

The removal of short-chain and long-chain perfluoroalkyl acids and sulfonates via granular activated carbons: A comparative column study

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Abstract

Per- and polyfluoroalkyl substances (PFAS) are contaminants of emerging concern. Granular activated carbon (GAC) is an adsorbent currently in use in removing long-chain PFAS from water. However, there are few studies that examine short-chain PFAS removal with GAC. The present Rapid Small-Scale Column Test study compares the removal of a suite of both long- and short-chain PFAS from groundwater. The results show that re-agglomerated, bituminous coal-based GACs, whether virgin or reactivated, are capable of effectively removing long- and short-chain PFAS with four or more perfluorinated carbons. It was also observed that perfluorobutanoic acid, a perfluorinated carboxylic acid with just three perfluorinated carbons, reached breakthrough far more quickly than all other PFAS in this study. Coconut-based and pool reactivated carbons, respectively, lasted 9 to 18% as long, and 43 to 57% as long, as the coal-based carbons studied prior to breakthrough of the PFAS. The results indicate that properly selected GACs (reagglomerated bituminous coal-based, virgin or reactivated), are a viable treatment option for PFAS contamination in water for all but the shortest perfluorinated carboxylic acids.

1 | INTRODUCTION

1.1 | Background on per- and polyfluoroalkyl substances

Per- and polyfluoroalkyl substances (PFAS) are a class of man-made chemical compounds known to be resistant to heat, water, and oils and, as a result, are frequently incorporated into a variety of industrial and commercial products such as nonstick cookware, stain-resistant clothing, food packaging, and firefighting foams. While the unique properties of these substances provide tremendous benefit, these same properties create remedial challenges because PFAS are highly persistent in the environment and resistant to degradation (Organisation for Economic Co-operation and Development [OECD], 2013).

PFAS contain fluorine atoms bonded to a carbon atom chain; the carbon chain length and functional head group vary depending on the desired chemical properties. The carbon-fluorine bond is the shortest and strongest covalent chemical bond found in nature and is the basis for the unique and beneficial chemical properties of PFAS. The length and functional head group of PFAS can also impact the level of difficulty to remediate them. While thousands of different compounds of PFAS exist, most of the regulatory focus to date has been on

two “long-chain” perfluorinated compounds—perfluorooctanoic acid (PFOA) and perfluorooctane sulfonic acid (PFOS)—because of the long time and widespread use of these two compounds coupled with the well-established knowledge of their harmful toxicological effects. PFAS of shorter chain lengths than PFOA and PFOS have generally received less attention, but the recent and sudden awareness of the presence of these compounds in drinking water and wastewater sources has generated interest in understanding both their possible toxicological effects and potential remediation approaches. By convention, short-chain perfluoroalkyl carboxylic acids are those having fewer than seven perfluorinated carbon atoms (i.e., $C_nF_{2n+1}COOH$, $n < 7$) and short-chain perfluoroalkyl sulfonic acids are those having fewer than six perfluorinated carbon atoms (i.e., $C_nF_{2n+1}SO_3H$, $n < 6$; Buck et al., 2011).

1.2 | PFAS regulatory activity

In May 2016, the U.S. Environmental Protection Agency (USEPA) revised the provisional lifetime health advisories for PFOA and PFOS in drinking water from 400 nanograms per liter (ng/L) and 200 ng/L, respectively, to a combined concentration of 70 ng/L (USEPA, 2016a). While health advisories are nonenforceable and nonregulatory, the

intent is to provide guidance to public water system operators on the potential adverse health effects resulting from exposure to toxic chemicals. Peer-reviewed studies indicate that PFOA and PFOS exposure may result in cancer (testicular, kidney, etc.), birth defects, and hormone disruption, among other health related issues (Agency for Toxic Substances & Disease Registry [ATSDR], 2015; Lau, 2012).

To eliminate the possibility of drinking water contamination, many parties responsible for sites contaminated with PFAS are pursuing remediation efforts to eliminate future liability.

To ensure that the public is protected, an increasing number of state regulatory agencies have instituted or proposed regulatory limits for PFOA and PFOS at or below the USEPA's health advisory and several of those have included guidelines on short-chain PFAS as well. Moreover, many public drinking water systems are focused on voluntarily treating to nondetect levels of PFOA and PFOS and are increasingly concerned about other PFAS chemicals. To eliminate the possibility of drinking water contamination, many parties responsible for sites contaminated with PFAS are pursuing remediation efforts to eliminate future liability. This includes, for example, producers or past producers of PFAS working to remediate the soil and associated groundwater to avoid potential lawsuits from groundwater consumers or municipal water authorities.

