl chains with n = 6, 8, and 10 carbons, with a majority (>50%) comprised of n = 6. Today, all currently produced fluorotelomer-based surfactants (by Voluntary Stewardship Program signees; USEPA 2010) used in firefighting foams are based on C6 raw materials that contain a six-carbon perfluoroalkyl moiety, $C_6F_{13}–$. Historical accounts (Cortina and Korzeniowski 2008; Place and Field 2012; Backe et al. 2013; Kleiner 2016; Barzen-Hanson et al. 2017) note that the fluorotelomer chemistry employed in the AFFF products used either a perfluoroalkyl moiety with n = 6

or a mixture of n = 6 (C6) and n = 8 (C8). This clearly means (not implies) that the fluorotelomer-based fluorosurfactants used in the early Ansul/Ciba-Geigy and Eau et Feu (Atochem) consisted of either short-chain or both short- and long-chain fluorosurfactants, from the mid-1970s onward. Back in those days, as surprising as it can be for nonspecialists, some producers where able to pass the performance requirements of the most demanding standard in the world (MIL-F-24385 1969) by using fluorosurfactants made with a perfluoroalkyl moiety, C_nF_{2n+1}–, where n = 6 only.

Fluorotelomer chemistry (Figure 1.4 lower part) was used to produce both the Ciba-Geigy Lodyne™ and Atochem Forafac™ branded surfactants. Atochem's fluorosurfactant business was acquired by DuPont in 2002. This business was spun out into Chemours in 2015 and is now part of the Capstone™ line of products. Likewise, the Ciba-Geigy Lodyne firefighting fluorosurfactant and foam stabilizer business was acquired by the Chemguard Specialty Chemicals Division from Ciba Specialty Chemicals in 2003–2004. The Chemguard Chemicals Division became part of the Tyco group in 2011, and Tyco was acquired by Johnson Controls in 2016. Each of these chemistries used the basic fluorotelomer scheme. The Ciba-Geigy/Chemguard fluorotelomer-based surfactants were generally characterized by the $–CH_2CH_2–S–CH_2–$ linkage. Likewise the Atochem/DuPont/Chemours fluorotelomer-based fluorosurfactants contained the $–CH_2CH_2–SO_2–N–$ linkage. The Qualified Products List (QPL) discussed below has all short-chain fluorotelomer products on the current 2017 QPL list for military specification MIL-F-24385F, the current acceptance specification used in the US Department of Defense (USDOD) (MIL-F-24385 1969).

1.6 FIREFIGHTING FOAMS: TYPES AND COMPOSITION

Fires are classified by the proper extinguishing agent. Class A fires consist of ordinary combustibles, such as wood, paper, fabric, and most kinds of trash. Water is usually used to extinguish Class A fires. For Class A, high-expansion and fluorine-free foams (F3, nonfluorinated hydrocarbon surfactants) are used. A Class B fire arises from flammable liquids or flammable gases, petroleum greases, tars, oils, oil-based paints, solvents, lacquers, or alcohols. Using water on a Class B fire (e.g., an oil-based liquid fire) is extremely dangerous. Petroleum-based fires burn significantly hotter (≥350°F) than the boiling point of water (212°F). When water is placed on a petroleum-based fire, it instantly vaporizes to form steam, which expands rapidly, causing the liquid hydrocarbon liquid to splatter, cause burns, and spread the fire. Hence, AFFF is preferred for extinguishing high-hazard Class B fires.

In use, AFFF concentrate is mixed with either freshwater or seawater by a proportioning nozzle (Figure 1.6). The foam forms spontaneously upon ejection from the nozzle. A thin layer of AFFF foam rapidly spreads over the hydrocarbon fuel surface, acting as a thermal and evaporation barrier to inhibit and eventually extinguish combustion. The "film-forming" characteristic refers to the fact that both in application and even after the foam has dissipated, the aqueous layer formed from the AFFF concentrate mixture remains on the flammable liquid hydrocarbon surface, thereby preventing reignition. This occurs because the fluorosurfactant makes the surface tension of the water lower than the surface tension of the hydrocarbon, enabling it to float on the hydrocarbon surface at the air interface.

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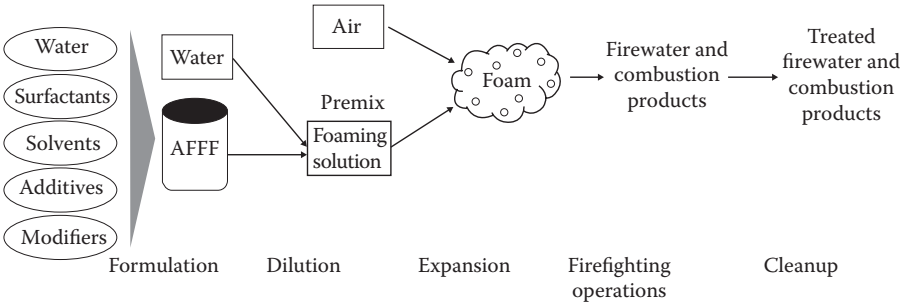


FIGURE 1.6 Schematic of AFFF concentrate and its use. It shows in pictorial form the ingredients used to concentrate foam to deploy to firewater.

1.6.1 Types of Firefighting Foam

There are a number of ways to classify firefighting foams. One way is by chemical type, and another is by expansion ratio. In general, expansion ratios are classified as (1) low-expansion foams (with an expansion ratio of ≤20), (2) medium-expansion foams (having an expansion ratio from 20 to 200), and (3) high-expansion foams (with expansion ratios of >200). We will focus on the two chemical types of firefighting foams: (1) F3 that are fluorine-free and (2) AFFF containing fluorosurfactants, with a focus on AFFF. Historically, nonfluorinated mechanical protein-type foams are derived from either hydrolyzed protein-rich animal or vegetable materials using alkali/lime. Vegetable materials were developed later and used in a "synthetic" formulation whose origin is from hydrocarbon-derived materials. Beginning in the 1960s, various fluorosurfactants were developed and incorporated into both types of synthetic and protein-based firefighting foam agents (Briggs 1996). Further, it is also well known that water-miscible liquids, such as polar solvents (e.g., alcohols and ketones), tend to destroy conventional foams. To overcome this characteristic, formulations were created that contained polymeric/gel-type materials, which help prevent foam destruction. These types of foam agents are called alcohol resistant (AR). For current-generation fluorine-free foams, there is basic F3, as well as alcohol-resistant fluorine-free foam (AR-FFF). It should be noted that protein, high-expansion, Class A, and most training foams have historically been fluorine-free. The types of firefighting foam are shown in Figure 1.7.

Fluorine-free foam (F3)	Fluorinated foams
· Basic fluorine-free foam (FFF)	· Fluoroprotein (FP)
· Protein	· Film-forming fluoroprotein (FFFP)
· Alcohol-resistant protein (AR-P)	· Alcohol-resistant film-forming fluoroprotein (AR-FFFP)
· Synthetic	· Aqueous film-forming foam (AFFF)
	· Alcohol-resistant aqueous film-forming foam (AR-AFFF)

FIGURE 1.7 Types of firefighting foam.

1.6.2 AFFF Ingredients and Composition

The precise chemical composition of AFFF formulations is generally proprietary confidential business information as the ingredients are viewed by AFFF manufacturers and formulators as providing unique competitive benefit to their products. A firefighting foam is obtained by the dilution of a foam concentrate in water (generally a 3% or 6% foam concentrate, v/v). This water–foam concentrate mixture is called a foaming solution or premix (Figure 1.6).

The foaming base of a foam concentrate consists of either a mixture of hydrocarbon surfactants or a hydrolyzed protein. Synthetic foam concentrates can be distinguished from protein foam concentrates according to the foaming base. To simplify matters, our discussion on ingredients and composition only involves synthetic foam concentrates made from surfactants. The types of firefighting foams are listed in Figure 1.7.

In general, AFFF concentrates contain four main ingredients: (1) water, (2) surfactants (to provide the foam and film-forming properties), (3) solvents, and (4) various additives and modifiers. As previously stated, water-soluble polymers are added to obtain AR-AFFF. Typical ingredients are shown in Figure 1.8. Water is the major ingredient and generally is more than 60% of the composition.

Typically, a synthetic foam concentrate designed to be diluted at 3% into water contains from 5% to 10% by weight of hydrocarbon surfactants. High-performance foam concentrates also contain one or several fluorosurfactants. Fluorosurfactants, given their relatively high expense and effectiveness at low concentration, are a minor but critical ingredient in AFFF. The fluorosurfactant plays a role in the formation of the foam as well as the formation of a water film at the surface of the solvent. In active matter, they account for 0.6–1.5 wt% of the total weight of a foam concentrate. Fluorosurfactants also provide oil repellency to firefighting foams, and this property is key when foams are used in larger-scale and real conditions.

The formation of the water film at the surface of the hydrocarbon has the result that the emission of solvent vapors is stopped or highly reduced. As those vapors are

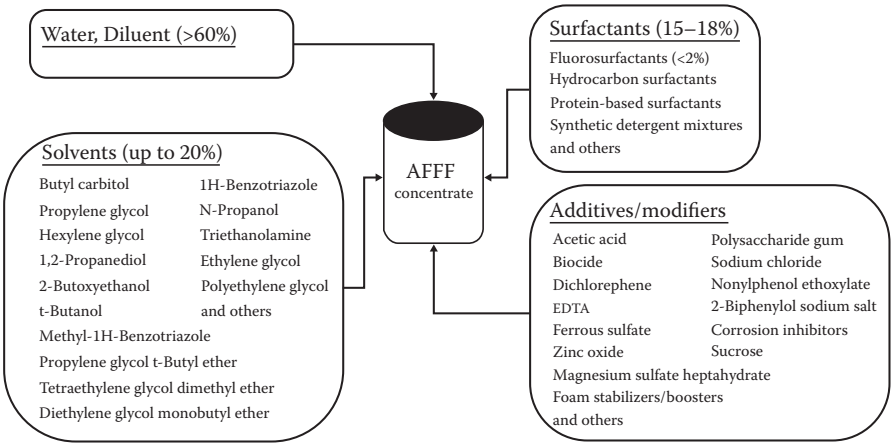


FIGURE 1.8 Type of ingredients for a 3% foam concentrate.

the fire source, it is very important to stop their emission. This water film formation occurs when the spreading coefficient (SC) of the foaming solution is positive. This coefficient is defined in Equation 1.1:

$$SC = \lambda s - (\lambda HC + \gamma L) \tag{1.1}$$

where γ_S is the surface tension of the solvent, γ_{HC} is the interfacial tension between the solvent and the foaming solution, and γ_L is the surface tension of the aqueous foaming solution.

The use of hydrocarbon surfactants reduces the solvent–foaming solution interfacial tension (γ_{HC}), and fluorinated surfactants provide the foaming solution with a particularly low surface tension (γ_L). Without a suitable choice of these two types of components, the spreading coefficient of a foaming solution on a solvent would be negative and extinction impossible.

There are debates in the industry about the existence of an aqueous film with AFFF at elevated temperature conditions, but foams having the capacity to form an aqueous film at regular temperature demonstrate, in most cases, higher performances than F3 foams. A foam concentrate also contains polar solvents (up to 20 wt%) that contribute to the foam stability. 1,2-Propanediol and similar types of solvents are used to reduce the freezing point of foam concentrates stored in refineries, civil airports, or drilling platforms.

In addition to the classic foam concentrates, AR foam concentrates are polyvalent and can be used to extinguish polar solvent fires, such as ethers, ketones, or alcohols. Polar solvents are miscible in water and can solubilize most hydrocarbon and fluorinated surfactants. This results in the almost instantaneous disappearance of the firefighting foam when it is placed at the surface of the solvent. The most common solution to obtain a stable foam in contact with the polar solvent involves the addition of water-soluble polymers such as polysaccharide to the foam concentrate. In contact with the polar solvent, the polymer precipitates to form a protective layer at the interface that isolates the foam from the polar solvent and prevents its destruction. Modern AR-AFFF also contains water-soluble polymers modified with fluorotelomers. Additives providing anticorrosion, antimicrobial activity, and other properties are all part of typical AFFF formulations.

A generalized view of a typical AFFF Type 3 (3%) concentrate composition based on data taken from company websites, safety data sheet documents, company fact sheets, and various reverse engineering efforts and analytical testing is shown in Figure 1.9 (Cortina 2012). In Figure 1.9, a representative composition of the foam that is actually applied to a fire, a 3% AFFF concentrate diluted with 97 parts water, is shown. In this example, the combination of surfactants and solvents is approximately 1% of the final solution as employed in firefighting.

1.7 QUALIFIED PRODUCTS LIST (QPL)

AFFF is a highly efficient firefighting agent for extinguishing flammable liquid (Class B) fires. AFFF concentrates used by the US military must meet the requirements set forth in military specification MIL-F-24385 (1969). The specification is

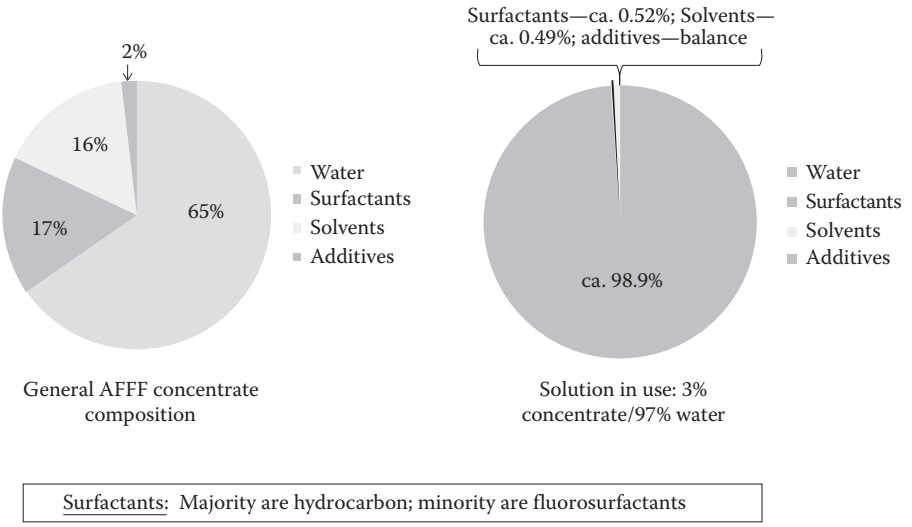


FIGURE 1.9 Pie chart showing the composition of a Type 3 (3%) AFFF concentrate.

under the control of the Naval Sea Systems Command, and the Naval Research Laboratory is the designated institution for certification evaluation for the USDOD's AFFF QPL. AFFF is used globally by the US military, by the militaries of other countries, and in the majority of civilian applications worldwide as either a 3% (Type 3) or a 6% (Type 6) concentrate. The military specification AFFF concentrates are effective against a wide variety of Class B liquid hydrocarbon fire threats. Special formulations exist for use against alcohol and other water-miscible fuels.

The early use of fluorosurfactants in firefighting foams (AFFF) is well documented in a Naval Research Laboratory report (Gipe and Peterson 1972). The first fluorosurfactant work was a collaborative effort between the US Navy and 3M and began around 1961. This effort resulted in the 1964 introduction of the first AFFF product into Navy firefighting service inventory, the ECF-based 3M product FC-183. FC-194, which replaced FC-183 about 3 years later, was less viscous and could be used at a 6% concentrate strength in freshwater with conventional foam-proportioning equipment. However, FC-194 had a significant drawback. FC-194 could not be used with seawater, essential for deployment on naval vessels. Subsequently, a seawater-compatible AFFF, FC-195, was developed, but it was never submitted for qualification under what became the well-known QPL and military specification MIL-F-24385, issued on November 21, 1969. The first AFFF on the QPL was FC-196 from 3M in 1969. Improvements and modifications to the AFFF agents continued. The next-generation AFFF, FC-200, had a pH of >7 and corrosion inhibitors added to limit any agent attack in seawater on the stainless steel storage vessels. FC-200 was QPL approved in January 1972. Of note, the FC-200 fluorine content (2.1% w/v) was significantly reduced in this formulation compared with its FC-196 predecessor (3.2% w/v).

For the first 4 years the QPL existed (1969–1973), 3M AFFFs containing ECF-based fluorosurfactants were the only products qualified under MIL-F-24385. 3M's

development partnership with the Navy lasted nearly 10 years (~1963–1973) before the next product and company became listed. AFFF containing fluorotelomer-based fluorosurfactants from National Foam was qualified on the QPL in late 1973, followed by Ansul using a Ciba-Geigy fluorotelomer-based fluorosurfactant in 1974–1975. These products had between 0.7% and 0.83% w/v fluorine, or less than half of fluorine of the ECF product FC-200. In 1981, two more products were added to the QPL: 3M's FC-206C and Ansul's AFC-5, which contained 0.94% w/v and 0.61% w/v fluorine, respectively (Barzen-Hanson et al. 2017). In 1993, 3M's Lightwater™ FC-203CF contained 1.8 wt% fluorine while National Foam's Aer-O-Water 3EM contained 0.85 wt% fluorine; both were listed on the QPL (Kleiner and Jho 2009). Each of these products had passed the US military's QPL specification MIL-F-24385. The data indicate that the AFFF containing fluorotelomer-based fluorosurfactants provided firefighting performance equal to that of the ECF-based AFFF products with substantially less fluorosurfactant content.

A chronology of the QPL is shown in Figure 1.10, with AFFF product descriptions given in Table 1.1. Even though ECF-based long-chain products (e.g., PFOS containing) are still in inventory at various sites globally (Darwin 2011), none are on the QPL today, in 2017. The reader is reminded that the short-chain (C4, perfluorobutane sulfonyl) ECF-based fluorosurfactants are not used in AFFF because, to date, no AFFF formulation containing them has been qualified. There has been significant changes in the QPL over the past 6 years (2011–2017) as the transition to AFFF containing short-chain C6 fluorotelomer-based surfactants has occurred.

With 3M's exit (Type 6 withdrawn January 3, 2007, and Type 3 withdrawn August 8, 2010) from long-chain ECF-based fluorosurfactant manufacture for AFFF in the last decade, National Foam is the longest-standing company on the list, having come onto the QPL in 1973, followed close behind by Ansul (today Johnson Controls). Both Angus and Buckeye came off the QPL within the past 2 years, while at the same time ICL/Auxquimia (2015; now Perimeter Solutions), Amerex/Solberg (2016), and Dafo Fomtec AB are recent additions. Chemguard (since 1997) and Fire Service Plus (Type 6 added May 2011) are also on the May 2017 QPL (Figure 1.10). It is also noteworthy

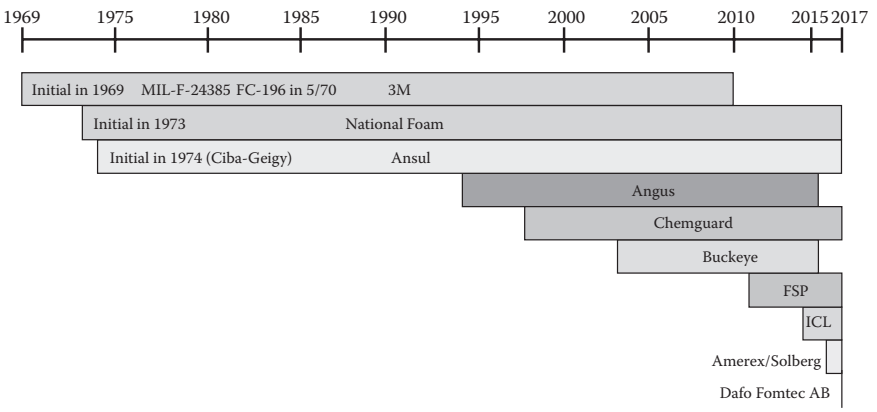


FIGURE 1.10 QPL chronology: 1969–2017 (MIL-F-24385) as of May 12, 2017.

TABLE 1.1
Qualified Products Database Comparison 2004–2017

		Test Date[a]		April 5, 2004		Test Date[a]	May 12, 2017	
Company	Chemistry Type	Type 3	Type 6	Type 3	Type 6	Type 3/6	Type 3	Type 6
3M Co.	E	NR	NR	FC-203CF	FC-206 CF			
Angus Fire	FT	February 18, 1994	–	Tridol M 3%	–			
Ansul, Inc.	FT	NR	NR	Ansulite 3%	Ansulite 6%			
Buckeye Fire Equipment	FT	November 12, 2003	–	Buckeye 3%	–			
Chemguard, Inc.	FT	January 30, 1997	July 25, 2001	Chemguard 3%	Chemguard 6%			
Kidde/National Foam	FT	NR	NR	Aer-O-Water 3-EM	Aer-O-Water 6-EM			
Fire Service Plus	FT					November 2, 2010	–	Fireade 2000-MIL 6 AFFF
National Foam	FT					May 2, 2016	Aer-O-Water 3-EM-C6 AFFF	Aer-O-Water 6-EM-C6 AFFF
	FT					May 2, 2016	Tridol-C6 M3 AFFF	Tridol-C6 M6 AFFF

(*Continued*)

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TABLE 1.1 (CONTINUED)
Qualified Products Database Comparison 2004–2017

		Test Date[a]		April 5, 2004		Test Date[a]	May 12, 2017	
Company	Chemistry Type	Type 3	Type 6	Type 3	Type 6	Type 3/6	Type 3	Type 6
Tyco Fire Products/ Ansul	FT					November 13, 2015	Ansulite AFC-3MS	Ansulite AFC-6MS
Amerex Corp./ Solberg Corp.	FT					February 29, 2016	Arctic 3% MIL-SPEC AFFF	Arctic 6% MIL-SPEC AFFF
Tyco Fire Products/ Chemguard	FT					November 13, 2015	Chemguard C306-MS	Chemguard C606-MS
ICL/Auxquimia	FT					November 3, 2015	Phos-Chek 3% AFFF	
Dafo Tomtec AB	FT					January 19, 2017	Fomtec AFFF 3%M	

Note: FT = fluorotelomer; NR = tests were conducted but no date was noted; E = ECF.

[a] Test dates/Naval Research Laboratory report listed when available from QPL list. Listing on actual QPL followed at a later date.

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TABLE 1.2
QPL List (March 2018) MIL Spec MIL-F 24385

Type 3, 3% AFFF		Type 6, 6% AFFF	
Product Name	**Supplier**	**Product Name**	**Supplier**
AER-O-WATER 3E-C6 AFFF	National Foam, Inc.	AER-O-WATER 6EM-C6 AFFF	National Foam, Inc.
ANSULITE AFC-3MS 3% AFFF	Tyco Fire Protection Products (JCI)	ANSULITE AFC-6MS 6% AFFF	TYCO Fire Protection Products (JCI)
ARCTIC 3% MIL-SPEC AFFF	Amerex Corp., The Solberg Company	ARCTIC 6% MILSPEC AFFF	Amerex Corp., The Solberg Company
CHEMGUARD C306-MS 3% AFFF	Tyco Fire Protection Products (JCI)	CHEMGUARD C606MS 6% AFFF	TYCO Fire Protection Products (JCI)
PHOS-CHEK 3% AFFF MS	ICL Performance Products (now Perimeter Solutions)	FIREADE MILSPEC 6 (Jan 2018)	Fire Service Plus, Inc.
TRIDOL-C6 M3 AFFF	National Foam, Inc.	TRIDOLC6 M6 AFFF	National Foam, Inc.
FOMTEC AFFF 3% M: SWE & USA	Dafo Foamtec AB	PHOS-CHEK 6% Milspec AFFF (12/17)	ICL Performance Products (now Perimeter Solutions)
FIREADE Milspec 3	Fire Service Plus, Inc.		

Source: http://qpldocs.dla.mil/search/parts.aspx?qpl=1910

that while three of the companies (National Foam, Ansul, and Chemguard) have been on the QPL for many years, there are an equal number of new entrants to qualify for arguably the world's most difficult fire standard. These new QPL entrants are Fire Service Plus (FSP); Solberg Corporation; ICL, who purchased Auxquimia's Class B foam business in 2014; Now Perimeter Solutions; and Dafo Fomtec AB. Current (2018) QPL AFFF products for Class B fires are shown in Table 1.2. The short-chain fluorosurfactants in all these products are C6 fluorotelomer-based products.

1.8 FIREFIGHTING FOAM SELECTION AND USE

The selection of a firefighting agent to extinguish a Class B fire is front and center today more so than it has been over the past 15 years due to the widespread environmental presence from historic use of AFFF (Willson 2016a). Numerous fire journal articles (and other media) have appeared debating the pros and cons of both general types of firefighting agents, F3 and AFFF containing fluorosurfactants. The selection decision requires a thoughtful consideration of multiple criteria, such as fire performance, life safety benefits, property protection, and environmental impact, including whether control of spent firewater is possible or practical. Balancing these criteria presents a set of difficult choices for regulators, firefighters, and those who routinely handle flammable liquids and require rapid fire extinguishment and control

to protect human life and property. A recent firefighting foam seminar in Singapore (SAA-IAFPA 2016) provided an open forum for discussion of these decision-making criteria, including numerous presentations highlighting critical insights into why AFFF is often chosen (Castro 2016; Jho 2016; Plant 2016; Willson 2016b). To illustrate the issues, performance, and fundamental property parameters, this section discusses foam properties (Table 1.3) and environmental properties (Table 1.4).

TABLE 1.3
Foam Properties: Comparison of AFFF and F3

Foam Property	Advantage	AFFF[a]	F3
Fuel repellency	Yes	Yes	No
Film formation	Yes	Yes	No
Foam spreading on fuel	Yes	Yes	No
Fuel spreading on foam	No	No	Yes
Fuel shedding	High	High	Low
Fuel pickup	Low	Low	High
Fuel emulsification	Low	Low	High
Flammability of contaminated foam	Low	Low	High
Degradation of contaminated foam	Low	Low	High
Heat resistance of foam	High	High	High

[a] Includes short-chain AFFF.

TABLE 1.4
Environmental Properties: Comparison of Short-Chain Formulations of AFFF and F3

Environmental Property	Advantage	AFFF[a]	F3
Aquatic toxicity	Low	Yes	No
Persistence	No	Yes	No
Reduced foam, water resources used, and wastewater generation	Yes	Yes	No
Reduced smoke and breakdown products generated	Yes	Yes	No
Risk to life safety	Low	Low	Higher
Escalation potential	Low	Low	High
Bioaccumulation	No	No[b]	No
Disposal through PTOW or WWTP	Yes	Yes[c]	Yes

Note: PTOW = publicly owned treatment works; WWTP = wastewater treatment plant.
[a] Includes short-chain AFFF.
[b] Short-chain AFFF.
[c] With pretreatment.

Critical foam properties required to fight a Class B fire that are evaluated in selecting a firefighting agent are presented in Table 1.3, comparing AFFF (containing short-chain fluorosurfactants) and F3. For example, in hydrocarbon-type Class B fires, a firefighting agent that has fuel repellency, sheds the fuel, forms a film, is not contaminated by the fuel, and rapidly spreads across the burning fuel surface is highly advantaged versus an agent that does not have these critical properties. These properties are clear advantages of AFFF containing fluorosurfactants that fundamentally differentiate AFFF from F3. Both AFFF and F3, in general, appear to have a high resistance to heat, although one can argue that the mechanism for this heat resistance is different. For AFFF, the fluorosurfactants used in the formulation have better chemical and heat resistance than hydrocarbon surfactants. F3 is given a high rating as it believed that the current generation of fluorine-free foams have high loadings of polysaccharides (sugars and gums) to boost drain times and thus trap or keep water longer in the foam matrix. The presence of the trapped water should provide greater heat resistance.

The environmental properties of short-chain AFFF and F3 are shown in Table 1.4. Whereas the foam properties show many clear advantages for AFFF (Table 1.3), the environmental properties require discussion and explanation. Aquatic toxicity data for firefighting foam products have been discussed (FFFC 2006; Plant 2016). The available data show that AFFF products have significantly lower acute aquatic toxicity than the F3 used in comparative fire testing. The basis is also shown in Table 1.5 (FFFC 2006). Both indicate very clearly that each of the three AFFF products have significantly lower toxicity in both the rainbow trout and the fathead minnows. A lower LC_{50} indicates the agent is more toxic.

Another important environmental property is persistence. The stability of the carbon–fluorine bonds in fluorosurfactants makes them and/or their terminal biodegradation products stable and therefore persistent in the environment (see Section 1.11 for a discussion). In contrast, F3s are made with hydrocarbon ingredients that are generally expected to biodegrade under normal forseeable environmental conditions and thus are not considered persistent.

The next five properties in Table 1.4, from reduced foam and water resources used, deserve some discussion. AFFF has been selected as the product with the more

TABLE 1.5
Aquatic Toxicity Comparison

96 h LC_{50} Test Fingerling Rainbow Trout		96 h LC_{50} Flow-Through Test Fathead Minnow	
Agent	**LC_{50} (mg/L)**	**Agent**	**LC_{50} (mg/L)**
Wetting agent	1.06	Wetting agent	0.887
F3-A	63	F3-A	171
F3-B	71	F3-B	171
MIL-SPEC AFFF	2176	MIL-SPEC AFFF	884
AR-AFFF	3536	AR-AFFF	1487
UL AFFF	5657	UL AFFF	1726

Note: UL = Underwriters Laboratories.

desirable firefighting characteristics when compared with F3. The advantage originates from the demonstrated performance of AFFF versus F3 when considering time to control the fire to overall extinguishment time. It is generally accepted that AFFF will knock down, control, and extinguish Class B fires faster than F3. Some argue that there are some fire test data that show equal performance in putting out a fire. However, the majority of fire test data show AFFF perform superiorly to F3 (see Section 1.9). That AFFF extinguishes a fire faster leads to lower foam usage, reduced water usage, and reduced smoke and breakdown products due the shorter control and extinguishment times. With reduced foam and water resources used, it follows that firewater runoff and wastewater generation are also reduced. In light of faster control and extinguishment times, the edge for life safety risk is also given to AFFF. Life safety risk includes firefighters and any other personnel in the fire's vicinity. In considering these properties as a whole, faster fire control in the view of many leads to a lower fire escalation potential, thus making AFFF the selection for use in a large-scale high-hazard Class B fire event.

The present-day short-chain C6 fluorotelomer-based products, raw material and degradation products, have been shown to have a more favorable toxicological and environmental profile than historic long-chain equivalents (FluoroCouncil 2017). Further, fluorotelomer-based C6 short-chain alternatives have been shown through various evaluations not to be bioaccumulative under current regulatory criteria (Conder et al. 2008). F3 is generally considered biodegradable and not bioaccumulative. The last environmental property, disposal, is very important. It is essential that all firefighting foams be properly disposed. Both the Fire Fighting Foam Coalition (FFFC) (2016a, 2016b) and FPAA (2017) have provided industry guidance for proper foam disposal. Foam, spent foam, and firewater must be disposed of according to local and state (and other government) authorities' requirements.

In summary, selection of a firefighting foam for Class B fires involves consideration of multiple criteria, as shown in Tables 1.3 and 1.4. Over the years, many have invoked the phrase "fit for purpose" as guidance for selection. If the firefighter needs rapid knockdown and control, rapid extinguishment, extended burnback times, and the best life safety performance and asset protection, the choice in 2017 is clearly AFFF containing fluorosurfactants. F3 performance has improved dramatically over the past few years and has been selected by some users instead of AFFF where performance meets their requirements for life safety and ultimate property and asset protection. F3s have achieved many internationally recognized certifications, but to date not MIL-F-24385. A research initiative funded by the Strategic Environmental Research and Development Program (SERDP) titled "Fluorine-Free Aqueous Film Forming Foam," dated October 29, 2015, seeks research proposals to identify a fluorine-free foam that meets or exceeds the performance criteria required in MIL-F-24385 (SERDP 2015).

1.9 AFFF AND F3: FIELD TEST PERFORMANCE AND EFFICACY

In addition to foam and environmental properties, actual efficacy comparisons in the field are essential to assess fire performance and select firefighting foam. The FFFC has published two studies comparing field test performance and the efficacy of

AFFF and F3 (FFFC 2014, 2017). In 2011, the Naval Research Laboratory presented field test results of AFFF agents and F3 (Williams et al. 2011). AFFF extinguishment times on 28 ft^2 pool fires tested at full strength were on average 77% faster for gasoline, 88% faster for methylcyclohexane (MCH), and 70% faster for heptane than those of F3. For isooctane, the tested AFFF was unable to form a film and the F3 extinguished the fire 10% faster, highlighting the importance of film formation to foam performance. The AFFF tests extinguished the heptane and gasoline fires in 21–28 seconds. The time required to pass the MIL-F-24385 is 30 seconds. The F3 was unable to extinguish any gasoline or heptane fire in less than 30 seconds. To be on the QPL, AFFF products must meet these requirements to be used for military applications. The Federal Aviation Administration (FAA 2011) requires all US airports to carry AFFF agents that meet MIL-F-24385 and are listed on the QPL. In another field study reported at the 2013 Reebok Foam Conference, VS Focum (2013) summarized the company's F3 development. The presentation contained side-by-side field test data done at the same facility under the same conditions comparing the fire performance of AFFF and F3. The results showed that AFFF performed significantly better than F3 in spray extinction tests (0.785 m^2) using heptane, gasoline, and Kerosene Jet A-1, and in Spray Pan Fire Out tests in sizes 0.25, 0.785, and 7.06 m^2, while the values were similar in the 4.52 m^2 test. The Naval Research Laboratory has conducted field tests comparing AFFF and F3 (Hinnant et al. 2017a, 2017b). In pool fire tests, AFFF achieved extinguishment in 18 seconds, compared with 40 seconds for the F3. In foam degradation tests, F3 degraded after 1–2 minutes, while AFFF lasted 35 minutes before degrading (longer is better). Similar results from a series of foam degradation tests on AFFF and F3 were published earlier in *International Fire Fighter* in 2012 (Jho 2012). At a 2016 foam seminar (SAA-IAFPA 2016), field fire test results conducted on five commercially available short-chain fluorotelomer-based AFFFs and five commercially available F3 were presented (Castro 2016). The tests were run on four fuels: gasoline, heptane, Jet A-1, and diesel. The results showed that AFFF performed significantly better than F3 on all fuels except diesel, where they both put out the fire. None of the F3s were able to extinguish Jet A-1.

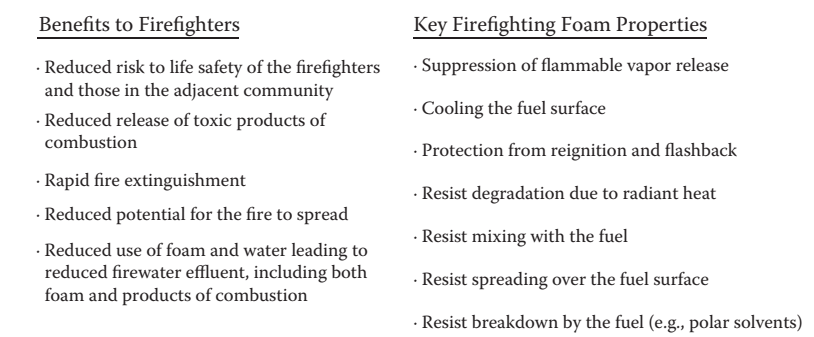


FIGURE 1.11 FPAA information bulletin: "Selection and Use of Firefighting Foam" (From FPAA, Selection and use of firefighting foams, Information bulletin, Version 2.0, January 2017. http://www.fpaa.com.au/.)

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Jet A-1 is the fuel used in International Civil Aviation Organization fire tests, which determine the acceptability of foams for airport use in many countries.

Lastly, the reader is directed to a recently published information bulletin titled "Selection and Use of Firefighting Foams," authored by the FPAA (2017). The FPAA has neatly articulated what a high level of firefighting performance provides firefighters, regardless of whether the foam contains fluorine (Figure 1.11). Additionally, the bulletin describes key foam properties that align with those presented in Table 1.3.

1.10 AFFF USE: BEST PRACTICES

It is imperative that the firefighting foam be fit for purpose. AFFFs should not be used for typical Class A fires (such as wood), forest fires and wildfires, residential fires, electrical fires (Class C), kitchens—commercial and residential, and computer, server, and any telecommunications equipment fires. In addition, AFFF products should not be used on a small car or truck fire where the potential for a large fuel spill is small and where water will suffice to extinguish the fire. AFFF is the foam of choice for fires where there is the potential for a significant flammable liquid hazard or where the rapid extinguishment of hydrocarbon fuel was necessary to avoid harm to people and/or property (e.g., armaments). This is why typical historic use of AFFFs has been in military operations, marine operations, and petroleum terminal, processing, and operations, as well as chemical and other industrial facilities. Considerations when determining the use of AFFF are many. The conversation appears to have shifted from performance to presence, and persistence is what really matters. Having perspective is critical in this discussion. Users of PFAS-containing products must consider the toxicological profile; the potential for exposure; the prevention of emissions; the protection of people, property, and the environment; and the use of risk-based analyses versus solely the precautionary principle (Figure 1.12) (Korzeniowski 2017).

AFFF safe handling, proper use, and disposal are essential. When it is appropriate to use AFFF and how a Class B fire is fought and extinguished are important. A universal consideration is minimizing human exposure and environmental release while protecting people and property. The potential environmental impact of Class B foam usage is critically important. There are three primary sources of potential environmental impact: (1) the fire's combustion products, (2) the AFFF foam itself, and (3) resulting firewater. Foam and firewater must be disposed of in compliance with local, state, and/or country regulations. All foams have the potential to contaminate surrounding areas where foam has been used and the potential to be harmful to the environment if the fire is not fought properly with the right products and if the firewater effluent is not managed appropriately.

Fluorosurfactants are highly stable and do not mineralize by biodegradation (see Section 1.11). As a result, AFFF emissions to the environment from historic use in fire training and firefighting have left a measurable environmental footprint, even decades after use according to recently published research (Anderson et al. 2016). The importance of proper use, handling, and disposal of AFFF has thus become a subject of intense discussion (Seow 2013; DEHP 2016a, 2016b; USEPA 2016). In

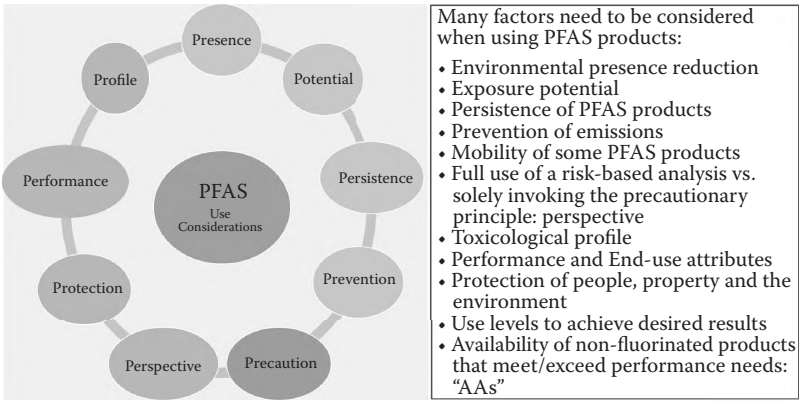


FIGURE 1.12 AFFF use considerations and best practices. (Adapted from FFFC, Best practice guidance for use of Class B firefighting foams, 2016. https://docs.wixstatic.com/ugd/331cad_188bf72c523c46adac082278ac019a7b.pdf. FFFC, Guidance for fluorinated firefighting foams, 2016. www.fffc.org.)

response, guidance has been developed and promulgated by manufacturers and regulators to AFFF users to cease the use of AFFF for training and only use it in active firefighting to protect people and property. The Umwelt Bundes Amt (UBA) (the German Environment Agency), in collaboration with firefighting foam manufacturers and firefighters, published guidance for the proper use, handling, and disposal of AFFF in 2013 (UBA 2013). The two industry groups, FFFC and FPAA, have done a significant amount of work to raise awareness for best practices and proper use and disposal of foam. The FFFC published both a best practice guidance document and a flyer detailing critical best practices in handling AFFF to fight Class B fires (FFFC 2016a, 2016b). As described earlier, the FPAA has also recently provided guidance on foam use (FPAA 2017). The FFFC, FPAA, and UBA documents should be consulted before use of Class B foam. In brief, everyone using AFFF should diligently work to eliminate foam discharge to the environment. A first way to ensure this is to be sure that all training foams are nonfluorinated. AFFF and firewater should be contained followed by treatment and approved disposal to ensure minimal or no releases to the environment. Advanced planning, preparation, and practice are ways to identify and prevent uncontrolled foam or firewater releases and thereby minimize the environmental impact from firefighting events. Lastly, active monitoring of foam generation and delivery systems for leaks and proper preventative maintenance of foam systems should be a requirement.

AFFF products containing fluorosurfactants provide the firefighter with the most effective agents on the market today. As set forth in the FFFC guidance document (FFFC 2016a, 2016b), not only is AFFF the product of choice for Class B fires, but also the use of the multipurpose AR-AFFF or AR-FFFP foam concentrates allows the firefighter to stock one product that can be used to extinguish either a hydrocarbon or a polar solvent fire. Not using the correct foam agent would result in ineffective

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Oxidation and Reduction Approaches for Treatment of Perfluoroalkyl Substances

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Short-Chain PFAS

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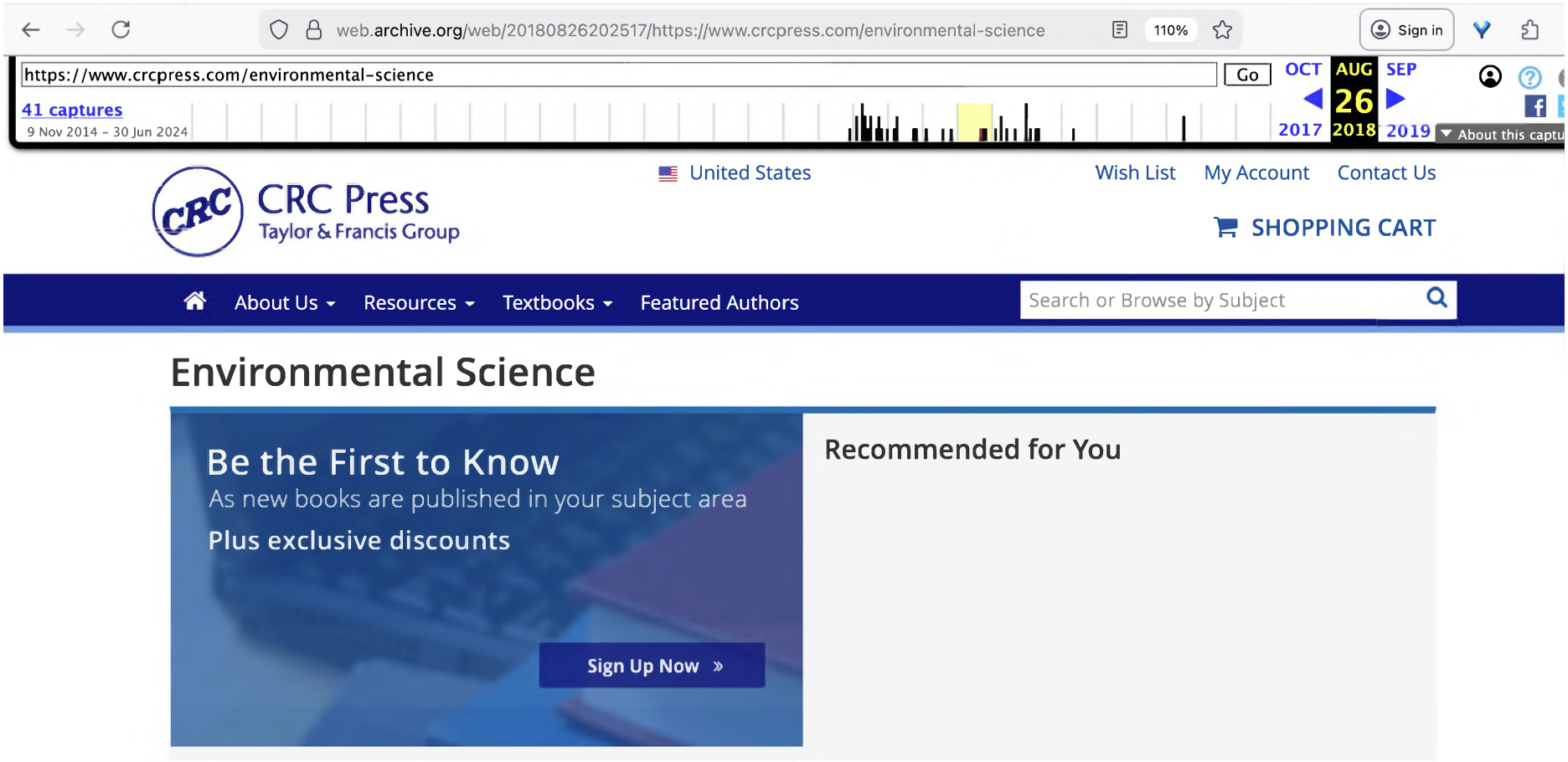
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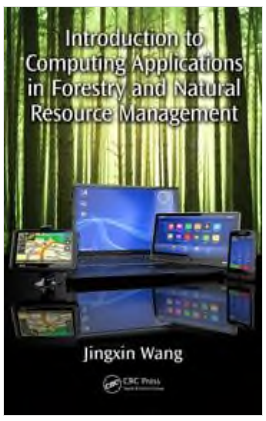


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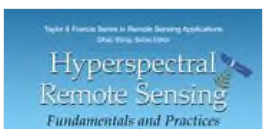


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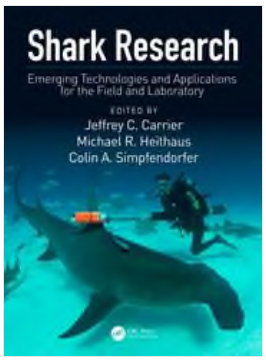
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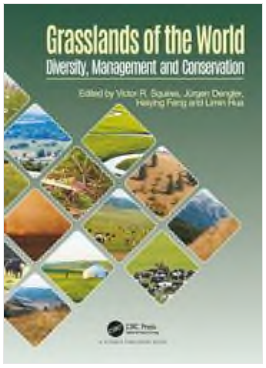
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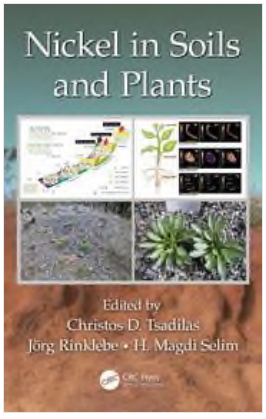
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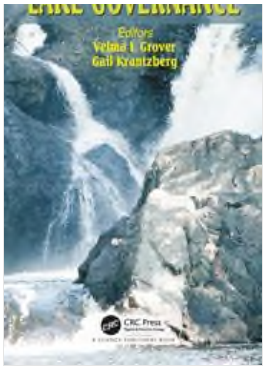
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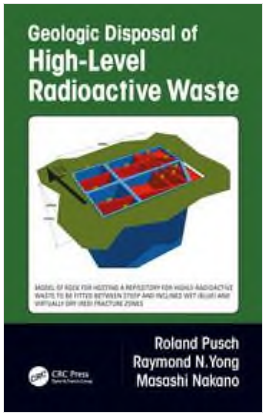
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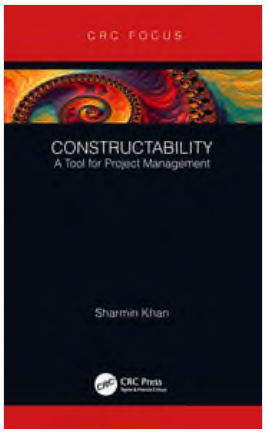
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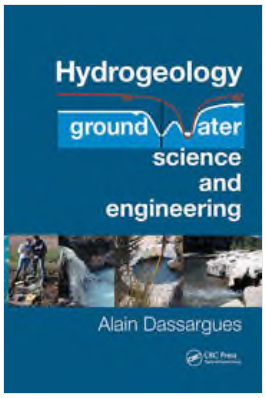


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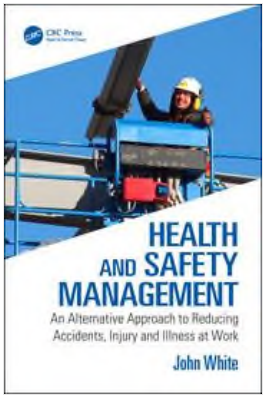
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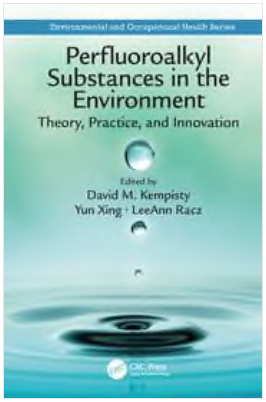
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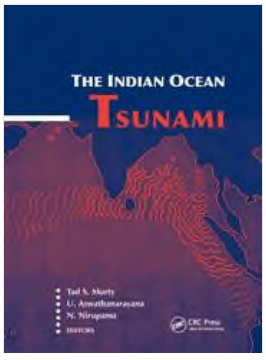
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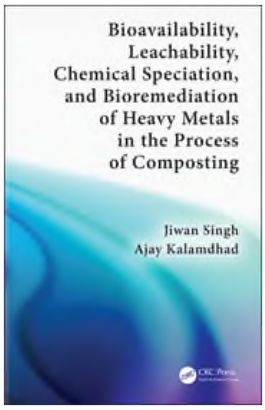


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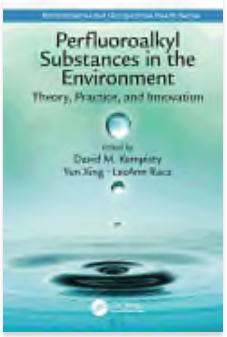
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Publication: Boca Raton, FL : CRC Press, Taylor & Francis Group,

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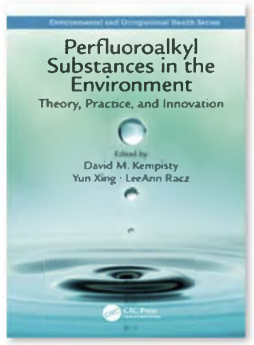
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Theory, Practice, and Innovation

Edited By David M. Kempisty, Yun Xing, LeeAnn Racz

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First Published	2018
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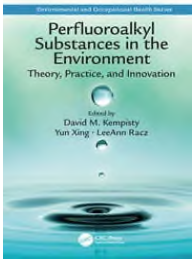
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- Detailed descriptions of conventional and state-of-the-art remediation technologies
- Practical recommendations for dealing with PFAS in a dynamic regulatory landscape
- Robust discussion of important sampling and analytical considerations

Perfluoroalkyl Substances in the Environment: Theory, Practice, and Innovation explores the challenges across the topical areas of regulation and management, to management strategies to effectively protect this and future generations.

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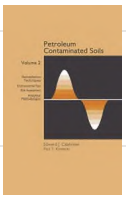
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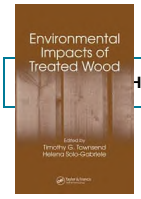


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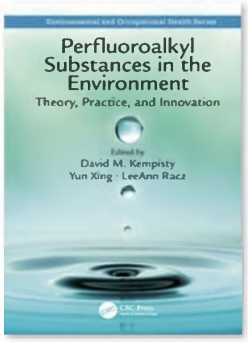
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Theory, Practice, and Innovation

Edited By David M. Kempisty, Yun Xing, LeeAnn Racz

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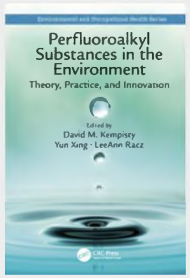


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Per- and polyfluorinated alkyl substances (PFAS), often referred to as per- (and poly) fluorinated compounds (PFCs), have been used for years in many everyday¾ and some lifesaving¾ products. However, their use has been linked to adverse health effects in humans, a problem compounded by their persistence in the environment. This book discusses the various challenges of PFAS in our environment today, including their historical use as well as their chemical and toxicological properties. It also presents robust discussion of analytical challenges and special considerations in sampling. The work goes on to give practical recommendations for dealing with these compounds in today's dynamic regulatory landscape and includes several chapters on various remediation techniques.

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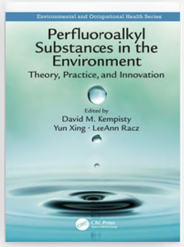
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Remediation Technologies and Methods for Per- and Polyfluoroalkyl Substances (PFAS)

1 Introduction

Remediation technologies exploit chemical and physical properties to immobilize, remove, or destroy the targeted contaminants. Certain PFAS have recently been the subject of regulatory actions and attempted soil, sediment, and water remediation. These compounds have unique chemical properties that require new remediation technologies or innovative combinations of existing technologies. The decision to remediate PFAS should be driven by applicable regulations and an appropriate risk assessment.

USEPA has compiled an online resource for PFAS that includes topics such as policy and guidance, chemistry and behavior, occurrence, toxicology, site characterization, and remediation technologies (USEPA 2017h). The National Groundwater Association (NGWA) has also published a resource on PFAS that includes information about remediation technologies (NGWA

ITRC has developed a series of fact sheets that summarize the latest science and emerging technologies related to PFAS. The purpose of this remediation fact sheet is to:

- provide an overview of remedial technologies and methods for treatment of solids (for instance, soil or sediment) and liquids (for instance, groundwater, leachate, or surface water);
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- describe processes for the treatment of PFAS that are now in use or are under development; and
- describe the challenges and limitations for each treatment technology.

1.1 PFAS Remediation Technologies Overview

Currently, full-scale PFAS treatment in water is limited to sorption using carbon, mineral media (for example, clay), or a combination of these. Additional pilot and bench-scale technologies are currently being tested. This fact sheet discusses the treatment technologies that have been successfully demonstrated through pilot testing at the field-scale. The accompanying tables summarize technologies that have only been tested in limited applications or in laboratory bench tests. Combining technologies may overcome limitations of any one given technology or expand the efficacy of each technology.

For each technology presented, this fact sheet discusses the following key elements:

- *Treatment Description* (according to each applicable media)
- *Treatment Mechanism* (for example, separation, sorption)
- *State of Development* – Is the technology at lab or bench-scale, field pilot-scale, or full-scale implementation? How many demonstration tests and what is the degree of commercialization for each technology?
- *Effectiveness* – Summary of demonstrated effectiveness on a broad range of PFAS (for example, Method 537 (Shoemaker, Grimmett and Boutin 2009) suite plus fluorotelomer sulfonates), a limited subset (for example, Third Unregulated Contaminant Monitoring Rule [UCMR3] list USEPA 2017f), or only perfluorooctane sulfonate (PFOS) and/or perfluorooctanoate (PFOA).
- *Sustainability Considerations* – Design considerations are evaluated including green remediation elements such as carbon footprint from energy usage, treatment media, and residual handling transport, as well as potential community impacts.

The current state of full-scale PFAS treatment in water is limited to sorption using carbon and/or mineral media (for instance, clay).

Two supporting tables comparing PFAS remediation technologies are available in a separate Excel file: Table 1, Solids Comparison, and Table 2, Liquids Comparison. The tables present all reported treatment technologies, including those tested only at the laboratory/bench scale. To be included in the tables, a technology must have been documented in a publicly available document. Some technologies, however, are only documented in literature supplied by the inventor/researcher/vendor of that technology, with no independent confirmation or peer-review process—be aware of possible biases. ITRC periodically updates the supporting tables and maintains the most current version on its website.

Air treatment is not included in this fact sheet because the current research is limited, and this topic is less applicable to site remediation projects.

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1.2 Factors affecting remedy selection

Site characteristics that affect PFAS remedy selection include the nature of the source, release pathways, affected receptors, and fate and transport in the environment (see *Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods; Environmental Fate and Transport;* and *Naming Conventions and Physical and Chemical Properties* fact sheets). Other site characteristics relevant to remedy selection may be identified as remedial technologies advance. Strategies for remediation of the broader class of PFAS may require complementary technologies, several of which are still being developed, as described in Tables 1 and 2.

Other factors affecting PFAS remedy selection include:

- **Characteristics of PFAS**. The wide-ranging chemical and physical characteristics of PFAS affect the remedy effectiveness. Key factors include ionic state (anionic, cationic, and zwitterionic), types of ionic groups (sulfonate or carboxylate), lipo- and hydrophobicity, nature and reactivity of alkyl groups, chain length and branching, partitioning coefficients, volatility, solubility, and acidity.
- **Changes in PFAS properties**. Chemical and physical properties resulting from naturally occurring processes or due to remedial actions for other (commingled) contaminants, such as chlorinated solvents, and petroleum hydrocarbons, can affect PFAS distribution and mobility in groundwater (McGuire et al. 2014). Example changes include:
  - The alkyl functional group of some PFAS may be more readily subject to chemical or biological transformation than the fully fluorinated aliphatic chain. This is the basis, for example, for the total oxidizable precursor (TOP) assay to estimate concentrations of precursor compounds (Houtz and Sedlak 2012).
  - Partial degradation of the carbon-carbon bonds in the aliphatic chain reported for some chemical remedies generates short-chain PFAS, which may be more mobile (Guelfo and Higgins 2013).
  - Modifications in aquifer properties (for example, redox, pH, or other geochemical characteristics) during remediation of comingled contaminants results in a conversion of PFAS to the more stable and mobile perfluorocarboxylic acids (PFCAs) (McKenzie et al. 2015, 2016).
- **Community acceptance**. Communities are often faced with trade-offs in terms of cost, level of clean-up, and residual contamination as part of remediation efforts. Stakeholder engagement and effective risk communication strategies are an important aspect of the overall remedial technology selection process, especially where off-site receptors are identified.

Factors specifically challenging for PFAS remediation include:

- Multiple ionic states
- Variable isomers
- Differing alkyl groups
- Past remediation effects
- Common co-contaminants

1.3 Technical Maturity

The treatment technologies described here are organized by degree of current confidence in the technology. Three levels of confidence are defined as follows:

- *Demonstrated Technologies*—Technologies that have been demonstrated under pilot or full-scale conditions and are well documented for multiple applications in peer-reviewed literature. These technologies are discussed in greatest detail.
- *Partially Demonstrated Technologies*—Technologies that have been documented in peer-reviewed literature by multiple researchers or practitioners but have only been executed at the laboratory or bench scale. These technologies are briefly mentioned.
- *Promising Technologies*—Technologies that have been demonstrated at the laboratory or field pilot-scale, but the results have not been rigorously peer reviewed. Often, these results are only reported by one group (for example, one university, practitioner, or vendor) or lack detailed validation of the treatment or mechanisms. These technologies are omitted from the text and presented only in the technology comparison tables (Tables 1 and 2).

Experimental techniques to treat PFAS are under development, but only those technologies that have some level of publicly available documentation demonstrating effectiveness are included here.

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2 Technologies for Treatment of Solids

Several technologies are currently available to remediate PFAS in solids. Most of these technologies have been demonstrated on soils only but may also apply to saturated sediments or sludge treatment.

Field-Demonstrated Treatment Technologies for PFAS in Solids

- Excavation and off-site landfilling or incineration
- Sorption/stabilization through ex situ soil mixing
- Ex situ thermal desorption and off-gas destruction

2.1 Capping

Technology Description: Capping places a cover over contaminated material such as landfill waste, contaminated soil, and sediments. Caps do not destroy or remove contaminants. The purpose of the cap is to prevent contact with the contamination and, depending on the design, to reduce or prevent further leaching of contaminants. For PFAS soil sites, the main purpose of a cap is to reduce or prevent further leaching of contaminants from soils to groundwater.

Treatment Mechanism: Caps isolate contaminants and prevent them from spreading and causing exposure by direct contact.

State of Development: Capping is a viable remediation method because it is simple technology that applies to most contaminants. Although capping has not been applied to a PFAS site, based on documented successes with other contaminants it offers promise for PFAS.

Effectiveness: Capping may prevent exposure and potentially reduce infiltration, but redevelopment options for the capped land surface may be limited. Caps are most effective when the seasonal high-water table is well separated vertically from the base of the contaminated solids. The PFAS contamination will remain at the site and therefore be a long-term liability. Contaminants could mobilize if site conditions change (for example, rising water table). Caps incur maintenance costs to maintain the integrity of the cap and require institutional controls to ensure the cap is not breached by future site uses or redevelopment.

Sustainability Considerations: The carbon footprint of capping includes earthwork equipment emissions and manufacturing and transporting capping material. Ecosystem restoration can be incorporated into the capping design (Lamb et al. 2014). Community impacts include truck hauling traffic and hindrance of redevelopment due to land use restrictions.

2.2 Excavation and Disposal

Technology Description: This remediation method includes removing contaminated soil and hauling it to a permitted landfill or incineration facility, then filling the excavated area with clean backfill.

Treatment Mechanism: This method isolates PFAS from receptors. Sometimes, PFAS-impacted soil is stabilized or solidified before disposal into a landfill. Disposal of PFAS-impacted soils or wastes into unlined landfills should be avoided. More studies are needed on interactions of PFAS with landfill linings.

Some states may require PFAS-contaminated solids to be treated at a Resource Conservation and Recovery Act (RCRA) permitted incinerator. The estimated temperature to destroy specific PFAS varies widely, from around 300° to greater than 1,000°C (Vecitis et al. 2008). Commercial incineration is often the only viable disposal option for media contaminated with PFAS.

State of Development: Solid wastes generated from environmental investigation and cleanup (for example, soil excavation, and investigation- and remediation-derived wastes) have been disposed at lined landfills, and therefore this is considered a well-demonstrated technology. PFAS have been reported in landfill leachate (Lang et al. 2017), although it is unlikely the source for these PFAS is investigation derived waste, as opposed to consumer product waste containing fluorochemicals. The combination of soil excavation and incineration is also considered to be a viable remedial option.

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Effectiveness: Excavation and disposal of PFAS-contaminated soil effectively removes a source area that may otherwise serve as a continuing source of groundwater contamination but does not result in destruction of the PFAS. Additionally, some nonhazardous waste landfills do not accept PFAS waste. Disposal of PFAS waste to landfills potentially adds to the PFAS contaminant load in the landfill leachate. In some states, the leachate is not analyzed or regulated for PFAS.

Sustainability Considerations: The carbon footprint for this approach includes earthwork equipment emissions, transporting contaminated soil and backfill, and manufacturing (such as resource extraction) of backfill material. Landfill incineration of the contaminated soil is energy intensive. Truck hauling traffic affects the local community. Guidance is available for performing a sustainability assessment for an excavation and disposal remedial design (Cappuyns and Kessen 2014; Goldenberg and Reddy 2014; Söderqvist et al. 2015; Song et al. 2018).

2.3 Sorption and Stabilization

Technology Description: Amendments added to the soil reduce or remove the potential for PFAS to mobilize from soil to groundwater.

A few commercially available sorbents have been specifically developed to immobilize PFAS in soil.

Treatment Mechanism: Amendments adsorb or stabilize PFAS. These amendments include activated carbon and carbon nanotubes (CNTs), resins, minerals, biomaterials, and molecularly imprinted polymers. The amendments bind to PFAS and thus reduce their release from soil.

State of Development: Sorption and stabilization techniques using carbon-based amendments are considered partially demonstrated technologies. Both granular activated carbon (GAC) and powdered activated carbon (PAC) rapidly remove PFAS from groundwater and surface water and can be applied to soil, but efficacy is reduced in the presence of organic co-contaminants (NGWA 2017). Performance also depends on PFAS chain length and functional group (Xiao et al. 2017). Laboratory tests should be conducted using site specific soils and the stabilizing amendment before field application to ensure success. Carbon amendments can be modified to enhance their sorption of PFAS. One patented amendment is carbon enhanced with aluminum hydroxide, kaolin clay, and other proprietary sorbents (USEPA 2017p).

Sorption and stabilization techniques using non-carbon-based sorbents, such as iron oxide minerals and modified organoclays (such as montmorillonite [Mt], hydrotalcite, and palygorskite) are promising, but only limited tests have been conducted. Minerals such as clays, silica, iron oxides, and zeolites have been used as sorbents for removing contaminants from groundwater and soil (Zhu et al. 2016; Rattanaoudom, Visvanathan, and Boontanon 2012; Zhou et al. 2010; Zhou, Pan, and Zhang 2013). The surface of organoclays can also be modified for enhanced PFOS and PFOA sorption (Zhou et al. 2010; Kambala and Maidu 2013; Zhu et al. 2016). Additionally, an amine-modified palygorskite clay sorbent has been patented for the treatment of PFOS and PFOA (Kambala and Maidu 2013).

Effectiveness: Carbon- and mineral-based sorption and stabilization techniques vary in their effectiveness according to site conditions and PFAS type. An example of a site condition that can affect sorption is high organic matter in soil, which can foul carbon sorbents with competing compounds. PFAS type affects sorption in that PFAS often occur as mixtures, including PFAS of different chain lengths with varying sorption characteristics. Sorption capacity was assessed in one study where CNTs were mixed with sediments at 4% weight per weight (w/w), and the sorption capacity of the sediment increased in comparison to untreated sediment (Kwadijk, Valzeboer, and Koelmans 2013).

Organoclays are used because they are environmentally benign, have a high sorption capacity, and can be easily modified to enhance their sorption capacity with mesopores. Organoclays have also been proven to work on several classes of contaminants (Zhu et al. 2016; Espana, Mallavarapu, and Naidu 2015; Zhou et al. 2008). The surface of organoclays are hydrophilic, and therefore ineffective for sorption of hydrophobic organic compounds like long-chain PFAS; however, modification with cations changes the surface to lipophilic. For example, Mt often exists as Na-Mt with a sodium cation on the surface, which is lipophilic and may be effective for long-chain PFAS.

Biomaterials such as chitosan, straw, and quarternized cotton do not perform as well as other sorbents, and the biomaterials may eventually degrade (Du et al. 2014).

Sorption and stabilization do not destroy PFAS, and information on the long-term stability of amendments for PFAS remediation is a data gap that currently limits their use. The amended soil can be mixed with concrete and other stabilizers to better trap the PFAS.

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Sustainability Considerations: The carbon footprint for sorption and stabilization includes emissions from earthwork equipment, manufacturing (for example, resource extraction), and transporting amendment material. Community impacts include hindrance of redevelopment due to land use restrictions. Resources are available for performing a sustainability assessment for sorption and stabilization remedial design (Goldenberg and Reddy 2014; Hou et al. 2016; Kuykendall and McMullan 2014).

2.4 Thermal

Technology Description: The mobilizing or destruction of chemicals using heat.

Treatment Mechanism: Heat is applied directly to the PFAS-contaminated soil. High temperatures can vaporize the chemicals or potentially destroy them. Vaporized chemicals can be captured and destroyed in off-gas treatment.

State of Development: Ex situ thermal treatment has been demonstrated at the field pilot-scale by a few technology vendors and is considered a partially demonstrated technology (Endpoint Consulting 2016; Enviropacific 2017). The use of this technology for PFAS-contaminated soil is still developing. Limited data sets are available, and several data gaps still exist. Additionally, no documented examples of in situ thermal treatment for PFAS-impacted soil have been identified.

Effectiveness: Results of one test indicated that the complete removal of a suite of nine PFAS was possible within 30 minutes but required temperatures over 900º C (Endpoint Consulting 2016). In another field pilot project, concentrations of 20 PFAS in soil were reduced to below reporting limits—greater than 99.9% reduction (Enviropacific 2017). At this time, it is unknown whether the pilot test resulted in volatilization or complete destruction. This test was performed at relatively lower temperatures (for example, approximately 450º C). There are still several data gaps related to thermal incineration of PFAS that should be considered when applying this technology. For example, the mass balance to assess whether PFAS are destroyed or simply mobilized is not completely understood.

Sustainability Considerations: Thermal treatment is an energy-intensive remediation method. Its carbon footprint includes the energy source and consumption during treatment system operation, as well as manufacturing and installation of heating system materials. Community impacts include managing the risks of potential vapor intrusion from volatile co-contaminants. Guidance is available for performing a sustainability assessment for thermal remedial design (Song et al. 2018; Vidonish et al. 2016).

3 Liquids Treatment

Several technologies are currently available for remediating PFAS in liquids. These technologies can be applied to drinking water supplies, groundwater, industrial wastewater, surface water, and other miscellaneous applications (such as landfill leachate). Influent concentrations of PFAS can vary by orders of magnitude for specific media or applications. These influent values, along with other general water quality parameters (for example, pH) can influence the performance and operating costs for the treatment technologies.

Field Demonstrated Treatment Technologies for Liquids

- Extraction and sorption with granular activated carbon or anion exchange resin
- Extraction and membrane filtration/reverse osmosis
- Extraction and precipitation/flocculation

3.1 Sorption

Many organic compounds can be treated by passing contaminated water through special granular media. The mechanism of this technology varies depending on the media, contaminant, and influent concentrations. There are two broad categories of PFAS sorption treatment: adsorption onto carbon media and ion exchange.

3.1.1 Granular Activated Carbon

Technology Description: Granular activated carbon (GAC) is made from organic materials, such as coal and coconut, which serve as effective adsorbent media because they are highly porous and provide a large surface area for contaminant contact. GAC treatment can be used for any aqueous-based treatment application (for example,

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municipal drinking water, groundwater, point-of-use residential, industrial wastewater, and landfill leachate). The GAC media is placed in packed-bed, flow-through vessels generally operated in series (lead-lag configuration). Either virgin or reactivated GAC can be used for most applications, but virgin GAC is the industry preference for more risk-averse drinking water applications. Commercial facilities in the United States conduct thermal reactivation of spent GAC, which can provide a more sustainable and less costly replacement option than virgin GAC and off-site disposal. To address concerns about using reactivated GAC, vendor bench-scale column studies can be performed prior to initial usage, and a quality control testing program can be implemented prior to delivery of each shipment.

Treatment Mechanism: Removal of PFAS from treated water by GAC is an adsorption process, as well as a physical mass transfer process from the aqueous phase onto solid media that does not involve any form of chemical degradation or transformation. Adsorption is a surface chemistry phenomenon by which an aqueous phase contaminant adheres to the surface of a granular media (via electrical, physical, or chemical processes), but does not penetrate it. The GAC adsorption capacity can vary considerably by media and contaminant. Adsorbent media must be removed and replaced when it becomes spent, meaning contaminants break through at concentrations above some established criteria. The spent media must be replaced and shipped off-site either for disposal (by landfilling or commercial incineration) or to be regenerated/reactivated for reuse consistent with applicable federal and state regulations.

State of Development: GAC is a demonstrated technology and is currently the most common water treatment method used for PFAS. Because of the limited treatment history and available technologies for PFAS, full-scale applications to date have mostly focused on higher priority private and public water supply and residential point-of-use treatment. Currently, only a few operating groundwater pump-and-treat applications use GAC treatment. Much of the published literature on GAC treatment of PFAS involves bench-scale and vendor column studies. Treatability data for full-scale operations involving different technologies, including GAC, have also been compiled and reported for several municipal wastewater treatment plants where PFAS have been detected in the influent.

Many sources in the literature support the use of GAC: Appleman et al. (2013); Szabo et al. (2017); and Woodard, Berry, and Newman (2017); and others cited in this section. These references also include more comprehensive bibliographies if further details are needed on specific topics or studies.

Effectiveness: GAC has been shown to reduce select PFAS to very low or nondetectable concentrations, on the order of nanograms per liter (equivalent to parts per trillion), with reported removal efficiencies in various references between 90% and >99%. The lower end of these reported GAC removal efficiencies may be the result of the faster breakthrough times for the short-chain PFAS (Xiao et al. 2017; Dickenson and Higgins 2016). Early municipal treatment plant sampling studies focused mostly on PFOA and PFOS, which are considered long-chain PFAS (defined as six carbon atoms or more) and currently drive most drinking water treatment decisions. Consequently, the shorter breakthrough times for PFAS with five carbon atoms or less were initially missed, and subsequently reported in the literature as lower GAC removal efficiencies.

Breakthrough and GAC usage should be checked for a variety of PFAS, not just PFOA or PFOS, including various chain-lengths.

Individual PFAS have different GAC usage capacities and corresponding breakthrough times. GAC removal capacity for PFOS is greater than PFOA, but both can be effectively captured. In general, PFAS containing five carbon atoms or less have higher GAC usage and much quicker breakthrough times than PFAS containing six carbon atoms or more with other factors being equal (such as influent concentration). Vendor column studies (Brewer 2017) with equivalent influent concentrations and empty contact bed times have shown that short-chain PFAS breakthrough times are approximately five times quicker than long-chain PFAS. Pilot and full-scale GAC treatment data show similar comparative breakthrough times (Brewer 2017).

In addition to usage capacity, several other factors affect GAC change-out frequency and cost for individual PFAS (for example, influent concentrations). Change-out frequency therefore cannot be predicted solely by the presence of specific short-chain PFAS. When concentrations of short-chain PFAS are much lower than concentrations of long-chain PFAS, GAC is still a cost-effective treatment for PFAS other than PFOA and PFOS. Because of the differences in GAC usage capacities between individual PFAS, treatability studies must evaluate the entire mixture of PFAS present in the influent to the extent practicable. Column studies are the best method to predict GAC performance and change-out frequency.

Column studies show that virgin GAC and thermally reactivated GAC have similar removal rates and breakthrough times (Brewer 2017). Based on vendor feedback (Mimna 2017), commercial thermal reactivation is performed at higher

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operating temperatures than steam or nitrogen regeneration systems and is capable of complete desorption and destruction of PFAS from spent GAC (Watanabe et al. 2016; Yamada et al. 2005). Also, vendor testing demonstrates that re-agglomerated bituminous coal provides better removal performance for PFAS than other types of GAC (Brewer 2017; Nowack 2017).

Sustainability Considerations: The carbon footprint for GAC includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. Spent single-use media requires incineration, which increase the carbon footprint. Regenerable media presents sustainability benefits because the media is reused; however, in drinking water applications, virgin material achieves greater confidence in treatment. Resources are available for performing a sustainability assessment for sorption remedial design (Amini et al. 2015: Choe et al. 2013, 2015; Dominguez-Ramos et al. 2014; Favara et al. 2016; Maul et al. 2014; Rahman et al. 2014; Ras and von Blottnitz 2012).

3.1.2 Biochar

Technical Description: *Biochar* is a hybrid word derived from *biomass* and *charcoal*. Biochar is a carbon-rich, porous solid synthesized from biomass, such as wood or manure, through a high-temperature low-oxygen process called "pyrolysis" (Ahmad et al. 2014). Key factors controlling the properties of biochar (for example, pore size, chemical composition, and hydrophobicity) include the temperature of pyrolysis and biomass feedstock.

Treatment Mechanism: The properties of biochar are comparable to those of GAC for sorptive purposes. Like GAC, biochar can adsorb organic contaminants.

State of Development: Biochar is considered a partially demonstrated technology. Various laboratory experiments have evaluated the efficacy of biochar compared to other media, but no full-scale treatment systems are in place for the removal of PFAS. This work demonstrates that biochar is potentially viable for treatment of PFAS, but additional research is needed to fully establish viability and costs.

Effectiveness: Xiao et al. (2017) compared one GAC and two commercially available biochars for treating an aqueous film-forming foam (AFFF)-impacted water supply. Based on batch studies, they concluded that biochars with large surface areas could be an alternative to GAC, although variability in biochar properties relative to GAC may affect reliability. While biochar removal is effective in ultrapure water, when used to treat river water (with more complicated water chemistry), biochar is ineffective compared to ion exchange and GAC and exhibited significantly slower adsorption kinetics (Rahman 2014).

Sustainability Considerations: The carbon footprint of this technology includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. The use of waste material as a starting feedstock results in a lower overall carbon footprint, though reactivation of biochar is not currently feasible and energy-intensive incineration or landfilling are required, which offsets some of the sustainability benefits.

3.1.3 Ion Exchange

Technology Description: Ion exchange (IX) uses synthetic, polymeric media to remove PFAS from water. IX media are employed similarly to GAC and can be used in combination with GAC.

Both regenerable and nonregenerable IX media are available. Nonregenerable IX is a single-use, disposable medium. Regenerable IX theoretically can be used indefinitely, however, insufficient operational data are available to understand its long-term durability. IX regeneration is a chemical process; the only demonstrated successful regeneration solution is a solvent-brine solution (Amec Foster Wheeler 2017). The regenerant solution can be distilled for reuse. The distillate residue is a concentrated PFAS waste that can be managed by off-site treatment (for example, incineration or possibly chemical oxidation).

Treatment Mechanism: IX is the process by which ions of one substance are replaced by similarly charged ions of another substance. The term denotes purification, separation, and decontamination of aqueous and other ion-containing solutions with solid polymeric or mineralic ion exchangers. Many organic contaminants are ionic and can be removed through specialized ion exchange media. The media are often derived from organic polymers or plastic, and thus ion exchange media are referred to as "resins." Regeneration of ion exchange resins is accomplished with a chemical flush, typically a highly acidic or basic solution, brine solution, or solvent-brine solution, rendering the resin reusable.

To date, IX has used positively charged, anion exchange media to remove negatively charged PFAS molecules, via binding of the carboxylic and sulfonic acid "heads" of perfluoroalkyl acids (PFAAs). Such IX media are manufactured to

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be more selective for PFAS than for typical competing mineral anions such as sulfate and nitrate. The fluorinated carbon chain of the PFAS molecule can also adsorb to IX media. This dual-mechanism, ion exchange plus adsorption, can result in higher removal compared to adsorption alone (Yu et al. 2009).

State of Development: IX is a fully demonstrated technology. Column tests comparing both regenerable and single-use ion exchange media have shown IX to be effective for the removal of several PFAS (Woodard, Berry, and Newman 2017; Conte et al. 2015). Full-scale IX systems are currently in operation in Australia at Australian Defense sites, and a full-scale system was installed in the United States in 2017. The U.S. Department of Defense (DOD) recently funded a SERDP project, ER18-C2-1306: "Combined In Situ/Ex Situ Treatment Train for Remediation of Per- and Polyfluoroalkyl Substance (PFAS) Contaminated Groundwater" to optimize regenerable IX and on-site destruction with plasma under a number of laboratory conditions (SERDP-ESTCP 2017).

Effectiveness: IX is a demonstrated effective technology for removal of anionic PFAS. It has higher adsorption capacity for some PFAS and significantly faster reaction kinetics compared to GAC (Conte et al. 2015). The combination of these properties means an equivalent treatment system for IX is smaller and thus uses less media. Like GAC, usage capacities and corresponding breakthrough times vary depending on PFAS functional groups and chain length. Short chain PFAS may break through faster under certain influent conditions. However, certain single-use IX media have been identified that may have higher usage capacities for short-chain PFAS. An IX treatment system can also include multiple vessels in a lead-lag configuration, consisting of both single-use and regenerable IX media. This design optimizes removal properties and operating costs for a mixture of short and long-chain length PFAS.

Sustainability Considerations: The carbon footprint for IX includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. Spent single-use IX media requires incineration or other treatment for disposal, resulting in an increased carbon footprint. Regenerable IX media offers sustainability benefits because the media can be reused, but the process for regenerating requires energy and creates a concentrated waste stream which must be managed. Disposal or treatment of the regenerant stream can be problematic and expensive. Regeneration solutions may present exchange system and treated water corrosion issues if media are not rinsed thoroughly prior to being placed back in service.

3.2 Precipitation/Flocculation/Coagulation

Technology Description: Coagulation–flocculation is a common pretreatment approach used in wastewater treatment plants for removing various particles and dissolved constituents. Coagulants, either commodity or proprietary chemicals, can be added to water (conventional technology) or generated by anode-cathode reactions of metals plates inserted into the water (electrocoagulation).

Technology Mechanism: Coagulants assist in forming solids. Flocculation is typically conducted by adding a soluble polymer and slowly mixing to allow the particles to agglomerate and grow. Upon solid formation, constituents such as PFAS can be physically incorporated into, or sorbed onto, the flocculated particulate (which is known as co-precipitation). The precipitated solids are then separated from the water by sedimentation, filtration, or a combination of both processes. The solid material containing the PFAS requires disposal.

State of Development: Current literature only documents bench-scale study results on treating PFAS via precipitation, flocculation, or coagulation and therefore this is considered a partially developed technology. Evaluations have focused on conventional commodity chemical coagulation (for example, aluminum or ferric salts) and nonconventional coagulation (for example, proprietary chemical coagulants or electrocoagulation). Pilot and full-scale applications have not been documented in the United States (Birk and Alden 2017).

Unconventional precipitation (for example, electrocoagulation or advanced chemical precipitants) has shown more potential for direct PFAS treatment, but little data is available.

Effectiveness: Electrocoagulation reactors, which range from basic to sophisticated designs, are highly efficient, compact, relatively low-cost, and completely automatable (Baudequin et al. 2011; Lin et al. 2015a). Recent studies have found that PFAAs, such as PFOA and PFOS, can be quickly sorbed on the surface of zinc hydroxide particulates generated by electrocoagulation (Lin et al. 2015a).

One commercially available proprietary chemical product has been shown in tests to reduce PFAS in groundwater containing AFFF (Birk and Alden 2017; CH2M 2017). This research also shows that conventional (ferric salt) and proprietary chemicals in combination are more effective than either alone. Current data have been developed at elevated

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concentrations, but removal for lower (µg/L) concentrations has also been shown (CH2M 2017; Birk 2015). No available data show precipitation effectiveness at very low (ng/L) concentration ranges.

Conventional PFAS precipitation induced by coagulation and flocculation has shown limited applicability for complete treatment of PFAS. Therefore, conventional chemical precipitation could be considered mainly as a pretreatment technology to sorb or precipitate PFAS prior to final filtration or destruction. Also, if used to remove conventional parameters such as solids or natural organic material (NOM), pretreatment would allow PFAS removal technologies to achieve the desired treatment goals in a more cost-effective and technically feasible manner.

Sustainability Considerations: The carbon footprint of this technology includes energy source and consumption during treatment system operation, as well as manufacturing of treatment media. Additionally, extracted solids containing PFAS require final disposal or destruction.

3.3 Redox Manipulation

Technology Description: Redox manipulation is the process of changing the oxidation-reduction potential of water through addition of oxidizing or reducing amendments, or by adding energy to a system to create oxidizing or reducing free radicals. These changes affect the mobility or structure (transformation or destruction) of the PFAS. PFCAs are generally more amenable to redox manipulation than perfluoroalkane sulfonic acids (PFSAs). Redox manipulation may be effective at treating many co-contaminants and may also alter organic matter, which can affect PFAS mobility. These system changes may allow subsequent treatment steps to manage target PFAS more effectively.

Treatment Mechanisms: Redox manipulation mechanisms may include both redox transformation (oxidative, reductive, and nucleophilic processes) of PFAS and changes in redox conditions in the impacted media (for example, groundwater), resulting in changes in the mobility and sorption of PFAS (McKenzie et al. 2015; Arvaniti et al. 2015). Redox transformation involves the transfer of electrons between reactants. In oxidative processes, electrons are transferred to the reactive species (the oxidant) from the target (PFAS), whereas in reductive processes the opposite occurs. In nucleophilic processes, a reactant (the nucleophile) bonds with the PFAS compound and displays an atom or group of atoms from the PFAS molecule.

Susceptibility to redox transformation depends on reaction conditions and the reactive species involved. For some specific technologies, more than one transformation mechanism or reactive species may be involved. For example, plasma, sonolytic, and photolytic technologies may combine physical (high-temperature pyrolysis) and free radical attack processes. PFAS carbon chains do not easily transform, because their carbon-carbon bonds are shielded in part by the tightly bound fluorine atoms that surround the carbon chain (Kissa 2001). The carboxylic or sulfonic group "heads" of PFAS are commonly more susceptible to redox transformation than carbon chain "tails," resulting in partial transformation of the parent compound but not in cleavage of the aliphatic chain (Houtz and Sedlak 2012; Anumol et al. 2016). The tail and head structure are illustrated for PFOS and PFOA in the following figure.

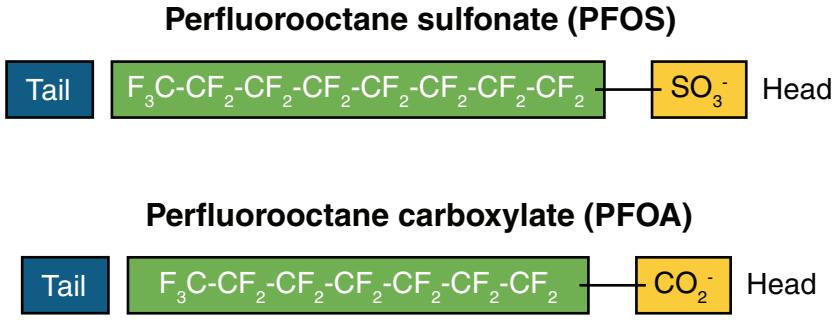


Figure 1. The tail and head structure of PFOS and PFOA molecules.

Reactions of zero valent metals with PFAS, generally considered a reductive process, may also affect mobility and sorption of PFAS in addition to (or rather than) PFAS transformation (Arvaniti et al. 2015). Complete mineralization/defluorination of PFAS via zero valent metal reactions has not been demonstrated to date; however, research in this area is ongoing.

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Combined redox and nucleophilic attack almost completely mineralize PFOA (Niu et al. 2012; Mitchell et al. 2014). A reactant formed in several redox manipulation approaches, the solvated electron (free electron in solution), shows promise for PFAS destruction (Park et al. 2009; Gu et al. 2016; Li et al. 2012; Stratton et al. 2017; Blotevogel, Giraud, and Borch 2018).

State of Development: Redox technologies have not been widely applied beyond laboratory bench-scale tests because of concerns that partial transformation (as opposed to complete destruction) will produce more mobile or toxic products. Therefore, redox technologies are considered a partially developed technology, with both successes and challenges. For example, chemical oxidation affects PFAA transport, but the direction (increased or decreased transport) and magnitude depend on reaction conditions (McKenzie et al. 2015). Apparent in situ destruction of PFAAs in groundwater at a fire training site has also been achieved using a combined ozone/persulfate approach (Eberle, Ball, and Boving 2017). The main difficulty in using redox-based technologies is achieving extremely low (ng/L) PFAS cleanup objectives, particularly for in situ remedies. Additionally, reactive species (for example, oxidizing radicals) interact with other compounds present in soil and groundwater that are more susceptible to oxidation and are at relatively higher concentrations than the target PFAS.

Despite these challenges, several options show promise for ex situ treatment, including electrochemical (Schaefer et al. 2015; Urtiaga et al. 2015), sonolytic (Vecitis et al. 2008; Rodriguez-Freire et al. 2015), plasma (Stratton et al. 2017), and reductive (Arvaniti et al. 2015) technologies. These approaches have successfully degraded an array of high-concentration PFAS at the laboratory scale. However, none of these technologies are sufficiently mature yet to assess PFAS treatment costs and overall effectiveness with confidence.

Effectiveness: A wide range of PFAS treatment methods based upon oxidation-reduction chemistries have been evaluated. These studies demonstrate the following:

- PFAS exhibit a wide range of reactivity toward destructive processes, based upon characteristics including chain length, nature of alkyl groups, and branched versus linear isomers.
- Treatment of PFAS at AFFF sites depends on the composition and production processes of the given AFFF formulation used at the site.
- Except for limited field pilot testing of one ozone and one persulfate-based chemical oxidation technology, none of the technologies summarized in Table 2 have matured beyond laboratory-scale studies.
- While promising, the relatively few field pilot applications have not yet been closely analyzed or duplicated in highly controlled and monitored studies.