By now in the United States, the manufacture of PFOA and PFOS has been voluntarily phased out beginning in 2006 and replaced with new PFAS compounds, many of which are shorter chain length versions of PFOA and PFOS (Renner, 2006; Ritter, 2010). There is comparatively less information available on the environmental and toxicological impacts of other PFAS compounds. While there are indications that shorter chain PFAS are less bioaccumulative (with the exception of perfluorohexane sulfonic acid [PFHxS] and less toxic than the longer chain compounds (ATSDR, 2015; Conder, Hoke, De Wolf, Russell, & Buck, 2008; Danish Environmental Protection Agency [Danish EPA], 2015), questions and concerns about their overall safety remain (Birnbaum & Grandjean, 2015; Wang, Cousins, Scheringer, & Hungerbuehler, 2015).

1.3 | Sources of exposure and impacted population

PFAS contamination comes from a variety of pathways with the most prevalent being groundwater contamination from aqueous film-forming foams (AFFFs) and wastewater contamination from manufacturers or users (Hu et al., 2016). AFFFs have been used in thousands

of emergency and training sites around the world including air force bases, other military sites, fire training centers, and airports. Releases of foam, whether from intended use or spills, cause the foam to seep into the soil and leach PFAS, thereby contaminating the groundwater or, in the case of surface water, storm water runoff can contaminate the source water. Because of the highly stable and migratory nature of the compounds, PFAS readily migrate from the source site in groundwater or surface water and, have been shown to have contaminated groundwater in residential areas and public drinking water systems.

Conventional water treatment processes such as coagulation/flocculation, filtration, ultraviolet oxidation, and chemical disinfection generally do not remove PFAS, resulting in contamination in the discharge water. Additionally, sludge from wastewater plants may contain PFAS, which could leach into the groundwater or surface water (Dickenson & Higgins, 2016).

1.4 | Available remediation technologies

Many public drinking water systems, private well owners, military bases, and manufacturers are pursuing remediation programs to ensure that the public has clean and safe drinking water and to eliminate future liability associated with PFAS contamination. The USEPA cites several options to reduce PFOA and PFOS concentration including changing blending ratios, providing bottled water, and closing contaminated wells (USEPA, 2016b). Bottled water will temporarily alleviate the problem, but it is also extremely expensive and not sustainable in the long term.

Well closure and blending can be quick and low cost solutions but may prove temporary since existing PFAS contamination remains in place and can eventually migrate to other nearby wells.

The USEPA identifies granular activated carbon (GAC) and reverse osmosis (RO) among viable remediation technologies for the removal of PFAS from source water (USEPA, 2018). The use of GAC for PFOA and PFOS removal has been practiced for over 15 years and is generally considered to be a proven and cost-effective option for the removal of PFOA and PFOS (Dickenson & Higgins, 2016). High pressure membrane technology (i.e., RO) is also an effective option for PFAS treatment. However, to date, RO technology is not widely applied for PFAS removal, largely because of the significantly higher capital and operating costs compared to GAC (Guo, Englehardt, & Wu, 2014). The RO process also results in a concentrated waste that needs to be disposed of in a safe manner. Anion exchange resins are also an option for the removal of PFAS. The drawbacks of this technology are principally the higher material cost and the requirement to incinerate the spent material when run in single use mode. The advantage is consistent performance independent of the PFAS chain length. The increased interest in ion exchange resins is due in large part to the limited data in the public domain on the efficacy of GAC for shorter chain PFAS.

1.5 | GAC for PFAS removal

GAC is a highly porous material used to adsorb harmful contaminants from both liquid and vapor streams. It is engineered to provide certain

physical properties, such as activity and pore size distribution, to effect changes in performance. Any carbonaceous material can be utilized for the manufacture of GAC, but the most common raw material sources are coal (bituminous, subbituminous, lignite), coconut, and wood. These raw materials are converted into activated carbon through high temperature steam activation or chemical activation. The raw material and activation methodology greatly influences the physical properties of the activated carbon product.

The performance of a GAC product will depend on the physical properties of the material (e.g., pore size distribution); the PFAS present, concentrations, and physical and chemical properties; the treatment objective (i.e., what contaminant is being removed to what concentration); and background source water properties (e.g., levels of natural organic matter). It is imperative to conduct performance testing to determine the best GAC media for a given application. Rapid Small-Scale Column Testing (RSSCT) is frequently utilized to compare the efficacy of various activated carbon products.

Despite the 15-year history of using GAC for removing PFOA, PFOS, and other perfluorinated compounds, published data are largely lacking on the efficacy of GAC for shorter chain PFAS compounds. The lack of knowledge and research is likely attributable to the relative lack of regulatory scrutiny for the shorter chain PFAS compounds.

1.6 | Short-chain PFAS study

The study detailed herein utilized RSSCT to determine the effectiveness of GAC for a suite of PFAS compounds (both short and long chain) and to compare the performance of different types of GAC media in the presence of typical total organic carbon (TOC) concentrations in groundwater. The PFAS chosen for this study were perfluorobutanoic acid (PFBA), perfluorobutane sulfonic acid (PFBS), perfluorohexanoic acid (PFHxA), PFHxS, PFOA, and PFOS. The former three are considered short-chain PFAS, while the latter three are considered long-chain PFAS.

2 | MATERIALS AND METHODS

2.1 | Background on RSSCT

RSSCT simulates a large scale, fixed GAC bed by using a miniature carbon-filled column to rapidly simulate the adsorption breakthrough curve. This technique has been shown to accurately simulate the carbon treatment of a range of waters and wastewaters under various conditions (Crittenden, Berrigan, & Hand, 1986). The principal advantage of the RSSCT procedure compared to a pilot-scale (e.g., 1-inch diameter) column adsorption test is its increased speed. Typically, an RSSCT can be completed in less than 1 to 15% of the time required for a 1-in. diameter study. The reduction in time needed for simulation is due to the much smaller particle sizes used in these studies, which greatly speeds up the kinetics of adsorption. At completion of the RSSCT study discussed next, the tests ran for ~100,000 bed volumes (BV), which simulated ~700 days of operation and the treatment of ~500 million gallons of water. The RSSCTs assumed constant

EXHIBIT 1 Description of carbons used for the study

GAC	Description	Supplier
Filtrisorb 400 (F400)	Virgin, re-agglomerated bituminous coal, 12 × 40 mesh	Calgon Carbon
OLC 12 × 40	Virgin, direct activated, coconut, 12 × 40 mesh	Calgon Carbon
CMR	Dedicated reactivated Filtrisorb 400, 12 × 40 mesh	Calgon Carbon
DSR	Pool reactivated, heterogeneous, 8 × 30 mesh	Calgon Carbon

diffusivity for different-sized carbons, and simulated a 10-foot diameter vessel with an empty-bed contact time (EBCT) of 15 min.

2.2 | Carbon preparation

The RSSCTs were conducted using four different GACs, including re-agglomerated bituminous coal-based carbon (Filtrisorb 400); coconut-based carbon (OLC 12 × 40); and two types of reactivated carbon, one from dedicated reactivation of Filtrisorb 400 from a water treatment plant (CMR) and the other from a variable and heterogeneous pool (DSR). These four GACs are manufactured by Calgon Carbon Corporation (Pittsburgh, Pennsylvania).

Dedicated, or custom, reactivation occurs when a particular site sends its spent carbon to be reactivated and, after the high-temperature reactivation process removes the contaminants, the same batch of carbon is returned to the site for further use. Pool reactivation occurs when spent carbons from a variety of sites are pooled together, reactivated, and used in appropriate applications (i.e., wastewater treatment and remediation). Exhibit 1 provides an overview of the four types of carbons studied.

Current production samples of each carbon were systematically resized to approximately 100 × 200 mesh using a Red Devil 1410 ball mill. The original carbon production samples were analyzed for apparent density (ASTM D2854), total ash (ASTM D2866), iodine number (ASTM D4607), and particle size distribution/screens (ASTM D2862). Exhibit 2 presents the physical characterization data for the various carbons.

The mean particle diameters of the sized carbons were determined using a Beckman Coulter LS13320 Particle Size Analyzer Brea, California, which records the particle size spectra by laser diffraction between 0.375 microns (μm) and 2,000 μm . The test carbons were oven-dried at 105°C for 16 hr, then allowed to cool in a desiccator until placed in a column. The dry, sized carbons were installed in the test columns. Prior to the introduction of the feed water, each carbon column was pre-wetted with ASTM Type II water for approximately 16 hr, in an upflow direction, at a flow rate comparable to that used in the test runs (a proven procedure that is effective at degassing the small-scale column carbon bed over many years of practicing the method). The column tests simulated a GAC system providing 15 min EBCT. Each test simulated the same EBCT by ensuring that the modeled flow rates and carbon volumes were identical. As a result,