Partial transformation can affect other physical and chemical characteristics of PFAS (see Section 1.2). For instance, persulfate-based transformation of PFAS may be incomplete (Houtz and Sedlak 2012) or negligible (McKenzie et al. 2015, 2016), whereas in other cases efficient transformation is reported (Hori et al. 2008; Lee et al. 2009; Liu et al. 2012; Park et al. 2016; Yin et al. 2016). Redox processes in the subsurface can generate unwanted byproducts (for instance, nitrate and bromate from chemical oxidation) depending on site and reaction conditions (Siegrist, Crimi, and Simpkin 2011). Site-specific treatability tests are recommended.

Sustainability Considerations: The carbon footprint for this technology includes energy source and consumption during treatment system operation, manufacturing of amendment materials, and manufacturing and installation of injection points (if implemented in situ). Electrochemical treatment options also require handling of hazardous investigation derived waste, which is a sustainability consideration.

3.4 Membrane Filtration

Membrane filtration refers to a variety of separation technologies based on the nominal size of the membrane pores. Types of membranes include reverse osmosis (RO), nanofiltration (NF), microfiltration (MF), and ultrafiltration (UF). Low pressure membranes such as MF and UF cannot reject PFAS since their pore sizes are larger than the effective diameter of the PFAS molecules (about 1 nm) (Tsai et al. 2010; Rahman et al. 2014). For that reason, MF and UF are not discussed here. Although bench-scale studies indicate that the membrane molecular weight cut-off (MWCO) of NF/RO is probably the most important factor for removal of PFAS for these technologies, other factors, such as ionic charge, may also influence performance.

Two terms, “salt passage” and “salt rejection,” generally describe how membrane systems perform. Salt passage is the percentage of dissolved constituents (contaminants) in the feedwater allowed to pass through the membrane. The opposite of salt passage, salt rejection, is the percentage of feed water that does not pass through the membrane. In

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general, NF membranes have lower rejection rates (95%) than RO (> 99%), because NF membranes have larger pores (Rahman et al. 2014 provides an excellent review of many of the relevant studies). As with all other treatment options, bench-scale and pilot-scale testing are required to understand the field applicability and establish essential detailed design criteria such as pretreatment needs and cost effectiveness.

3.4.1 Reverse Osmosis

Technology Description: RO membranes are effective in removing most organic and inorganic compounds from water solutions. In recent years, new polymer chemistry and manufacturing processes have improved efficiency, lowering operating pressures and reducing costs. As a result, RO membranes are increasingly used by industry to concentrate or remove chemicals. RO is commonly used around the world in household drinking water purification systems, the production of bottled mineral water, self-contained water purification units (for the U.S. military), and industrial applications (for example, water supply to cooling towers, boilers, and deionized water). The largest application of RO is in desalination.

Reverse osmosis is most useful as one element within a treatment train, where the reject water is further treated or captured.

Treatment Mechanism: RO separates compounds from water solutions by passing pressurized water across a semipermeable membrane. Treated water (permeate) passes through the membrane and the rejected water (concentrate) is collected for disposal or discharge, depending on the nature of the compounds and particles present.

State of Development: RO has been studied in bench-scale studies and pilot plants for wastewater and drinking water PFAS applications and is considered a partially developed technology. Conventional and advanced treatments have been studied in several pilot plants and drinking water treatment plants, demonstrating both treatments operating simultaneously as well as the effectiveness of traditional drinking and wastewater treatment methods alongside PFAS-specific technologies.

Effectiveness: Influent pretreatment is critical for RO membranes because of their spiral-wound design. Membranes are highly susceptible to fouling (loss of production capacity) because some accumulated material cannot be removed from the membrane surface.

RO removal of PFAS from various waters has been studied (for example, semiconductor wastewater, drinking water, surface water, and reclaimed water), combined with NF in some cases. PFOS removal > 99% was achieved using four different types of membranes over a wide range of feed concentrations, from 0.5 to 1500 mg/L (Tang et al. 2006). Another study tested five RO and three NF membranes at feed concentrations of 10 mg/L PFOS over four days (Tang et al. 2007). The PFOS rejection and permeate flux performances were > 99% for RO and 90 to 99% for NF. The use of RO and NF as advanced drinking water treatments is still limited, but both technologies have been shown to be successful for the removal of longer-chain (> C5) PFAAs (Loi-Brugger et al. 2008; Tang et al. 2006). Conventional and advanced treatment efficiencies to remove PFOA and PFOS from surface water of the Llobregat River in northeast Spain were also studied. Results were compared in several pilot plants, and in a drinking water treatment plant that operates with UF and RO treatment alongside traditional treatment processes (Flores et al. 2013).

Another study examined the fate of PFSAs and PFCAs in two water reclamation plants that further treat water from wastewater treatment plants in Australia (Thompson et al. 2011). Plant A used adsorption and filtration methods alongside ozonation, while Plant B used membrane processes and an advanced oxidation process, to produce purified recycled water. At both facilities, PFOS, perfluorohexane sulfonate (PFHxS), perfluorohexanoate (PFHxA), and PFOA were the most frequently detected PFAS. Comparing the two reclamation facilities, Plant A showed some removal during the adsorption/filtration stages. Overall, however, Plant A failed to completely remove PFOS and the PFCAs shorter than perfluorononanoic acid (PFNA) in chain length. All PFAS present were removed by RO at Plant B from the finished water to concentrations below detection and reporting limits (0.4–1.5 ng/L).

Sustainability Considerations: The carbon footprint for this technology includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. RO requires power for high-pressure pumps and the management of concentrate, which can be energy intensive. The contaminant-rich brine rejected by RO must be disposed of appropriately. The removal of nontarget minerals from the treated water may increase its corrosivity, and posttreatment corrosion control measures are needed in most cases. Resources are available for performing a sustainability assessment for membrane filtration remedial design (Ras and von Blottnitz 2012).

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3.4.2 Nanofiltration

Technology Description: NF is a form of membrane technology that is pressure-driven and shown to be effective in the removal of PFAS (Tang et al. 2007). This method provides high water flux at low operating pressure (Izadpanah and Javidnia 2012). Like RO, NF is easy to operate and reliable for the removal of chemicals.

Technology Mechanism: Nanometer-sized membrane pores are used to separate compounds in a process similar to RO, but NF does not remove smaller ions such as chloride and sodium.

State of Development: NF is considered a partially developed technology because available data on the removal of PFAS are limited to laboratory-scale tests performed on flat sheet membrane coupons.

Effectiveness: No studies have reported either pilot or full-scale performance of NF membranes. Therefore, variations in performance due to fouling, flux, and concentration distributions in standard spiral-wound membrane configurations have not been characterized.

NF membranes tested include the Dow membranes NF-270, NF-200, and NF-90 and the SUEZ (formerly GE Water & Process Technologies) DK membrane. Reported rejections were generally > 95% for PFAS with molecular weights ranging from 214 g/mol to 713 g/mol (Steinle-Darling and Reinhard 2008; Appleman et al. 2013). However, lower rejections were observed for perfluoropentanoate (PFPeA) and perfluorooctane sulfonamide (FOSA)—about 70 and 90%, respectively (Steinle-Darling and Reinhard 2008). Recovery and salt passage information was not reported in these studies. Scaling up these results is challenging because rejection is affected by recovery, which can vary from one application to another.

Salt passage for PFOS was reported to range from < 1% for the tighter NF-90 membrane to about 6% for the looser NF-270 and DK membranes (Tang et al. 2007). PFOS salt passage also was correlated to sodium chloride salt passage, a common specification for membrane manufacturers (Tang et al. 2007). Salt passage incorporates both rejection and membrane recovery, therefore is a more useful parameter for predicting full-scale performance than rejection.

Pilot and full-scale testing of the selected NF membrane for PFAS removal is a crucial step when considering this treatment process. Choosing membranes with MWCO smaller than the targeted PFAS is also a key design consideration.

Appropriate disposal or treatment of the membrane concentrate stream is another design factor, especially when using high-pressure membranes for inland communities. Furthermore, NF membrane fouling mechanisms are poorly understood and further research is needed to develop cost-effective cleaning methods to restore membrane performance (Al-Amoudi and Lovitt 2007). As with other forms of filtration, pretreatment strategies to avoid fouling and membrane fabrication drive performance results.

Sustainability Considerations: The carbon footprint for this technology includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. NF requires power for pumps (generally less than RO), and the management of concentrate, which can be energy intensive.

4 References and Acronyms

The references cited in this fact sheet, and the other ITRC PFAS fact sheets, are included in one combined list that is available on the ITRC web site. The combined acronyms list is also available on the ITRC web site.



Per- and Polyfluoroalkyl Substances (PFAS) Team Contacts

Robert Mueller • New Jersey Department of Environmental Protection
609-984-3910 • Bob.Mueller@dep.nj.gov

Virginia Yingling • Minnesota Department of Health
651-343-2890 • virginia.yingling@state.mn.us

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This Interstate Technology and Regulatory Council (ITRC) online document includes fact sheets prepared by the ITRC PFAS team. The team is currently working on the associated Technical and Regulatory Guidance Document.

ITRC has developed six **fact sheets** to summarize the latest science and emerging technologies for per- and polyfluoroalkyl substances (PFAS). The fact sheets are tailored to the needs of state regulatory program personnel who are tasked with making informed and timely decisions regarding PFAS-impacted sites. The content is also useful to consultants and parties responsible for the release of these contaminants, as well as public and tribal stakeholders. The fact sheets in the series are:

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6. Remediation Technologies and Methods.

An **introductory document** has also been prepared. This web site also includes a combined **references list** and **acronyms list**.

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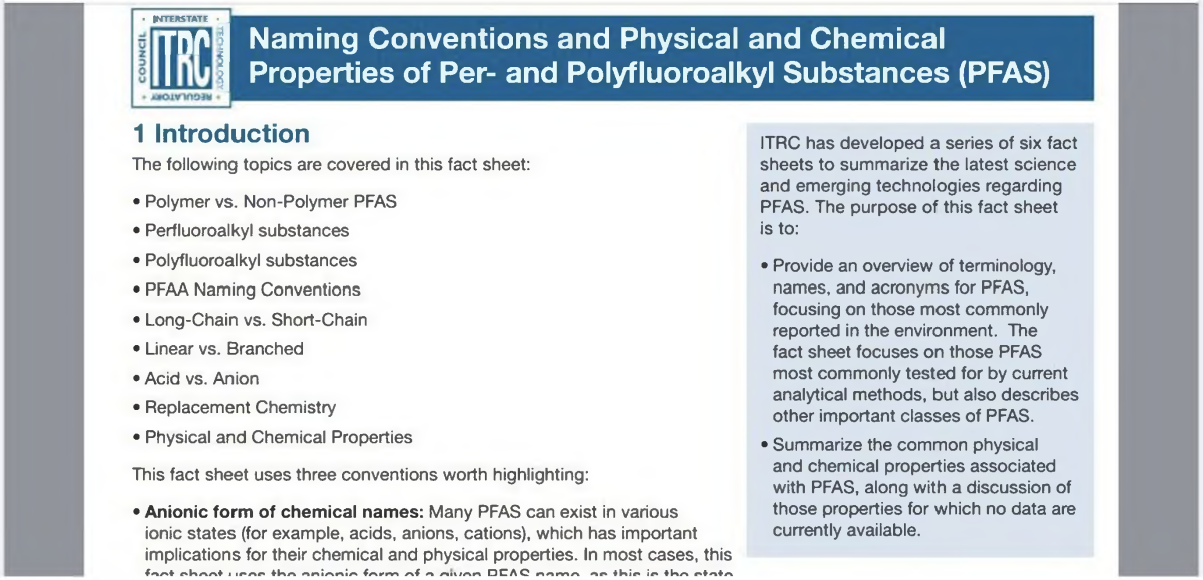
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The first three fact... **See more**



Naming Conventions and Physical and Chemical Properties of Per- and Polyfluoroalkyl Substances (PFAS)

1 Introduction

The following topics are covered in this fact sheet:

- Polymer vs. Non-Polymer PFAS
- Perfluoroalkyl substances
- Polyfluoroalkyl substances
- PFAA Naming Conventions
- Long-Chain vs. Short-Chain
- Linear vs. Branched
- Acid vs. Anion
- Replacement Chemistry
- Physical and Chemical Properties

This fact sheet uses three conventions worth highlighting:

- **Anionic form of chemical names:** Many PFAS can exist in various ionic states (for example, acids, anions, cations), which has important implications for their chemical and physical properties. In most cases, this

ITRC has developed a series of six fact sheets to summarize the latest science and emerging technologies regarding PFAS. The purpose of this fact sheet is to:

- Provide an overview of terminology, names, and acronyms for PFAS, focusing on those most commonly reported in the environment. The fact sheet focuses on those PFAS most commonly tested for by current analytical methods, but also describes other important classes of PFAS.
- Summarize the common physical and chemical properties associated with PFAS, along with a discussion of those properties for which no data are currently available.

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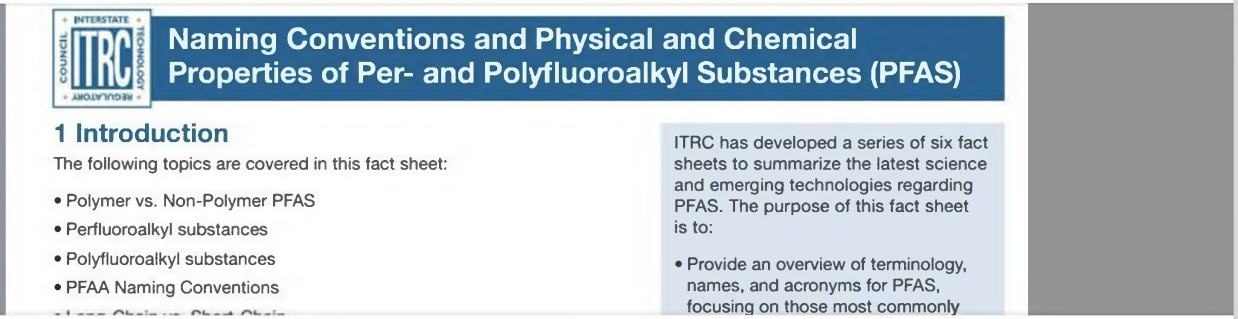
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The first three fact sheets were released last week. Here is the Naming Conventions and Physical and Chemical Properties Sheet, which contains some useful information.

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This fact sheet uses three conventions worth highlighting:

- **Anionic form of chemical names:** Many PFAS can exist in various ionic states (for example, acids, anions, cations), which has important implications for their chemical and physical properties. In most cases, this fact sheet uses the anionic form of a given PFAS name, as this is the state in which most PFAS exist in the environment.

focusing on those most commonly reported in the environment. The fact sheet focuses on those PFAS most commonly tested for by current analytical methods, but also describes other important classes of PFAS.

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1. Naming Conventions and Physical and Chemical Properties
2. Regulations, Guidance, and Advisories
3. History and Use
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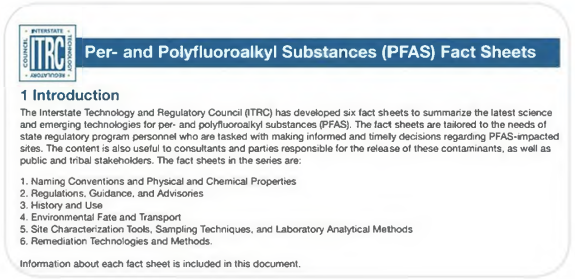
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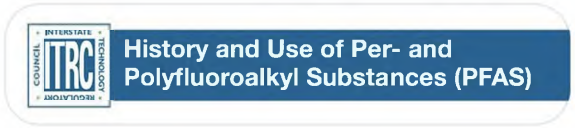
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Information about each fact sheet is included in this document.

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1) Environmental Fate and Transport
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The last fact sheet on Aqueous Film Forming Foam is set to be published August 2018. Access the fact sheets here: **See less**



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11/30/2018 - BECOME AN ITRC PFAS TRAINING SPONSOR! (/web/20190719230817/http://www.itrcweb.org/Content/images/News/Sponsorship_Ad.jpg)

11/20/2018 - ITRC 2019 Online Training Schedule is Available (/web/20190719230817/http://www.itrcweb.org/Documents/TeamResources_OutreachMaterials/ITRC-2019-Classes-040419.pdf)

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10/30/2018 - 2018 IAP and Team Membership Registration Will Close on Friday, November 2, 2018 (/web/20190719230817/http://www.itrcweb.org/about/2018-IAP-and-Team-Membership-Registration-Will-Close)

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10/9/2018 - ITRC Releases Aqueous Film-Forming Foam (AFFF) Fact Sheet (/web/20190719230817/http://www.itrcweb.org/about/AFFF-Fact-Sheet)

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9/27/2018 - Successful PFAS Fall Meeting! (/web/20190719230817/http://www.itrcweb.org/about/Successful-PFAS-Fall-Meeting)

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8/2/2018 - PFAS Fact Sheets in Spanish / Fichas Técnicas de las PFAS en Español (/web/20190719230817/http://www.itrcweb.org/About/PFAS-Fact-Sheets-in-Spanish)

7/20/2018 - Sharing Methane Detection Technologies at ARPA-E (/web/20190719230817/http://www.itrcweb.org/About/News Item - Sharing Methane Detection Technologies at ARPA-E)

7/13/2018 - Updated Information on PFAS Standards and Guidance Values (/web/20190719230817/http://www.itrcweb.org/About/2018-Updated-Information-on-PFAS-standards-and_Guidance-Values)

6/29/2018 - Welcoming Janet Waldron as the new In Situ Co-Lead! (/web/20190719230817/http://www.itrcweb.org/About/Waldron)

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6/15/2018 - Battelle Shares its Appreciation for ITRC! (/web/20190719230817/http://www.itrcweb.org/About/battelle)

6/11/2018 - ITRC Supports Environmental Collaboration in China (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Supports-Environmental-Collaboration-in-China)

6/5/2018 - ITRC Welcomes Randy Chapman as the new Team Leader Liaison (/web/20190719230817/http://www.itrcweb.org/About/Randy-Chapman)

5/25/2018 - ITRC Training Seminar on Characterization and Remediation of Fractured Rock in Kansas City, MO (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Training-Seminar-MO)

5/16/2018 - ITRC's Biannual Newsletter Showcasing Winter 2017 & Spring 2018's Key Events (https://web.archive.org/web/20190719230817/https://mailchi.mp/a30db717f7ee/itrconnect-spring-2018)

5/10/2018 - ITRC Identifies Emerging Contaminants and Public Water as Top Priority Issues (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Identifies-Emerging-Contaminants)

5/9/2018 - ITRC Announces New Co-Chair (/web/20190719230817/http://www.itrcweb.org/About/2018-ITRC-Announces-New-Co-Chair)

5/3/2018 - ITRC Congratulates Award Winners! (/web/20190719230817/http://www.itrcweb.org/About/2017Awards)

4/26/2018 - ITRC Releases Newest Guidance on Quality Considerations for Multiple Aspects of Munitions Response Sites (/web/20190719230817/http://www.itrcweb.org/About/QCMR-1-Release)

4/26/2018 - ITRC Annual Meeting (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Annual-Meeting-2018)

4/6/2018 - ITRC at the 28th Annual International Conference on Soil, Water, Energy, and Air (/web/20190719230817/http://www.itrcweb.org/About/28-International-Conference)

3/20/2018 - ITRC is thrilled to release three new PFAS fact sheets! (/web/20190719230817/http://www.itrcweb.org/About/three-new-PFAS-fact-sheets)

3/14/2018 - ITRC Releases Newest Guidance on LNAPL Update (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Releases-Newest-Guidance-on-LNAPL-Update)

3/13/2018 - ITRC Co-Chair Voting (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Co-Chair-Voting)

2/27/2018 - Plenary Speakers Announced for ITRC Annual Meeting! (/web/20190719230817/http://www.itrcweb.org/About/Plenary-Speakers-Announced-for-ITRC-Annual-Meeting)

2/16/2018 - ITRC Annual Meeting Registration Reminder (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Annual-Meeting-Registration-Reminder)

2/9/2018 - ITRC in Israel (/web/20190719230817/http://www.itrcweb.org/About/ITRC-in-Israel)

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1/16/2018 - The 2018 ITRC Annual Meeting (/web/20190719230817/http://www.itrcweb.org/About/The-2018-ITRC-Annual-Meeting)

12/04/2017 - Registration is open for 2018 ITRC Teams (/web/20190719230817/http://www.itrcweb.org/About/Registration-is-open-for-2018-ITRC-Teams)

12/06/2017 - ITRC Releases Newest Guidance on Characterization and Remediation of Fractured Rock (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Releases-Newest-Guidance-on-Characterization-and-Remediation-of-Fractured-Rock)

11/21/2017 - ITRC Releases Newest Guidance on Bioavailability in Contaminated Soils (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Releases-Newest-Guidance-Bioavailabilit-Contaminated-Soils)

11/21/2017 - ITRC 2018 Training Schedule is Available (/web/20190719230817/http://www.itrcweb.org/Documents/TeamResources_OutreachMaterials/ITRC-2018-Classes-071118.pdf)

ITRC's Biannual Newsletter Showcasing Summer & Fall 2017's Key Events (https://web.archive.org/web/20190719230817/http://mailchi.mp/7eec415323cb/itrconnect-fall-2017)

ITRC Releases Three PFAS Fact Sheets (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Releases-Three-PFAS-Fact-Sheets)

Funding Available for Environmental Research and Development (/web/20190719230817/http://www.itrcweb.org/About/Funding-Available-for-Environmental-Research-and-Development-2)

2018 Registration Open for ITRC's Industry Affiliates Program (IAP) (/web/20190719230817/http://www.itrcweb.org/About/ITRC-2018 IAP-registration)

ITRC Team Publishes Guidance on Complex Sites (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Team-Publishes-Guidance-on-Complex-Sites)

ITRC Congratulates all of the 2017 Industry Recognition Award Winners (/web/20190719230817/http://www.itrcweb.org/IAP/Industry_Recognition_2017_Awards)

ITRC Announces Two New Teams for 2018 (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Announces-Two-New-Teams-2018)

PFAS Meeting Brings Together Many Attendees and Notable Speakers (/web/20190719230817/http://www.itrcweb.org/About/2017-PFAS-Meeting)

ITRC Goes to Rhode Island for Summer Meeting (/web/20190719230817/http://www.itrcweb.org/About/Rhode-Island-for-Summer-Meeting)

ITRC Promotes Federal Partnerships (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Promotes-Federal-Partnerships)

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ITRC Attends ECOS STEP Meeting (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Attends-ECOS-STEP-Meeting)

ITRC Promotes Environmental Initiatives in China (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Promotes-Environmental-Initiatives-in-China)

Registration now open for ITRC PVI classroom training October 10-12, 2017 in Ann Arbor, Michigan. (https://web.archive.org/web/20190719230817/https://www.regonline.com/ITRC-PVI-MI)

ITRC Welcomes Summer Interns (/web/20190719230817/http://www.itrcweb.org/About/2017-ITRC-Welcomes-Summer-Interns)

ITRC Spring 2017 Newsletter Now Available! (/web/20190719230817/http://www.itrcweb.org/About/2017-Spring-Newsletter)

ITRC Thrilled to Announce Two New Board Members: Sara Pearson and Naji Akladiss! (/web/20190719230817/http://www.itrcweb.org/About/2017-New-Board-Members-Announced)

New Co-Chairs and Governance (/web/20190719230817/http://www.itrcweb.org/About/New-Co-Chairs-and-Governance)

ITRC Announces Award Winners (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Announces-Award-Winners)

ITRC Annual Meeting – Post Meeting Materials Available (/web/20190719230817/http://www.itrcweb.org/Meetings/2017_Annual)

2017 ITRC Annual Meeting (/web/20190719230817/http://www.itrcweb.org/About/2017-ITRC-Annual-Meeting)

Lifetime Achievement Award Nominees (/web/20190719230817/http://www.itrcweb.org/About/2017-Lifetime-Achievement-Award-Nominees)

ITRC Announces First 2017 Annual Meeting Breakfast Sponsor - Tehama LLC (/web/20190719230817/http://www.itrcweb.org/About/Tehama-LLC)

Welcome New ITRC Staff (/web/20190719230817/http://www.itrcweb.org/About/Welcome-New-ITRC-Staff-2017)

Long-Term Contaminant Management Using Institutional Controls (IC-1) and Long-Term Stewardship Plan Tool (/web/20190719230817/http://www.itrcweb.org/About/IC-1-Plan-Tool)

ITRC Announces First 2017 Annual Meeting Sponsor - Brisea Group, Inc. (/web/20190719230817/http://www.itrcweb.org/About/Brisea-Group)

ITRC Publishes New Guidance: Long-Term Contaminant Management Using Institutional Control (/web/20190719230817/http://www.itrcweb.org/About/Publish-Institutional-Controls)

Registration is open for 2017 ITRC Teams! (/web/20190719230817/http://www.itrcweb.org/About/Registration-open-2017-ITRC-Teams)

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ITRC 2017 Training Schedule is Available (/web/20190719230817/http://www.itrcweb.org/

Documents/TeamResources_OutreachMaterials/ITRC-2017-Classes-081417.pdf)

2017 Industry Affiliates Program (IAP) Registration Now Open (/web/20190719230817/http://www.itrcweb.org/About/2017-IAP-registration)

ITRConnect - Winter 2016 Newsletter (/web/20190719230817/http://www.itrcweb.org/About/ITRConnect-Winter-2016-Newsletter)

ITRC Publishes Guidance Document on Geospatial Analysis for Optimization (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Publishes-GRO-1)

ITRC's GSMC Document Cited as a Resource on ADEC's Solid Waste Program Monitoring Webpage (/web/20190719230817/http://www.itrcweb.org/About/ITRC-GSMC-1-document)

ITRC goes to China! (/web/20190719230817/http://www.itrcweb.org/About/ITRC-goes-to-China)

TPH Risk Evaluation Team Hosts Petroleum Metabolites Session During Fall Team Meetings (/web/20190719230817/http://www.itrcweb.org/About/TPH-Fall-Meeting)

ITRC Co-Hosts the 2016 RCRA Summit and ASTSWMO Annual Meeting (/web/20190719230817/http://www.itrcweb.org/About/2016-RCRA-Summit-and-ASTSWMO-Annual-Meeting)

ITRC Team Leaders Present at California Professional Environmental Marketing Association Meeting (/web/20190719230817/http://www.itrcweb.org/About/California-Professional-Environmental-Marketing-Association-Meeting)

ITRC Announces the Appointment of New Board of Advisors - Mr. Douglas Bacon (UT) and Mr. Richard Spiese (VT). (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Announces-Appointment-New-Board-Advisors)

Practical Assessment and Optimization of Redox-Based Groundwater Remediation Technologies (/web/20190719230817/http://www.itrcweb.org/About/SERDP-and-ESTCP-Webinar-Series-83016)

Free Online Course to Grow Your Understanding of Environmental Remediation Technologies (/web/20190719230817/http://www.itrcweb.org/About/Free-Online-Course)

Follow ITRC on Social Media (/web/20190719230817/http://www.itrcweb.org/About/Follow-ITRC-on-Social-Media)

ITRC Guidance Documents Instrumental in Saving Time and Reducing Costs at Complex Superfund Site (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Cuts-Cleanup-Time-and-Saves-Money)

ITRC Announces Board of Advisors Appointments (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Announces-Board-of-Advisors-Appointments)

ITRC 2015 Industry Recognition Awards (/web/20190719230817/http://www.itrcweb.org/IAP/Industry_Recognition_2015_Awards)

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ITRC Publishes Guidance Document on Geophysical Classification for Munitions Response (GCMR) (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Publishes-Guidance-Document-Geophysical-Classification-Munitions-Response)

ECOS Names Patricia Catherwood Reyes ITRC Director (/web/20190719230817/http://www.itrcweb.org/About/ECOS-Names-Patricia-Catherwood-Reyes-ITRC-Director)

ITRC Aids in Fairbanks Cleanup (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Aids-in-Fairbanks-Cleanup)

ITRC Releases Two New LNAPL Success Stories (/web/20190719230817/http://www.itrcweb.org/About/New-LNAPL-Success-Stories-62015)

ITRC's LNAPL Training Part 1: An Improved Understanding of LNAPL Behavior in the Subsurface - State of Science vs. State of Practice is now available in Spanish – (PDF download) (/web/20190719230817/http://www.itrcweb.org/Documents/Training/LNAPL1/RIC-SPANISH-ITRC-LNAPL-Part1-062414ibt.pdf)

ITRC Publishes New Resource on Integrated DNAPL Site Characterization and Tools Selection (ISC-1) (/web/20190719230817/http://www.itrcweb.org/About/DNAPL-Site-Characterization-Web-based-Guidance-Document)

Celebrate the 20th Anniversary with us by Viewing our Anniversary Slide Show! (/web/20190719230817/http://www.itrcweb.org/Meetings/2015_Spring)

ITRC Publishes New Resource to Support Decision Making Using Site-Specific Risk Assessments (/web/20190719230817/http://www.itrcweb.org/About/RISK-3)

ITRC Featured by U.S. Department of Energy (U.S. DOE) for Work on Cost-Saving Technology (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Featured-by-US-DOE-for-Work-on-Cost-Saving-Technology)

ITRC Pushes Innovation Window: Announces New Remediation-Plus/Stormwater BMP Team (/web/20190719230817/http://www.itrcweb.org/About/New-Remediation-Plus-Stormwater-BMP-Team)

ITRC Publishes New Guidance Document on Petroleum Vapor Intrusion (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Publishes-New-Guidance-Document-Petroleum-Vapor-Intrusion)

ITRC Publishes New Guidance Document on Contaminated Sediments Remediation (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Publishes-New-Guidance-Document-on-Contaminated-Sediments-Remediation)

Follow ITRC on LinkedIn (/web/20190719230817/http://www.itrcweb.org/About/Follow-ITRC-on-LinkedIn)

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ITRC Publishes New Web Based Guidance Document on Groundwater Statistics for Monitoring

Compliance (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Publishes-Based-Guidance-Documents-on--Groundwater-Statistics)

ITRC Publishes New Web Based Guidance Document on Biochemical Reactors for Mining Influenced Water (/web/20190719230817/http://www.itrcweb.org/About/ITRC-Publishes-Based-Guidance-Document-on-Biochemical-Reactors)

ITRC Industry Recognition Awards for 2014 (/web/20190719230817/http://www.itrcweb.org/IAP/Industry_Recognition_2014_Awards)

2013 ITRC Awards (/web/20190719230817/http://www.itrcweb.org/About/2013_Awards)

ITRC Industry Recognition Awards for 2013 (/web/20190719230817/http://www.itrcweb.org/IAP/Industry_Recognition_2013_Awards)

ECOS Passes Resolution Urging Federal Funding Support for ITRC (/web/20190719230817/http://www.itrcweb.org/About/The-Environmental-Council-of-the-States-(ECOS)-passes-resolution-urging-federal-funding-support-for-ITRC)

Forbes Online Editorial Recognizes ITRC for Providing Needed Educational Resources on Environmental Molecular Diagnostics (/web/20190719230817/http://www.itrcweb.org/About/ITRC-providing-needed-educational-resources)

ITRC is Referenced in Two Seminal Publications on Cleaning Up Complex, Highly Contaminated Waste Sites (/web/20190719230817/http://www.itrcweb.org/About/ITRC-referenced-in-two-seminal-publications)

ITRC Biofuels Team Members Provide Insight for "Inside EPA Weekly Report" (/web/20190719230817/http://www.itrcweb.org/Documents/news/PA-DEP-As-EPA-Backs-IEPA-04-12.pdf)

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APPENDIX 1005H



ITRC is thrilled to release three new PFAS fact sheets!

(March 20, 2018) The Interstate Technology and Regulatory Council (ITRC) is thrilled to release three new PFAS fact sheets! The fact sheets are:

- Environmental Fate and Transport
- Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods
- Remediation Technologies and Methods

The Wayback Machine - https://web.archive.org/web/20190825090656/https://itrcweb.org/About/thr…

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- Remediation Technologies and Methods

ITRC has developed this series of fact sheets to summarize the latest science and emerging technologies for per- and polyfluoroalkyl substances (PFAS). The fact sheets are tailored to the needs of state regulatory program personnel who are tasked with making informed and timely decisions regarding PFAS-impacted sites. The content is also useful to consultants and parties responsible for the release of these contaminants, as well as community stakeholders.

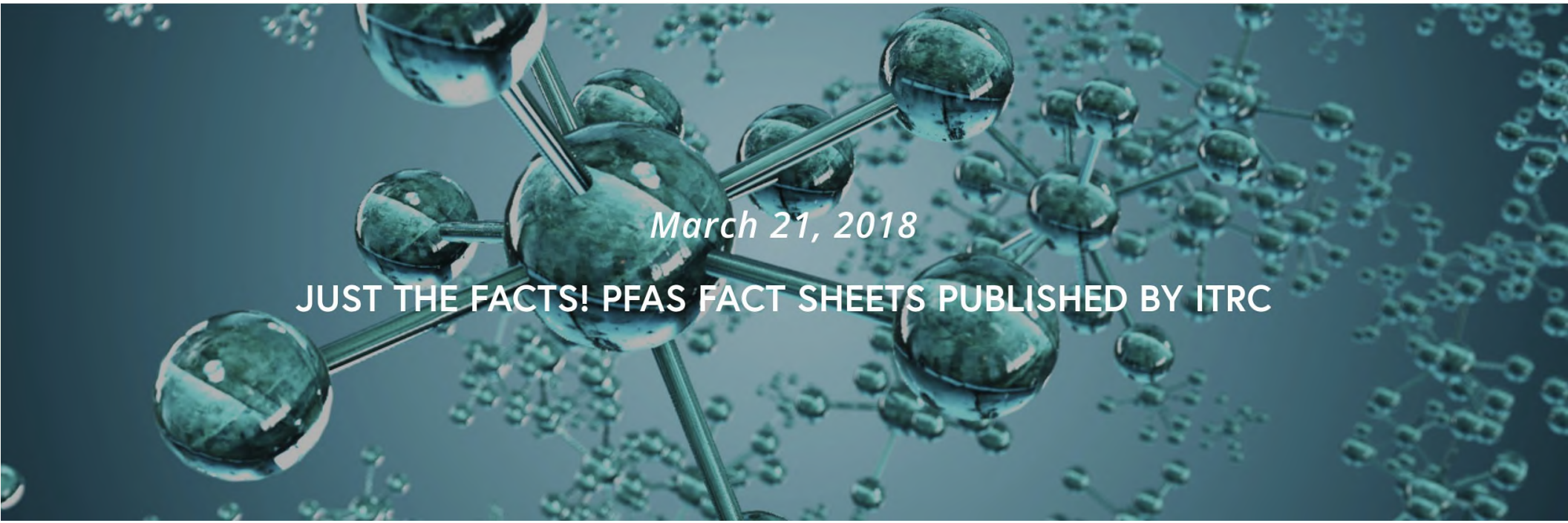
Click HERE (https://web.archive.org/web/20190825090656/https://pfas-1.itrcweb.org/fact-sheets/) to access the fact sheets. The last fact sheet on Aqueous Film Forming Foam is set to be published August 2018.

APPENDIX 1005I

www.meritlabs.com/blog/2018/3/21/just-the-facts-pfas-fact-sheets-published-by-itrc

Click here for information on our PFAS laboratory testing services

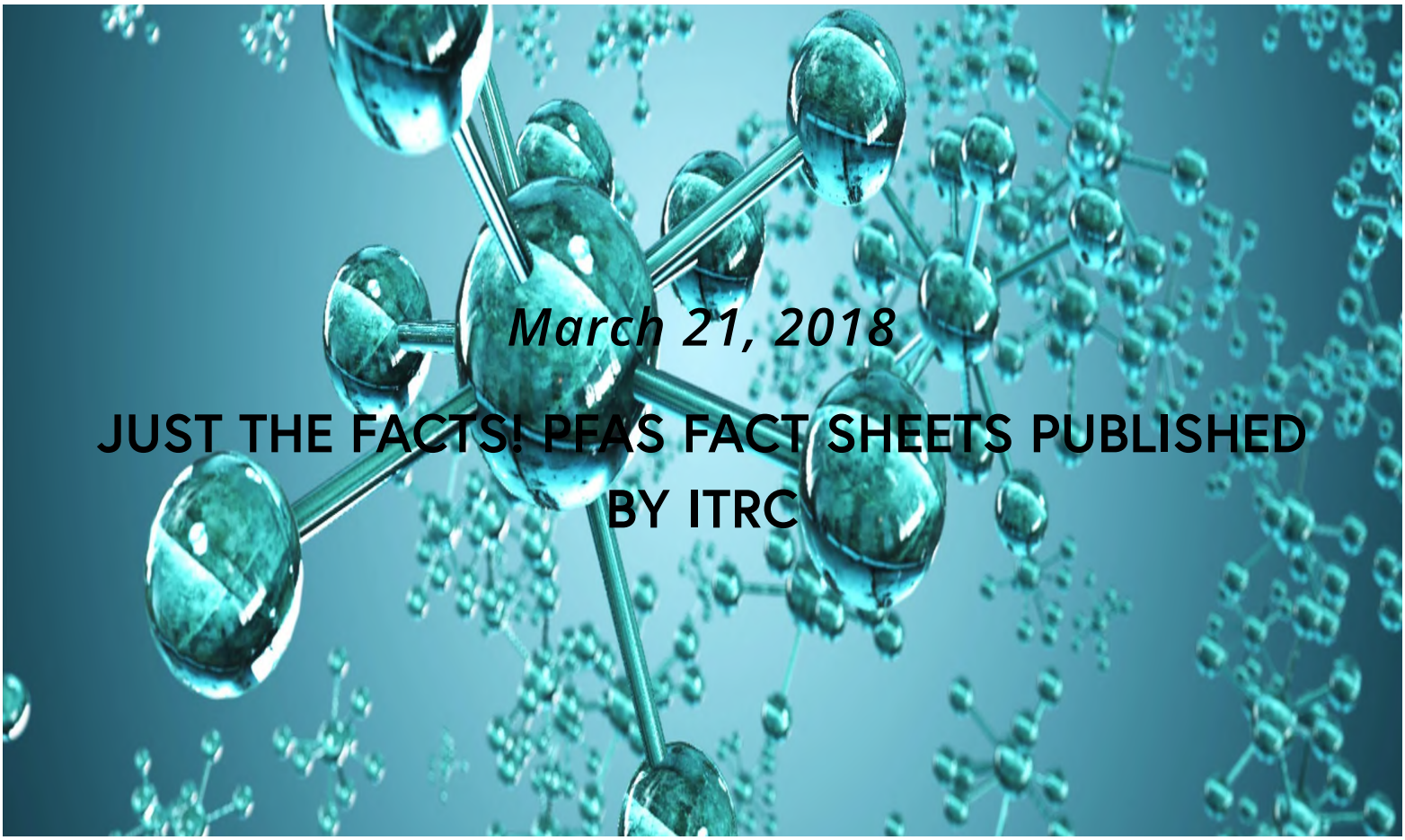
Merit Laboratories, Inc.



March 21, 2018

JUST THE FACTS! PFAS FACT SHEETS PUBLISHED BY ITRC

(/pfas)
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PFAS (/blog/category/PFAS), Merit Labs (/blog/category/Merit+Labs)

Just the Facts! PFAS Fact Sheets Published by ITRC

The Interstate Regulatory Technology Council (ITRC) just published three new fact sheets on Per- and Polyfluoroalkyl Substances (PFAS) along with one updated fact sheet. The four fact sheets address important topics related to PFAS remediation technologies, sample collection, laboratory methods, environmental fate and transport, and more. These four PFAS updates from ITRC are the most current guidance available and will provide environmental professionals with detailed information on PFAS:

- Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods (https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_site_characterization_3_15_18.pdf)
- Remediation Technologies and Methods (https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf)
- Naming Conventions and Physical and Chemical Properties (https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_naming_conventions_3_16_18.pdf)
- Environmental Fate and Transport (https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_fate_and_transport_3_16_18.pdf)

The fact sheet on sample collection and laboratory methods (https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_site_characterization_3_15_18.pdf) will guide environmental professionals through specific PFAS sampling procedures and method selection. The sample collection requirements for PFAS that are covered include groundwater, surface water, pore water, and soil. According to the ITRC, sampling conducted to determine PFAS concentrations in water, soil, sediment, air, biota and other sources is similar to that for other chemical compounds with several additional specific considerations and protocols. If regulatory procedures, methods, or guidelines are inconsistent with the needs of a PFAS sampling program, then the governing agency should be contacted directly to determine an alternate approach or if an exception can be made. Other considerations for PFAS sampling include low laboratory detection limits, state and federal screening levels, and in some cases, cleanup criteria and potential for background concentrations of PFAS in the environment.

The fact sheet on Remediation Technologies and Methods (https://pfas-1.itrcweb.org/wp-content/uploads/2018/03/pfas_fact_sheet_remediation_3_15_18.pdf) explores the treatment techniques for solid and liquid matrices. Solid remediation treatment covered include capping, excavation and disposal, sorption and stabilization, and thermal techniques. The liquid techniques discussed include sorption (e.g., granular activated carbon), precipitation/flocculation/coagulation, redox manipulation, and membrane filtration.

Contact Merit Laboratories (http://www.meritlabs.com/) for additional information on PFAS and learn how we can support your PFAS projects. We take pride in being your technical experts in the environmental laboratory industry and are here to assist.

Tagged: PFAS (/blog/tag/PFAS), PFOA (/blog/tag/PFOA), PFOS (/blog/tag/PFOS), Merit Laboratories (/blog/tag/Merit+Laboratories)

♥ 1 Likes Share

Newer Post

Helping You Clear the Air on TO-15 Project Set-up (/blog/2018/3/23/helping-you-clear-the-air-on-to-15-project-set-up)

Older Post

Michigan's PFAS Industrial Pretreatment Program Initiative on the Fast Track (/blog/2018/3/16/michigans-pfas-industrial-pretreatment-program-initiative-on-the-fast-track)

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EAST LANSING, MICHIGAN

48823, USA

517.332.0167

INFO@MERITLABS.COM

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APPENDIX 1005J

www.facebook.com/groups/353457201741568/permalink/433432190410735/?rdid=QyM 120%

VPN

Johnsie Lang's Post

North Carolina Stop GenX and Nafion in Water · Join

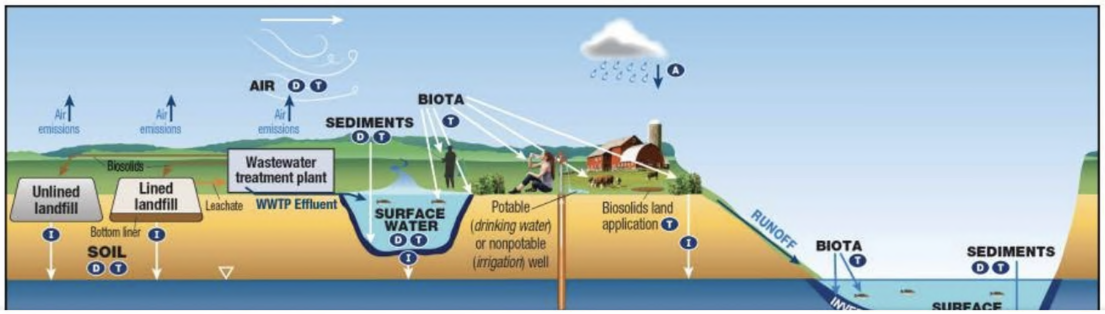
Johnsie Lang · Admin · March 22, 2018 ·

The Interstate Technology and Regulatory Council (ITRC) released three new PFAS fact sheets. The fact sheets are:

Environmental Fate and Transport
Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods
Remediation Technologies and Methods

I helped write the landfill section!

https://pfas-1.itrcweb.org/.../pfas_fact_sheet_fate_and...



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Johnsie Lang's Post

Environmental Fate and Transport
Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods
Remediation Technologies and Methods

I helped write the landfill section!

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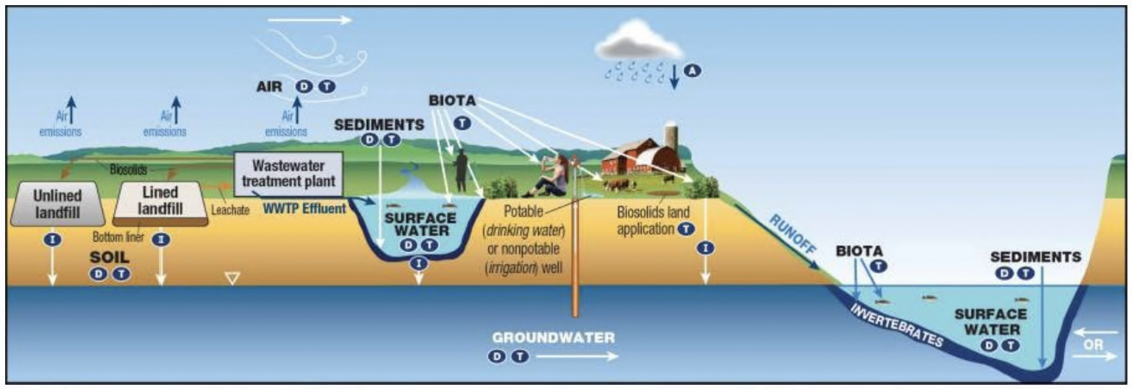


Figure 3. Conceptual site model for landfills and WWTPs.
(Source: Adapted from figure by L. Trozzolo, TRC, used with permission)

APPENDIX 1005K

www.facebook.com/CleanCapeFear/posts/pfbid02iCEmLpNueUjCGQn6D1BC1Bk3kRdu3p 120%

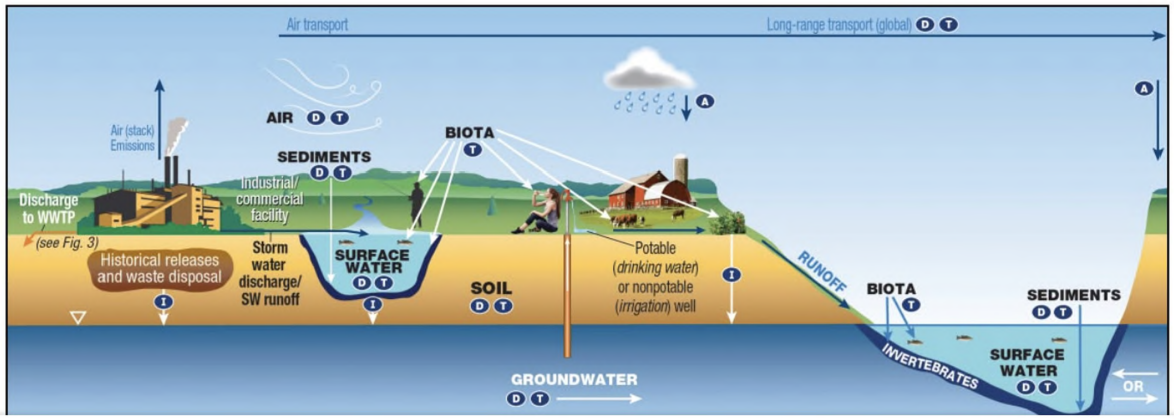
Clean Cape Fear's Post

Clean Cape Fear
March 31, 2018 ·

The ITRC (Interstate Technology Regulatory Council) released three new PFAS fact sheets this month. I really like this figure from the Environmental Fate and Transport Sheet showing PFAS contamination from industrial sites.

https://pfas-1.itrcweb.org/.../pfas_fact_sheet_fate_and...

https://pfas-1.itrcweb.org/fact-sheets/



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Clean Cape Fear's Post

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https://pfas-1.itrcweb.org/fact-sheets/

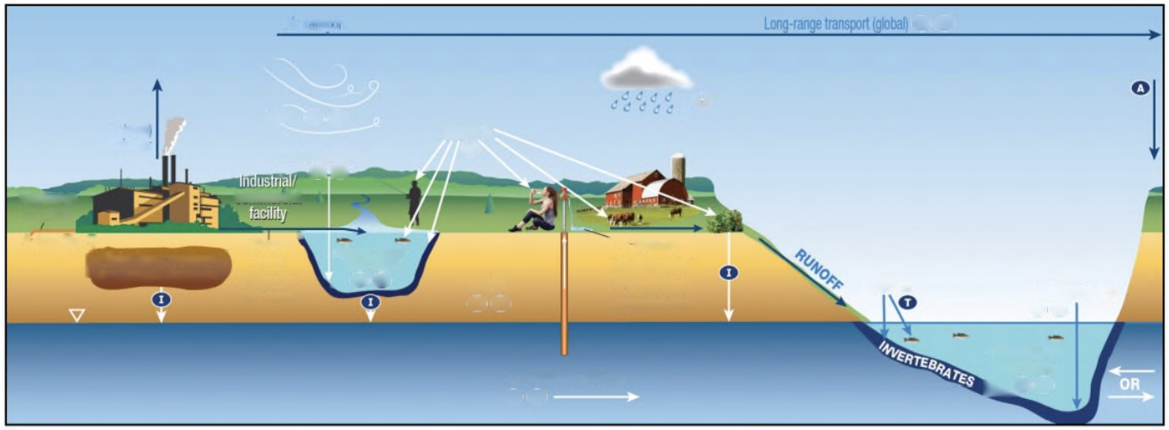


Figure 2. Conceptual site model for industrial sites.

2

EX-1040
Norit Americas, Inc. v. Calgon Carbon Corporation
0367

APPENDIX 1005L

clu-in.org/download/newsltrs/techdirect/td042018.pdf

1 of 7 Automatic Zoom

U.S. ENVIRONMENTAL PROTECTION AGENCY

TechDirect, April 1, 2018

Welcome to TechDirect! Since the March 1 message, TechDirect gained 70 new subscribers for a total of 39,527. If you feel the service is valuable, please share TechDirect with your colleagues. Anyone interested in subscribing may do so on CLU-IN at https://clu-in.org/techdirect . All previous issues of TechDirect are archived there. The TechDirect messages of the past can be searched by keyword or can be viewed as individual issues.

TechDirect's purpose is to identify new technical, policy and guidance resources related to the assessment and remediation of contaminated soil, sediments and groundwater.

Mention of non-EPA documents or presentations does not constitute a U.S. EPA endorsement of their contents, only an acknowledgment that they exist and may be relevant to the TechDirect audience.

U.S. ENVIRONMENTAL PROTECTION AGENCY

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Welcome to TechDirect! Since the March 1 message, TechDirect gained 70 new subscribers for a total of 39,527. If you feel the service is valuable, please share TechDirect with your colleagues. Anyone interested in subscribing may do so on CLU-IN at https://clu-in.org/techdirect . All previous issues of TechDirect are archived there. The TechDirect messages of the past can be searched by keyword or can be viewed as individual issues.

TechDirect's purpose is to identify new technical, policy and guidance resources related to the assessment and remediation of contaminated soil, sediments and groundwater.

Mention of non-EPA documents or presentations does not constitute a U.S. EPA endorsement of their contents, only an acknowledgment that they exist and may be relevant to the TechDirect audience.

> Upcoming Live Internet Seminars

Environmental Sequence Stratigraphy and the new EPA Remediation Geology Paradigm - April 3, 2018, 12:00PM-1:30PM EDT (16:00-17:30 GMT). This webinar will highlight a recently released EPA Technical Issue Paper serving as a practical guide on the application of the geologic principles of sequence stratigraphy and facies models to the characterization of stratigraphic heterogeneity at hazardous waste sites. This technology is applicable to sites underlain by clastic aquifers (intermixed gravels/sands/silts/clays). The scientific principles and methods presented in this document will help to bring clarity to the challenges posed by stratigraphic heterogeneity at complex contaminated sites thereby facilitating site management strategies. These methods can be applied to both new site investigations as well as reviewing existing site data and updating the Conceptual Site Model (CSM). High resolution site characterization (HRSC) is EPA s preferred approach for characterizing sites and developing a detailed CSM. The application of Environmental Sequence Stratigraphy (ESS) allows the practitioner to place high resolution data and other environmental subsurface data in a geologic and hydrogeologic context. This advanced geologic approach evaluates subsurface site information in the context of the site s geologic depositional and erosional history. Application of ESS benefits groundwater remediation projects by improving the ability to: define groundwater flow paths and preferential contaminant migration pathways, map and predict contaminant mass transport and storage zones, identify data gaps to conduct high resolution site characterization, determine appropriate locations for monitoring and remediation wells, determine appropriate well construction design (e.g., screened intervals), improve efficiency of remediating and monitoring contaminated groundwater, and focus your site strategy to save significant time and money. For more information and to register, see https://clu-in.org/live.

ITRC Petroleum Vapor Intrusion: Fundamentals of Screening, Investigation, and Management - April 5, 2018, 1:00PM-3:15PM EDT (17:00-19:15 GMT). Chemical

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contaminants in soil and groundwater can volatilize into soil gas and migrate through unsaturated soils of the vadose zone. Vapor intrusion (VI) occurs when these vapors migrate upward into overlying buildings through cracks and gaps in the building floors, foundations, and utility conduits, and contaminate indoor air. If present at sufficiently high concentrations, these vapors may present a threat to the health and safety of building occupants. Petroleum vapor intrusion (PVI) is a subset of VI and is the process by which volatile petroleum hydrocarbons (PHCs) released as vapors from light nonaqueous phase liquids (LNAPL), petroleum-contaminated soils, or petroleum-contaminated groundwater migrate through the vadose zone and into overlying buildings. The ITRC Technical and Regulatory Guidance Web-Based Document, Petroleum Vapor Intrusion: Fundamentals of Screening, Investigation, and Management (PVI-1, 2014) and this associated Internet-based training provides regulators and practitioners with consensus information based on empirical data and recent research to support PVI decision making under different regulatory frameworks. The PVI assessment strategy described in this guidance document enables confident decision making that protects human health for various types of petroleum sites and multiple PHC compounds. This guidance provides a comprehensive methodology for screening, investigating, and managing potential PVI sites and is intended to promote the efficient use of resources and increase confidence in decision making when evaluating the potential for vapor intrusion at petroleum-contaminated sites. By using the ITRC guidance document, the vapor intrusion pathway can be eliminated from further investigation at many sites where soil or groundwater is contaminated with petroleum hydrocarbons or where LNAPL is present. For more information and to register, see https://www.itrcweb.org or https://clu-in.org/live.

ITRC Long-term Contaminant Management Using Institutional Controls - April 10, 2018, 1:00PM-3:15PM EDT (17:00-19:15 GMT). Institutional controls (ICs) are administrative or legal restrictions that provide protection from exposure to contaminants on a site. When ICs are jeopardized or fail, direct exposure to human health and the environment can occur. While a variety of guidance and research to date has focused on the implementation of ICs, ITRC's Long-term Contaminant Management Using Institutional Controls (IC-1, 2016) guidance and this associated training class focuses on post-implementation IC management, including monitoring, evaluation, stakeholder communications, enforcement, and termination. The ITRC guidance and training will assist those who are responsible for the management and stewardship of ICs. After attending the training, participants will be able to: describe best practices and evolving trends for IC management at individual sites and across state agency programs; use this guidance to improve IC reliability and prevent IC failures, improve existing, or develop new, IC Management programs, identify the pros and cons about differing IC management approaches; use the tools to establish an LTS plan for specific sites; and use the elements in the tools to understand the information that should populate an IC registry or data management system. For more information and to register, see http://www.itrcweb.org or http://clu-in.org/live.

NAVFAC Open Environmental Restoration Resource (OER2) Webinar: Munitions Response Program Update and Lessons Learned - April 25, 2018, 2:00 PM ET (18:00 GMT). This topic will present updates in the form of case studies and lessons learned from completed and ongoing NAVFAC Munitions Response Program (MRP) projects. The session will include an update on the Defense Advanced Geophysical Classification Accreditation Program (DAGCAP) and discussion of using the vertical conceptual site model (VCSM) on munitions projects.
For more information and to register, see
https://cc.readytalk.com/registration/#/?meeting=yw2u9d02kl1v&campaign=j3h70wnxcxsh.

ITRC Remediation Management of Complex Sites - April 26, 2018, 1:00PM-3:15PM EDT (17:00-19:15 GMT). This training course and associated ITRC guidance: Remediation Management of Complex Sites (RMCS-1, 2017), provide a recommended holistic process for management of challenging sites, termed "adaptive site management."

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By participating in this training course we expect you will learn to apply the ITRC guidance document to: identify and integrate technical and nontechnical challenges into a holistic approach to remediation; use the Remediation Potential Assessment to identify whether adaptive site management is warranted due to site complexity; understand and apply adaptive site management principles; develop a long-term performance-based action plan; apply well-demonstrated techniques for effective stakeholder engagement; access additional resources, tools, and case studies most relevant for complex sites; and communicate the value of the guidance to regulators, practitioners, community members, and others. For more information and to register, see https://www.itrcweb.org or https://clu-in.org/live.

Mining Webinar Series: Successful Implementation of Biologically-Based Passive Remediation Systems -Tuesday, May 1, 2018; 2:00-3:30 PM EST (18:00-19:00 GMT). Metal mines at their end of active operations must prepare for the potential long-term treatment of mining-influenced water (MIW). This webinar will highlight how biologically-based passive remediation technologies have been shown to be a cost-effective option for treating MIW, especially mildly- to moderately-impacted waters at low- to moderate-flow rates such as may be found at remote or closed mine sites. Presenters will share an overview of biologically-based passive remediation technologies, their applicability to mining site conditions and illustrate the remedial design and evaluation process with a current treatability study example. In addition, two case study examples of mining sites with operating biologically-based passive treatment systems will be presented: Iron King Mine near Jerome, Arizona, and Garfield Mine near Delta, Colorado. For more information and to register, see https://clu-in.org/live.

ITRC Bioavailability of Contaminants in Soil: Considerations for Human Health Risk Assessment - May 3, 2018, 1:00PM-3:15PM EDT (17:00-19:15 GMT). The basis for this training course is the ITRC guidance: Bioavailability of Contaminants in Soil: Considerations for Human Health Risk Assessment (BCS-1). This guidance describes the general concepts of the bioavailability of contaminants in soil, reviews the state of the science, and discusses how to incorporate bioavailability into the human health risk assessment process. The target audience for this guidance and training course are: project managers interested in decreasing uncertainty in the risk assessment which may lead to reduced remedial action costs, and risk assessors new to bioavailability or those who want additional confidence and training in the current methods and common practices for using bioavailability assessment to more accurately determine human health risk at a contaminated site. As a participant in this training you should learn to: apply the decision process to determine when a site-specific bioavailability assessment may be appropriate, use the ITRC Review Checklist to develop or review a risk assessment that includes soil bioavailability, consider factors that affect arsenic, lead and PAH bioavailability, select appropriate methods to evaluate soil bioavailability, and use tools to develop site-specific soil bioavailability estimates and incorporate them into human health risk assessment. For more information and to register, see https://www.itrcweb.org or https://clu-in.org/live.

Combined Remedies: The New Normal - Attentive Use of the "Right" Tools - May 21, 2018, 1:00PM-2:00PM EDT (17:00-18:00 GMT).
Combining remedies is becoming increasingly prevalent across the spectrum of hazardous waste sites from relatively simple to more complex. This trend is driven by 1) a larger remedial tool box; and 2) by increased understanding that contamination occurs in different phases and concentrations, and in different subsurface compartments. Combining remedies is only part of the story. Flexible, attentive, adaptive deployment is equally important as surprises are not uncommon and conditions change during remedy implementation. This webinar will discuss the underlying principles of attentive deployment of a variety of remedial technologies along with informative case studies - with particular emphasis on NPL sites. For more information and to register, see https://clu-in.org/live.

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Highlight from the CLU-IN Seminar Archives. Each edition of TechDirect highlights a previously recorded internet seminar from our archives that may be of interest to our readers. We welcome your feedback on this addition to TechDirect.

Analytical Tools and Methods: Session I - Field-ready Biosensors to Assess Bioavailability and Toxicity, Sponsored by: NIEHS Superfund Research Program, Archived: Monday, April 17, 2017 (2 hours). This webinar series highlighted innovative analytical tools and methods developed and used by Superfund Research Program (SRP) grantees. The presenters featured the benefits of these new tools and methods compared to conventional methods. They also included information about how the technology has helped to facilitate ongoing SRP research. In the first session, researchers described their tools to assess bioavailability/toxicity for more effective human and/or environmental monitoring. Treatment assessments and water quality monitoring that rely only on measuring the reduction of target contaminant concentrations are often insufficient because they do not consider the complex and broader risks that specific contaminants or mixtures and their transformation products pose to the environment and human health. For more information or to replay Session 1, visit https://clu-in.org/conf/tio/SRPAnalyticalT&M1_041717/.

> New Documents and Web Resources

ITRC Releases Three PFAS Fact Sheets. ITRC has released three new PFAS fact sheets:

- Environmental Fate and Transport
- Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods
- Remediation Technologies and Methods

ITRC has developed this series of fact sheets to summarize the latest science and emerging technologies for per- and polyfluoroalkyl substances (PFAS). The fact sheets are tailored to the needs of state regulatory program personnel who are tasked with making informed and timely decisions regarding PFAS-impacted sites. The content is also useful to consultants and parties responsible for the release of these contaminants, as well as community stakeholders. View or download at https://pfas-1.itrcweb.org/fact-sheets/.

Superfund Research Program Science Digest - Technology Profile, March 2018. As part of an SRP small business project, researchers are developing a technology to expedite removal of per- and polyfluoroalkyl substances (PFAS) from soil and groundwater. Read more at https://www.niehs.nih.gov/research/supported/centers/srp/science_digest/2018/3/technology/technology_profile.cfm

Technology Innovation News Survey Corner. The Technology Innovation News Survey contains market/commercialization information; reports on demonstrations, feasibility studies and research; and other news relevant to the hazardous waste community interested in technology development. Recent issues, complete archives, and subscription information is available at https://clu-in.org/products/tins/. The following resources were included in recent issues:

- Groundwater Source Zone EISB Construction Completion Report, Casper PCE Plumes Orphan Site, Casper, Wyoming
- Estimating the High-Arsenic Domestic-Well Population in the Conterminous United States
- Dioxin Degradation and Metal Biovolatilization at a Former Wood Treating Site
- NMR-Based Sensors for In Situ Monitoring of Changes in Groundwater Chemistry

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- Integration of Filtration and Advanced Oxidation: Development of a Membrane Liquid-Phase Plasma Reactor
- Removal Action Completion Report: Electrical Resistance Heating in Source Area, Former Mercury Cleaners Site Area, 1419 16th Street, Sacramento, California
- Post-Remediation Evaluation of EVO Treatment: How Can We Improve Performance?
- Pilot Studies Report: Former Schilling Air Force Base, Salina, Kansas
- Pilot Study Implementation Report: Lower Columbia Slough-Pacific Meats Cleanup Site, Portland, Oregon
- Rapid Assessment of Remedial Effectiveness and Rebound in Fractured Bedrock
- Ecosystem Services at Superfund Sites: Reuse and the Benefit to Community
- Validation of Passive Sampling Devices for Monitoring of Munitions Constituents in Underwater Environments: Technology User's Manual

EUGRIS Corner. New Documents on EUGRIS, the platform for European contaminated soil and water information. More than 16 resources, events, projects and news items were added to EUGRIS in March 2018. These can be viewed at http://www.eugris.info/whatsnew.asp . Then select the appropriate month and year for the updates in which you are interested. The following resource was posted on EUGRIS:

Removal Action Completion Report Electrical Resistance Heating in Source Area Former Mercury Cleaners Site Area (2018). Mercury Cleaners operated as a dry cleaner from 1947 to 2014. The facility used a variety of dry cleaning solvents, and the site requires soil and groundwater treatment to remediate the impacts of petroleum-based Stoddard Solvent and of PCE and its daughter products. Cleanup began in 2015 with a soil vapor extraction (SVE) system pilot test, which is currently operating. To address soil contaminated with PCE above residential screening levels in an area ~40 ft by 40 ft in plan, a source area removal action was implemented in November 2016 using in situ electrical resistance heating (ERH) thermal desorption. Comprising 15 electrode/vapor recovery wells located within the source area, the ERH system was designed to volatize VOCs in the subsurface for SVE system removal and treatment. The goal of the removal action was to reduce contaminant mass by > 99% and lower concentrations of TPH, PCE, and other VOCs below the soil and groundwater cleanup goals. ERH system operation ended on July 7, 2017. Although source area cleanup goals were not met completely, significant PCE mass was removed from the source area. View or download at http://geotracker.waterboards.ca.gov/esi/uploads/geo_report/5028631806/T10000003411.PDF.

> Conferences and Symposia

EPA to present at 11th Symposium on Design and Construction Issues at Hazardous Waste Sites, Philadelphia, PA, April 18-20, 2018. EPA staff will present at the Symposium on a range of topics including Approaches to Managing Large Superfund Sites, Site Investigation and Characterization: Applications in Design and Remedial Action and Employing Adaptive Techniques to Expedite Site Completion. For more information, see https://www.same.org/Get-Connected/Find-a-Post/Philadelphia/Meetings-Events/ModuleID/1671/ItemID/1730/mctl/EventDetails.

Groundwater High-Resolution Site Characterization (HRSC), Tallahassee, FL, May 1-2, 2018. This training course focuses on groundwater characterization and discusses (1) the impacts of subsurface heterogeneity on the investigation and cleanup of groundwater and related media, (2) the need for scale-appropriate measurements and adequate data density, and (3) the tools and strategies that are available to overcome the impacts of subsurface heterogeneity. After taking this course, participants will be armed

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with information that will allow them to improve their subsurface investigation approaches and develop more realistic and comprehensive conceptual site models (CSM). CSMs developed based on HRSC strategies and tools will decrease site uncertainty, improve the remedy selection process for groundwater remedies, and better enable the evaluation, design, and implementation of targeted in situ and ex situ groundwater remedies. The Groundwater HRSC course is an advanced 2-day course. The recommended audience includes EPA, federal, state, tribal and private industry technical project managers, practitioners and other stakeholders involved in groundwater investigation and remediation. For more information and to register, see https://trainex.org/hrsc.

EPA to Convene National Leadership Summit to Take Action on PFAS, Washington, D.C., May 22-23, 2018. During the summit, participants will work together to share information on ongoing efforts to characterize risks from PFAS and develop monitoring and treatment/cleanup techniques; identify specific near-term actions, beyond those already underway, that are needed to address challenges currently facing states and local communities and develop risk communication strategies that will help communities to address public concerns with PFAS. For more information, see https://www.epa.gov/pfas/pfas-national-leadership-summit-and-engagement.

Incremental Sampling, Dallas, TX, July 26-27, 2018. This training course focuses on the theory and application of ITRC's Incremental Sampling Methodology (ISM), composite sampling designs, and hybrids of the two. IS hybrid designs are useful to address multiple project goals simultaneously. Since "representativeness" is a key aspect of data quality and ISM/IS data are demonstrably more representative than most discrete data, it will be argued that IS data are indeed "better" than non-IS data. The course will answer questions such as: what is the difference between ITRC's ISM and EPA's Incremental Sampling (IS) strategies? Is there written EPA guidance? What features should an ISM or IS design have? Can IS give project risk assessors the data they want, while simultaneously meeting the RPM's own data needs for characterization or remedial design? How are background concentrations determined and comparisons to background handled using IS? Do we know whether IS "worked" for the project? For more information and to register, see https://trainex.org/offeringslist.cfm?courseid=1621.

Best Practices for Site Characterization Throughout the Remediation Process, Dallas, TX, July 23-26, 2018. This training course is based on best management practices (BMP) implemented by the U.S. EPA, partnership organizations, federal and state partners, and consultants. Participants will learn how to streamline projects in a legal, technically sound, and cost-effective manner. By taking the course, participants achieve the following objectives: integrate best practices into traditional project activities, effectively collect and communicate critical project information, design dynamic work strategies, recognize and overcome the challenges presented while implementing a dynamic work strategy, and use BMPs to support all phases of the environmental cleanup life cycle. For more information and to register, see https://trainex.org/offeringslist.cfm?courseid=1515.

NOTE: For TechDirect, we prefer to concentrate mainly on new documents and the Internet live events. However, we do support an area on CLU-IN where announcement of conferences and courses can be regularly posted. We invite sponsors to input information on their events at https://clu-in.org/courses . Likewise, readers may visit this area for news of upcoming events that might be of interest. It allows users to search events by location, topic, time period, etc.

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APPENDIX 1005M

tgpc.texas.gov/gwissues/minutes/TGPC_GWIminutes21Mar2018.pdf
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Automatic Zoom

GROUNDWATER ISSUES SUBCOMMITTEE
OF THE
TEXAS GROUNDWATER PROTECTION COMMITTEE
RECORD OF MEETING
Third Quarter Meeting, Fiscal Year 2018

Meeting Date: March 21, 2018
Meeting No.: 13

Place: TCEQ Campus, Bldg F
Room: 2210

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MEETING ATTENDANCE

Subcommittee Members	Affiliation
Kathy McCormack (Co-Chair)	Texas Commission on Environmental Quality (TCEQ)
Alan Cherepon	TCEQ
Peter Pope (Co-Chair)	Railroad Commission of Texas (RRC)
Janie Hopkins	Texas Water Development Board (TWDB)
Deba Dutta	Texas Department of State Health Service (DSHS)
David T. Villarreal	Texas Department of Agriculture (TDA)
T.J. Helton	Texas State Soil and Water Conservation Board (TSSWCB)
Sarah Schlessinger	Texas Alliance of Groundwater Districts (TAGD)
John Tracy	Texas A&M AgriLife Research (AgriLife Research)
Ken Rainwater	Texas Tech University (TTU)
Cindy Loeffler	Texas Parks and Wildlife Department (TPWD)

Agency Staff	Affiliation	Program
Abiy Berehe	TCEQ	Water Availability Division
Leon Byrd	TCEQ	Water Availability Division
Beth Hood	TAGD	
Cindy Hooper	TCEQ	Water Availability Division
Peggy Hunka	TCEQ	Water Availability Division
Michael Parr	TCEQ	Legal-Environmental Law Division

Interested Parties
None

MEETING HANDOUTS

1. Texas Groundwater Protection Committee (TGPC) Groundwater Issues (GWI) Subcommittee Meeting Agenda, March 21, 2018
2. TGPC GWI Subcommittee Meeting Record, December 13, 2017
3. TGPC Agricultural Chemicals (AgChem) Task Force (TF) Status Report, March 21, 2018
4. Proposed FY 18 Pesticide Monitoring Plan
5. TGPC Publications Status Report, March 21, 2018
6. TGPC *Texas Groundwater Protection Strategy* ("*Strategy*") Update Task Force (TF) Status Report, March 21, 2018
7. National Ground Water Association (NGWA) *Groundwater and PFAS: State of Knowledge and Practice*, 2017 (pages 1 – 3, only), http://www.ngwa.org/Media-Center/news/Pages/Groundwater-and-PFAS-State-of-Knowledge.aspx
8. Interstate Technology and Regulatory Council (ITRC) PFAS Fact Sheets webpage (https://pfas-1.itrcweb.org/fact-sheets/)
9. Proposed *Texas Groundwater Protection Strategy* update
10. email template to TGPC member management regarding the proposed *Strategy* update
11. TCEQ management concurrence letter template regarding the proposed *Strategy* update

The following item was also on display on the handout table:

1. TGPC Public Information Act Policy

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MEETING RECORD OF MARCH 21, 2018

1. CALL TO ORDER AND INTRODUCTIONS

Peter Pope (RRC), Co-Chair of the TGPC GWI Subcommittee, called the meeting to order at approximately 9:39 AM CDT. GWI Subcommittee members were introduced. The representatives from the Bureau of Economic Geology of The University of Texas at Austin (UTBEG), Texas Department of Licensing and Regulation (TDLR), United States Geological Survey (USGS), and Texas A&M AgriLife Extension Service (AgriLife Extension) were not present at the meeting. The representative from TPWD was not present at the beginning of the meeting. Janie Hopkins (TWDB) noted an error in the previous meeting minutes concerning the major aquifers that they would be sampling in 2018, and these minutes were updated after the meeting.

Cindy Loeffler (TPWD) arrived at approximately 9:40 AM CDT and was present for the remainder of the meeting.

2. STATUS REPORTS

Agricultural Chemicals Task Force

Alan Cherepon (TCEQ) reported on the Agricultural Chemicals Task Force:

- There had been one Agricultural Chemicals Task Force meeting (via email, phone, or in person) since the last GWI Subcommittee meeting.
- The Middle-Of-Year report was being reviewed and would be emailed and sent to U.S. Environmental Protection Agency (EPA) Region 6 in Dallas as soon as approved for delivery (the target date was April 1, 2018).
- The proposed FY2018 pesticide monitoring plan required modification and included the following tasks:
    - Continue with cooperative pesticide monitoring with TWDB, primarily for atrazine and possibly 2,4-D and glyphosate; this year, TWDB would be sampling in the Carrizo-Wilcox Aquifer and several minor aquifers, and a total of 150 samples were anticipated; and,
    - Shallow water wells would be sampled for atrazine and related pesticides used on corn and sorghum cropland areas from Williamson to Ellis Counties, primarily east of I-35. This area had not been previously monitored by TCEQ and was close enough to TCEQ's main office to limit travel expenses. An urban metropolitan area with sufficient water wells to sample for some previously un-monitored urban pesticides would not actually be monitored, after all, because the laboratory could not accommodate the analyses, and a cluster of Resource Conservation and Recovery Act (RCRA) and Superfund pesticide-related sites could not be identified in order to collect samples from nearby private water wells.
- TCEQ monitoring was not scheduled to begin until late spring/early summer 2018, while Cooperative monitoring was scheduled to begin sometime in April 2018.
- The 2017 Cooperative and on-going monitoring results had been entered into the Interagency Pesticide Database (IPD) and had been sent to the U.S. EPA and Region 6 U.S. EPA.

- The 12/14/16 to 9/20/17 USGS data had been quality checked and entered into the IPD; this included 64 wells (27 new), 69 new samples, and 15,377 analytes.

Alan Cherepon (TCEQ) concluded the discussion by referencing the Proposed FY 18 Pesticide Monitoring Plan in the handout package.

TGPC Publications

Abiy Berehe (TCEQ) reported on the following TGPC publications:

- Groundwater Assessment for the *2018 Texas Integrated Report for Clean Water Act Sections 305(b) and 303(d)* ("*Texas Integrated Report*"):
  - The 2018 report preparation was underway and anticipated to be completed by April 1, 2018.
- *Joint Groundwater Monitoring and Contamination Report – 2017* ("*Joint Report*", TCEQ publication SFR-056):
  - Staff were working on the 2017 *Joint Report* and it would be completed by June 2018.
- *Activities and Recommendations of the Texas Groundwater Protection Committee: Report to the 86th Legislature* ("*Legislative Report*", TCEQ publication SFR-047)
  - The Legislative Report Subcommittee was activated to work on the referenced report on January 10, 2018, there had been one Legislative Report Subcommittee meeting since the last GWI Subcommittee meeting, and the next Legislative Report Subcommittee meeting was scheduled for April 18, 2018;
  - The Legislative Report Subcommittee had started working on the draft report and its recommendations to the 86th Legislature, and the draft report would be presented at the July 2018 TGPC meeting; with TGPC approval, the final draft report would then be routed for upper management review and concurrence; and,
  - It was planned that the final report would be distributed to the Legislature in December 2018 before the session begins in January 2019.
- *Texas Groundwater Protection Strategy* ("*Strategy*", TCEQ publication AS-188):
  - There had been two *Strategy* Update Task Force meetings since the last GWI Subcommittee meeting; the final draft was to be reviewed, and possibly approved, at that GWI Subcommittee meeting.

Texas Groundwater Protection Strategy Update Task Force

Kathy McCormack (TCEQ) reported on the *Strategy* Update Task Force:

- The *Strategy* Update Task Force had met on December 21, 2017 and discussed the revised draft;
- The *Strategy* Update Task Force had met on February 21, 2018 and reviewed the proof from TCEQ Agency Communications; and,
- As requested by the *Strategy* Update Task Force, TCEQ Agency Communications made three minor changes to the proof resulting in the final draft to be reviewed at that GWI Subcommittee meeting.

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3. SET FUTURE MEETING DATE

The next TGPC GWI Subcommittee meeting was scheduled for Wednesday, June 13, 2018, at 9:30 AM CDT, in TCEQ's Building F, Conference Room 2210.

4. DISCUSS CURRENT GROUNDWATER ISSUES

Water Supply Enhancement Program

There was no discussion of this topic at the meeting.

Hydraulic Fracturing

There was no discussion of this topic at the meeting.

Emerging Contaminants

Kathy McCormack (TCEQ) referred members to the first three pages of the recent NGWA *Groundwater and PFAS: State of Knowledge and Practice* report that was in the handout package.

Deba Dutta (DSHS) referred members to the ITRC PFAS Fact Sheets webpage that was in the handout package. Three new fact sheets had recently been released:

- Environmental Fate and Transport;
- Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods; and,
- Remediation Technologies and Methods.

Six ITRC PFAS fact sheets were now available. The last ITRS PFAS fact sheet on Aqueous Film Forming Foam was scheduled to be published in August 2018.

Kathy McCormack (TCEQ) noted a recent news article about 19 private drinking water wells and one public water well at the former Reese Air Force Base in Lubbock County that were contaminated with perflourinated compounds. Although the Air Force was currently providing bottled water to affected well owners, Ken Rainwater (TTU) indicated that the City of Lubbock would be providing a clean drinking water line to affected homes.

David Villarreal (TDA) reported that there were several new pesticides being investigated under experimental use permits for feral hogs and invasive species in surface water applications.

Alan Cherepon (TCEQ) asked about chlorination disinfection by-products; perhaps this issue would be added to the next GWI Subcommittee *Activity Plan*.

Saline Groundwater Trends and Impacts in Texas

There was no discussion of this topic at the meeting.

Agricultural Chemicals

There was no additional discussion of this topic at the meeting.

Border Groundwater

John Tracy (AgriLife Research) reported that:

- Federal funding for the Transboundary Aquifer Assessment Program (https://wrrc.arizona.edu/TAAP) should be approved;
- The *International Law of Transboundary Groundwater Resources* book (https://www.barnesandnoble.com/w/the-international-law-of-transboundary-groundwater-resources-gabriel-eckstein/1122551411?ean=9781138842991&st=PLA&sid=BNB_DRS_New+Core+Shopping+Books_00000000&2sid=Google_&sourceId=PLGoP164972&gclid=EAIaIQobChMIxei1qLaA2gIVFXdeCh1crQToEAQYASABEgJHu_D_BwE) by Gabriel Eckstein had recently been published;
- A book review by Rosario Sanchez of the *International Law of Transboundary Groundwater Resources* had recently been published (https://journals.tdl.org/twj/index.php/twj/article/view/7076/pdf);
- They had received 60 abstracts for their transboundary conference on July 9 – 11, 2018 in Fort Worth, but only two of them were related to groundwater quality; and,
- They hoped to publish a resource paper with Lessons Learned from the conference (covering, for example, intrastate to international issues, science, ecological services, management and governance structures, etc.).

Aquifer Storage and Recovery

Kathy McCormack (TCEQ) reported that UTBEG had indicated that there were still many questions to be answered before they would be ready to start working on their Aquifer Storage and Recovery (ASR) white paper for the GWI Subcommittee. They also suggested that TWDB should be involved with the white paper because the TWDB Innovative Water group was active in ASR projects relating to the State Water Plan.

Cindy Loeffler (TPWD) noted that the Edwards Aquifer Habitat Conservation Plan (HCP) had some ASR aspects (http://www.eahcp.org/index.php/flow_protection/asr) and that there was an "ASR for Texas" conference on April 12, 2018 in Austin.

John Tracy (AgriLife Research) remarked that the U.S. Department of the Interior (DOI) Bureau of Reclamation had a High Plains States Groundwater Recharge Demonstration Program Project (https://www.usbr.gov/projects/index.php?id=520).

Sarah Schlessinger (TAGD) added that the Barton Springs / Edwards Aquifer Conservation District (BSEACD) had a managed recharge program (e.g., http://bseacd.org/2017/09/status-report-for-aquifer-storage-and-recovery-pilot-project-ruby-ranch-water-supply-corporation-hays-county-central-texas/).

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Abandoned Water Wells

Sarah Schlessinger (TAGD) reported that, while a similar bill was vetoed at the last legislative session, the Texas House Committee on Natural Resources now had an Interim Charge to "Study the hazards presented by abandoned and deteriorated groundwater wells, and make recommendations to address the contamination and other concerns these wells may represent." Sarah Schlessinger noted that the Bandera River Authority and Conservation District already had the authority to plug an abandoned or deteriorated water wells without a water well driller license.

Failing Septic Systems

There was no discussion of this topic at the meeting.

Others

Cindy Loeffler (TPWD) reported that the Texas Hornshell (*Popenaias popeii*) freshwater mussel was recently listed by the U.S. DOI Fish and Wildlife Service (FWS) as endangered, and that there would be a CPA Freshwater Mussel Working Group meeting on April 3, 2018 in Austin. Examples of the implications of an Endangered Species Act (ESA) listing include:

- The Edwards Aquifer HCP was created in response to lawsuits which ultimately led to the Edwards Aquifer Authority working with U.S. FWS on an an incidental take permit so that they could continue their activities;
- The BSEACD wanted to proactively pursue an incidental take permit in their HCP which is now before U.S. FWS for approval; and,
- Alternatively, a Candidate Conservation Agreement between potentially affected parties and U.S. FWS can provide some assurances about the impacts of ESA listing.

Kathy McCormack (TCEQ) added that a "Multiple freshwater mussel species of the Brazos River, Colorado River, and Guadalupe River basins" final report had recently been issued by the Texas Comptroller of Public Accounts (CPA).

The GWI Subcommittee took a meeting break at approximately 10:39 – 10:45 AM CDT. A quorum of designated TGPC representatives/alternates was present at the meeting and they were reminded not to discuss TGPC business during the break.

5. BUSINESS DISCUSSION AND POSSIBLE ACTION

Draft *Texas Groundwater Protection Strategy* Update

Peter Pope (RRC) presented the final draft of the proposed *Strategy* update. David Villarreal (TDA) proposed, Sarah Schlessinger (TAGD) seconded, and all remaining members approved the proposed *Strategy* update. The proposed *Strategy* update would be presented to the TGPC for approval at their April 18, 2018 meeting.

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Kathy McCormack (TCEQ) reviewed two additional items in the handout package regarding the proposed *Strategy* update: an email template to TGPC member management and a TCEQ management concurrence letter template. Both of these files would be sent to TGPC members after the meeting; they could be edited as needed for their management and the required communication procedures at their agency or organization. These Word files had been requested by those that needed to get started early in order to meet the anticipated June 1st deadline for the management concurrence letter.

Deactivate *Texas Groundwater Protection Strategy* Update Task Force

Peter Pope (RRC) proposed, David Villarreal (TDA) seconded, and all remaining members approved the deactivation of the *Strategy* Update Task Force.

Draft *How is Groundwater Quality Affected by Failing On-site Sewage Facilities (OSSFs) ?* White Paper Outline

Kathy McCormack (TCEQ) reported that Anish Jantrania (AgriLife Extension) had indicated that he would provide a draft *How is Groundwater Quality Affected by Failing On-site Sewage Facilities (OSSFs) ?* white paper at the next meeting.

Initial Discussion of White Papers Under Development

In order to help with the development of their white paper, Deba Dutta (DSHS) reported that they may give a presentation related to the health impacts of Per- and Polyfluoroalkyl Substances (PFAS) at a future meeting.

John Tracy (AgriLife Research) reported that they would be working on the Border Groundwater white paper this summer.

6. ANNOUNCEMENTS

Peter Pope (RRC) announced that the Northwest Environmental Training Center (NWETC, https://www.nwetc.org/) would be offering an Emerging Contaminants Workshop (https://www.nwetc.org/course-catalog/chem-410-sept-13-2018) on September 13, 2018 in Austin.

Sarah Schlessinger (TAGD) announced their 2018 Texas Groundwater Summit on August 28 – 30, 2018 in San Antonio.

Janie Hopkins (TWDB) announced that:

- John Dupnik was their new Deputy Executive Administrator for Water Science and Conservation;
- Brooke Paup was their new Board member; and,
- Peter Lake was their new Board Chairman.

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Deba Dutta (DSHS) noted that the week before the meeting had been the annual National Groundwater Awareness Week.

7. PUBLIC COMMENT

There was no public comment at the meeting.

8. ADJOURN

Peter Pope (RRC) adjourned the meeting at approximately 11:26 AM CDT.

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Article

Monthly Variations in Perfluorinated Compound Concentrations in Groundwater

Megan Steele [1], Converse Griffith [1] and Christin Duran [2,*]

[1] UES, Force Health Branch, United States Air Force School of Aerospace Medicine, 711 Human Performance Wing, Dayton, OH 45431, USA; megan.steele.1.ctr@us.af.mil (M.S.); converse.griffith.ctr@us.af.mil (C.G.)
[2] Force Health Branch, United States Air Force School of Aerospace Medicine, 711 Human Performance Wing, Dayton, OH 45433, USA
* Correspondence: christin.duran@us.af.mil; Tel.: +1-937-937-3790

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Abstract: Large-scale manufacturing of poly- and perfluorinated compounds in the second half of the 20th century has led to their ubiquity in the environment, and their unique structure has made them persistent contaminants. A recent drinking water advisory level issued by the United States Environmental Protection Agency lowered the advisory level concentration of perfluorooctanesulfonic acid (PFOS) and perfluorooctanoic acid (PFOA) from 200 nanograms per liter and 400 nanograms per liter, respectively, to 70 nanograms per liter separately or combined. Small temporal variations in PFOS and PFOA concentrations could be the difference between meeting or exceeding the recommended limit. In this study, newly sampled data from a contaminated military site in Alaska and historical data from former Pease Air Force Base were collected. Data were evaluated to determine if monthly variations within PFOS and PFOA existed. No statistically significant temporal trend was observed in the Alaska data, while the results from Pease, although statistically significant, showed the spread of observed contaminant concentrations around the fitted line is broad (as indicated by the low R^2 values), indicating that collection date has little value in predicting contaminant concentrations. Though not currently the subject of a US EPA health advisory, data on perfluorobutanesulfonic acid (PFBS), perfluorohexane sulfonic acid (PFHxS), perfluoroheptanoic acid (PFHpA), and perfluorononanoic acid (PFNA) were collected for each site and their average concentrations evaluated.

Keywords: PFAS; PFOS; PFOA; perfluorooctane sulfonate; perfluorooctanoic acid

1. Introduction

Per- and polyfluoroalkyl substances (PFAS) have a carbon chain backbone bonded to either fluorine or functional groups [1]. This structure makes PFAS both hydrophobic and oleophobic, properties that are essential for making stain- and stick-resistant products, such as fast food wrappers and stain-resistant fabric [2]. The wetting properties that allow these compounds to evenly coat a surface make them ideal additives for aqueous firefighting foam (AFFF), since unbroken coverage of spilled fuel can be achieved through a relatively small amount of PFAS [3]. At 105.4 kcal/mol, the carbon-fluorine bond is one of the strongest covalent bonds, making the compounds highly resistant to change by chemical or physical means [4,5]. While this bond property is advantageous in high-temperature or chemically aggressive processes, it makes PFAS persistent organic pollutants once they have reached the environment [6].

While an entire class of PFAS exist, two specific compounds have been singled out for attention: Perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS). These two compounds are ubiquitous, present in 95% of American citizens' serum [7], and found at low levels in the soil of

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Antarctica [8]. Both of these compounds have been observed to bioaccumulate in birds [9], and mammals [10] while PFOS is shown to bioaccumlate in fish though PFOA is not [11]. Just as they are long lasting in the environment, they persist within the serum of exposed humans. A five-year study of former workers from a PFAS production facility found the half-life of PFOA and PFOS in humans to be 3.5 years and 4.8 years, respectively, with an average serum concentration of 799 ng/mL for PFOS and 691 ng/mL for PFOA [12,13]. Human exposure to PFAS has been shown to occur through air, drinking water, food, and indoor dust from treated furniture [14].

PFOS has been internationally recognized as a persistent organic pollutant under the Stockholm Convention since 2009 [15], and though no decision has been reached, both PFOA and PFHxA are under investigation. While not included in the Stockholm Convention, short chain PFAS are of growing concern, since manufacturing focus has shifted to shorter chain compounds, and these compounds tend to be present in water, due to their poor sorbtion to soil [16].

Prior studies have indicated potential health effects from PFAS exposure to include low infant birth weights [17], changing cholesterol [18], reduced immune response for vaccinations [19], and thyroid disruption [20]. The Environmental Protection Agency (EPA) included PFOS and PFOA in the third Unregulated Contaminant Monitoring Rule (UCMR3) to determine the scale of contamination across the United States [21]. The data were collected from 2013–2015 and they served as a baseline of likely exposures that were used to inform potential regulatory decisions. In 2016, based on new research, the EPA issued a drinking water health advisory for levels 70 parts per trillion of PFOS and PFOA, individually and combined [22]. The 2016 drinking water health advisory significantly lowered the 2009 provisional health advisory values of 200 parts per trillion of PFOS and 400 parts per trillion of PFOA [22]. By creating a lower, combined health advisory level, water systems previously below the bounds of the 2009 advisory may no longer meet the more stringent guidance. Traditional removal techniques for water contaminants, such as flocculation, sand filtration, or photodegradation without additional chemicals are ineffective against PFAS [23,24]. To combat high levels of long chain PFAS, water systems install activated carbon filters or reverse osmosis systems, both of which are costly [25].

Across the Department of Defense (DOD), PFAS have been included as part of Class B firefighting foam, a type of AFFF that meets military standards [26]. Class B firefighting foam has been in use since the 1960s as both part of suppression systems within a hangar, as well as for training exercises [27]. Incidental runoff from these areas introduced a plethora of PFAS into the surrounding soil. The predominate PFAS of concern in legacy AFFF is PFOS, though PFOA is often present in lesser amounts as it was a contaminant commonly found in the finished product. Once PFOS has reached the soil, it is strongly sorbed leading it to leach slowly into the water table; however, PFOA is not sorbed as strongly as PFOS and shorter chain PFAS are even less well, leading to a faster transit to the groundwater [28]. PFOS plume movement is slow and often takes decades, as demonstrated at Wurtsmith and Tyndall Air Force Bases (AFBs) [29]. AFFF use is common enough to DOD sites that it has been shown there is a 35% increased chance of PFOS contamination within the watershed if a military base is present [30].

At a sufficiently large installation, the U.S. DOD is the water purveyor within those boundaries. The potential water customers include military members and their families who live in government housing, as well as any civilians whose work require them spend their days on site. While it is known that PFOS and PFOA concentrations in groundwater do not show large variance over time [28,31,32], it is not known if small variations occur from month to month. Considering the prevalence of AFFF at DOD sites, if small temporal variations of PFOS and PFOA concentrations in groundwater occur, due to weather patterns, it could place an extra burden on the water filtration system throughout the year. The aim of this study was to determine if PFOS or PFOA concentrations change monthly. If a trend is present, it will also better inform the water purveyors—so they can pay close attention effluent concentration and monitor their treatment beds for signs of breakthrough, due to higher than usual PFAS concentrations. To this end, two Air Force sites with prior legacy AFFF use are examined for changes in the PFOS and PFOA concentrations within the groundwater from month to month.

2. Materials and Methods

Data were derived from two sources: Historical data from former Pease AFB and recently collected data from a contaminated military base in Alaska.

PFAS data from former Pease AFB were downloaded from the publicly available Portsmouth Water Works site. Due to widespread contamination, four production wells were monitored monthly for 3.5 years while 13 sentry wells were monitored monthly from between 4 months and 3.5 years [33]. Data were generated by consulting firms employed by the Air Force Civil Engineer Center under the oversight of the EPA. Samples were analyzed by an accredited laboratory that used EPA method 537 for the determination of PFAS in water [34]. The minimum detection limit provided by the lab was no higher than 5 nanograms per liter [35].

Monthly samples from a military installation in Alaska were collected from July 2016 through March 2017. Six wells from around the installation were sampled each month. The wells were selected based on historical sample data that indicated contamination of PFOS and PFOA was present. Water that had passed through granulated active charcoal beds and was ready for distribution was also monitored, denoted as FG. During collection, a new set of gloves was donned, all clothes worn to the site were previously laundered several times, and no cosmetic or lotions were worn. In keeping with the guidance provided in EPA document 600/R-08/092, samples were collected in one-time use polypropylene bottles, immediately put on ice and shipped overnight to a third party lab. Care was taken to avoid contact with Teflon or other known sources of PFAS.

Analysis of samples was carried out through Pace Analytical, a lab certified by the EPA to analyze compounds found in the UCMR3 list. Samples were evaluated using the EPA 537 method using liquid chromatography/tandem mass spectrometry. Each batch of samples were submitted with a field blank to ensure contamination did not occur from improper sampling technique or ambient PFAS levels. The adjusted method detection limit for PFOS was 8 ng/L and for PFOA was 0.4 ng/L. Each batch of samples were reported with quality control data, which included a method blank, a spiked laboratory control sample, and laboratory control sample with duplicate.

In order to determine if there was a relationship between sampling date and PFOS or PFOA concentration, a polynomial (cubic) regression was used. Polynomial regression is an extension of simple linear regression, which includes, in addition to the linear term, squared and cubed terms of the predictor variable [36]. This extension of simple linear regression allows for potential cyclical changes to also be observed. The regression line minimizes the sum of the squared differences between the observations and the predicted value. The null hypothesis is that there is no relationship between date and concentration. To evaluate the hypothesis with the correlated data, the Newey-West variance estimator was used [37]. This estimator was used, because samples were taken from the same wells, which are on the same aquifer, so they cannot be treated as truly independent. The behavior of one well can impact the other, depending on distance and how much one well draws. Therefore, the usual variance estimator, which assumes independence among observations, is inappropriate. The variance was expressed as an R^2 value, which is a number that summarizes how well the independent variable (collection date) accounts for variation in the concentrations. The R^2 value ranges from zero to one, where one describes a perfect relationship.

3. Results

Data for PFOS and PFOA concentrations for each site are presented by analyte. A separate cubic regression plot is shown for Pease and for the Alaskan installation. Each month, the concentration of PFOS or PFOA for every well sampled is plotted vertically. Individual wells are denoted by a uniquely colored circle. Each plot contains a red line representing the cubic regression line generated from all the data collected at the site. Wells could not be considered independent, since they drew from a single aquifer. A dashed green line is shown to indicate the current public health advisory stipulated by the U.S. EPA.

3.1. PFOA Data

PFOA concentrations in each well were plotted by month (Figure 1). Tabular data are available in Supplementary Materials (Tables S1 and S2). Overall, the values at Pease AFB were an order of magnitude lower than those found at the Alaskan site. When comparing variation in all of the wells collectively over time, there was no association between collection date and PFOA at the Alaskan installation ($p = 0.83$). At Pease, the relationship was statistically significant ($p < 0.001$). As can be seen in the plot below, there is considerable spread in the observed concentrations around the fitted line. It is expected that if time played a large role in the concentration changes, the slope of the regression line would be more pronounced and the date would explain more of the variation; however, in the Pease data, the date explained only 10% of the variation in PFOA concentrations ($R^2 = 0.10$); thus, 90% of the variation could not be accounted for by collection date.

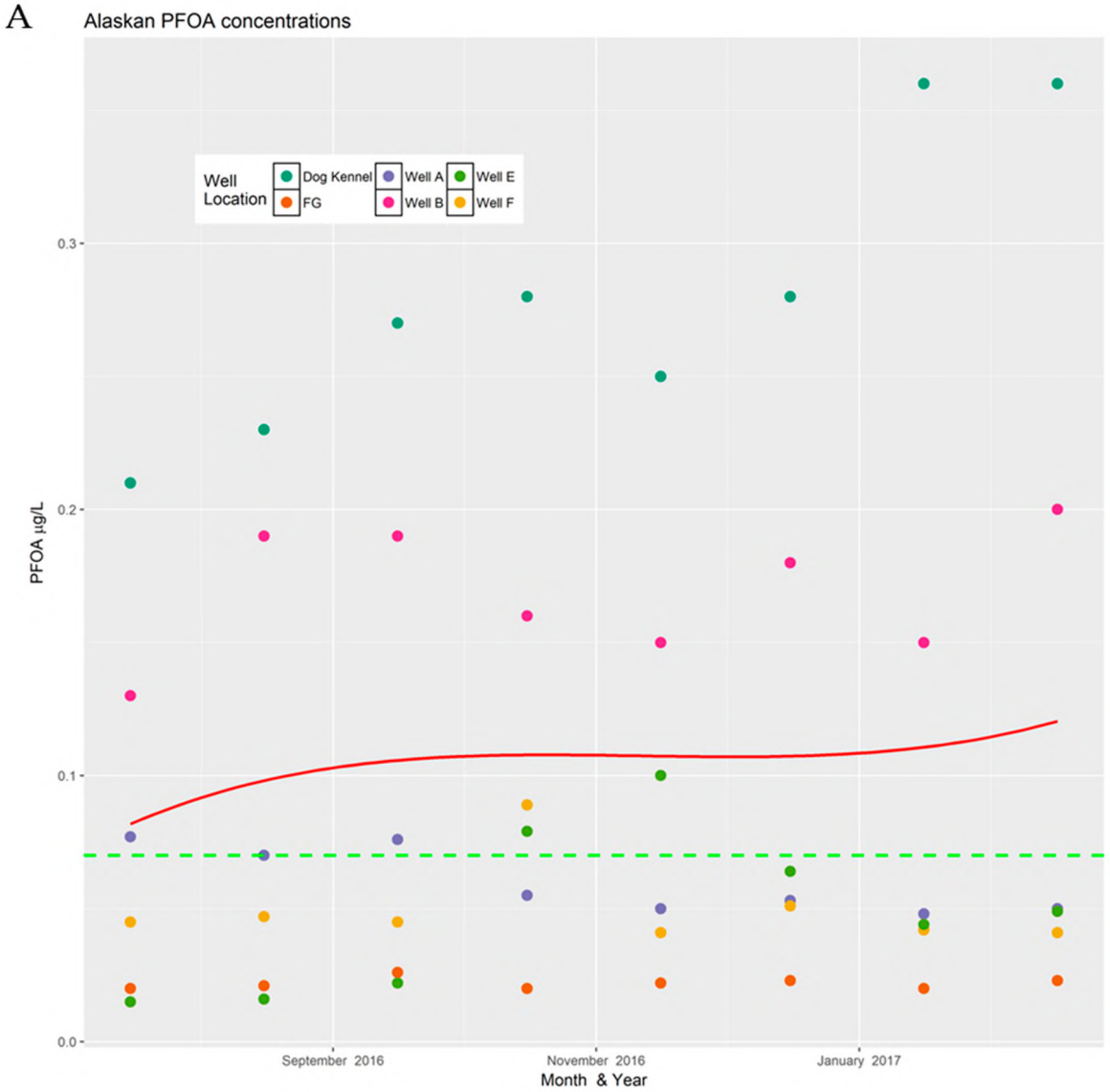


Figure 1. *Cont.*

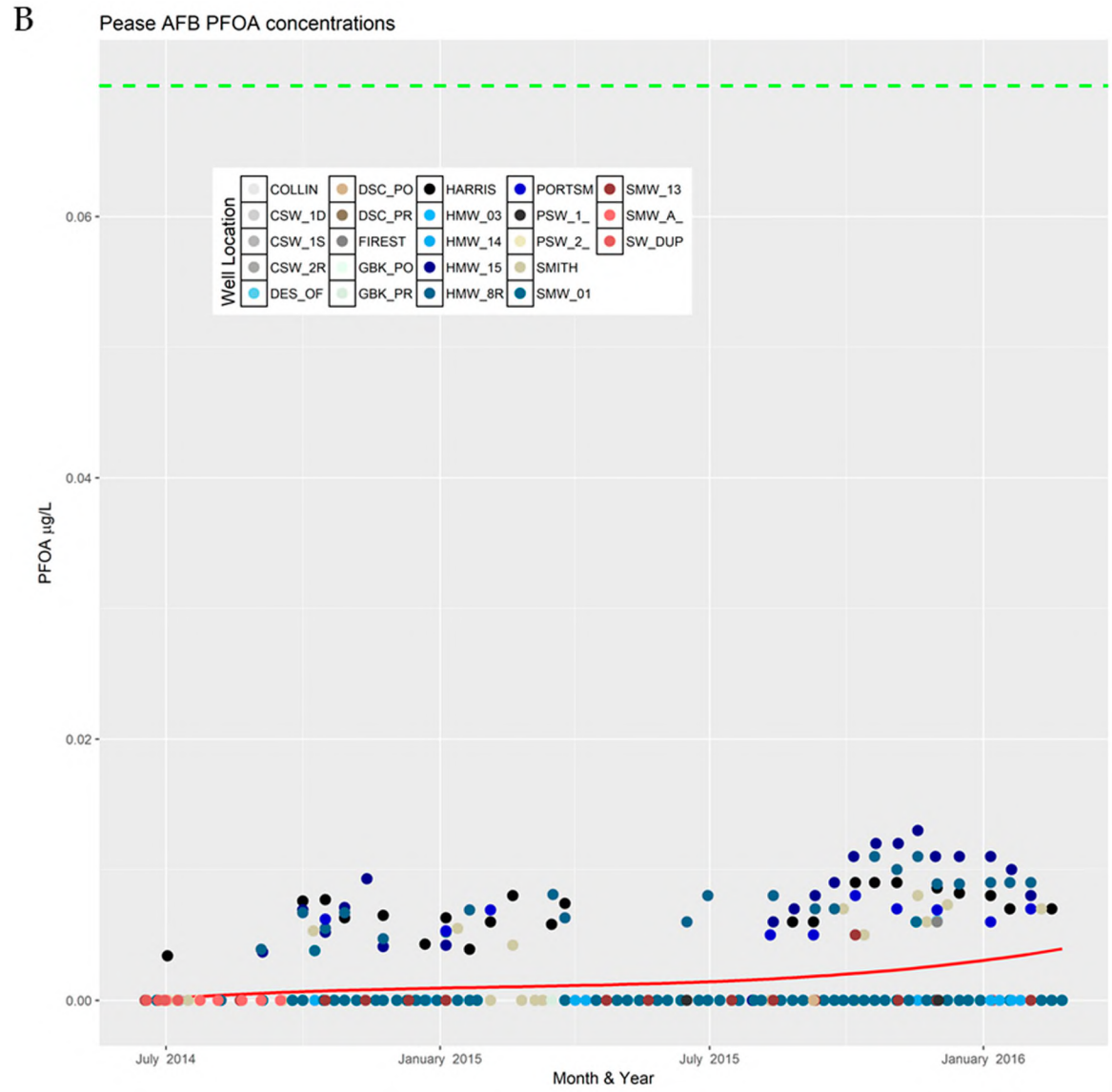


Figure 1. The above chart shows the variance in perfluorooctanoic acid (PFOA) concentrations at two different military installations. (**A**) Alaska military base; (**B**) Pease Air Force Base (AFB). Missing data were excluded from the plots.

3.2. PFOS Data

PFOS concentrations in each well were plotted by month (Figure 2). Tabular data are available in Supplementary Materials (Tables S1 and S2). The relationship between collection date and PFOS at the Alaskan installation was non-significant at (p = 0.49), while at Pease AFB the relationship was statistically significant ($p < 0.001$), but explained little of the variation in PFOS concentrations (R^2 = 0.04), leaving 96% of the variation unexplained by the collection date. These values are higher than the reported PFOA concentrations, suggesting the primary source of contamination at the Alaskan site is from AFFF. Previous studies have found a similar pattern in concentrations when studying AFFF [38,39].

Concentrations at five of the wells sampled at the Alaskan installation were above the EPA health advisory level for at least one measurement. Interestingly, PFOS concentrations were consistently highest at the dog kennel well, followed by well B. This is likely due to the proximity of the wells to point sources, caused by prior firefighting exercise.

For wells A, E, and F, the concentrations were above the health advisory level for one to two out of eight measurements. Therefore, the use of a single annual measurement should consider the possibility for random variation in the reading that could affect future EPA compliance. The total span in the measured concentration across months was 29, 222, and 2990 ng/L for wells A, E, and F, respectively.

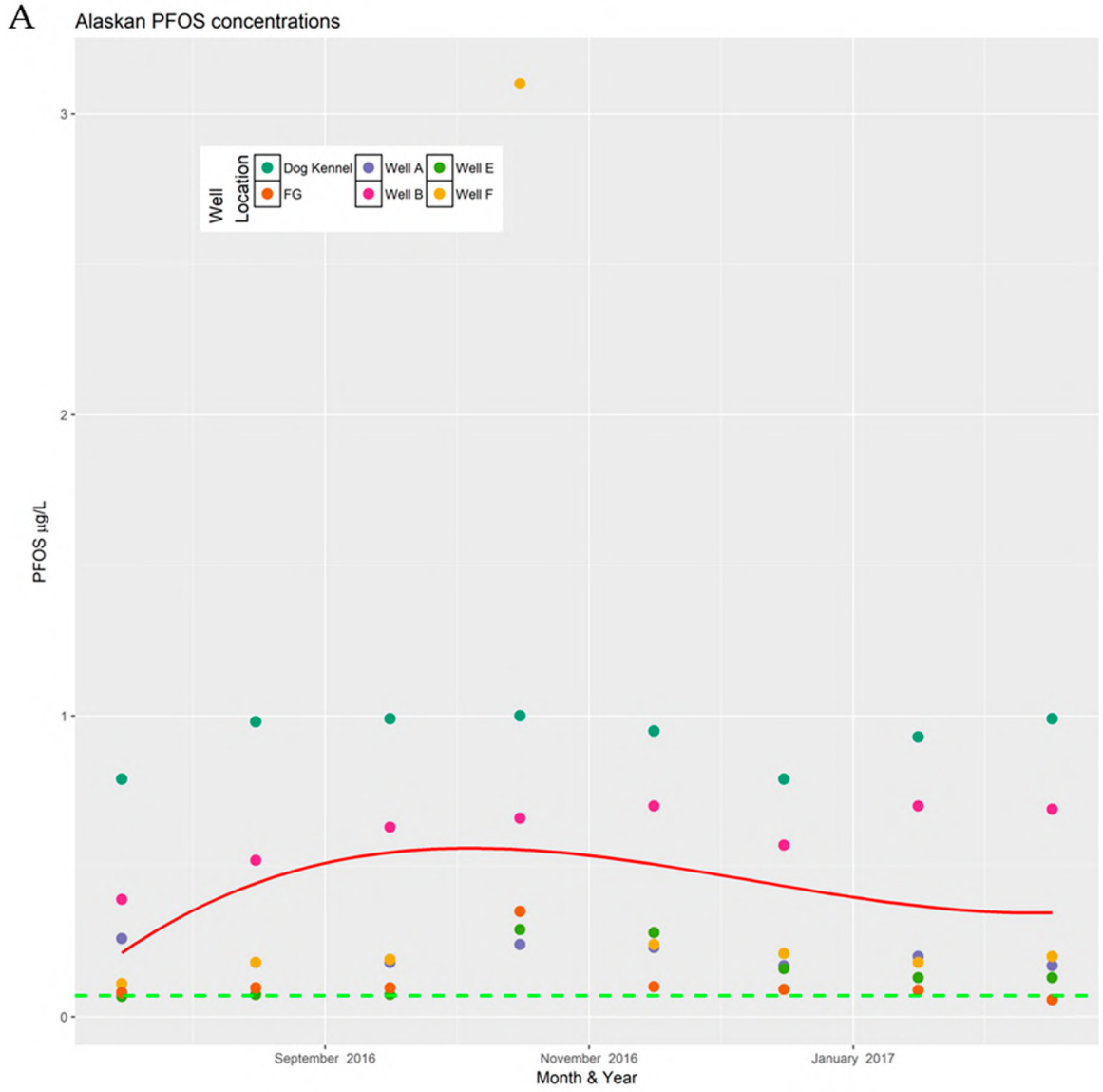


Figure 2. *Cont.*

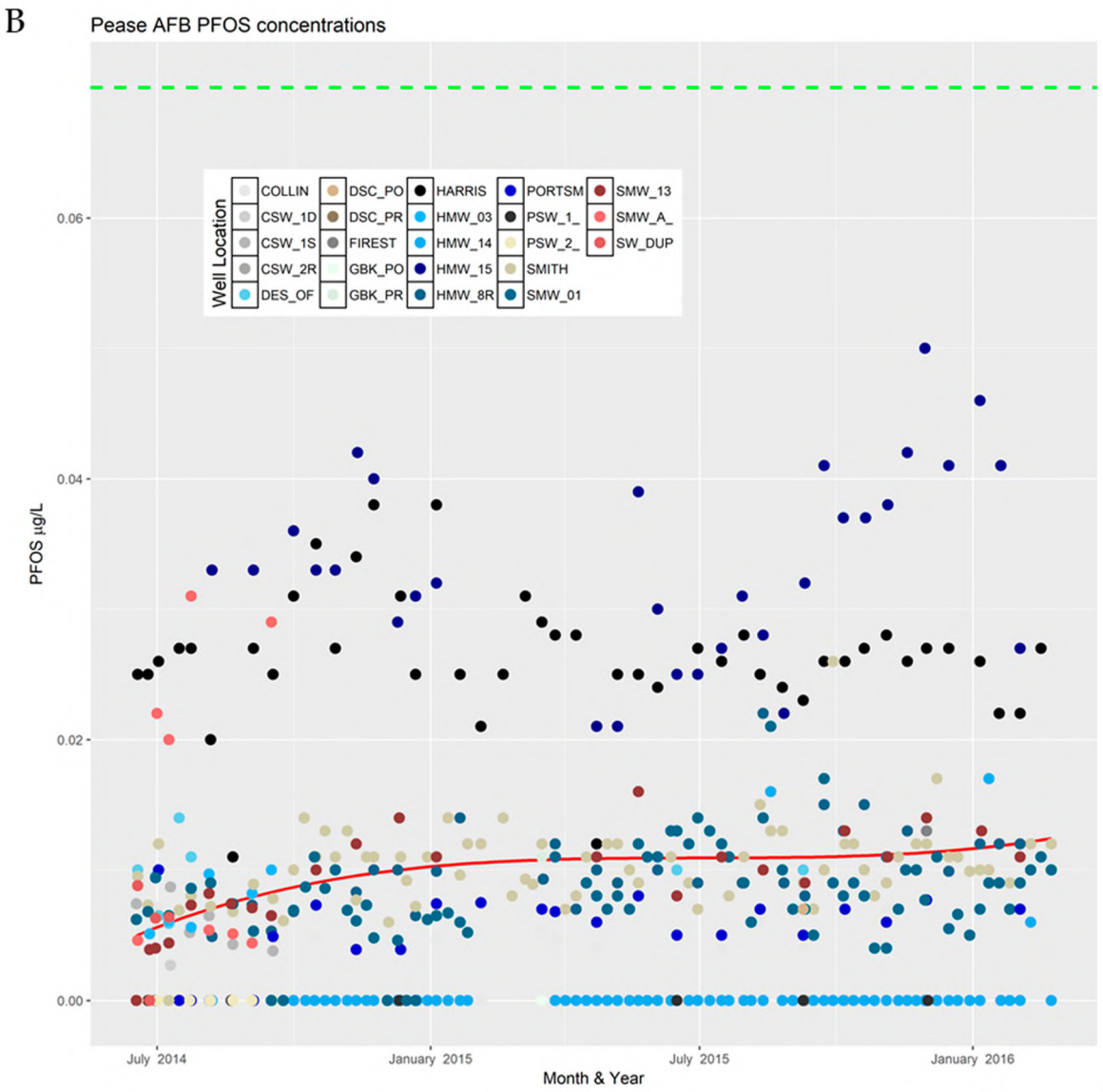


Figure 2. The above chart shows the variance in perfluorooctanesulfonic acid (PFOS) concentrations, over 8 months at, two different military installations. (**A**) Alaska military base; (**B**) Pease AFB. Missing data were excluded from the plots. Because of the numerous data points for the Pease data, only points representing concentrations above 0.02 µg/L were labeled on the plot.

Data on perfluorobutanesulfonic acid (PFBS), perfluorohexane sulfonic acid (PFHxS), perfluoroheptanoic acid (PFHpA), and perfluorononanoic acid (PFNA) were collected (Figure 3). When reviewed, if the analytes were present, they showed similar trends to those observed with PFOS and PFOA.

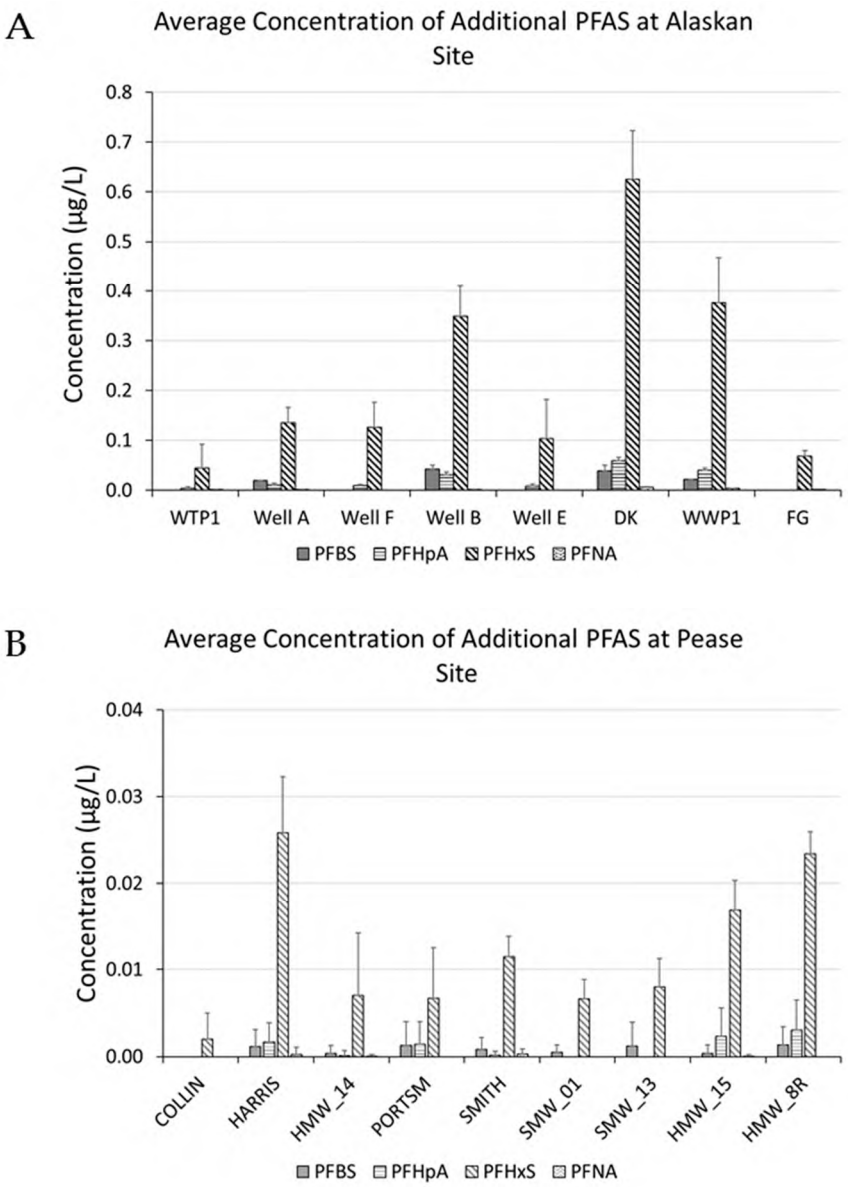


Figure 3. The above chart shows the average and standard deviation in additionally sampled polyfluoroalkyl substances (PFAS) concentrations during the same time frame. (**A**) Alaska military base; (**B**) Pease AFB. Missing data were excluded from the plots. Because of the numerous data points for the Pease data, only wells that consistently had positive identification of the PFAS are shown. PFBS, perfluorobutanesulfonic acid; PFHpA, perfluoroheptanoic acid; PFHxS, perfluorohexane sulfonic acid; PFNA, perfluorononanoic acid.

4. Discussion

Based on the statistical model detailed above, there was not a statistically significant correlation between PFOS and PFOA concentration and collection date at the Alaskan installation. At Pease AFB there were statistically, but not practically significant correlations. As can be seen by the wide variation in observed concentrations above and below the collection date concentration regression line, collection date is of little value in predicting contaminant concentrations, as one would expect given R^2 values of 0.10 and 0.04 for PFOA and PFOS, respectively. Therefore, the Pease results have no practical importance and monthly sampling would not be useful for wastewater or drinking water managers in the monitoring of PFOS or PFOA. This is similar to the results of a previous study that found seasonal trends were not observed in PFOS and PFOA concentrations from wastewater

treatment facility effluents [40]. This result is also compatible with the findings published in literature that showed long chain PFAS concentrations vary insignificantly over years [41]. While not part of current EPA health advisories, the concentration of PFHxS observed was comparable to the PFOS concentration at both sites. PFHxS is currently under consideration by the Stockholm Convention for classifying it as a persistent organic pollutant.

The United States Government Accountability Office published a report in October 2017 outlining the state of the DOD response to PFAS contamination [42]. As of May 2016, the military departments of the DOD have all restricted use of legacy AFFF to emergencies to avoid unnecessary releases into the environment. Testing of alternative AFFF without PFOS as a component is in progress by the Navy. DOD funding is currently being used to explore AFFF that does not use PFAS at all, although testing is anticipated to take until 2020. This final item, the investigation of AFFF that does not require PFAS to operate, is paramount, since there is concern that the proliferation of replacement PFAS only differs minutely from PFOS and PFOA [43]. Within the European Union, AFFF that contains PFOS was banned in 2011. Other PFASs have been identified as substances of concern, under the European chemicals regulation, REACH. A number of PFOS and PFOA AFFFs are available, though adoption of any of these alternatives by the U.S. DOD must follow a rigorous review.

There were significant limitations to this study. Both Pease and the Alaskan military installation draw their water from a single aquifer, so wells within the field cannot truly be treated as independent from one another, complicating analysis of the concentration changes observed. The longest monitored site in Pease was 2.5 years. Ideally, the study would include five years of data collected at two-week intervals to produce more conclusive results. Both time and financial constraints did not allow for this reality.

5. Conclusions

PFAS are persistent, pervasive contaminants that have been found on all continents and in the serum of many animals that have been studied [44–46]. They resist degradation while in the environment and require more sophisticated purification techniques to remove from water, once they have been introduced [25,47,48]. There is a growing body of evidence to suggest low-level exposure to PFAS has long-term effects on humans [49,50]. The inclusion of PFOS and PFOA in the UCMR3 and the reduction in suggested concentration for the EPA's drinking water health advisory have given these compounds public visibility in the United States [51–53].

The results of this study agree with the consensus for short-term laboratory tests that PFAS concentrations do not vary significantly on the scale of weeks or months, although the results presented here are drawn from true environmental samples [54]. Conversion from traditional water treatment techniques to more sophisticated techniques is an expensive undertaking. In the United States, the Defense Environmental Restoration Program requires the DOD to clean up current and former installations, including remediating contaminated land after it has been transferred to another party if contamination is discovered after the transfer has taken place [42].

The Government Accountability Office report also outlines the number of current and former military installations suspected to be contaminated with legacy PFAS: 203 installations in the Air Force, 127 installations in the Navy, and 61 installations in the Army [52]. As new AFFF are introduced, the risk of exposure to different PFAS than PFOS or PFOA increases. Comprehensive monitoring of PFAS is needed, since human health effects for shorter chain PFAS is still of concern. No one-size-fits-all remediation strategy exists. Several competing in situ mediation techniques exist in varying stages of development [55,56]. Some water treatment techniques are mature enough to treat contaminated sites prior to distribution to consumers, but these treatments do not address the underlying contamination. The longevity of these contaminants in the environment requires strategic treatment for individual sites. The Air Force has identified more potentially contaminated sites than both the Navy and Army combined, so proactive investigation is critical in the coming years.

Supplementary Materials: The following are available online at http://www.mdpi.com/2305-6304/6/3/56/s1, Table S1: PFOA concentrations in ng/L at former Pease AFB; Table S2: PFOS concentrations in ng/L at former Pease AFB.

Author Contributions: Conceptualization, M.S. and C.D.; Methodology, M.S. and C.G.; Software, C.G.; Validation, M.S., C.D. and C.G.; Formal Analysis, C.G.; Investigation, M.S.; Resources, C.D.; Data Curation, C.G.; Writing-Original Draft Preparation, M.S.; Writing-Review & Editing, C.D.; Visualization, C.G. and C.D.; Supervision, C.D.; Project Administration, M.S.; Funding Acquisition, C.D.

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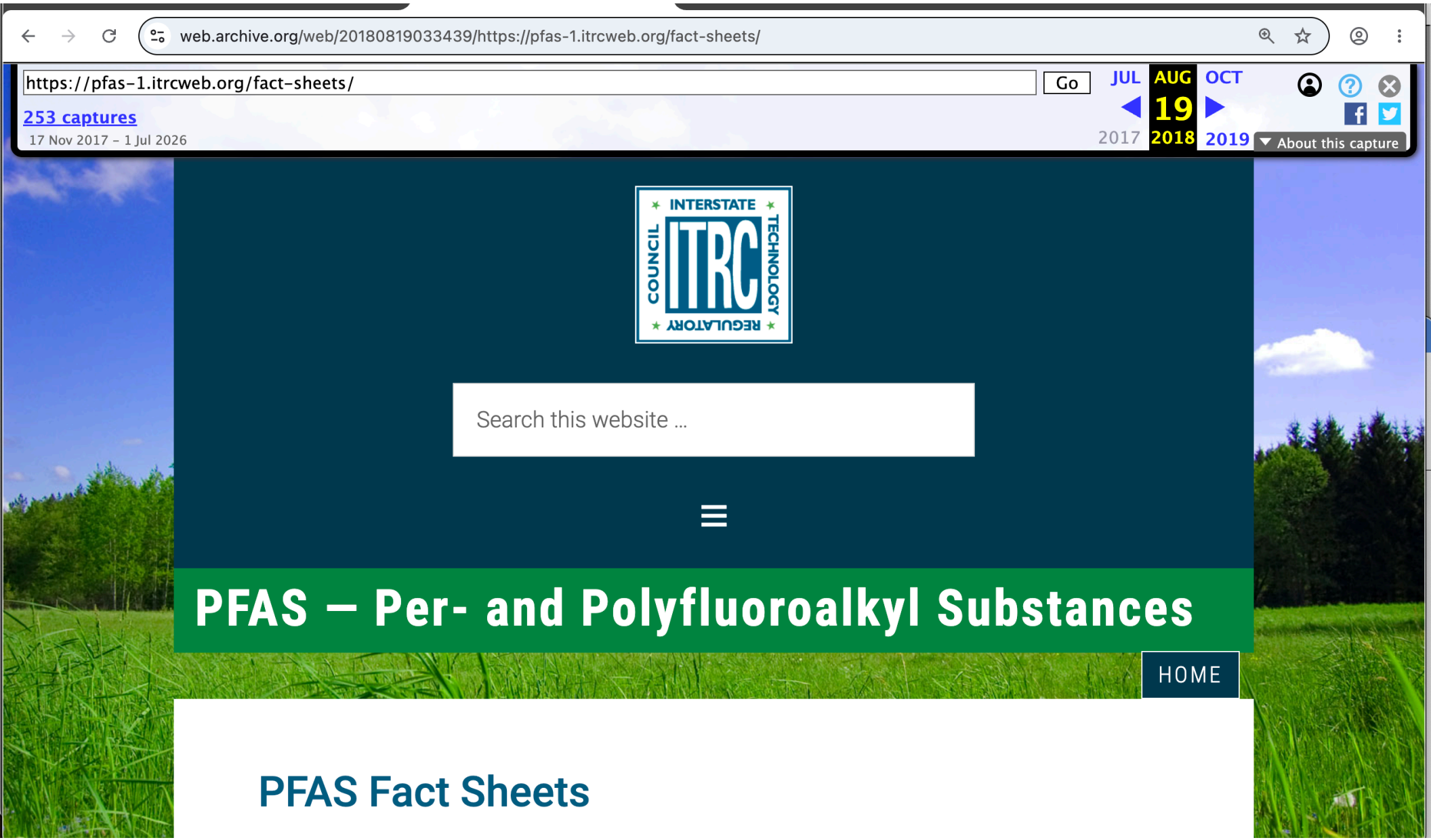
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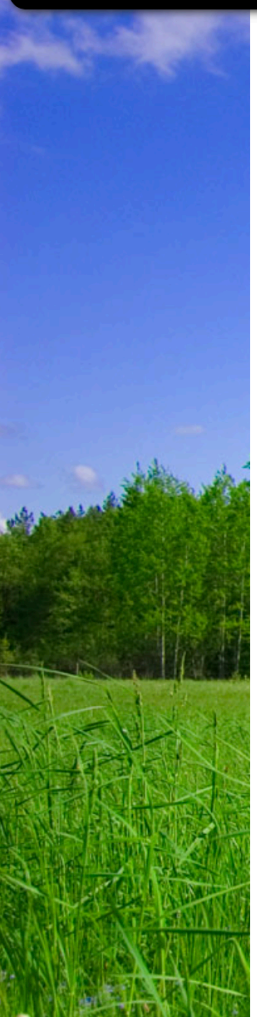


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APPENDIX 1005O


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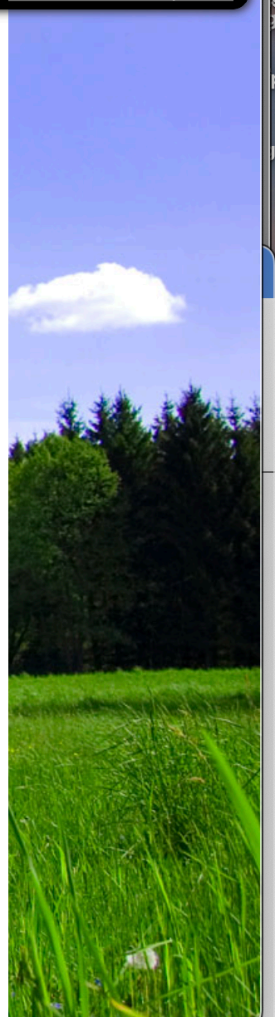


PFAS Fact Sheets

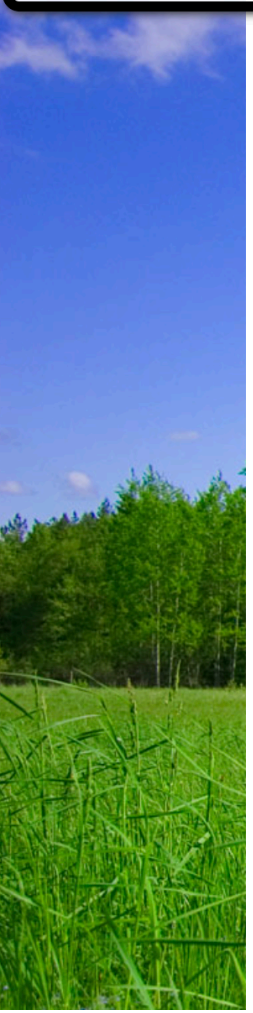
This page includes in the links for the ITRC PFAS fact sheets. The fact sheets are available as PDF files. Several tables of supporting information are published separately so that they can be updated periodically by ITRC. The fact sheet user should visit this page to access the current versions of the files.

An Introductory document (Spanish Version) has been prepared that briefly describes the contents of each of the fact sheets. An Introductory document has been prepared that briefly describes the contents of each of the fact sheets. This web site also includes a combined references list and acronyms list.

- Naming Conventions and Physical and Chemical Properties (updated 3-16-18)
- Regulations, Guidance, and Advisories (updated 1-4-18)
  - Section 4 Tables Excel file – (updated 7-16-18)
    - Table 4-1 presents the available PFAS water values established by the USEPA, each pertinent state, or country (Australia, Canada and Western European countries)
    - Table 4-2 presents the available PFAS soil values established by the USEPA,



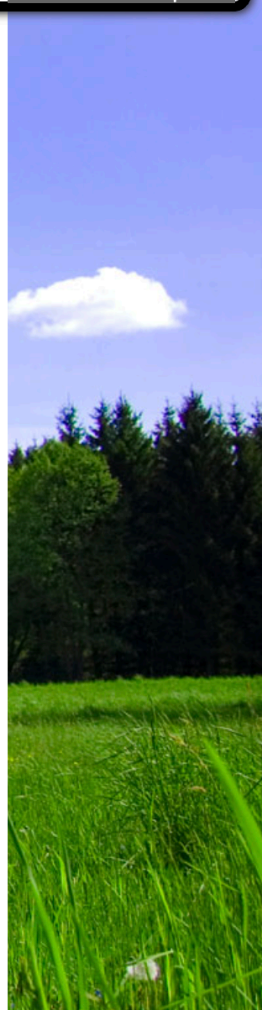
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each pertinent state, or country (Australia, Canada and Western European countries)

- Section 5 Tables Excel file (published November 2017)
  - Table 5-1 summarizes the differences in the PFOA values for drinking water in the United States.
  - Table 5-2 summarizes the differences in the PFOS values for drinking water in the United States.

- Regulación, Orientación, y Asesoramiento para sustancias Per- y Polifluroalquiladas (PFAS) (Spanish Version)
- History and Use (published 11-13-17)
- Historia y Uso (Spanish Version)
- Environmental Fate and Transport (published 3-16-18)
  - Table 3-1 Log Koc values for select PFAS Excel file (published April 2018)
- Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods (published 3-15-18)
- Remediation Technologies and Methods (published 3-15-18)
  - Remediation Comparison Tables Excel file (published April 2018)
    - Table 1 – Solids Comparison



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- Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods (published 3-15-18)
- Remediation Technologies and Methods (published 3-15-18)
  - Remediation Comparison Tables Excel file (published April 2018)
    - Table 1 – Solids Comparison
    - Table 2 – Liquids Comparison
- Aqueous Film Forming Foam (to be published August 2018)

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APPENDIX 1005P

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Remediation Technologies and Methods for Per- and Polyfluoroalkyl Substances (PFAS)

1 Introduction

Remediation technologies exploit chemical and physical properties to immobilize, remove, or destroy the targeted contaminants. Certain PFAS have recently been the subject of regulatory actions and attempted soil, sediment, and water remediation. These compounds have unique chemical properties that require new remediation technologies or innovative combinations of existing technologies. The decision to remediate PFAS should be driven by applicable regulations and an appropriate risk assessment.

USEPA has compiled an online resource for PFAS that includes topics such as policy and guidance, chemistry and behavior, occurrence, toxicology, site characterization, and remediation technologies (USEPA 2017h). The National Groundwater Association (NGWA) has also published a resource on PFAS that includes information about remediation technologies (NGWA

ITRC has developed a series of fact sheets that summarize the latest science and emerging technologies related to PFAS. The purpose of this remediation fact sheet is to:

- provide an overview of remedial technologies and methods for treatment of solids (for instance, soil or sediment) and liquids (for instance, groundwater, leachate, or surface water);
- describe processes for the treatment of PFAS that are now in use or are

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- provide an overview of remedial technologies and methods for treatment of solids (for instance, soil or sediment) and liquids (for instance, groundwater, leachate, or surface water);
- describe processes for the treatment of PFAS that are now in use or are under development; and
- describe the challenges and limitations for each treatment technology.

1.1 PFAS Remediation Technologies Overview

Currently, full-scale PFAS treatment in water is limited to sorption using carbon, mineral media (for example, clay), or a combination of these. Additional pilot and bench-scale technologies are currently being tested. This fact sheet discusses the treatment technologies that have been successfully demonstrated through pilot testing at the field-scale. The accompanying tables summarize technologies that have only been tested in limited applications or in laboratory bench tests. Combining technologies may overcome limitations of any one given technology or expand the efficacy of each technology.

For each technology presented, this fact sheet discusses the following key elements:

- *Treatment Description* (according to each applicable media)
- *Treatment Mechanism* (for example, separation, sorption)
- *State of Development* – Is the technology at lab or bench-scale, field pilot-scale, or full-scale implementation? How many demonstration tests and what is the degree of commercialization for each technology?
- *Effectiveness* – Summary of demonstrated effectiveness on a broad range of PFAS (for example, Method 537 (Shoemaker, Grimmett and Boutin 2009) suite plus fluorotelomer sulfonates), a limited subset (for example, Third Unregulated Contaminant Monitoring Rule [UCMR3] list USEPA 2017f), or only perfluorooctane sulfonate (PFOS) and/or perfluorooctanoate (PFOA).
- *Sustainability Considerations* – Design considerations are evaluated including green remediation elements such as carbon footprint from energy usage, treatment media, and residual handling transport, as well as potential community impacts.

The current state of full-scale PFAS treatment in water is limited to sorption using carbon and/or mineral media (for instance, clay).

Two supporting tables comparing PFAS remediation technologies are available in a separate Excel file: Table 1, Solids Comparison, and Table 2, Liquids Comparison. The tables present all reported treatment technologies, including those tested only at the laboratory/bench scale. To be included in the tables, a technology must have been documented in a publicly available document. Some technologies, however, are only documented in literature supplied by the inventor/researcher/vendor of that technology, with no independent confirmation or peer-review process—be aware of possible biases. ITRC periodically updates the supporting tables and maintains the most current version on its website.

Air treatment is not included in this fact sheet because the current research is limited, and this topic is less applicable to site remediation projects.

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1.2 Factors affecting remedy selection

Site characteristics that affect PFAS remedy selection include the nature of the source, release pathways, affected receptors, and fate and transport in the environment (see *Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods; Environmental Fate and Transport;* and *Naming Conventions and Physical and Chemical Properties* fact sheets). Other site characteristics relevant to remedy selection may be identified as remedial technologies advance. Strategies for remediation of the broader class of PFAS may require complementary technologies, several of which are still being developed, as described in Tables 1 and 2.

Other factors affecting PFAS remedy selection include:

- **Characteristics of PFAS**. The wide-ranging chemical and physical characteristics of PFAS affect the remedy effectiveness. Key factors include ionic state (anionic, cationic, and zwitterionic), types of ionic groups (sulfonate or carboxylate), lipo- and hydrophobicity, nature and reactivity of alkyl groups, chain length and branching, partitioning coefficients, volatility, solubility, and acidity.
- **Changes in PFAS properties**. Chemical and physical properties resulting from naturally occurring processes or due to remedial actions for other (commingled) contaminants, such as chlorinated solvents, and petroleum hydrocarbons, can affect PFAS distribution and mobility in groundwater (McGuire et al. 2014). Example changes include:
  - The alkyl functional group of some PFAS may be more readily subject to chemical or biological transformation than the fully fluorinated aliphatic chain. This is the basis, for example, for the total oxidizable precursor (TOP) assay to estimate concentrations of precursor compounds (Houtz and Sedlak 2012).
  - Partial degradation of the carbon-carbon bonds in the aliphatic chain reported for some chemical remedies generates short-chain PFAS, which may be more mobile (Guelfo and Higgins 2013).
  - Modifications in aquifer properties (for example, redox, pH, or other geochemical characteristics) during remediation of comingled contaminants results in a conversion of PFAS to the more stable and mobile perfluorocarboxylic acids (PFCAs) (McKenzie et al. 2015, 2016).
- **Community acceptance**. Communities are often faced with trade-offs in terms of cost, level of clean-up, and residual contamination as part of remediation efforts. Stakeholder engagement and effective risk communication strategies are an important aspect of the overall remedial technology selection process, especially where off-site receptors are identified.

Factors specifically challenging for PFAS remediation include:

- Multiple ionic states
- Variable isomers
- Differing alkyl groups
- Past remediation effects
- Common co-contaminants

1.3 Technical Maturity

The treatment technologies described here are organized by degree of current confidence in the technology. Three levels of confidence are defined as follows:

- *Demonstrated Technologies*—Technologies that have been demonstrated under pilot or full-scale conditions and are well documented for multiple applications in peer-reviewed literature. These technologies are discussed in greatest detail.
- *Partially Demonstrated Technologies*—Technologies that have been documented in peer-reviewed literature by multiple researchers or practitioners but have only been executed at the laboratory or bench scale. These technologies are briefly mentioned.
- *Promising Technologies*—Technologies that have been demonstrated at the laboratory or field pilot-scale, but the results have not been rigorously peer reviewed. Often, these results are only reported by one group (for example, one university, practitioner, or vendor) or lack detailed validation of the treatment or mechanisms. These technologies are omitted from the text and presented only in the technology comparison tables (Tables 1 and 2).

Experimental techniques to treat PFAS are under development, but only those technologies that have some level of publicly available documentation demonstrating effectiveness are included here.

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2 Technologies for Treatment of Solids

Several technologies are currently available to remediate PFAS in solids. Most of these technologies have been demonstrated on soils only but may also apply to saturated sediments or sludge treatment.

Field-Demonstrated Treatment Technologies for PFAS in Solids

- Excavation and off-site landfilling or incineration
- Sorption/stabilization through ex situ soil mixing
- Ex situ thermal desorption and off-gas destruction

2.1 Capping

Technology Description: Capping places a cover over contaminated material such as landfill waste, contaminated soil, and sediments. Caps do not destroy or remove contaminants. The purpose of the cap is to prevent contact with the contamination and, depending on the design, to reduce or prevent further leaching of contaminants. For PFAS soil sites, the main purpose of a cap is to reduce or prevent further leaching of contaminants from soils to groundwater.

Treatment Mechanism: Caps isolate contaminants and prevent them from spreading and causing exposure by direct contact.

State of Development: Capping is a viable remediation method because it is simple technology that applies to most contaminants. Although capping has not been applied to a PFAS site, based on documented successes with other contaminants it offers promise for PFAS.

Effectiveness: Capping may prevent exposure and potentially reduce infiltration, but redevelopment options for the capped land surface may be limited. Caps are most effective when the seasonal high-water table is well separated vertically from the base of the contaminated solids. The PFAS contamination will remain at the site and therefore be a long-term liability. Contaminants could mobilize if site conditions change (for example, rising water table). Caps incur maintenance costs to maintain the integrity of the cap and require institutional controls to ensure the cap is not breached by future site uses or redevelopment.

Sustainability Considerations: The carbon footprint of capping includes earthwork equipment emissions and manufacturing and transporting capping material. Ecosystem restoration can be incorporated into the capping design (Lamb et al. 2014). Community impacts include truck hauling traffic and hindrance of redevelopment due to land use restrictions.

2.2 Excavation and Disposal

Technology Description: This remediation method includes removing contaminated soil and hauling it to a permitted landfill or incineration facility, then filling the excavated area with clean backfill.

Treatment Mechanism: This method isolates PFAS from receptors. Sometimes, PFAS-impacted soil is stabilized or solidified before disposal into a landfill. Disposal of PFAS-impacted soils or wastes into unlined landfills should be avoided. More studies are needed on interactions of PFAS with landfill linings.

Some states may require PFAS-contaminated solids to be treated at a Resource Conservation and Recovery Act (RCRA) permitted incinerator. The estimated temperature to destroy specific PFAS varies widely, from around 300° to greater than 1,000°C (Vecitis et al. 2008). Commercial incineration is often the only viable disposal option for media contaminated with PFAS.

State of Development: Solid wastes generated from environmental investigation and cleanup (for example, soil excavation, and investigation- and remediation-derived wastes) have been disposed at lined landfills, and therefore this is considered a well-demonstrated technology. PFAS have been reported in landfill leachate (Lang et al. 2017), although it is unlikely the source for these PFAS is investigation derived waste, as opposed to consumer product waste containing fluorochemicals. The combination of soil excavation and incineration is also considered to be a viable remedial option.

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Effectiveness: Excavation and disposal of PFAS-contaminated soil effectively removes a source area that may otherwise serve as a continuing source of groundwater contamination but does not result in destruction of the PFAS. Additionally, some nonhazardous waste landfills do not accept PFAS waste. Disposal of PFAS waste to landfills potentially adds to the PFAS contaminant load in the landfill leachate. In some states, the leachate is not analyzed or regulated for PFAS.

Sustainability Considerations: The carbon footprint for this approach includes earthwork equipment emissions, transporting contaminated soil and backfill, and manufacturing (such as resource extraction) of backfill material. Landfill incineration of the contaminated soil is energy intensive. Truck hauling traffic affects the local community. Guidance is available for performing a sustainability assessment for an excavation and disposal remedial design (Cappuyns and Kessen 2014; Goldenberg and Reddy 2014; Söderqvist et al. 2015; Song et al. 2018).

2.3 Sorption and Stabilization

Technology Description: Amendments added to the soil reduce or remove the potential for PFAS to mobilize from soil to groundwater.

A few commercially available sorbents have been specifically developed to immobilize PFAS in soil.

Treatment Mechanism: Amendments adsorb or stabilize PFAS. These amendments include activated carbon and carbon nanotubes (CNTs), resins, minerals, biomaterials, and molecularly imprinted polymers. The amendments bind to PFAS and thus reduce their release from soil.

State of Development: Sorption and stabilization techniques using carbon-based amendments are considered partially demonstrated technologies. Both granular activated carbon (GAC) and powdered activated carbon (PAC) rapidly remove PFAS from groundwater and surface water and can be applied to soil, but efficacy is reduced in the presence of organic co-contaminants (NGWA 2017). Performance also depends on PFAS chain length and functional group (Xiao et al. 2017). Laboratory tests should be conducted using site specific soils and the stabilizing amendment before field application to ensure success. Carbon amendments can be modified to enhance their sorption of PFAS. One patented amendment is carbon enhanced with aluminum hydroxide, kaolin clay, and other proprietary sorbents (USEPA 2017p).

Sorption and stabilization techniques using non-carbon-based sorbents, such as iron oxide minerals and modified organoclays (such as montmorillonite [Mt], hydrotalcite, and palygorskite) are promising, but only limited tests have been conducted. Minerals such as clays, silica, iron oxides, and zeolites have been used as sorbents for removing contaminants from groundwater and soil (Zhu et al. 2016; Rattanaoudom, Visvanathan, and Boontanon 2012; Zhou et al. 2010; Zhou, Pan, and Zhang 2013). The surface of organoclays can also be modified for enhanced PFOS and PFOA sorption (Zhou et al. 2010; Kambala and Maidu 2013; Zhu et al. 2016). Additionally, an amine-modified palygorskite clay sorbent has been patented for the treatment of PFOS and PFOA (Kambala and Maidu 2013).

Effectiveness: Carbon- and mineral-based sorption and stabilization techniques vary in their effectiveness according to site conditions and PFAS type. An example of a site condition that can affect sorption is high organic matter in soil, which can foul carbon sorbents with competing compounds. PFAS type affects sorption in that PFAS often occur as mixtures, including PFAS of different chain lengths with varying sorption characteristics. Sorption capacity was assessed in one study where CNTs were mixed with sediments at 4% weight per weight (w/w), and the sorption capacity of the sediment increased in comparison to untreated sediment (Kwadijk, Valzeboer, and Koelmans 2013).

Organoclays are used because they are environmentally benign, have a high sorption capacity, and can be easily modified to enhance their sorption capacity with mesopores. Organoclays have also been proven to work on several classes of contaminants (Zhu et al. 2016; Espana, Mallavarapu, and Naidu 2015; Zhou et al. 2008). The surface of organoclays are hydrophilic, and therefore ineffective for sorption of hydrophobic organic compounds like long-chain PFAS; however, modification with cations changes the surface to lipophilic. For example, Mt often exists as Na-Mt with a sodium cation on the surface, which is lipophilic and may be effective for long-chain PFAS.

Biomaterials such as chitosan, straw, and quarternized cotton do not perform as well as other sorbents, and the biomaterials may eventually degrade (Du et al. 2014).

Sorption and stabilization do not destroy PFAS, and information on the long-term stability of amendments for PFAS remediation is a data gap that currently limits their use. The amended soil can be mixed with concrete and other stabilizers to better trap the PFAS.

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Sustainability Considerations: The carbon footprint for sorption and stabilization includes emissions from earthwork equipment, manufacturing (for example, resource extraction), and transporting amendment material. Community impacts include hindrance of redevelopment due to land use restrictions. Resources are available for performing a sustainability assessment for sorption and stabilization remedial design (Goldenberg and Reddy 2014; Hou et al. 2016; Kuykendall and McMullan 2014).

2.4 Thermal

Technology Description: The mobilizing or destruction of chemicals using heat.

Treatment Mechanism: Heat is applied directly to the PFAS-contaminated soil. High temperatures can vaporize the chemicals or potentially destroy them. Vaporized chemicals can be captured and destroyed in off-gas treatment.

State of Development: Ex situ thermal treatment has been demonstrated at the field pilot-scale by a few technology vendors and is considered a partially demonstrated technology (Endpoint Consulting 2016; Enviropacific 2017). The use of this technology for PFAS-contaminated soil is still developing. Limited data sets are available, and several data gaps still exist. Additionally, no documented examples of in situ thermal treatment for PFAS-impacted soil have been identified.

Effectiveness: Results of one test indicated that the complete removal of a suite of nine PFAS was possible within 30 minutes but required temperatures over 900º C (Endpoint Consulting 2016). In another field pilot project, concentrations of 20 PFAS in soil were reduced to below reporting limits—greater than 99.9% reduction (Enviropacific 2017). At this time, it is unknown whether the pilot test resulted in volatilization or complete destruction. This test was performed at relatively lower temperatures (for example, approximately 450º C). There are still several data gaps related to thermal incineration of PFAS that should be considered when applying this technology. For example, the mass balance to assess whether PFAS are destroyed or simply mobilized is not completely understood.

Sustainability Considerations: Thermal treatment is an energy-intensive remediation method. Its carbon footprint includes the energy source and consumption during treatment system operation, as well as manufacturing and installation of heating system materials. Community impacts include managing the risks of potential vapor intrusion from volatile co-contaminants. Guidance is available for performing a sustainability assessment for thermal remedial design (Song et al. 2018; Vidonish et al. 2016).

3 Liquids Treatment

Several technologies are currently available for remediating PFAS in liquids. These technologies can be applied to drinking water supplies, groundwater, industrial wastewater, surface water, and other miscellaneous applications (such as landfill leachate). Influent concentrations of PFAS can vary by orders of magnitude for specific media or applications. These influent values, along with other general water quality parameters (for example, pH) can influence the performance and operating costs for the treatment technologies.

Field Demonstrated Treatment Technologies for Liquids

- Extraction and sorption with granular activated carbon or anion exchange resin
- Extraction and membrane filtration/reverse osmosis
- Extraction and precipitation/flocculation

3.1 Sorption

Many organic compounds can be treated by passing contaminated water through special granular media. The mechanism of this technology varies depending on the media, contaminant, and influent concentrations. There are two broad categories of PFAS sorption treatment: adsorption onto carbon media and ion exchange.

3.1.1 Granular Activated Carbon

Technology Description: Granular activated carbon (GAC) is made from organic materials, such as coal and coconut, which serve as effective adsorbent media because they are highly porous and provide a large surface area for contaminant contact. GAC treatment can be used for any aqueous-based treatment application (for example,

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municipal drinking water, groundwater, point-of-use residential, industrial wastewater, and landfill leachate). The GAC media is placed in packed-bed, flow-through vessels generally operated in series (lead-lag configuration). Either virgin or reactivated GAC can be used for most applications, but virgin GAC is the industry preference for more risk-averse drinking water applications. Commercial facilities in the United States conduct thermal reactivation of spent GAC, which can provide a more sustainable and less costly replacement option than virgin GAC and off-site disposal. To address concerns about using reactivated GAC, vendor bench-scale column studies can be performed prior to initial usage, and a quality control testing program can be implemented prior to delivery of each shipment.

Treatment Mechanism: Removal of PFAS from treated water by GAC is an adsorption process, as well as a physical mass transfer process from the aqueous phase onto solid media that does not involve any form of chemical degradation or transformation. Adsorption is a surface chemistry phenomenon by which an aqueous phase contaminant adheres to the surface of a granular media (via electrical, physical, or chemical processes), but does not penetrate it. The GAC adsorption capacity can vary considerably by media and contaminant. Adsorbent media must be removed and replaced when it becomes spent, meaning contaminants break through at concentrations above some established criteria. The spent media must be replaced and shipped off-site either for disposal (by landfilling or commercial incineration) or to be regenerated/reactivated for reuse consistent with applicable federal and state regulations.

State of Development: GAC is a demonstrated technology and is currently the most common water treatment method used for PFAS. Because of the limited treatment history and available technologies for PFAS, full-scale applications to date have mostly focused on higher priority private and public water supply and residential point-of-use treatment. Currently, only a few operating groundwater pump-and-treat applications use GAC treatment. Much of the published literature on GAC treatment of PFAS involves bench-scale and vendor column studies. Treatability data for full-scale operations involving different technologies, including GAC, have also been compiled and reported for several municipal wastewater treatment plants where PFAS have been detected in the influent.

Many sources in the literature support the use of GAC: Appleman et al. (2013); Szabo et al. (2017); and Woodard, Berry, and Newman (2017); and others cited in this section. These references also include more comprehensive bibliographies if further details are needed on specific topics or studies.

Effectiveness: GAC has been shown to reduce select PFAS to very low or nondetectable concentrations, on the order of nanograms per liter (equivalent to parts per trillion), with reported removal efficiencies in various references between 90% and >99%. The lower end of these reported GAC removal efficiencies may be the result of the faster breakthrough times for the short-chain PFAS (Xiao et al. 2017; Dickenson and Higgins 2016). Early municipal treatment plant sampling studies focused mostly on PFOA and PFOS, which are considered long-chain PFAS (defined as six carbon atoms or more) and currently drive most drinking water treatment decisions. Consequently, the shorter breakthrough times for PFAS with five carbon atoms or less were initially missed, and subsequently reported in the literature as lower GAC removal efficiencies.

Breakthrough and GAC usage should be checked for a variety of PFAS, not just PFOA or PFOS, including various chain-lengths.

Individual PFAS have different GAC usage capacities and corresponding breakthrough times. GAC removal capacity for PFOS is greater than PFOA, but both can be effectively captured. In general, PFAS containing five carbon atoms or less have higher GAC usage and much quicker breakthrough times than PFAS containing six carbon atoms or more with other factors being equal (such as influent concentration). Vendor column studies (Brewer 2017) with equivalent influent concentrations and empty contact bed times have shown that short-chain PFAS breakthrough times are approximately five times quicker than long-chain PFAS. Pilot and full-scale GAC treatment data show similar comparative breakthrough times (Brewer 2017).

In addition to usage capacity, several other factors affect GAC change-out frequency and cost for individual PFAS (for example, influent concentrations). Change-out frequency therefore cannot be predicted solely by the presence of specific short-chain PFAS. When concentrations of short-chain PFAS are much lower than concentrations of long-chain PFAS, GAC is still a cost-effective treatment for PFAS other than PFOA and PFOS. Because of the differences in GAC usage capacities between individual PFAS, treatability studies must evaluate the entire mixture of PFAS present in the influent to the extent practicable. Column studies are the best method to predict GAC performance and change-out frequency.

Column studies show that virgin GAC and thermally reactivated GAC have similar removal rates and breakthrough times (Brewer 2017). Based on vendor feedback (Mimna 2017), commercial thermal reactivation is performed at higher

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operating temperatures than steam or nitrogen regeneration systems and is capable of complete desorption and destruction of PFAS from spent GAC (Watanabe et al. 2016; Yamada et al. 2005). Also, vendor testing demonstrates that re-agglomerated bituminous coal provides better removal performance for PFAS than other types of GAC (Brewer 2017; Nowack 2017).

Sustainability Considerations: The carbon footprint for GAC includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. Spent single-use media requires incineration, which increase the carbon footprint. Regenerable media presents sustainability benefits because the media is reused; however, in drinking water applications, virgin material achieves greater confidence in treatment. Resources are available for performing a sustainability assessment for sorption remedial design (Amini et al. 2015: Choe et al. 2013, 2015; Dominguez-Ramos et al. 2014; Favara et al. 2016; Maul et al. 2014; Rahman et al. 2014; Ras and von Blottnitz 2012).

3.1.2 Biochar

Technical Description: *Biochar* is a hybrid word derived from *biomass* and *charcoal*. Biochar is a carbon-rich, porous solid synthesized from biomass, such as wood or manure, through a high-temperature low-oxygen process called "pyrolysis" (Ahmad et al. 2014). Key factors controlling the properties of biochar (for example, pore size, chemical composition, and hydrophobicity) include the temperature of pyrolysis and biomass feedstock.

Treatment Mechanism: The properties of biochar are comparable to those of GAC for sorptive purposes. Like GAC, biochar can adsorb organic contaminants.

State of Development: Biochar is considered a partially demonstrated technology. Various laboratory experiments have evaluated the efficacy of biochar compared to other media, but no full-scale treatment systems are in place for the removal of PFAS. This work demonstrates that biochar is potentially viable for treatment of PFAS, but additional research is needed to fully establish viability and costs.

Effectiveness: Xiao et al. (2017) compared one GAC and two commercially available biochars for treating an aqueous film-forming foam (AFFF)-impacted water supply. Based on batch studies, they concluded that biochars with large surface areas could be an alternative to GAC, although variability in biochar properties relative to GAC may affect reliability. While biochar removal is effective in ultrapure water, when used to treat river water (with more complicated water chemistry), biochar is ineffective compared to ion exchange and GAC and exhibited significantly slower adsorption kinetics (Rahman 2014).

Sustainability Considerations: The carbon footprint of this technology includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. The use of waste material as a starting feedstock results in a lower overall carbon footprint, though reactivation of biochar is not currently feasible and energy-intensive incineration or landfilling are required, which offsets some of the sustainability benefits.

3.1.3 Ion Exchange

Technology Description: Ion exchange (IX) uses synthetic, polymeric media to remove PFAS from water. IX media are employed similarly to GAC and can be used in combination with GAC.

Both regenerable and nonregenerable IX media are available. Nonregenerable IX is a single-use, disposable medium. Regenerable IX theoretically can be used indefinitely, however, insufficient operational data are available to understand its long-term durability. IX regeneration is a chemical process; the only demonstrated successful regeneration solution is a solvent-brine solution (Amec Foster Wheeler 2017). The regenerant solution can be distilled for reuse. The distillate residue is a concentrated PFAS waste that can be managed by off-site treatment (for example, incineration or possibly chemical oxidation).

Treatment Mechanism: IX is the process by which ions of one substance are replaced by similarly charged ions of another substance. The term denotes purification, separation, and decontamination of aqueous and other ion-containing solutions with solid polymeric or mineralic ion exchangers. Many organic contaminants are ionic and can be removed through specialized ion exchange media. The media are often derived from organic polymers or plastic, and thus ion exchange media are referred to as "resins." Regeneration of ion exchange resins is accomplished with a chemical flush, typically a highly acidic or basic solution, brine solution, or solvent-brine solution, rendering the resin reusable.

To date, IX has used positively charged, anion exchange media to remove negatively charged PFAS molecules, via binding of the carboxylic and sulfonic acid "heads" of perfluoroalkyl acids (PFAAs). Such IX media are manufactured to

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be more selective for PFAS than for typical competing mineral anions such as sulfate and nitrate. The fluorinated carbon chain of the PFAS molecule can also adsorb to IX media. This dual-mechanism, ion exchange plus adsorption, can result in higher removal compared to adsorption alone (Yu et al. 2009).

State of Development: IX is a fully demonstrated technology. Column tests comparing both regenerable and single-use ion exchange media have shown IX to be effective for the removal of several PFAS (Woodard, Berry, and Newman 2017; Conte et al. 2015). Full-scale IX systems are currently in operation in Australia at Australian Defense sites, and a full-scale system was installed in the United States in 2017. The U.S. Department of Defense (DOD) recently funded a SERDP project, ER18-C2-1306: "Combined In Situ/Ex Situ Treatment Train for Remediation of Per- and Polyfluoroalkyl Substance (PFAS) Contaminated Groundwater" to optimize regenerable IX and on-site destruction with plasma under a number of laboratory conditions (SERDP-ESTCP 2017).

Effectiveness: IX is a demonstrated effective technology for removal of anionic PFAS. It has higher adsorption capacity for some PFAS and significantly faster reaction kinetics compared to GAC (Conte et al. 2015). The combination of these properties means an equivalent treatment system for IX is smaller and thus uses less media. Like GAC, usage capacities and corresponding breakthrough times vary depending on PFAS functional groups and chain length. Short chain PFAS may break through faster under certain influent conditions. However, certain single-use IX media have been identified that may have higher usage capacities for short-chain PFAS. An IX treatment system can also include multiple vessels in a lead-lag configuration, consisting of both single-use and regenerable IX media. This design optimizes removal properties and operating costs for a mixture of short and long-chain length PFAS.

Sustainability Considerations: The carbon footprint for IX includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. Spent single-use IX media requires incineration or other treatment for disposal, resulting in an increased carbon footprint. Regenerable IX media offers sustainability benefits because the media can be reused, but the process for regenerating requires energy and creates a concentrated waste stream which must be managed. Disposal or treatment of the regenerant stream can be problematic and expensive. Regeneration solutions may present exchange system and treated water corrosion issues if media are not rinsed thoroughly prior to being placed back in service.

3.2 Precipitation/Flocculation/Coagulation

Technology Description: Coagulation–flocculation is a common pretreatment approach used in wastewater treatment plants for removing various particles and dissolved constituents. Coagulants, either commodity or proprietary chemicals, can be added to water (conventional technology) or generated by anode-cathode reactions of metals plates inserted into the water (electrocoagulation).

Technology Mechanism: Coagulants assist in forming solids. Flocculation is typically conducted by adding a soluble polymer and slowly mixing to allow the particles to agglomerate and grow. Upon solid formation, constituents such as PFAS can be physically incorporated into, or sorbed onto, the flocculated particulate (which is known as co-precipitation). The precipitated solids are then separated from the water by sedimentation, filtration, or a combination of both processes. The solid material containing the PFAS requires disposal.

State of Development: Current literature only documents bench-scale study results on treating PFAS via precipitation, flocculation, or coagulation and therefore this is considered a partially developed technology. Evaluations have focused on conventional commodity chemical coagulation (for example, aluminum or ferric salts) and nonconventional coagulation (for example, proprietary chemical coagulants or electrocoagulation). Pilot and full-scale applications have not been documented in the United States (Birk and Alden 2017).

Unconventional precipitation (for example, electrocoagulation or advanced chemical precipitants) has shown more potential for direct PFAS treatment, but little data is available.

Effectiveness: Electrocoagulation reactors, which range from basic to sophisticated designs, are highly efficient, compact, relatively low-cost, and completely automatable (Baudequin et al. 2011; Lin et al. 2015a). Recent studies have found that PFAAs, such as PFOA and PFOS, can be quickly sorbed on the surface of zinc hydroxide particulates generated by electrocoagulation (Lin et al. 2015a).

One commercially available proprietary chemical product has been shown in tests to reduce PFAS in groundwater containing AFFF (Birk and Alden 2017; CH2M 2017). This research also shows that conventional (ferric salt) and proprietary chemicals in combination are more effective than either alone. Current data have been developed at elevated

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concentrations, but removal for lower (µg/L) concentrations has also been shown (CH2M 2017; Birk 2015). No available data show precipitation effectiveness at very low (ng/L) concentration ranges.

Conventional PFAS precipitation induced by coagulation and flocculation has shown limited applicability for complete treatment of PFAS. Therefore, conventional chemical precipitation could be considered mainly as a pretreatment technology to sorb or precipitate PFAS prior to final filtration or destruction. Also, if used to remove conventional parameters such as solids or natural organic material (NOM), pretreatment would allow PFAS removal technologies to achieve the desired treatment goals in a more cost-effective and technically feasible manner.

Sustainability Considerations: The carbon footprint of this technology includes energy source and consumption during treatment system operation, as well as manufacturing of treatment media. Additionally, extracted solids containing PFAS require final disposal or destruction.

3.3 Redox Manipulation

Technology Description: Redox manipulation is the process of changing the oxidation-reduction potential of water through addition of oxidizing or reducing amendments, or by adding energy to a system to create oxidizing or reducing free radicals. These changes affect the mobility or structure (transformation or destruction) of the PFAS. PFCAs are generally more amenable to redox manipulation than perfluoroalkane sulfonic acids (PFSAs). Redox manipulation may be effective at treating many co-contaminants and may also alter organic matter, which can affect PFAS mobility. These system changes may allow subsequent treatment steps to manage target PFAS more effectively.

Treatment Mechanisms: Redox manipulation mechanisms may include both redox transformation (oxidative, reductive, and nucleophilic processes) of PFAS and changes in redox conditions in the impacted media (for example, groundwater), resulting in changes in the mobility and sorption of PFAS (McKenzie et al. 2015; Arvaniti et al. 2015). Redox transformation involves the transfer of electrons between reactants. In oxidative processes, electrons are transferred to the reactive species (the oxidant) from the target (PFAS), whereas in reductive processes the opposite occurs. In nucleophilic processes, a reactant (the nucleophile) bonds with the PFAS compound and displays an atom or group of atoms from the PFAS molecule.

Susceptibility to redox transformation depends on reaction conditions and the reactive species involved. For some specific technologies, more than one transformation mechanism or reactive species may be involved. For example, plasma, sonolytic, and photolytic technologies may combine physical (high-temperature pyrolysis) and free radical attack processes. PFAS carbon chains do not easily transform, because their carbon-carbon bonds are shielded in part by the tightly bound fluorine atoms that surround the carbon chain (Kissa 2001). The carboxylic or sulfonic group "heads" of PFAS are commonly more susceptible to redox transformation than carbon chain "tails," resulting in partial transformation of the parent compound but not in cleavage of the aliphatic chain (Houtz and Sedlak 2012; Anumol et al. 2016). The tail and head structure are illustrated for PFOS and PFOA in the following figure.

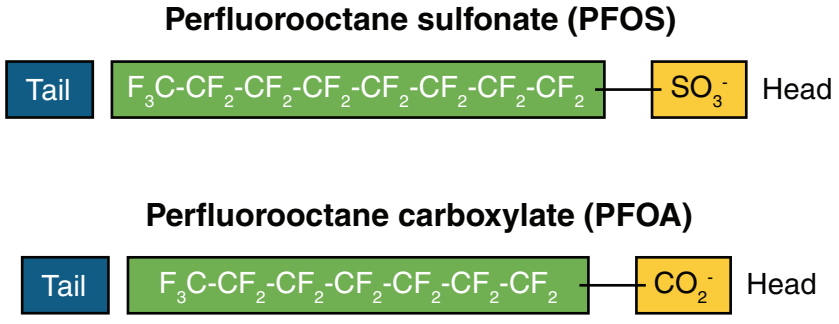


Figure 1. The tail and head structure of PFOS and PFOA molecules.

Reactions of zero valent metals with PFAS, generally considered a reductive process, may also affect mobility and sorption of PFAS in addition to (or rather than) PFAS transformation (Arvaniti et al. 2015). Complete mineralization/defluorination of PFAS via zero valent metal reactions has not been demonstrated to date; however, research in this area is ongoing.

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Combined redox and nucleophilic attack almost completely mineralize PFOA (Niu et al. 2012; Mitchell et al. 2014). A reactant formed in several redox manipulation approaches, the solvated electron (free electron in solution), shows promise for PFAS destruction (Park et al. 2009; Gu et al. 2016; Li et al. 2012; Stratton et al. 2017; Blotevogel, Giraud, and Borch 2018).

State of Development: Redox technologies have not been widely applied beyond laboratory bench-scale tests because of concerns that partial transformation (as opposed to complete destruction) will produce more mobile or toxic products. Therefore, redox technologies are considered a partially developed technology, with both successes and challenges. For example, chemical oxidation affects PFAA transport, but the direction (increased or decreased transport) and magnitude depend on reaction conditions (McKenzie et al. 2015). Apparent in situ destruction of PFAAs in groundwater at a fire training site has also been achieved using a combined ozone/persulfate approach (Eberle, Ball, and Boving 2017). The main difficulty in using redox-based technologies is achieving extremely low (ng/L) PFAS cleanup objectives, particularly for in situ remedies. Additionally, reactive species (for example, oxidizing radicals) interact with other compounds present in soil and groundwater that are more susceptible to oxidation and are at relatively higher concentrations than the target PFAS.

Despite these challenges, several options show promise for ex situ treatment, including electrochemical (Schaefer et al. 2015; Urtiaga et al. 2015), sonolytic (Vecitis et al. 2008; Rodriguez-Freire et al. 2015), plasma (Stratton et al. 2017), and reductive (Arvaniti et al. 2015) technologies. These approaches have successfully degraded an array of high-concentration PFAS at the laboratory scale. However, none of these technologies are sufficiently mature yet to assess PFAS treatment costs and overall effectiveness with confidence.

Effectiveness: A wide range of PFAS treatment methods based upon oxidation-reduction chemistries have been evaluated. These studies demonstrate the following:

- PFAS exhibit a wide range of reactivity toward destructive processes, based upon characteristics including chain length, nature of alkyl groups, and branched versus linear isomers.
- Treatment of PFAS at AFFF sites depends on the composition and production processes of the given AFFF formulation used at the site.
- Except for limited field pilot testing of one ozone and one persulfate-based chemical oxidation technology, none of the technologies summarized in Table 2 have matured beyond laboratory-scale studies.
- While promising, the relatively few field pilot applications have not yet been closely analyzed or duplicated in highly controlled and monitored studies.

Partial transformation can affect other physical and chemical characteristics of PFAS (see Section 1.2). For instance, persulfate-based transformation of PFAS may be incomplete (Houtz and Sedlak 2012) or negligible (McKenzie et al. 2015, 2016), whereas in other cases efficient transformation is reported (Hori et al. 2008; Lee et al. 2009; Liu et al. 2012; Park et al. 2016; Yin et al. 2016). Redox processes in the subsurface can generate unwanted byproducts (for instance, nitrate and bromate from chemical oxidation) depending on site and reaction conditions (Siegrist, Crimi, and Simpkin 2011). Site-specific treatability tests are recommended.

Sustainability Considerations: The carbon footprint for this technology includes energy source and consumption during treatment system operation, manufacturing of amendment materials, and manufacturing and installation of injection points (if implemented in situ). Electrochemical treatment options also require handling of hazardous investigation derived waste, which is a sustainability consideration.

3.4 Membrane Filtration

Membrane filtration refers to a variety of separation technologies based on the nominal size of the membrane pores. Types of membranes include reverse osmosis (RO), nanofiltration (NF), microfiltration (MF), and ultrafiltration (UF). Low pressure membranes such as MF and UF cannot reject PFAS since their pore sizes are larger than the effective diameter of the PFAS molecules (about 1 nm) (Tsai et al. 2010; Rahman et al. 2014). For that reason, MF and UF are not discussed here. Although bench-scale studies indicate that the membrane molecular weight cut-off (MWCO) of NF/RO is probably the most important factor for removal of PFAS for these technologies, other factors, such as ionic charge, may also influence performance.

Two terms, “salt passage” and “salt rejection,” generally describe how membrane systems perform. Salt passage is the percentage of dissolved constituents (contaminants) in the feedwater allowed to pass through the membrane. The opposite of salt passage, salt rejection, is the percentage of feed water that does not pass through the membrane. In

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general, NF membranes have lower rejection rates (95%) than RO (> 99%), because NF membranes have larger pores (Rahman et al. 2014 provides an excellent review of many of the relevant studies). As with all other treatment options, bench-scale and pilot-scale testing are required to understand the field applicability and establish essential detailed design criteria such as pretreatment needs and cost effectiveness.

3.4.1 Reverse Osmosis

Technology Description: RO membranes are effective in removing most organic and inorganic compounds from water solutions. In recent years, new polymer chemistry and manufacturing processes have improved efficiency, lowering operating pressures and reducing costs. As a result, RO membranes are increasingly used by industry to concentrate or remove chemicals. RO is commonly used around the world in household drinking water purification systems, the production of bottled mineral water, self-contained water purification units (for the U.S. military), and industrial applications (for example, water supply to cooling towers, boilers, and deionized water). The largest application of RO is in desalination.

Reverse osmosis is most useful as one element within a treatment train, where the reject water is further treated or captured.

Treatment Mechanism: RO separates compounds from water solutions by passing pressurized water across a semipermeable membrane. Treated water (permeate) passes through the membrane and the rejected water (concentrate) is collected for disposal or discharge, depending on the nature of the compounds and particles present.

State of Development: RO has been studied in bench-scale studies and pilot plants for wastewater and drinking water PFAS applications and is considered a partially developed technology. Conventional and advanced treatments have been studied in several pilot plants and drinking water treatment plants, demonstrating both treatments operating simultaneously as well as the effectiveness of traditional drinking and wastewater treatment methods alongside PFAS-specific technologies.

Effectiveness: Influent pretreatment is critical for RO membranes because of their spiral-wound design. Membranes are highly susceptible to fouling (loss of production capacity) because some accumulated material cannot be removed from the membrane surface.

RO removal of PFAS from various waters has been studied (for example, semiconductor wastewater, drinking water, surface water, and reclaimed water), combined with NF in some cases. PFOS removal > 99% was achieved using four different types of membranes over a wide range of feed concentrations, from 0.5 to 1500 mg/L (Tang et al. 2006). Another study tested five RO and three NF membranes at feed concentrations of 10 mg/L PFOS over four days (Tang et al. 2007). The PFOS rejection and permeate flux performances were > 99% for RO and 90 to 99% for NF. The use of RO and NF as advanced drinking water treatments is still limited, but both technologies have been shown to be successful for the removal of longer-chain (> C5) PFAAs (Loi-Brugger et al. 2008; Tang et al. 2006). Conventional and advanced treatment efficiencies to remove PFOA and PFOS from surface water of the Llobregat River in northeast Spain were also studied. Results were compared in several pilot plants, and in a drinking water treatment plant that operates with UF and RO treatment alongside traditional treatment processes (Flores et al. 2013).

Another study examined the fate of PFSAs and PFCAs in two water reclamation plants that further treat water from wastewater treatment plants in Australia (Thompson et al. 2011). Plant A used adsorption and filtration methods alongside ozonation, while Plant B used membrane processes and an advanced oxidation process, to produce purified recycled water. At both facilities, PFOS, perfluorohexane sulfonate (PFHxS), perfluorohexanoate (PFHxA), and PFOA were the most frequently detected PFAS. Comparing the two reclamation facilities, Plant A showed some removal during the adsorption/filtration stages. Overall, however, Plant A failed to completely remove PFOS and the PFCAs shorter than perfluorononanoic acid (PFNA) in chain length. All PFAS present were removed by RO at Plant B from the finished water to concentrations below detection and reporting limits (0.4–1.5 ng/L).

Sustainability Considerations: The carbon footprint for this technology includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. RO requires power for high-pressure pumps and the management of concentrate, which can be energy intensive. The contaminant-rich brine rejected by RO must be disposed of appropriately. The removal of nontarget minerals from the treated water may increase its corrosivity, and posttreatment corrosion control measures are needed in most cases. Resources are available for performing a sustainability assessment for membrane filtration remedial design (Ras and von Blottnitz 2012).

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3.4.2 Nanofiltration

Technology Description: NF is a form of membrane technology that is pressure-driven and shown to be effective in the removal of PFAS (Tang et al. 2007). This method provides high water flux at low operating pressure (Izadpanah and Javidnia 2012). Like RO, NF is easy to operate and reliable for the removal of chemicals.

Technology Mechanism: Nanometer-sized membrane pores are used to separate compounds in a process similar to RO, but NF does not remove smaller ions such as chloride and sodium.

State of Development: NF is considered a partially developed technology because available data on the removal of PFAS are limited to laboratory-scale tests performed on flat sheet membrane coupons.

Effectiveness: No studies have reported either pilot or full-scale performance of NF membranes. Therefore, variations in performance due to fouling, flux, and concentration distributions in standard spiral-wound membrane configurations have not been characterized.

NF membranes tested include the Dow membranes NF-270, NF-200, and NF-90 and the SUEZ (formerly GE Water & Process Technologies) DK membrane. Reported rejections were generally > 95% for PFAS with molecular weights ranging from 214 g/mol to 713 g/mol (Steinle-Darling and Reinhard 2008; Appleman et al. 2013). However, lower rejections were observed for perfluoropentanoate (PFPeA) and perfluorooctane sulfonamide (FOSA)—about 70 and 90%, respectively (Steinle-Darling and Reinhard 2008). Recovery and salt passage information was not reported in these studies. Scaling up these results is challenging because rejection is affected by recovery, which can vary from one application to another.

Salt passage for PFOS was reported to range from < 1% for the tighter NF-90 membrane to about 6% for the looser NF-270 and DK membranes (Tang et al. 2007). PFOS salt passage also was correlated to sodium chloride salt passage, a common specification for membrane manufacturers (Tang et al. 2007). Salt passage incorporates both rejection and membrane recovery, therefore is a more useful parameter for predicting full-scale performance than rejection.

Pilot and full-scale testing of the selected NF membrane for PFAS removal is a crucial step when considering this treatment process. Choosing membranes with MWCO smaller than the targeted PFAS is also a key design consideration.

Appropriate disposal or treatment of the membrane concentrate stream is another design factor, especially when using high-pressure membranes for inland communities. Furthermore, NF membrane fouling mechanisms are poorly understood and further research is needed to develop cost-effective cleaning methods to restore membrane performance (Al-Amoudi and Lovitt 2007). As with other forms of filtration, pretreatment strategies to avoid fouling and membrane fabrication drive performance results.

Sustainability Considerations: The carbon footprint for this technology includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. NF requires power for pumps (generally less than RO), and the management of concentrate, which can be energy intensive.

4 References and Acronyms

The references cited in this fact sheet, and the other ITRC PFAS fact sheets, are included in one combined list that is available on the ITRC web site. The combined acronyms list is also available on the ITRC web site.



Per- and Polyfluoroalkyl Substances (PFAS) Team Contacts

Robert Mueller • New Jersey Department of Environmental Protection
609-984-3910 • Bob.Mueller@dep.nj.gov

Virginia Yingling • Minnesota Department of Health
651-343-2890 • virginia.yingling@state.mn.us

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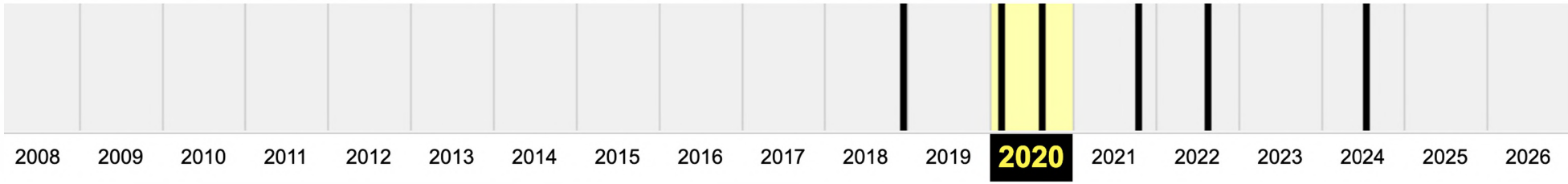
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An Introductory document (Spanish Version) has been prepared that briefly describes the contents of each of the fact sheets. An Introductory document has been prepared that briefly describes the contents of each of the fact sheets. This web site also includes a combined references list and acronyms list.