EXHIBIT 2 Activated carbon characterization data

	Filtrisorb 400	OLC 12 × 40	CMR	DSR 8 × 30
Apparent density (g/mL)	0.561	0.539	0.527	0.557
Ash (%)	7.8	2.1	9.9	8.1
Iodine number (mg/g)	1,000	1,149	932	800
Particle size distribution (percent on each U.S. mesh screen size)	12 × 40	12 × 40	12 × 40	8 × 30
8				3.5
10				6.1
12	0.6	0.4	0.1	10.3
16	17.3	19.6	13.5	33.3
20	34.0	41.6	39.4	24.1
30	15.8	26.8	36.2	15.2
−30				7.5
40	3.0	9.3	10.2	
−40	1.1	2.3	0.6	

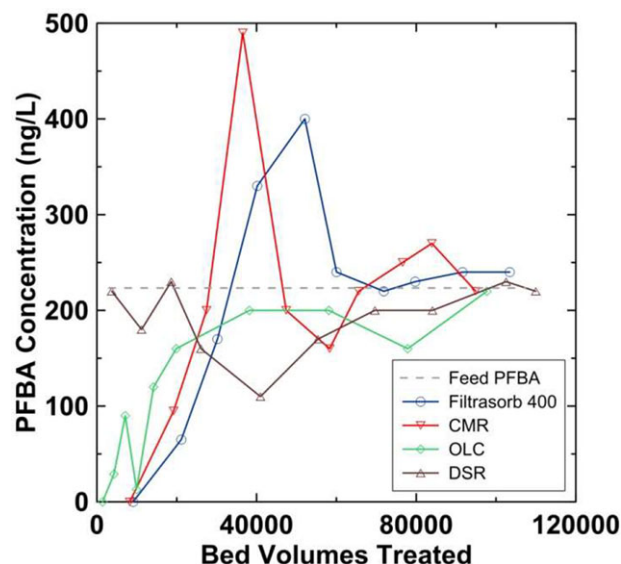
EXHIBIT 3 Water quality data

Test	Units	Result
TSS (total suspended solids)	mg/L	<0.50
TDS (total dissolved solids)	mg/L	670
TOC (total organic carbon)	mg/L	0.16
Conductivity	μS/cm	916
pH	N/A	7.98
Iron (Fe total)	mg/L	<0.02
Hardness (total)	mg/L (as CaCO ₃)	460
Alkalinity (methyl orange)	mg/L (as CaCO ₃)	140
Common anions (IC)		
Fluoride (F [−])	mg/L	0.11
Chloride (Cl [−])	mg/L	28.3
Bromide (Br [−])	mg/L	<0.20
Nitrate (as NO ₃ [−])	mg/L	4.24
Phosphate (PO ₄ ^{3−})	mg/L	<0.50
Sulfate (SO ₄ ^{2−})	mg/L	326

due to differences in carbon densities and original particle size, the weights of the carbons simulated varied slightly.

2.3 | Influent water quality

Untreated groundwater from the Warrington Water District located in Pennsylvania was used for the study. Standard water quality parameters of the water sample prior to the addition of PFAS are shown in Exhibit 3. Total suspended solids and total dissolved solids were analyzed by Test America Laboratories, Inc., Pittsburgh, Pennsylvania; the remaining parameters were measured in Calgon Carbon Corporation's analytical laboratory. The groundwater was dosed to establish

**EXHIBIT 4** Removal of perfluorobutanoic acid using granular activated carbon at 15 min empty-bed contact time

a concentration of 200 ng/L of each of the PFAS compounds studied (accomplished by creating 100 mg/L stock solutions made of deionized water). The individual neat PFAS compounds were purchased from Sigma-Aldrich (St. Louis, Missouri). Depending upon the volume of the feed to be prepared, a measured volume of the stock solution was added to the water to achieve the desired concentration.

2.4 | RSSCT sampling and analytical

Effluent samples were collected via automated sample collectors. Column effluent samples designated for PFAS analyses were collected in high-density polyethylene bottles containing Trizma[®] preservative (St. Louis, Missouri) and shipped on ice (overnight) to the Eurofins Eaton Analytical (EEA) laboratory located in Monrovia, California. A chain of custody identifying the samples accompanied the shipment. These steps were taken to ensure no PFAS contamination from external sources and there was no evaporative loss throughout sample transport.

Flow rates of the RSSCTs were closely monitored throughout the study. Composite samples of the RSSCT effluent, minus discrete samples collected for PFAS analysis, were measured on a daily basis. From these data, adjustments to flow rate were made and an overall average flow rate was calculated for each column.