- Naming Conventions and Physical and Chemical Properties (updated 3-16-18)
- Naming Conventions and Physical and Chemical Properties (Spanish Version)
- Regulations, Guidance, and Advisories (updated 1-4-18)
    - Section 4 Tables Excel file – (updated February 2020)
        - Table 4-1 presents the available PFAS water values established by the USEPA, each pertinent state, or country (Australia, Canada and Western European countries)
        - Table 4-2 presents the available PFAS soil values established by the USEPA, each pertinent state, or country (Australia, Canada and Western European countries)
    - Section 5 Tables Excel file (updated January 2019)
        - Table 5-1 summarizes the differences in the PFOA values for drinking water in the United States.
        - Table 5-2 summarizes the differences in the PFOS values for drinking water in the United States.
- Regulación, Orientación, y Asesoramiento para sustancias Per- y Polifluroalquiladas (PFAS) (Spanish Version)
- History and Use (published 11-13-17)
- Historia y Uso (Spanish Version)
- Environmental Fate and Transport (published 3-16-18)
    - Table 3-1 Log Koc values for select PFAS Excel file (published April 2018)

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- Table 3-1 Log Koc values for select PFAS Excel file (published April 2018)
- Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods (published 3-15-18)
- Remediation Technologies and Methods (published 3-15-18)
  - Remediation Comparison Tables Excel file (published April 2018)
    - Table 1 – Solids Comparison
    - Table 2 – Liquids Comparison
- Aqueous Film-Forming Foam (published October 2018)

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Remediation Technologies and Methods for Per- and Polyfluoroalkyl Substances (PFAS)

1 Introduction

Remediation technologies exploit chemical and physical properties to immobilize, remove, or destroy the targeted contaminants. Certain PFAS have recently been the subject of regulatory actions and attempted soil, sediment, and water remediation. These compounds have unique chemical properties that require new remediation technologies or innovative combinations of existing technologies. The decision to remediate PFAS should be driven by applicable regulations and an appropriate risk assessment.

USEPA has compiled an online resource for PFAS that includes topics such as policy and guidance, chemistry and behavior, occurrence, toxicology, site characterization, and remediation technologies (USEPA 2017h). The National Groundwater Association (NGWA) has also published a resource on PFAS that includes information about remediation technologies (NGWA 2017).

ITRC has developed a series of fact sheets that summarize the latest science and emerging technologies related to PFAS. The purpose of this remediation fact sheet is to:

- provide an overview of remedial technologies and methods for treatment of solids (for instance, soil or sediment) and liquids (for instance, groundwater, leachate, or surface water);
- describe processes for the treatment of PFAS that are now in use or are under development; and

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- describe processes for the treatment of PFAS that are now in use or are under development; and
- describe the challenges and limitations for each treatment technology.

1.1 PFAS Remediation Technologies Overview

Currently, full-scale PFAS treatment in water is limited to sorption using carbon, mineral media (for example, clay), or a combination of these. Additional pilot and bench-scale technologies are currently being tested. This fact sheet discusses the treatment technologies that have been successfully demonstrated through pilot testing at the field-scale. The accompanying tables summarize technologies that have only been tested in limited applications or in laboratory bench tests. Combining technologies may overcome limitations of any one given technology or expand the efficacy of each technology.

For each technology presented, this fact sheet discusses the following key elements:

- *Treatment Description* (according to each applicable media)
- *Treatment Mechanism* (for example, separation, sorption)
- *State of Development* – Is the technology at lab or bench-scale, field pilot-scale, or full-scale implementation? How many demonstration tests and what is the degree of commercialization for each technology?
- *Effectiveness* – Summary of demonstrated effectiveness on a broad range of PFAS (for example, Method 537 (Shoemaker, Grimmett and Boutin 2009) suite plus fluorotelomer sulfonates), a limited subset (for example, Third Unregulated Contaminant Monitoring Rule [UCMR3] list USEPA 2017f), or only perfluorooctane sulfonate (PFOS) and/or perfluorooctanoate (PFOA).
- *Sustainability Considerations* – Design considerations are evaluated including green remediation elements such as carbon footprint from energy usage, treatment media, and residual handling transport, as well as potential community impacts.

The current state of full-scale PFAS treatment in water is limited to sorption using carbon and/or mineral media (for instance, clay).

Two supporting tables comparing PFAS remediation technologies are available in a separate Excel file: Table 1, Solids Comparison, and Table 2, Liquids Comparison. The tables present all reported treatment technologies, including those tested only at the laboratory/bench scale. To be included in the tables, a technology must have been documented in a publicly available document. Some technologies, however, are only documented in literature supplied by the inventor/researcher/vendor of that technology, with no independent confirmation or peer-review process—be aware of possible biases. ITRC periodically updates the supporting tables and maintains the most current version on its website.

Air treatment is not included in this fact sheet because the current research is limited, and this topic is less applicable to site remediation projects.

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1.2 Factors affecting remedy selection

Site characteristics that affect PFAS remedy selection include the nature of the source, release pathways, affected receptors, and fate and transport in the environment (see *Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods; Environmental Fate and Transport;* and *Naming Conventions and Physical and Chemical Properties* fact sheets). Other site characteristics relevant to remedy selection may be identified as remedial technologies advance. Strategies for remediation of the broader class of PFAS may require complementary technologies, several of which are still being developed, as described in Tables 1 and 2.

Other factors affecting PFAS remedy selection include:

- **Characteristics of PFAS**. The wide-ranging chemical and physical characteristics of PFAS affect the remedy effectiveness. Key factors include ionic state (anionic, cationic, and zwitterionic), types of ionic groups (sulfonate or carboxylate), lipo- and hydrophobicity, nature and reactivity of alkyl groups, chain length and branching, partitioning coefficients, volatility, solubility, and acidity.
- **Changes in PFAS properties**. Chemical and physical properties resulting from naturally occurring processes or due to remedial actions for other (commingled) contaminants, such as chlorinated solvents, and petroleum hydrocarbons, can affect PFAS distribution and mobility in groundwater (McGuire et al. 2014). Example changes include:
  - The alkyl functional group of some PFAS may be more readily subject to chemical or biological transformation than the fully fluorinated aliphatic chain. This is the basis, for example, for the total oxidizable precursor (TOP) assay to estimate concentrations of precursor compounds (Houtz and Sedlak 2012).
  - Partial degradation of the carbon-carbon bonds in the aliphatic chain reported for some chemical remedies generates short-chain PFAS, which may be more mobile (Guelfo and Higgins 2013).
  - Modifications in aquifer properties (for example, redox, pH, or other geochemical characteristics) during remediation of comingled contaminants results in a conversion of PFAS to the more stable and mobile perfluorocarboxylic acids (PFCAs) (McKenzie et al. 2015, 2016).
- **Community acceptance**. Communities are often faced with trade-offs in terms of cost, level of clean-up, and residual contamination as part of remediation efforts. Stakeholder engagement and effective risk communication strategies are an important aspect of the overall remedial technology selection process, especially where off-site receptors are identified.

Factors specifically challenging for PFAS remediation include:

- Multiple ionic states
- Variable isomers
- Differing alkyl groups
- Past remediation effects
- Common co-contaminants

1.3 Technical Maturity

The treatment technologies described here are organized by degree of current confidence in the technology. Three levels of confidence are defined as follows:

- *Demonstrated Technologies*—Technologies that have been demonstrated under pilot or full-scale conditions and are well documented for multiple applications in peer-reviewed literature. These technologies are discussed in greatest detail.
- *Partially Demonstrated Technologies*—Technologies that have been documented in peer-reviewed literature by multiple researchers or practitioners but have only been executed at the laboratory or bench scale. These technologies are briefly mentioned.
- *Promising Technologies*—Technologies that have been demonstrated at the laboratory or field pilot-scale, but the results have not been rigorously peer reviewed. Often, these results are only reported by one group (for example, one university, practitioner, or vendor) or lack detailed validation of the treatment or mechanisms. These technologies are omitted from the text and presented only in the technology comparison tables (Tables 1 and 2).

Experimental techniques to treat PFAS are under development, but only those technologies that have some level of publicly available documentation demonstrating effectiveness are included here.

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2 Technologies for Treatment of Solids

Several technologies are currently available to remediate PFAS in solids. Most of these technologies have been demonstrated on soils only but may also apply to saturated sediments or sludge treatment.

Field-Demonstrated Treatment Technologies for PFAS in Solids

- Excavation and off-site landfilling or incineration
- Sorption/stabilization through ex situ soil mixing
- Ex situ thermal desorption and off-gas destruction

2.1 Capping

Technology Description: Capping places a cover over contaminated material such as landfill waste, contaminated soil, and sediments. Caps do not destroy or remove contaminants. The purpose of the cap is to prevent contact with the contamination and, depending on the design, to reduce or prevent further leaching of contaminants. For PFAS soil sites, the main purpose of a cap is to reduce or prevent further leaching of contaminants from soils to groundwater.

Treatment Mechanism: Caps isolate contaminants and prevent them from spreading and causing exposure by direct contact.

State of Development: Capping is a viable remediation method because it is simple technology that applies to most contaminants. Although capping has not been applied to a PFAS site, based on documented successes with other contaminants it offers promise for PFAS.

Effectiveness: Capping may prevent exposure and potentially reduce infiltration, but redevelopment options for the capped land surface may be limited. Caps are most effective when the seasonal high-water table is well separated vertically from the base of the contaminated solids. The PFAS contamination will remain at the site and therefore be a long-term liability. Contaminants could mobilize if site conditions change (for example, rising water table). Caps incur maintenance costs to maintain the integrity of the cap and require institutional controls to ensure the cap is not breached by future site uses or redevelopment.

Sustainability Considerations: The carbon footprint of capping includes earthwork equipment emissions and manufacturing and transporting capping material. Ecosystem restoration can be incorporated into the capping design (Lamb et al. 2014). Community impacts include truck hauling traffic and hindrance of redevelopment due to land use restrictions.

2.2 Excavation and Disposal

Technology Description: This remediation method includes removing contaminated soil and hauling it to a permitted landfill or incineration facility, then filling the excavated area with clean backfill.

Treatment Mechanism: This method isolates PFAS from receptors. Sometimes, PFAS-impacted soil is stabilized or solidified before disposal into a landfill. Disposal of PFAS-impacted soils or wastes into unlined landfills should be avoided. More studies are needed on interactions of PFAS with landfill linings.

Some states may require PFAS-contaminated solids to be treated at a Resource Conservation and Recovery Act (RCRA) permitted incinerator. The estimated temperature to destroy specific PFAS varies widely, from around 300° to greater than 1,000°C (Vecitis et al. 2008). Commercial incineration is often the only viable disposal option for media contaminated with PFAS.

State of Development: Solid wastes generated from environmental investigation and cleanup (for example, soil excavation, and investigation- and remediation-derived wastes) have been disposed at lined landfills, and therefore this is considered a well-demonstrated technology. PFAS have been reported in landfill leachate (Lang et al. 2017), although it is unlikely the source for these PFAS is investigation derived waste, as opposed to consumer product waste containing fluorochemicals. The combination of soil excavation and incineration is also considered to be a viable remedial option.

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Effectiveness: Excavation and disposal of PFAS-contaminated soil effectively removes a source area that may otherwise serve as a continuing source of groundwater contamination but does not result in destruction of the PFAS. Additionally, some nonhazardous waste landfills do not accept PFAS waste. Disposal of PFAS waste to landfills potentially adds to the PFAS contaminant load in the landfill leachate. In some states, the leachate is not analyzed or regulated for PFAS.

Sustainability Considerations: The carbon footprint for this approach includes earthwork equipment emissions, transporting contaminated soil and backfill, and manufacturing (such as resource extraction) of backfill material. Landfill incineration of the contaminated soil is energy intensive. Truck hauling traffic affects the local community. Guidance is available for performing a sustainability assessment for an excavation and disposal remedial design (Cappuyns and Kessen 2014; Goldenberg and Reddy 2014; Söderqvist et al. 2015; Song et al. 2018).

2.3 Sorption and Stabilization

Technology Description: Amendments added to the soil reduce or remove the potential for PFAS to mobilize from soil to groundwater.

A few commercially available sorbents have been specifically developed to immobilize PFAS in soil.

Treatment Mechanism: Amendments adsorb or stabilize PFAS. These amendments include activated carbon and carbon nanotubes (CNTs), resins, minerals, biomaterials, and molecularly imprinted polymers. The amendments bind to PFAS and thus reduce their release from soil.

State of Development: Sorption and stabilization techniques using carbon-based amendments are considered partially demonstrated technologies. Both granular activated carbon (GAC) and powdered activated carbon (PAC) rapidly remove PFAS from groundwater and surface water and can be applied to soil, but efficacy is reduced in the presence of organic co-contaminants (NGWA 2017). Performance also depends on PFAS chain length and functional group (Xiao et al. 2017). Laboratory tests should be conducted using site specific soils and the stabilizing amendment before field application to ensure success. Carbon amendments can be modified to enhance their sorption of PFAS. One patented amendment is carbon enhanced with aluminum hydroxide, kaolin clay, and other proprietary sorbents (USEPA 2017p).

Sorption and stabilization techniques using non-carbon-based sorbents, such as iron oxide minerals and modified organoclays (such as montmorillonite [Mt], hydrotalcite, and palygorskite) are promising, but only limited tests have been conducted. Minerals such as clays, silica, iron oxides, and zeolites have been used as sorbents for removing contaminants from groundwater and soil (Zhu et al. 2016; Rattanaoudom, Visvanathan, and Boontanon 2012; Zhou et al. 2010; Zhou, Pan, and Zhang 2013). The surface of organoclays can also be modified for enhanced PFOS and PFOA sorption (Zhou et al. 2010; Kambala and Maidu 2013; Zhu et al. 2016). Additionally, an amine-modified palygorskite clay sorbent has been patented for the treatment of PFOS and PFOA (Kambala and Maidu 2013).

Effectiveness: Carbon- and mineral-based sorption and stabilization techniques vary in their effectiveness according to site conditions and PFAS type. An example of a site condition that can affect sorption is high organic matter in soil, which can foul carbon sorbents with competing compounds. PFAS type affects sorption in that PFAS often occur as mixtures, including PFAS of different chain lengths with varying sorption characteristics. Sorption capacity was assessed in one study where CNTs were mixed with sediments at 4% weight per weight (w/w), and the sorption capacity of the sediment increased in comparison to untreated sediment (Kwadijk, Valzeboer, and Koelmans 2013).

Organoclays are used because they are environmentally benign, have a high sorption capacity, and can be easily modified to enhance their sorption capacity with mesopores. Organoclays have also been proven to work on several classes of contaminants (Zhu et al. 2016; Espana, Mallavarapu, and Naidu 2015; Zhou et al. 2008). The surface of organoclays are hydrophilic, and therefore ineffective for sorption of hydrophobic organic compounds like long-chain PFAS; however, modification with cations changes the surface to lipophilic. For example, Mt often exists as Na-Mt with a sodium cation on the surface, which is lipophilic and may be effective for long-chain PFAS.

Biomaterials such as chitosan, straw, and quarternized cotton do not perform as well as other sorbents, and the biomaterials may eventually degrade (Du et al. 2014).

Sorption and stabilization do not destroy PFAS, and information on the long-term stability of amendments for PFAS remediation is a data gap that currently limits their use. The amended soil can be mixed with concrete and other stabilizers to better trap the PFAS.

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Sustainability Considerations: The carbon footprint for sorption and stabilization includes emissions from earthwork equipment, manufacturing (for example, resource extraction), and transporting amendment material. Community impacts include hindrance of redevelopment due to land use restrictions. Resources are available for performing a sustainability assessment for sorption and stabilization remedial design (Goldenberg and Reddy 2014; Hou et al. 2016; Kuykendall and McMullan 2014).

2.4 Thermal

Technology Description: The mobilizing or destruction of chemicals using heat.

Treatment Mechanism: Heat is applied directly to the PFAS-contaminated soil. High temperatures can vaporize the chemicals or potentially destroy them. Vaporized chemicals can be captured and destroyed in off-gas treatment.

State of Development: Ex situ thermal treatment has been demonstrated at the field pilot-scale by a few technology vendors and is considered a partially demonstrated technology (Endpoint Consulting 2016; Enviropacific 2017). The use of this technology for PFAS-contaminated soil is still developing. Limited data sets are available, and several data gaps still exist. Additionally, no documented examples of in situ thermal treatment for PFAS-impacted soil have been identified.

Effectiveness: Results of one test indicated that the complete removal of a suite of nine PFAS was possible within 30 minutes but required temperatures over 900º C (Endpoint Consulting 2016). In another field pilot project, concentrations of 20 PFAS in soil were reduced to below reporting limits—greater than 99.9% reduction (Enviropacific 2017). At this time, it is unknown whether the pilot test resulted in volatilization or complete destruction. This test was performed at relatively lower temperatures (for example, approximately 450º C). There are still several data gaps related to thermal incineration of PFAS that should be considered when applying this technology. For example, the mass balance to assess whether PFAS are destroyed or simply mobilized is not completely understood.

Sustainability Considerations: Thermal treatment is an energy-intensive remediation method. Its carbon footprint includes the energy source and consumption during treatment system operation, as well as manufacturing and installation of heating system materials. Community impacts include managing the risks of potential vapor intrusion from volatile co-contaminants. Guidance is available for performing a sustainability assessment for thermal remedial design (Song et al. 2018; Vidonish et al. 2016).

3 Liquids Treatment

Several technologies are currently available for remediating PFAS in liquids. These technologies can be applied to drinking water supplies, groundwater, industrial wastewater, surface water, and other miscellaneous applications (such as landfill leachate). Influent concentrations of PFAS can vary by orders of magnitude for specific media or applications. These influent values, along with other general water quality parameters (for example, pH) can influence the performance and operating costs for the treatment technologies.

Field Demonstrated Treatment Technologies for Liquids

- Extraction and sorption with granular activated carbon or anion exchange resin
- Extraction and membrane filtration/reverse osmosis
- Extraction and precipitation/flocculation

3.1 Sorption

Many organic compounds can be treated by passing contaminated water through special granular media. The mechanism of this technology varies depending on the media, contaminant, and influent concentrations. There are two broad categories of PFAS sorption treatment: adsorption onto carbon media and ion exchange.

3.1.1 Granular Activated Carbon

Technology Description: Granular activated carbon (GAC) is made from organic materials, such as coal and coconut, which serve as effective adsorbent media because they are highly porous and provide a large surface area for contaminant contact. GAC treatment can be used for any aqueous-based treatment application (for example,

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municipal drinking water, groundwater, point-of-use residential, industrial wastewater, and landfill leachate). The GAC media is placed in packed-bed, flow-through vessels generally operated in series (lead-lag configuration). Either virgin or reactivated GAC can be used for most applications, but virgin GAC is the industry preference for more risk-averse drinking water applications. Commercial facilities in the United States conduct thermal reactivation of spent GAC, which can provide a more sustainable and less costly replacement option than virgin GAC and off-site disposal. To address concerns about using reactivated GAC, vendor bench-scale column studies can be performed prior to initial usage, and a quality control testing program can be implemented prior to delivery of each shipment.

Treatment Mechanism: Removal of PFAS from treated water by GAC is an adsorption process, as well as a physical mass transfer process from the aqueous phase onto solid media that does not involve any form of chemical degradation or transformation. Adsorption is a surface chemistry phenomenon by which an aqueous phase contaminant adheres to the surface of a granular media (via electrical, physical, or chemical processes), but does not penetrate it. The GAC adsorption capacity can vary considerably by media and contaminant. Adsorbent media must be removed and replaced when it becomes spent, meaning contaminants break through at concentrations above some established criteria. The spent media must be replaced and shipped off-site either for disposal (by landfilling or commercial incineration) or to be regenerated/reactivated for reuse consistent with applicable federal and state regulations.

State of Development: GAC is a demonstrated technology and is currently the most common water treatment method used for PFAS. Because of the limited treatment history and available technologies for PFAS, full-scale applications to date have mostly focused on higher priority private and public water supply and residential point-of-use treatment. Currently, only a few operating groundwater pump-and-treat applications use GAC treatment. Much of the published literature on GAC treatment of PFAS involves bench-scale and vendor column studies. Treatability data for full-scale operations involving different technologies, including GAC, have also been compiled and reported for several municipal wastewater treatment plants where PFAS have been detected in the influent.

Many sources in the literature support the use of GAC: Appleman et al. (2013); Szabo et al. (2017); and Woodard, Berry, and Newman (2017); and others cited in this section. These references also include more comprehensive bibliographies if further details are needed on specific topics or studies.

Effectiveness: GAC has been shown to reduce select PFAS to very low or nondetectable concentrations, on the order of nanograms per liter (equivalent to parts per trillion), with reported removal efficiencies in various references between 90% and >99%. The lower end of these reported GAC removal efficiencies may be the result of the faster breakthrough times for the short-chain PFAS (Xiao et al. 2017; Dickenson and Higgins 2016). Early municipal treatment plant sampling studies focused mostly on PFOA and PFOS, which are considered long-chain PFAS (defined as six carbon atoms or more) and currently drive most drinking water treatment decisions. Consequently, the shorter breakthrough times for PFAS with five carbon atoms or less were initially missed, and subsequently reported in the literature as lower GAC removal efficiencies.

Breakthrough and GAC usage should be checked for a variety of PFAS, not just PFOA or PFOS, including various chain-lengths.

Individual PFAS have different GAC usage capacities and corresponding breakthrough times. GAC removal capacity for PFOS is greater than PFOA, but both can be effectively captured. In general, PFAS containing five carbon atoms or less have higher GAC usage and much quicker breakthrough times than PFAS containing six carbon atoms or more with other factors being equal (such as influent concentration). Vendor column studies (Brewer 2017) with equivalent influent concentrations and empty contact bed times have shown that short-chain PFAS breakthrough times are approximately five times quicker than long-chain PFAS. Pilot and full-scale GAC treatment data show similar comparative breakthrough times (Brewer 2017).

In addition to usage capacity, several other factors affect GAC change-out frequency and cost for individual PFAS (for example, influent concentrations). Change-out frequency therefore cannot be predicted solely by the presence of specific short-chain PFAS. When concentrations of short-chain PFAS are much lower than concentrations of long-chain PFAS, GAC is still a cost-effective treatment for PFAS other than PFOA and PFOS. Because of the differences in GAC usage capacities between individual PFAS, treatability studies must evaluate the entire mixture of PFAS present in the influent to the extent practicable. Column studies are the best method to predict GAC performance and change-out frequency.

Column studies show that virgin GAC and thermally reactivated GAC have similar removal rates and breakthrough times (Brewer 2017). Based on vendor feedback (Mimna 2017), commercial thermal reactivation is performed at higher

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operating temperatures than steam or nitrogen regeneration systems and is capable of complete desorption and destruction of PFAS from spent GAC (Watanabe et al. 2016; Yamada et al. 2005). Also, vendor testing demonstrates that re-agglomerated bituminous coal provides better removal performance for PFAS than other types of GAC (Brewer 2017; Nowack 2017).

Sustainability Considerations: The carbon footprint for GAC includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. Spent single-use media requires incineration, which increase the carbon footprint. Regenerable media presents sustainability benefits because the media is reused; however, in drinking water applications, virgin material achieves greater confidence in treatment. Resources are available for performing a sustainability assessment for sorption remedial design (Amini et al. 2015: Choe et al. 2013, 2015; Dominguez-Ramos et al. 2014; Favara et al. 2016; Maul et al. 2014; Rahman et al. 2014; Ras and von Blottnitz 2012).

3.1.2 Biochar

Technical Description: *Biochar* is a hybrid word derived from *biomass* and *charcoal*. Biochar is a carbon-rich, porous solid synthesized from biomass, such as wood or manure, through a high-temperature low-oxygen process called "pyrolysis" (Ahmad et al. 2014). Key factors controlling the properties of biochar (for example, pore size, chemical composition, and hydrophobicity) include the temperature of pyrolysis and biomass feedstock.

Treatment Mechanism: The properties of biochar are comparable to those of GAC for sorptive purposes. Like GAC, biochar can adsorb organic contaminants.

State of Development: Biochar is considered a partially demonstrated technology. Various laboratory experiments have evaluated the efficacy of biochar compared to other media, but no full-scale treatment systems are in place for the removal of PFAS. This work demonstrates that biochar is potentially viable for treatment of PFAS, but additional research is needed to fully establish viability and costs.

Effectiveness: Xiao et al. (2017) compared one GAC and two commercially available biochars for treating an aqueous film-forming foam (AFFF)-impacted water supply. Based on batch studies, they concluded that biochars with large surface areas could be an alternative to GAC, although variability in biochar properties relative to GAC may affect reliability. While biochar removal is effective in ultrapure water, when used to treat river water (with more complicated water chemistry), biochar is ineffective compared to ion exchange and GAC and exhibited significantly slower adsorption kinetics (Rahman 2014).

Sustainability Considerations: The carbon footprint of this technology includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. The use of waste material as a starting feedstock results in a lower overall carbon footprint, though reactivation of biochar is not currently feasible and energy-intensive incineration or landfilling are required, which offsets some of the sustainability benefits.

3.1.3 Ion Exchange

Technology Description: Ion exchange (IX) uses synthetic, polymeric media to remove PFAS from water. IX media are employed similarly to GAC and can be used in combination with GAC.

Both regenerable and nonregenerable IX media are available. Nonregenerable IX is a single-use, disposable medium. Regenerable IX theoretically can be used indefinitely, however, insufficient operational data are available to understand its long-term durability. IX regeneration is a chemical process; the only demonstrated successful regeneration solution is a solvent-brine solution (Amec Foster Wheeler 2017). The regenerant solution can be distilled for reuse. The distillate residue is a concentrated PFAS waste that can be managed by off-site treatment (for example, incineration or possibly chemical oxidation).

Treatment Mechanism: IX is the process by which ions of one substance are replaced by similarly charged ions of another substance. The term denotes purification, separation, and decontamination of aqueous and other ion-containing solutions with solid polymeric or mineralic ion exchangers. Many organic contaminants are ionic and can be removed through specialized ion exchange media. The media are often derived from organic polymers or plastic, and thus ion exchange media are referred to as "resins." Regeneration of ion exchange resins is accomplished with a chemical flush, typically a highly acidic or basic solution, brine solution, or solvent-brine solution, rendering the resin reusable.

To date, IX has used positively charged, anion exchange media to remove negatively charged PFAS molecules, via binding of the carboxylic and sulfonic acid "heads" of perfluoroalkyl acids (PFAAs). Such IX media are manufactured to

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be more selective for PFAS than for typical competing mineral anions such as sulfate and nitrate. The fluorinated carbon chain of the PFAS molecule can also adsorb to IX media. This dual-mechanism, ion exchange plus adsorption, can result in higher removal compared to adsorption alone (Yu et al. 2009).

State of Development: IX is a fully demonstrated technology. Column tests comparing both regenerable and single-use ion exchange media have shown IX to be effective for the removal of several PFAS (Woodard, Berry, and Newman 2017; Conte et al. 2015). Full-scale IX systems are currently in operation in Australia at Australian Defense sites, and a full-scale system was installed in the United States in 2017. The U.S. Department of Defense (DOD) recently funded a SERDP project, ER18-C2-1306: "Combined In Situ/Ex Situ Treatment Train for Remediation of Per- and Polyfluoroalkyl Substance (PFAS) Contaminated Groundwater" to optimize regenerable IX and on-site destruction with plasma under a number of laboratory conditions (SERDP-ESTCP 2017).

Effectiveness: IX is a demonstrated effective technology for removal of anionic PFAS. It has higher adsorption capacity for some PFAS and significantly faster reaction kinetics compared to GAC (Conte et al. 2015). The combination of these properties means an equivalent treatment system for IX is smaller and thus uses less media. Like GAC, usage capacities and corresponding breakthrough times vary depending on PFAS functional groups and chain length. Short chain PFAS may break through faster under certain influent conditions. However, certain single-use IX media have been identified that may have higher usage capacities for short-chain PFAS. An IX treatment system can also include multiple vessels in a lead-lag configuration, consisting of both single-use and regenerable IX media. This design optimizes removal properties and operating costs for a mixture of short and long-chain length PFAS.

Sustainability Considerations: The carbon footprint for IX includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. Spent single-use IX media requires incineration or other treatment for disposal, resulting in an increased carbon footprint. Regenerable IX media offers sustainability benefits because the media can be reused, but the process for regenerating requires energy and creates a concentrated waste stream which must be managed. Disposal or treatment of the regenerant stream can be problematic and expensive. Regeneration solutions may present exchange system and treated water corrosion issues if media are not rinsed thoroughly prior to being placed back in service.

3.2 Precipitation/Flocculation/Coagulation

Technology Description: Coagulation–flocculation is a common pretreatment approach used in wastewater treatment plants for removing various particles and dissolved constituents. Coagulants, either commodity or proprietary chemicals, can be added to water (conventional technology) or generated by anode-cathode reactions of metals plates inserted into the water (electrocoagulation).

Technology Mechanism: Coagulants assist in forming solids. Flocculation is typically conducted by adding a soluble polymer and slowly mixing to allow the particles to agglomerate and grow. Upon solid formation, constituents such as PFAS can be physically incorporated into, or sorbed onto, the flocculated particulate (which is known as co-precipitation). The precipitated solids are then separated from the water by sedimentation, filtration, or a combination of both processes. The solid material containing the PFAS requires disposal.

State of Development: Current literature only documents bench-scale study results on treating PFAS via precipitation, flocculation, or coagulation and therefore this is considered a partially developed technology. Evaluations have focused on conventional commodity chemical coagulation (for example, aluminum or ferric salts) and nonconventional coagulation (for example, proprietary chemical coagulants or electrocoagulation). Pilot and full-scale applications have not been documented in the United States (Birk and Alden 2017).

Unconventional precipitation (for example, electrocoagulation or advanced chemical precipitants) has shown more potential for direct PFAS treatment, but little data is available.

Effectiveness: Electrocoagulation reactors, which range from basic to sophisticated designs, are highly efficient, compact, relatively low-cost, and completely automatable (Baudequin et al. 2011; Lin et al. 2015a). Recent studies have found that PFAAs, such as PFOA and PFOS, can be quickly sorbed on the surface of zinc hydroxide particulates generated by electrocoagulation (Lin et al. 2015a).

One commercially available proprietary chemical product has been shown in tests to reduce PFAS in groundwater containing AFFF (Birk and Alden 2017; CH2M 2017). This research also shows that conventional (ferric salt) and proprietary chemicals in combination are more effective than either alone. Current data have been developed at elevated

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concentrations, but removal for lower (µg/L) concentrations has also been shown (CH2M 2017; Birk 2015). No available data show precipitation effectiveness at very low (ng/L) concentration ranges.

Conventional PFAS precipitation induced by coagulation and flocculation has shown limited applicability for complete treatment of PFAS. Therefore, conventional chemical precipitation could be considered mainly as a pretreatment technology to sorb or precipitate PFAS prior to final filtration or destruction. Also, if used to remove conventional parameters such as solids or natural organic material (NOM), pretreatment would allow PFAS removal technologies to achieve the desired treatment goals in a more cost-effective and technically feasible manner.

Sustainability Considerations: The carbon footprint of this technology includes energy source and consumption during treatment system operation, as well as manufacturing of treatment media. Additionally, extracted solids containing PFAS require final disposal or destruction.

3.3 Redox Manipulation

Technology Description: Redox manipulation is the process of changing the oxidation-reduction potential of water through addition of oxidizing or reducing amendments, or by adding energy to a system to create oxidizing or reducing free radicals. These changes affect the mobility or structure (transformation or destruction) of the PFAS. PFCAs are generally more amenable to redox manipulation than perfluoroalkane sulfonic acids (PFSAs). Redox manipulation may be effective at treating many co-contaminants and may also alter organic matter, which can affect PFAS mobility. These system changes may allow subsequent treatment steps to manage target PFAS more effectively.

Treatment Mechanisms: Redox manipulation mechanisms may include both redox transformation (oxidative, reductive, and nucleophilic processes) of PFAS and changes in redox conditions in the impacted media (for example, groundwater), resulting in changes in the mobility and sorption of PFAS (McKenzie et al. 2015; Arvaniti et al. 2015). Redox transformation involves the transfer of electrons between reactants. In oxidative processes, electrons are transferred to the reactive species (the oxidant) from the target (PFAS), whereas in reductive processes the opposite occurs. In nucleophilic processes, a reactant (the nucleophile) bonds with the PFAS compound and displays an atom or group of atoms from the PFAS molecule.

Susceptibility to redox transformation depends on reaction conditions and the reactive species involved. For some specific technologies, more than one transformation mechanism or reactive species may be involved. For example, plasma, sonolytic, and photolytic technologies may combine physical (high-temperature pyrolysis) and free radical attack processes. PFAS carbon chains do not easily transform, because their carbon-carbon bonds are shielded in part by the tightly bound fluorine atoms that surround the carbon chain (Kissa 2001). The carboxylic or sulfonic group "heads" of PFAS are commonly more susceptible to redox transformation than carbon chain "tails," resulting in partial transformation of the parent compound but not in cleavage of the aliphatic chain (Houtz and Sedlak 2012; Anumol et al. 2016). The tail and head structure are illustrated for PFOS and PFOA in the following figure.

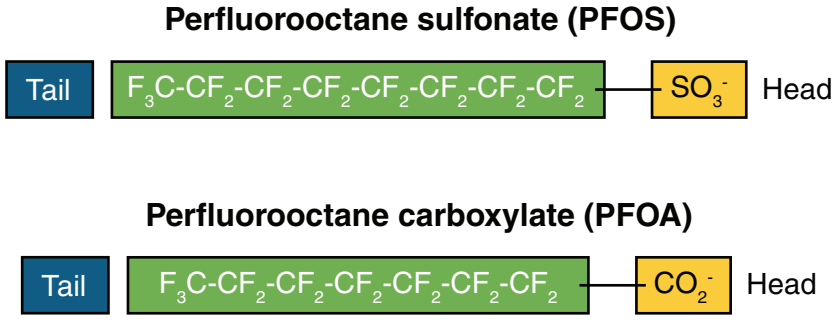


Figure 1. The tail and head structure of PFOS and PFOA molecules.

Reactions of zero valent metals with PFAS, generally considered a reductive process, may also affect mobility and sorption of PFAS in addition to (or rather than) PFAS transformation (Arvaniti et al. 2015). Complete mineralization/defluorination of PFAS via zero valent metal reactions has not been demonstrated to date; however, research in this area is ongoing.

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Combined redox and nucleophilic attack almost completely mineralize PFOA (Niu et al. 2012; Mitchell et al. 2014). A reactant formed in several redox manipulation approaches, the solvated electron (free electron in solution), shows promise for PFAS destruction (Park et al. 2009; Gu et al. 2016; Li et al. 2012; Stratton et al. 2017; Blotevogel, Giraud, and Borch 2018).

State of Development: Redox technologies have not been widely applied beyond laboratory bench-scale tests because of concerns that partial transformation (as opposed to complete destruction) will produce more mobile or toxic products. Therefore, redox technologies are considered a partially developed technology, with both successes and challenges. For example, chemical oxidation affects PFAA transport, but the direction (increased or decreased transport) and magnitude depend on reaction conditions (McKenzie et al. 2015). Apparent in situ destruction of PFAAs in groundwater at a fire training site has also been achieved using a combined ozone/persulfate approach (Eberle, Ball, and Boving 2017). The main difficulty in using redox-based technologies is achieving extremely low (ng/L) PFAS cleanup objectives, particularly for in situ remedies. Additionally, reactive species (for example, oxidizing radicals) interact with other compounds present in soil and groundwater that are more susceptible to oxidation and are at relatively higher concentrations than the target PFAS.

Despite these challenges, several options show promise for ex situ treatment, including electrochemical (Schaefer et al. 2015; Urtiaga et al. 2015), sonolytic (Vecitis et al. 2008; Rodriguez-Freire et al. 2015), plasma (Stratton et al. 2017), and reductive (Arvaniti et al. 2015) technologies. These approaches have successfully degraded an array of high-concentration PFAS at the laboratory scale. However, none of these technologies are sufficiently mature yet to assess PFAS treatment costs and overall effectiveness with confidence.

Effectiveness: A wide range of PFAS treatment methods based upon oxidation-reduction chemistries have been evaluated. These studies demonstrate the following:

- PFAS exhibit a wide range of reactivity toward destructive processes, based upon characteristics including chain length, nature of alkyl groups, and branched versus linear isomers.
- Treatment of PFAS at AFFF sites depends on the composition and production processes of the given AFFF formulation used at the site.
- Except for limited field pilot testing of one ozone and one persulfate-based chemical oxidation technology, none of the technologies summarized in Table 2 have matured beyond laboratory-scale studies.
- While promising, the relatively few field pilot applications have not yet been closely analyzed or duplicated in highly controlled and monitored studies.

Partial transformation can affect other physical and chemical characteristics of PFAS (see Section 1.2). For instance, persulfate-based transformation of PFAS may be incomplete (Houtz and Sedlak 2012) or negligible (McKenzie et al. 2015, 2016), whereas in other cases efficient transformation is reported (Hori et al. 2008; Lee et al. 2009; Liu et al. 2012; Park et al. 2016; Yin et al. 2016). Redox processes in the subsurface can generate unwanted byproducts (for instance, nitrate and bromate from chemical oxidation) depending on site and reaction conditions (Siegrist, Crimi, and Simpkin 2011). Site-specific treatability tests are recommended.

Sustainability Considerations: The carbon footprint for this technology includes energy source and consumption during treatment system operation, manufacturing of amendment materials, and manufacturing and installation of injection points (if implemented in situ). Electrochemical treatment options also require handling of hazardous investigation derived waste, which is a sustainability consideration.

3.4 Membrane Filtration

Membrane filtration refers to a variety of separation technologies based on the nominal size of the membrane pores. Types of membranes include reverse osmosis (RO), nanofiltration (NF), microfiltration (MF), and ultrafiltration (UF). Low pressure membranes such as MF and UF cannot reject PFAS since their pore sizes are larger than the effective diameter of the PFAS molecules (about 1 nm) (Tsai et al. 2010; Rahman et al. 2014). For that reason, MF and UF are not discussed here. Although bench-scale studies indicate that the membrane molecular weight cut-off (MWCO) of NF/RO is probably the most important factor for removal of PFAS for these technologies, other factors, such as ionic charge, may also influence performance.

Two terms, “salt passage” and “salt rejection,” generally describe how membrane systems perform. Salt passage is the percentage of dissolved constituents (contaminants) in the feedwater allowed to pass through the membrane. The opposite of salt passage, salt rejection, is the percentage of feed water that does not pass through the membrane. In

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general, NF membranes have lower rejection rates (95%) than RO (> 99%), because NF membranes have larger pores (Rahman et al. 2014 provides an excellent review of many of the relevant studies). As with all other treatment options, bench-scale and pilot-scale testing are required to understand the field applicability and establish essential detailed design criteria such as pretreatment needs and cost effectiveness.

3.4.1 Reverse Osmosis

Technology Description: RO membranes are effective in removing most organic and inorganic compounds from water solutions. In recent years, new polymer chemistry and manufacturing processes have improved efficiency, lowering operating pressures and reducing costs. As a result, RO membranes are increasingly used by industry to concentrate or remove chemicals. RO is commonly used around the world in household drinking water purification systems, the production of bottled mineral water, self-contained water purification units (for the U.S. military), and industrial applications (for example, water supply to cooling towers, boilers, and deionized water). The largest application of RO is in desalination.

Reverse osmosis is most useful as one element within a treatment train, where the reject water is further treated or captured.

Treatment Mechanism: RO separates compounds from water solutions by passing pressurized water across a semipermeable membrane. Treated water (permeate) passes through the membrane and the rejected water (concentrate) is collected for disposal or discharge, depending on the nature of the compounds and particles present.

State of Development: RO has been studied in bench-scale studies and pilot plants for wastewater and drinking water PFAS applications and is considered a partially developed technology. Conventional and advanced treatments have been studied in several pilot plants and drinking water treatment plants, demonstrating both treatments operating simultaneously as well as the effectiveness of traditional drinking and wastewater treatment methods alongside PFAS-specific technologies.

Effectiveness: Influent pretreatment is critical for RO membranes because of their spiral-wound design. Membranes are highly susceptible to fouling (loss of production capacity) because some accumulated material cannot be removed from the membrane surface.

RO removal of PFAS from various waters has been studied (for example, semiconductor wastewater, drinking water, surface water, and reclaimed water), combined with NF in some cases. PFOS removal > 99% was achieved using four different types of membranes over a wide range of feed concentrations, from 0.5 to 1500 mg/L (Tang et al. 2006). Another study tested five RO and three NF membranes at feed concentrations of 10 mg/L PFOS over four days (Tang et al. 2007). The PFOS rejection and permeate flux performances were > 99% for RO and 90 to 99% for NF. The use of RO and NF as advanced drinking water treatments is still limited, but both technologies have been shown to be successful for the removal of longer-chain (> C5) PFAAs (Loi-Brugger et al. 2008; Tang et al. 2006). Conventional and advanced treatment efficiencies to remove PFOA and PFOS from surface water of the Llobregat River in northeast Spain were also studied. Results were compared in several pilot plants, and in a drinking water treatment plant that operates with UF and RO treatment alongside traditional treatment processes (Flores et al. 2013).

Another study examined the fate of PFSAs and PFCAs in two water reclamation plants that further treat water from wastewater treatment plants in Australia (Thompson et al. 2011). Plant A used adsorption and filtration methods alongside ozonation, while Plant B used membrane processes and an advanced oxidation process, to produce purified recycled water. At both facilities, PFOS, perfluorohexane sulfonate (PFHxS), perfluorohexanoate (PFHxA), and PFOA were the most frequently detected PFAS. Comparing the two reclamation facilities, Plant A showed some removal during the adsorption/filtration stages. Overall, however, Plant A failed to completely remove PFOS and the PFCAs shorter than perfluorononanoic acid (PFNA) in chain length. All PFAS present were removed by RO at Plant B from the finished water to concentrations below detection and reporting limits (0.4–1.5 ng/L).

Sustainability Considerations: The carbon footprint for this technology includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. RO requires power for high-pressure pumps and the management of concentrate, which can be energy intensive. The contaminant-rich brine rejected by RO must be disposed of appropriately. The removal of nontarget minerals from the treated water may increase its corrosivity, and posttreatment corrosion control measures are needed in most cases. Resources are available for performing a sustainability assessment for membrane filtration remedial design (Ras and von Blottnitz 2012).

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3.4.2 Nanofiltration

Technology Description: NF is a form of membrane technology that is pressure-driven and shown to be effective in the removal of PFAS (Tang et al. 2007). This method provides high water flux at low operating pressure (Izadpanah and Javidnia 2012). Like RO, NF is easy to operate and reliable for the removal of chemicals.

Technology Mechanism: Nanometer-sized membrane pores are used to separate compounds in a process similar to RO, but NF does not remove smaller ions such as chloride and sodium.

State of Development: NF is considered a partially developed technology because available data on the removal of PFAS are limited to laboratory-scale tests performed on flat sheet membrane coupons.

Effectiveness: No studies have reported either pilot or full-scale performance of NF membranes. Therefore, variations in performance due to fouling, flux, and concentration distributions in standard spiral-wound membrane configurations have not been characterized.

NF membranes tested include the Dow membranes NF-270, NF-200, and NF-90 and the SUEZ (formerly GE Water & Process Technologies) DK membrane. Reported rejections were generally > 95% for PFAS with molecular weights ranging from 214 g/mol to 713 g/mol (Steinle-Darling and Reinhard 2008; Appleman et al. 2013). However, lower rejections were observed for perfluoropentanoate (PFPeA) and perfluorooctane sulfonamide (FOSA)—about 70 and 90%, respectively (Steinle-Darling and Reinhard 2008). Recovery and salt passage information was not reported in these studies. Scaling up these results is challenging because rejection is affected by recovery, which can vary from one application to another.

Salt passage for PFOS was reported to range from < 1% for the tighter NF-90 membrane to about 6% for the looser NF-270 and DK membranes (Tang et al. 2007). PFOS salt passage also was correlated to sodium chloride salt passage, a common specification for membrane manufacturers (Tang et al. 2007). Salt passage incorporates both rejection and membrane recovery, therefore is a more useful parameter for predicting full-scale performance than rejection.

Pilot and full-scale testing of the selected NF membrane for PFAS removal is a crucial step when considering this treatment process. Choosing membranes with MWCO smaller than the targeted PFAS is also a key design consideration.

Appropriate disposal or treatment of the membrane concentrate stream is another design factor, especially when using high-pressure membranes for inland communities. Furthermore, NF membrane fouling mechanisms are poorly understood and further research is needed to develop cost-effective cleaning methods to restore membrane performance (Al-Amoudi and Lovitt 2007). As with other forms of filtration, pretreatment strategies to avoid fouling and membrane fabrication drive performance results.

Sustainability Considerations: The carbon footprint for this technology includes energy source and consumption during treatment system operation, as well as manufacturing/disposal of treatment media. NF requires power for pumps (generally less than RO), and the management of concentrate, which can be energy intensive.

4 References and Acronyms

The references cited in this fact sheet, and the other ITRC PFAS fact sheets, are included in one combined list that is available on the ITRC web site. The combined acronyms list is also available on the ITRC web site.



Per- and Polyfluoroalkyl Substances (PFAS) Team Contacts

Robert Mueller • New Jersey Department of Environmental Protection
609-984-3910 • Bob.Mueller@dep.nj.gov

Virginia Yingling • Minnesota Department of Health
651-343-2890 • virginia.yingling@state.mn.us

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PFAS Fact Sheets

This page includes links for the ITRC PFAS fact sheets. The fact sheets are available as PDF files. Several tables of supporting information are published separately so that they can be updated periodically by ITRC. The fact sheet user should visit this page to access the current versions of the files.

The references list for the fact sheets is included here as a separate PDF. An acronyms list is available on the website.

- Naming Conventions and Physical and Chemical Properties (updated April 2020)
- Regulations, Guidance, and Advisories (updated April 2020)
  - PFAS Water and Soil Values Table Excel file– (updated March 2020)
    - The Water Table includes presents the available PFAS water values established by the USEPA, each pertinent state, or country (Australia, Canada and Western European countries)
    - The Soil Table includes the available PFAS soil values established by the USEPA, each pertinent state, or country (Australia, Canada and Western European countries)
  - Basis for PFOA and PFOS Values Tables Excel file (updated March 2020)
    - The PFOA Table summarizes the differences in the PFOA values for drinking water in the United States.
    - The PFOS Table summarizes the differences in the PFOS values for drinking water in the United States. Regulación, Orientación, y Asesoramiento para sustancias Per- y Polifluroalquiladas (PFAS)

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- History and Use (updated April 2020)
- Environmental Fate and Transport (published April 2020)
    - Physical and Chemical Properties Table for select PFAS Excel file (updated April 2020)
- Site Characterization Considerations, Sampling Precautions, and Laboratory Analytical Methods (published April 2020)
- Remediation Technologies and Methods – the fact sheet has been withdrawn. The user is directed to the guidance document section 12 for updated information about Treatment Technologies
- Aqueous Film-Forming Foam (published April 2020)

PFAS Fact Sheets Explainer Videos

The ITRC PFAS team has developed a series of explainer videos that are intended to provide an overview of the subject matter explored in the fact sheets. The following links will redirect you to ITRC's PFAS explainer videos on Youtube:

- ITRC PFAS Team Introduction
- PFAS Naming Conventions
- PFAS History and Use
- PFAS Fate and Transport
- PFAS Remediation
- PFAS Lab Analytical Methods
- Aqueous Film Forming Foam

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APPENDIX 1006A

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The physical nature and manufacture of activated carbon

by G.J. McDOUGALL*

SYNOPSIS

After defining activated carbon, the author describes its structure and outlines the physical characteristics distinguishing one type of activated carbon from another. The adsorptive properties of these carbons, the raw materials used, and the manufacturing processes—chemical activation, and physical or thermal activation—are outlined. The high-temperature thermal route (which is the most important for the products employed in gold recovery) using coconut shells or coal as the raw material is then discussed in some detail.

SAMEVATTING

Na 'n omskrywing van geaktiveerde koolstof bespreek die skrywer die struktuur daarvan en gee in hooftrekke die fisiese eienskappe wat een soort geaktiveerde koolstof van 'n ander onderskei. Die adsorpsie-eienskappe van hierdie koolstowwe, die grondstowwe wat gebruik word, en die verwaardigingsprosesse—chemiese aktivering en fisiese of termiese aktivering—word in hooftrekke behandel. Die termiese hoëtemperatuurroete (die belangrikste roete wat betref die produkte wat in goudherwinning gebruik word) wat van klapperdoppe of steenkool as die grondstof gebruik maak, word daarna redelik in besonderhede bespreek.

Introduction

Carbonized wood, which can be regarded as a crude form of activated carbon by present-day standards, has been in use for many thousands of years. In about 1500 B.C., the Egyptians, who were aware of the adsorbent properties of wood charcoal, used it for medicinal purposes and as a purifying agent, as did the ancient Hindus, who filtered their drinking water through charcoal[1].

The decolorization and gas-adsorption properties of various carbon products were first investigated in the late nineteenth century, their development having gained impetus from the need for gas adsorbents to protect people against the poisonous gases used in World War I, and also from new applications that arose in the sugar industry and that are related to decolorization.

The ability of activated carbon to adsorb gold from chloride solutions was first reported[2] in 1847. This information attracted considerable interest and, in 1880, Davis[3] patented a process in which wood charcoal was used to recover gold from chloride leach liquors. This process was subsequently to become popular, particularly in Australia.

Shortly after the discovery by MacArthur and the Forrest brothers in 1890 that cyanide was a good solvent for gold[4], Johnson[5] (1894) patented the use of wood charcoal for the recovery of gold from cyanide solutions. Since then, major developments have occurred in the technology relating to the manufacture of activated carbons, which are currently employed commercially in a wide range of industries.

Important applications relate to the use of activated carbons to render water potable by the removal of taste, colour, odour, and undesirable organic impurities, in the treatment of domestic and industrial waste water, in the removal of colour from various types of sugar syrups, in a variety of gas-phase applications, and in the purification of many chemical and pharmaceutical products and foodstuffs. Activated carbon also recently found increased application in the field of hydrometallurgy, particularly in the recovery of gold, silver and, to a lesser extent, molybdenum[6-8].

Despite the commercial importance of activated carbon, little is known outside the industry itself about the raw materials that are suitable for conversion into activated carbon, the methods of manufacture, and the properties that make a product superior for a particular use and apparently unsuitable for another.

In South Africa, the gold-mining industry is the largest single consumer of activated carbon, and it was felt that a paper dealing with general aspects relating to the manufacture of activated carbons, as well as with the properties of the resultant products, would be of interest to personnel in the industry.

What is Activated Carbon?

Activated carbon is a generic term for a family of highly porous carbonaceous materials, none of which can be characterized by a structural formula or by chemical analysis[1]. The volume of the pores in activated carbons is generally defined as being greater than 0,2 ml/g, and the internal surface area is generally larger than 400 m^2/g as measured by the nitrogen BET method. The width of the pores varies from 3 Å to several thousand angstroms, and the pores are generally classified for convenience in terms of their diameters:

Macropores	500	to 20 000	Å
Transitional pores (mesopores)	100	to 500	Å
Micropores	8	to 100	Å.

In cross-section, the pores appear to be cylindrical or rectangular in shape, but can also occur in a variety of

* NCP, P.O. Box 284, Bedfordview, 2008 Transvaal.
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irregular shapes. Pores may also have constrictions or bottlenecks.

Macropores, which generally contribute very little to the total internal surface area of the product, depend mainly on the nature of the carbonaceous raw material employed and the preliminary manufacturing process, viz grinding and agglomeration of the raw material. Their major function is to serve as transport arteries that make the internal parts of the carbon granules readily accessible to the molecules being adsorbed. The transitional pores account for about 5 per cent, and the micropores for about 95 per cent, of the internal surface area. The micropores are largely the product of the activation process.

Activated carbons are commercially available in numerous forms: powders, granular chips in various size ranges, and shaped or moulded products extruded into rod-like shapes, which are commonly 0,8 to 6 mm in diameter by 3 to 10 mm in length.

Structure

X-ray-diffraction studies have shown that the structure of thermally activated carbon is similar to that of graphite[9,10]. Ideal graphite, as shown in Fig. 1, consists of layers of fused hexagons held approximately 3,35 Å apart by Van der Waals forces, so that the carbon atoms in any one plane lie above the centres of the hexagons in the layer immediately below it. The lattice is of the ABAB type.

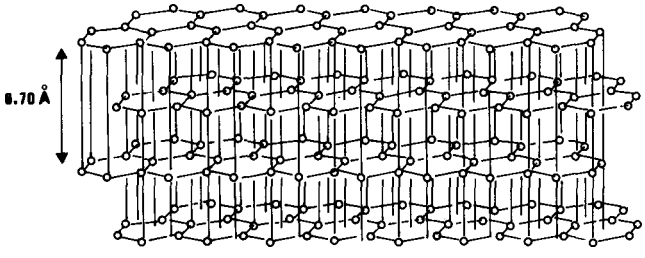


Fig. 1—Schematic representation of the structure of graphite; the circles denote the positions of carbon atoms, while the horizontal lines represent carbon-to-carbon bonds (after Bockris[9])

The proposed structure of thermally activated carbon is depicted in Fig. 2. The carbon is believed to be composed of tiny graphite-like platelets, only a few carbon atoms thick and 20 to 100 Å in diameter, which form the walls of open cavities of molecular dimensions, i.e. the pore system. However, the hexagonal carbon rings, many of which have undergone cleavage, are randomly orientated, and lack the directional relationship with one another that is present in single graphite crystals. The overall structure is therefore very disordered, and is often referred to as 'turbostratic'. Furthermore, the separation between the layers is greater than that found in graphite, viz 3,60 Å.

Because of the high level of structural imperfections in activated carbons, there are many possibilities for reactions with carbon atoms at the edges of the planar layers. As a result, oxygen-containing organic functional groups, which are located mostly at the edges of broken graphitic ring systems, are present on the surface of the carbon.

Although the exact chemical structures of these surface oxides are not known with certainty, it has been suggested that the surface oxides depicted in Fig. 3 are the oxides most often present in thermally activated carbons[10]. Basic surface groups have also been identified, and it appears that the nature of the surface groups depends upon conditions during and after manufacture[6].

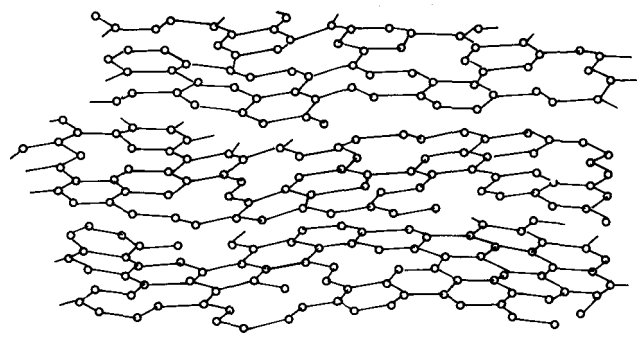


Fig. 2—Schematic representation of the proposed structure of activated carbon; oxygen-containing functional groups are located at the edges of broken graphitic ring systems (after Bockris[9])

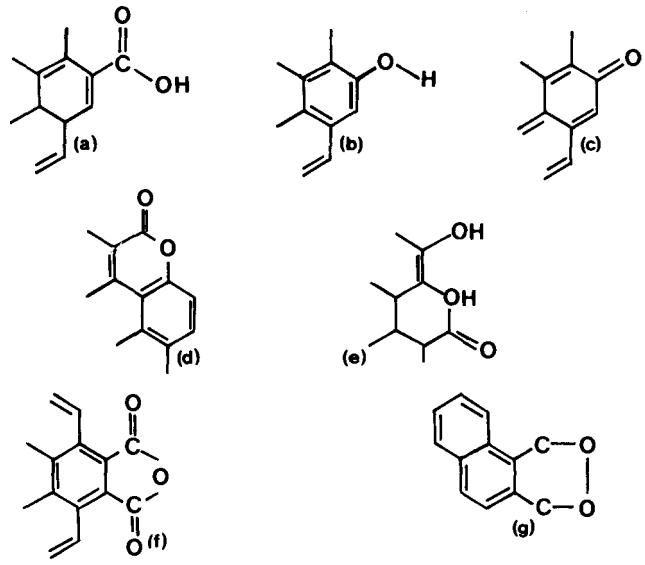


Fig. 3—Structure of some surface oxides[10]: (a) carboxylic acid (b) phenolic hydroxyl (c) quinone-type carbonyl groups (d) normal lactone (e) fluorescein-type lactones (f) carboxylic acid anhydrides (g) cyclic peroxides

Characterization of Activated Carbons

The different types of activated carbon can be distinguished from one another only by reference to certain physical and chemical properties.

Important physical properties are the number and size distribution of the pores, bulk density, dry impact hardness, wet abrasion resistance, and particle-size distribution. From the chemical point of view, comparisons are based on the ability of the different products to adsorb various selected substances from the gaseous phase, e.g. benzene, carbon tetrachloride, and nitrogen, as well as from the aqueous phase, e.g. iodine, molasses, phenol, methylene blue, and tannin.

For example, the nitrogen BET value expresses the surface area in square metres per gram of adsorbent that

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can be covered by nitrogen in a monomolecular layer. Activated carbons typically yield values between 400 and 1500 m^2/g, the former value corresponding to what is termed a low-activity product and the latter to a high-activity product. Such surface-area dimensions alone are insufficient to characterize a carbon product, simply because the nitrogen molecule is very small and can therefore penetrate into pores that are not accessible to other larger molecules. Hence, for the practical assessment of activated carbons, the adsorptive capability of the various products for compounds of large molecular dimensions, viz molasses, methylene blue, and tannin, is determined under standard experimental conditions.

The extent to which the various activated carbons adsorb molecules of a different size can be regarded as an indication of the distribution of their internal accessible volume among pores of different sizes. Hence, the small iodine molecule is adsorbed in pores down to 10 Å in diameter so that the iodine number is an indication of the surface area of the pores in that size range. At the other end of the scale, molasses, a large polysaccharide molecule, is adsorbed only in pores larger than about 30 Å in diameter, and the molasses number gives an indication of the number of pores in that size range[8].

A more sophisticated method by which information about the pore-size distribution can be gained involves measurement of the pressure required to force liquid mercury into the activated carbon. This, in turn, can be related to the size of the pore into which the mercury is being forced. The pore-size distributions shown in Figs 9 and 11 were obtained by this method.

Those physical and chemical properties most often employed by manufacturers of activated carbon to characterize their products are listed in Table I. Typical values are indicated for a high-activity coconut-shell carbon, which is activated thermally using steam, and which is usually employed in gas-phase applications and in processes for the recovery of gold from cyanide leach liquors.

TABLE I
PHYSICAL PROPERTIES AND CHEMICAL-ADSORPTION CHARACTERISTICS OF A THERMALLY ACTIVATED COCONUT-SHELL CARBON TYPICAL OF THOSE EMPLOYED IN GOLD-RECOVERY APPLICATIONS

Physical properties	
Particle density (mercury displacement)	0,80 to 0,85 g/ml
Bulk density	0,48 to 0,54 g/ml
Pore volume	0,70 to 0,80 ml/g
Ball-pan hardness (ASTM)	97 to 99%
Particle-size distribution	1,18 to 2,36 mm
Ash content	2 to 4%
Moisture content	1 to 4%
Chemical-adsorption characteristics	
Surface area (nitrogen BET)	1050 to 1200 m^2/g
Iodine number*	1000 to 1150 mg/g
Carbon tetrachloride number†	60 to 70%
Benzene number	36 to 40%

* The iodine number indicates the mass of iodine adsorbed out of aqueous solution under standard experimental conditions by 1 g of activated carbon

† The carbon tetrachloride number indicates the mass of carbon tetrachloride vapour adsorbed under standard experimental conditions by 1 g of activated carbon, expressed as a percentage

Adsorptive Properties

It is generally conceded that activated carbon owes its adsorptive properties primarily to its large internal surface area, as well as to its pore-size distribution, and that the external surface area and the nature of the surface oxides play minor roles[6,10]. The size of the pores developed during activation therefore has an important influence on adsorption behaviour because the pores act as a screen. This screen prevents the adsorption of large molecules, but promotes the adsorption of an adsorbate that fits snugly into the pores, making the maximum number of contacts with the adsorbed molecule, and thus maximizing ΔH as defined in equation (1):

$$\Delta H = \Delta G + T\Delta S, \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where

ΔH is the change in free enthalpy,
ΔG is the change in free energy,
ΔS is the change in entropy, and
T is the temperature of the system.

Fig. 4 attempts to illustrate this concept for the case in which two adsorbate molecules in a solvent, which is not shown, compete with each other for adsorbent surface or sites. The external surface can provide only a certain amount of access to the inner pores, and it is likely that the main role of the surface oxides is to impart a hydrophilic character to the predominantly hydrophobic carbon skeleton. This would account for the affinity of activated carbon for many polar and non-polar organic and inorganic species[8].

Activated carbon functions by adsorption, i.e. by the adhesion of certain substances to the internal surface constituting the walls of the pores. Hence, the greater the adsorption surface available, the better is the adsorption function.

Adsorption occurs as the result of an imbalance of the forces that act upon the carbon atoms constituting the surface of the pore wall. Such an imbalance is inherent in all surfaces and, in an attempt to rectify it, molecules are adsorbed from the gaseous or aqueous phases, and are attracted and held to the surface. Surface adsorption, regardless of the energy of the interaction, must always proceed with a negative change in free energy (ΔG), as well as a decrease in entropy (ΔS), which, according to equation (1), must result in a negative change in free enthalpy (ΔH). Hence, adsorption is always an exothermic process[10].

There are two types of adsorption process: physical and chemical adsorption. Physical adsorption involves weak Van der Waals forces (also dipole–dipole interactions and hydrogen bonding), and the processes are generally reversible. Chemical adsorption refers to processes involving homopolar forces (as in ionic or covalent bonds), and such processes are generally irreversible. Nevertheless, in most instances, the adsorptions can be classified as being physical in nature.

In general, the factors that influence the adsorptive behaviour of activated carbon from aqueous solution are temperature (adsorption generally decreases with increasing temperature); the pH value of the solution (carbon generally has a low affinity for ions, particularly those with a high charge-to-surface ratio, and pH can affect

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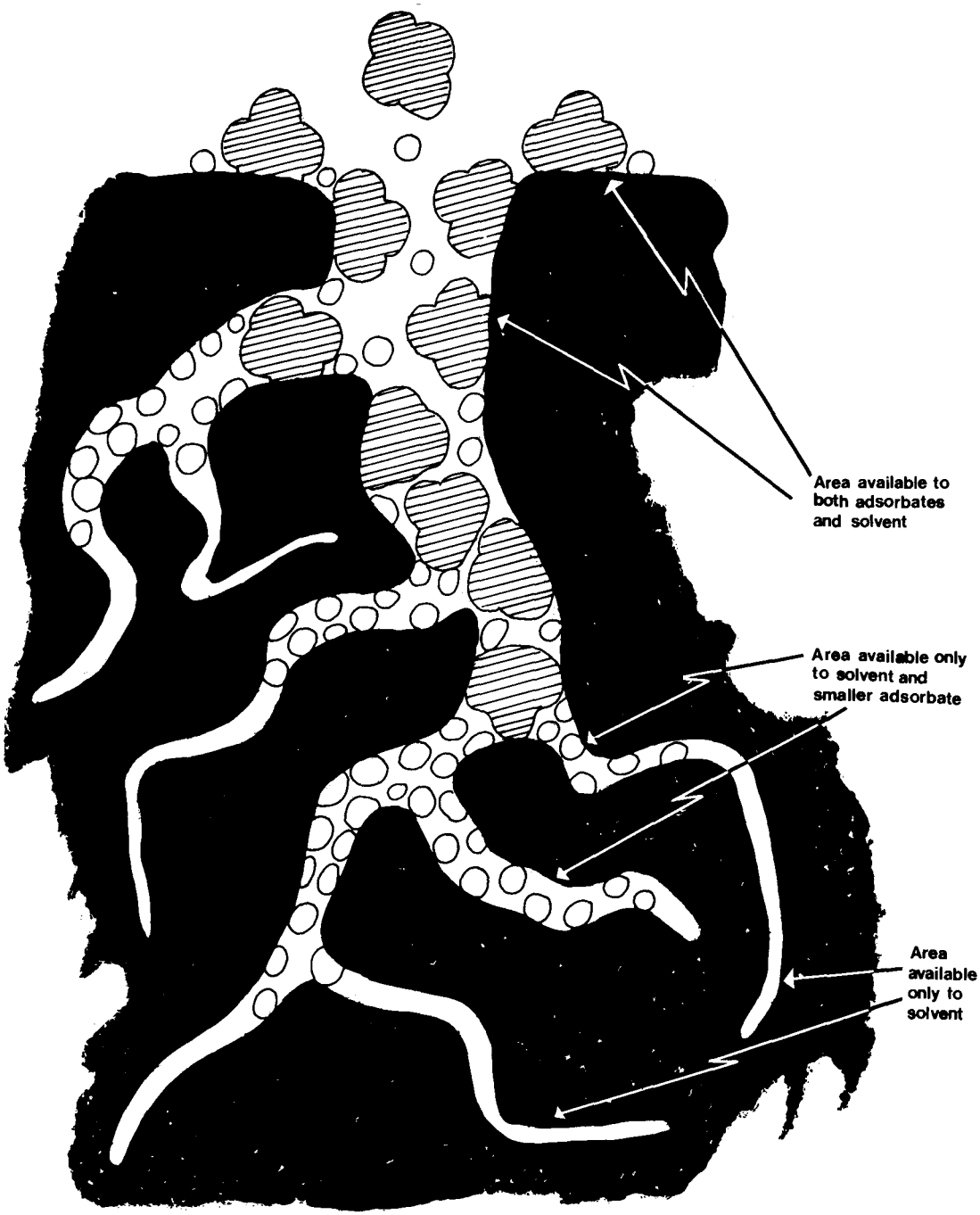


Fig. 4—Concept of molecular screening in micropores (after Pittsburgh Activated Carbon Brochure[11])

ionicity); the chemical nature of the species present and their relative concentration; and the nature of the activated carbon and its particle-size distribution. For molecules to be adsorbed, they must reach the internal surface of the macropores by diffusion. Therefore, the reaction time will be influenced by the length of the diffusion path, and the kinetics of adsorption will increase with decreasing particle size.

Activated carbon is a very versatile substrate with respect to its interactions with various organic and inorganic compounds. It is able to function as a simple adsorbent that is akin to synthetic polymeric adsorbents, since molecular compounds can be physically adsorbed onto its large internal surface area, and be retained there by forces of the Van der Waals type[7,8]. It can also function as a reducing agent or, in the presence of excess oxygen, as an oxidation catalyst[1,7,10]. As a result, the adsorption of organic and inorganic species onto carbon may occur by several mechanisms, and their identification, particularly in the extraction of certain metal complexes from solution, is extremely difficult as in the extraction of the gold dicyanoaurate anion[12-14].

Because carbon is generally a non-polar material, it adsorbs organic compounds in preference to polar inorganic species. For example, small highly charged cations such as Ni^{2+} and anions such as Cl^- are not adsorbed by activated carbon to any significant extent[13].

The adsorption of organic species onto carbon usually

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occurs by simple mechanisms that are fairly well understood in terms of a balance between hydrogen-bonding forces in solution and Van der Waals attraction between the carbon surface and the organic compounds. The driving force for the adsorption of organic molecules onto carbon is similar to that responsible for the salting-out of organic materials dissolved in water, where the addition of a simple inorganic salt causes the organic material to separate out from the aqueous phase[8].

These considerations have given rise to the statement frequently made that adsorption onto activated carbon is a fight against solubility. Applications involving organic materials will therefore be most attractive when the adsorbate has high molecular mass, low polarity, and a low degree of ionization, because these properties generally signify low aqueous solubility. Hence, activated carbons are excellent adsorbents for a considerable number of aliphatic compounds, and particularly for aromatic compounds like those commonly found in pesticides and herbicides. As such, activated carbons are employed extensively, not only in commercial applications that require the removal of contaminating organic compounds from aqueous effluents, but also in purification and recovery processes[6,10].

Because granular activated carbons are expensive, their application is usually economically viable only if the carbon can be regenerated and therefore recycled in the process.

The regeneration of granular activated carbon is generally accomplished using steam, hot solvents, or thermal routes. Regeneration by extraction with steam can be applied if the carbon contains substances that volatilize at temperatures below 150°C. For substances of higher boiling points that pyrolyse on heating, thermal reactivation at 700 to 950°C is employed. Steam is usually injected into the kiln, and any pyrolysed organic substances, i.e. non-volatile carbonaceous materials, that may be present in the carbon are burnt away by the reaction shown in equation (2):

$$C + H_2O \rightleftarrows CO + H_2 \ (\Delta H = 29 \text{ kcal/mol}). \ .. \ (2)$$

This treatment cleans the pyrolysed contaminant carbon from the walls of the pores, and returns the carbon to its original activity. It should be noted that the pyrolysed residue is indistinguishable from the original carbon, and care should therefore be taken to ensure that not too much of the original carbon matrix is burnt away during thermal reactivation.

Regeneration can also be accomplished by treatment of the spent carbon with a solvent in which the adsorbate is highly soluble. The preferred route will depend on whether recovery of the adsorbate is desired or not. If it is, regeneration using steam or solvent extraction is preferred.

Manufacture of Activated Carbons

The most important step in the production of activated carbon is activation. This is the process by which the chosen raw material or carbonized raw material is converted into a finely crystalline form of carbon that is permeated by the greatest possible number of randomly distributed pores of various shapes and sizes. These pores give rise to the extended internal surface area. In the manufacture of activated carbon, it is desirable to develop the maximum accessible surface area that is commensurate with economic viability and product application[10,15-17].

The products of simple coking or carbonizing (such as that used in the early days) do not meet present-day standards. This can be attributed to the fact that the available surface area of pulverized charcoal, which is essentially external surface area, is only 2 to 4 m^2/g—as against typically 1000 m^2/g for a granular activated-carbon product—measured by the nitrogen BET method.

Raw Materials

Activated carbon can be manufactured from any carbonaceous raw material, and numerous patents have been granted for processes using such diverse starting materials as various ranks of coal (peat, lignite, bituminous coal, and anthracite), coconut shells, peach and apricot stones, waste tyres, wood, sawdust, petroleum heavy oil, cellulose, rice husks, corn cobs, sugar, bones, etc.[10,16,17]. All the starting materials used in relatively large-scale manufacture are readily available and, hence, are reasonably priced[15]. In the Western World, the companies entering the business in the past 30 years have tended to concentrate on using coal (mostly lignite, bituminous coal, and anthracite), whereas, in the developing world, new entrants have concentrated on using coconut-shell charcoal.

Since the manufacturing process involves the removal of volatile matter from the raw material, the economic relationship between the price, availability, and quality of the raw material on the one hand, and its volatile content on the other, is important. For example, one of the advantages of bituminous coal and anthracite is their relatively low volatile content (as shown in Table II) and, hence, their high yield of product[15].

TABLE II
FIXED CARBON CONTENT OF RAW MATERIALS COMMONLY EMPLOYED IN THE MANUFACTURE OF ACTIVATED CARBON (AFTER HOLDEN[12])

Material	Approximate carbon content, %
Soft wood	40
Hard wood	40
Coconut shell	40
Lignite	60
Bituminous coal	75
Anthracite	90

In the developing world, a major consideration is the availability of waste materials such as coconut shells and rice husks, which, despite having very high volatile contents and, hence, giving low yields of activated carbon, are so inexpensive that the economics involved in their use are often favourable.

Although the activation procedure and the after-treatment employed mainly determine the chemical nature of the surface oxides and the surface area of the resultant product, the structure of the pores and the pore-size distribution are largely predetermined by the nature of the starting material.

This paper focuses on the various steps involved in the

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manufacture of activated carbon from coconut-shell charcoal and from coal, as well as on the properties of the resultant products.

Chemical Versus Physical Manufacturing Processes

Although the general outlines of the various manufacturing processes are well known, the exact details of the procedures employed by the manufacturers are jealously guarded secrets. In general, the methods most commonly employed for the manufacture of activated carbon are of two main types: the wet-chemical process (basically a single-stage process), and the physical or thermal–gas process (essentially a two-stage process).

As would be expected, chemical methods use chemicals for activation, whereas thermal processes use gases (usually carbon dioxide and air), vapours (steam), or mixtures of gases and vapours[15-17].

Chemical Activation

The wet-chemical process is generally employed to convert uncarbonized cellulosic material, primarily wood, into activated carbon. The most popular activating agent is phosphoric acid, although zinc chloride and sulphuric acid are also well-known agents. Other chemicals also suggested for use include sodium carbonate, sodium and calcium hydroxide, and the chloride salts of magnesium, calcium, ferric iron, and aluminium. These chemicals have in common only their strength as dehydrating agents.

The process using sawdust as the starting material involves mixing of the raw material and the dehydrating agent into a paste. The paste is then dried and carbonized in some type of kiln, usually a rotary kiln, at between 200 and 650°C. Upon calcination, the impregnated chemicals dehydrate the raw material, resulting in charring and aromatization of the carbon skeleton, with the concomitant creation of a porous structure and extended surface area.

This procedure yields a power product. If a granular product is required, granular materials are simply impregnated with the activating agent, and the same general procedure as that described above is followed. However, these granular products are generally soft (unless they are manufactured from the powder after suitable pelletization), which limits their application.

After carbonization of the paste, the activating agents are usually recovered and recycled for economic reasons. All the commonly used chemical activators impose certain constraints on the overall activation process and the equipment employed, or on the quality of the product. Mixtures of sulphuric acid and wood cannot be heated above 200°C, and the product, after the sulphuric acid has been recovered by leaching, has adsorptive properties only when wet. Activation with phosphoric acid requires temperatures in the range 375 to 500°C, and the reagent is readily recovered by leaching with water. However, corrosion of the equipment is a major problem. Activation with zinc chloride occurs in the temperature range 550 to 650°C and, although most of the zinc chloride is recovered by leaching with dilute hydrochloric acid, problems may arise in the application of the product because it retains traces of zinc salts. The activated carbon produced with phosphoric acid and zinc chloride can be dried with little, if any, loss of adsorptive capacity.

The activity of the product can be controlled by alteration of the proportion of raw material to chemical reagent. For phosphoric acid, the proportion is usually between 1:0,5 and 1:4. The activity, as measured by the number of pores created, increases as the proportion of the chemical activator increases, and is also affected by the temperature and residence time in the kiln.

Physical or Thermal Activation

The first step in the production of activated carbon by the thermal route is carbonization, which is the formation of a char from the source material. Carbonization is generally accomplished by heating of the source material—as lumps or pre-sized material, or in moulded form, e.g. as briquettes or extrudates—in an inert atmosphere such as flue gas to a temperature that must not exceed 700°C so that dehydration and devolatilization of many of the carbon atoms can occur in a controlled manner. The main purpose of carbonization is to reduce the volatile content of the source material (a fixed carbon content of 80 per cent or higher is desirable) to convert it to a suitable form for activation. Rearrangement of the carbon atoms into graphitic-like structures also occurs during carbonization.

As previously mentioned, the process by which the carbonized product develops an extended surface area and a porous structure of molecular dimensions is referred to as *activation*. This step is generally conducted at temperatures between 800 and 1100°C in the presence of a suitable oxidizing agent such as steam, air, carbon dioxide, or any mixture of these gases. The active oxygen in the activating agent burns away the more reactive components of the carbon skeleton as carbon monoxide and carbon dioxide, depending on the oxidizing agent employed. Although the burning-out of the carbon skeleton to create new pores is believed to occur at different rates on different parts of the exposed surface, the detailed mechanism of this process is not clearly understood.

According to Smisek and Cerny[16], gasification of the carbonaceous material by steam and carbon dioxide occurs in accordance with the endothermic reactions shown in equations (2) and (3):

$$C + H_2O \rightleftharpoons CO + H_2 \ (\Delta H = 29 \text{ kcal/mol}). \ldots (2)$$

$$C + CO_2 \rightleftharpoons 2CO \ (\Delta H = 39 \text{ kcal/mol}). \ldots\ldots (3)$$

The reaction of water vapour with carbon is accompanied by the secondary reaction of water–gas formation, which is catalysed by the carbon surface as shown in equation (4):

$$CO + H_2O \rightleftharpoons CO_2 + H_2 \ (\Delta H = -10 \text{ kcal/mol}). \quad (4)$$

Because the reactions of carbon with steam and carbon dioxide are endothermic, the activation process lends itself to accurate control of the conditions in the kiln. External heating is required to drive reactions (2) and (3), and to maintain the reaction temperatures. However, as shown in equation (5), the reaction of carbon with air (oxygen) is extremely exothermic:

$$C + O_2 \rightleftharpoons CO_2 \ (\Delta H = -97 \text{ kcal/mol}). \ldots\ldots (5)$$

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This reaction is difficult to control, and excessive burn-off of the external carbon surface can easily occur, resulting in a decrease in average particle size, which obviously reduces the yield of final product. The reaction of steam with carbon is catalysed by certain chemicals (e.g. the oxides and carbonates of alkali metals, iron, copper, and other metals), and some commercial operations use these chemicals as catalysts. Therefore, as combustion proceeds, preferential etching occurs, which results in the development of a large internal surface area and the creation of a pore structure.

The art of the manufacturer of activated carbon lies in conducting the activation process in such a way that combustion of the carbonized raw material occurs internally, and not from the exterior of the granules. The term *burn-off* is used to denote the degree of activation, which is the loss of char (in percentage by mass) that is allowed to occur. The burn-off (B) and activation yield (Y) are related as indicated in equation (6):

$$B = 100 - Y. \quad \ldots\ldots (6)$$

Carbonization and activation used to be carried out separately, but there is currently an increasing tendency for the two processes to be conducted in the same kiln. A wide variety of furnaces and kilns are used for carbonization and activation, including rotary kilns fired directly or indirectly, multiple-hearth furnaces, fluidized-bed reactors, and vertical retorts, each of which has advantages for a particular purpose. The indirectly fired rotary kiln, for example, is relatively large and expensive, but avoids a large discharge of inert gases to the afterburner. The fluidized-bed reactor is extremely compact, but is generally used only for the activation of abrasion-resistant raw materials because of the attrition that occurs during fluidization.

The effect of the heat treatment of a carbonaceous material on its resultant structure is as follows[16]. As the temperature of carbonization increases, polymerization occurs and, irrespective of whether the starting material is composed of long-chain aliphatic or aromatic molecules, a system of planar condensed benzene-ring molecules is formed. A solid then forms (this is possible at a range of setting temperatures) as a result of the formation of C – C bonds between neighbouring planes. From 400 to 700°C, these condensed ring systems grow gradually, but all the peripheral carbon atoms are attached by chemical bonds to hydrogen atoms or to hydrocarbon groups. Hence, these substances are termed condensed molecular solids, and have high electrical resistivity, which is typical of chemically activated carbons. However, between 700 and 800°C, many of the hydrogen atoms or hydrocarbon groups are expelled from the material, leaving small crystallites having a structure similar to that of graphite. The evolution of gases coincides with a drastic reduction in electrical resistivity.

Moulded forms of the raw material, e.g. briquettes or extrudates, are obtained as follows. The wood charcoal, coal, or peat used in the raw material is pulverized, and the powder is mixed with a coking binder. The hot mixture is then pressed into briquettes, or is extruded through dies into rod-like shapes of a selected diameter. Powder carbons are obtained by the crushing and grinding of granular material.

If cognizance is taken of the many combinations of source material and conditions for carbonization and activation that can be employed in the manufacturing process, it is apparent that a large number of activated-carbon products can be obtained. Because, as previously discussed, no clearly defined chemical formulae can be determined for these products, the only way in which they can be distinguished from one another is by reference to selected physical and chemical properties.

Properties of Activated Carbons

The most significant differences between chemically and thermally activated carbons relate to pore-size distribution (Fig. 9) and semiconductor properties.

Chemically activated carbons are generally characterized by a macroporous pore-size distribution, and these products therefore generally have a high molasses number and comparatively low nitrogen BET and iodine numbers[18]. Such activated carbons are used mainly in applications that require the removal of large molecules from solutions, e.g. in the decolorization of sugar syrups. The absence of semiconductor properties in chemically activated carbons almost certainly relates to the fact that the temperature generally employed in the manufacture of these carbons is too low for the creation in the final product of graphitic zones, which are presumably responsible for the semiconductor–redox properties of thermally activated products[14,19].

The reduction potential of thermally activated carbons, measured[12] by a graphite-rod technique, has been found to be typically above −0,14 V versus the saturated calomel electrode at a pH value of 6. These carbons can therefore function as reducing agents, and many of the intriguing properties of thermally activated carbons, particularly their redox and catalytic properties, are believed to be related to their semiconductor properties.

It is interesting to note that chemically activated carbons have a poor affinity for the gold dicyanoaurate anion. This may be indicative of the important role played in the mechanism of gold extraction from a cyanide medium by the graphitic zones that are believed to exist in thermally activated carbon[14,20].

Since the high-temperature thermal route is employed most often for the manufacture of granular or shaped activated carbon products, and is also the most important route for the manufacture of products suitable for use in gold-recovery applications, that method will be focused on here. Furthermore, any detailed discussion of typical process routes for the preparation of activated carbon will be confined to those using coconut shells and coal as raw materials.

Process Route Using Coconut Shells

A typical flowsheet for the manufacture of activated carbon from presized or unsized char is depicted in Fig. 5. A similar route would be used for the manufacture of activated carbon from peach and apricot stones, or from any other hard, abrasion-resistant char, e.g. hard woods, which have sufficient inherent porosity to permit the activating agent to penetrate into the interior of the granule and simultaneously allow the products of the oxidation–gasification reactions to escape.

Details pertaining to the important unit operations that

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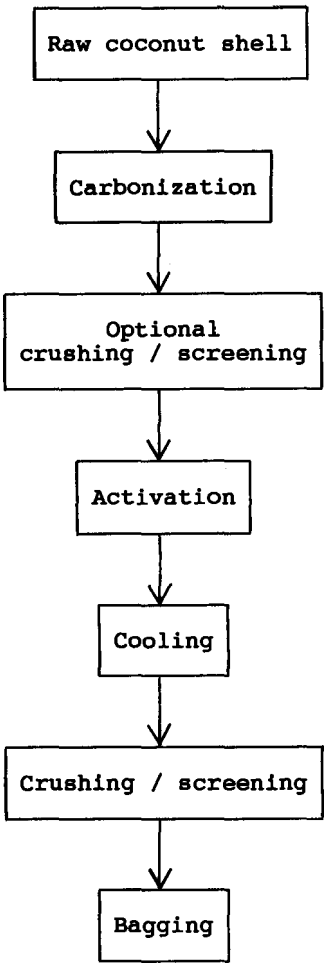


Fig. 5—Flowsheet for the manufacture of activated carbon from coconut-shell char (after Yehaskel[17])

constitute the process are discussed below.

Carbonization

A large quantity of coconut shells are available in countries such as the Philippines, Sri Lanka, and the South East Asian Islands as a byproduct of the coconut-oil and desiccated-coconut industries. Since coconut shells are eminently suitable for the production of very hard granular activated carbon, a viable industry for the manufacture of activated carbon has arisen in the Philippines and Sri Lanka. Because of its close proximity to the Philippines, Japan is a large importer of coconut-shell char and, hence, a manufacturer of activated coconut-shell carbon. As a matter of interest, the cigarette industry in Japan is the single largest consumer of activated coconut-shell carbon, since, in order to minimize the incidence of cancer due to smoking, the law requires fine carbon granules to be used in cigarette filters to adsorb the volatile organic materials emanating from the burning tobacco.

In the coconut-shell industry, carbonization is generally a small-scale, primitive, and crude operation that is conducted in the heart of the coconut plantations by individual farmers in, for example, 200-litre drums cut in half. Copra is placed in the bottom of the drum, which is then filled with coconut shells and ignited. The drum is covered with a sheet of metal, and the coconut shells are allowed to char for a couple of hours in an atmosphere that is essentially depleted of air. The char is sold to central depots, usually established by the various local manufacturers or exporters of char. Although charring is also sometimes done on a larger scale in pits, or simply in trenches dug in the sand on beaches, it is very much a cottage industry. Quality control in relation to char is conducted on the premises of the manufacturer using various techniques based on density separation.

As was mentioned earlier, carbonization is essentially a devolatilization step, which produces a char with a significantly higher content of fixed carbon (between 70 and 85 per cent) than is present in raw coconut shells.

Activation

Prior to activation, the carbonized material may be crushed, sized, and freed from extraneous materials such as sand, or large chunks of it may be fed directly to the activation stage. Activation is usually conducted in a directly fired rotary kiln, where the product is activated at between 800 and 1000°C in the presence of steam, air, flue gas, or mixtures of these, for a period of time.

The development of a highly porous product is generally associated with the degree of burn-off or loss in mass of the char, i.e. the extent to which the gasification reactions shown in equations (2) and (3) have been allowed to proceed.

In the activation of carbonaceous materials, there are at least five primary variables that can affect the surface area and pore-size distribution of the product and, hence, its adsorptive properties:

(1) the nature of the starting material or quality of the char,
(2) the composition of the activation atmosphere,
(3) the flowrate of the activating gases,
(4) the temperature of activation, and
(5) the duration of the burn-off period.

The interrelationship between the various activation parameters is complex, and therefore the qualitative representations given in Figs. 6 to 8 are only simplistic qualitative representations. The data presented, which are not accurate, serve only to indicate relative trends, and are equally applicable to any type of carbonaceous raw material.

The nature of the source material has a marked effect on certain properties of the final product, e.g. the pore-size distribution and hardness, as will be discussed in greater detail later. The composition of the activation atmosphere is most important, not only in terms of the reactions referred to earlier, but because of its effect on the nature of the surface oxides that form in the carbon during activation. This affects the hydrophobic/hydrophilic balance and, hence, the adsorptive behaviour and selectivity trends of the product.

Fig. 6 indicates that the yield of final product, which is inversely proportional to the extent of burn-off, decreases as the residence time in the kiln is increased, and is also markedly dependent on the temperature of activation, which obviously affects the rate of burn-off or gasification.

The activity of the product as measured by nitrogen BET adsorption is approximately proportional to the degree of burn-off up to a certain point, as is apparent from Fig. 7, and corresponds to the formation of microporosity in the product. However, a point is reached

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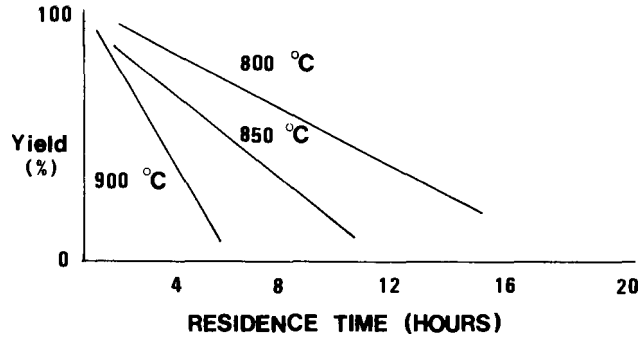


Fig. 6—Qualitative relationship between yield of activated carbon and residence time in the kiln at various temperatures

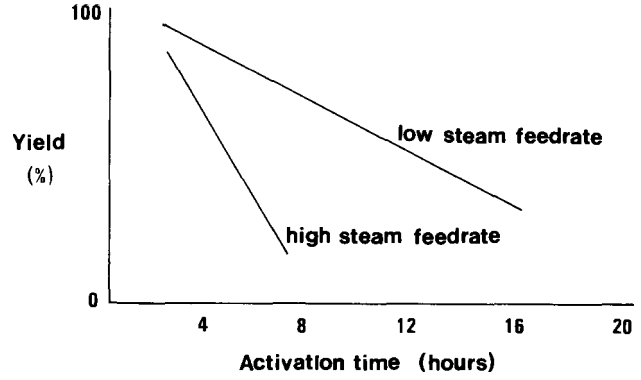


Fig. 8—Qualitative relationship between yield of activated carbon and residence time in the kiln at high and low feed rates of steam

where further burn-off results in a reduction in the nitrogen BET surface, and this phenomenon relates to the conversion of micropores into mesopores and macropores by the burning-away of the pore walls. There is usually a concomitant loss in the structural strength of the product.

Fig. 8 depicts the relationship between yield and residence time in the kiln as a function of the steam feed rate. In this case, it is apparent that higher steam feed rates enhance the rate of burn-off, thereby decreasing the yield of final product.

Once the desired level of activity has been achieved, the product is cooled to below the ignition point of carbon in air (usually less than 400°C), and bagged directly or after further crushing and sizing.

Because of the adsorptive properties of activated carbon, it is important that the product should be sealed from the atmosphere. Activated carbon is therefore usually packed directly into polyethylene bags and sealed before being placed in a second, stronger bag made of a material such as woven polypropylene.

Characteristics of Activated Coconut-shell Carbon

One of the most important characteristics of activated carbon made from coconut shells is that it is extremely microporous, the major portion of its pore volume being represented by pores with a radius of 10 Å, as can be seen from the pore-size distribution curve shown in Fig. 9. For comparative purposes, a curve of typical pore-size distribution for a chemically activated wood carbon is also shown; this curve indicates the presence of a high content of mesopores and macropores.

For this reason, coconut-shell products are ideal for the adsorption of small gaseous molecules that fit snugly

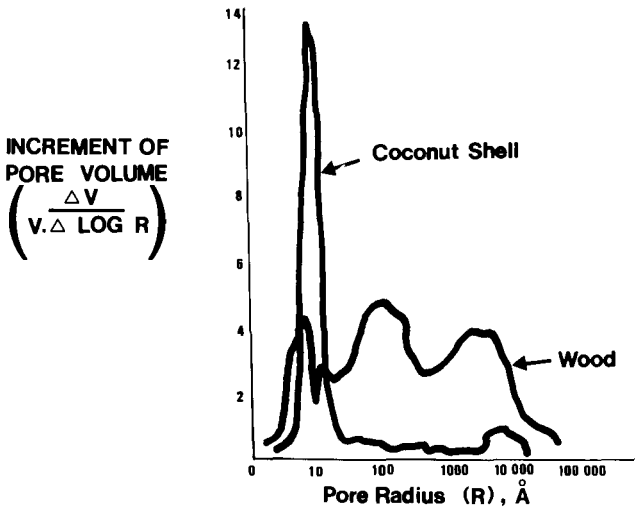


Fig. 9—Pore-size distributions in typical thermally activated coconut-shell carbon and chemically activated wood carbon

into the micropores, and are therefore extensively employed in gas-phase and solvent-recovery applications in general[6,8,10]. However, coconut-shell products generally fare poorly in applications involving the treatment of industrial effluents because the molecules that have to be adsorbed are often too large to penetrate into the microporous carbon matrix, and are therefore adsorbed less efficiently.

The extremely good impact hardness and wet abrasion resistance of coconut-shell carbons, combined with their

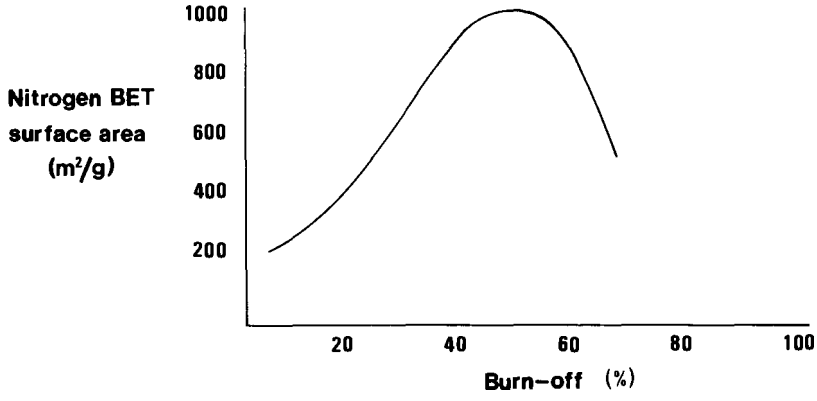


Fig. 7—Qualitative relationship between nitrogen BET surface area and loss in mass of char during activation

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high adsorptive capacity for the small gold–dicyanoaurate complex, makes these carbons ideal for applications involving gold recovery, particularly in carbon-in-pulp (CIP) and carbon-in-leach (CIL) processes.

The nature of the surface oxides formed in the carbon, and the activity of the product as measured, for instance, by the extent to which selected molecules from the gaseous or aqueous phase are adsorbed, are determined by the activation parameters, but the structure of the pores and the pore-size distribution are predetermined to a large extent by the source material[10,16]. This is apparent from photomicrographs of the pore structure of activated carbons manufactured from coconut shells and peach pips, and indicates that the structure of the original source material, viz the cellular structure of the coconut shell and the peach pips, is still present in the carbon skeleton of the final product[8].

Therefore, the inherent cellular structure of raw materials of vegetable origin imposes a constraint on the degree to which the properties of the product can be modified for special applications. For example, the micropores of the cellular structure of coconut shell can be converted into macropores by burning-away of the cell walls, thus changing the adsorption properties of the final product. Unfortunately, this type of modification is accompanied by a loss in the structural integrity and, hence, the strength of the product, as well as a decrease in product yield.

Process Routes Using Coal

Fig. 10 illustrates the various unit operations that are generally involved in the manufacture of granular activated carbons from coal[17].

Process routes using coal as the starting material are significantly more complex than those generally employed in the manufacture of coconut-shell carbons, as is indicated by the large number of unit operations involved.

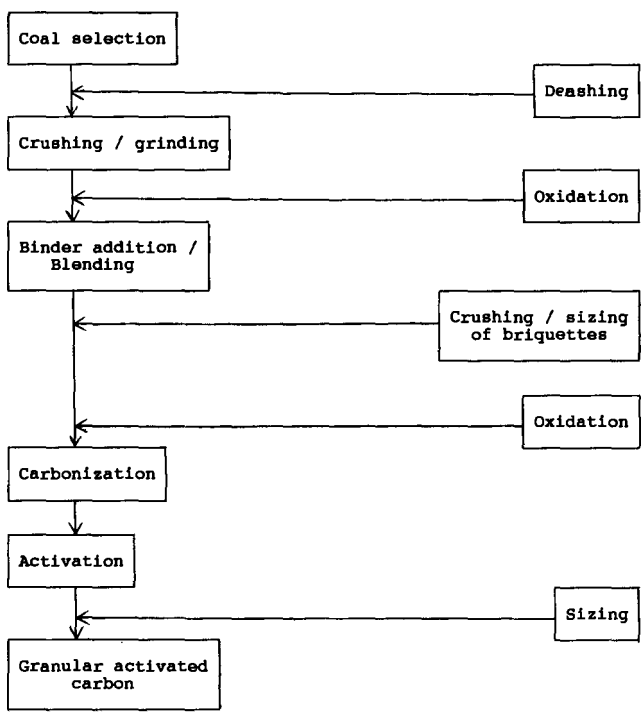


Fig. 10—Flowsheet showing the unit operations that are generally involved in the manufacture of granular activated carbons from coal (after Yehaskel[17])

Owing to the wide availability of abundant quantities of coal, particularly in the Western World, some of the largest commercial production facilities are based on coal as a raw material. However, the particular processing route employed depends mainly on the nature of the coal and the form in which the product is required (crushed or extruded), and hundreds of patents have been registered to describe the numerous process routes developed over the years for the manufacture of granular carbons from various ranks of coal (peat, brown coal, bituminous coal, lignite, coking coal, and anthracite)[17].

A low ash content is one of the most important requirements of a coal that is used for activated carbon. The major reasons for this relate to the fact that the ash content may catalyse certain undesirable activation effects, and is also believed to adversely affect the strength of the final product. Therefore, with high-ash coals, deashing with mineral acid may be necessary. The details of the other unit operations are discussed in the following sections.

Crushing, Grinding, and Oxidation

The inherent porosity of most ranks of coal is very low, and therefore the coal used as a starting material requires pretreatment to increase its porosity. The coal is initially crushed and milled by conventional means into a fine powder, after which it is reconstituted by briquetting or extrusion with the aid of a coking binder such as coal-tar pitch. This procedure may result in up to a tenfold increase in porosity over that of the coal as received from the mine. This porosity allows for uniform gasification of the coal granules during activation by providing channels that permit the oxidizing gases to enter the particle, while allowing the reaction products to escape.

Oxidation of the coal, either as a powder or after granulation, is often an important intermediate stage, depending on the nature of the coal. For example, the agglomerating property of a high-rank medium-volatile bituminous coal is destroyed if the coal is oxidized at a temperature below its fusion point, i.e. between 200 and 280°C. This treatment alters organic components in the coal, thereby rendering the particles non-agglomerative, and can be conducted either in a fluidized bed (coal powder) or in a rotary kiln (granules). The average residence time for oxidation varies between 10 minutes and 1 hour. Careful temperature control is critical because, above about 375°C, the characteristic particle shape is destroyed, and the particles may fuse. Steam is often injected into the kiln for purposes of temperature control.

Briquetting and Extrusion

Granular activated carbons can be crushed or pressed. The products of crushed granular coal are produced by briquetting, the briquette being crushed and sized prior to oxidation or carbonization, or only after activation. The particle-size range is usually between 0,2 and 3,5 mm.

The second type of granular carbon, viz pressed or extruded carbon, is usually produced in uniform cylindrical shapes. These are always formed prior to carbonization and activation. In the process, the coal powder

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is blended with a coking binder, usually coal-tar pitch, although numerous other types of binders are also employed commercially, at a temperature above the softening point of the pitch. The resultant plastic mass is then extruded from a die, and the rod is cut into pieces of uniform length. The diameter of the cylinders usually varies between 0,8 and 4,0 mm, and the length is from one to three times greater than the diameter.

Carbonization and Activation

General aspects related to carbonization and activation are no different from those discussed previously for coconut shells, and are therefore not repeated here.

Characteristics of Coal-based Activated Carbons

Coal-based activated-carbon products are characterized by a tridisperse pore-size distribution, i.e. they contain micropores, mesopores, and macropores, as is apparent from Fig. 11. For this reason, they constitute a very versatile range of products and are generally employed in a wide variety of aqueous-phase applications requiring the removal of a range of molecules varying significantly in molecular size, e.g. in the treatment of effluent water.

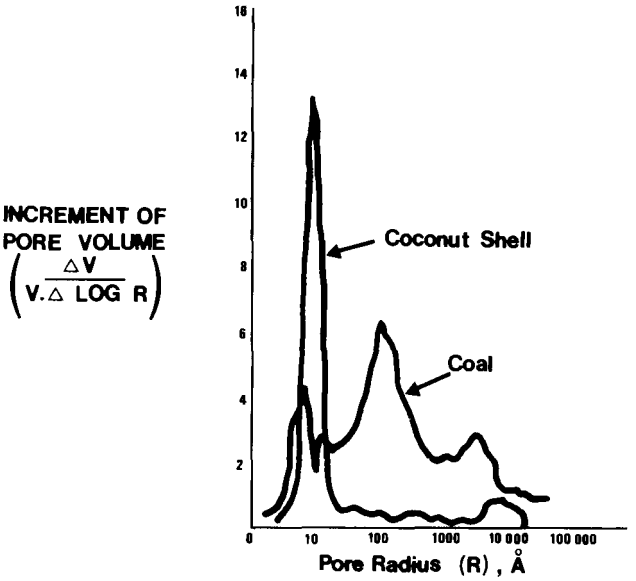


Fig. 11—Pore-size distributions for a typical thermally activated coal-based carbon and a coconut-shell carbon (after Fornwalt *et al*[18])

When cognizance is taken of the many variables involved in the manufacture of coal-based products (the rank of the coal, the binder chosen, the briquetting or extrusion technology used, the combination of activation parameters chosen, the fineness of grind employed, etc.), it is obvious that the scope for modification of the product is significant. For example, coal-based products cannot be modified to simulate the properties of coconut-shell or wood-based products, although any modifications between these extremes are possible.

It is also generally accepted that the abrasion resistance and impact hardness of these reconstituted products, based on current manufacturing technology, is with few exceptions not as good as those found in products using coconut shells as the raw material.

Selection of an Activated Carbon for Gold Recovery

The primary requisites[19] for a granular activated carbon intended for use in gold-recovery applications are as follows:

(a) the carbon must have a high gold activity, and
(b) the carbon must be tough and resistant to abrasion, for this will minimize the loss of gold associated with fine abraded carbon in the tailings.

Since activated carbons manufactured from coconut shells best meet the above requirements, they are widely employed in gold-recovery applications. Other important parameters relate to particle size (the rate of gold extraction increases with decreasing particle size), the initial and equilibrium gold concentrations, the surface area of the product, the pH value and temperature of the adsorption medium, etc.[12,13,19].

Conclusions

The main objective in the manufacture of granular activated carbon is the development of an optimum pore structure associated with a high surface area—with minimum loss of the carbon content through carbonization and oxidation—and of a product with sufficient structural strength to withstand normal usage without excessive attrition of the particles.

The manufacture of high-quality activated-carbon products is significantly more complex than has been outlined here because of the number of variables involved in the manufacturing process, and the complex interrelationship between those variables. As discussed, when unsophisticated methods of manufacture are employed, each raw material tends to produce its own characteristic type of pore structure. Nevertheless, this natural tendency can be altered considerably by special treatment requiring considerable effort and know-how.

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Mine valuation and grade control

The South African Institute of Mining and Metallurgy will be holding a 2-day school on 6th and 7th June, 1991, on the topic **Mine Valuation and Grade Control.**

Objective

The objective of this School is to present the practical application of valuation and grade control as they apply to the accounting for metal and grade of broken ore from the *in situ* to the final saleable product.

The latest developments in this field relating to the mining of precious metals, coal, and base minerals will be covered.

Programme

Sessions will be held on the following topics:

- Reserves (Chairman: Dr I. Clark)
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- Stockpiling (Chairman: Mr N. de Wet)
- Plant (Chairman: Mr J. Shirley)
- Quality Control (Chairman: Mr D.J. van Niekerk).

Contact Address

Further information is available from

Pamela Smith
SAIMM
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Marshalltown 2107.
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Mine productivity and safety

The South African Institute of Mining and Metallurgy will be holding a one-day colloquium devoted to **Communications and Monitoring to Improve Mine Productivity and Safety.**

This Colloquium will cover a range of topics dealing with the following systems planned or in operation in underground and surface mines:

- Voice communications and telemetry underground
- Rock and personnel tracking
- Environmental monitoring and alarm
- Rope-load monitoring
- Equipment-condition monitoring
- Shaft management.

The Colloquium will be held on 21st May, 1991, at the BIFSA Conference Centre, Halfway House, Midrand.

Enquiries should be addressed to

Pamela Smith
SAIMM
P.O. Box 61127
Marshalltown 2107.
Tel.: (011) 834-1273/7; Fax: (011) 838-5923
Telex: 4-86431.

Bacterial oxidation

The South African Institute of Mining and Metallurgy will be holding a colloquium on **Bacterial Oxidation** in June 1991 in Johannesburg.

The papers will be published in a bound volume, which will be available to all delegates on registration at the Colloquium. The language of the Colloquium will be English.

The final circular will contain detailed information on the Colloquium, as well as the registration forms.

All correspondence should be addressed to

Pamela Smith
SAIMM
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Marshalltown 2107.
Tel.: (011) 834-1273/7; Fax: (011) 838-5923
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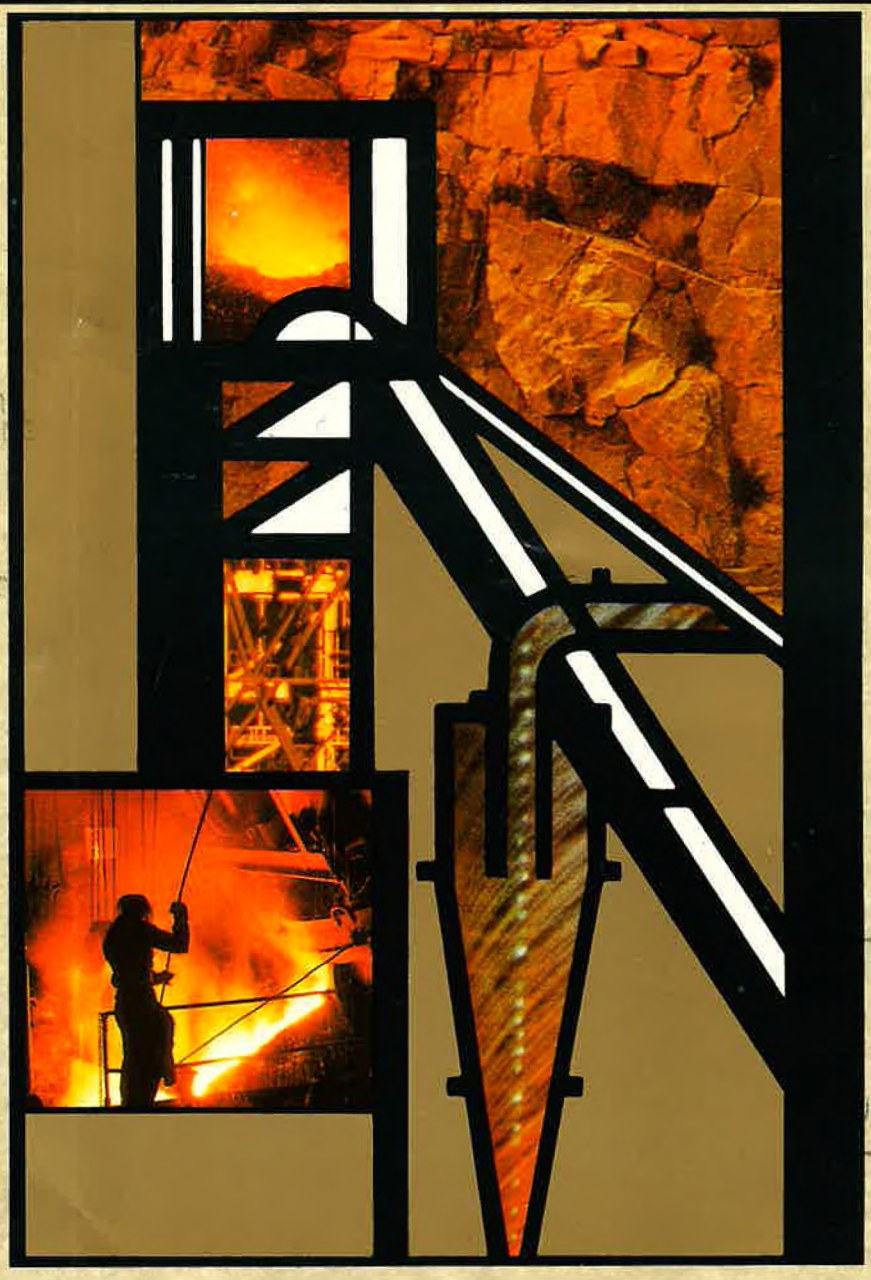
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The physical nature and manufacture of activated carbon

by G.J. McDOUGALL*

SYNOPSIS

After defining activated carbon, the author describes its structure and outlines the physical characteristics distinguishing one type of activated carbon from another. The adsorptive properties of these carbons, the raw materials used, and the manufacturing processes—chemical activation, and physical or thermal activation—are outlined. The high-temperature thermal route (which is the most important for the products employed in gold recovery) using coconut shells or coal as the raw material is then discussed in some detail.

SAMEVATTING

Na 'n omskrywing van geaktiveerde koolstof bespreek die skrywer die struktuur daarvan en gee in hooftrekke die fisiese eienskappe wat een soort geaktiveerde koolstof van 'n ander onderskei. Die adsorpsie-eienskappe van hierdie koolstowwe, die grondstowwe wat gebruik word, en die verwaardigingsprosesse—chemiese aktivering en fisiese of termiese aktivering—word in hooftrekke behandel. Die termiese hoëtemperatuurroete (die belangrikste roete wat betref die produkte wat in goudherwinning gebruik word) wat van klapperdoppe of steenkool as die grondstof gebruik maak, word daarna redelik in besonderhede bespreek.

Introduction

Carbonized wood, which can be regarded as a crude form of activated carbon by present-day standards, has been in use for many thousands of years. In about 1500 B.C., the Egyptians, who were aware of the adsorbent properties of wood charcoal, used it for medicinal purposes and as a purifying agent, as did the ancient Hindus, who filtered their drinking water through charcoal[1].

The decolorization and gas-adsorption properties of various carbon products were first investigated in the late nineteenth century, their development having gained impetus from the need for gas adsorbents to protect people against the poisonous gases used in World War I, and also from new applications that arose in the sugar industry and that are related to decolorization.

The ability of activated carbon to adsorb gold from chloride solutions was first reported[2] in 1847. This information attracted considerable interest and, in 1880, Davis[3] patented a process in which wood charcoal was used to recover gold from chloride leach liquors. This process was subsequently to become popular, particularly in Australia.

Shortly after the discovery by MacArthur and the Forrest brothers in 1890 that cyanide was a good solvent for gold[4], Johnson[5] (1894) patented the use of wood charcoal for the recovery of gold from cyanide solutions. Since then, major developments have occurred in the technology relating to the manufacture of activated carbons, which are currently employed commercially in a wide range of industries.

Important applications relate to the use of activated carbons to render water potable by the removal of taste, colour, odour, and undesirable organic impurities, in the treatment of domestic and industrial waste water, in the removal of colour from various types of sugar syrups, in a variety of gas-phase applications, and in the purification of many chemical and pharmaceutical products and foodstuffs. Activated carbon also recently found increased application in the field of hydrometallurgy, particularly in the recovery of gold, silver and, to a lesser extent, molybdenum[6–8].

Despite the commercial importance of activated carbon, little is known outside the industry itself about the raw materials that are suitable for conversion into activated carbon, the methods of manufacture, and the properties that make a product superior for a particular use and apparently unsuitable for another.

In South Africa, the gold-mining industry is the largest single consumer of activated carbon, and it was felt that a paper dealing with general aspects relating to the manufacture of activated carbons, as well as with the properties of the resultant products, would be of interest to personnel in the industry.

What is Activated Carbon?

Activated carbon is a generic term for a family of highly porous carbonaceous materials, none of which can be characterized by a structural formula or by chemical analysis[1]. The volume of the pores in activated carbons is generally defined as being greater than 0,2 ml/g, and the internal surface area is generally larger than 400 m^2/g as measured by the nitrogen BET method. The width of the pores varies from 3 Å to several thousand angstroms, and the pores are generally classified for convenience in terms of their diameters:

Macropores	500 to 20 000 Å
Transitional pores (mesopores)	100 to 500 Å
Micropores	8 to 100 Å.

In cross-section, the pores appear to be cylindrical or rectangular in shape, but can also occur in a variety of

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irregular shapes. Pores may also have constrictions or bottlenecks.

Macropores, which generally contribute very little to the total internal surface area of the product, depend mainly on the nature of the carbonaceous raw material employed and the preliminary manufacturing process, viz grinding and agglomeration of the raw material. Their major function is to serve as transport arteries that make the internal parts of the carbon granules readily accessible to the molecules being adsorbed. The transitional pores account for about 5 per cent, and the micropores for about 95 per cent, of the internal surface area. The micropores are largely the product of the activation process.

Activated carbons are commercially available in numerous forms: powders, granular chips in various size ranges, and shaped or moulded products extruded into rod-like shapes, which are commonly 0,8 to 6 mm in diameter by 3 to 10 mm in length.

Structure

X-ray-diffraction studies have shown that the structure of thermally activated carbon is similar to that of graphite[9,10]. Ideal graphite, as shown in Fig. 1, consists of layers of fused hexagons held approximately 3,35 Å apart by Van der Waals forces, so that the carbon atoms in any one plane lie above the centres of the hexagons in the layer immediately below it. The lattice is of the ABAB type.

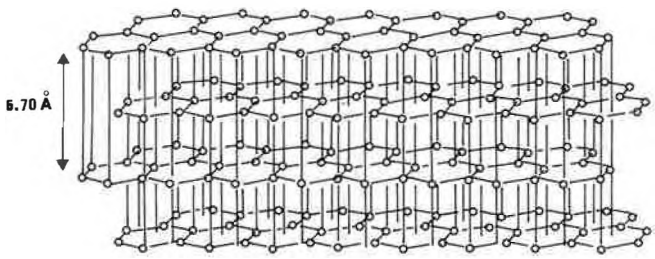


Fig. 1—Schematic representation of the structure of graphite; the circles denote the positions of carbon atoms, while the horizontal lines represent carbon-to-carbon bonds (after Bockris[9])

The proposed structure of thermally activated carbon is depicted in Fig. 2. The carbon is believed to be composed of tiny graphite-like platelets, only a few carbon atoms thick and 20 to 100 Å in diameter, which form the walls of open cavities of molecular dimensions, i.e. the pore system. However, the hexagonal carbon rings, many of which have undergone cleavage, are randomly orientated, and lack the directional relationship with one another that is present in single graphite crystals. The overall structure is therefore very disordered, and is often referred to as 'turbostratic'. Furthermore, the separation between the layers is greater than that found in graphite, viz 3,60 Å.

Because of the high level of structural imperfections in activated carbons, there are many possibilities for reactions with carbon atoms at the edges of the planar layers. As a result, oxygen-containing organic functional groups, which are located mostly at the edges of broken graphitic ring systems, are present on the surface of the carbon.

Although the exact chemical structures of these surface oxides are not known with certainty, it has been suggested that the surface oxides depicted in Fig. 3 are the oxides most often present in thermally activated carbons[10]. Basic surface groups have also been identified, and it appears that the nature of the surface groups depends upon conditions during and after manufacture[6].

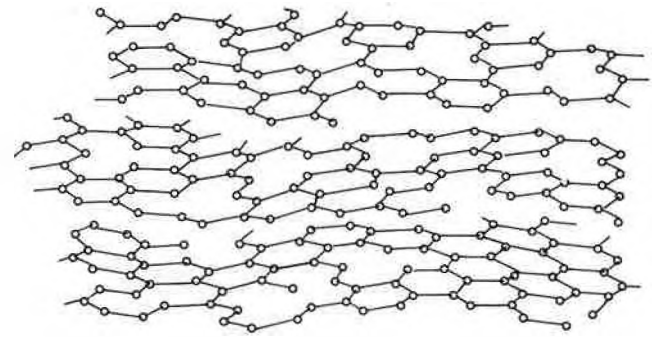


Fig. 2—Schematic representation of the proposed structure of activated carbon; oxygen-containing functional groups are located at the edges of broken graphitic ring systems (after Bockris[9])

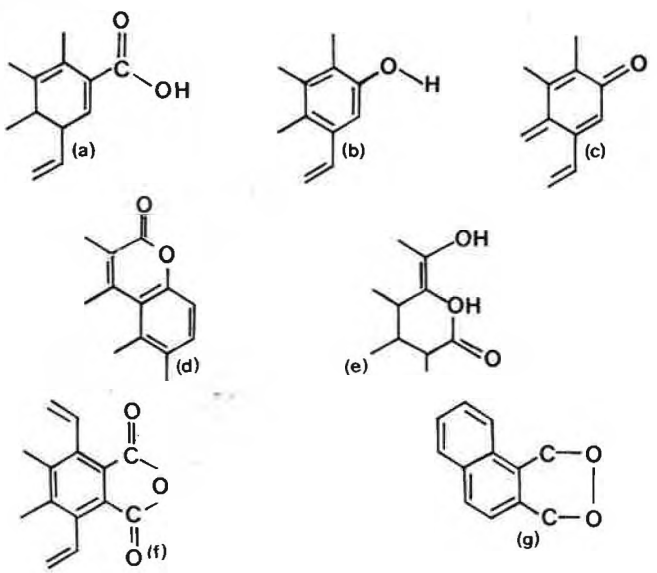


Fig. 3—Structure of some surface oxides[10]: (a) carboxylic acid (b) phenolic hydroxyl (c) quinone-type carbonyl groups (d) normal lactone (e) fluorescein-type lactones (f) carboxylic acid anhydrides (g) cyclic peroxides

Characterization of Activated Carbons

The different types of activated carbon can be distinguished from one another only by reference to certain physical and chemical properties.

Important physical properties are the number and size distribution of the pores, bulk density, dry impact hardness, wet abrasion resistance, and particle-size distribution. From the chemical point of view, comparisons are based on the ability of the different products to adsorb various selected substances from the gaseous phase, e.g. benzene, carbon tetrachloride, and nitrogen, as well as from the aqueous phase, e.g. iodine, molasses, phenol, methylene blue, and tannin.

For example, the nitrogen BET value expresses the surface area in square metres per gram of adsorbent that

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can be covered by nitrogen in a monomolecular layer. Activated carbons typically yield values between 400 and 1500 m^2/g, the former value corresponding to what is termed a low-activity product and the latter to a high-activity product. Such surface-area dimensions alone are insufficient to characterize a carbon product, simply because the nitrogen molecule is very small and can therefore penetrate into pores that are not accessible to other larger molecules. Hence, for the practical assessment of activated carbons, the adsorptive capability of the various products for compounds of large molecular dimensions, viz molasses, methylene blue, and tannin, is determined under standard experimental conditions.

The extent to which the various activated carbons adsorb molecules of a different size can be regarded as an indication of the distribution of their internal accessible volume among pores of different sizes. Hence, the small iodine molecule is adsorbed in pores down to 10 Å in diameter so that the iodine number is an indication of the surface area of the pores in that size range. At the other end of the scale, molasses, a large polysaccharide molecule, is adsorbed only in pores larger than about 30 Å in diameter, and the molasses number gives an indication of the number of pores in that size range[8].

A more sophisticated method by which information about the pore-size distribution can be gained involves measurement of the pressure required to force liquid mercury into the activated carbon. This, in turn, can be related to the size of the pore into which the mercury is being forced. The pore-size distributions shown in Figs 9 and 11 were obtained by this method.

Those physical and chemical properties most often employed by manufacturers of activated carbon to characterize their products are listed in Table I. Typical values are indicated for a high-activity coconut-shell carbon, which is activated thermally using steam, and which is usually employed in gas-phase applications and in processes for the recovery of gold from cyanide leach liquors.

TABLE I
PHYSICAL PROPERTIES AND CHEMICAL-ADSORPTION CHARACTERISTICS OF A THERMALLY ACTIVATED COCONUT-SHELL CARBON TYPICAL OF THOSE EMPLOYED IN GOLD-RECOVERY APPLICATIONS

Physical properties	
Particle density (mercury displacement)	0,80 to 0,85 g/ml
Bulk density	0,48 to 0,54 g/ml
Pore volume	0,70 to 0,80 ml/g
Ball-pan hardness (ASTM)	97 to 99%
Particle-size distribution	1,18 to 2,36 mm
Ash content	2 to 4%
Moisture content	1 to 4%
Chemical-adsorption characteristics	
Surface area (nitrogen BET)	1050 to 1200 m^2/g
Iodine number*	1000 to 1150 mg/g
Carbon tetrachloride number†	60 to 70%
Benzene number	36 to 40%

* The iodine number indicates the mass of iodine adsorbed out of aqueous solution under standard experimental conditions by 1 g of activated carbon

† The carbon tetrachloride number indicates the mass of carbon tetrachloride vapour adsorbed under standard experimental conditions by 1 g of activated carbon, expressed as a percentage

Adsorptive Properties

It is generally conceded that activated carbon owes its adsorptive properties primarily to its large internal surface area, as well as to its pore-size distribution, and that the external surface area and the nature of the surface oxides play minor roles[6,10]. The size of the pores developed during activation therefore has an important influence on adsorption behaviour because the pores act as a screen. This screen prevents the adsorption of large molecules, but promotes the adsorption of an adsorbate that fits snugly into the pores, making the maximum number of contacts with the adsorbed molecule, and thus maximizing ΔH as defined in equation (1):

$$\Delta H = \Delta G + T\Delta S, \quad \text{..... (1)}$$

where

ΔH is the change in free enthalpy,
ΔG is the change in free energy,
ΔS is the change in entropy, and
T is the temperature of the system.

Fig. 4 attempts to illustrate this concept for the case in which two adsorbate molecules in a solvent, which is not shown, compete with each other for adsorbent surface or sites. The external surface can provide only a certain amount of access to the inner pores, and it is likely that the main role of the surface oxides is to impart a hydrophilic character to the predominantly hydrophobic carbon skeleton. This would account for the affinity of activated carbon for many polar and non-polar organic and inorganic species[8].

Activated carbon functions by adsorption, i.e. by the adhesion of certain substances to the internal surface constituting the walls of the pores. Hence, the greater the adsorption surface available, the better is the adsorption function.

Adsorption occurs as the result of an imbalance of the forces that act upon the carbon atoms constituting the surface of the pore wall. Such an imbalance is inherent in all surfaces and, in an attempt to rectify it, molecules are adsorbed from the gaseous or aqueous phases, and are attracted and held to the surface. Surface adsorption, regardless of the energy of the interaction, must always proceed with a negative change in free energy (ΔG), as well as a decrease in entropy (ΔS), which, according to equation (1), must result in a negative change in free enthalpy (ΔH). Hence, adsorption is always an exothermic process[10].

There are two types of adsorption process: physical and chemical adsorption. Physical adsorption involves weak Van der Waals forces (also dipole–dipole interactions and hydrogen bonding), and the processes are generally reversible. Chemical adsorption refers to processes involving homopolar forces (as in ionic or covalent bonds), and such processes are generally irreversible. Nevertheless, in most instances, the adsorptions can be classified as being physical in nature.

In general, the factors that influence the adsorptive behaviour of activated carbon from aqueous solution are temperature (adsorption generally decreases with increasing temperature); the pH value of the solution (carbon generally has a low affinity for ions, particularly those with a high charge-to-surface ratio, and pH can affect

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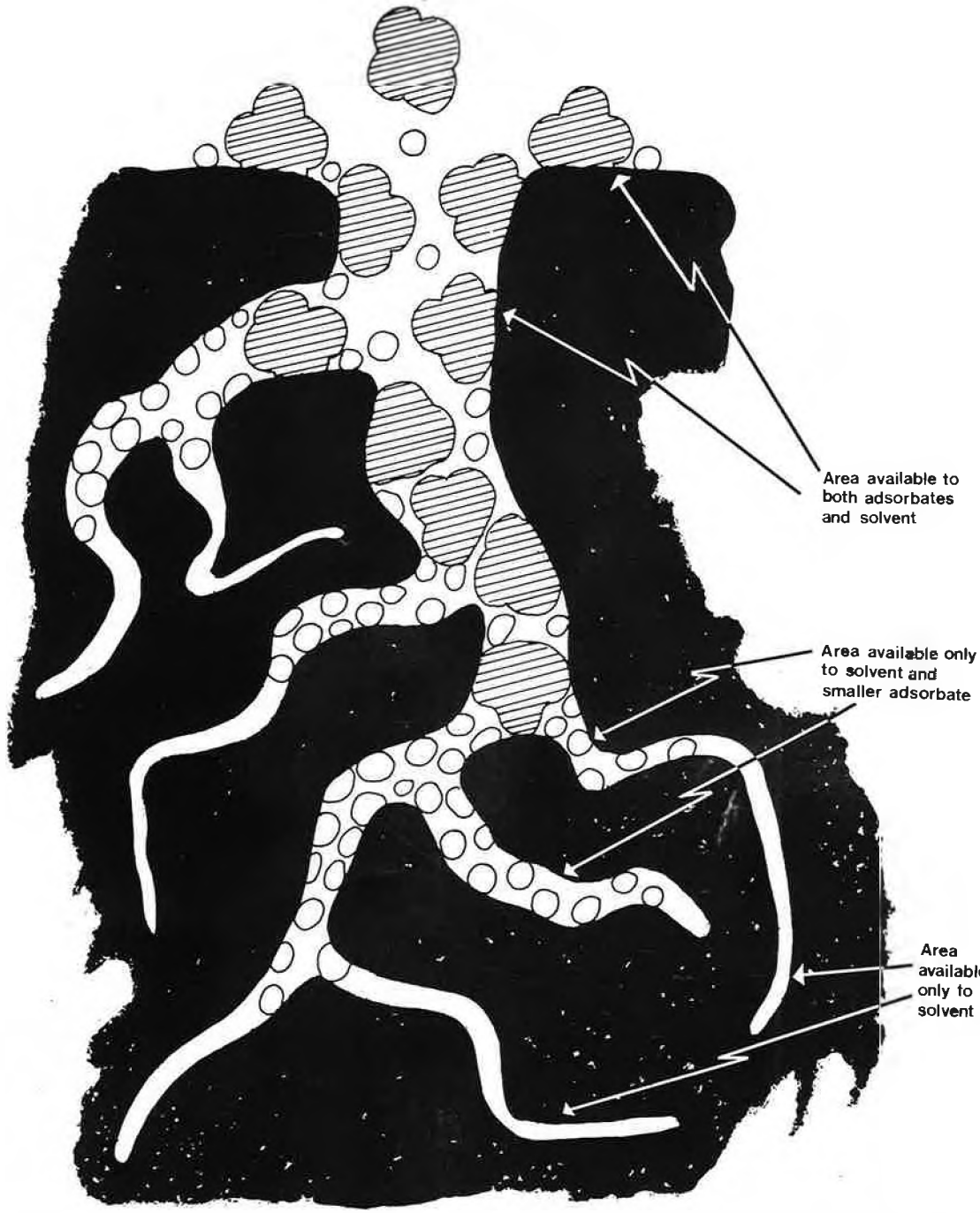


Fig. 4—Concept of molecular screening in micropores (after Pittsburgh Activated Carbon Brochure[11])

ionicity); the chemical nature of the species present and their relative concentration; and the nature of the activated carbon and its particle-size distribution. For molecules to be adsorbed, they must reach the internal surface of the macropores by diffusion. Therefore, the reaction time will be influenced by the length of the diffusion path, and the kinetics of adsorption will increase with decreasing particle size.

Activated carbon is a very versatile substrate with respect to its interactions with various organic and inorganic compounds. It is able to function as a simple adsorbent that is akin to synthetic polymeric adsorbents, since molecular compounds can be physically adsorbed onto its large internal surface area, and be retained there by forces of the Van der Waals type[7,8]. It can also function as a reducing agent or, in the presence of excess oxygen, as an oxidation catalyst[1,7,10]. As a result, the adsorption of organic and inorganic species onto carbon may occur by several mechanisms, and their identification, particularly in the extraction of certain metal complexes from solution, is extremely difficult as in the extraction of the gold dicyanoaurate anion[12–14].

Because carbon is generally a non-polar material, it adsorbs organic compounds in preference to polar inorganic species. For example, small highly charged cations such as Ni^{2+} and anions such as Cl^- are not adsorbed by activated carbon to any significant extent[13].

The adsorption of organic species onto carbon usually

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occurs by simple mechanisms that are fairly well understood in terms of a balance between hydrogen-bonding forces in solution and Van der Waals attraction between the carbon surface and the organic compounds. The driving force for the adsorption of organic molecules onto carbon is similar to that responsible for the salting-out of organic materials dissolved in water, where the addition of a simple inorganic salt causes the organic material to separate out from the aqueous phase[8].

These considerations have given rise to the statement frequently made that adsorption onto activated carbon is a fight against solubility. Applications involving organic materials will therefore be most attractive when the adsorbate has high molecular mass, low polarity, and a low degree of ionization, because these properties generally signify low aqueous solubility. Hence, activated carbons are excellent adsorbents for a considerable number of aliphatic compounds, and particularly for aromatic compounds like those commonly found in pesticides and herbicides. As such, activated carbons are employed extensively, not only in commercial applications that require the removal of contaminating organic compounds from aqueous effluents, but also in purification and recovery processes[6,10].

Because granular activated carbons are expensive, their application is usually economically viable only if the carbon can be regenerated and therefore recycled in the process.

The regeneration of granular activated carbon is generally accomplished using steam, hot solvents, or thermal routes. Regeneration by extraction with steam can be applied if the carbon contains substances that volatilize at temperatures below 150°C. For substances of higher boiling points that pyrolyse on heating, thermal reactivation at 700 to 950°C is employed. Steam is usually injected into the kiln, and any pyrolysed organic substances, i.e. non-volatile carbonaceous materials, that may be present in the carbon are burnt away by the reaction shown in equation (2):

$$C + H_2O \rightleftarrows CO + H_2 \ (\Delta H = 29 \text{ kcal/mol}). \ .. \ (2)$$

This treatment cleans the pyrolysed contaminant carbon from the walls of the pores, and returns the carbon to its original activity. It should be noted that the pyrolysed residue is indistinguishable from the original carbon, and care should therefore be taken to ensure that not too much of the original carbon matrix is burnt away during thermal reactivation.

Regeneration can also be accomplished by treatment of the spent carbon with a solvent in which the adsorbate is highly soluble. The preferred route will depend on whether recovery of the adsorbate is desired or not. If it is, regeneration using steam or solvent extraction is preferred.

Manufacture of Activated Carbons

The most important step in the production of activated carbon is activation. This is the process by which the chosen raw material or carbonized raw material is converted into a finely crystalline form of carbon that is permeated by the greatest possible number of randomly distributed pores of various shapes and sizes. These pores give rise to the extended internal surface area. In the manufacture of activated carbon, it is desirable to develop the maximum accessible surface area that is commensurate with economic viability and product application[10,15-17].

The products of simple coking or carbonizing (such as that used in the early days) do not meet present-day standards. This can be attributed to the fact that the available surface area of pulverized charcoal, which is essentially external surface area, is only 2 to 4 m^2/g—as against typically 1000 m^2/g for a granular activated-carbon product—measured by the nitrogen BET method.

Raw Materials

Activated carbon can be manufactured from any carbonaceous raw material, and numerous patents have been granted for processes using such diverse starting materials as various ranks of coal (peat, lignite, bituminous coal, and anthracite), coconut shells, peach and apricot stones, waste tyres, wood, sawdust, petroleum heavy oil, cellulose, rice husks, corn cobs, sugar, bones, etc.[10,16,17]. All the starting materials used in relatively large-scale manufacture are readily available and, hence, are reasonably priced[15]. In the Western World, the companies entering the business in the past 30 years have tended to concentrate on using coal (mostly lignite, bituminous coal, and anthracite), whereas, in the developing world, new entrants have concentrated on using coconut-shell charcoal.

Since the manufacturing process involves the removal of volatile matter from the raw material, the economic relationship between the price, availability, and quality of the raw material on the one hand, and its volatile content on the other, is important. For example, one of the advantages of bituminous coal and anthracite is their relatively low volatile content (as shown in Table II) and, hence, their high yield of product[15].

TABLE II
FIXED CARBON CONTENT OF RAW MATERIALS COMMONLY EMPLOYED IN THE MANUFACTURE OF ACTIVATED CARBON (AFTER HOLDEN[12])

Material	Approximate carbon content, %
Soft wood	40
Hard wood	40
Coconut shell	40
Lignite	60
Bituminous coal	75
Anthracite	90

In the developing world, a major consideration is the availability of waste materials such as coconut shells and rice husks, which, despite having very high volatile contents and, hence, giving low yields of activated carbon, are so inexpensive that the economics involved in their use are often favourable.

Although the activation procedure and the after-treatment employed mainly determine the chemical nature of the surface oxides and the surface area of the resultant product, the structure of the pores and the pore-size distribution are largely predetermined by the nature of the starting material.

This paper focuses on the various steps involved in the

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manufacture of activated carbon from coconut-shell charcoal and from coal, as well as on the properties of the resultant products.

Chemical Versus Physical Manufacturing Processes

Although the general outlines of the various manufacturing processes are well known, the exact details of the procedures employed by the manufacturers are jealously guarded secrets. In general, the methods most commonly employed for the manufacture of activated carbon are of two main types: the wet-chemical process (basically a single-stage process), and the physical or thermal–gas process (essentially a two-stage process).

As would be expected, chemical methods use chemicals for activation, whereas thermal processes use gases (usually carbon dioxide and air), vapours (steam), or mixtures of gases and vapours[15-17].

Chemical Activation

The wet-chemical process is generally employed to convert uncarbonized cellulosic material, primarily wood, into activated carbon. The most popular activating agent is phosphoric acid, although zinc chloride and sulphuric acid are also well-known agents. Other chemicals also suggested for use include sodium carbonate, sodium and calcium hydroxide, and the chloride salts of magnesium, calcium, ferric iron, and aluminium. These chemicals have in common only their strength as dehydrating agents.

The process using sawdust as the starting material involves mixing of the raw material and the dehydrating agent into a paste. The paste is then dried and carbonized in some type of kiln, usually a rotary kiln, at between 200 and 650°C. Upon calcination, the impregnated chemicals dehydrate the raw material, resulting in charring and aromatization of the carbon skeleton, with the concomitant creation of a porous structure and extended surface area.

This procedure yields a power product. If a granular product is required, granular materials are simply impregnated with the activating agent, and the same general procedure as that described above is followed. However, these granular products are generally soft (unless they are manufactured from the powder after suitable pelletization), which limits their application.

After carbonization of the paste, the activating agents are usually recovered and recycled for economic reasons. All the commonly used chemical activators impose certain constraints on the overall activation process and the equipment employed, or on the quality of the product. Mixtures of sulphuric acid and wood cannot be heated above 200°C, and the product, after the sulphuric acid has been recovered by leaching, has adsorptive properties only when wet. Activation with phosphoric acid requires temperatures in the range 375 to 500°C, and the reagent is readily recovered by leaching with water. However, corrosion of the equipment is a major problem. Activation with zinc chloride occurs in the temperature range 550 to 650°C and, although most of the zinc chloride is recovered by leaching with dilute hydrochloric acid, problems may arise in the application of the product because it retains traces of zinc salts. The activated carbon produced with phosphoric acid and zinc chloride can be dried with little, if any, loss of adsorptive capacity.

The activity of the product can be controlled by alteration of the proportion of raw material to chemical reagent. For phosphoric acid, the proportion is usually between 1:0,5 and 1:4. The activity, as measured by the number of pores created, increases as the proportion of the chemical activator increases, and is also affected by the temperature and residence time in the kiln.

Physical or Thermal Activation

The first step in the production of activated carbon by the thermal route is carbonization, which is the formation of a char from the source material. Carbonization is generally accomplished by heating of the source material—as lumps or pre-sized material, or in moulded form, e.g. as briquettes or extrudates—in an inert atmosphere such as flue gas to a temperature that must not exceed 700°C so that dehydration and devolatilization of many of the carbon atoms can occur in a controlled manner. The main purpose of carbonization is to reduce the volatile content of the source material (a fixed carbon content of 80 per cent or higher is desirable) to convert it to a suitable form for activation. Rearrangement of the carbon atoms into graphitic-like structures also occurs during carbonization.

As previously mentioned, the process by which the carbonized product develops an extended surface area and a porous structure of molecular dimensions is referred to as *activation*. This step is generally conducted at temperatures between 800 and 1100°C in the presence of a suitable oxidizing agent such as steam, air, carbon dioxide, or any mixture of these gases. The active oxygen in the activating agent burns away the more reactive components of the carbon skeleton as carbon monoxide and carbon dioxide, depending on the oxidizing agent employed. Although the burning-out of the carbon skeleton to create new pores is believed to occur at different rates on different parts of the exposed surface, the detailed mechanism of this process is not clearly understood.

According to Smisek and Cerny[16], gasification of the carbonaceous material by steam and carbon dioxide occurs in accordance with the endothermic reactions shown in equations (2) and (3):

$$C + H_2O \rightleftharpoons CO + H_2 \quad (\Delta H = 29 \text{ kcal/mol}). \quad (2)$$

$$C + CO_2 \rightleftharpoons 2CO \quad (\Delta H = 39 \text{ kcal/mol}). \quad (3)$$

The reaction of water vapour with carbon is accompanied by the secondary reaction of water–gas formation, which is catalysed by the carbon surface as shown in equation (4):

$$CO + H_2O \rightleftharpoons CO_2 + H_2 \quad (\Delta H = -10 \text{ kcal/mol}). \quad (4)$$

Because the reactions of carbon with steam and carbon dioxide are endothermic, the activation process lends itself to accurate control of the conditions in the kiln. External heating is required to drive reactions (2) and (3), and to maintain the reaction temperatures. However, as shown in equation (5), the reaction of carbon with air (oxygen) is extremely exothermic:

$$C + O_2 \rightleftharpoons CO_2 \quad (\Delta H = -97 \text{ kcal/mol}). \quad (5)$$

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This reaction is difficult to control, and excessive burn-off of the external carbon surface can easily occur, resulting in a decrease in average particle size, which obviously reduces the yield of final product. The reaction of steam with carbon is catalysed by certain chemicals (e.g. the oxides and carbonates of alkali metals, iron, copper, and other metals), and some commercial operations use these chemicals as catalysts. Therefore, as combustion proceeds, preferential etching occurs, which results in the development of a large internal surface area and the creation of a pore structure.

The art of the manufacturer of activated carbon lies in conducting the activation process in such a way that combustion of the carbonized raw material occurs internally, and not from the exterior of the granules. The term *burn-off* is used to denote the degree of activation, which is the loss of char (in percentage by mass) that is allowed to occur. The burn-off (B) and activation yield (Y) are related as indicated in equation (6):

$$B = 100 - Y. \quad (6)$$

Carbonization and activation used to be carried out separately, but there is currently an increasing tendency for the two processes to be conducted in the same kiln. A wide variety of furnaces and kilns are used for carbonization and activation, including rotary kilns fired directly or indirectly, multiple-hearth furnaces, fluidized-bed reactors, and vertical retorts, each of which has advantages for a particular purpose. The indirectly fired rotary kiln, for example, is relatively large and expensive, but avoids a large discharge of inert gases to the afterburner. The fluidized-bed reactor is extremely compact, but is generally used only for the activation of abrasion-resistant raw materials because of the attrition that occurs during fluidization.

The effect of the heat treatment of a carbonaceous material on its resultant structure is as follows[16]. As the temperature of carbonization increases, polymerization occurs and, irrespective of whether the starting material is composed of long-chain aliphatic or aromatic molecules, a system of planar condensed benzene-ring molecules is formed. A solid then forms (this is possible at a range of setting temperatures) as a result of the formation of C – C bonds between neighbouring planes. From 400 to 700°C, these condensed ring systems grow gradually, but all the peripheral carbon atoms are attached by chemical bonds to hydrogen atoms or to hydrocarbon groups. Hence, these substances are termed condensed molecular solids, and have high electrical resistivity, which is typical of chemically activated carbons. However, between 700 and 800°C, many of the hydrogen atoms or hydrocarbon groups are expelled from the material, leaving small crystallites having a structure similar to that of graphite. The evolution of gases coincides with a drastic reduction in electrical resistivity.

Moulded forms of the raw material, e.g. briquettes or extrudates, are obtained as follows. The wood charcoal, coal, or peat used in the raw material is pulverized, and the powder is mixed with a coking binder. The hot mixture is then pressed into briquettes, or is extruded through dies into rod-like shapes of a selected diameter. Powder carbons are obtained by the crushing and grinding of granular material.

If cognizance is taken of the many combinations of source material and conditions for carbonization and activation that can be employed in the manufacturing process, it is apparent that a large number of activated-carbon products can be obtained. Because, as previously discussed, no clearly defined chemical formulae can be determined for these products, the only way in which they can be distinguished from one another is by reference to selected physical and chemical properties.

Properties of Activated Carbons

The most significant differences between chemically and thermally activated carbons relate to pore-size distribution (Fig. 9) and semiconductor properties.

Chemically activated carbons are generally characterized by a macroporous pore-size distribution, and these products therefore generally have a high molasses number and comparatively low nitrogen BET and iodine numbers[18]. Such activated carbons are used mainly in applications that require the removal of large molecules from solutions, e.g. in the decolorization of sugar syrups. The absence of semiconductor properties in chemically activated carbons almost certainly relates to the fact that the temperature generally employed in the manufacture of these carbons is too low for the creation in the final product of graphitic zones, which are presumably responsible for the semiconductor–redox properties of thermally activated products[14,19].

The reduction potential of thermally activated carbons, measured[12] by a graphite-rod technique, has been found to be typically above $-0,14$ V versus the saturated calomel electrode at a pH value of 6. These carbons can therefore function as reducing agents, and many of the intriguing properties of thermally activated carbons, particularly their redox and catalytic properties, are believed to be related to their semiconductor properties.

It is interesting to note that chemically activated carbons have a poor affinity for the gold dicyanoaurate anion. This may be indicative of the important role played in the mechanism of gold extraction from a cyanide medium by the graphitic zones that are believed to exist in thermally activated carbon[14,20].

Since the high-temperature thermal route is employed most often for the manufacture of granular or shaped activated carbon products, and is also the most important route for the manufacture of products suitable for use in gold-recovery applications, that method will be focused on here. Furthermore, any detailed discussion of typical process routes for the preparation of activated carbon will be confined to those using coconut shells and coal as raw materials.

Process Route Using Coconut Shells

A typical flowsheet for the manufacture of activated carbon from presized or unsized char is depicted in Fig. 5. A similar route would be used for the manufacture of activated carbon from peach and apricot stones, or from any other hard, abrasion-resistant char, e.g. hard woods, which have sufficient inherent porosity to permit the activating agent to penetrate into the interior of the granule and simultaneously allow the products of the oxidation–gasification reactions to escape.

Details pertaining to the important unit operations that

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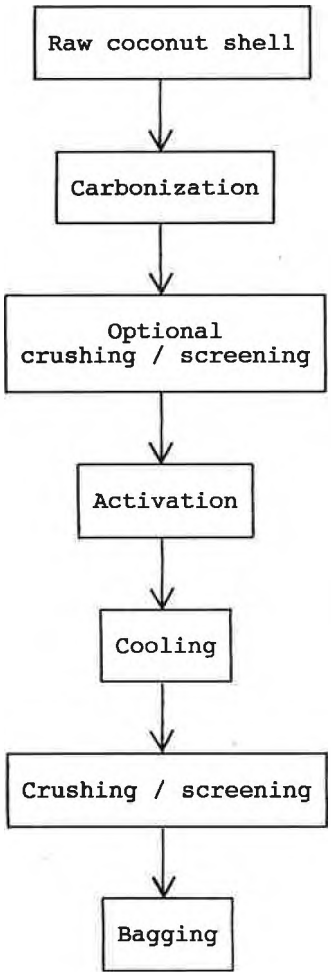


Fig. 5—Flowsheet for the manufacture of activated carbon from coconut-shell char (after Yehaskel[17])

constitute the process are discussed below.

Carbonization

A large quantity of coconut shells are available in countries such as the Philippines, Sri Lanka, and the South East Asian Islands as a byproduct of the coconut-oil and desiccated-coconut industries. Since coconut shells are eminently suitable for the production of very hard granular activated carbon, a viable industry for the manufacture of activated carbon has arisen in the Philippines and Sri Lanka. Because of its close proximity to the Philippines, Japan is a large importer of coconut-shell char and, hence, a manufacturer of activated coconut-shell carbon. As a matter of interest, the cigarette industry in Japan is the single largest consumer of activated coconut-shell carbon, since, in order to minimize the incidence of cancer due to smoking, the law requires fine carbon granules to be used in cigarette filters to adsorb the volatile organic materials emanating from the burning tobacco.

In the coconut-shell industry, carbonization is generally a small-scale, primitive, and crude operation that is conducted in the heart of the coconut plantations by individual farmers in, for example, 200-litre drums cut in half. Copra is placed in the bottom of the drum, which is then filled with coconut shells and ignited. The drum is covered with a sheet of metal, and the coconut shells are allowed to char for a couple of hours in an atmosphere that is essentially depleted of air. The char is sold to central depots, usually established by the various local manufacturers or exporters of char. Although charring is also sometimes done on a larger scale in pits, or simply in trenches dug in the sand on beaches, it is very much a cottage industry. Quality control in relation to char is conducted on the premises of the manufacturer using various techniques based on density separation.

As was mentioned earlier, carbonization is essentially a devolatilization step, which produces a char with a significantly higher content of fixed carbon (between 70 and 85 per cent) than is present in raw coconut shells.

Activation

Prior to activation, the carbonized material may be crushed, sized, and freed from extraneous materials such as sand, or large chunks of it may be fed directly to the activation stage. Activation is usually conducted in a directly fired rotary kiln, where the product is activated at between 800 and 1000°C in the presence of steam, air, flue gas, or mixtures of these, for a period of time.

The development of a highly porous product is generally associated with the degree of burn-off or loss in mass of the char, i.e. the extent to which the gasification reactions shown in equations (2) and (3) have been allowed to proceed.

In the activation of carbonaceous materials, there are at least five primary variables that can affect the surface area and pore-size distribution of the product and, hence, its adsorptive properties:

(1) the nature of the starting material or quality of the char,
(2) the composition of the activation atmosphere,
(3) the flowrate of the activating gases,
(4) the temperature of activation, and
(5) the duration of the burn-off period.

The interrelationship between the various activation parameters is complex, and therefore the qualitative representations given in Figs. 6 to 8 are only simplistic qualitative representations. The data presented, which are not accurate, serve only to indicate relative trends, and are equally applicable to any type of carbonaceous raw material.

The nature of the source material has a marked effect on certain properties of the final product, e.g. the pore-size distribution and hardness, as will be discussed in greater detail later. The composition of the activation atmosphere is most important, not only in terms of the reactions referred to earlier, but because of its effect on the nature of the surface oxides that form in the carbon during activation. This affects the hydrophobic/hydrophilic balance and, hence, the adsorptive behaviour and selectivity trends of the product.

Fig. 6 indicates that the yield of final product, which is inversely proportional to the extent of burn-off, decreases as the residence time in the kiln is increased, and is also markedly dependent on the temperature of activation, which obviously affects the rate of burn-off or gasification.

The activity of the product as measured by nitrogen BET adsorption is approximately proportional to the degree of burn-off up to a certain point, as is apparent from Fig. 7, and corresponds to the formation of microporosity in the product. However, a point is reached

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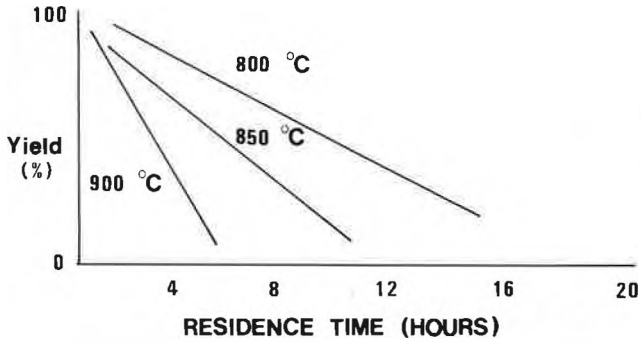


Fig. 6—Qualitative relationship between yield of activated carbon and residence time in the kiln at various temperatures

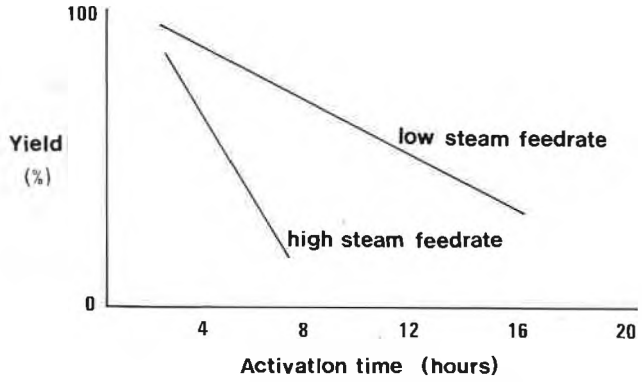


Fig. 8—Qualitative relationship between yield of activated carbon and residence time in the kiln at high and low feed rates of steam

where further burn-off results in a reduction in the nitrogen BET surface, and this phenomenon relates to the conversion of micropores into mesopores and macropores by the burning-away of the pore walls. There is usually a concomitant loss in the structural strength of the product.

Fig. 8 depicts the relationship between yield and residence time in the kiln as a function of the steam feed rate. In this case, it is apparent that higher steam feed rates enhance the rate of burn-off, thereby decreasing the yield of final product.

Once the desired level of activity has been achieved, the product is cooled to below the ignition point of carbon in air (usually less than 400°C), and bagged directly or after further crushing and sizing.

Because of the adsorptive properties of activated carbon, it is important that the product should be sealed from the atmosphere. Activated carbon is therefore usually packed directly into polyethylene bags and sealed before being placed in a second, stronger bag made of a material such as woven polypropylene.

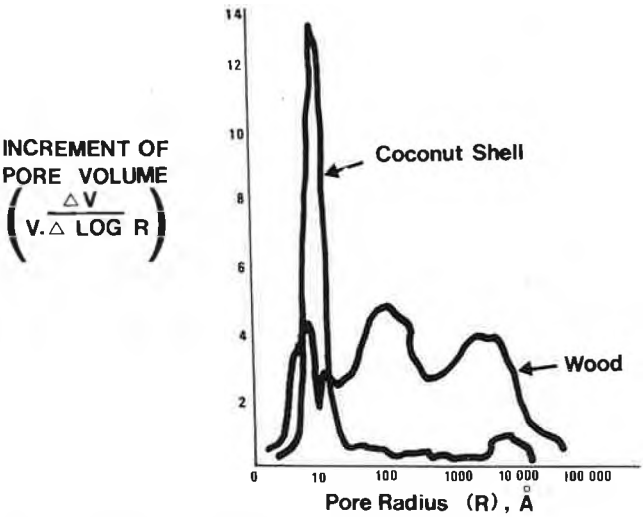


Fig. 9—Pore-size distributions in typical thermally activated coconut-shell carbon and chemically activated wood carbon

Characteristics of Activated Coconut-shell Carbon

One of the most important characteristics of activated carbon made from coconut shells is that it is extremely microporous, the major portion of its pore volume being represented by pores with a radius of 10 Å, as can be seen from the pore-size distribution curve shown in Fig. 9. For comparative purposes, a curve of typical pore-size distribution for a chemically activated wood carbon is also shown; this curve indicates the presence of a high content of mesopores and macropores.

For this reason, coconut-shell products are ideal for the adsorption of small gaseous molecules that fit snugly into the micropores, and are therefore extensively employed in gas-phase and solvent-recovery applications in general[6,8,10]. However, coconut-shell products generally fare poorly in applications involving the treatment of industrial effluents because the molecules that have to be adsorbed are often too large to penetrate into the microporous carbon matrix, and are therefore adsorbed less efficiently.

The extremely good impact hardness and wet abrasion resistance of coconut-shell carbons, combined with their

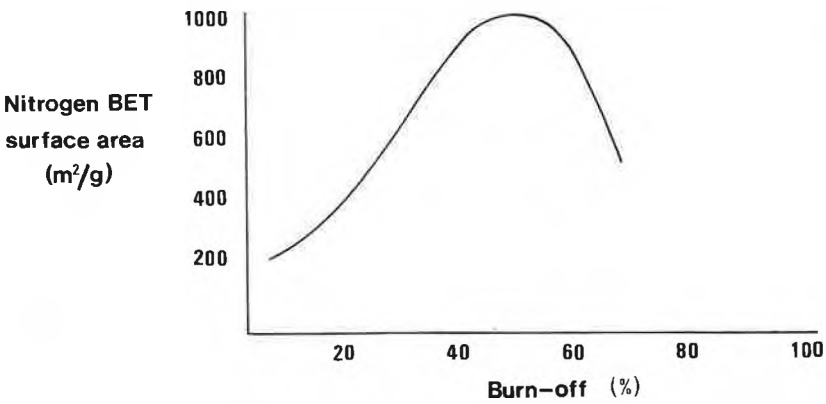


Fig. 7—Qualitative relationship between nitrogen BET surface area and loss in mass of char during activation

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high adsorptive capacity for the small gold–dicyanoaurate complex, makes these carbons ideal for applications involving gold recovery, particularly in carbon-in-pulp (CIP) and carbon-in-leach (CIL) processes.

The nature of the surface oxides formed in the carbon, and the activity of the product as measured, for instance, by the extent to which selected molecules from the gaseous or aqueous phase are adsorbed, are determined by the activation parameters, but the structure of the pores and the pore-size distribution are predetermined to a large extent by the source material[10,16]. This is apparent from photomicrographs of the pore structure of activated carbons manufactured from coconut shells and peach pips, and indicates that the structure of the original source material, viz the cellular structure of the coconut shell and the peach pips, is still present in the carbon skeleton of the final product[8].

Therefore, the inherent cellular structure of raw materials of vegetable origin imposes a constraint on the degree to which the properties of the product can be modified for special applications. For example, the micropores of the cellular structure of coconut shell can be converted into macropores by burning-away of the cell walls, thus changing the adsorption properties of the final product. Unfortunately, this type of modification is accompanied by a loss in the structural integrity and, hence, the strength of the product, as well as a decrease in product yield.

Process Routes Using Coal

Fig. 10 illustrates the various unit operations that are generally involved in the manufacture of granular activated carbons from coal[17].

Process routes using coal as the starting material are significantly more complex than those generally employed in the manufacture of coconut-shell carbons, as is indicated by the large number of unit operations involved.

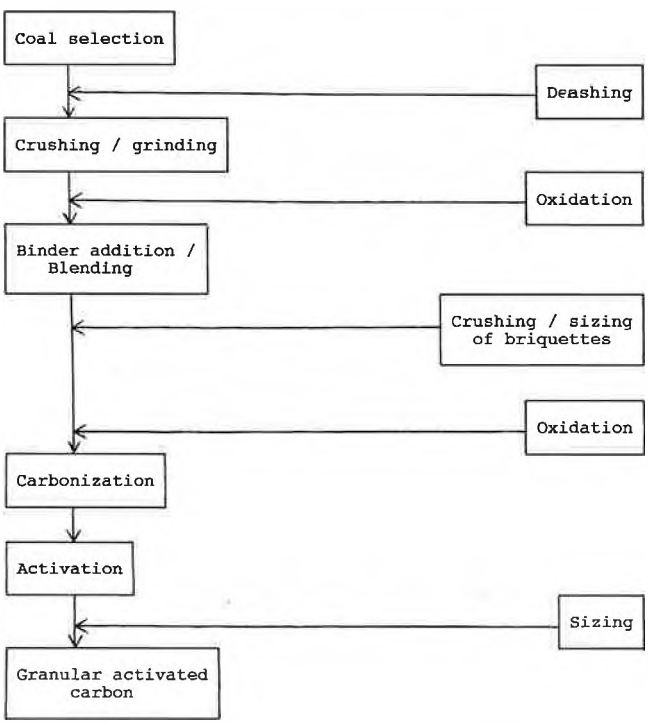


Fig. 10—Flowsheet showing the unit operations that are generally involved in the manufacture of granular activated carbons from coal (after Yehaskel[17])

Owing to the wide availability of abundant quantities of coal, particularly in the Western World, some of the largest commercial production facilities are based on coal as a raw material. However, the particular processing route employed depends mainly on the nature of the coal and the form in which the product is required (crushed or extruded), and hundreds of patents have been registered to describe the numerous process routes developed over the years for the manufacture of granular carbons from various ranks of coal (peat, brown coal, bituminous coal, lignite, coking coal, and anthracite)[17].

A low ash content is one of the most important requirements of a coal that is used for activated carbon. The major reasons for this relate to the fact that the ash content may catalyse certain undesirable activation effects, and is also believed to adversely affect the strength of the final product. Therefore, with high-ash coals, de-ashing with mineral acid may be necessary. The details of the other unit operations are discussed in the following sections.

Crushing, Grinding, and Oxidation

The inherent porosity of most ranks of coal is very low, and therefore the coal used as a starting material requires pretreatment to increase its porosity. The coal is initially crushed and milled by conventional means into a fine powder, after which it is reconstituted by briquetting or extrusion with the aid of a coking binder such as coal-tar pitch. This procedure may result in up to a tenfold increase in porosity over that of the coal as received from the mine. This porosity allows for uniform gasification of the coal granules during activation by providing channels that permit the oxidizing gases to enter the particle, while allowing the reaction products to escape.

Oxidation of the coal, either as a powder or after granulation, is often an important intermediate stage, depending on the nature of the coal. For example, the agglomerating property of a high-rank medium-volatile bituminous coal is destroyed if the coal is oxidized at a temperature below its fusion point, i.e. between 200 and 280°C. This treatment alters organic components in the coal, thereby rendering the particles non-agglomerative, and can be conducted either in a fluidized bed (coal powder) or in a rotary kiln (granules). The average residence time for oxidation varies between 10 minutes and 1 hour. Careful temperature control is critical because, above about 375°C, the characteristic particle shape is destroyed, and the particles may fuse. Steam is often injected into the kiln for purposes of temperature control.

Briquetting and Extrusion

Granular activated carbons can be crushed or pressed. The products of crushed granular coal are produced by briquetting, the briquette being crushed and sized prior to oxidation or carbonization, or only after activation. The particle-size range is usually between 0,2 and 3,5 mm.

The second type of granular carbon, viz pressed or extruded carbon, is usually produced in uniform cylindrical shapes. These are always formed prior to carbonization and activation. In the process, the coal powder

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is blended with a coking binder, usually coal-tar pitch, although numerous other types of binders are also employed commercially, at a temperature above the softening point of the pitch. The resultant plastic mass is then extruded from a die, and the rod is cut into pieces of uniform length. The diameter of the cylinders usually varies between 0,8 and 4,0 mm, and the length is from one to three times greater than the diameter.

Carbonization and Activation

General aspects related to carbonization and activation are no different from those discussed previously for coconut shells, and are therefore not repeated here.

Characteristics of Coal-based Activated Carbons

Coal-based activated-carbon products are characterized by a tridisperse pore-size distribution, i.e. they contain micropores, mesopores, and macropores, as is apparent from Fig. 11. For this reason, they constitute a very versatile range of products and are generally employed in a wide variety of aqueous-phase applications requiring the removal of a range of molecules varying significantly in molecular size, e.g. in the treatment of effluent water.

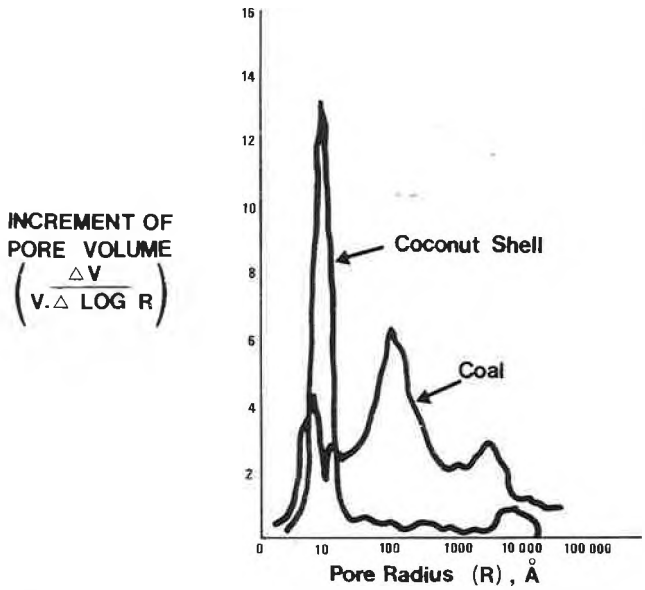


Fig. 11—Pore-size distributions for a typical thermally activated coal-based carbon and a coconut-shell carbon (after Fornwalt *et al*[18])

When cognizance is taken of the many variables involved in the manufacture of coal-based products (the rank of the coal, the binder chosen, the briquetting or extrusion technology used, the combination of activation parameters chosen, the fineness of grind employed, etc.), it is obvious that the scope for modification of the product is significant. For example, coal-based products cannot be modified to simulate the properties of coconut-shell or wood-based products, although any modifications between these extremes are possible.

It is also generally accepted that the abrasion resistance and impact hardness of these reconstituted products, based on current manufacturing technology, is with few exceptions not as good as those found in products using coconut shells as the raw material.

Selection of an Activated Carbon for Gold Recovery

The primary requisites[19] for a granular activated carbon intended for use in gold-recovery applications are as follows:

(a) the carbon must have a high gold activity, and
(b) the carbon must be tough and resistant to abrasion, for this will minimize the loss of gold associated with fine abraded carbon in the tailings.

Since activated carbons manufactured from coconut shells best meet the above requirements, they are widely employed in gold-recovery applications. Other important parameters relate to particle size (the rate of gold extraction increases with decreasing particle size), the initial and equilibrium gold concentrations, the surface area of the product, the pH value and temperature of the adsorption medium, etc.[12,13,19].

Conclusions

The main objective in the manufacture of granular activated carbon is the development of an optimum pore structure associated with a high surface area—with minimum loss of the carbon content through carbonization and oxidation—and of a product with sufficient structural strength to withstand normal usage without excessive attrition of the particles.

The manufacture of high-quality activated-carbon products is significantly more complex than has been outlined here because of the number of variables involved in the manufacturing process, and the complex interrelationship between those variables. As discussed, when unsophisticated methods of manufacture are employed, each raw material tends to produce its own characteristic type of pore structure. Nevertheless, this natural tendency can be altered considerably by special treatment requiring considerable effort and know-how.

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Mine valuation and grade control

The South African Institute of Mining and Metallurgy will be holding a 2-day school on 6th and 7th June, 1991, on the topic **Mine Valuation and Grade Control.**

Objective

The objective of this School is to present the practical application of valuation and grade control as they apply to the accounting for metal and grade of broken ore from the *in situ* to the final saleable product.

The latest developments in this field relating to the mining of precious metals, coal, and base minerals will be covered.

Programme

Sessions will be held on the following topics:

- Reserves (Chairman: Dr I. Clark)
- Dilution (Chairman: Dr G.A. Fourie)
- Mineral Flow (Chairman: Mr L.V. Estment)
- Stockpiling (Chairman: Mr N. de Wet)
- Plant (Chairman: Mr J. Shirley)
- Quality Control (Chairman: Mr D.J. van Niekerk).

Contact Address

Further information is available from

Pamela Smith
SAIMM
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Tel.: (011) 834-1273/7; Fax: (011) 838-5923
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Mine productivity and safety

The South African Institute of Mining and Metallurgy will be holding a one-day colloquium devoted to **Communications and Monitoring to Improve Mine Productivity and Safety.**

This Colloquium will cover a range of topics dealing with the following systems planned or in operation in underground and surface mines:

- Voice communications and telemetry underground
- Rock and personnel tracking
- Environmental monitoring and alarm
- Rope-load monitoring
- Equipment-condition monitoring
- Shaft management.

The Colloquium will be held on 21st May, 1991, at the BIFSA Conference Centre, Halfway House, Midrand.

Enquiries should be addressed to

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Bacterial oxidation

The South African Institute of Mining and Metallurgy will be holding a colloquium on **Bacterial Oxidation** in June 1991 in Johannesburg.

The papers will be published in a bound volume, which will be available to all delegates on registration at the Colloquium. The language of the Colloquium will be English.

The final circular will contain detailed information on the Colloquium, as well as the registration forms.

All correspondence should be addressed to

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Dr. Richard Mimna
Director of Business Innovation

Eric Forrester
Applications Engineer

Granular Activated Carbon:
A Proven Solution for PFC Removal



January 30, 2017

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Granular Activated Carbon:
A Proven Solution for PFC Removal



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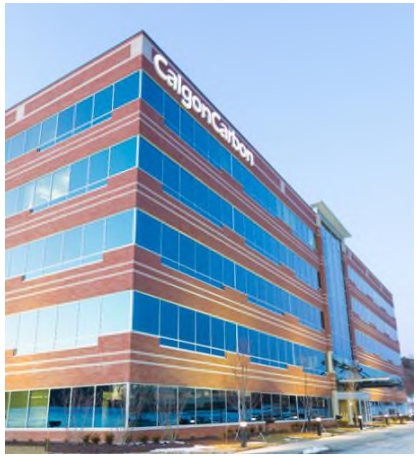
Introduction

- Calgon Carbon Corporation is the world's largest producer of Granular Activated Carbon (GAC)
- Our mission: pure water, clean air, better living
- We solve purification and separation problems with an array of technologies (Activated Carbon, IX, UV, AOP, Perlites, D.E.)
- Water treatment is core competency with a diverse product portfolio

$535 million
2015 net sales

70+ years
experience

1,400+ employees



23 offices
sales and service

21 facilities
manufacturing, reactivation, equipment

240 patents

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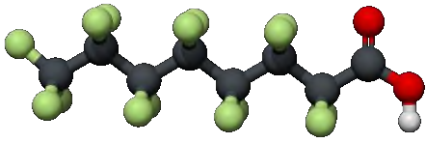
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Protecting Communities from Perfluorinated Compounds

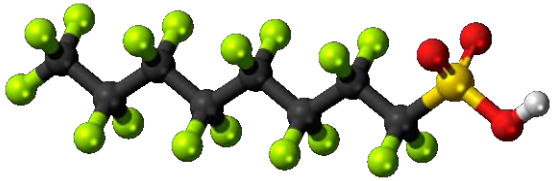
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PFC Overview

Man made fully fluorinated organic compounds



PFOA Molecule



PFOS Molecule

Health Advisory:
70 ppt
Combined
PFOA / PFOS

Why PFCs Are a Problem

- Contaminates drinking water and food
- Highly persistent / resistant to degradation
- Accumulate in the body

Where they come from….



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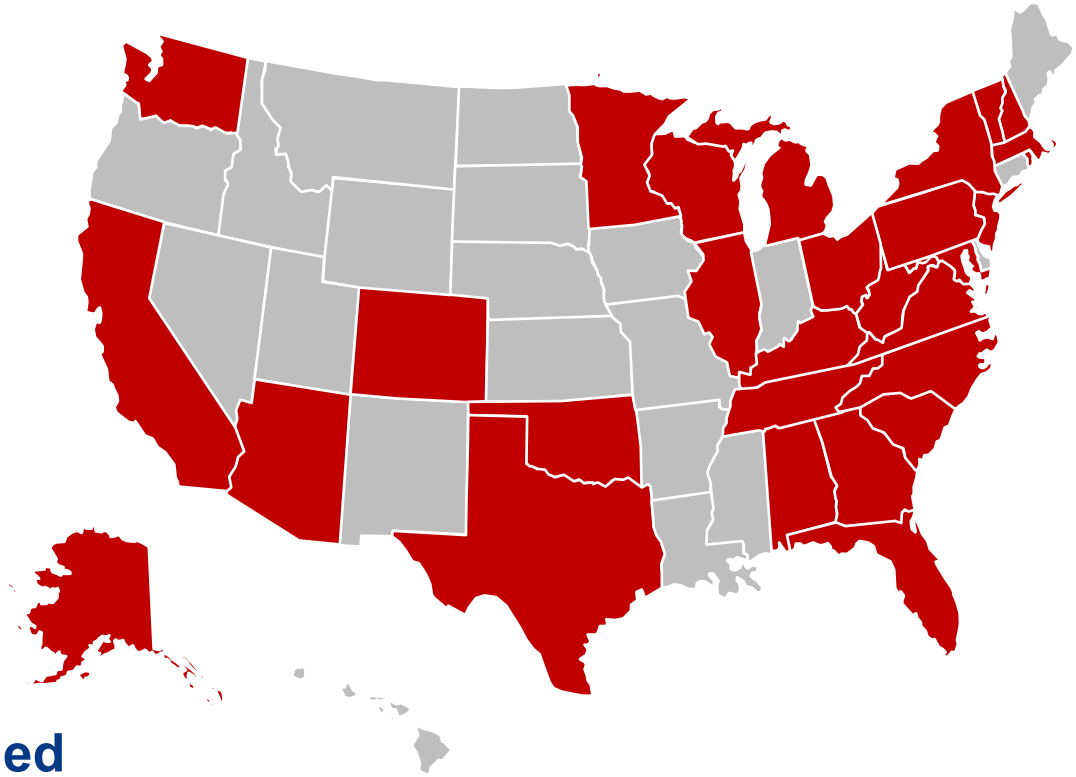
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Where are PFCs?

U.S. Drinking Water Contaminated by PFCs*

According to the U.S. EPA:

- 94 Public drinking water systems with PFC's
- Across 28 States(Red Highlight)
- 6.5 Million Americans affected
- Not including private wells



Contaminated non-drinking water sources are not included

….THE PROBLEM IS ONLY GROWING!

*source http://news10.com/2016/06/02/pfoa-by-the-numbers-a-widespread-contamination-and-how-it-affects-your-health/

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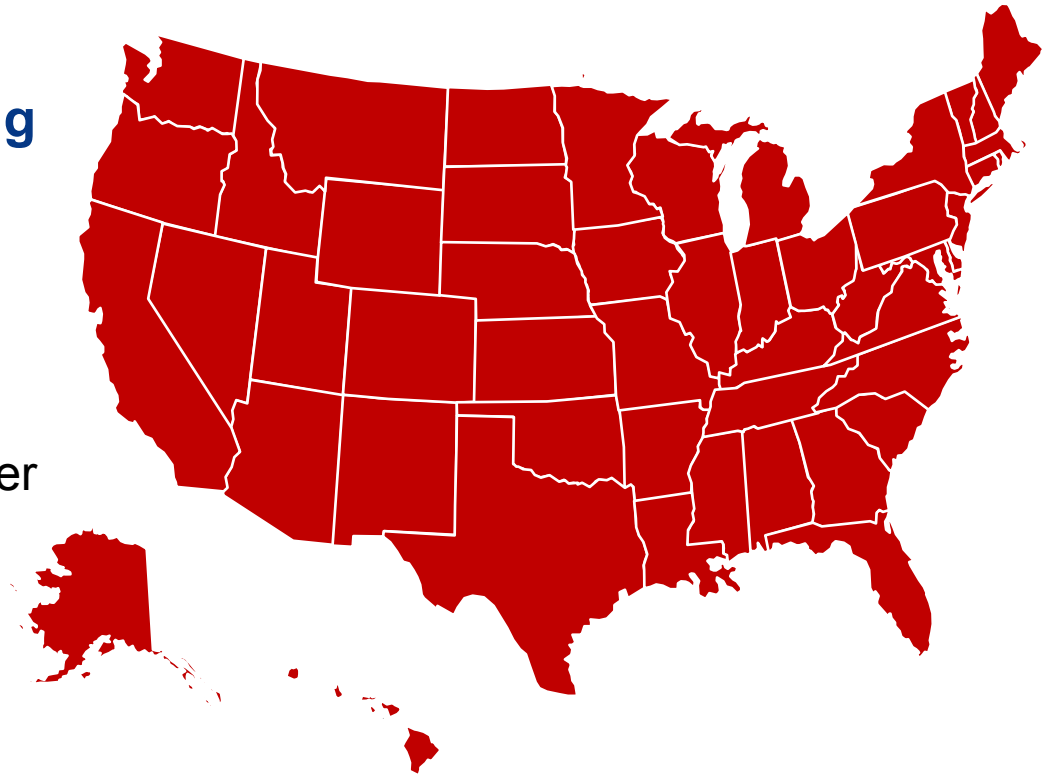
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Where are PFCs?

Potentially PFC Contaminated DoD Sites *

Based on the use of fire fighting foams….

- DoD identified over 200 bases potentially contaminated
- Across 49 States(Red Highlight)
- Testing currently underway
- May impact adjacent municipal water utilities



*source http://news10.com/2016/06/02/pfoa-by-the-numbers-a-widespread-contamination-and-how-it-affects-your-health/

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How GAC Can Help

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Economic Benefits of GAC



The average family of 4 consumes 65,700 gallons of water annually

Tap water filtered by GAC costs a family of 4 roughly $2.00/Month (per AWWA)

The average 2015 price for bottled water is $1.22/gallon

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GAC is a Proven Technology



Leading technology for removal of PFCs from drinking water and groundwater

>15 years and >20 large installations in municipal/ industrial segments & > 1000 POET GAC systems treating residential well sites

GAC is safe & environmentally responsible - safest way to treat is to remove contaminants

Cost effective & simultaneously removes other emerging contaminants which addresses future compliance requirements

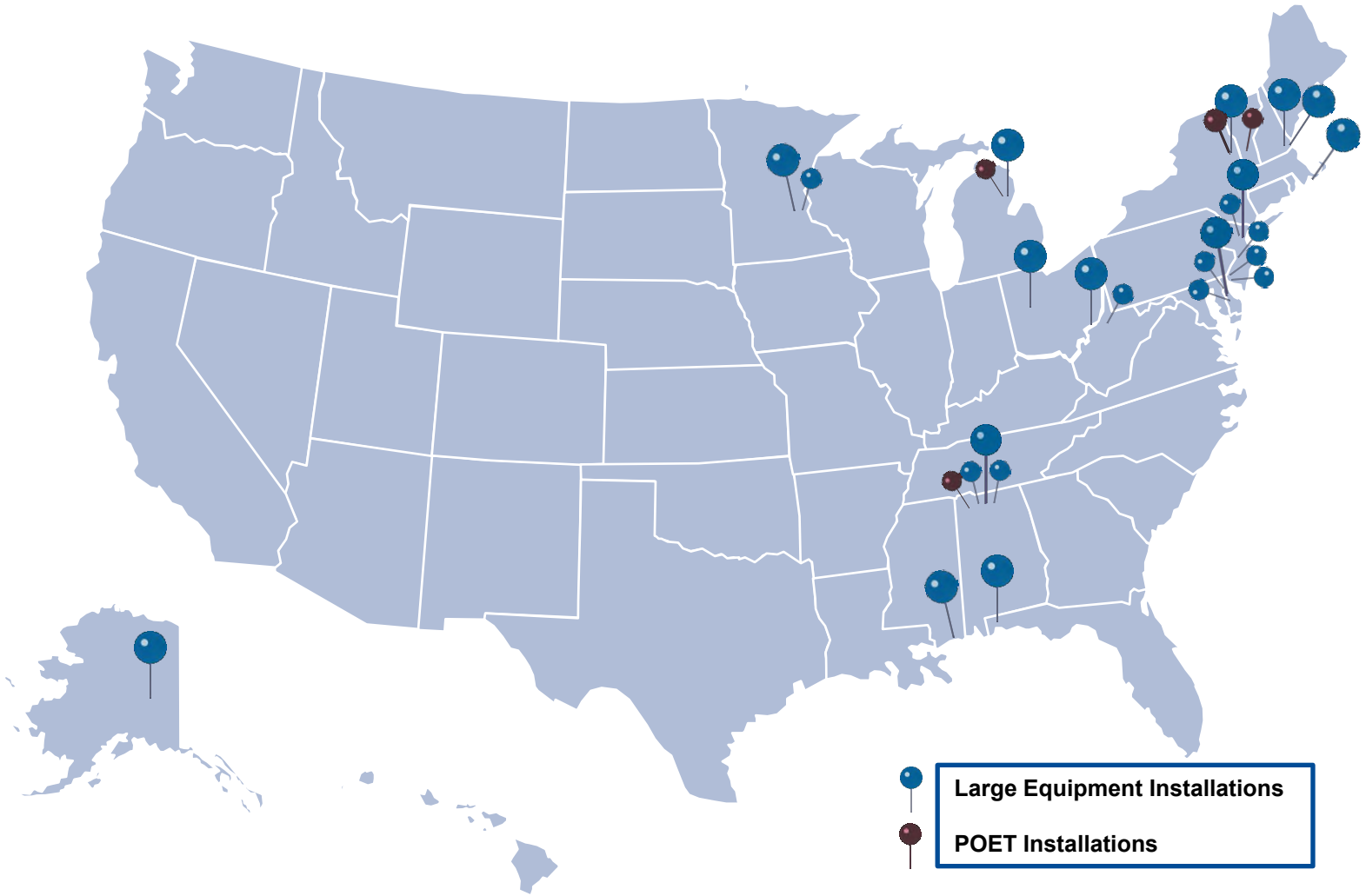
Reactivation of spent GAC thermally destroys adsorbed contaminants including PFC's

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Calgon Carbon PFC Treatment Locations



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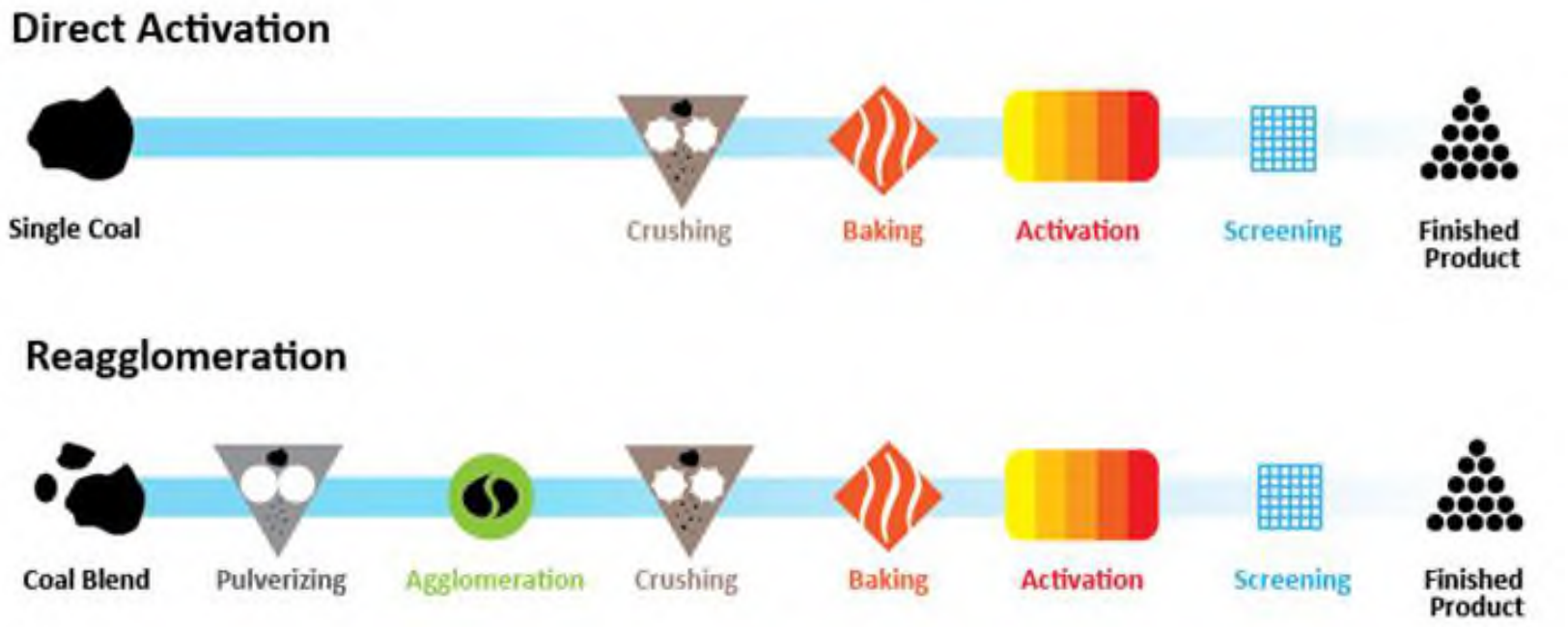
Why FILTRASORB® GAC is superior

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FILTRASORB® GAC

Domestically mined, domestically manufactured GAC

Bituminous coal-based re-agglomerated GAC has shown to have better performance in water applications than direct activated coal and coconut based carbons



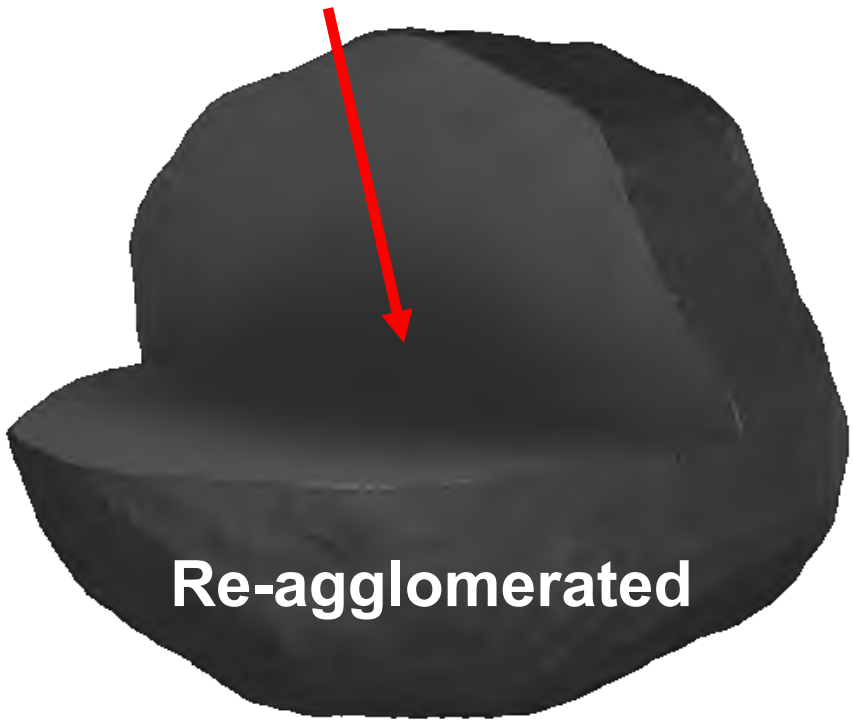
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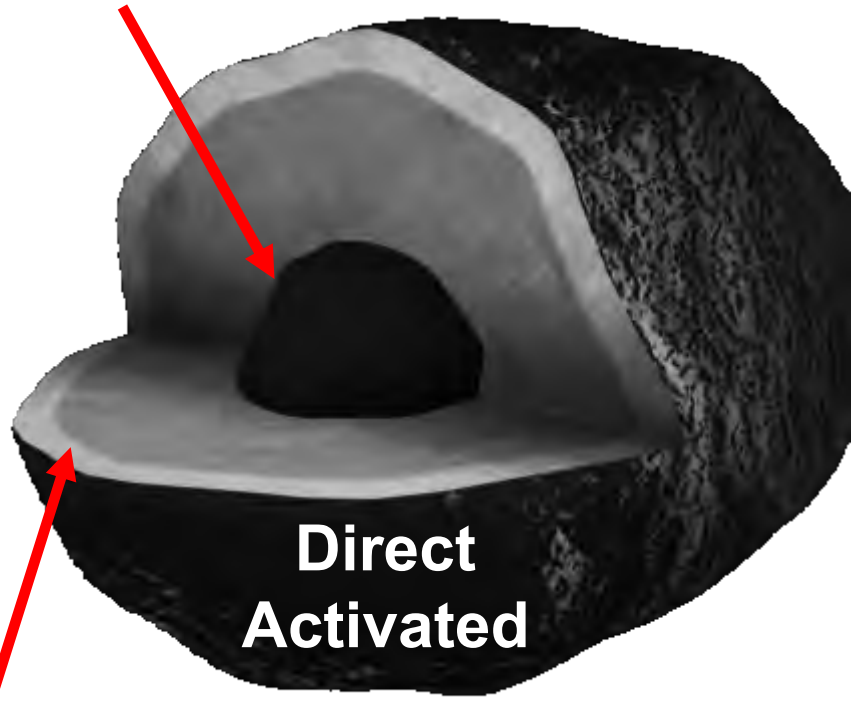
Granular Activated Carbon

Product differences: Re-agglomeration versus Direct Activation

Even Activation



Low Activation



Over activation in the outer Part of the granule

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Activated Carbon Starting Materials









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The Importance Of Testing

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Importance Of Testing

- **PFCs/PFASs are found in trace amounts in water**
- **Testing is recommended – customer raw water sample**
  - Isotherm
    - Quick test method for feasibility
  - Accelerated Column Tests (ACT)
    - Simulates full scale performance
    - Provides important information: carbon type, breakthrough data, usage rates
    - System design is critical
- **Drinking water applications**
  - Typically low concentrations (ppb, ppt)
  - Background TOC can be 10-100X the PFC/PFAS concentration
  - Modeling such scenarios is difficult (even with other, non-PFC compounds)
  - ACT or RSSCT is beneficial
- **If client timing does not permit testing, CCC recommends as a start: 10 minutes EBCT (Empty Bed Contact Time) per adsorber, with 2 adsorbers in series**

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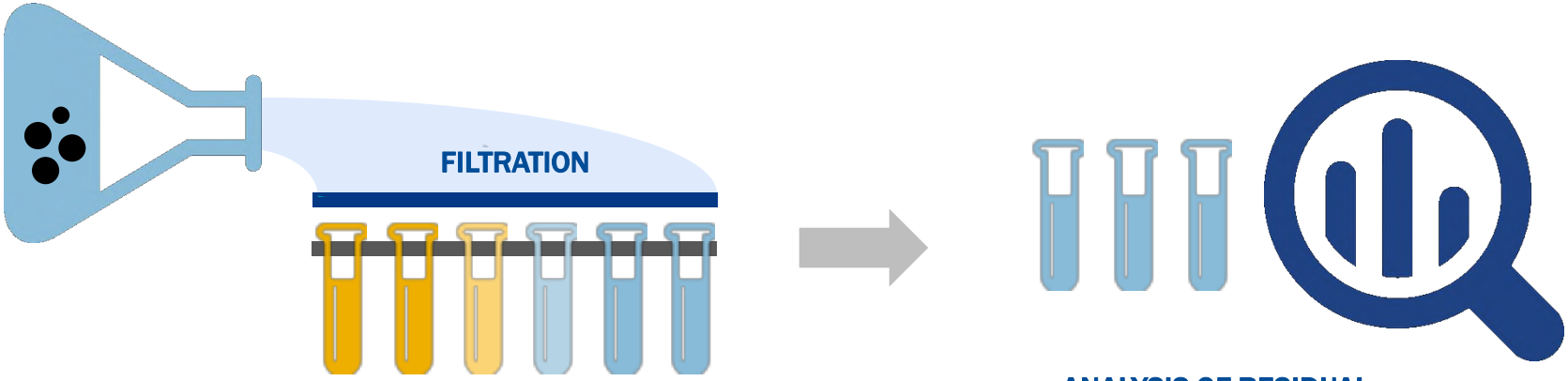
Isotherms



100 ML OF SOLUTION TO BE TREATED

SOLUTION IN CONTACT WITH INCREASING AMOUNT OF CARBON

EQUILIBRIUM REACHED



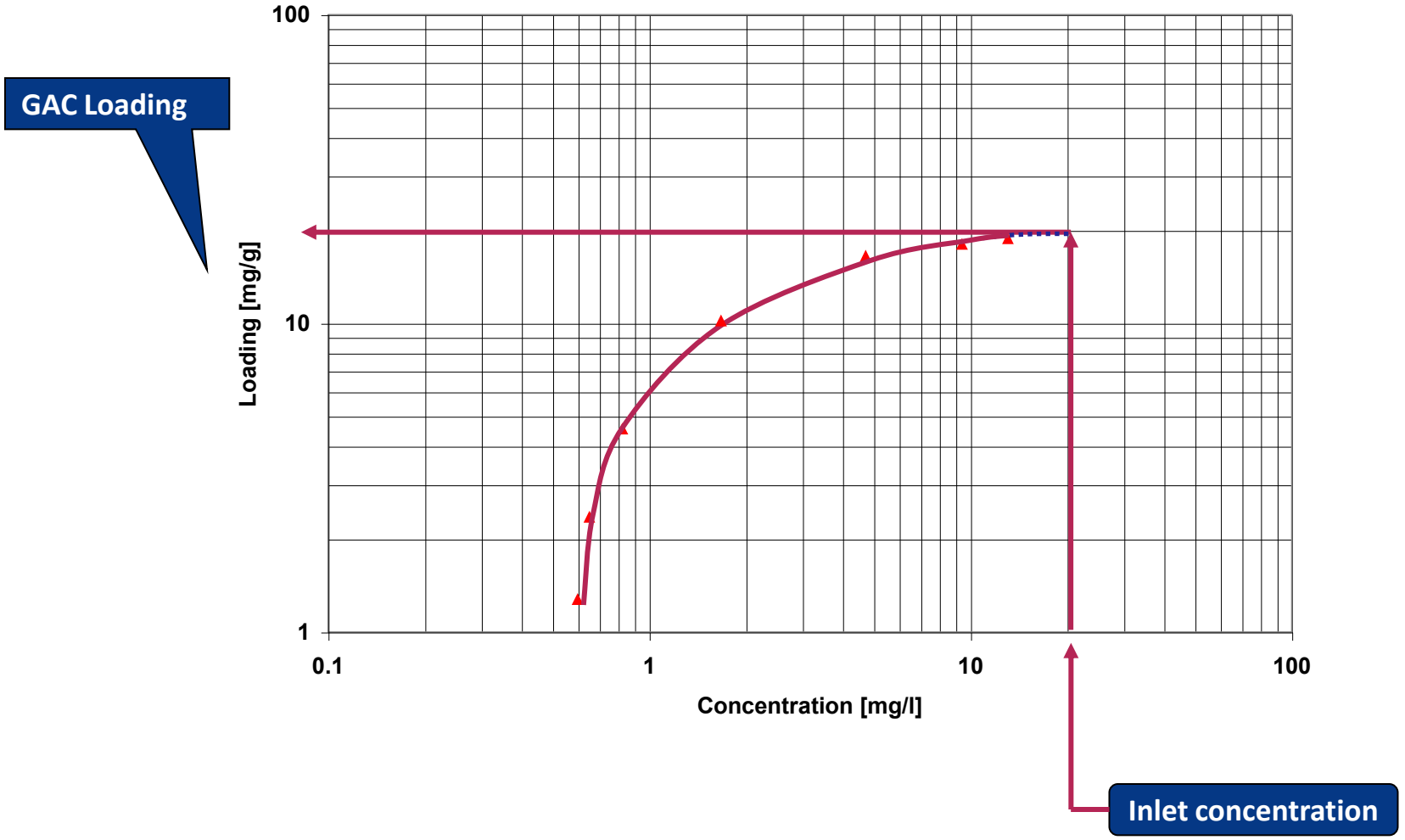
ANALYSIS OF RESIDUAL CONCENTRATION

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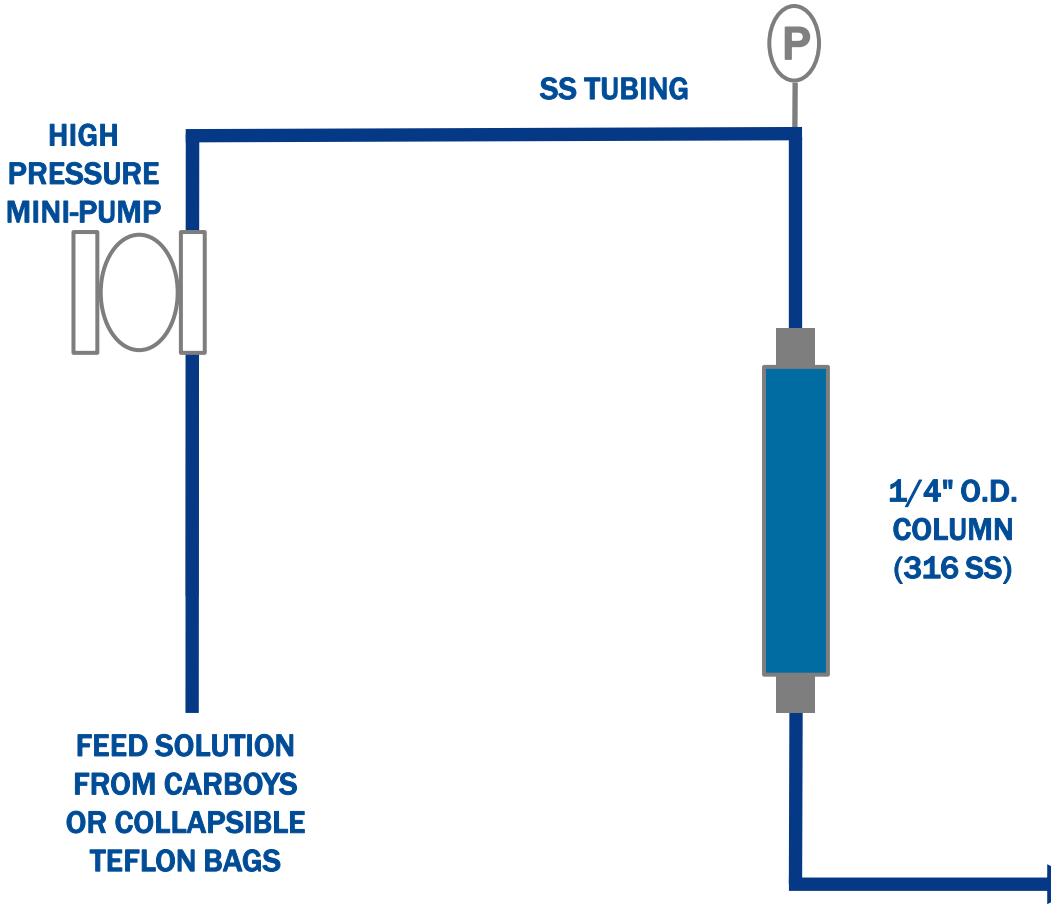
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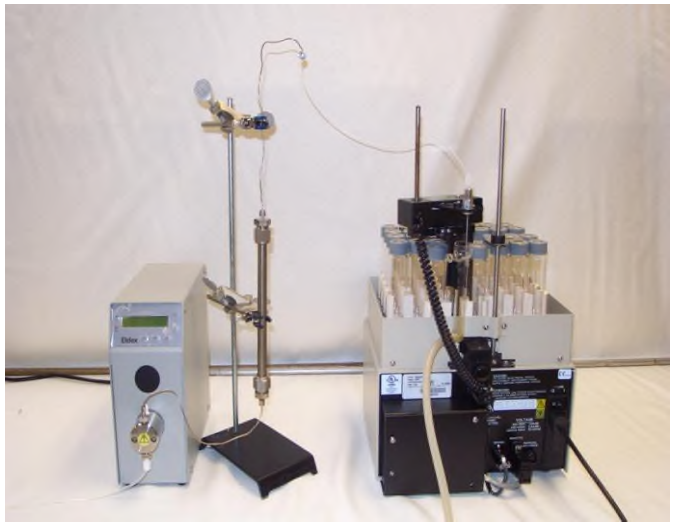
Isotherm



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RSSCTs / ACTs Test Methods

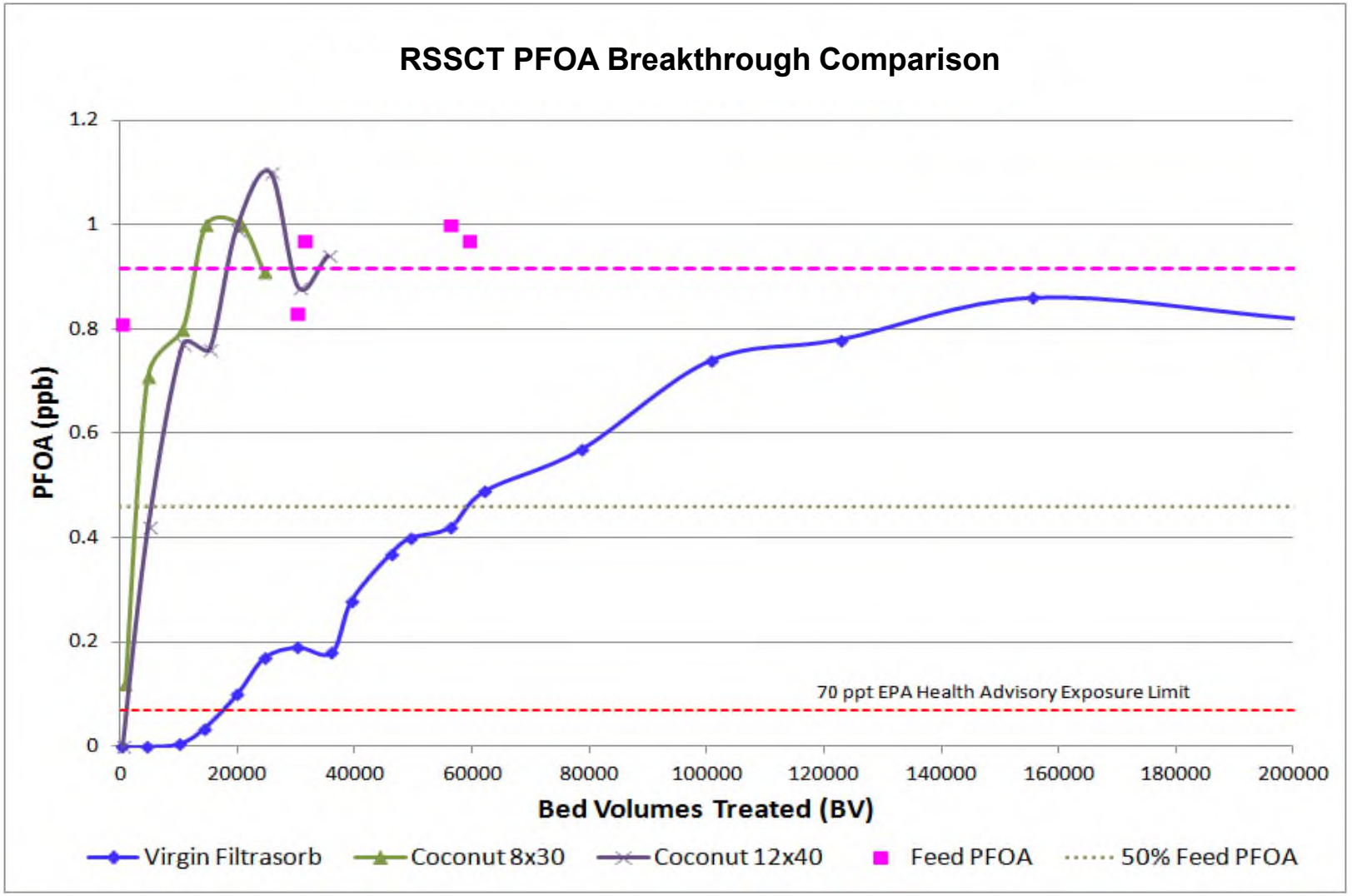




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FILTRASORB® Comparative Performance Data

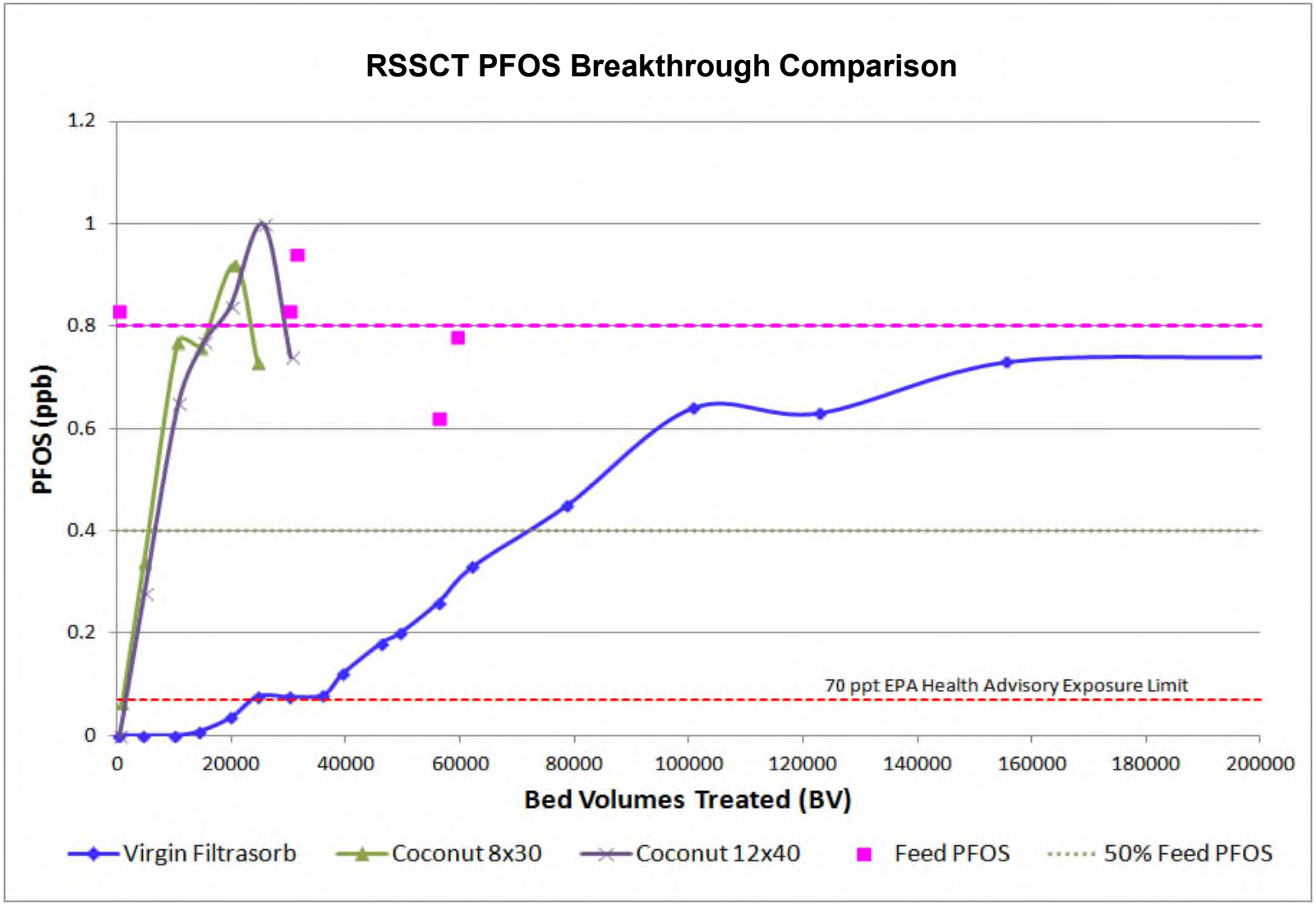


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FILTRASORB® Comparative Performance Data



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Conclusions from Analysis

- Bituminous coal-based re-agglomerated GAC significantly outperforms coconut based GAC
- Lab analysis supports bituminous coal-based re-agglomerated GAC is the best product for PFC removal



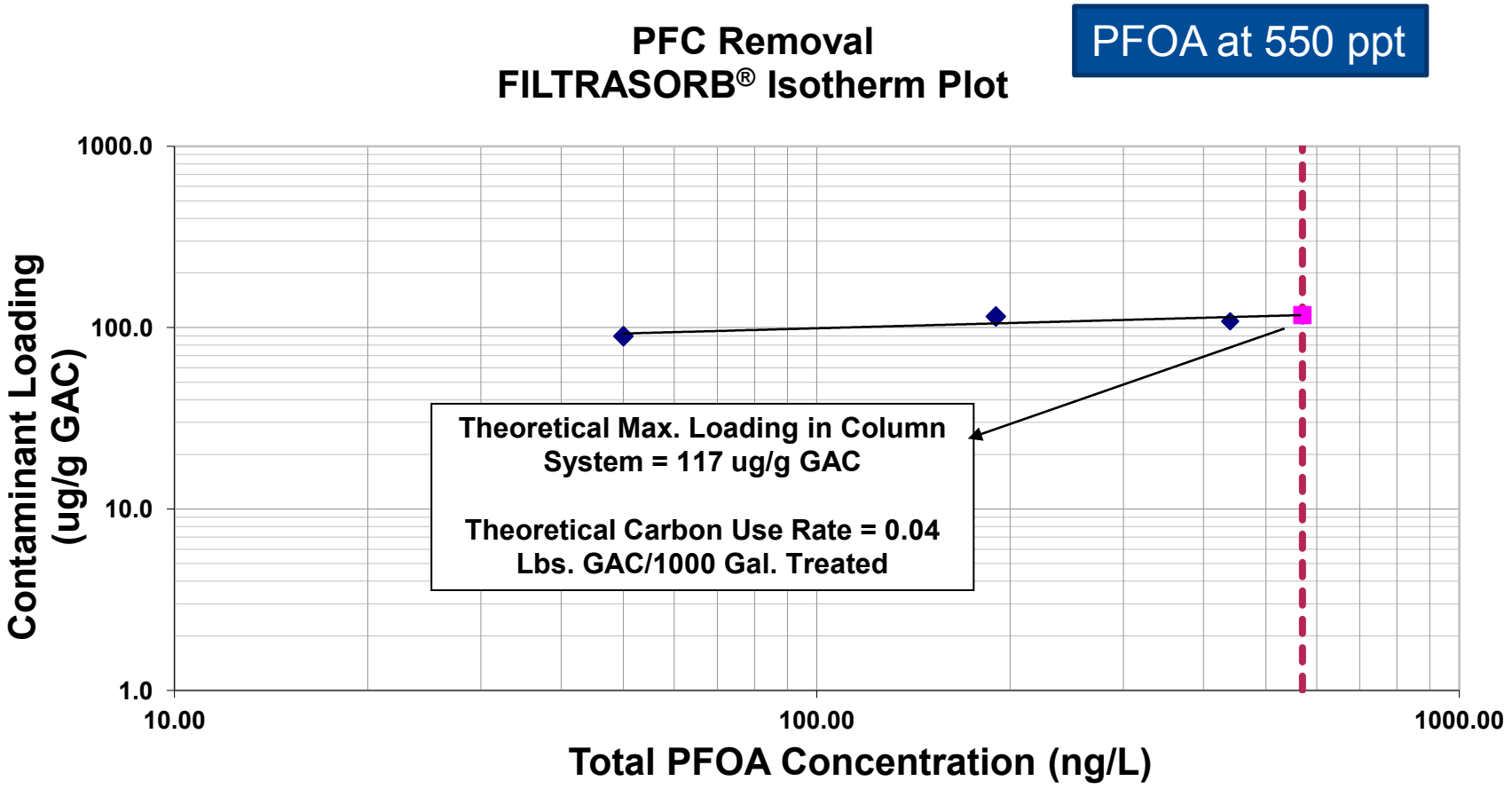
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Case Study

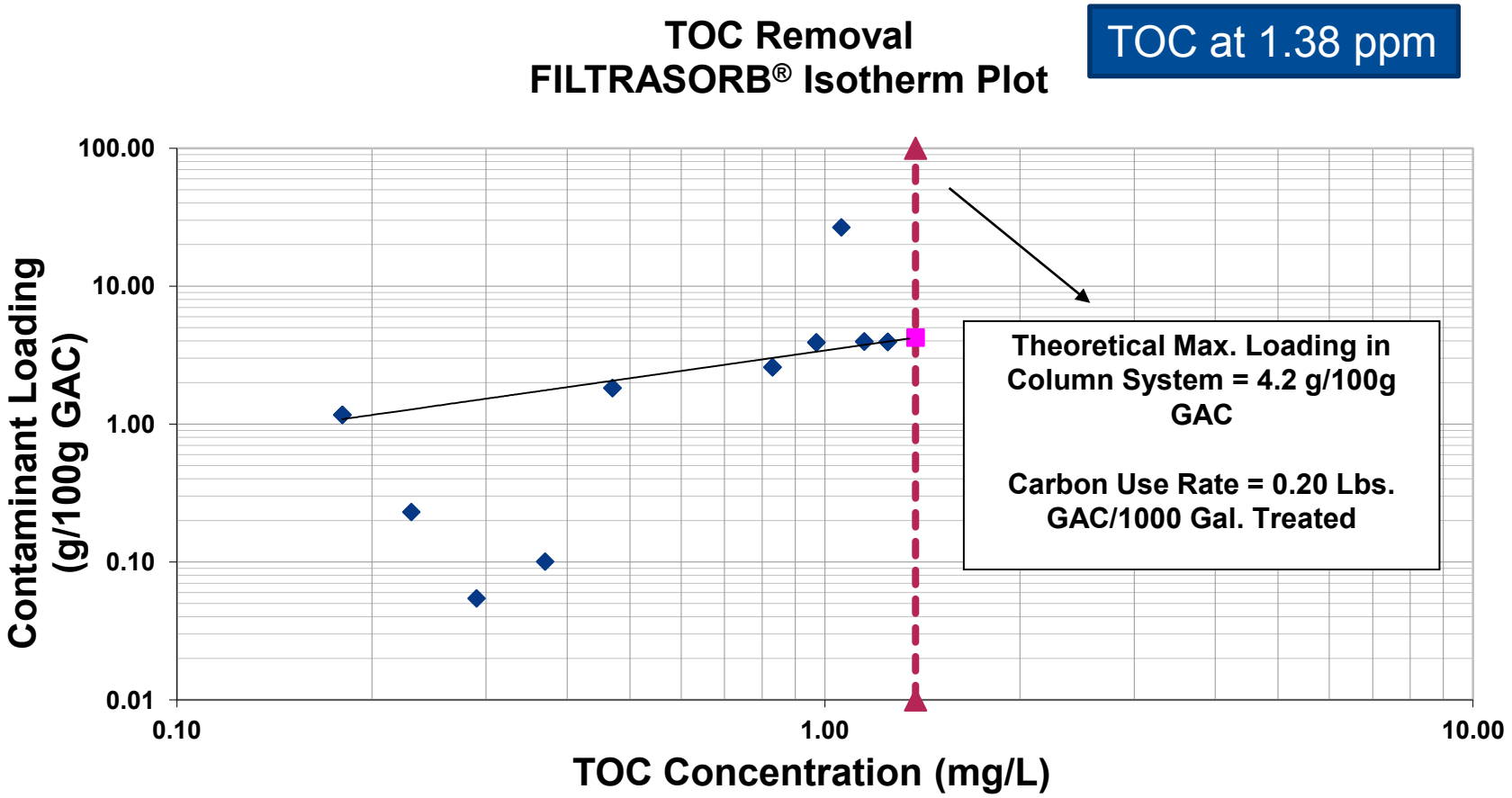
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PFC Isotherm- NY Water Source



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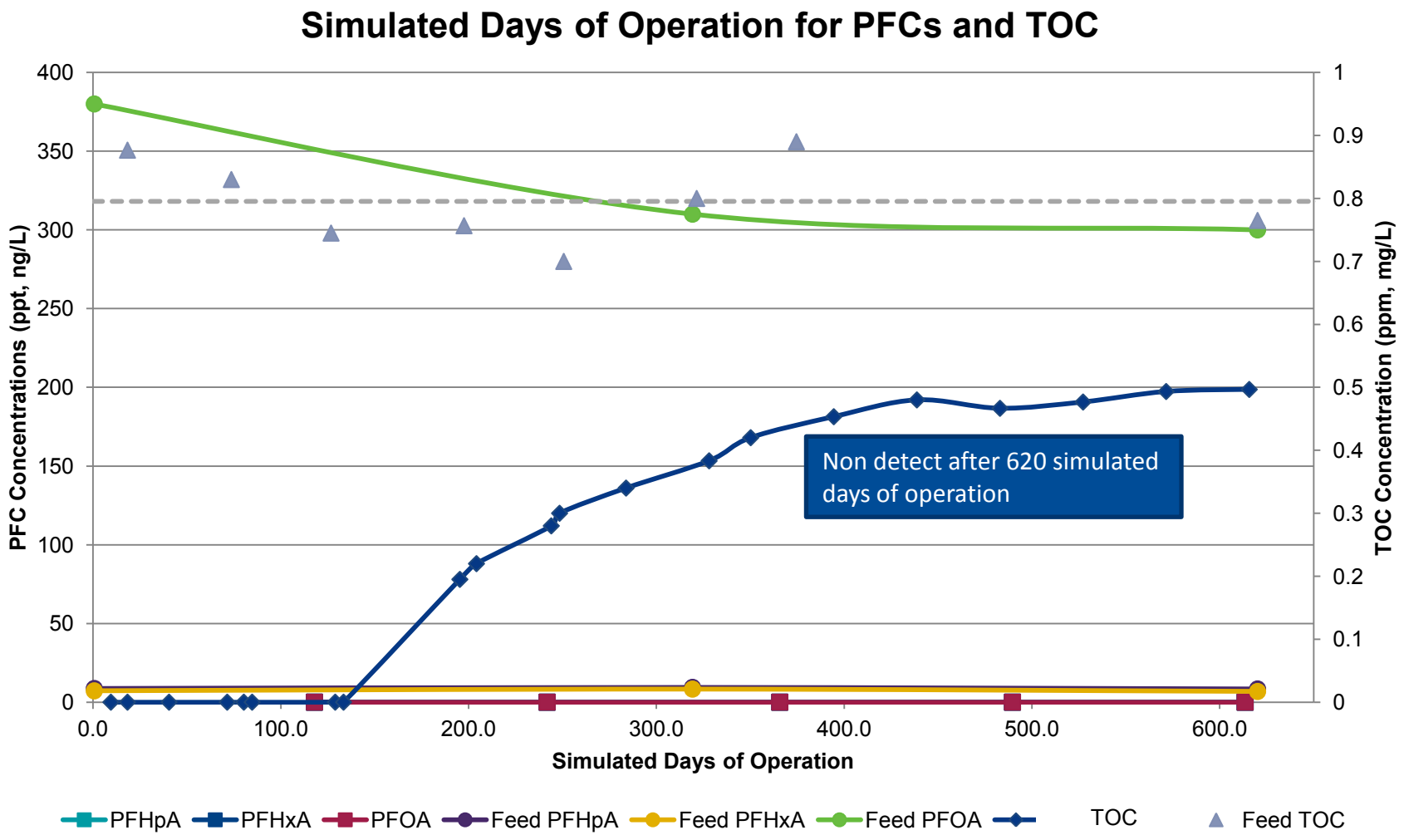
TOC Isotherm- Same NY Water Source



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PFC/TOC Customer ACT Data



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Conclusions from Case Study

- FILTRASORB® can remove PFCs to non-detectable levels
- TOC does not appear to compete strongly with PFC
- FILTRASORB® is effective for > 620 simulated days of operation
- Temporary System: 2 x 10’ diameter vessels, 20,000 lbs GAC each
- Permanent System: 2 x 12’ diameter vessels, 40,000 lbs GAC each

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Equipment and Reactivation

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Carbon Adsorption Equipment

Numerous "base" options - customized for customer's requirements…

Model 8



Model 12



Model 14



Model 10



Model 12-40

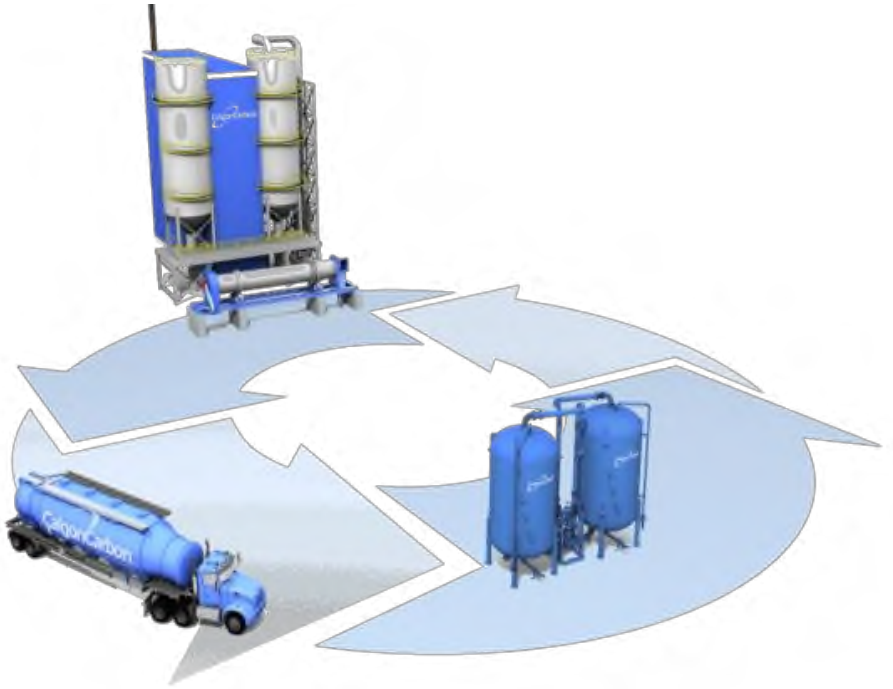


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Reactivation of GAC

- **Carbon reactivation is a thermal treatment process in which adsorbed chemical constituents are removed from spent activated carbon**
- **The desorbed chemical constituents are thermally destroyed in the reactivation process**
- **Carbon is then reusable**
- **Frequency of reactivation is dependent on application**

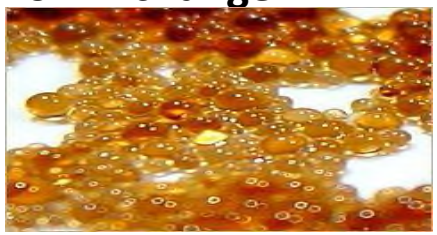


CCC has over 10 years of experience reactivating carbons with PFCs

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PFC Removal: GAC vs. RO vs. IX

Treatment Option	Pros	Cons
GAC 	•Significantly lower capital costs •Significantly lower O&M costs •Reactivation saves cost, destroys PFCs, & removes liability •Established BAT for a long list of organic contaminants	•High NOM can increase use rates
RO 	•Removes salts / inorganics that GAC cannot	•Concentrated waste water disposal liabilities & costs •More energy / CO_2 intensive •High maintenance cleaning and replacement of fouled membranes •Removes healthy minerals
Ion Exchange 	•Resin can be regenerated •May be more economical for certain source waters (i.e. high levels of nitrate)	•High cost of reagent •Regeneration produces disposal liabilities & costs

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CCC Advantage

CCC has offered the preferred and total treatment solution for PFC removal for 15 years with the FILTRASORB® product line

GAC is the leading technology for removal of PFCs

Calgon Carbon is ready to respond immediately with technical services, equipment and carbon supply

Spent carbon can be returned to CCC for reactivation thus thermally destroying the PFCs and eliminating future liability

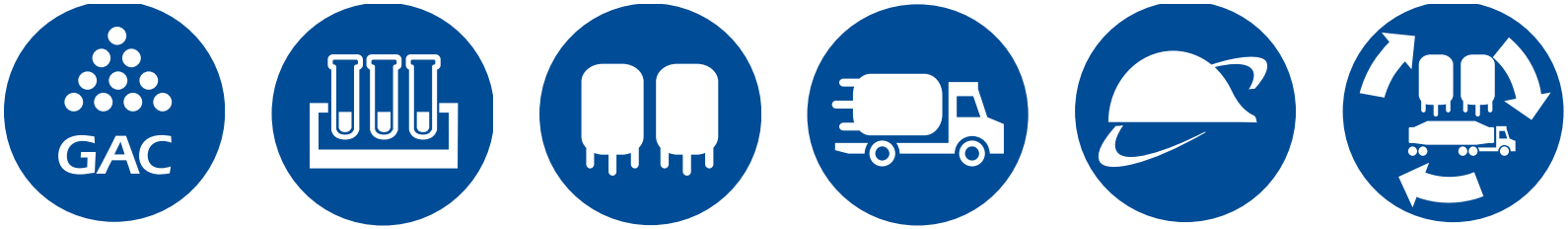


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Calgon Carbon Solutions



- Provide a proven and cost-effective packaged solution
- Perform laboratory and field testing
- Ensure proper equipment design
- Rapid deployment of temporary treatment systems
- Experienced sales, field service and applications engineering teams
- Carbon reactivation services

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Questions

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Thank you.

www.calgoncarbon.com/PFOA

Send additional questions to: pfcsolutions@calgoncarbon.com

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APPENDIX 1007B

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PERFLUORINATED COMPOUNDS (PFCS), PERFLUOROOCTANE SULFONATE (PFOS) AND PERFLUOROOCTANOIC ACID (PFOA)



The Wayback Machine - https://web.archive.org/web/20170222172255/http://www.calgoncarbon.com/PFOA/

PERFLUORINATED COMPOUNDS (PFCS), PERFLUOROOCTANE SULFONATE (PFOS) AND PERFLUOROOCTANOIC ACID (PFOA)



WHAT ARE PERFLUORINATED COMPOUNDS (PFCS)?

- Manmade fully fluorinated compounds which are not naturally found in the environment.
- Are used in a variety of products such as fire fighting foams, coating additives as a surface-activeagent.
- PFOS (perfluorooctane sulfonate) and PFOA (Perfluorooctanoic acid) are the most commonly produced PFCs.
- Large amounts of PFCs have been produced during past manufacturing processes and released to the air, soil and water.

CONTACT US

CASE STUDIES

MORE INFORMATION

Click to download the brochure

DOWNLOAD NOW

WHY ARE PFCS HARMFUL?

- PFOA and PFOS are persistent and mobile in the atmosphere and aqueous environments, due to their chemical stability and low volatility.
- Animal toxicology studies indicate potential developmental, reproductive and systematic effects.
- After oral exposure, PFCs accumulate and adsorb in the serum, kidney, and liver.
- Resistant to direct oxidation, photolytic degradation, biodegradation and air stripping/ vapor extraction, making their removal difficult.

WHAT IS THE EPA'S STANCE?

- PFOS and PFOA are on the EPA's Contaminant Candidate List 4 (CCL 4).
- The EPA has taken steps to further investigate PFOAs and related chemicals as well as to reduce their emissions

and use in products.

WHAT IS THE SOLUTION?

•Granular activated carbon (GAC) filters have been recognized as effective technologies for reducing perfluorinated compounds from water.

•Incineration of any concentrated PFC waste is required for complete destruction of PFOS and PFOA. Spent activated carbon containing adsorbed species can be thermally reactivated , destroying the adsorbed contaminants and allowing the activated carbon to be recycled and reused.

•Recent Accelerated Column Tests (ACTs) of Calgon Carbon type Filtrasorb 400 and Filtrasorb 600virgin GAC shows successful removal of perfluorinated compounds including Perfluorobutanoic Acid (PFBA), Perfluoropentanoic Acid (PFPA), Perfluorohexanoic Acid (PFHxA), Perfluoroheptanoic Acid (PFHtA), Perfluorooctanoic Acid (PFOA), Perfluorodeconaoic Acid (PFDA).

•The strong fluorine-carbon bond and low vapor pres-sure makes PFOA/PFOS resistant to a number of conventional water treatment technologies, including direct oxidation, biodegradation, air stripping, vapor extraction and direct photolysis (UV).

•Since each water source contains different combinations and levels of PFCs, as well as TOCs, it is advised that a lab or pilot test (such as an Accelerated Column Test or ACT) be performed on a representative water sample to determine the adsorption zone needed, as well as the estimated carbon exhaustion rate to properly design an activated carbon adsorption system.

WHAT CAN CALGON CARBON DO FOR YOU?

•Provide proven and cost effective solutions with activated carbon adsorption technology that cover a broad range of applications and system sizes.

•Temporary and permanent GAC systems that can be rapidly deployed.

•Our technical and engineering team can perform laboratory and field tests as needed, and tailor solutions for various application and customer needs. CCC has carbon reactivation sites globally that will thermally destroy the PFCs and enable the reuse of the activated carbon.

Webinar: Granular Activated Carbon – A Proven Solution for PFC Removal

Filtrasorb Granular Activated Carbon

Field Tests and Pilot Systems to Test Water

Case Study: GAC Removes PFOA from Drinking Water

Reactivation of Spent Carbon Containing PFCs

Water World Article on PFOAs



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Dr. Richard Mimna
Director of Business Innovation

Eric Forrester
Applications Engineer

Granular Activated Carbon:
A Proven Solution for PFC Removal



January 30, 2017

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WATER RESEARCH



International Water Association

Volume 51 (2014)

EDITOR-IN-CHIEF

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Delft University of Technology

AIMS AND SCOPE

Water Research publishes refereed, original research papers on all aspects of the science and technology of water quality and its management worldwide. A broad outline of the journal's scope includes:

- Treatment processes for water and wastewaters, municipal, agricultural and industrial, including residuals management.
- Water quality standards and the analysis, monitoring and assessment of water quality by chemical, physical and biological methods.
- Studies on inland, tidal or coastal waters and urban water, including surface and ground waters, and point and non-point sources of pollution.
- The limnology of lakes, impoundments and rivers.
- Solid and hazardous waste management, including source characterization and the effects and control of leachates and gaseous emissions.
- Environmental restoration, including soil and groundwater remediation.
- Analysis of the interfaces between sediments and water, and water/atmosphere interactions.
- The application of mathematical and modelling techniques.
- Public health and risk assessment.



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Treatment of poly- and perfluoroalkyl substances in U.S. full-scale water treatment systems

Timothy D. Appleman [a], Christopher P. Higgins [a,*], Oscar Quiñones [b], Brett J. Vanderford [b], Chad Kolstad [c], Janie C. Zeigler-Holady [b], Eric R.V. Dickenson [a,b,**]

[a] Civil and Environmental Engineering Department, Colorado School of Mines, 1500 Illinois Street, Golden, CO 80401, USA

[b] Applied Research and Development Center, Water Quality Research and Development Division, Southern Nevada Water Authority, Henderson, NV 89015, USA

[c] Minnesota Department of Health, St. Paul, MN 55155, USA

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ABSTRACT

The near ubiquitous presence of poly- and perfluoroalkyl substances (PFASs) in humans has raised concerns about potential human health effects from these chemicals, some of which are both extremely persistent and bioaccumulative. Because some of these chemicals are highly water soluble, one major pathway for human exposure is the consumption of contaminated drinking water. This study measured concentrations of PFASs in 18 raw drinking water sources and 2 treated wastewater effluents and evaluated 15 full-scale treatment systems for the attenuation of PFASs in water treatment utilities throughout the U.S. A liquid-chromatography tandem mass-spectrometry method was used to enable measurement of a suite of 23 PFASs, including perfluorocarboxylic acids (PFCAs) and perfluorosulfonic acids (PFSAs). Despite the differences in reporting levels, the PFASs that were detected in >70% of the source water samples (n = 39) included PFSAs, perfluorobutane sulfonic acid (74%), perfluorohexane sulfonic acid (79%), and perfluorooctane sulfonic acid (84%), and PFCAs, perfluoropentanoic acid (74%), perfluorohexanoic acid (79%), perfluoroheptanoic acid (74%), and perfluorooctanoic acid (74%). More importantly, water treatment techniques such as ferric or alum coagulation, granular/micro-/ultrafiltration, aeration, oxidation (i.e., permanganate, ultraviolet/hydrogen peroxide), and disinfection (i.e., ozonation, chlorine dioxide, chlorination, and chloramination) were mostly ineffective in removing PFASs. However, anion exchange and granular activated carbon treatment preferably removed longer-chain PFASs and the PFSAs compared to the PFCAs, and reverse osmosis demonstrated significant removal for all the PFASs, including the smallest PFAS, perfluorobutanoic acid.

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* *Corresponding author.* Tel.: +1 720 984 2116.
** *Corresponding author.* Tel.: +1 702 856 3668.
E-mail addresses: chiggins@mines.edu (C.P. Higgins), eric.dickenson@snwa.com (E.R.V. Dickenson).

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1. Introduction

Poly- and perfluoroalkyl substances (PFASs) are a group of chemicals that have been used directly in or as part of the manufacturing of a wide variety of industrial and consumer products including, but not limited to, firefighting foams, paper and cardboard coating materials employed in food packaging, ScotchGard™, and Teflon™. One class of PFASs are the perfluoroalkyl acids (PFAAs), which are stable chemicals made of a carbon backbone surrounded by fluorine atoms and a terminal acid group, such as carboxylic or sulfonic acid. These PFAAs are extremely resistant to degradation and thus stable in both water and soil and highly persistent in the environment (Buck et al., 2011; ATSDR, 2009).

Unlike most other persistent and bioaccumulative organic pollutants (e.g. dioxin, polychlorinated biphenyls), PFAAs are water soluble. A number of PFAAs have been detected in U.S. surface waters, including lakes, rivers, and tributaries, in the ng/L range or lower (Furdui et al., 2008; Mak et al., 2009; Nakayama et al., 2007; Plumlee et al., 2008; Post et al., 2012; ATSDR, 2009), and they have also been detected in ground waters at similar concentrations (Plumlee et al., 2008; Post et al., 2009). In one study (Sinclair and Kannan, 2006), several PFAAs were detected in effluents from wastewater treatment plants in New York, with concentrations in the low ng/L range for all of the chemicals tested, except for perfluorooctanoic acid (PFOA), which had median concentrations for each utility ranging from 67 ng/L to 697 ng/L. The high persistence of PFAAs in the environment and widespread detection has created a concern for the possible exposure to animals and humans. PFOA, perfluorononanoic acid (PFNA), perfluorooctane sulfonic acid (PFOS), and perfluorohexane sulfonic acid (PFHxS) are found in the serum of virtually all U.S. residents (Kato et al., 2011). Some of these chemicals, including PFOA and PFOS, have been detected in the blood of animals in remote regions of the world (Houde et al., 2006). Due to concerns about widespread occurrence and potential health effects, major U.S. manufacturers of PFOS and PFHxS stopped production in 2002 (Butenhoff et al., 2009), while the worldwide use of PFOA and longer chain PFCA homologues is currently being phased out by their major manufacturers (Lindstrom et al., 2011). Replacements for these phased-out compounds include less extensively studied shorter chain PFASs, such as PFHxA and other fluorinated compounds, which generally are more rapidly excreted in humans and animals than the longer chain-length compounds (DuPontTM, 2011; Gordon, 2011). However, these shorter-chain PFASs are still highly persistent in the environment (Ritter, 2010; Gordon, 2011).

As far as potential human exposure is concerned, some of these chemicals have also been detected in U.S. finished/tap waters in the low ng/L range (Post et al., 2009; Quiñones and Snyder, 2009), and higher levels (hundreds of ng/L to several μg/L) of some PFASs have been found in finished drinking water, particularly near sites of industrial use (Emmett et al., 2006; Rumsby et al., 2009; Skutlarek et al., 2006). Drinking water exposure is one pathway that may have contributed to increased concentrations in the serum of humans in most developed countries (Post et al., 2012; ATSDR, 2009) and serum levels of PFOA, PFOS, and PFHxS were increased in communities with contaminated drinking water (ATSDR, 2013; Hölzer et al., 2008; Post et al., 2009). Other potential routes of human exposure include ingestion from food, food packaging, treated carpets, upholstery, and clothing, house dust, protective sprays, and waxes sold as consumer products (Post et al., 2012).

Human epidemiological studies have found associations with numerous health endpoints resulting from exposure, including drinking water exposure (Post et al., 2012; Saikat et al., 2013). An independent panel of prominent environmental epidemiologists concluded that probable links exist between six adverse health conditions and PFOA exposure in a community with contaminated drinking water, including kidney cancer and testicular cancer (C8 2012, Vieira et al., 2013). A number of associations of PFAA exposure with health effects have been reported in the general population, including a study that found an association between PFOA and PFOS exposures and a reduced humoral immune response to routine childhood immunizations in children aged 5 and 7 years (Grandjean et al., 2012).

In 2009, the U.S. Environmental Protection Agency (EPA) established Provisionary Health Advisory (PHA) values for PFOA and PFOS of 0.4 and 0.2 μg/L, respectively, in response to an emergency situation in Decatur, Alabama, which were meant to protect from short term exposure (USEPA, 2013). In addition, PFOS and PFOA have been added to the EPA's Contaminant Candidate List 3 of chemicals under consideration for future drinking water regulation in the U.S. (USEPA, 2009). These two chemicals as well as perfluoroheptanoic acid (PFHpA), PFNA, perfluorobutane sulfonic acid (PFBS), and PFHxS were also added to the EPA's Unregulated Contaminant Monitoring Rule 3 (UCMR 3) which requires nationwide monitoring by public water suppliers to provide occurrence data needed for regulatory decision making (Post et al., 2012; USEPA, 2012). As some PFAAs have been shown to have a potential to harm humans, it is imperative that treatment options be examined for their ability to remove these chemicals from contaminated water sources.

Previous studies assessed the occurrence of PFAAs in raw and finished waters of full-scale conventional drinking water treatment trains (Post et al., 2009; Quiñones and Snyder, 2009), where the latter study examined treatment systems that consisted of coagulation/flocculation, filtration, ozonation, chlorination, and chloramination processes (Quiñones and Snyder, 2009). These studies suggested these treatment systems were ineffective towards PFAA removal, but confirmation of these results has not been shown at other types of full-scale systems or for a wider spectrum of PFASs. To date, some less commonly employed processes, such as anion exchange (AIX), granular activated carbon (GAC), nanofiltration (NF), and reverse osmosis (RO) have been evaluated at the bench scale and showed promise in the removal of some of these chemicals (Deng et al., 2010; Lampert et al., 2007; Steinle-Darling and Reinhard, 2008; Tang et al., 2006; Appleman et al., 2013). GAC and RO have also been evaluated at the full-scale in a few studies performed outside of the U.S. (Eschauzier et al., 2012; Flores et al., 2013; Takagi et al., 2011; Thompson et al., 2011), where GAC was somewhat ineffec-

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tive for the shorter chain-length PFAAs, though RO was able to remove all PFAAs present in the feed water. The performance of AIX has yet to be validated at the full-scale, and demonstration of full-scale GAC and RO performance has been limited in the U.S.

The objective of this study was to evaluate the ability of a wide spectrum of full-scale treatment techniques to remove PFASs from contaminated raw water or potable reuse sources. The novelty of this study lies in the targeted examination of a wide range of U.S. full-scale treatment techniques, including conventional processes and advanced technologies, such as GAC, AIX, RO, advanced oxidation process (AOP), ultrafiltration (UF), dissolved air flotation (DAF), and river bank filtration (RBF). In addition, the removal of a wide range of PFASs, including all six PFASs on the UCMR 3 List, was evaluated. This study also brings new information on the occurrence levels and distribution of an expanded list of PFASs (including some compounds that were not previously examined that have the potential to degrade into PFAAs) in raw and treated waters throughout the U.S., though water sources thought to be potentially impacted by PFASs were specifically targeted. To this end, a large suite of 23 PFASs [Table S1 in the Supplemental Information (SI)] was analyzed in raw water, finished drinking water, potable reuse product water, and at various steps along the treatment train. To enable this, samples were collected during multiple sampling events for 15 full-scale treatment systems throughout the U.S., including 2 potable reuse treatment systems.

2. Materials and methods

2.1. Site selection

The majority of the utilities chosen for this study (Table S2) were selected because they were either known from previous monitoring or expected based on their source waters to contain detectable PFASs (i.e., impacted by upstream wastewater effluent discharge). As a result, occurrence data presented herein are not meant to be representative of the national occurrence of PFASs in North American drinking water source waters. PFASs concentrations were measured in 20 source waters located in the states of Wisconsin (1), Oklahoma (1), Alaska (1), California (2), Alabama (1), Colorado (2), Ohio (1), Nevada (1), Minnesota (1) and New Jersey (9). The source waters included 11 surface waters, 6 groundwaters, 1 surface water and groundwater blend and 2 treated wastewaters. The specific treatment processes employed by each utility was another criterion for the site selection process, which includes somewhat common treatment techniques such as coagulation followed by sedimentation or DAF and/or filtration (i.e., granular, ultrafiltration, microfiltration), aeration and oxidation/disinfection (chlorine, chlorine dioxide, ozone, chloramination, potassium permanganate), as well as less commonly employed treatment techniques such as RBF, GAC, AIX, AOP, and RO. Synoptic grab samples were taken before and after each treatment process (except for RBF), with almost all treatment processes evaluated on at least two separate occasions. A 3% average error for duplicate samples was calculated and a 10% error associated with grab sampling was assumed, since composite samples were not collected. Additional details on sampling and the treatment trains employed at each utility can be found in the SI as well as sampling data associated with each sampling event. Source water was sampled at all sites, and individual treatment steps and finished or potable reuse product waters were evaluated at 15 treatment systems: Utilities 4, 5, 7, 8, and 10-20 (Table 1).

2.2. Sample extraction and analysis

Chemicals and Reagents. With the exception of the analysis of water samples from Utility 20, analytical standards and isotopically labeled standards for all PFASs measured in this study (Table S1) were procured from Wellington Laboratories (Guelph, Ontario, Canada). This analytical suite of 23 chemicals included 9 perfluorocarboxylic acids (PFCAs), 4 perfluorosulfonic acids (PFSAs), perfluorooctane sulfonamide (FOSA), 2 perfluorosulfonamidoacetic acids, 4 fluorotelomer unsaturated carboxylic acids (FTUCAs), and 3 fluorotelomer sulfonic acids (FTSAs). The list of precursor chemicals in this study is not comprehensive, and was limited by the method applied. Whenever possible, matched isotope standards were used for quantitation of each PFAS. As discussed below, samples from Utility 20 were analyzed separately using an alternative protocol, as detailed in the SI. Working stock PFAS solutions and calibration standards were prepared in methanol and appropriate dilutions were made for automated solid phase extraction (ASPE) spiking solutions. All solutions and standards were stored at −20 °C. Trace analysis grade methanol and methyl *tert*-butyl ether (MTBE) were obtained from Burdick and Jackson (Muskegon, MI, USA). Ascorbic acid was purchased from Mallinckrodt Chemicals (Phillipsburg, NJ,

Table 1 – Treatment trains evaluated in this study.

Utility ID	Treatment train
4	MF/RO/UV-AOP/DI/Cl_2
5	AIX/COAG/FLOC/SED/MF/Cl_2
7	RBF/ARR/SOFT/SCC/UV-AOP/G-FIL (Biological)/GAC
8	SED/COAG/FLOC/SED/G-FIL/GAC/Cl_2
10	MF/UF/RO/UV-AOP
11	AER/COAG/FLOC/SED/G-FIL/ClO_2
12	O_3/DAF/Cl_2/CLM
13	UV/Cl_2
14	AIX/APT/Cl_2
15	Cl_2/MnO_4/G-FIL
16	ClO_2/Cl_2
17	MnO_4/O_3/Cl_2
18	APT/GAC/Cl_2
19	Cl_2
20	GAC/Cl_2

AER: Aeration, AIX: Anion Exchange, APT: Aeration Packed Tower, ARR: Aquifer Recharge and Recovery, CLM: Chloramination, Cl_2: Hypocholorous/Hypocholorite, ClO_2: Chlorine Dioxide, COAG: Coagulation, DAF: Dissolved Air Flotation, DI: Direct Injection, O_3: Ozone, FLOC: Flocculation, GAC: Granular Activated Carbon Filtration, G-FIL: Granular Filtration, GW: Ground Water, MF: Microfiltration, MnO_4: Permanganate, RBF: River Bank Filtration, RO: Reverse Osmosis, SCC: Solids Contact Clarifier, SED: Sedimentation, SOFT: Softening, UF: Ultrafiltration, UV: UV Photolysis, UV-AOP: UV Photolysis with Advanced Oxidation (Hydrogen Peroxide).

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USA) and concentrated sulfuric acid was obtained from EM Scientific (Merck KGaA, Darmstadt, Germany). Reagent grade water was prepared with a Milli-Q Gradient water purification system (Millipore, Billerica, MA, USA).

2.3. Sample analysis

Samples were prepared using ASPE, with ASPE extracts analyzed by liquid-chromatography tandem mass-spectrometry (LC-MS/MS) using a previously reported method (Quiñones and Snyder, 2009), adapted and expanded to include all analytes of interest. Details of the ASPE and LC-MS/MS methods as well as the extensive quality assurance and quality control measures [e.g., method reporting limits (MRLs)] are provided in the SI.

3. Results and discussion

3.1. Source and finished water occurrence data

Though the primary objective of this study was to evaluate the removal of PFASs during full-scale water treatment, meeting such an objective required some understanding of PFAS occurrence in source and finished waters. End-product PFAAs were frequently detected in both the source and finished waters of many of the utilities sampled in this study. The three most commonly detected PFASs in the source water samples (n = 39) were PFOS (84%) (MRL = 0.25 ng/L), PFHxA (79%) (MRL = 0.5 ng/L), and PFHxS (79%) (MRL = 0.25 ng/L), though the MRLs of the PFASs did vary. Other chemicals that were frequently detected in the source water samples included perfluoropentanoic acid (PFPeA) (74%) (MRL = 2 ng/L), PFHpA (74%) (MRL = 0.5 ng/L), PFOA (74%) (MRL = 5 ng/L), PFNA (66%) (MRL = 0.5 ng/L), and PFBS (74%) (MRL = 0.25 ng/L). Interestingly, PFHxA and PFPeA were frequently detected, but they are not included in the UCMR 3. Also, the occurrence frequency of PFOA could be higher considering its MRL is 10–20 times higher. Among the PFCAs, perfluorobutanoic acid (PFBA) (MRL = 5 ng/L) and perfluorodecanoic acid (PFDA) (MRL = 0.25 ng/L) were only present in 34% of the samples, though the MRL for PFBA is 10–20 times higher, whereas longer chain PFCAs, perfluoroundecanoic acid (PFUnA) (MRL = 0.5 ng/L) and perfluorododecanoic acid (PFDoA) (MRL = 0.25 ng/L), were only detected in 13% and 11% of samples, respectively. 6:2 fluorotelomer sulfonic acid (6:2 FTSA) (MRL = 0.5 ng/L), *N*-methyl perfluorooctane sulfonamidoacetic acid (*N*-MeFOSAA) (MRL = 0.25 ng/L), *N*-ethyl perfluorooctane sulfonamidoacetic acid (*N*-EtFOSAA) (MRL = 0.25 ng/L), and FOSA (MRL = 0.25 ng/L) were also detected in 38%, 29%, 24% and 16% of samples, respectively. Several chemicals, such as perfluorodecane sulfonic acid (PFDS) (MRL = 0.1 ng/L), the FTUCAs (MRLs = 2 ng/L), 4:2 fluorotelomer sulfonic acid (4:2 FTSA) (MRL = 0.5 ng/L), and 8:2 fluorotelomer sulfonic acid (FTSA) (MRL = 0.5 ng/L) were not detected above MRLs in any of the samples. PFAS detection frequency in the finished waters from conventional treatment trains containing coagulation/flocculation, filtration (i.e., granular, ultrafiltration, microfiltration), and/or disinfection was similar to the source waters, except for PFDoA which was not detected in any finished water sample.

The range of concentrations found in the source and finished or potable reuse product waters from the sampling campaigns in this study as well as the ranges of North American values from a detailed, though not comprehensive, literature survey can be found in Table S5. Ranges include all of the concentrations of the source and finished or product samples measured in this study and are grouped by the utility's raw water source (i.e. surface waters, ground waters, blend of surface and groundwater, or Utilities 4 and 10 whose "raw water source" is 100% treated wastewater effluent). In addition, Tables S6 and S7 contain the concentrations of all samples analyzed in this study. Data from Utility 20 had higher levels than the other sites because its raw water source is highly contaminated by an industrial source. These data are not presented in Table S5, but are displayed in a different format because there are more data for this site. The concentration ranges measured in this study were similar for the chemicals that had previously been quantified in other studies in North America for sites that were believed not to be heavily contaminated by industrial sources. The highest concentrations measured were in the source water of Utility 10 at 370 and 220 ng/L of PFPeA and PFOA, respectively. Utility 10, as well as Utilities 4 and 7, generally had higher concentrations of most PFASs, which corresponds with all of these being highly wastewater-impacted sources, where Utilities 4 and 10 are 100% impacted. *N*-MeFOSAA, *N*-EtFOSAA, FOSA, FTUCAs and FTSAs (except 6:2 FTSA) were not detected in ground waters, but FOSA, *N*-MeFOSAA, and *N*-EtFOSAA were in the surface waters of Utilities 5, 7, and 17 (up to 1.7, 0.9, and 2.0 ng/L, respectively) and the treated wastewater effluents of Utilities 4 and 10 (up to 0.42, 1.1, and 0.43 ng/L, respectively).

The detected PFASs in finished waters from drinking water treatment systems (except for Utility 20), were in the low ng/L range, where the highest detection was 62 ng/L of PFHxA at site 19. The MRLs in this study (Table S3) were much lower than the EPA method for UCMR 3, which are 10 ng/L for PFHpA, 20 ng/L for PFOA, and 40 ng/L for PFOS (USEPA, 2012). Three PFAAs were found in some samples to contain higher concentrations than the UCMR 3 MRLs. Four utilities (Utility 5, 14, 16 and 19), were above the 10 ng/L level for PFHpA with values of 14, 13, 11, and 34 ng/L, respectively, in at least one of their finished water samples. Six utilities were above the 20 ng/L level for PFOA: Utility 5 (two samples: 32 and 50 ng/L), Utility 11 (33 ng/L), Utility 14 (21 ng/L), Utility 15 (38 ng/L), Utility 18 (two samples: 24 and 27 ng/L), and Utility 19 (57 ng/L). Only Utility 5 was above the 40 ng/L level for PFOS in one of the two finished water samples taken (measured at 61 ng/L). However, none of the finished water samples exceeded the MRLs of 20, 90, and 30 ng/L for PFNA, PFBS, and PFHpA, respectively, on the UCMR 3 list. Of the six chemicals that will be monitored under the UCMR 3, the results here suggest that PFHpA, PFOA, and PFOS, could be the most frequently detected. PFOA, PFOS, PFBA, and PFBS were measured at levels below U.S. EPA and state guidance levels, with the exception of one utility. Utility 19 in New Jersey was over (at 57 ng/L) the state guidance level of 40 ng/L for PFOA. Collectively, these data suggest that if existing public health guidelines for these four chemicals are applied,

the observed levels will be below the guidance levels for the majority of the utilities examined in this study.

3.2. *Full scale treatment efficacy*

Fig. 1 displays the removal efficacy of the well-studied PFAAs, PFOA and PFOS, as a result of the various types of treatments employed by the utilities. Similar plots for all measured PFAAs can be found in the SI (Figures S1-1 to S1-11). Each treatment type had its influent and effluent sampled twice, except for Utilities 7, 11, and 15 which were sampled once, and the average and standard error of the two sampling campaigns are displayed.

3.2.1. *Coagulation/physical separation*

The efficacy of PFAS removal from coagulation followed by sedimentation or DAF and/or filtration was evaluated. Added coagulants included aluminum sulfate and polymer (Utility 8), aluminum sulfate (Utility 11), and polyaluminum chloride (PACl; Utility 12). Coagulation followed by sedimentation did not lead to PFAA removal, but where DAF was used instead of sedimentation (such as at Utility 12), a 49% removal of PFOS was observed (Fig. 1). Similarly, PFNA was also removed, albeit to a lesser extent (29%) by DAF. However, the detected shorter-chain PFCAs and PFSAs were not well removed by DAF. It is possible that the longer-chain PFAAs may have a higher affinity for the air/water interface resulting from the DAF air bubbles and are subsequently skimmed off with the surface scum. Improved removal of these longer-chain PFAAs by DAF should be studied further. Other physical separation technologies such as granular filtration with sand (Utility 8) and an anthracite/sand combination (Utility 12) had little impact on PFAA removal. An increase in PFOS concentration was observed after the coagulation/sedimentation/granular filtration processes at Utilities 8 and 11 However, the average level was within the error of duplicate sampling events for Utility 8, whereas for Utility 11, the data presented are only from one sampling campaign.

With respect to low pressure membrane filtration, the MF system at Utility 4 was not effective; in fact, a slight increase in PFOS concentration was observed at this utility, though it is within the error of duplicate sampling events. The system uses polypropylene membranes with 0.2 micron rated pore size. Utility 10 splits their flow and runs the water in parallel through an MF system with a Microza MF Model UNA 620 A membrane having a nominal pore size of 0.1 micron, and a UF system with a nominal pore size of 0.02 micron. This MF/UF system was also mostly ineffective, however partial reductions were observed, i.e., 24% of PFOS, 44% of PFDoA and 42% of FOSA. Given the large MF/UF pore sizes and the molecular weights of these compounds (499–614 g/mol), they should pass through these membranes with ease and it is unknown why partial reductions of these chemicals were observed.

3.2.2. *Oxidation/Aeration/Disinfection*

Oxidation and disinfection processes were evaluated at Utilities 7, 11–15, 17, and 18 and included ozonation, aeration packed towers, potassium permanganate, ultraviolet (UV) treatment, AOP (UV/H_2O_2), chlorination (Cl_2) with and without chloramination, and chlorine dioxide. All of these processes proved mostly ineffective at all of the utilities (Fig. 1). In fact, in many cases, the concentration of PFCAs and PFSAs were consistently slightly higher following oxidative treatments.

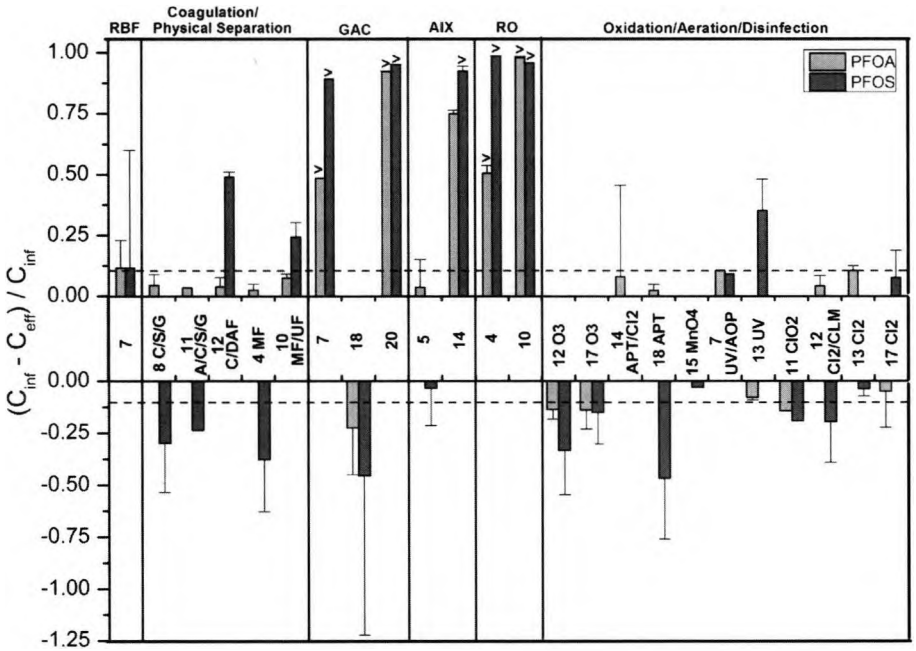


Fig. 1 – PFOA and PFOS Removal by Treatment: A greater than symbol " > " denotes samples where the concentration was below the MRL in the effluent, but the MRL was used as a conservative estimate for the percent removal. The dashed lines at ±10% represent the range of error based on the average analytical variability multiplied by a conservative safety factor (~3×) for the possible error associated with collecting grab samples before and after treatments. C_{inf}: influent concentration. C_{eff}: effluent concentration, A: Aeration, C: Coagulation, S: Sedimentation, G: Granular Filtration.

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However, these data are generally within the range of error associated with sampling and analytical variability (i.e., ±10%).

The lack of removal of PFAAs via these full-scale processes is not surprising, as PFAAs are generally resistant to oxidation. AOPs, which utilize the hydroxyl radical, such as alkaline ozonation, peroxone, Fenton's reagent, and UV/hydrogen peroxide, have been shown ineffective towards PFOA and PFOS at the bench-scale (Hori et al., 2004; Moriwaki et al., 2005; Schroder and Meesters, 2005). Other oxidation/reduction technologies, such as photocatalytic oxidation, photochemical oxidation, photochemical reduction, persulfate radical, thermally-induced reduction, and sonochemical pyrolysis, have been shown to be effective at degrading some PFAAs in water (Hori et al., 2005; Lazerte et al., 1953; Moriwaki et al., 2005; Yamamoto et al., 2007). However, most of these technologies are not employed in current drinking water treatment practices.

One exception in this group was the UV system (80 mJ/cm^2) at Utility 13, which interestingly resulted in partial removal of PFHxS (34%) and PFOS (35%) (Fig. 1). UV photolysis has been demonstrated to be effective at degrading PFOS and PFOA (Chen et al., 2007; Chen, 2006; Hori et al., 2004; Yamamoto et al., 2007). However, the UV system at Utility 13 did not show removal of the PFCAs or the smaller chain sulfonate, PFBS. In addition, the UV/hydrogen peroxide process at Utility 7 operated at a high dose (>500 mJ/cm^2) for trace organic contaminant removal was not effective. Given these mixed results, one might expect minimal removal, if any, of PFAAs in contaminated drinking water as a result of current UV treatment practices.

3.2.3. RBF

RBF was tested twice at Utility 7, with a travel time of approximately 10 days. Although minimal removal was observed for PFOA and PFOS, removal of the other chemicals was variable. For example, some chemicals showed removal, such as PFHxA (20%), PFDA (19%), and N-MeFOSAA (68%), while other chemicals actually increased in concentration, such as PFBA (−103% removal), PFHpA (−31%), PFBS (−63%), and FOSA (−20%). This variation is possibly due to variability in influent concentrations from the wastewater effluent impacted river, combined with the fact that raw and post-RBF samples were not collected synoptically for the 10 day travel time. Another potential explanation is the transformation of precursor compounds into end products through the riverbank. One example of the influent variation is for PFOS, which had the largest difference in concentration between samples of the raw water with levels at 7.3 ng/L during the first sampling event and 35 ng/L during the second sampling event just six weeks later. Similar variation was observed in a study on infiltrated water from the infiltrated Rhine River water in the Netherlands (Eschauzier et al,. 2010).

3.2.4. *AIX treatment*

Two AIX treatments were examined at the full-scale in this study, though they were not designed for PFAS removal. Utility 14 added Purolite FerrIX A33e media to a softener vessel which is the first treatment process in the train (Table 1). The iron infused AIX resin is designed for arsenic removal, and is only changed out once performance decreases in this aspect. The system was installed in December 2011, but is only used when the arsenic concentration is high enough that removal is necessary; therefore the resin for the two sampling events was at most 5 and 9 months old. Interestingly, the resin was successful in reducing some of the PFAS levels. In particular, PFHpA was partially removed (46%), as were PFOA (75%), and PFBS (81%). PFNA, which was only detected in one of the two raw water samples, exhibited >67% removal, whereas PFHxS and PFOS exhibited >97%, and >92% removals, respectively. The PFSAs were preferably removed over the PFCAs. Examining the removal efficiencies of both campaigns in Table 2, there appears to be a chain length effect for this particular ion exchange treatment, with the smaller chain PFCAs (<314 g/mol), i.e., PFBA and PFHxA, exhibiting little to no removal, and the larger chain ones (>364 g/mol), i.e., PFHpA and PFOA, showing partial to significant removal. Similarly, in batch tests, an AIX resin (Siemens A-714) demonstrated >99% removal of PFOS and PFOA (Lampert et al., 2007), while other bench-scale studies (Deng et al., 2010; Senevirathna et al., 2010) showed AIX resins capable of removing PFOS. It is possible that certain AIX resins can target PFAS sorption by ion exchange and/or hydrophobic interactions.

A magnetic anion exchange treatment process was also examined at Utility 5, where it is employed upfront in the treatment train (Table 1) to target total organic carbon. Unlike Utility 14 and previous bench-scale work, this process showed little to no removal of any of the chemicals at Utility 5. This may have been due to the operational method employed for this treatment process. The AIX system at Utility 5 uses an upflow fluidized-bed reactor with continuous regeneration (approximately 13 regenerations in a 24 h period). Continual regeneration, as opposed to a complete resin replacement, or insufficient capacity due to improper operation and/or kinetics may be causes of its ineffectiveness at the full-scale. Previous studies have shown that conventional methods of AIX regeneration are ineffective for PFBS and PFOS (Carter and Farrell, 2010) and significant kinetic limitations can occur (Deng et al., 2010; Lampert et al., 2007).

The results from Utility 14's AIX column operation and past bench-scale studies indicate AIX is a promising technology for the removal of PFASs, despite what was observed at Utility 5. In order for AIX to be employed at the full-scale for PFAS removal, further AIX research is needed to identify which AIX resins are most suitable for PFAS removal, the selectivities of resins for different PFAS, suitable regeneration techniques, and required frequency for resin changes. In addition, a better understanding of sorption kinetics and competition with other anions and natural organic matter is necessary.

3.2.5. *GAC treatment*

Four full-scale GAC systems were examined. Details for these GAC systems at Utilities 7, 8, 18, and 20 can be found in the SI. PFAS levels at Utility 8 were too close to the MRLs, and as a result, GAC performance for this system was not analyzed. Utility 20 is of particular interest, as it specifically employs GAC for PFAS removal from groundwater. The system utilizes Calgon F600 (coal-based) media, and is set up with two contactors, a lead and a lag, that run in series with a flow between 1.4 and 1.5 m^3/min, and an empty bed contact time (EBCT) of

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Table 2 – Percent removal for most effective PFAS treatment technologies.

Site	#4	#4	#10	#10	#14	#14	#7	#20
Treatment	RO	RO	RO	RO	AIX	AIX	GAC	GAC
Sample date	12/6/2011	2/22/2012	1/9/2012	3/6/2012	5/30/2012	9/19/2012	8/21/2012	4/25/2007–4/22/2008
PFBA	>90%	>82%	N/A	>95%	−9%	0%	33%	−17%
PFPeA	>79%	>82%	>99%	>98%	0%	0%	74%	>22%
PFHxA	>97%	>98%	>99%	>99%	14%	−14%	91%	>68%
PFHpA	>81%	>86%	>98%	>95%	54%	38%	>89%	N/A
PFOA	>54%	>47%	>98%	>98%	76%	73%	>48%	>92%
PFNA	>87%	>87%	>98%	>95%	N/A	>67%	>37%	N/A
PFDA	>76%	>67%	>99%	>99%	N/A	N/A	N/A	N/A
PFUnA	N/A	N/A	>77%	>71%	N/A	N/A	N/A	N/A
PFDoA	N/A	N/A	>87%	>84%	N/A	N/A	N/A	N/A
PFBS	>93%	>98%	>96%	>94%	83%	80%	>96%	N/A
PFHxS	>95%	>94%	>96%	>90%	>97%	>98%	>96%	>41%
PFOS	>98%	>99%	>96%	>96%	>90%	>94%	>89%	>95%
PFDS	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
FOSA	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
N-MeFOSAA	>43%	>36%	>84%	>79%	N/A	N/A	N/A	N/A
N-EtFOSAA	N/A	N/A	>55%	>58%	N/A	N/A	N/A	N/A

N/A – not available.

approximately 13 min in each contactor. For this system, the removal efficacy reported in Table 2 was based on the average removal over the course of one year for the lag basin (4/25/2007–4/22/2008). During this period the lag effluent concentrations fell below the detection limits for all chemicals, with the exception of PFBA.

Fig. 2 displays the breakthrough of five different PFASs in the lead GAC bed over a five year period at Utility 20. Similar initial breakthrough curves of PFBA, PFPeA, and PFHxA within the first two years were observed previously (Eschauzier et al., 2011). Every three months corresponds to approximately 10,000 bed volumes (BVs). The BV axis stops at 70,000 BVs because the system was first altered on 8/13/2008 (or ~69,000 BVs). Three important dates during the operation of this system, noted on the horizontal axis in Fig. 2, are 09/22/2008, 10/05/2009, and 10/11/2010. On the first two dates, the flow was redirected so that the lag vessel became the lead, and the original lead vessel had its carbon replaced with virgin carbon. On 10/11/2010, all of the carbon in the system (both lead and lag vessels) was replaced with virgin carbon. These dates are reflected by the sharp decline in effluent concentrations for PFHxA, PFOA, and PFOS in the lead vessel. PFBA only exhibited a sharp decline when all of the carbon in the system was replaced. On 12/27/2007, the effluent value of PFHxA had a sharp decline back to below its MRL, but this observation is believed to be an outlier, as the GAC was not altered in anyway on this date. PFHxS was detected in the influent over the timespan, but all lead effluent samples were below the detection limits. Other than PFHxS, PFOS was the slowest to break through and PFBA was the fastest. The chemicals PFHxA and PFOA appear to exhibit similar initial breakthrough times in the lead GAC, but the smaller chain PFHxA reaches full breakthrough at a much faster rate. These findings are in general agreement with several bench-scale studies of the removal of PFASs using coal-based GAC that showed GAC was capable of removing PFOA, PFBS, and PFOS (Carter and Farrell, 2010; Lampert et al., 2007; Senevirathna et al., 2010; Appleman et al., 2013).

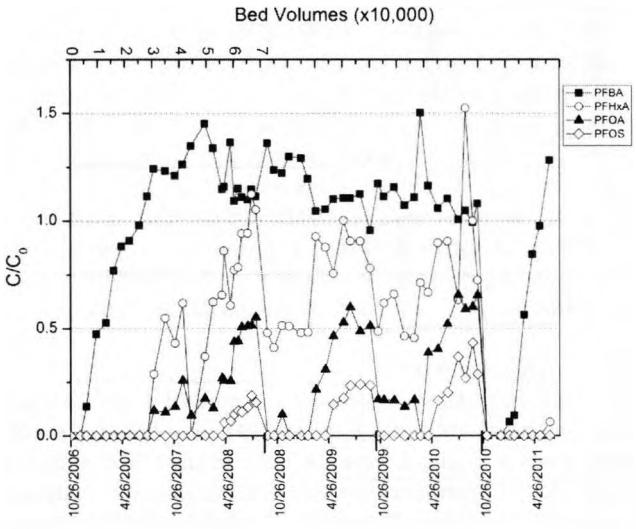


Fig. 2 – Breakthrough of PFASs in Utility 20 GAC System. Every three months corresponds to approximately 10,000 BVs. BV axis stops at 70,000 BVs because the system was first altered on 8/13/2008 (or ~69,000 BVs).

As can be seen in Fig. 2, PFBA exhibited breakthrough after about two months of operation, and was above 1.0 for its C/C_0 value from July 2007 to October 2010 (when all of the carbon was replaced). A similar result of increased effluent concentration was observed with PFBS in another study (Eschauzier et al., 2012). This is believed to be due to competitive effects with other sorbing species (perhaps longer chain PFASs and/or organic matter) leading to desorption and release of sorbed PFBA over time, a phenomena that was observed for PFBA (Gellrich and Knepper, 2011). The average influent concentrations were 1.45 ± 0.18 μg/L, so it is unlikely that the $C/C_0 > 1$ was caused by influent variability. This chemical could be a strong candidate for the Critical Compound for GAC

treatment in the removal of PFASs when present in the raw water source.

Utility 18 uses Calgon F300 (coal-based) GAC to treat surface water, but unlike Utility 20, the system is not specifically designed for PFAS removal. For the first sampling event, little change in the concentrations was observed for most of these chemicals. Interestingly, for the second sampling campaign, the concentrations were higher for some of the PFASs [i.e., PFOA (45%), PFDA (34%), and PFOS (122%)], in its effluent than the influent. The GAC for this system had not been reactivated or replaced with virgin carbon in over six years. Thus, these results suggest the GAC had been spent and the second sampling campaign may indicate leaching of chemicals. This finding is important in that it highlights the importance of carbon replacement and/or regeneration for ensuring removal of PFASs by GAC. This was also seen in the Takagi et al. (2011) study where fresh carbon was deemed effective for PFOA and PFOS removal, but the same carbon tested one year later was no longer effective at the removal of these chemicals.

Utility 7 also employs GAC treatment with six parallel GAC contact chambers containing 24.3 m^3 of Norit (coal-based) GAC300, an empty bed contact time of ~10 min, and an average flow of about 114 m^3/d. This GAC system was specifically designed to target the removal of trace (ng/L) organic contaminants. The removal percentages for this site are displayed in Table 2. The longer chain PFCAs (PFHpA, PFOA, and PFNA), as well as the PFSAs (PFBS, PFHxS, and PFOS), were all removed to below MRLs. Three shorter chain PFCAs (PFBA, PFPeA, and PFHxA), had partial removal at 33%, 74%, and 91%, respectively, which corresponds to the increased affinity of longer-chain PFCAs for organic carbon as observed in previous studies (Eschauzier et al., 2012; Appleman et al., 2013; Higgins and Luthy, 2006).

The overall results indicate for the longer chain PFASs, GAC systems were effective at Utilities 7 and 20. Utilities 7 and 20 appear to have chain length dependent PFAS breakthrough, and also the sulfonic acids being removed for a longer period of time than the carboxylic acids. Utility 20 operated its lead vessels for approximately 10 months before initial breakthrough of PFHxA or PFOA, and for 18 months before PFOS started to breakthrough. It is important to point out that the presence of dissolved organic matter either in groundwater or surface water can have an effect on the breakthrough behavior (Appleman et al., 2013). However, if the shorter-chain PFBA is targeted for removal, an alternative treatment strategy would need to be employed.

3.2.6. *RO treatment*

Two U.S. California potable water reuse plants employing RO were examined in this study (Utilities 4 and 10). Utility 4 uses polyamide Hydranautics ESPA2 membranes in a three stage array with a 12 gfd flux rate and 85% recovery, and Utility 10 uses Toray and Hydranautics RO membranes with an RO flux rate of 12 gfd and 80% recovery. All PFASs were below the MRLs in the collected samples immediately following the RO systems, making this the most effective form of treatment evaluated in this study. Results in a bench-scale study (Tang et al., 2006) of RO membranes showed similar rejection of PFOS (>99%), and also in a full-scale study in Australia (Thompson et al., 2011) where all PFAAs present in the feed (PFHxA, PFHpA, PFOA, PFNA, PFDA, PFBS, PFHxS, and PFOS) were below their limit of reporting values in the RO permeate.

Effective rejection of PFASs by RO is especially important for the shortest chain PFAS in this study, PFBA, which proved to be recalcitrant through all other treatment techniques evaluated at the full-scale. Despite RO's effectiveness, RO would likely be the most costly method for removal. Less costly membrane systems, such as NF (or tight NF), could prove to be just as capable of rejecting PFASs as compared to RO in full-scale plants, since NF has been deemed potentially effective (>95%) in bench-scale experiments using NF270 membranes (Steinle-Darling and Reinhard, 2008; Appleman et al., 2013). Another important factor to consider with these membrane treatment systems is the disposal of the concentrate, which will contain the rejected PFASs, and add to the cost of operation of these systems.

4. Conclusion

A study on available treatment technologies for the treatment of PFAS was conducted at full-scale U.S. water treatment plants. This study demonstrates that full-scale conventional treatments in the U.S., such as coagulation followed by physical separation processes, and chemical oxidation, aeration and disinfection, were unable to remove PFASs. This will become an issue for some utilities if low ng/L regulatory levels are promulgated for these chemicals. In this event, utilities with significant PFAS levels in their raw water sources will need to examine additional mitigation strategies, such as alternative treatment technologies. The levels of several PFASs, such as PFOA and PFOS, were reduced by current day RO, GAC, and AIX practices at the full-scale. However, GAC and AIX were less effective at removing the shorter chain PFASs, whereas RO treatment was effective for even the smallest PFAS studied, PFBA.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.watres.2013.10.067

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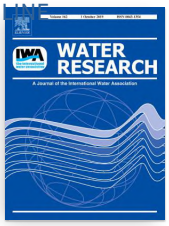
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APPENDIX 1009B

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JOURNAL

Water research

International Association on Water Pollution Research.
Oxford, Pergamon Press.; v. 1- Jan. 1967-; 1967
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Language(s)	English
Description	v. illus.
Frequency	Twenty no. a year, 2002-
Identifier(s)	ISSN : 0043-1354 (Print) ISSN : 1879-2448 (Electronic) OCLC : (OCoLC)01769499
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Water Res. 2014 Mar 15:51:246-55. doi: 10.1016/j.watres.2013.10.067. Epub 2013 Nov 7.

Treatment of poly- and perfluoroalkyl substances in U.S. full-scale water treatment systems

Timothy D Appleman [1], Christopher P Higgins [2], Oscar Quiñones [3], Brett J Vanderford [3], Chad Kolstad [4], Janie C Zeigler-Holady [3], Eric R V Dickenson [5]

Affiliations

PMID: 24275109 DOI: 10.1016/j.watres.2013.10.067

Abstract

The near ubiquitous presence of poly- and perfluoroalkyl substances (PFASs) in humans has raised concerns about potential human health effects from these chemicals, some of which are both extremely persistent and bioaccumulative. Because some of these chemicals are highly water soluble, one major pathway for human exposure is the consumption of contaminated drinking water. This study measured concentrations of PFASs in 18 raw drinking water sources and 2 treated wastewater effluents and evaluated 15 full-scale treatment systems for the attenuation of PFASs in water treatment utilities throughout the U.S. A liquid-chromatography tandem mass-spectrometry method was used to enable measurement of a suite of 23 PFASs, including perfluorocarboxylic acids (PFCAs) and perfluorosulfonic acids (PFSAs). Despite the differences in reporting levels, the PFASs that were detected in >70% of the source water samples (n = 39) included PFSAs, perfluorobutane sulfonic acid (74%), perfluorohexane sulfonic acid (79%), and perfluorooctane sulfonic acid (84%), and PFCAs, perfluoropentanoic acid (74%), perfluorohexanoic acid (79%), perfluoroheptanoic acid (74%), and perfluorooctanoic acid (74%). More importantly, water treatment techniques such as ferric or alum coagulation, granular/micro-/ultra- filtration, aeration, oxidation (i.e., permanganate, ultraviolet/hydrogen peroxide), and disinfection (i.e., ozonation, chlorine dioxide, chlorination, and chloramination) were mostly ineffective in removing PFASs. However, anion exchange and granular activated carbon treatment preferably removed longer-chain PFASs and the PFSAs compared to the PFCAs, and reverse osmosis demonstrated significant removal for all the PFASs, including the smallest PFAS, perfluorobutanoic acid.

Keywords: Anion exchange; Full-scale; Granular actived carbon; Occurrence; PFOA; PFOS; Reverse osmosis; Treatment.

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TI - Treatment of poly- and perfluoroalkyl substances in U.S. full-scale water treatment systems.

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AB - The near ubiquitous presence of poly- and perfluoroalkyl substances (PFASs) in humans has raised concerns about potential human health effects from these chemicals, some of which are both extremely persistent and bioaccumulative. Because some of these chemicals are highly water soluble, one major pathway for human exposure is the consumption of contaminated drinking water. This study measured concentrations of PFASs in 18 raw drinking water sources and 2 treated wastewater effluents and evaluated 15 full-scale treatment systems for the attenuation of PFASs in water treatment utilities throughout the U.S. A liquid-chromatography tandem mass-spectrometry method was used to enable measurement of a suite of 23 PFASs, including perfluorocarboxylic acids (PFCAs) and perfluorosulfonic acids (PFSAs). Despite the differences in reporting levels, the PFASs that were detected in >70% of the source water samples (n = 39)

included PFSAs, perfluorobutane sulfonic acid (74%), perfluorohexane sulfonic acid (79%), and perfluorooctane sulfonic acid (84%), and PFCAs, perfluoropentanoic acid (74%), perfluorohexanoic acid (79%), perfluoroheptanoic acid (74%), and perfluorooctanoic acid (74%). More importantly, water treatment techniques such as ferric or alum coagulation, granular/micro-/ultra- filtration, aeration, oxidation (i.e., permanganate, ultraviolet/hydrogen peroxide), and disinfection (i.e., ozonation, chlorine dioxide, chlorination, and chloramination) were mostly ineffective in removing PFASs. However, anion exchange and granular activated carbon treatment preferably removed longer-chain PFASs and the PFSAs compared to the PFCAs, and reverse osmosis demonstrated significant removal for all the PFASs, including the smallest PFAS, perfluorobutanoic acid.

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FAU - Appleman, Timothy D

AU - Appleman TD

AD - Civil and Environmental Engineering Department, Colorado School of Mines, 1500 Illinois Street, Golden, CO 80401, USA.

FAU - Higgins, Christopher P

AU - Higgins CP

AD - Civil and Environmental Engineering Department, Colorado School of Mines, 1500 Illinois Street, Golden, CO 80401, USA. Electronic address: chiggins@mines.edu.

FAU - Quiñones, Oscar

AU - Quiñones O

AD - Applied Research and Development Center, Water Quality Research and Development Division, Southern Nevada Water Authority, Henderson, NV 89015, USA.

FAU - Vanderford, Brett J

AU - Vanderford BJ

AD - Applied Research and Development Center, Water Quality Research and Development Division, Southern Nevada Water Authority, Henderson, NV 89015, USA.

FAU - Kolstad, Chad

AU - Kolstad C

AD - Minnesota Department of Health, St. Paul, MN 55155, USA.

FAU - Zeigler-Holady, Janie C

AU - Zeigler-Holady JC

AD - Applied Research and Development Center, Water Quality Research and Development Division, Southern Nevada Water Authority, Henderson, NV 89015, USA.

FAU - Dickenson, Eric R V

AU - Dickenson ER

AD - Civil and Environmental Engineering Department, Colorado School of Mines, 1500 Illinois Street, Golden, CO 80401, USA; Applied Research and Development Center, Water Quality Research and Development Division, Southern Nevada Water Authority, Henderson, NV 89015, USA. Electronic address: eric.dickenson@snwa.com.

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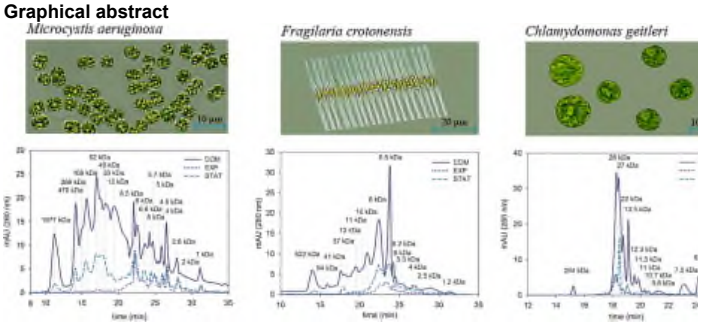
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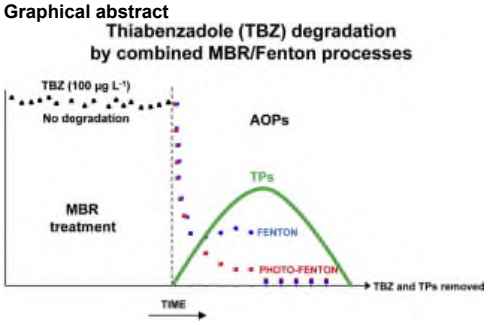
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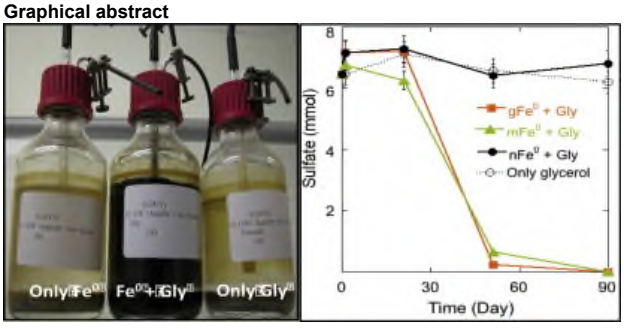
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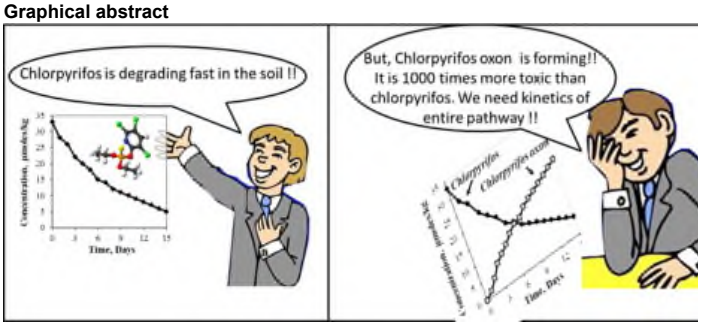
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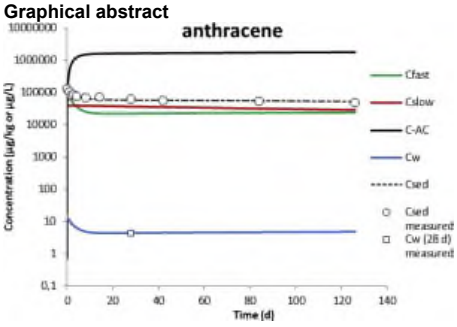
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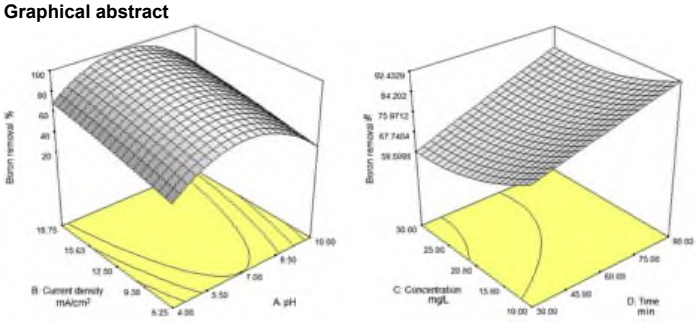
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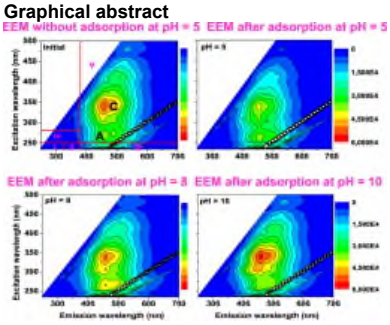
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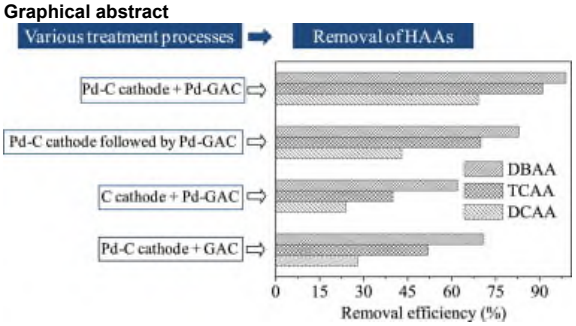
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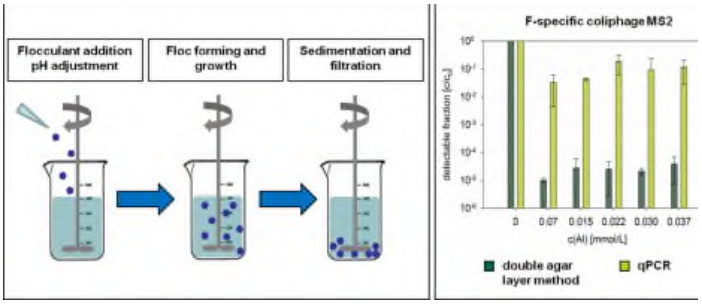
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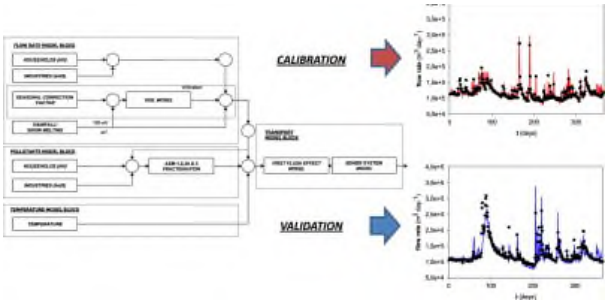
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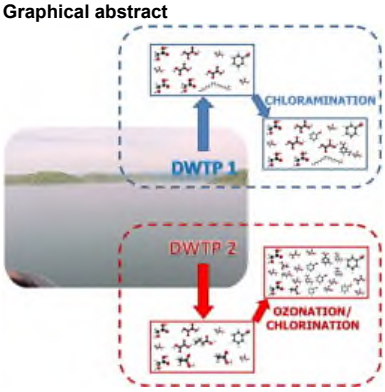
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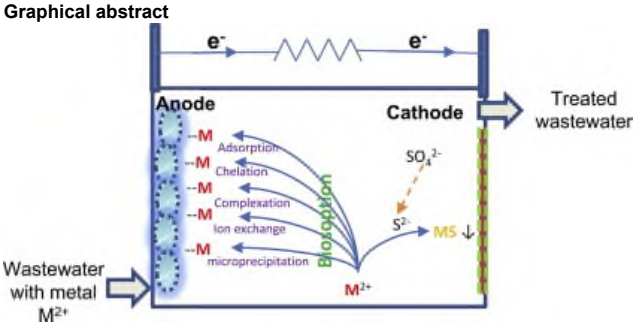
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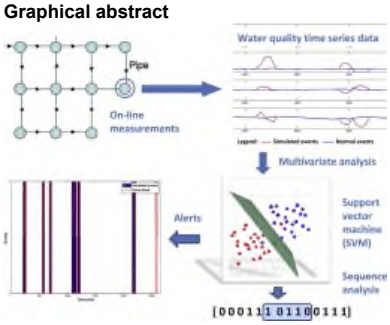
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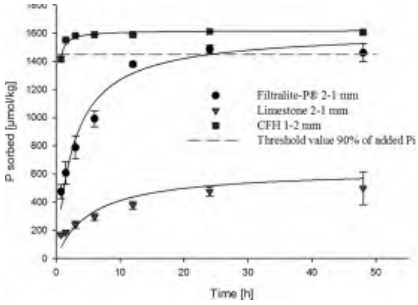
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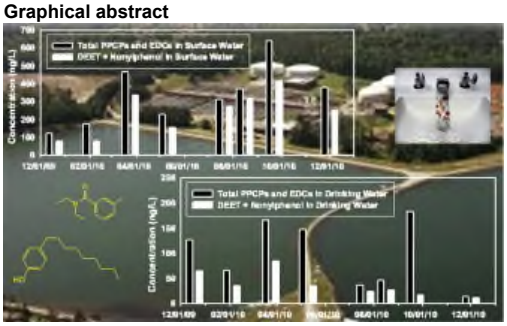
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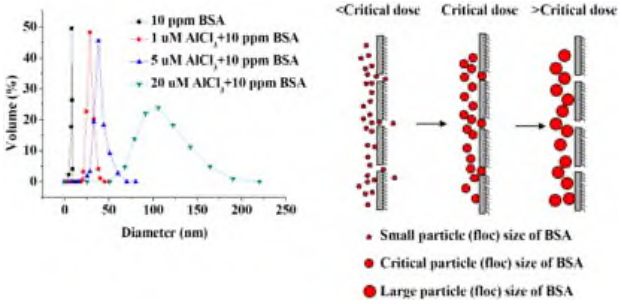
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Effect of Calcium in Field-Spent GACs on Pore Development During Regeneration

Author(s): Fred S. Cannon, Vernon L. Snoeyink, Ramon G. Lee, Gérard Dagois and James R. DeWolfe

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Effect of Calcium in Field-Spent GACs on Pore Development During Regeneration

Fred S. Cannon, Vernon L. Snoeyink, Ramon G. Lee, Gérard Dagois, and James R. DeWolfe

Thermal regenerations were conducted on a granular activated carbon (GAC) that had become exhausted (spent) in a water treatment plant where it had been in service nearly four years. This GAC contained 2 percent calcium. With this field-loaded calcium intact, proper regeneration restored the spent GAC to the same pore structure and surface area that was exhibited by several virgin GACs. When calcium appeared inside the spent GAC, it always caused micropores to be converted to small mesopores during thermal oxidation. In contrast, if the calcium was leached out of the spent GAC with acid, thermal oxidation increased only the micropore volume. Several regeneration variables were tested, including pyrolysis temperature (650–950°C), oxidation temperature (650–950°C), oxidant gas (steam and CO_2), oxidant flow rate, and oxidant extent. All regenerated products of the spent GAC that had been taken to the same targeted apparent density displayed the same surface area and pore structure except those regenerated at low temperatures (650–750°C) with both steam and CO_2 together: these produced carbons with lower volumes of small pores and lower surface areas.

The primary role of granular activated carbon (GAC) in water treatment is to adsorb organic compounds that could potentially harm humans or impart tastes and odors. While the GAC is in service, however, it can also accumulate metals. When the GAC's capacity to adsorb organic compounds becomes exhausted (spent), the GAC can be thermally regenerated for reuse.

This metals accumulation could be high when the GAC serves as a filter adsorber, i.e., when GAC replaces anthracite coal or sand in a conventional filter bed. In such cases the GAC is downstream of iron, aluminum, or lime coagulation process, or a softening process, and it could become heavily laden with these metals.

The goal of this research was to explore how these accumulated metals affected thermal regeneration and to determine whether thermal regeneration could restore the spent GAC's virgin adsorption capacity despite the metals.

When a GAC becomes spent, water utility managers face a major decision. Should the spent GAC be disposed of in a landfill or incinerated and replaced with virgin GAC, or should the GAC be regenerated so it can be reused? If utility managers select the landfill option, they waste a renewable resource and contribute to a national waste disposal dilemma. Moreover, they may be asked some day to prove that the adsorbed organic compounds have not subsequently desorbed from the GAC in the landfill and contaminated the groundwater.

On the other hand, if they select regeneration, they can reuse the GAC rather than discard it. The regeneration process employs the same methods as those used to manufacture virgin GAC or incinerate spent GAC.

Regeneration potentially offers the most economical, energy-efficient, and environmentally sound option. However, any cost or energy analysis would be very sensitive to the adsorption capacity of regenerated carbon relative to virgin carbon. GAC bed volume losses would also weigh heavily, because any lost GAC volume would have to be made up with equal volumes of virgin carbon.

One practical research goal, then, has been to create a thermally regenerated GAC product that manifested the same adsorption properties as its virgin counterpart. As an alternative to monitoring adsorption capacities for the myriad organic compounds present in water, the authors substituted nitrogen pore volumes and surface areas. It was inferred that these parameters were somewhat usefully correlated to adsorption capacities for organic compounds.

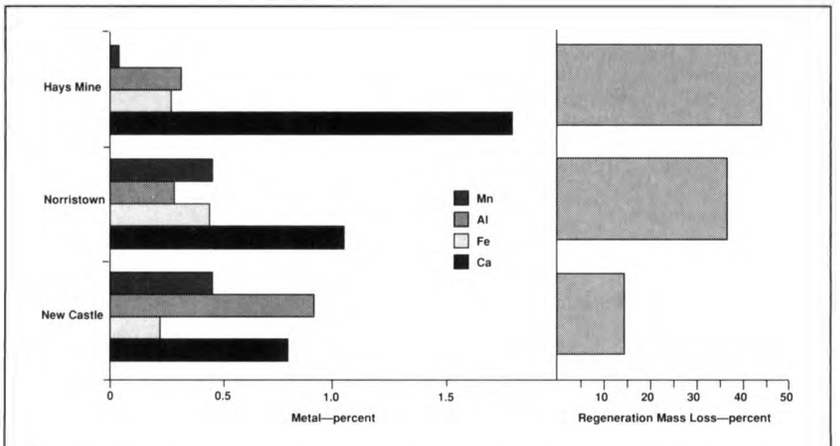


Figure 1. Metals content and regeneration mass loss for spent GACs from three water treatment plants in Pennsylvania *(regeneration at 850°C, with 15 min oxidation in moderate flows or steam and CO_2)*

Regeneration method and strategies

Thermal regeneration includes both a pyrolysis step and an oxidation step. During pyrolysis, the spent GAC is exposed to an inert gas environment at 650–850°C. This step vaporizes the GAC's water, volatilizes volatile organic compounds, and converts nonvolatile organic compounds to char. Volatilization vacates pores, and charring creates very narrow fissures between aromatic units that had previously been associated with one another. Char is an amorphous carbon pyrolysis product generally devoid of oxygen and hydrogen. A larger percentage of the mass of aromatic and unsaturated aliphatic compounds is usually

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converted to char than that of saturated aliphatics.[1,2]

During oxidation, the second step of regeneration, the pyrolyzed GAC is exposed to an environment of steam or CO_2 or both at 700–900°C. These gases partly gasify the solid GAC internal surface and slightly widen the pyrolyzation fissures. One could say that optimum oxidation would widen these fissures sufficiently to accommodate organic carbon adsorption but not so excessively that the solid layer between one fissure and another would be destroyed. If excessive, one wide pore with two adsorption surfaces would be created where two narrower pores with four adsorption surfaces had previously existed.

Metals accumulation and catalysis

Metals analyses of spent GACs[3-5] from American Water Works Service Company (AWWSC) and Compagnie Générale des Eaux (CGE) water treatment plants showed significant metal buildup during use (see representative data in Figure 1; not all data shown herein). In spent GACs, calcium contents were the highest (0.4–7 percent), followed by aluminum (0.3–3.7 percent), iron (nonpyritic—0.1–0.5 percent), manganese (0.02–0.6 percent), and magnesium (0.06–0.3 percent). Calcium, iron, manganese, and magnesium are not known to be harmful to human health.

These metals appear naturally in raw waters. Calcium, iron, and aluminum may be added during chemical treatment. The metals can become loaded onto GAC via chemical precipitation, chelation with natural organic matter, or ion exchange onto the GAC surface. Buildup could be particularly significant when GAC replaces anthracite filtration in a GAC filter–adsorber.[6]

Of these metals, calcium is the most catalytic[3,4,7,8] (Figure 1). Moreover, iron catalyzes gasification dramatically when sulfur is absent, but it has only a marginal effect when sulfur is present.[3,4,9] Sulfur always appears in GAC, either from the pyrite that commonly occurs in coal or from organically bound sulfur. Coal-based GACs contain 0.5–0.8 percent sulfur. Screening experiments also showed that aluminum, manganese, and magnesium are not catalytic.[3,4] Other metals exhibit low concentrations (<0.03 percent), most notably potassium, sodium, nickel, strontium, and barium. At higher concentrations, these five metals measurably catalyze carbon gasification.[10]

Literature review

The following review surveys how uncatalyzed and catalyzed oxidation have influenced pore development and adsorption behavior. It also addresses how pore structure potentially influences adsorption behavior by comparing pore sizes with molecular dimensions.

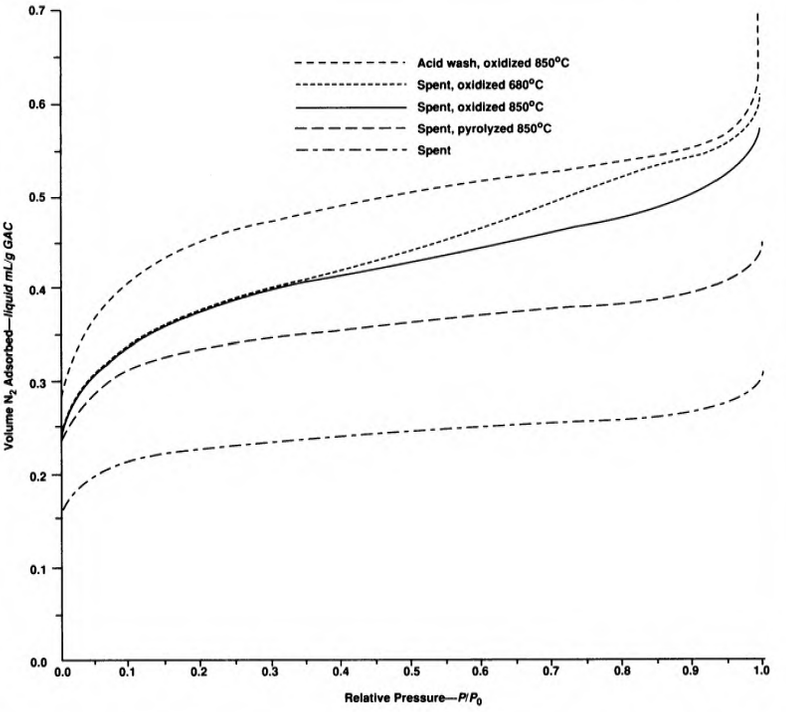


Figure 2. Nitrogen adsorption isotherms for spent Hays Mine GAC treated as shown *(oxidation in moderate flows of steam and CO_2 to an apparent density of 0.43 g/mL)*

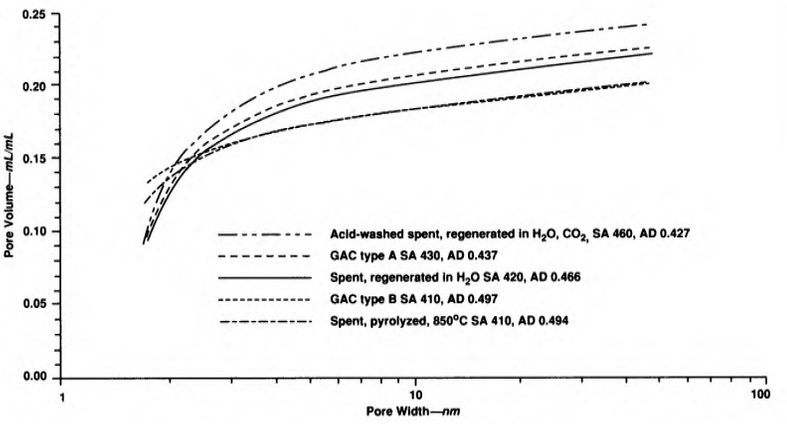


Figure 3. Cumulative pore volume distribution and surface area (m^2/mL) for spent Hays Mine GAC pyrolyzed and regenerated at 850°C to the apparent density shown *(compared with virgin GAC types A and B)*

Size comparisons of GAC pores versus organic compounds. Pore sizes have been categorized into three ranges:[11] micropores that are less than 2 nm in width, mesopores that are 2–50 nm in width, and macropores that are larger than 50 nm in width. In comparison, dissolved organic molecules that predominate in natural freshwaters are mostly within the 0.5–5-nm-diameter range. Single-ring aromatic compounds have dimensions in the range of 0.5–0.6 nm,[12] and 1,000-Dalton organic molecules span 1.3 nm (Table 1).

Several researchers have proposed correlations between adsorbate molecular size and adsorbent pore size, including Kasaoka et al,[19] who observed that adsorption dramatically increased when the carbon's mean pore size exceeded the molecule's second widest dimension by 1.5–2 times. To test this, adsorption was monitored onto an array of carbons that had progressively wider pore sizes. The pores were widened by subjecting a microporous parent carbon to progressively greater oxidation. Methylene blue's adsorption capacity, for example,

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TABLE 1
Molecular and pore size comparisons

Size—*nm*	Parameter	Reference
0.13–0.15	Bond length between carbon atoms	13
0.2	Calcium ionic diameter*	13
0.4 × 0.4	Surface area for condensed N_2	14
0.3–0.4	Carbon graphitic interlayer spacing	15,16
0.5–0.6	*p*-nitrophenol width	17
0.5	I_2 diameter	13
0.6	Diameter of 100-Dalton molecule	12
1–2	Estimated diameter, aquatic fulvic and humic compounds, MW of 800–30,000 Daltons	12,18
1.3	Diameter of 1,000-Dalton molecule	12
1.5–1.9	Estimated critical pore diameter for methylene blue adsorption	17,19
<2	Micropore width	19
2.9	Diameter of 10,000-Dalton molecule	12
6.2	Diameter of 100,000-Dalton molecule	12
2–50	Mesopore width	11
>50	Macropore width	11
450	Blue light wavelength	—

*Hydrated calcium is several times wider.

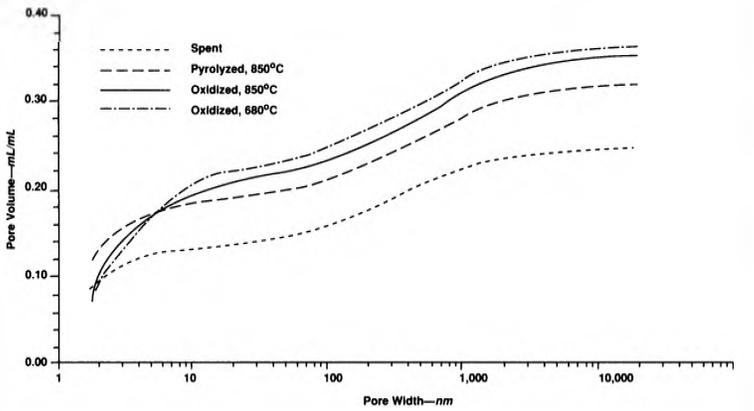


Figure 4. Cumulative pore volume distribution for spent Hays Mine GAC that was pyrolyzed and oxidized in steam and CO_2 to an apparent density of 0.43 g/mL

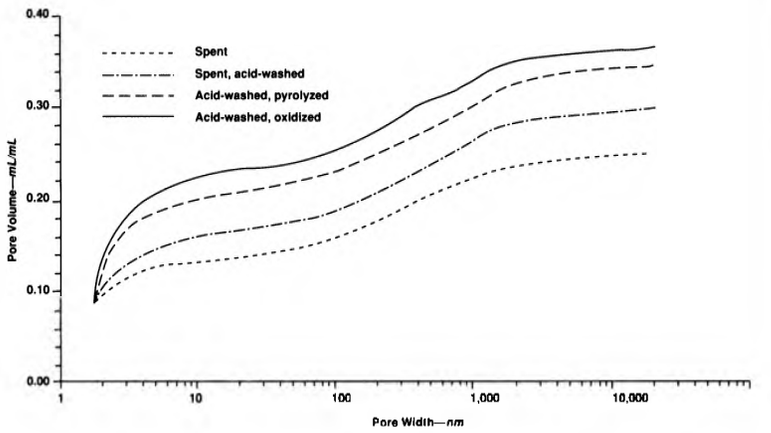


Figure 5. Cumulative pore volume distribution for acid-washed spent Hays Mine GAC that was pyrolyzed and oxidized at 850°C in steam and CO_2 to an apparent density of 0.43 g/mL

increased abruptly when the carbon's mean pore size surpassed 1.5 nm.

Although further research regarding size correlations is warranted, the literature suggests that the low-molecular-weight organic compounds that appear in water supplies would be small enough to fit into medium-sized GAC micropores but not into the narrowest micropores. Low-molecular-weight compounds might include small carboxylic acids, phenols, amino acids, small hydrocarbons, and a number of the regulated synthetic and halogenated organic compounds. Indeed, Hölzel and Sontheimer[20] and Sontheimer et al[21] propose that a large number of small organic compounds adsorb into pores smaller than 2 nm in width. In comparison, moderate-sized waterborne compounds such as cellulose and carbohydrates have dimensions that would allow them to fit only into the larger micropores or mesopores.

While evaluating several raw water samples from Southern California, Amy et al[22] observed the following apparent molecular sizes for dissolved organic carbon (DOC) composition: 5–30 percent of the DOC was contained among >30,000-Dalton molecules, 10–30 percent was among 10,000- to 30,000-Dalton molecules, 20–35 percent was among 1,000- to 10,000-Dalton molecules, and 20–65 percent was among <1,000-Dalton molecules. According to Perry and Chilton,[12] these would have corresponding diameter ranges of >4.5 nm, 2.9–4.5 nm, 1.3–2.9 nm, and <1.3 nm, respectively. MacCarthy and Suffet[18] and Thurman[23] have also characterized the typical molecular weights of river water DOC.

If these organic compounds could only adsorb into pores that were 1.5–2 times greater than their molecular dimension, then only the 20–65 percent of the organic carbon that occurred within <1,000-Dalton molecules could potentially fit within micropores. The remainder would potentially fit into nothing smaller than mesopores. Most of the specific synthetic organic compounds targeted for removal are smaller than 1,000 Daltons.

Pore structure and adsorptive capacity following uncatalyzed oxidation. With catalysts absent, the pore structure and adsorptive capacity of spent GAC have been adequately restored by thermal regeneration, as reported by several researchers. Waer et al[24] and Waer[25] tested GACs that were regenerated without catalysts being present and found the GACs could be restored to their virgin surface area, micropore volume, iodine capacity, *p*-nitrophenol capacity, and methylene blue capacity. Best adsorptive properties and pore structure occurred when apparent density (AD) of the regenerated GAC was equal to or slightly lower than that of the virgin GAC.

Jüntgen[26] regained virgin phenol capacity in repetitive (uncatalyzed) regenerations of GAC. Chiharaet al[27] restored sucrose adsorption capacity after one (uncatalyzed) regeneration, but capacity deteriorated in subsequent regenerations.

Uncatalyzed oxidation has also effectively developed superior activated carbons from lignocellulosic chars without destroying the carbon micropores. This was found in experiments by Cannon and Roberts[28] and Cannon[29] that employed a prune-pit char. They attained the highest pore volumes and adsorption capacities when the char was oxidized the longest

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in CO_2. As oxidation mass loss increased from 37 to 67 percent, the micropore volume increased from 0.27 to 0.47 mL/g, the mesopore volume increased from 0.14 to 0.35 mL/g, the N_2-BET surface area increased from 930 to 1,700 m^2/g, and the adsorption capacity for filtered wastewater secondary effluent DOC increased from 13 to 63 mg DOC/g powdered activated carbon. Although no metals analysis was conducted on this char, fruit pits are generally known to contain very little ash.[30] When comparing these prune-pit carbons to GAC type A,* it was observed that DOC adsorption capacity correlated better to the lower range of mesopore volume than to micropore volume. Other parameters may have also influenced adsorption capacity.

Calcium effect on micropore structure and adsorptive properties. Calcium accelerates the gasification rate and thus allows a desired mass loss to be achieved in less time or at lower temperature than when calcium is not present. But uncontrolled calcium catalysis can adversely affect pore structure and surface area.

Sebastiani[31] regenerated spent GAC from a GAC contactor that served the Upper Occoquan Sewage Authority in Virginia. This contactor followed activated sludge treatment and multimedia filtration. The coal-based spent GAC contained 0.85 percent calcium, compared with 0.2 percent in the corresponding virgin GAC, and had an AD of 0.51 g/mL. This compared with an AD of 0.43 for a virgin GAC type A (12- × 16-mesh fraction, 1.7 mm × 1.18 mm). Sebastiani compared the regeneration products of spent GAC with acid-washed spent GAC and found that when both had been regenerated to an AD of 0.43, they exhibited identical DOC isotherms for wastewater secondary effluent, although their pore size distributions were presumably different. Moreover, the DOC isotherms for these two GACs were statistically undistinguishable from isotherms that employed virgin GAC or ones that employed spent GAC that had been regenerated to ADs of 0.47 or 0.41. These all had different pore size distributions.

Juhola[32] measured the adsorption properties of regenerated spent GAC from an advanced wastewater treatment plant. The spent GAC had accumulated roughly 1.5–2.5 percent ash above virgin levels. Regenerations to virgin AD at 930°C resulted in 10–15 percent lower iodine adsorption numbers than in virgin GAC type A and 0–20 percent higher molasses numbers. Regenerations with steam alone generally caused higher iodine numbers than with CO_2 alone. In comparison, the regeneration product of acid-washed GAC exhibited iodine adsorption capacity 4–10 percent lower than virgin GAC and molasses numbers 0–13 percent lower. Iodine has been perceived to adsorb primarily in large micropores and molasses in small mesopores.

In Juhola's experiments, micropore volume appeared to be restored to virgin values for a spent GAC that was regenerated at 930°C in steam alone or for an acid-washed GAC regenerated in steam and CO_2. However, micropore volume was not restored for a spent GAC that was regenerated in CO_2 alone.[32] None of these three regeneration conditions restored the 2–5-nm pores or surface area to virgin values. These pore structure analyses were conducted using water adsorption isotherms (the modeling method was not reported). It is not known how these would compare with nitrogen adsorption isotherms. As the spent GAC experienced three field-loadings and regeneration cycles, iodine capacity decreased (0–10 percent each cycle), ash content increased (1 percent each cycle), and molasses numbers increased (0–20 percent each cycle).

Van Vliet and Venter[33] were able to restore only 70 percent of virgin micropore volume (on a unit bed volume basis) after a fourth regeneration cycle. The spent GAC came from a wastewater secondary effluent GAC contactor, and steam oxidation was applied in an infrared furnace. Optimum regeneration conditions were 8 min oxidation at 800°C. Data were not available on the pore size distribution following the third regeneration, just prior to the fourth loading and regeneration cycle. Although ash analyses were not provided, the pore structure behavior suggested catalytic influence.

Linares-Solano et al[34] found that uncatalyzed CO_2 gasification caused an increase in micropores but no increase in mesopores, whereas calcium-catalyzed CO_2 gasification caused a dramatic increase in mesopores and a slight decrease in micropores. These GAC samples were loaded with calcium acetate and then were oxidized in a thermogravimetric analyzer (TGA) at 900°C. Catalyzed oxidation also caused a considerable increase in "supermicropore" volumes, defined as the difference between the N_2 and CO_2 micropore volumes. Linares-Solano et al did not assign a pore size to these. In comparison, calcium-catalyzed steam oxidation (at 0.02-MPa pressure) created a pore distribution that was similar to uncatalyzed steam oxidation and exhibited a reactivity that equaled uncatalyzed reactivity. Linares-Solano et al attributed this unusually low catalytic activity to the low partial pressure of steam.

*Filtrasorb 400, Calgon Carbon Corp., Pittsburgh, Pa.

TABLE 2
Comparison of regeneration products for Hays Mine GAC

Parameter	Spent, Hays Mine	Spent, Pyrolyzed	Spent, Pyrolyzed, Mildly Oxidized	Acid-Washed, Pyrolyzed, Oxidized	Type A	Type B*
Regeneration conditions						
Temperature—*°C*		850	850	850		
Oxidation time—*min*	0	0	3	35		
Mass loss—*percent*	0	13.7	18.6	24.5		
Volume loss—*percent*	0	3.8	3.4	6.8		
Oxidant employed—*g H_2O/g GAC*	0	0	0.4	4.7†		
Product characterization						
Apparent density—*g/mL*	0.548	0.494	0.466	0.428	0.437	0.497
Ash-free apparent density—*g-C/mL*	0.492	0.440	0.414	0.406	0.410	0.473
Micropore volume—*mL/mL*	0.100	0.134	0.124	0.134	0.130	0.142
Mesopore volume—*mL/mL*	0.049	0.065	0.097	0.107	0.094	0.057
(Volumetric) surface area—*m^2/mL*	300	410	420	460	430	410
(Gravimetric) surface area—*m^2/g-GAC*	540	820	900	1,080	990	820
Ash content—*percent*	9.9	10.8	11.1	5.1	6.22	4.92

*This virgin GAC type B sample was not from the same batch that became spent at Hays Mine, Pa.
†Plus 6.5 g CO_2/g GAC

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Full-scale and pilot-scale regenerations at the Cincinnati Water Works have been reported by Miller and Hartman[35] and by De Marco et al.[36] These data indicated that after virgin GACs had become spent at this water treatment plant and then regenerated through one cycle, the regenerated product GACs exhibited 7–14 percent lower iodine numbers and BET surface areas than their virgin predecessors (on a volumetric basis of comparison). Following the second, third, and fourth regeneration cycles, there was no further decline in iodine numbers. Although calcium content was not specifically monitored during these tests, the data depicted considerable ash buildup.

Pore blockage by calcium and other inorganic metals has not been noticeable within activated carbons and chars. Linares-Solano et al[37] and Hippo and Walker[38] saw no change in pore structure and a slight increase in N_2-BET surface area when 12 percent calcium and an array of other metals were loaded by chelation or ion exchange into porous chars.

In summary, calcium-catalyzed oxidation has generally been found to slightly deteriorate GAC micropores, surface areas, and iodine adsorption capacity. This deterioration has been the least when the spent GACs were regenerated to their virgin AD. If the spent GACs were acid-washed before regeneration, they exhibited less micropore deterioration and in some cases gained micropore volume.

Recognizing this research, the authors have sought the most favorable conditions for regenerating a spent GAC that contained high levels of calcium and therefore have subjected a spent GAC that contained calcium to a wide array of oxidant gases, oxidant flow rates, oxidation temperatures, pyrolysis temperatures, and extents of oxidation.

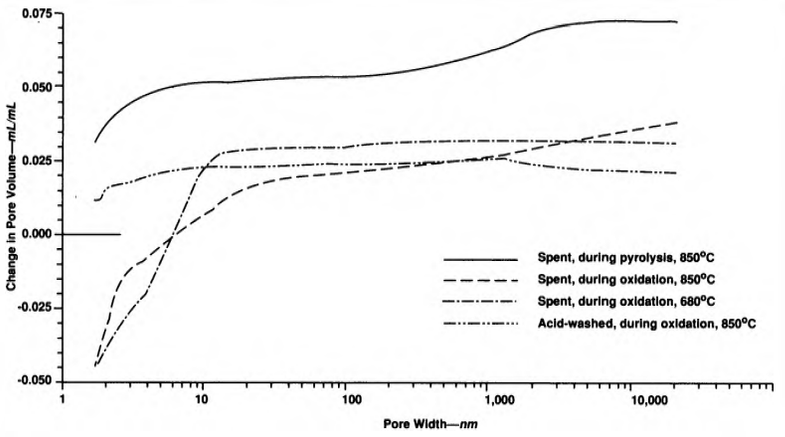


Figure 6. Change in cumulative pore volume distribution for spent Hays Mine GAC during pyrolysis and for pyrolyzed GACs during oxidation in steam and CO_2 to an apparent density of 0.43 g/mL

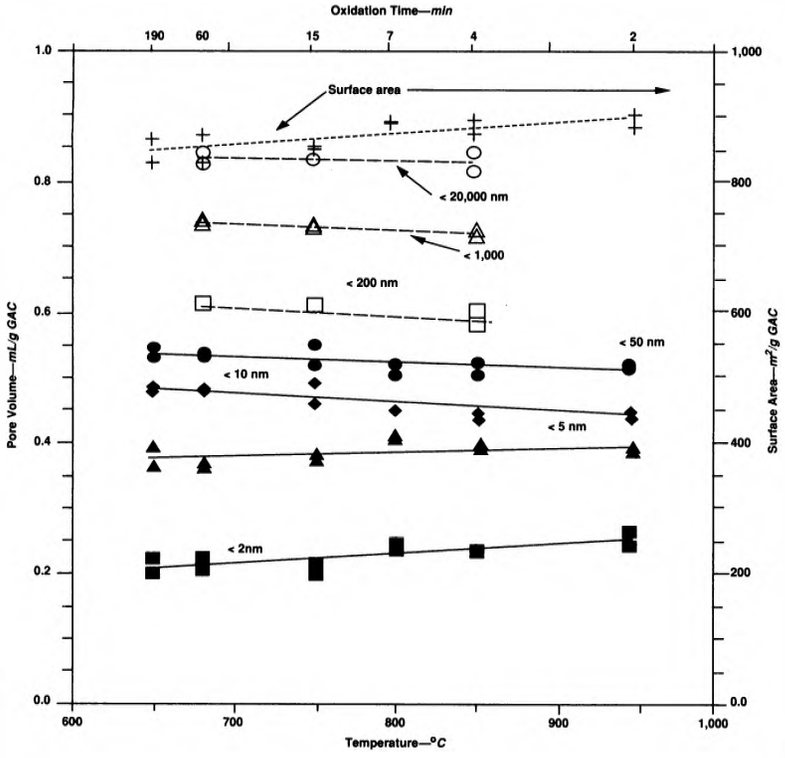


Figure 7. Pore volume distribution and surface area for spent Hays Mine GAC regenerated in steam and CO_2 at various temperatures to an apparent density of 0.43 g/mL

Materials and methods

Spent and acid-washed GACs were regenerated in a fluidized-bed furnace, and the regenerated products were compared with virgin GACs by employing nitrogen adsorption isotherms and mercury porosimetry. Spent adsorptive capacity was monitored for select samples by measuring iodine, *p*-nitrophenol, and methylene blue adsorption.

Spent and virgin GACs. Experiments were conducted with a spent GAC from the Hays Mine, Pa., water treatment plant (subsequently called "spent GAC") and with virgin GAC types B and A.* Both are bituminous-based carbons, and both GAC type A and type B are commonly used in water and wastewater treatment applications.

The Hays Mine plant serves Pittsburgh, Pa., and treats Monongahela River water. At this plant, GAC type B had been used in a filter adsorber for three years and eight months prior to sampling. The spent GAC contained 1.8 percent calcium and 9.9 percent ash and had an AD of 0.55 g/mL.

Acid-washing. Acid-washing was accomplished by passing 10 or more bed volumes of 1 *M* HCl up through a GAC filter bed followed by 10 or more bed volumes of deionized distilled water. The HCl washing extracted 98 percent of the GAC's calcium; 90 percent of its available aluminum, manganese, and magnesium; and 50 percent of its iron. The HCl did not appear to extract pyritic iron, sulfur, or aluminosilicates.[39]

Fluidized-bed regenerations. The fluidized-bed regeneration furnace was as described by Waer et al.[24] Standard ("moderate") steam–CO_2 fluidized-bed regenerations included 10 min of pyrolysis in nitrogen followed by oxidation in 8

*Type B—Filtrasorb 200 and type A—Filtrasorb 400, Calgon Carbon Corp., Pittsburgh, Pa.

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g H_2O/g GAC-h, 11 g CO_2/g GAC-h, and 30 g N_2/g GAC-h. In these regenerations, 10 g of 16- × 20-mesh (1.18- × 0.85-mm mesh) GAC was used, and the changes in mass, unstratified bed volume, and AD were monitored per ASTM D 2854-83.[40] This narrow mesh range was employed to minimize variations in oxidation rate that could have resulted from variations in grain size.

Thermogravimetric analysis. Thermogravimetric analyses were conducted with a unit* that continuously monitored mass loss of 10–20-mg GAC samples while they were being pyrolyzed in nitrogen or oxidized in 20 mL/min CO_2 plus N_2 at preprogrammed temperatures. Steam was not used because TGAs are limited to very low water flow rates.[34] Moreover, volume losses and changes in AD could not be monitored during TGA experiments.

Metals analysis. Metals analysis was conducted by ashing GAC in a 600°C muffle furnace for 12–16 h, dissolving the ash in 25 mL concentrated HCl for 4–8 h, filtering the solution through 0.45-μm filter paper, and then diluting it to 100 mL with deionized distilled water. This solution was analyzed for metals by employing an inductively coupled plasma (ICP) unit† in the University of Illinois Chemistry Department. Hydrochloric acid is expected to dissolve all pertinent metals except for aluminosilicates, pyrites (FeS_2), and some claybound iron.[39,40] All ashings and metals analyses were duplicated, and values from duplicate ashings agreed within ±10 percent.

Sulfur was analyzed using an automated total sulfur analyzer‡ at the University of Illinois Forestry Department[41] or at the Illinois State Geological Survey. Samples were duplicated, and values agreed within ±10 percent.

Apparent density, mass loss, and volume loss. The AD of GAC was obtained by dividing the mass of a GAC sample by the volume that the sample occupies when randomly stratified. For this research project, AD measurements were performed according to ASTM procedure D 2854-83[40] with the modification that 5- to 10-g samples were vibrated into a 25-mL graduated cylinder.

The mass loss resulting from the thermal regeneration process was determined for a batch-type process by weighing the carbon on an analytical balance before and after the regeneration. Mass loss has been expressed as a percentage of unregenerated GAC mass. For samples regenerated in duplicate, the average difference in mass loss between two samples was 0.8 percent (for example, replicate regenerations could have exhibited 25.0 and 25.8 percent mass loss).

The volume of a randomly stratified GAC sample was measured by vibrating a GAC sample into a graduated cylinder as described in the AD procedure. By measuring the volume that GAC samples were occupying before and after thermal regeneration, the percentage of volume loss could be calculated and expressed as a percentage of the spent GAC volume.

For samples regenerated in duplicate, the average difference in volume loss between two samples was 0.7 percent (for example, average replicate regenerations could have exhibited 4.0 and 4.7 percent volume loss).

The volume loss occurring during regeneration is an important parameter because it determines the amount of virgin GAC that needs to be purchased to refill the GAC bed to its design volume.

Iodine number, p-nitrophenol, and methylene blue capacity. The iodine number has been defined as the mass of iodine (in milligrams) adsorbed by 1 g of carbon when the residual iodine concentration is 0.02 N. At the University of Illinois, the authors followed ASTM method D4607-86[40] for measuring the iodine number. However, both the initial concentration of the iodine solution and the amount of carbon were reduced by half to conserve carbon needed for subsequent analyses. *p*-nitrophenol and methylene blue adsorption capacities were determined from isothermal measurements.[25]

Nitrogen adsorption at 77 K. Nitrogen adsorption isotherms were obtained at 77.15 K, the boiling point of liquid N_2 at 1 atm (0.1 MPa) of pressure. These isotherms provided the information required to calculate the BET surface area as well as the micropore and mesopore volume of GAC samples. N_2 adsorption

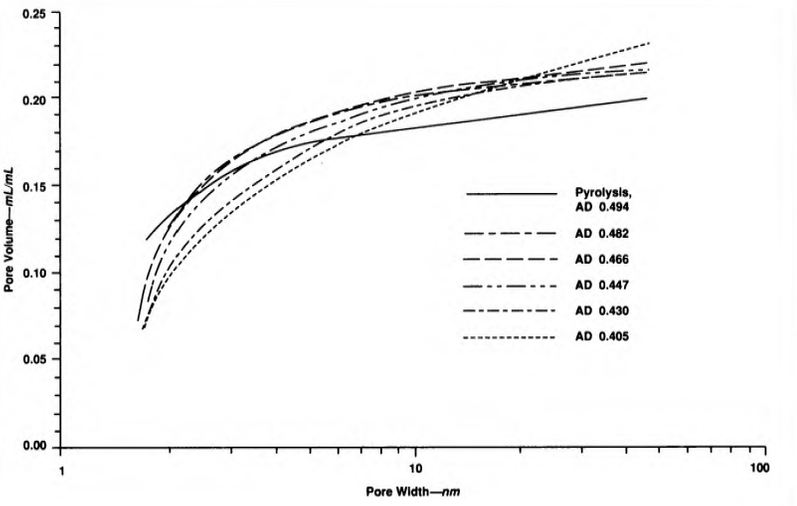


Figure 8. Pore volume distribution for spent Hays Mine GAC regenerated at 850°C to various apparent densities in steam

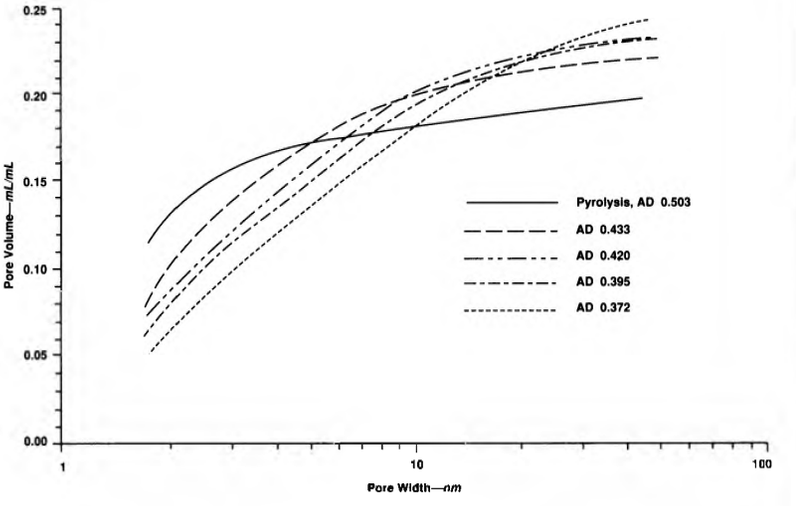


Figure 9. Pore volume distribution for spent Hays Mine GAC regenerated at 750°C to various apparent densities in steam and CO_2

*PC Series TGA7, Perkin-Elmer, Norwalk, Conn.
†Plasma P 2000, Perkin-Elmer, Norwalk, Conn.
‡SC-132, LECO, St. Joseph, Mich.

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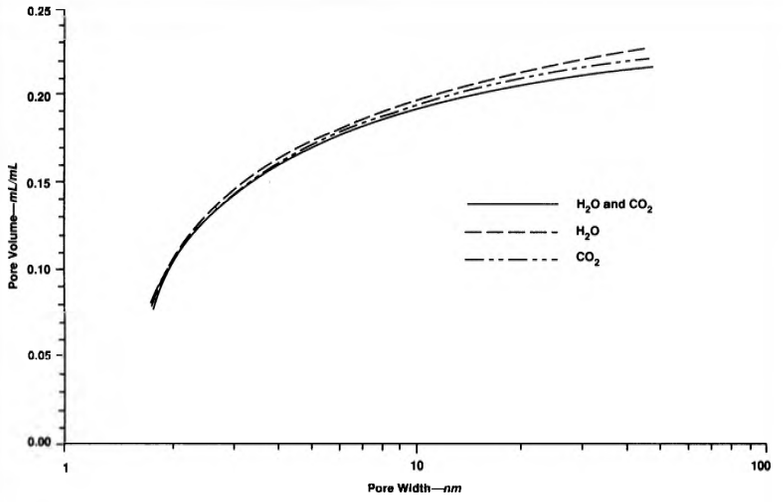


Figure 10. Pore volume distribution for spent Hays Mine GAC regenerated to an apparent density of 0.43 at 850°C in steam, CO_2, and steam plus CO_2

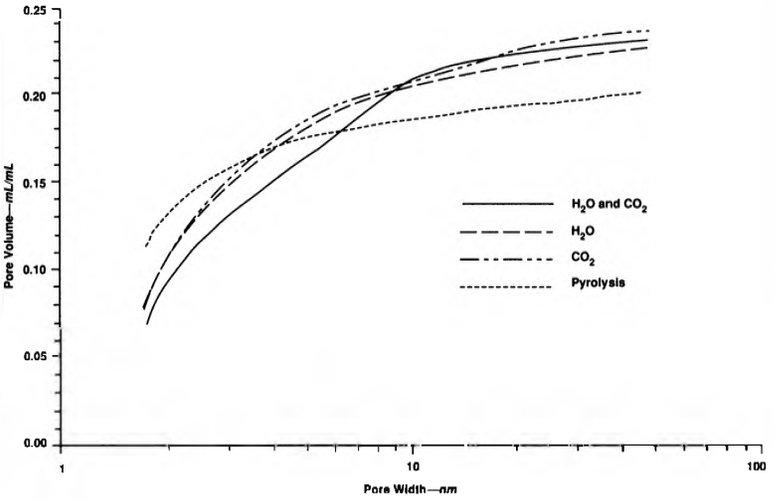


Figure 11. Pore volume distribution for spent Hays Mine GAC regenerated to an apparent density of 0.43 g/mL at 680°C in steam, CO_2, and steam plus CO_2

experiments were conducted on a nitrogen surface analyzer* at the Center for Cement Composite Materials at the University of Illinois. This instrument measured the nitrogen volumes adsorbed versus relative pressures, in which relative pressure was the equilibrium pressure (P) divided by the saturation pressure of N_2 (P_0) at 77 K.

For each GAC sample, the nitrogen surface analyzer automatically monitored roughly 45 adsorption equilibrium points, which spanned relative pressures from 0.005 to 1.00. For each of these data points, a known volume of gaseous nitrogen was pulsed into a glass bulb that contained 0.13–0.17 g of sample. This glass bulb was immersed in liquid nitrogen. As the gaseous nitrogen within the bulb condensed and adsorbed into the GAC sample (at a density approximately equal to that of liquid nitrogen), the pressure within the bulb dropped to an equilibrium value. This pressure was automatically recorded, and then another pulse of gaseous nitrogen was introduced to repeat the adsorption cycle.

The authors have included representative isotherm examples in Figure 2, which represents experiments that are considered further in the "Results and discussion" section. Several researchers[14,24,25,42,43] have proposed models that interpret these isotherms to derive the GAC surface area and micropore and mesopore volumes. In most cases, N_2 isotherms were obtained for duplicate regenerations. Typical differences in pore volumes and surface areas were within 5 percent for duplicate regenerations.

Surface area. Total surface area was computed using the model of Brunauer et al,[14] by employing the portion of the nitrogen data that spanned relative pressures from 0.005 to 0.124. The Barrett et al model (BJH model)[42] provided mesopore surface areas, and mercury porosimetry provided macropore surface areas.†

Mesopore volume. The mesopore volume range contains pores with diameters spanning from 2 to 50 nm as defined by the International Union of Pure and Applied Chemistry (IUPAC).[11] The total mesopore volume and the mesopore volume distribution were calculated from the data of the adsorption leg of the N_2 isotherm via the BJH model.[42]

Barrett et al[42] presumed that nitrogen adsorbed by two simplified mechanisms: (1) capillary condensation within small pores and (2) layered physical adsorption onto larger pore walls. Relative pressure dictated the pore size where condensation and layered adsorption occurred, and trigonometric functions distinguished between them. Barrett et al[42] built their model on the Kelvin equation, which identified the relative pressure that caused nitrogen condensation within the "inner capillary volume" of a pore. The "inner capillary volume" was defined as the vacant volume that was not occupied by either carbon or layered nitrogen at the preceding relative pressure. Thus, the inner capillary width equaled the pore width minus twice the layered nitrogen thickness.

Barrett et al championed their model for situations in which pores were "cylindrical, or more precisely, when the pore volume and the capillary volume were related to each other as the square of some adequate measure of their cross sections."[42] In this article the authors propose that the wedge-shaped pores found in GAC conform to this criterion. The cross-section dimensions computed by this method may not, however, represent true pore widths, and they therefore offer relative rather than quantitative comparisons. Thus, throughout this article, the term "2-nm pores" refers to pores having a computed cross-section dimension of 2 nm.

The equation used for determining nitrogen layer thickness, t_N, in the BJH model was provided by Waer:[25]

$$t_N = 24.6164/(0.034 - \log [P/P_0])^{0.429} \quad (1)$$

For duplicate analyses of duplicated regenerations, the difference in mesopore volumes was usually found to be within 5 percent.

Micropore volume. The recovery of GAC micropore volume within a regenerated product is especially important for restoring the adsorption capacity of low-molecular-weight organic compounds.

*2400 ASAP, Micromeritics, Norcross, Ga.
†Autopore II Mercury Porosimeter, Micromeritics, Norcross, Ga.

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Micropores are defined by IUPAC as pores having diameters <2 nm.[11] To determine micropore volume, a procedure was developed that expanded the scope of the BJH model.[42] As developed during the research, the authors propose that at a relative pressure of 0.12, all pores smaller than 2 nm are filled with nitrogen by capillary condensation,[44,45] and all surfaces in pores larger than 2 nm have adsorbed a 0.4-nm-thick layer of nitrogen.[24,25] The micropore volume could then be computed by the procedure:

1. Convert gaseous volume of N_2 adsorbed to equivalent liquid N_2 volume (V_{total}) at a relative pressure of 0.12 (cm^3 at STP/g GAC). The density of liquid N_2 at 77.15 K is 0.808 g/cm^3.[13]

2. Derive the adsorbed layer volume (V_{layer}) in pores larger than 2 nm at a relative pressure of 0.12 by multiplying the mesopore plus macropore surface areas by the thickness of the adsorbed liquid N_2 layer (0.4 nm). The mesopore surface area was determined from the nitrogen isotherm data by use of the BJH model; the macropore surface area was determined by mercury porosimetry.

3. Compute the micropore volume (V_{micro}) by taking the difference between the total and layered volumes (Eq 2):

$$V_{micro} = V_{total} - V_{layer} \quad (2)$$

For the coal-based virgin and regenerated GACs, V_{micro} generally amounted to two thirds of V_{total}, and V_{layer} amounted to roughly one third of V_{total}. For analyses of duplicated regenerations, the difference in micropore volumes was usually within 5 percent.

Total pore volume under 50 nm. Similarly, a relative pressure of 0.96 filled all pores smaller than 50 nm per the Kelvin equation[43] and blanketed macropore surfaces with a 1.4-nm-thick adsorption layer per Waer et al[24] and Waer.[25] Thus their difference equaled the <50-nm volume. These computations were cross-checked by comparing micropore volumes (at $P/P_0 = 0.12$), mesopore volumes (by the BJH model), and volume below 50 nm (at $P/P_0 = 0.96$). The last value consistently exceeded the sum of the first two values by only 0–7 percent. This good agreement implied model accuracy or consistently compensating inaccuracies. One cannot independently distinguish between these possibilities. Nonetheless, this modified BJH model has been considered useful when making comparisons among GAC samples.

Several researchers[7,24,25,43] have employed the thickness–volume (t–V) method when computing micropore volumes. The t–V method has relied on the same assumptions that have been used in this research. However, after several trials, the authors abandoned the t–V method because (1) the authors could not interpret all data with it and (2) it apparently overestimated micropore volumes. When t–V micropore volumes that were computed from data were added to corresponding BJH mesopore volumes, they exceeded the under-50-nm volume by 10 to 25 percent.

An example of one of the curves that could not be interpreted by the t–V method is labeled in Figure 2 as the "Spent, oxidized 680°C" curve. No single micropore volume could be computed for this because it was described by two t–V slopes between relative pressures of 0.2 and 0.93.

Representative nitrogen adsorption isotherms. Several representative nitrogen isotherms have been shown on a unit mass basis in Figure 2 as they appeared before they were mathematically manipulated. The BJH model results (see subsequent discussion) depicted the same trends as these isotherms, as expected.

Mercury porosimetry. IUPAC defines macropores as pores with diameters >50 nm.[11] Pore volume distributions in the macropore range up to pore diameters of 20 μm (20,000 nm) were obtained using mercury intrusion porosimetry (MIP)* at the Center for Cement Composite Materials at the University of Illinois. In MIP, mercury is forced into GAC pores at pressures up to 60,000 psi. Increasing pressures correspond to decreasing pore diameters so that a pore volume distribution is obtained. The cumulative intrusion volume was plotted against the corresponding pore diameters.

For duplicated regenerations, mercury porosimetry agreement was within ±6 percent.

Comparisons per unit bed volume. Throughout this article, the authors have reported GAC data per unit of unstratified bed volume. The volume is that occupied by the GAC including the volume of voids between grains (ASTM D2584-83).[40] When values were measured on a mass basis, they were converted to a unit bed volume basis by multiplying the values by their corresponding ADs.

The unit bed volume basis was preferred over the unit mass basis for two reasons: (1) the capacity per unit volume is more directly related to the capacity of a fixed-bed adsorber and (2) comparisons based on a unit bed volume more accurately depict how pore development progresses. This latter reason was true for three reasons. First, the volume of GAC grains changed very little during regeneration, whereas a considerable fraction of the solid mass was volatilized or gasified. For example, when coal-based regenerated samples experienced 25–29 percent mass loss, they correspondingly lost only 3–9 percent of their volume. Second, most of the mass loss was internal, and third, some of the bed volume loss appeared to be caused by abrasion, which probably did not change pore size distribution per unit volume.

Results and discussion

Objectives. Specific objectives of the experiments described were to (1) restore a field-spent GAC to virgin pore structure and adsorptive properties while the calcium remained within the GAC, (2) understand how calcium in a field-spent GAC affected pore development during regeneration, (3) determine whether pretreatment with acid substantially improved the regeneration product, and (4) determine how an array of different regeneration conditions affected field-spent GAC pore structure.

Regeneration to restore virgin properties.

Spent GAC. Virgin pore structure could be virtually restored by appropriate thermal regeneration of the spent Hays Mine GAC while its 1.8 percent calcium remained intact. When the spent GAC was merely pyrolyzed at 850°C, its pore structure became nearly identical to that of virgin GAC type B, with 13 percent more mesopores, 6 percent fewer micropores, and the same surface area (Figure 3 and Table 2). Pyrolysis caused 14–16 percent

*Autopore II Mercury Porosimeter, Micromeritics, Norcross, Ga.

TABLE 3

Adsorption capacity of spent Hays Mine GAC regenerated at various temperatures to an apparent density of 0.43–0.44*

Regeneration Temperature °C	Iodine Number mg I_2/g C	Methylene Blue Capacity† mg MB/g C	p-Nitrophenol Capacity‡ mg PNP/gC	Ash Content percent
650	980	198	140	11.5
680	940	201	160	11.5
750	960	207	160	11.5
850	1,010	214	165	11.5
910	930	201	160	11.5
Pyrolyzed, 900°C§	960	200	175	10.8
Acid-washed, oxidized, 850°C	1,060	218	160	4.4
Virgin type A			195**	6.2

*Adsorption data listed on the unit basis of ash-free carbon mass
†For initial concentrations of 1.2 g/L methylene blue and 4.0 g/L GAC, 30-min adsorption
‡For equilibrium PNP concentration of 3 mg/L
§Apparent density following pyrolysis was 0.48 g/mL.
**Data provided by C. Feizoulof, University of Illinois

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mass loss and 3–4 percent bed volume loss. This same trend in pore structure was also observed at other pyrolysis temperatures, as discussed later in this article. The authors did not test whether these pore structures could be sustained through several regeneration cycles. (The virgin GAC type B tested in the laboratory was from a different batch than the GAC type B used at Hays Mine.) The curves shown in Figure 3 and in all subsequent figures represent cumulative pore volume distributions.

In comparison, the regenerated GAC exhibited a pore structure most like GAC type A when it had received 10 min of pyrolysis at 850°C and 2–3 min of oxidation with moderate steam flows at 850°C. These products had surface areas, micropore volumes, and mesopore volumes that were within 2–5 percent of values for GAC type A (Figure 3 and Table 2). Mild oxidation to an AD of 0.466–0.484 g/mL achieved these results and employed 0.27–0.40 g H_2O/g GAC. Corresponding mass loss was 14.5–18.5 percent, and volume loss was 3–4 percent. At 0.466, the AD of this regenerated GAC was 7 percent higher than GAC type A's AD, but the ash-free apparent densities were the same at 0.41 g ash-free carbon/mL. Although GAC type A is not the same GAC as was used initially, it has been considered a high-grade, popular GAC among water utilities.

Thus, within the range of statistical significance, the pore structures of two popular virgin GACs could be attained by employing controlled regeneration of a spent GAC, even though 1.8 percent calcium remained within this spent GAC.

Comparison with acid-washed GAC. Spent GAC was washed in 10 bed volumes of 1 *M* HCl (subsequently called "acid-washed GAC") and then pyrolyzed and oxidized in moderate flows of steam and CO_2 at 850°C for 35 min. This GAC product had 7–9 percent higher surface area, micropore volume, and mesopore volume than the regenerated product (with AD 0.47) that had not been acid-washed (Figure 3 and Table 2). The AD of the acid-washed product, at 0.43 g GAC/mL, was lower than the 0.47 AD for the mildly oxidized spent product previously discussed. But on an ash-free basis, the two products had the same AD of 0.41 g ash-free carbon/mL.

Attaining this level of oxidation with acid-washed GAC required 4.7 g H_2O/g GAC, 6.5 g CO_2/g GAC, and 35 min of oxidation time. These values were an order of magnitude greater than those necessary when the spent GAC was loaded with calcium. This difference highlighted how calcium dramatically improved oxidation efficiency, a factor that could offer advantages during full-scale, carefully controlled regenerations.

When all issues are weighed for the spent Hays Mine GAC, the slight improvements in pore structure gained by acid-washing would not justify the disadvantages incurred by using acid. Disadvantages include longer regeneration times, higher steam or CO_2 consumption, higher capital expense, stricter safety precautions, and acid waste streams that must be disposed of. Indeed, the calcium catalyst would allow a higher GAC throughput through a regeneration furnace, which probably would offset the potential need for more frequent regeneration cycles. This overall recommendation regarding acid-washing would also apply to another coal-based GAC with 0.9 percent calcium that has been studied at the University of Illinois,[31] but it might not apply to several wood-based GACs that contained 6 percent and 3 percent calcium studied at the University of Illinois.[3,4,8]

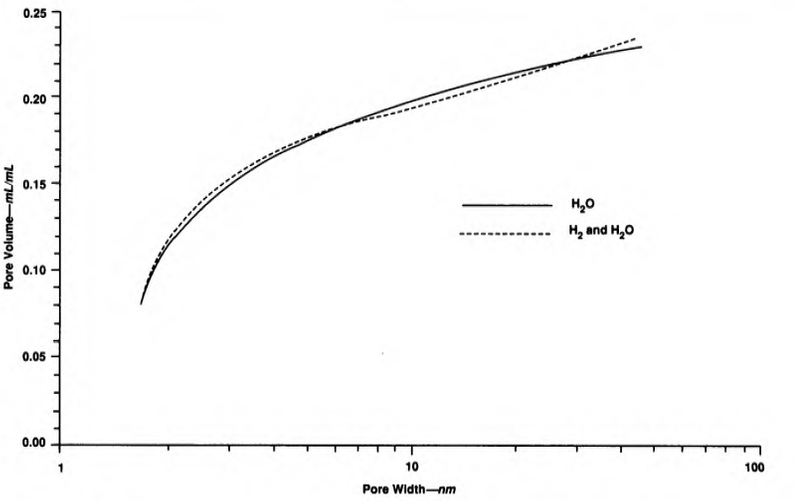


Figure 12. Pore volume distribution for spent Hays Mine GAC regenerated to an apparent density of 0.43 at 850°C in steam and H_2 plus H_2O

Pore development progression. Spent GAC and acid-washed GAC had similar pore size distributions following pyrolysis, but the pore size distributions differed dramatically following oxidation. For both GACs, pyrolysis increased the micropores, small mesopores, and 600–2,000-nm macropore volumes. During oxidation, calcium-loaded spent GAC always lost some micropore volume, whereas acid-washed GAC always gained micropores. These trends are observed in Figures 4–6, which show BJH nitrogen adsorption data for pores <50 nm and mercury porosimetry data for pores >50 nm. Figure 6 shows the change in cumulative pore volume that occurred during a single regeneration step. For example, the curve appearing in Figure 6 labeled "Spent, during oxidation, 850°C" represents the difference between the curves labeled "Oxidized, 850°C" and "Pyrolyzed, 850°C" in Figure 4.

An AD of 0.43 was targeted in these (and other subsequently discussed) experiments. This AD was intentionally slightly low so trends could be exaggerated and accurately quantified. This AD was achieved following 25–29 percent regeneration mass loss and 4–9 percent bed volume loss.

Pyrolysis. During pyrolysis, most porosity developed in pores <10 nm wide (Figures 4–6). This presumably represented the volatilization of organic matter from within the small pores and the release of oxygen functional groups from the carbon surface. Pores also developed in the range 600–2,000 nm. The 600–2,000-nm pore development had consistent magnitude whether spent GAC was pyrolyzed at 850°C or 680°C or whether acid-washed GAC was pyrolyzed at 850°C. (The latter two cases are not shown.) Development of pores in this range could have biological significance, because the narrow dimension of bacteria can be as small as 1,000–2,000 nm (1–2 μm).

Spent GAC oxidation. Oxidation always caused spent GAC to lose micropore volume and gain mesopore volume, but the temperature and oxidant dictated where mesopores developed. When CO_2 and steam were combined, 850°C oxidation caused pore widening throughout all pore ranges from 2 to 20,000 nm, but 680°C oxidation with both gases caused pore volume to increase solely between 6 and 10 nm. This implied that steam and CO_2 together attacked different carbon sites at low temperatures than they did at high temperatures.

At both temperatures, volumes declined in pores <5 nm, but the decline was greater at the lower temperature. The <5-nm pore size range is believed to be

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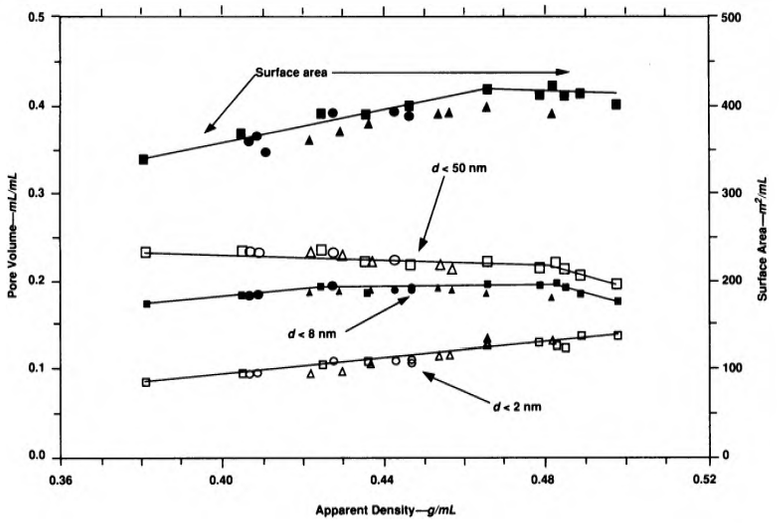


Figure 13. Pore volume distribution and surface area for spent Hays Mine GAC regenerated in steam at 850°C to various apparent densities *(triangles—low flow, prepyrolyzed at 950°C, squares—moderate flow, circles—high flow)*

important, and for most water treatment applications the pore volume distribution at 680°C would probably be inferior.

The effect of widening micropores implied that calcium catalyst either appeared in the micropores or facilitated oxidant transfer into the micropores along the carbon surface. If dissolved calcium had been conveyed into the GAC in association with a small anion during water treatment service, the calcium presumably would have fit into micropores, even in hydrated form (Table 1).

Acid-washed GAC oxidation. Oxidation of acid-washed GAC increased its micropore volumes but did not affect its mesopores or macropores (Figures 5 and 6). Only the pyrolysis step developed 2–10-nm mesopores for this acid-washed GAC. After the GAC was acid-washed and pyrolyzed, it took 35 min of oxidation to attain a 0.43 AD, compared with 4 min if it was not acid-washed, when both oxidations employed moderate steam and CO_2 flows. The ninefold rate increase implies that when the catalyst was present, it induced nearly all the oxidation that occurred. Thus, the spent GAC pore size that was widened probably correlated to the pore size that hosted the calcium. The larger micropore volume for acid-washed GAC suggested that steam and CO_2 could penetrate micropores more deeply than calcium could.

Potential blockage of pores by metals. The metals present in ash itself did not significantly impair pore structure by themselves. To test this, several spent Hays Mine GACs were acid-washed after they had been regenerated. The GACs' surface areas, micropore volumes, and mesopore volumes remained unchanged by the acid-washing when compared on an ash-free mass basis.

However, the metals that accumulate within GAC during water treatment service could potentially block pores by other possible association mechanisms. When humic compounds and metals become chelated to one another, they can form flocs[46] that would be large enough to mask the GAC's macropores. If this phenomenon occurred, it might prevent smaller organic compounds from subsequently penetrating through this mask into the GAC's smaller internal pores. Similarly, the hydrated water molecules associated with metals might span wide enough dimensions to clog small pores.

Because the nitrogen adsorption preparatory procedure includes a dehydration step, this method would not detect such hydrated water pore constriction. Perhaps subsequent research will provide perspective on these possibilities.

Volume and mass balance. Volume and mass balances were conducted for the oxidations shown in Figure 6 by using the equation

$$\Delta U_{OX} = \Delta PV_{OX} \times SD_N + \Delta BV_{OX} \times AD_{ur} \quad (3)$$

in which ΔU_{OX}—mass loss during oxidation per unit volume (g/mL unregenerated GAC), ΔPV_{OX}—change in internal pore volume during oxidation (mL/mL), SD_N—nitrogen skeletal density (g/mL), ΔBV_{OX}—bed volume loss during oxidation (mL/mL unregenerated GAC), and AD_{ur}—AD of unregenerated GAC (g/mL).

Balancing units were grams GAC lost/millilitres unregenerated bed volume. The nitrogen skeletal density was the mass of GAC/volume of carbon solid plus voids that nitrogen could not enter. This was calculated as 2.0 g/mL and was derived from nitrogen adsorption and mercury porosimetry experiments. Eq 3 balanced within 6–10 percent for all three of these oxidations. This was believed to be good, considering the experimental errors inherent for each parameter in this equation.

It was determined that 70–85 percent of the mass loss occurred internally and 15–30 percent occurred externally (i.e., outside 20,000-nm-diameter pores). The spent GAC that was oxidized at 850°C lost the least mass externally, and the acid-washed GAC lost the most externally. When regeneration was prolonged, a higher portion of the external mass loss could be attributed to the agitation-induced attrition of micron-sized aggregates, and a lower proportion could be attributed to burnout by the oxidizing gases. For the acid-washed GAC, nearly all of the external losses appeared to be caused by attrition and none by oxidizing gases.

Temperature effect on combined steam and CO_2 oxidation. ***Effect on pore volumes.*** When steam and CO_2 were combined, pore development during oxidation to a uniform 0.43 AD varied with temperature. With decreasing temperature from 944°C to 650°C, micropore volume decreased, 2–10-nm pore volume increased, and 10–1,000-nm pore volume decreased (Figure 7). The total pore volume (<20,000 nm) remained constant at all temperatures, as would be expected when regenerating to the same AD. Oxidation times required to achieve this AD ranged from 2 min at 944°C to 190 min at 650°C. The Figure 7 data are presented on a unit mass basis, because all these GACs were regenerated to the same AD, the same trends would have been observed on a unit bed volume basis.

Effect on surface area. Decreasing temperature correlated to decreasing total N_2-BET surface area: from 890 m^2/g GAC at 944°C to 850 m^2/g GAC at 650°C (Figure 7). However, the BJH mesopore surface area increased with declining temperature from 250 m^2/g GAC at 944°C to 310 m^2/g GAC at 650°C. Macropore surface areas generally ranged between 3 to 5 m^2/g GAC as determined by mercury porosimetry.

From these and similar analyses, it is discerned that roughly one third of the total surface area in these coal-based regenerated and virgin GACs appeared within the mesopore range, roughly two thirds was in the micropore range, and less than 1 percent was in the macropore range.

Mesopore surface area might relate to adsorption performance when adsorbing large fulvic or humic acids or other high-molecular-weight compounds that would be too large to penetrate micropores; whereas total surface area may correlate to adsorption performance for the organic compounds that would be small enough to fit within micropores (see pre-

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vious discussion). Many of the regulated synthetic organic compounds have low molecular weights.

Adsorption capacity for I_2, PNP, and methylene blue. Adsorption capacities for iodine, *p*-nitrophenol, and methylene blue were compared for (1) spent GAC that had been oxidized to an AD of 0.43–0.44 over the 650–910°C temperature range; (2) spent GAC that had been pyrolyzed at 900°C (AD 0.48); and (3) acid-washed GAC that had been regenerated at 850°C to AD 0.435. Capacities were reported on an ash-free mass basis of comparison. Within the accuracy of measurement, the adsorption capacities were roughly equal for all these GACs and exhibited no consistent temperature-related trend (Table 3). This suggested that the range of these pore size distributions was not wide enough to detect significant differences among adsorption capacities.

Temperature effect with decreasing apparent density. Nitrogen pore volume distribution was monitored as AD declined with progressively increasing oxidation of the spent GAC. When oxidation occurred at 850°C with moderate steam flows, the pore distribution progressed as shown in Figure 8. When oxidation occurred at 750°C with moderate flows of both steam and CO_2, the pore distribution progressed as shown in Figure 9. At 750°C, pores widened solely within the <15-nm range, where the distributions were linear and parallel.

At 850°C, in contrast, the pore development occurred throughout the 2–50-nm region (which could be monitored by nitrogen adsorption) and resulted in curvilinear pore distributions. Initial steam oxidations to an AD of 0.482–0.466 widened some micropores to the 2–5-nm size and markedly increased the total pore volume <5 nm. Continued oxidation appeared to widen these pores to progressively larger sizes. Perhaps much of the easily oxidized char was gasified during the first few minutes, and extended oxidation attacked primarily the GAC skeletal structure.

Effect of oxidant gas. ***Steam, CO_2, or steam plus CO_2.*** No pore structure differences appeared whether steam, CO_2, or steam plus CO_2 was employed at 850°C (Figure 10). Moreover, 680°C oxidations caused this same pore distribution when steam alone or CO_2 alone was employed (Figure 11). However, when steam and CO_2 were used together at 680°C, a different distribution resulted, which was linear between 2 and 10 nm (Figure 11). These distinctions were manifested as a trend throughout the 650–950°C range when steam and CO_2 were used together: as temperature decreased, the destruction of small pores became more pronounced (Figure 7).

In light of these pore distributions, coupled with experimental findings pertaining to the catalyzed reaction kinetics,[3,4,5] the authors have proposed that the manner of pore development was influenced by which reaction step was rate limiting. At higher temperatures (>800°C), the water–gas shift reaction has been proposed to limit overall rate, and pore development was characterized by nonfocused gasification that was dispersed throughout all pore size ranges.

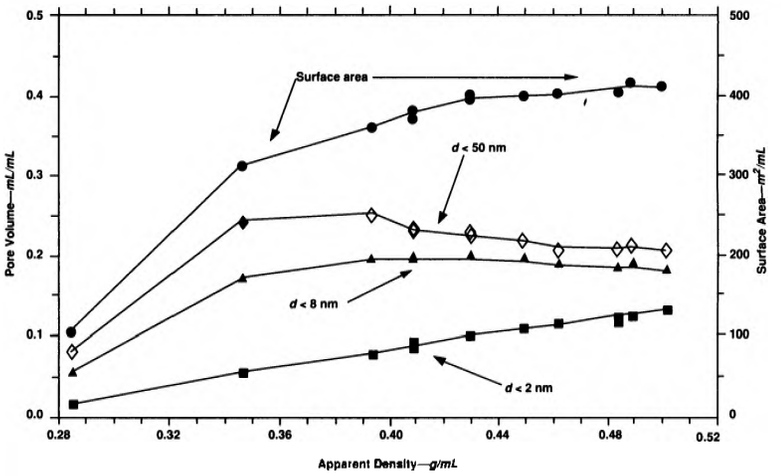


Figure 14. Pore volume distribution and surface area for spent Hays Mine GAC regenerated in moderate flow of steam at 680°C to various apparent densities

In contrast, when lower temperatures (<750°C) were employed, the gasification of solid -C(O) to gaseous CO has been proposed to limit overall rate, and pore development was characterized by a focused attack on the pores <5 nm, which caused more pore development in the 5–10-nm range.[3–5]

Reduced gas environment: steam plus H_2. Gasification was also tested with a hydrogen–steam mixture. These experiments were motivated by the findings of Matsumoto and Walker,[47] which showed that hydrogen inhibited calcium catalysis. In tests, 5 g H_2/g GAC/h plus 5 g H_2O/g GAC/h was applied to spent GAC for 160 min at 850°C before it reached the targeted AD of 0.43. This required more than an order of magnitude longer time than had been necessary to achieve this AD when the H_2 was excluded and steam was used alone. Nonetheless, the pore distribution following this gasification was nearly identical to that following steam alone (Figure 12). The surface area, at 390 m^2/mL, was also about the same as that following steam oxidation. Thus, H_2 dramatically inhibited calcium catalysis and the rate of carbon gasification, but this inhibition did not influence the product pore structure.

Effect of steam flow rate. Steam flow rate made no difference to pore development when oxidations were carried to any given AD (Figures 13, 14, and 15). Three flows were tested: a moderate rate of 8 g H_2O/g GAC/h, a high rate of 50 g H_2O/g GAC/h, and a low rate of 0.4 g H_2O/g GAC/h. The moderate flow rate was within the range commonly employed at full-scale regeneration facilities; the high rate was an order of magnitude greater; and the low rate was an order of magnitude lower. The low flow reaction rate was slowed even further by prepyrolyzing these GAC samples at 950°C. High-temperature prepyrolysis sintered the calcium and decreased its surface area of reaction.[7,8,48,49]

The oxidation rates varied considerably. For example, achieving an AD of 0.43 required 2 min at the highest flow, 9 min at the moderate flow, and 45 min at the lowest flow. Despite this, all flow rates created nearly identical pore volume distributions and surface areas when compared at the same ADs (Figures 13 and 15). As an exception, the GAC prepyrolyzed at 950°C exhibited 10 percent lower surface areas than the others. This probably reflected more on the high-temperature sintering effects than on the low-flow oxidation effects. Because pore size distribution was the same at a given AD over this wide range of flow rates, it could be reasoned that pore diffusion limitations were not manifested. This reasoning was confirmed by diffusion computations.[3,4]

During steam oxidation, temperature also did not affect the pore size distribution. Whether 680 or 850°C was used, the same pore distributions and surface areas resulted at a given AD (compare Figures 13 and 14). The 680°C oxidation to AD 0.43 took 100 min. Thus, steam alone apparently attacked the same carbon sites at 680°C as it did at 850°C.

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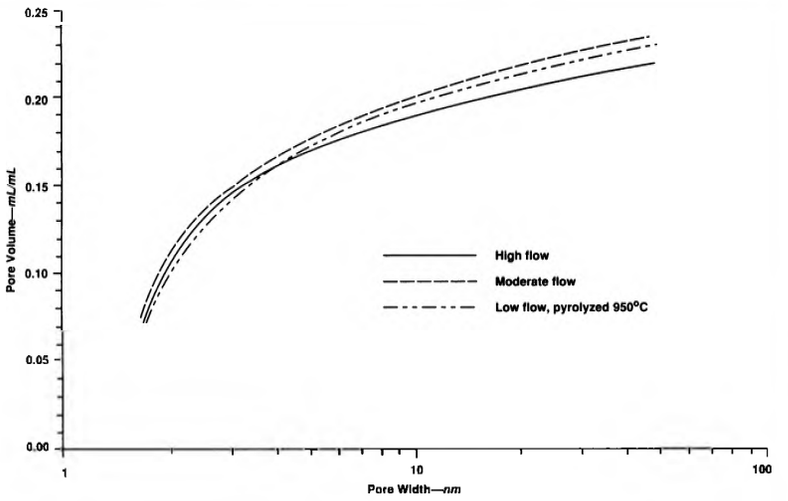


Figure 15. Effect of steam flowrate on pore volume distribution of spent Hays Mine GAC regenerated to an apparent density of 0.43 g/mL *(pyrolysis at 850°C,* **except as indicated)**

As AD decreased, the pore structure progressed through stages that were common for all three flow rates and both temperatures. Progressing from an initial 0.49 AD (for pyrolyzed GAC) down to the range of 0.44–0.47, the 2–8-nm pore volume increased slightly while the <2-nm pore volume decreased slightly and surface area remained fairly constant. This corresponded to 14–18 percent mass loss. With further oxidation to AD 0.40–0.42 and 26–28 percent mass loss, micropore volumes continued to decline, 2–8-nm pore volumes remained unchanged, 8–50-nm pore volumes increased, and surface area declined. This represented the widening of micropores to progressively larger mesopores.

Finally, below ADs in the range of 0.38–0.40 (corresponding to 28–35 percent mass loss), micropores and surface areas had deteriorated dramatically (with losses of 20–40 percent), indicating that the GAC was severely overregenerated. Severe overregeneration took only 13 min of moderate steam oxidation at 850°C or 1.7 g H_2O/g GAC, which was within the range of common oxidation conditions employed at full-scale regeneration facilities. This highlights how important it is to tailor regeneration conditions to the GAC's calcium content. A regeneration strategy that works well for a low-calcium GAC may seriously deteriorate a catalyst-laden GAC.

Effect of pyrolysis. ***Pyrolysis alone.*** Pyrolysis alone, without oxidation, most effectively restored micropores. If the spent pyrolyzed GAC was also oxidized, it always lost a fraction of its micropores. Moreover, pyrolyzed spent GAC possessed high adsorption capacity for iodine, PNP, and methylene blue (Table 3). It would be useful to determine whether this quality could still be restored after several adsorption and pyrolysis cycles.

Mere pyrolysis at any temperature between 650 and 950°C created products that were nearly identical to one another and were also similar to virgin GAC type B. Pore structure similarities can be seen by comparing the representative curves for pyrolysis at 850, 750, and 680°C in Figures 8, 9, and 11 and by comparing the product of 850°C pyrolysis with GAC type B in Figure 3. Surface areas throughout the 650–950°C pyrolysis temperature span were also consistently the same, ranging between 390 and 410 m^2/mL (790–840 m^2/g GAC).

Pyrolysis temperature did affect GAC mass loss and volume loss, however. With an increase from 650 to 950°C, bed volume loss in 10 min increased from 2 percent to 5 percent, mass loss increased from 10 percent to 16 percent, and AD decreased from 0.50 to 0.48.

There was no attempt to optimize pyrolysis times in these experiments, because the rate of pyrolytic mass loss appeared unrelated to the calcium content. Ten minutes was used merely because it allowed ample time to stabilize temperature, complete volatilization, and facilitate quantitative comparisons during oxidation. However, shorter pyrolysis times would suffice for full-scale regenerations. When spent GAC was pyrolyzed in the TGA, volatilization appeared complete within 1–3 min following 5 min of temperature rampup time.

Similarly, when spent GAC was pyrolyzed at 900°C in a fluidized bed for 3, 10, and 30 min, mass losses were 14.2, 14.7, and 16.9 percent, respectively; volume losses were 3.5, 3.5, and 4.0 percent; and final ADs were 0.49, 0.48, and 0.47 g/mL. This compared with a spent-GAC AD of 0.55. This implied that the volatilization was virtually complete within 3 min. The temperature rampup procedure added another 2–5 min of pyrolysis time to those values listed previously.

Effect of pyrolysis temperature on subsequent oxidation. High-temperature pyrolysis influenced subsequent oxidation behavior by dramatically slowing the oxidation rate and slightly affecting the resulting pore distribution. When spent GAC was pyrolyzed at 900°C before being oxidized at 750°C, its oxidation rate was half as fast as when it was pyrolyzed at 750°C before being oxidized at 750°C. Moreover, when pyrolyzed at 900°C and oxidized at 750°C to AD 0.42, it exhibited a curvilinear pore volume distribution in the small-mesopore range and linear distribution in the large-mesopore range (Figure 16). This small-mesopore distribution imitated that of spent GAC that had been pyrolyzed at 920°C and oxidized at 910°C rather than one that had been both pyrolyzed and oxidized at 750°C.

Radovic et al[48] suggested that high-temperature pyrolysis caused calcium oxide crystallites to sinter and congregate into larger pores and become less reactive catalysts. The experiments described in this article confirmed the lower reactivities but were not designed to identify where the calcium congregated. All three of these regenerations achieved the same surface area of 360–380 m^2/mL (850–890 m^2/g GAC). Thus, high-temperature presintering influenced the subsequent oxidation rate, but not in a manner that substantially improved the regenerated GAC quality.

Summary and conclusions

Metals accumulation in spent GAC. In summary, spent GACs from water treatment plants were found to contain high concentrations of calcium (0.4–7 percent) and moderate concentrations of iron (nonpyritic: 0.1–0.5 percent), aluminum (0.3–3.7 percent), manganese (0.02–0.6 percent), and magnesium (0.06–0.3 percent). Calcium, iron, manganese, and magnesium are not known to be harmful to human health. Other metals appeared in low concentrations (<0.03 percent). The GAC with the highest calcium loadings often came from water treatment plants in which lime treatment preceded the GAC filter adsorbers. However, lime treatment did not cause calcium to accumulate at all the plants where it was employed.

Acid-washing in 1 *M* HCl extracted most of these metals, and acid-washed spent GAC exhibited far lower oxidation reaction rates than spent GAC that had not been acid-washed. Of these metals extracted by acid, calcium was isolated as the one that catalyzed oxidation the most vigorously during thermal regenerations.

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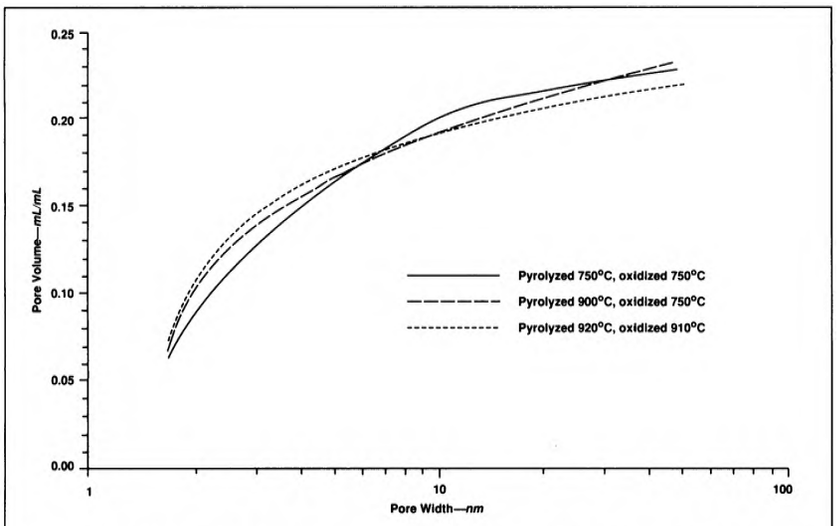


Figure 16. Effect of pyrolysis temperature on pore volume distribution for spent Hays Mine GAC oxidized at 750°C in steam and CO_2 to an apparent density of 0.42 g/mL

Restoration of pore structure in calcium's presence. The authors therefore evaluated how field-loaded calcium affected pore structure following an array of regeneration conditions. It was determined that when a spent GAC type B was thermally regenerated under the proper conditions, its virgin pore characteristics could be restored despite the presence of 1.8 percent calcium. This implied that the regeneration option for this calcium-loaded GAC provided potential as a viable alternative to discarding or incinerating spent GAC and replacing it with virgin GAC.

Pyrolysis of this spent GAC at 850°C created a product that had 6 percent fewer micropores, 13 percent more mesopores, and the same surface area as a virgin GAC type B. Comparisons have been made on a unit volume basis. Mere pyrolysis caused 14–16 percent mass loss and 3–4 percent bed volume loss.

If this pyrolyzed spent GAC also received 2–3 min of steam oxidation at 850°C, the product's characteristics became nearly identical to virgin GAC type A, with virtually the same micropore volume, surface area, and mesopore volume. This regeneration corresponded to 14–19 percent mass loss and 3–4 percent volume loss and resulted in an AD of 0.46–0.48 g/mL. The authors believe this regeneration strategy was not the only one that could have produced these pore characteristics for this spent GAC. The same pore structure would be expected when regenerations achieved this same AD with just steam or just CO_2 at any temperature or with both steam and CO_2 at temperatures above 800°C.

Influence of temperature on pore structure. The spent GAC was regenerated to a uniform AD of 0.43 g/mL under a range of regeneration conditions. All conditions caused decreased micropore volumes, decreased surface areas, and increased mesopore volumes compared with their pyrolyzed counterparts. Moreover, all but one condition caused pores to widen throughout the mesopore range. The exception occurred when both steam and CO_2 were used at temperatures below 750°C. These oxidations caused lower total surface areas, higher mesopore surface area, lower micropore volumes, higher 2–10-nm pore volumes, and a linear pore size distribution between 2 and 10 nm. These low-temperature oxidations primarily widened pores of the <5-nm range, forming pores in the 5–10-nm range. They caused negligible increase in pores >10 nm. The <5-nm pores are probably the most important for many water treatment applications. Therefore, the author recommends that field regeneration strategies avoid low temperatures (<800°C) when both steam and CO_2 are employed together and the spent GAC is loaded with calcium.

Acid-washing. By treating the spent GAC with 1 *M* HCl, the authors could produce a slightly superior regeneration product. The authors recommend that the slight improvement would not justify the added nuisance and expense incurred by employing an acid-washing step before regenerating coal-based GACs that contain <2 percent calcium.

Influence of rate-limiting step on pore development. When both steam and CO_2 were combined, the manner of pore development was proposed to be influenced by which reaction step was rate-limiting.[3–5] At higher temperatures (>800°C), the water–gas shift reaction appeared to limit overall rate, which caused nonfocused gasification that was dispersed throughout all pore size ranges.

In contrast, when lower temperatures (<750°C) were employed, the gasification of solid -C(O) to gaseous CO appeared to limit overall rate, which resulted in a focused attack on the pores <5 nm and caused more pore development in the 5–10-nm range.

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About the authors: *Fred S. Cannon is an assistant professor of environmental engineering at Pennsylvania State University, Department of Civil and Environmental Engineering, University Park, PA 16802. At the time of the research described in this article, he was completing his PhD candidacy at the University of Illinois, Champaign–Urbana. He has 10 years of consulting engineering experience. He has degrees from the University of California, Davis (BSCE), Stanford University (MS and DE), and University of Illinois, Champaign–Urbana (PhD). He is a member of AWWA. Vernon L. Snoeyink is a professor in the Department of Civil and Environmental Engineering, University of Illinois, 205 N. Mathews Ave., Champaign–Urbana, IL 61801. Ramon G. Lee is director of research and technology at the American Water Works Service Co. (AWWSC), 1025 Laurel Oak Rd., Voorhees, NJ 08043. Gérard Dagois is the senior executive vice-president of PICA, Levallois Cedex, France. James R. DeWolfe is an engineer with Gannett-Fleming Consulting Engineers, P.O. Box 1963, Harrisburg, PA 17105. At the time of this research he was with AWWSC.*

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Cannon, Fred S. ; Snoeyink, Vernon L. ; Lee, Ramon G. ; Dagois, Gérard ; DeWolfe, James R.

Journal - American Water Works Association, 1993-03, Vol.85 (3), p.76-89

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Keywords Applied sciences ; Drinking water and swimming-pool water. Desalination ; Exact sciences and technology ; Granular Activated Carbon ; Micropores ; Q1 ; Research & Technology ; Surface areas ; Water treatment and pollution

Description Thermal regenerations were conducted on a granular activated carbon (GAC) that had become exhausted (spent) in a water treatment plant where it had been in service nearly four years. This GAC contained 2 percent calcium. With this field-loaded calcium intact, proper regeneration restored the spent GAC to the same pore structure and surface area that was exhibited by several virgin GACs. When calcium appeared inside the spent GAC, it always caused micropores to be converted to small mesopores during thermal oxidation. In contrast, if the calcium was leached out of the spent GAC with acid, thermal oxidation increased only the micropore volume. Several regeneration variables were tested, including pyrolysis temperature (650-950°C), oxidation temperature (650-950°C), oxidant gas (steam and CO_2), oxidant flow rate, and oxidant extent. All regenerated products of the spent GAC that had been taken to the same targeted apparent density displayed the same surface area and pore structure except those regenerated at low temperatures (650-750°C) with both steam and CO_2 together: these produced carbons with lower volumes of small pores and lower surface areas. Se condujeron regeneraciones termales en un carbón granular activado (GAC) que había sido usado en una planta de tratamiento de aguas durante cerca de cuatro años y que contenía un 2 porciento de calcio. Con este calcio cargado intacto, una regeneración propia restauró el GAC usado con la misma estructura porosa y área superficial que mostraban varios GAC vírgenes. Cuando el calcio aparecía dentro del GAC usado, áquel siempre hacía que los microporos se convirtieran en mesoporos pequeños durante la oxidación termal. Por el contrario, si el calcio era sacado del GAC por medio de ácidos, la oxidación termal aumentaba solamente el volumen microporoso. Se probaron algunas variables de regeneración, incluyendo la temperatura de pirólisis (650-950°C), la temperatura de oxidación (650-950°C), el gas oxidante (vapor y CO_2), la velocidad de flujo del oxidante, y la capacidad del oxidante. Todos los productos regenerados del GAC usado (cargado con calcio) que habían sido tomados con la misma densidad aparente requerida mostraron la misma área superficial y estructura poroso excepto esos que se regeneraron a temperaturas menores (650-750°C) con vapor y CO_2 juntos: estos produjeron carbones con menores volúmenes de poros pequeños y menores áreas superficiales.

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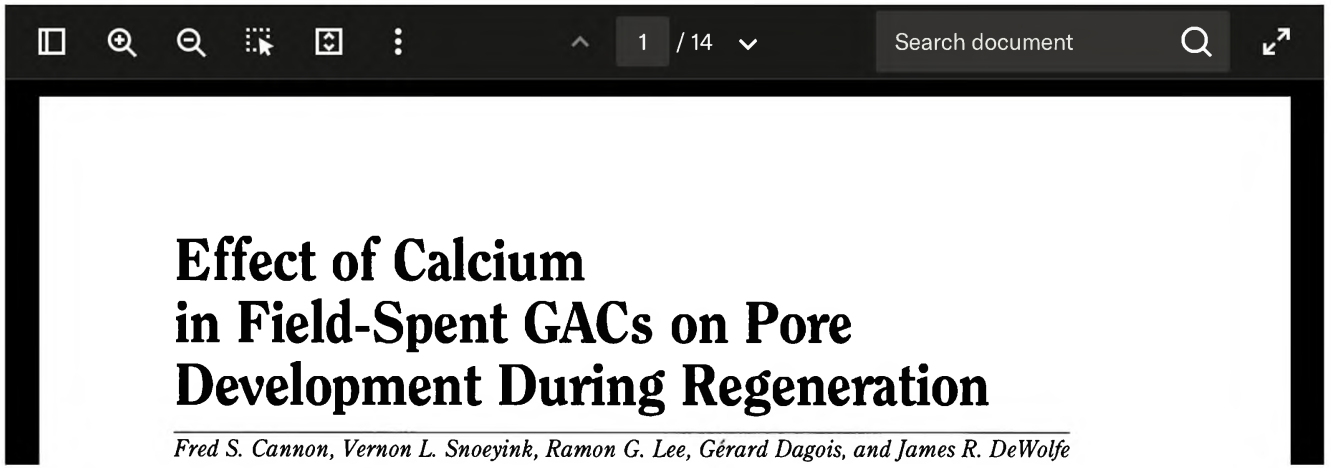
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APPENDIX 1013A

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Comparison of Activated Carbons for Removal of Perfluorinated Compounds From Drinking Water

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Comparison of Activated Carbons for Removal of Perfluorinated Compounds From Drinking Water

JAMES D. McNAMARA,[1] RALPH FRANCO,[2] RICHARD MIMNA,[2] AND LEO ZAPPA[2]

[1]Retired
[2]Calgon Carbon Corporation, Moon Township, Pa.

This article describes the challenge of treating drinking waters contaminated by perfluorinated compounds, especially perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS). A goal of the study was to establish the relative effectiveness for perfluorinated compound removal by the two primary types of granular activated carbons (GACs) commonly used in the treatment of municipal drinking water: bituminous coal-based re-agglomerated GAC and coconut-based direct activated GAC. The effectiveness of GACs in removing PFOA and PFOS to nondetectable levels is demonstrated through the use of rapid small-scale column testing. Results demonstrate that bituminous coal-based re-agglomerated carbons provide considerably greater removal capacity of the targeted compounds compared with the coconut-based direct activated carbon. In support of these findings, summaries of additional third-party test work and field installations are cited. In addition, the authors provide an overview of reactivation of activated carbon to improve the economics of the technology.

Keywords: *granular activated carbon, perfluorinated compounds, perfluorooctanoic acid, perfluorooctane sulfonate*

Perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS), among other perfluorinated compounds (PFCs), have been used in the production of various fluoropolymers. These compounds, as well as the fluoropolymers made from them, have been used in stain-resistant fabrics, nonstick cookware, firefighting foams, and other applications. Although dozens of PFCs of varying chain lengths and compositions have been detected in water, the US Environmental Protection Agency (USEPA) has shown particular concern regarding the longer-chained PFCs with eight or more carbons, such as PFOA and PFOS (USEPA 2016). Because of their ubiquitous use in the past, PFOA and PFOS usually are detected more frequently than other PFCs and at higher concentrations. They are also more toxic and more bioaccumulative than shorter-chain PFCs, such as the hexyl and butyl analogues of PFOA and PFOS.

Given their stability and solubility in water, PFCs have now become widely distributed throughout the environment, particularly in water systems. Although PFOA is not reported as an analyte in the US Geological Survey National Water Quality Assessment Program and is not monitored by USEPA's National Aquatic Resource Surveys, it has been detected in many studies of US source waters including the Tennessee River, Mississippi River, Ohio River, Lake Erie, Lake Ontario, and many others (USEPA 2016).

BACKGROUND

PFOA and PFOS health concerns and occurrence. The International Agency for Research on Cancer (IARC 2016) has classified PFOA as possibly carcinogenic to humans (group 2B) on the basis of limited evidence of testicular and kidney cancers in humans and limited evidence in laboratory animals. Although no similar cancer classifications have been developed for PFOS, there is still evidence of developmental effects attributed to toxicity (suspected particularly in the developing fetus and newborns).

Exposure data collected by the National Health and Nutrition Examination Survey 2003–2004 found PFOA in human serum in 99.7% of the population (CDC 2005). Since then, levels in human serum have declined because of the phaseout of PFOA and PFOS. All manufacture and import of PFOS in the United States was voluntarily ceased by 2002. Similarly, all manufacture and import of PFOA in the United States was voluntarily halted by 2015. The USEPA (2017) included PFOA and PFOS as candidates for possible regulation under the Safe Drinking Water Act (SDWA). In accordance with the requirements of the SDWA to determine whether to regulate a new contaminant, PFOA was added to the third Contaminant Candidate List (USEPA 2009a) and was included as one of the 30 contaminants to be studied as part of the third

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Unregulated Contaminant Monitoring Rule (USEPA 2012). Under the rule, large (and some smaller) water suppliers are required to monitor PFOA in their waters for a one-year period. This monitoring was conducted between 2013 and 2015. Results showed that PFOA was reported at or above the minimum reporting level of 20 ng/L by about 2% of the public water utilities. Approximately 0.3% of those surveyed reported levels at or above the USEPA's recently issued health advisory level of 70 ng/L, and approximately 1% reported results of combined levels of PFOA and PFOS above the health advisory level (USEPA 2016). This means that the drinking water supplies of more than six million people in the United States have concentrations of these compounds at levels exceeding the health advisory limit (Hu et al. 2016).

PFOA and PFOS have been found worldwide in soil, groundwater, surface water, and wastewater. Reported concentrations in water are typically in the nanograms-per-liter range, but much higher concentrations in micrograms per liter or even milligrams per liter are found in waters adjacent to industries where PFCs are in use (Ahrens 2011) and sites where aqueous film-forming foams have been used for firefighting and crash training (Anderson et al. 2016, Rumsby et al. 2009). For example, in communities downstream of a fluoropolymer manufacturing facility in West Virginia, PFOA was detected at concentrations ranging from 30 to 3,550 ng/L in public drinking water, and as high as 22,100 ng/L in private wells (Emmett et al. 2006). PFCs are mobile and leach into groundwater, whereas surface water sources are affected by PFCs present in treated wastewater that feeds into surface water sources, such as rivers. Most conventional wastewater processes are not effective in removing PFCs, thereby leading to the presence of these compounds in treated wastewater effluent (ASTSWMO 2015).

To date there is no established primary drinking water regulation for PFCs—only the lifetime drinking water health advisory levels set by USEPA and several individual states. These levels are summarized in Table 1 (AWWA 2016).

PFOA and PFOS properties. PFOA and PFOS are synthetic fully fluorinated organic acids. PFOA ($C_8HF_{15}O_2$) and PFOS ($C_8HF_{17}O_3S$) are strong acids generally present in their ionic forms, perfluorooctanate (pK_a = 2.8) and perfluorooctane sulfonate (pK_a = 3.27 calculated), and both exhibit significant solubility in water (PFOA: 9,500 mg/L, PFOS: 570 mg/L). These compounds are highly stable because of their saturation of fluorine–carbon bonds. Their vapor pressures are very low, and the boiling points of PFOA and PFOS are 372 and 271°F, respectively.

Because of this combination of properties, these compounds are highly mobile in surface water and groundwater and resistant to most types of degradation. Their properties also make these compounds resistant to removal by most standard treatment technologies.

APPLICATION OF GRANULAR ACTIVATED CARBON FOR PFC TREATMENT

Currently the most researched and installed technology for PFC removal is granular activated carbon (GAC). GAC has been shown to be effective at removing compounds such as PFOA and PFOS and at favorable loading capacities.

Reported lab studies. Appleman and colleagues (2013) tested several activated carbons with and without the

TABLE 1 PFC concentration guidelines

Agency	Matrix	Contaminant µg/L
USEPA	Drinking water	Lifetime health advisory: PFOA and PFOS (combined or individually)—0.070
State	**Matrix**	**Contaminant µg/L**
Illinois	Groundwater	PFOA—0.400, PFOS—0.200
Maine	Groundwater	PFOA—0.060, PFOS—0.100
Michigan	Surface water	PFOA—0.420, PFOS—0.012
Minnesota	Drinking water and fish consumption	PFOA—0.610 (lake), PFOS—0.012 (lake) PFOA—0.720 (river), PFOS—0.006 (river)
New Jersey	Drinking water	PFOA—0.040
North Carolina	Groundwater	PFOA—2
Vermont	Drinking water	PFOA—0.020

Source: AWWA 2016

PFC—perfluorinated compound, PFOA—perfluorooctanoic acid, PFOS—perfluorooctane sulfonate, USEPA—US Environmental Protection Agency

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presence of dissolved organic carbon (DOC) for perfluoroalkyl acid (PFAA) removal at levels of 1 µg/L. A coal-based GAC[1] was shown to have the highest adsorptive capacity, exhibiting less than 20% breakthrough of all PFCs at 125,000 bed volumes (BVs) treated. All carbons experienced a 20% breakthrough at 10,000 BVs when DOC was added.

Qiu et al. (2007) conducted isotherms using a coal-based GAC,[2] a coconut-based GAC,[3] a peat-based GAC,[4] and a wood-based GAC.[5] The coal-based GAC was found to have the best overall performance for the PFCs tested. In general, all products performed better for the longer-chain PFCs, which include PFOA and PFOS. The presence of natural organic matter (NOM) was shown to adversely affect the adsorption capacity of these products.

Rahman (2014) conducted isotherm tests for the adsorption of three PFCs with the coal-based GAC,[2] a coconut-based GAC,[6] and two wood-based GACs,[7,8] along with a biochar[9] and a bone char.[10] The coal-based GAC was the superior product for the three targeted compounds, and even rivaled two different ion exchange resins in similar tests conducted by Rahman et al. Treatment of surface waters containing NOM negatively influenced the adsorptive capacities of all media but not to such an extent as to make the technologies infeasible.

Field experience. In full-scale field operation, activated carbon has proved to be a cost-effective means for meeting PFC effluent targets.

Little Hocking, Ohio. The public water supply was contaminated with concentrations of PFOA as high as 8.5 µg/L by discharges from a nearby manufacturing plant. A GAC plant began operation in 2007 to treat incoming groundwater with an average of 3.3 µg/L of PFOA. Carbon is exchanged at approximately three-month intervals. No detectable PFOA has been found entering the water supply for the town (Cummings et al. 2015).

Oakdale, Minn. Groundwater contamination with several PFCs from a manufacturing site led to the installation of ten 10,000 lb-capacity carbon vessels with a flow capacity of 2,000 gpm. The lead vessels of two vessel trains are changed when concentrations in the outlet reach 50% of the inlet level. By replacing carbon on the basis of PFOA breakthrough, the plant was able to treat 1.9 bil gal over 23 months, which amounted to a cost of $0.12/1,000 gal treated (Bachmeier 2015).

New Jersey American Water: Logan System Birch Creek. GAC absorbers' were designed to provide an empty bed contact time (EBCT) of 15 min using coal-based re-agglomerated GAC to remove PFOA and perfluorononanoic acid from groundwater at influent concentrations of 18–72 ng/L. The contaminants are removed to below reporting levels of 5 ng/L (Engineering Performance Solutions 2010).

Hoosick Falls, N.Y. Two 20,000 lb vessels in lead–lag configuration were in temporary use to treat drinking water contaminated from an industrial site. Sampling of the public water supply before the installation detected PFOA at concentrations between 400 and 1,000 ng/L (Hoosick Falls 2016). The temporary system treated nearly 210 mil gal of groundwater in 290 days with no breakthrough in system effluent. A permanent system consisting of two 40,000 lb vessels filled with coal-based GAC has been effectively reducing PFOA levels from over 400 ng/L to below 2 ng/L since December 2016.

Anglian Water, United Kingdom. Five GAC filters with coal-based GAC producing an EBCT of 65–110 min are used to treat groundwater with 3.7 µg/L PFOS and other PFCs emanating from a nearby airbase. The carbon is reactivated annually after treating about 5,500 BVs of water. The breakthrough of PFOS and PFOA would occur at 8,000–9,000 BVs (Rumsby et al. 2009).

Reactivation of GAC. A key feature in GAC use is the thermal reactivation of the spent product by which it is essentially remanufactured for reuse in the application. Not only does the use of reactivation greatly improve the economics of applying GAC, but it also eliminates the need to dispose of spent carbon and its contaminant loadings, removing any future liabilities associated with those wastes. This process has been widely used at several municipal treatment plants and commercial facilities across the United States. In the reactivation process, the activated carbon is heated to temperatures of at least 800°C using steam as a selective oxidant. It should be noted that in testing of activated carbons containing PFCs, Watanabe et al. (2016) found no residual perfluoroalkyl substances on activated carbon when heated to 700°C in nitrogen. During reactivation, the adsorbed organic material either boils off the activated carbon or is broken down to a char in the carbon pores. The selective oxidation process restores the pore structure of the product and makes it suitable for reuse. All organic emissions are subsequently burned in the system's afterburner, and acid gases (including hydrogen fluoride) are neutralized in alkaline scrubbers.

CHARACTERIZATION OF GAC PORE STRUCTURES

As in all activated carbon adsorption applications, the carbon's performance in adsorbing PFCs from groundwaters should be influenced by the quality and characteristics of the GAC used. An extensive range of GACs are available for water treatment, and these differ by the source of carbonaceous starting material, the method of manufacture, and level of activation. There are no "bad" activated carbons, only "poorly applied" activated carbons; therefore, the challenge is to match the qualities and characteristics of the various activated carbons available to the specific water treatment task at hand. In the current study, performance testing was conducted to compare the PFC removal performance of two types of GACs, both of which are often used in the treatment of groundwater contamination. For inclusion in this study, the authors selected two bituminous, coal-based

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re-agglomerated, steam-activated carbon products[11,12] and two direct steam-activated coconut-shell-based carbons[13,14] (Table 2).

The coal-based re-agglomeration process involves the following five steps: pulverization of the coal, addition of a carbonaceous binder, reforming of a briquette in the press room, sizing based on final product dimensions, and baking and steam activation. The direct activation process requires three steps: low-temperature charring of raw coconut shells, sizing of char on the basis of final product dimensions, and steam activation.

Re-agglomeration during coal-based carbon manufacture is a technique that has been used for many years to improve certain characteristics of GAC. In short, re-agglomeration provides "man-made" cracks and crevices that resemble the transport pore structure in the granulated carbon. The transport pore structure can be defined as those pores that are greater than 100 angstroms (Å) in diameter and have very limited adsorption energy but provide the important pathways by which contaminants travel to the adsorption pores located throughout the granule. Not only does the re-agglomeration process increase the number and volume of transport pores in the final product, but it also provides pathways that allow a more homogenous activation during the manufacturing process. This results in a more consistent adsorption pore size distribution ranging from the very outer surface of the granule to the innermost points of the particle. One of the re-agglomerated coal-based carbons was tested in its virgin state. The second coal-based carbon was a custom municipal reactivated (CMR) GAC that before use was the same product as the virgin carbon tested. The CMR carbon was in service for 12 to 18 months at an unnamed drinking water treatment facility, removing surface water NOM and total organic carbon (TOC) for the prevention–reduction of disinfection byproducts post-chlorination. On the basis of the age of the custom reactivation pool, it was estimated that the CMR carbon used in this study had undergone three reactivation cycles. Custom reactivation is conducted with steam at approximately 800°C, well above the 700°C threshold reported by Watanabe et al. (2016). The direct activated coconut

TABLE 2 Activated carbon types used in RSSCT study

GAC Type	Description
Virgin bituminous coal, 12 × 40	Moderate-density bituminous re-agglomerated coal-based GAC, 12 × 40 mesh
Coconut, 8 × 30	Direct activated coconut-based GAC, 8 × 30 mesh
Coconut, 12 × 40	Direct activated coconut-based GAC, 12 × 40 mesh
Reactivated bituminous coal, 12 × 40	CMR re-agglomerated GAC, 12 × 40 mesh

CMR—custom municipal reactivated, GAC—granular activated carbon, RSSCT—rapid small-scale column testing

The four GACs tested in the RSSCT study were manufactured and supplied by Calgon Carbon Corporation, Moon Township, Pa.

TABLE 3 Activated carbon characterization data

Parameter	Virgin Bituminous Coal 12 × 40	Coconut 8 × 30	Coconut 12 × 40	Reactivated Bituminous Coal 12 × 40
Apparent density—*g/cc*	0.532	0.533	0.539	0.527
Ash—*%*	8	2.3	2.1	9.9
Iodine number—*mg/g*	948	1,039	1,149	932
TCN—*g/cc*	11.1	15.1	17.7	10.4
Molasses number	173	144	138	178
Screen mesh size—*weight % on pan*				
8	0	0.5	0	0
10	0	15.8	0	0
12	4.1	32.4	0.3	0.3
16	50.3	30.1	34.4	29.9
20	30.6	14.5	38.6	43.9
30	12.2	6.6	18.9	20
–30	0	0.1	0	0
40	2.4	0	7.4	5.3
–40	0.4	0	0.4	0.6

TCN—trace capacity number

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carbons selected for inclusion in the comparison study described here were an 8 × 30 mesh product[13] and a 12 × 40 mesh product.[14]

The following common standard carbon tests were conducted on each of the test carbons, including apparent density (ASTM International 2014a), iodine number (ASTM International 2014b), ash (ASTM International 2011), trace capacity number (TCN) test method (Nowicki 2014), and molasses number. Results are shown in Table 3.

Total adsorbent characterization via temperature influence correction (TACTIC) analyses (Greenbank et al. 1991) were performed to generate "fingerprint" pore size distribution comparisons for the GACs used in the testing. TACTIC analyses have been used by Calgon Carbon Corporation to characterize adsorption pore-size distributions for 35 years. During the test, the adsorbent is exposed to a pure gas adsorbate over a large range of temperatures. The adsorption of the gas requires more pore energy as the temperature increases. The energy of the pore is directly related to its size or average diameter. The weight of the carbon and adsorbed gas is precisely measured as the temperature is slowly increased and decreased, logging approximately 1,000 adsorption data points covering seven orders of magnitude in relative pressure and three orders of magnitude in carbon loading. The data are fit to a sixth-order polynomial equation, which can be used to directly compare adsorbents for a common application. Figure 1, parts A and B, shows the differential pore volume measured versus the adsorption potential energy (x-axis), which is directly related to pore size (secondary x-axis).

Although all four carbons exhibited very similar total cumulative adsorption pore volumes (approximately 40 cc/100 g), the distribution of the adsorption pores as shown in the graph (Figure 1, part A) of the differential pore volumes (pores of up to 100 Å in diameter) is much different for the re-agglomerated coal-based GACs versus the direct activated coconut GACs. The coconut-based GACs exhibited higher pore volumes for pores with diameters ≤10 Å, while the re-agglomerated coal-based carbons showed higher pore volumes in the larger pores (10–100 Å in diameter). Although TACTIC analysis does not measure the transport pore structure discussed previously, the slopes of the differential curves between 10 and 30 Å provide an estimation of the pore volumes of transport pores ranging from 100 to 1,300 Å in diameter. Therefore, the TACTIC curves suggest the coconut-based GACs have significantly less transport pore structure than the re-agglomerated coal-based GACs. This was also confirmed by the molasses number values for each of the GACs presented in Table 3, showing the coconut GACs between 138 and 144, while the coal-based GACs exhibited molasses numbers of 173 and 178. The molasses number test method has long been used in the activated carbon industry to measure a carbon's relative performance in color removal applications. The molasses test is a relatively good measure of adsorption kinetics, which is directly related to the amount of transport structure in an activated carbon.

Part B of Figure 1 is an expanded view of part A and shows the differences in pore volumes in the higher-energy pore structure range. The curves clearly indicate that the coconut-based GACs showed higher volume in the high-energy adsorption pores than the coal-based GACs. The TCN test method, developed by Calgon Carbon to measure the performance of activated carbons for the removal of very soluble, low-molecular-weight contaminants present at low concentrations (Nowicki 2014), also confirmed the coconut-based GACs' superior pore volume in the higher-energy pore region, exhibiting high TCN values of 15.1 and 17.7 g/cc versus values of 11.1 and 10.4 g/cc for the coal-based GACs.

In terms of GAC treatment, typical PFC removal applications would be considered to be trace removal type applications, i.e., the adsorption of very soluble organic compounds at very low contaminant concentrations. This would suggest that GACs exhibiting higher volumes of higher-energy pore structure would outperform GACs having larger adsorption pores and lower overall adsorptive energy.

EXPERIMENTAL STUDY

In this research, a laboratory study was conducted to determine the relative efficacy of four different GAC products for the removal of PFOA and PFOS. The study detailed here used rapid small-scale column testing (RSSCT) to determine the relative adsorbability of PFOA and PFOS in the presence of typical TOC concentrations in groundwater, demonstrate the effectiveness of GAC for the control of these compounds, and compare the performance of re-agglomerated coal-based GACs with that of coconut-based GACs.

EXPERIMENTAL METHODS

RSSCT design. The RSSCT design simulated a fixed GAC bed providing a 10-min EBCT; RSSCT design parameters are listed in Table 4.

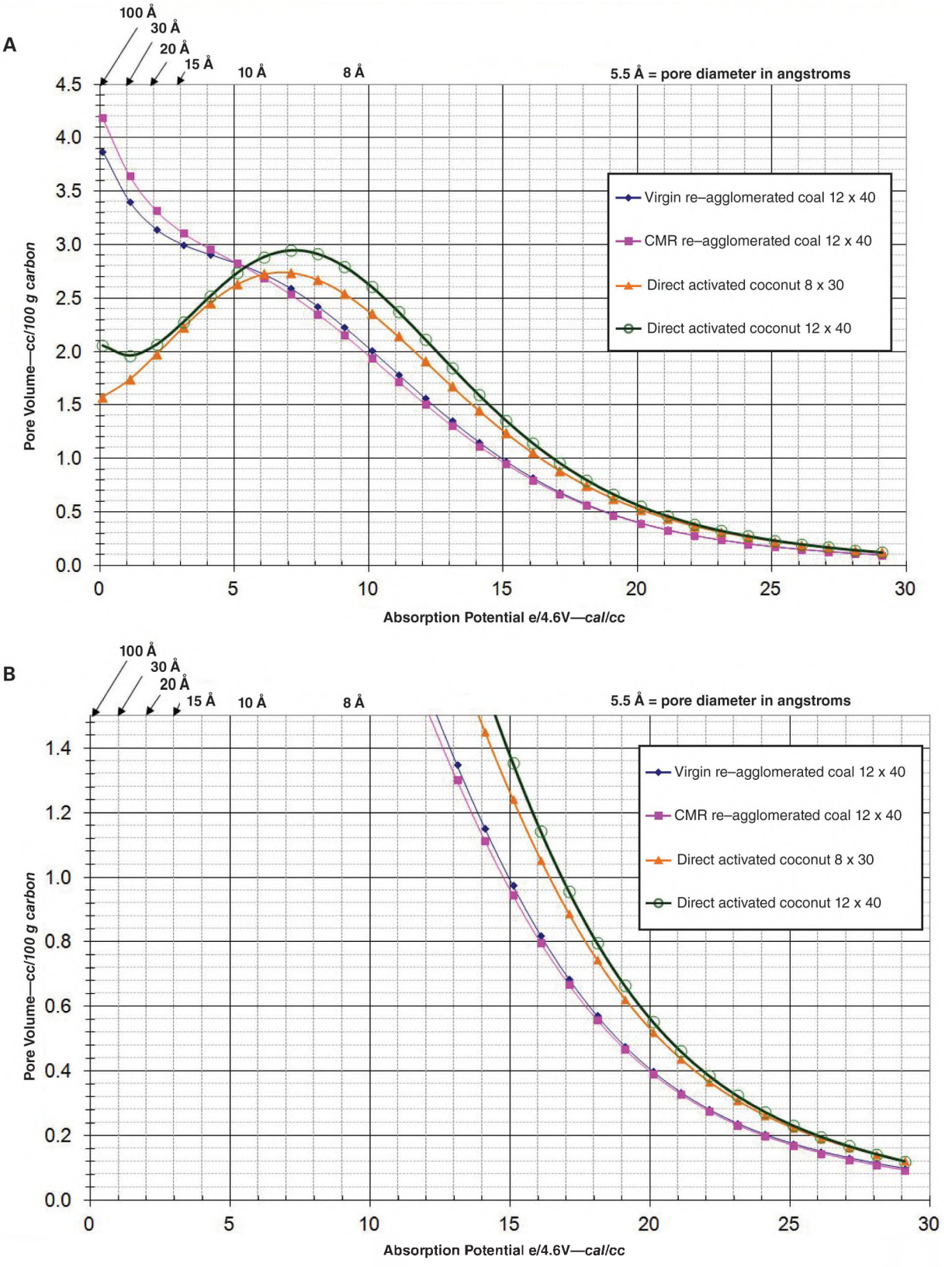
The RSSCT procedure uses a miniature carbon-filled column to rapidly simulate the adsorption breakthrough curve that would be obtained by treating an aqueous stream in a large adsorption system. This technique has been shown (Crittenden et al. 1986) to accurately simulate the carbon treatment of a wide range of waters and wastewaters under various conditions.

The principle advantage of the RSSCT procedure compared with the 1 in.-diameter column adsorption test is its increased speed. Typically, an RSSCT can be completed in less than 1–15% of the time required for a 1 in.-diameter study.

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FIGURE 1 TACTIC differential adsorption pore size/pore energy distribution curves (A) and close-up view (B)



CMR—custom municipal reactivated, TACTIC—total adsorbent characterization via temperature influence correction

Part B of the figure is an expanded view of part A showing the differences in pore volumes in the higher-energy pore structure range.

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TABLE 4 RSSCT design parameters

Parameter	Virgin Bituminous Coal 12 × 40	Coconut 8 × 30	Coconut 12 × 40	Reactivated Bituminous Coal 12 × 40
Particle diameter ($d_{p,LC}$)—*mm*	1.11	1.52	1.09	1.06
Particle diameter ($d_{p,SC}$)—*mm*	0.1229	0.119	0.1193	0.1209
AD—*g/mL*	0.532	0.533	0.539	0.527
Small column diameter—*m*	0.622	0.622	0.622	0.622
Scaling factor ($d_{p,LC}/d_{p,SC}$)	81.3	162.5	82.9	76.8
$EBCT_{SC}$—*min*	0.123	0.062	0.121	0.130
$Reynolds_{LC}$	12.70	17.38	12.45	12.14
$Velocity_{SC}$—*m/h*	16.5	16.5	16.5	16.5
$Length_{SC}$—*m*	0.0338	0.0169	0.0332	0.0358
Q—*mL/min*	8.36	8.36	8.36	8.36
Weight $carbon_{SC}$—*g*	0.5466	0.2741	0.5433	0.5736
Volume of $carbon_{SC}$—*mL*	1.027	0.514	1.008	1.088

AD—apparent density, EBCT—empty bed contact time, RSSCT—rapid small-scale column testing

Q is the flow rate, and $d_{p,SC}$ and $d_{p,LC}$ are the particle sizes for the small- and large-scale GACs, respectively.

The differing scale factors and operating conditions for the coconut 8 × 30 calculated using the RSSCT procedures are attributable to its larger mesh size compared with the 12 × 40 carbons.

To predict the volume breakthrough curve for the full-scale adsorber, the RSSCT results must be multiplied by the volume scale factor determined for each carbon type. The time breakthrough curve for the full-scale adsorber can be calculated by either of two methods. In the first method, the predicted volumes calculated by multiplying the RSSCT results by the volume scale factor are divided by the flow rate of the full-scale system. In the second method, the run time is multiplied by the scale factor determined for each carbon type.

Comparisons between small and full scale can be made using the following equations:

$$\frac{EBCT_{SC}}{EBCT_{LC}} = \left[\frac{d_{p,SC}}{d_{p,LC}}\right]^{2-x} = \frac{t_{SC}}{t_{LC}} \quad (1)$$

$$\frac{V_{SC}}{V_{LC}} = \frac{d_{p,LC}}{d_{p,SC}} \quad (2)$$

$$M_{SC} = EBCT_{LC}\left[\frac{d_{p,SC}}{d_{p,LC}}\right]^{2-x} Q_{SC} \times \rho_{LC} \quad (3)$$

where $d_{p,SC}$ and $d_{p,LC}$ are the particle sizes for the small and large GACs, respectively, and x is the diffusivity constant, which can be used (0 for constant or 1 for proportional diffusivity). In the current study, it was assumed that the intraparticle diffusivities do not change with particle size; therefore a constant diffusivity factor ($x = 0$) was used. In addition, t_{SC} and t_{LC} are the corresponding elapsed times in the small- and large-scale column tests, respectively; V_{SC} and V_{LC} are the velocities in the RSSCT and large-scale columns, respectively; M_{SC} and Q_{SC} are the mass of carbon and flow rate, respectively, in the small-scale column; and ρ_{LC} is the apparent density of the full-scale carbon.

RSSCT carbon preparation. The RSSCTs were conducted using four different GACs—two re-agglomerated bituminous coal-based GACs and two direct activated coconut-based GACs (Table 2).

Current production samples of the two re-agglomerated bituminous coal-based GACs (virgin and CMR) and the 8 × 30 and 12 × 40 standard coconut-based GACs were obtained for this study. In preparation for the RSSCT, each GAC was systematically resized to approximately 100 × 200 mesh using a ball mill.[15] The sized GACs were dry-screened to 100 × 200 mesh before particle size analysis.

The mean particle diameters of the sized carbons were determined using a particle size analyzer[16] that records the particle size spectra by laser diffraction between 0.375 and 2,000 µm. The particle size distribution as well as the mean particle diameter of the sized carbons (the $d_{p,SC}$ value used in the RSSCT calculations) are presented in Figure 2.

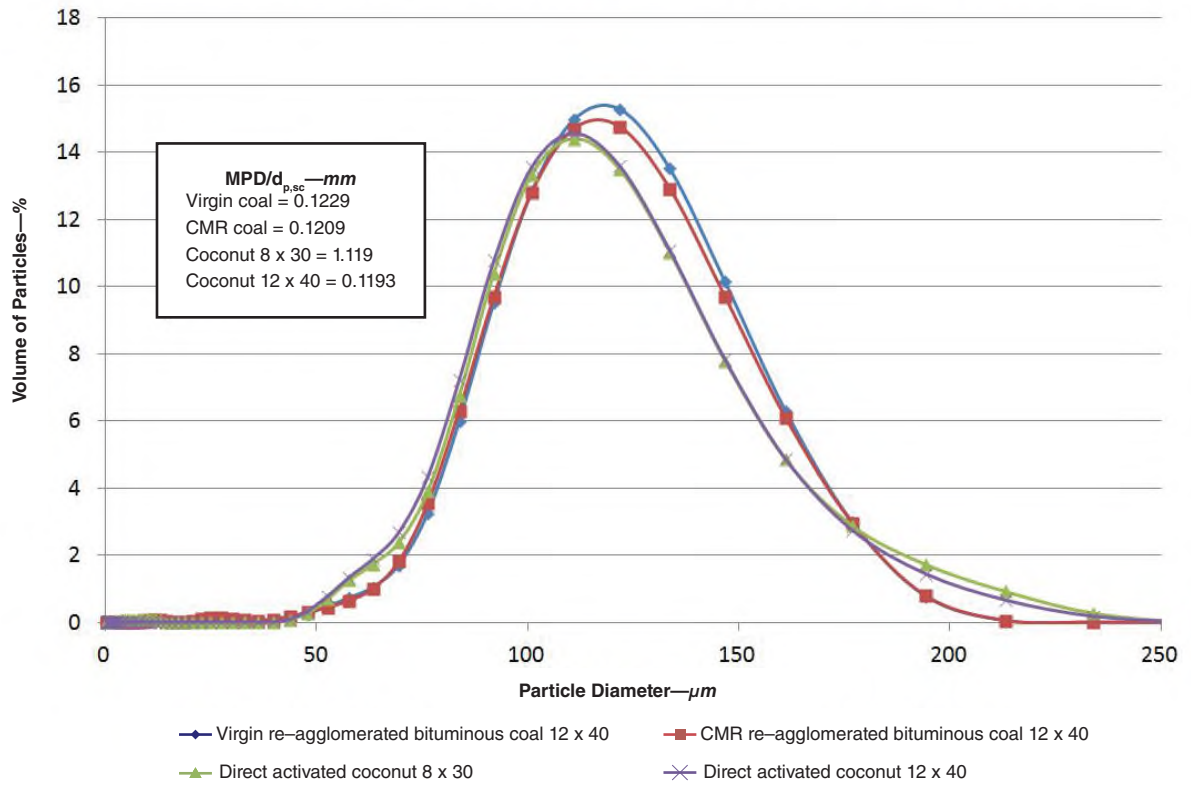
The dry, sized carbons were installed in the test columns. Before the introduction of the feedwater, each carbon column was pre-wetted with purified water[17] for approximately 16 h in an upflow direction, at a flow rate comparable to that used in the test runs. This procedure has proved very effective at degassing the small-scale column carbon bed over many years of practicing the method.

RSSCT feedwater preparation. Municipal tap water (which does not contain PFCs at any detectable

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FIGURE 2 RSSCT carbon size distribution curves for test carbons



CMR—custom municipal reactivated, $d_{p,SC}$—small-scale particle size, MPD—mean particle diameter, RSSCT—rapid small-scale column test

concentrations) was received in eight 55 gal drums from Center Township, Pa. This water was from the municipality's groundwater sources (after chlorination) and was not treated with GAC at the time of the sampling. The chlorine level in the municipality distribution system averages <1 mg/L as reported in the township's 2015 consumer confidence report. A standard water quality analysis was performed before addition/spiking of PFCs (Table 5).

Stock solutions of 100 mg/L PFOA and PFOS were prepared using deionized water and potassium PFOS or PFOA, both purchased from a supplier[18] and used as received. A calculated and precisely measured volume of the stock solutions was added to the feedwater containers to target 1 µg/L concentrations each of PFOA and PFOS. The feedwater averaged 0.8 µg/L PFOS and 0.92 µg/L PFOA after spiking. Although these concentrations are well above the USEPA health advisory level, and it is impossible to select a single concentration that is representative of all PFC removal scenarios, they are representative of concentrations that have been reported in both drinking water and remediation applications. Furthermore, these levels ensured a good analytical comparison among the

TABLE 5 Water quality data

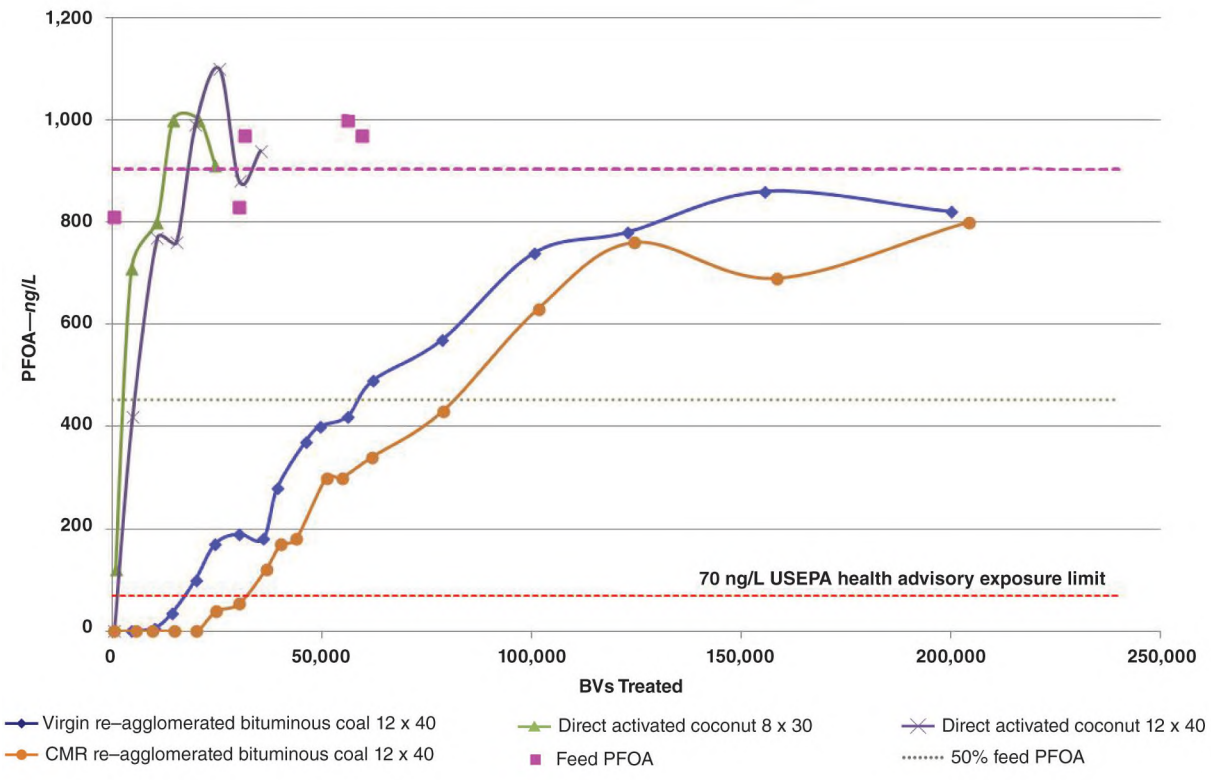
Test	Result
TSS—*mg/L*	<1
TDS—*mg/L*	470
TOC—*mg/L*	1.42
Conductivity—*µS/cm*	815
pH	7.86
Total Fe—*mg/L*	<0.03
Total hardness—*mg/L as* $CaCO_3$	300
Alkalinity (methyl orange)—*mg/L as* $CaCO_3$	145
Common Anions (IC)	Result
Fluoride (F^-)—*mg/L*	0.17
Chloride (Cl^-)—*mg/L*	105.7
Bromide (Br^-)—*mg/L*	<0.2
Nitrate (as NO_3^-)—*mg/L*	4.37
Phosphate (PO_4^-)—*mg/L*	<0.5
Sulfate (SO_4^-)—*mg/L*	114.9

$CaCO_3$—calcium carbonate, Fe—iron, IC—ion chromatography, TDS—total dissolved solids, TOC—total organic carbon, TSS—total suspended solids

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FIGURE 3 RSSCT breakthrough curves of PFOA



BV—bed volume, CMR—custom municipal reactivated, PFOA—perfluorooctanoic acid, RSSCT—rapid small-scale column test, USEPA—US Environmental Protection Agency

GACs as well as a reasonable testing time frame that would not require months to reach breakthrough.

RSSCT sampling and analyses. Effluent samples were collected eight times daily for five days and three times daily thereafter at regular intervals via automated sample collectors. Column effluent samples designated for PFC analyses were placed in high-density polyethylene bottles with preservative[19] and shipped overnight on ice to a commercial laboratory[20] for analysis according to method 537 (USEPA 2009b).

The flow rates of the RSSCTs were closely monitored throughout the study. In addition to periodic flow rate checks, the RSSCT effluent water not placed in individual sample bottles was collected in a larger composite container and was gravimetrically measured to establish an accurate average flow rate over the course of the study.

RESULTS AND DISCUSSION

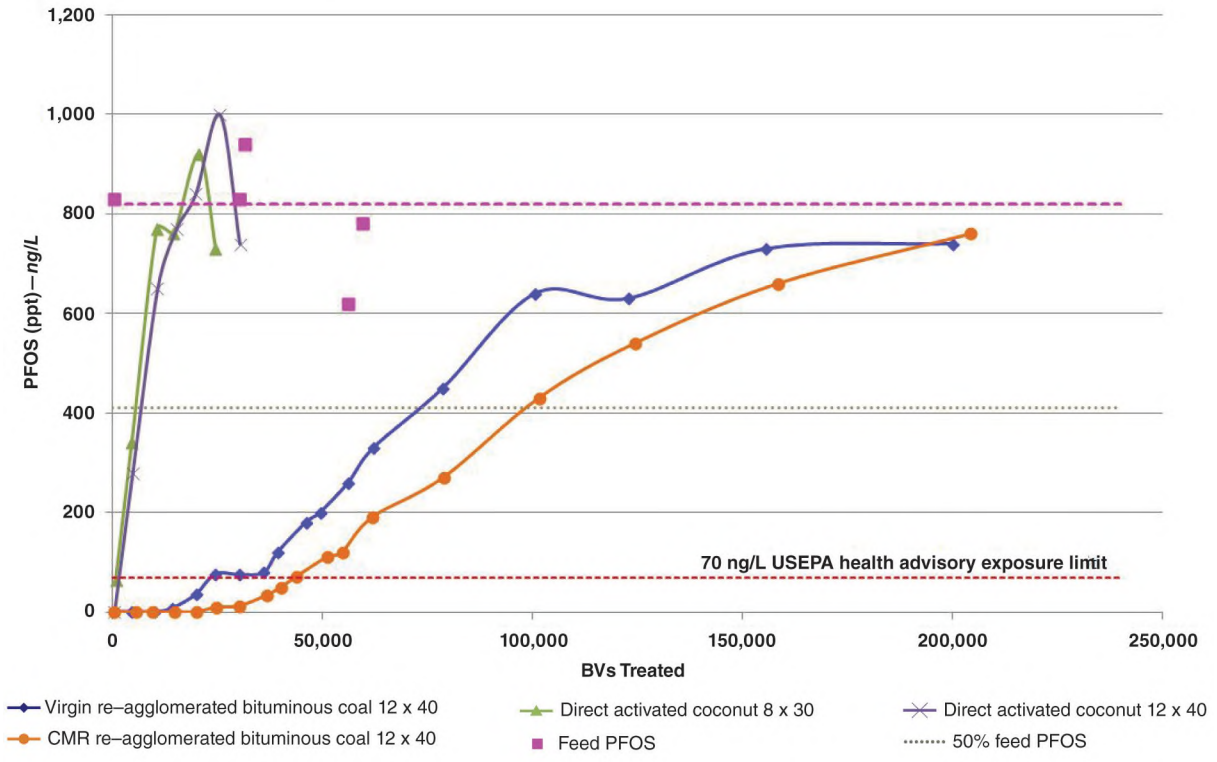
The RSSCT simulated a GAC treatment system that provided an EBCT of 10 min. The weight of the carbon in the simulated system was approximately 23,500 lb. It was assumed that the full-scale system consisted of a 10 ft.-diameter adsorber operating at 530 gpm. Each RSSCT was run until the column effluent contained a minimum of 50% of the feed PFC concentrations. The longest of these ran for ~200,000 BVs, which simulated ~1,400 days of operation and the treatment of ~1 bil gal of water. The resulting breakthrough curves are shown in Figures 3 and 4 for PFOA and PFOS, respectively.

All four GACs tested performed better at removing PFOS than PFOA. Comparatively, the re-agglomerated bituminous coal-based carbons greatly outperformed the coconut-based carbons for removal of both PFOA and PFOS. The differences and similarities of the GACs used in this study were discussed previously in the section on application of GAC for PFC treatment. On the basis of the characteristics of the two coconut-based GACs used in this study, it would be reasonable to expect coconut-based GACs to perform well for the removal of PFC contaminants from water. However, the coconut-based GACs could not effectively remove PFCs for any reasonable treatment period. These GACs experienced rapid initial breakthrough and

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FIGURE 4 RSSCT breakthrough curves of PFOS



BV—bed volume, CMR—custom municipal reactivated, PFOS—perfluorooctane sulfonate, RSSCT—rapid small-scale column test, USEPA—US Environmental Protection Agency

reached loading saturation much more quickly than the coal-based GACs. The reason for the poor performance of the coconut-based GACs in this application did not appear to be attributable to inadequate adsorption pore structure but rather to an inadequate transport pore structure. Although they possessed fewer high-energy adsorption pores and approximately the equivalent overall adsorption pore volume of the coconut-based GACs, the re-agglomerated coal-based GACs (both virgin and reactivated) were able to better use the adsorption pores available because they had a much larger and well-developed transport pore structure, attributable at least in part to the re-agglomeration manufacturing process.

The reactivated coal-based GAC was observed to outperform even the virgin coal-based GAC. In theory, repeated reactivation reduces adsorption pore volume, decreases carbon structure, and increases transport pore volume. It would appear that through at least three reactivation cycles, the increase in transport pore volume of the reactivated coal-based GAC resulted in even better PFC removal performance than that of its virgin counterpart, despite the loss of some adsorption pore volume.

Comparisons of the four carbons' performance in terms of service life are shown in Tables 6 and 7 for a single-vessel design and a lead–lag dual-vessel design, respectively. The single-vessel design change-out criterion was assumed at the 70 ng/L breakthrough of PFOA, the USEPA health advisory exposure limit for PFCs. For a lead–lag design, the change-out criterion was set at 50% breakthrough in the lead bed. During the period when the first bed would be achieving high loadings and eluting up to 50% of the PFC present in the influent water, the lag bed would consistently produce an effluent water below 70 ng/L PFOA.

It should be noted that the presence of other adsorbing organic compounds present in groundwaters from different sources/regions could compete with the adsorption of PFCs, changing the maximum loadings and subsequent service lives of full-scale treatment systems. For that reason, the carbon use rates and predicted service lives presented in this study may not match those in other source waters, even when PFC concentrations are similar. Therefore, performance testing is recommended when accurate carbon use rates and system size information are required.

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TABLE 6 Carbon service life estimates for PFOA in single-vessel design

Activated Carbon	BVs Treated *n*	Simulated Days *n*	Water Treated *gal*	Carbon Use Rate *lb/1,000 gal*	Total PFC Loading at Initial PFOA Breakthrough *µg/g*
Coconut, 8 × 30	300	2	1,590,000	14.84	1.3
Coconut, 12 × 40	500	3	2,650,000	9.01	1.5
Virgin bituminous coal, 12 × 40	17,200	119	91,160,000	0.26	50
CMR bituminous coal, 12 × 40	31,000	215	164,300,000	0.14	100

BV—bed volume, CMR—custom municipal reactivated, *n*—number, PFC—perfluorinated compound, PFOA—perfluorooctanoic acid

TABLE 7 Carbon service life estimates for PFOA in lead–lag, dual-vessel design

Activated Carbon	BVs Treated *n*	Simulated Days *n*	Water Treated *gal*	Carbon Use Rate *lb/1,000 gal*	Total PFC Loading at 50% PFOA Breakthrough *µg/g*
Coconut, 8 × 30	3,000	21	15,900,000	1.48	7
Coconut, 12 × 40	5,400	38	28,620,000	0.83	12
Virgin bituminous coal, 12 × 40	59,200	411	313,800,000	0.075	157
CMR bituminous coal, 12 × 40	82,000	592	434,650,000	0.054	220

BV—bed volume, CMR—custom municipal reactivated, *n*—number, PFC—perfluorinated compound, PFOA—perfluorooctanoic acid

CONCLUSION

The study described here yielded the following findings.

- The re-agglomerated bituminous coal-based GACs (both virgin and reactivated versions) outperformed the coconut-shell-based GACs in the column studies reported here. The performance ranking of the carbons tested were in agreement with other published isotherm results (Rahman 2014, Qiu et al. 2007), although in the current case, using this particular groundwater, coconut carbons demonstrated near non-effective PFOA and PFOS removal performance. It is likely that the abundant adsorption pore structure of the coconut-based GACs was not accessible because of the relatively narrow transport pore structure of direct activated coconut-based GACs, relative to re-agglomerated coal-based GACs. Although the re-agglomerated bituminous coal-based GACs contained fewer high-energy pores relative to the coconut-based carbons, the larger transport pore structure allowed access to the adsorption pores that were effective at adsorbing the PFC contaminants.
- The CMR bituminous coal-based re-agglomerated GAC used in many municipal water treatment applications proved to be very effective at removing PFOA and PFOS after repeated cycles of high temperature reactivation and even outperformed its virgin counterpart. At this time, it remains unknown if there is a greater number of reactivation cycles beyond which PFC removal performance might begin to decline, but reactivation would clearly provide an economic benefit for the effective control of PFC contaminants in groundwater.
- The carbon use rates exhibited by the coal-based re-agglomerated GAC products indicated that cost-effective systems and operating costs can be achieved for the treatment of PFC-contaminated waters. In this case, the 411-day service life of the virgin bituminous coal-based re-agglomerated GAC translated into an operational cost of $0.12/1,000 gal treated and capital costs of approximately $275,000/mgd treated (excluding auxiliary systems). If custom reactivation is included, operating costs drop to $0.08/1,000 gal treated (again, based on the data set described here). Total actual capital and operating costs will depend on source water quality, contaminant concentrations, and other requirements that can influence the facility design and site preparation.

SUMMARY

Adsorption via bituminous coal-based re-agglomerated GAC appears to be the most widely used and cost-effective technology to remove PFCs from water. To date, it is the only technology applied specifically for the treatment of PFC-laden drinking water at full scale (Dickenson & Higgins 2016). Furthermore, with the ability to minimize

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treatment residuals and recycle the spent media, additional cost benefits can be realized.

Differing performance results have been found at various application sites because of different combinations of the PFCs and background NOM. Testing can be used to determine the expected use rates for activated carbon at specific locations.

ENDNOTES

[1]FILTRASORB® 300, Calgon Carbon Corporation, Moon Township, Pa.
[2]FILTRASORB® 400, Calgon Carbon Corporation, Moon Township, Pa.
[3]Diasorb W10-30, Calgon Carbon Japan, Tokyo
[4]PK1-3, Cabot Norit Activated Carbon, Marshall, Tex.
[5]Wako GAC, Wako Pure Chemical Industries, Osaka, Japan
[6]AquaCarb CX 1230®, Evoqua Water Technologies, Warrendale, Pa.
[7]C-Gran®, Cabot Norit Activated Carbon, Marshall, Tex.
[8]WV B30®, Mead Westvaco, North Charleston, S.C.
[9]Dairy manure-based biochar, Char Technologies, Toronto, Ont., Canada
[10]Fija Fluor, Apelsa Carbon, Jalisco, Mexico
[11]FILTRASORB®, Calgon Carbon Corporation, Pittsburgh, Pa.
[12]Custom Reactivated FILTRASORB®, Calgon Carbon Corporation, Pittsburgh, Pa.
[13]TN5, Calgon Carbon Corporation, Pittsburgh, Pa.
[14]OLC 12 × 40, Calgon Carbon Corporation, Pittsburgh, Pa.
[15]Red Devil ball mill, RADIA, Plymouth, Minn.
[16]LS13320, Beckman Coulter, Brea, Calif.
[17]Milli-Q®, MilliporeSigma, Burlington, Mass.
[18]Sigma-Aldrich, St. Louis, Mo.
[19]Trizma®, Sigma-Aldrich, St. Louis, Mo.
[20]Eurofins Eaton Analytical, Monrovia, Calif.

ABOUT THE AUTHORS



James D. McNamara *(to whom correspondence may be addressed) can be contacted at jmcnam5001@aol.com. He holds a BS degree in chemistry from Wayland Baptist College, Plainview, Tex., and an MS degree in forensic chemistry from the University of Pittsburgh, Pittsburgh, Pa. He worked for Calgon Carbon Corporation for 36 years, evaluating activated carbon performance for a wide range of liquid- and gas-phase applications, and retired in 2017.* ***Ralph Franco*** *is director of municipal products,* ***Richard Mimna*** *is director of business innovation, and* ***Leo Zappa*** *is director of sales (Municipal Business Unit) at Calgon Carbon Corporation, Moon Township, Pa.*

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Comparison of Activated Carbons for Removal of Perfluorinated Compounds From Drinking Water

James D. McNamara, Ralph Franco, Richard Mimna, Leo Zappa

Pages: E2-E14 | First Published: 06 February 2018

Abstract | **Full text** | **PDF** | **References** | **Request permissions**

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Laurence B. Winn

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Comparison of Activated Carbons for Removal of Perfluorinated Compounds From Drinking Water

McNamara, James D. ; Franco, Ralph ; Mimna, Richard ; Zappa, Leo

Journal - American Water Works Association, 2018-01, Vol.110 (1), p.E2-E14

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Title	Comparison of Activated Carbons for Removal of Perfluorinated Compounds From Drinking Water
Creator	McNamara, James D. > Franco, Ralph > Mimna, Richard > Zappa, Leo >
Is Part Of	

EX-1040
Norit Americas, Inc. v. Calgon Carbon Corporation
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Subject	Journal - American Water Works Association, 2018-01, Vol.110 (1), p.E2-E14 Carbon, Activated › Fluorocarbons › Perfluorooctane sulfonate › Perfluorooctanoic acid › Water utilities ›
Keywords	granular activated carbon ; perfluorinated compounds
Description	This article describes the challenge of treating drinking waters contaminated by perfluorinated compounds, especially perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS). A goal of the study was to establish the relative effectiveness for perfluorinated compound removal by the two primary types of granular activated carbons (GACs) commonly used in the treatment of municipal drinking water: bituminous coal-based re-agglomerated GAC and coconut-based direct activated GAC. The effectiveness of GACs in removing PFOA and PFOS to nondetectable levels is demonstrated through the use of rapid small-scale column testing. Results demonstrate that bituminous coal-based re-agglomerated carbons provide considerably greater removal capacity of the targeted compounds compared with the coconut-based direct activated carbon. In support of these findings, summaries of additional third-party test work and field installations are cited. In addition, the authors provide an overview of reactivation of activated carbon to improve the economics of the technology.
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Journal AWWA, Vol. 110, No. 1 (January 2018), pp. E2-E14 (13 pages)

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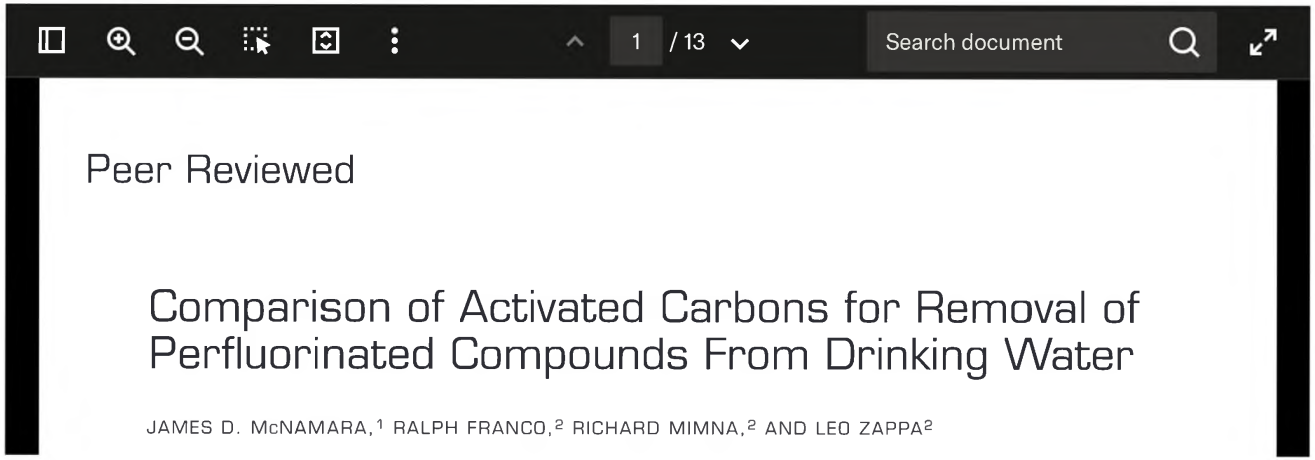
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Comparison of Activated Carbons for Removal of Perfluorinated Compounds From Drinking Water

JAMES D. McNAMARA,[1] RALPH FRANCO,[2] RICHARD MIMNA,[2] AND LEO ZAPPA[2]

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Creator	American Water Works Association. ›
Subject	Water-supply engineering -- Periodicals. ›
MESH subjects	Water Supply ›
Genre	Periodicals › periodicals ›
Other title	Journal AWWA
Related titles	Earlier title : Journal of the American Water Works Association. › Earlier title : Journal of the American Water Works Association › Available in other form: Journal (American Water Works Association) › Available in other form: Journal (American Water Works Association) › Has supplement : AWWA ... buyers' guide and publications catalog (American Water Works Association) › Has supplement : Buyers' guide (American Water Works Association) › Has supplement : Buyers' guide and publications catalog (American Water Works Association) ›
Publisher	New York, N.Y. : The Association Vol. 40, no. 1 (Jan. 1948)-
Date	c1948-
Format	v. : ill. ; 23-28 cm.
Frequency	Monthly
Identifier	LC : 86641260 ISSN : 0003-150X
Notes	Chemical abstracts 0009-2258 Annual special no. published separately with title: Buyers' guide (American Water Works Association), <1980- >; AWWA ... buyers' guide and publications catalog, <1985- >; Buyers' guide and publications catalog (American Water Works Association), <1989- >.
General Notes	Title from cover. Imprint varies: Denver, CO, <Aug. 1985- >
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580    ##$aAnnual special no. published separately with title:
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Comparison of Activated Carbons for Removal of Perfluorinated Compounds From Drinking Water

McNamara, James D. ; Franco, Ralph ; Mimna, Richard ; Zappa, Leo | Chichester, UK: John Wiley & Sons, Ltd | This article describes the challenge of treating drinking waters contaminated by perfluorinated compounds, especially perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS). A goal of the study was to establish the relative effectiveness for perfluorinated compound removal by the two primary types of granular activated carbons (GACs) commonly used in the treatment of municipal drinking water: bituminous coal-based re-agglomerated GAC and coconut-based direct activated GAC. The effectiveness of GACs in removing PFOA and PFOS to nondetectable levels is demonstrated through the use of rapid small-scale column testing. Results demonstrate that bituminous coal-based re-agglomerated carbons provide considerably greater removal capacity of the targeted compounds compared with the coconut-based direct activated carbon. In support of these findings, summaries of additional third-party test work and field installations are cited. In addition, the authors provide an overview of reactivation of activated carbon to improve the economics of the technology.
Journal - American Water Works Association, 2018-01, Vol.110 (1), p.E2-E14

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Title	Comparison of Activated Carbons for Removal of Perfluorinated Compounds From Drinking Water
Creator	McNamara, James D. › Franco, Ralph › Mimna, Richard › Zappa, Leo ›
Is Part Of	Journal - American Water Works Association, 2018-01, Vol.110 (1), p.E2-E14 ›
Subject	Carbon, Activated › Fluorocarbons › Perfluorooctane sulfonate › Perfluorooctanoic acid ›

Description	Water utilities › This article describes the challenge of treating drinking waters contaminated by perfluorinated compounds, especially perfluorooctanoic acid (PFOA) and perfluorooctane sulfonate (PFOS). A goal of the study was to establish the relative effectiveness for perfluorinated compound removal by the two primary types of granular activated carbons (GACs) commonly used in the treatment of municipal drinking water: bituminous coal-based re-agglomerated GAC and coconut-based direct activated GAC. The effectiveness of GACs in removing PFOA and PFOS to nondetectable levels is demonstrated through the use of rapid small-scale column testing. Results demonstrate that bituminous coal-based re-agglomerated carbons provide considerably greater removal capacity of the targeted compounds compared with the coconut-based direct activated carbon. In support of these findings, summaries of additional third-party test work and field installations are cited. In addition, the authors provide an overview of reactivation of activated carbon to improve the economics of the technology.
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Timothy Appleman
Water research.2013, Vol. 51, p. 246-55

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Nanofiltration and granular activated carbon treatment of perfluoroalkyl acids.
Christopher Higgins
Journal of hazardous materials.2013, Vol. 260, p. 740-6

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Rg Higgins
Environmental science & technology.2006, Vol. 40(23), p. 7251-7256

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Reactivation Services Frequently Asked Questions:

How is spent carbon reactivated?

Spent carbon is reactivated in the same manner that it is manufactured. In multiple hearth furnaces or rotary kilns, the spent carbon is heated to temperatures above 1,700°F utilizing steam as a selective oxidant. Adsorbed organics are either volatilized or pyrolysed to a carbon char. The high- temperature steam reaction serves to develop the pore volume of the activated carbon and restore its adsorptive capacity.

Reactivation Services Frequently Asked Questions:

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How are the emissions from the reactivation process treated?

Reactivation furnaces are maintained under negative pressure ensuring there will be no leaks to the outside environment. The furnace off-gas is passed through an afterburner to destroy any unburned organics that remain. The emission stream is then passed through a chemical scrubber to remove any acidic gases that may have formed. A final treatment is through baghouse filters that remove any particulate matter that remains in the stream.

Will reactivated carbon work for my application?

In most applications, reactivated carbon will perform nearly as well as virgin carbon. Calgon Carbon can provide an assessment of the standard reactivated carbon products against virgin carbon for most applications using computer modeling programs. Details can be submitted by clicking here to complete an Application Questionnaire. If required, laboratory evaluation by accelerated column testing or pilot scale trials can be conducted.

What is the advantage of reactivating spent carbon?

The reactivation process recycles the spent carbon for reuse. Adsorbed organics are destroyed and the activated carbon's adsorptive capacity is restored. The cost and long-term liability of disposal are avoided. Reactivated carbon also represents a cost savings over virgin carbons. The adsorptive capacity of the spent carbon can be restored at a lower cost than the manufacture of activated carbon from raw material.

Is reactivated carbon suitable for use in drinking water?

Spent carbons from drinking water and other food grade applications can be reactivated for reuse in their prior applications if these carbons are segregated for custom reactivation at a food grade facility. Reactivated carbon from industrial applications or reactivated at a non-food grade operation is classified as non-food grade as a general practice.

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If spent carbon is returned, can it be replaced with virgin carbon?

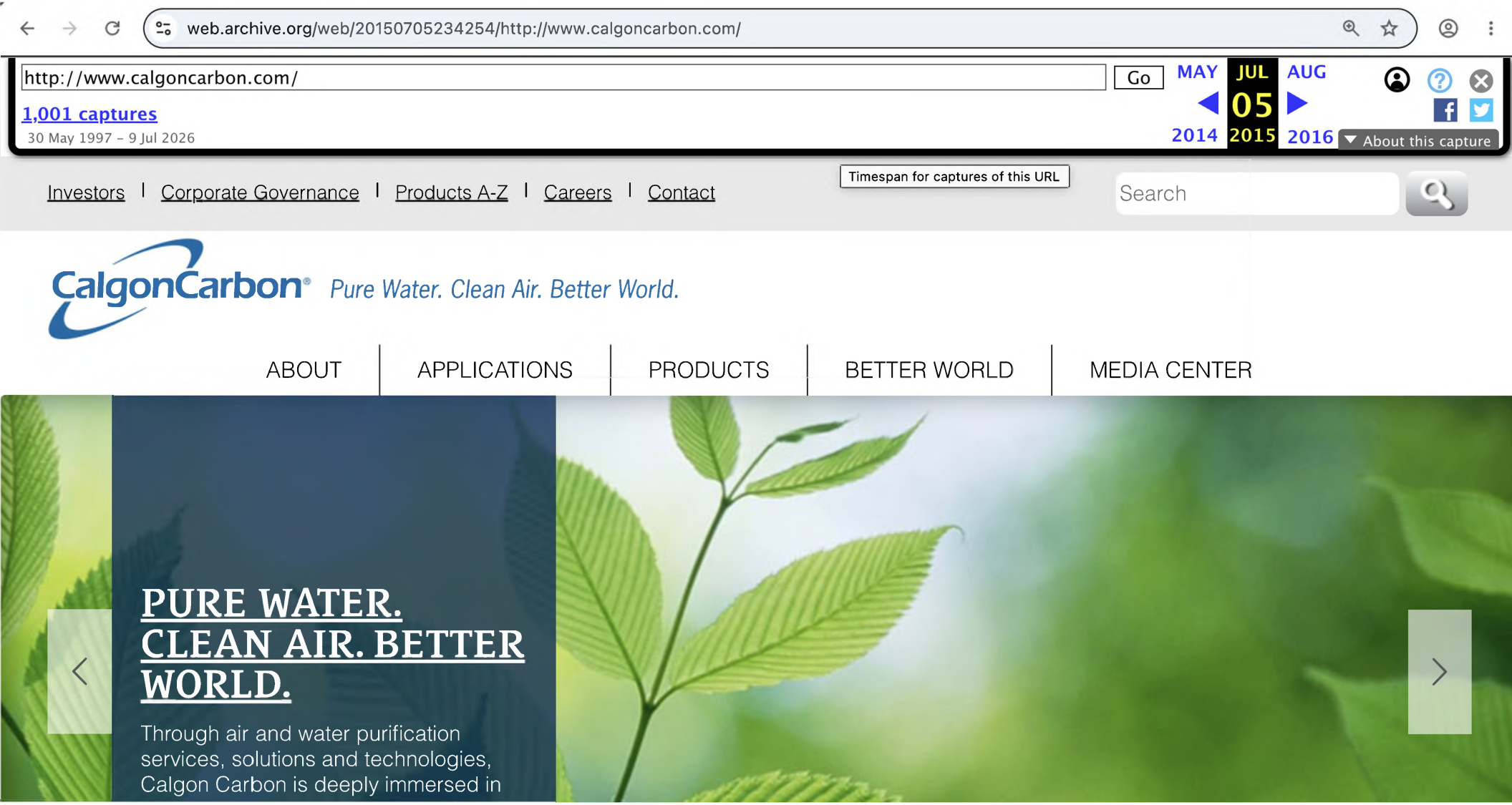
Yes, Calgon Carbon will reactivate the spent carbon and supply this later to a non-food grade application. The spent carbon can be replaced with a variety of virgin carbons that would suit the needs of the application.

Can hazardous spent carbons be returned for reactivation?

Yes, in most cases. The Catlettsburg, KY and Pittsburgh, PA plants are fully permitted to accept RCRA hazardous wastes. There are a few RCRA codes such as some characteristic hazards that are unacceptable by permit. Some compounds such as PCB's are also not approved for return.

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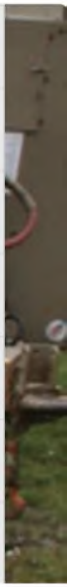
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REACTIVATION SERVICES

Calgon Carbon Corporation is an environmentally conscious company - sustainability has vital implications in what we do every single day.

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REACTIVATION SERVICES

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Calgon Carbon Corporation is an environmentally conscious company - sustainability has vital implications in what we do every single day. Recycling and reactivation are important parts of those initiatives. In fact, we have been recycling for years by reactivating spent carbon.

Features & Benefits:

After an activated carbon's adsorptive capacity has been exhausted, it can be returned to Calgon Carbon for thermal reactivation. With high temperature reactivation followed by off-gas treatment, the adsorbed organic compounds are destroyed and reactivated carbon can be safely and cost-effectively recycled back to facilities for continued use. A number of important steps are involved in the reactivation process:

1. Spent activated carbon is heated in furnaces devoid of oxygen using steam as a selective oxidant.
2. Adsorbed organics are either volatilized from the activated carbon or pyrolysed to a carbon char.
3. Volatilized organics are destroyed in the furnace's afterburner.

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4. Acid gases are removed by means of a chemical scrubber.
5. The high-temperature reaction with steam serves to restore the adsorptive capacity of the activated carbon.

Through reactivation, the spent granular activated carbon can be recycled for reuse, virtually eliminating the costs and long-term liability associated with disposal.

A significant benefit of reactivation and recycling is that the CO2 footprint associated with reactivation is significantly lower than that associated with the production, supply and use of virgin activated carbon. With carbon reactivation facilities in the United States, Europe and Asia, Calgon Carbon makes it easy to help your company meet sustainability goals. Reactivated carbon is also an efficient, cost-effective alternative to virgin where appropriate.

Calgon Carbon Reactivation Services include the reactivation of spent granular activated carbon on a custom basis or a pool basis. Customers in the municipal drinking water, food application or industrial sectors can have their spent carbon reactivated with the resultant product being returned to them (Custom Reactivation). Alternatively, customers may put their spent carbon into a generic pool for reactivation, making this a cost-effective product alternative when getting your own material returned is not required or a cost-effective recycling option as opposed to disposal when virgin material is required.

Calgon Carbon's custom municipal reactivation (CMR) products, custom food grade reactivation (CFR) products and custom industrial reactivation (CIR) products are put through the same quality testing system as the original virgin materials.

Reactivation Services Frequently Asked Questions

Carbon Acceptance Frequently Asked Questions

More Information on Spent Carbon Acceptance

Adsorbate Profile Document

Adsorbate Profile Document Instructions

Manifest Information and the EPA's summary of the Hazardous Waste Manifest System

Land Disposal Restriction Form Information and Land Disposal Notification & Certification Form

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Applications:



MUNICIPAL WATER TREATMENT



ENVIRONMENTAL WATER TREATMENT



ENVIRONMENTAL AIR TREATMENT



INDUSTRIAL PROCESSES



FOOD & BEVERAGE



MEDICAL

PRODUCTS: REACTIVATION SERVICES

+ MUNICIPAL WATER TREATMENT
+ ENVIRONMENTAL WATER TREATMENT
+ ENVIRONMENTAL AIR TREATMENT
+ INDUSTRIAL PROCESSES
+ FOOD AND BEVERAGE
+ MEDICAL

HYDE MARINE CALGON CARBON JAPAN CHEMVIRON

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About this capture

CalgonCarbon®
Pure Water. Clean Air.
Better World.

Reactivation Services Frequently Asked Questions:

How is spent carbon reactivated?

Spent carbon is reactivated in the same manner that it is manufactured. In multiple hearth furnaces or rotary kilns, the spent carbon is heated to temperatures above 1,700°F utilizing steam as a selective oxidant. Adsorbed organics are either volatilized or pyrolysed to a carbon char. The high- temperature steam reaction serves to develop the pore volume of the activated carbon and restore its adsorptive capacity.