The column effluent and feed samples were submitted to EEA for PFAS analysis by USEPA Method 537 (USEPA, 2009). Effluent samples were also measured for TOC, but no values above the 0.15 mg/L detection limit were observed.

3 | RESULTS AND DISCUSSION

3.1 | Results

Breakthrough curves of the four carbons tested for each individual PFAS tested are shown in Exhibits 4–9. Each graph

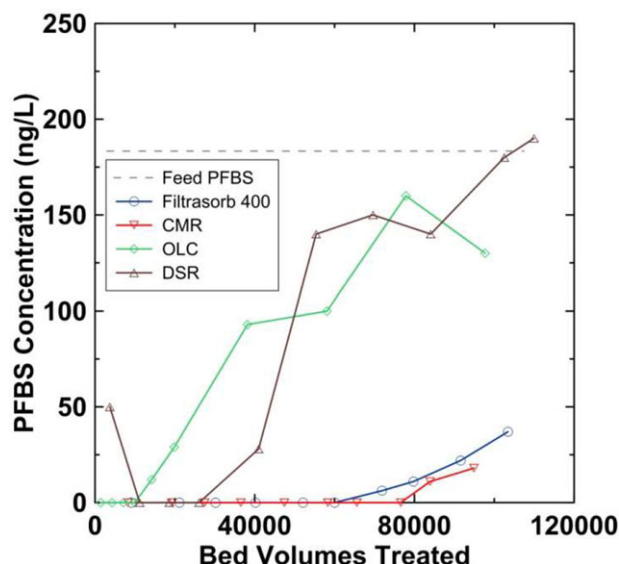


EXHIBIT 5 Removal of perfluorobutane sulfonic acid using granular activated carbon at 15 min empty-bed contact time

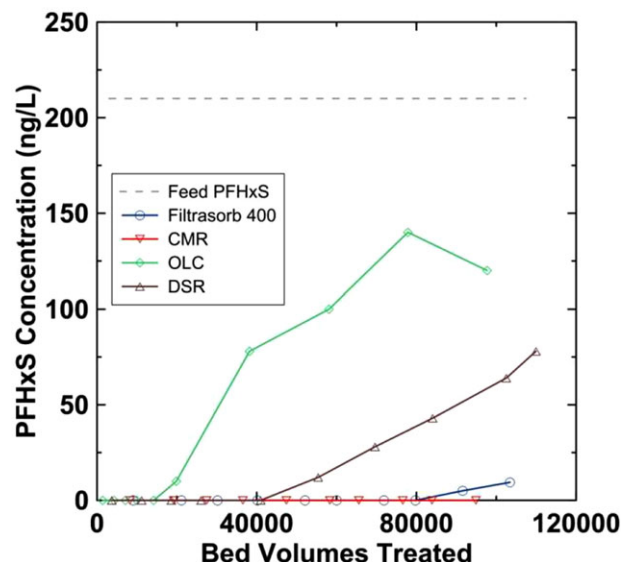


EXHIBIT 7 Removal of perfluorohexane sulfonic acid using granular activated carbon at 15 min empty-bed contact time

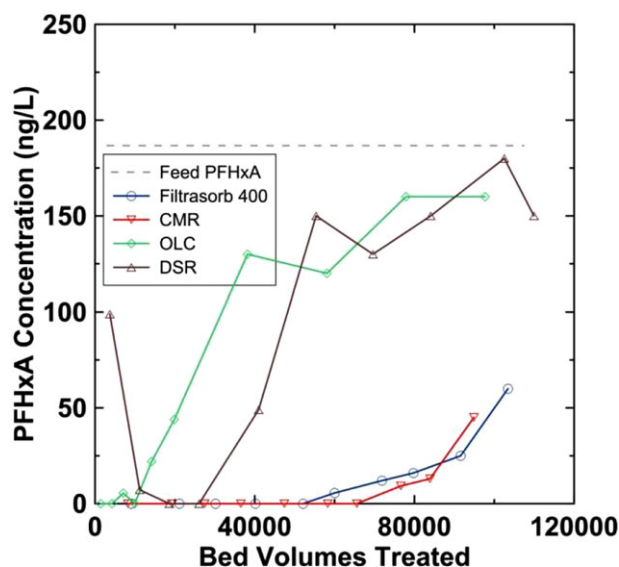


EXHIBIT 6 Removal of perfluorohexanoic acid using granular activated carbon at 15 min empty-bed contact time

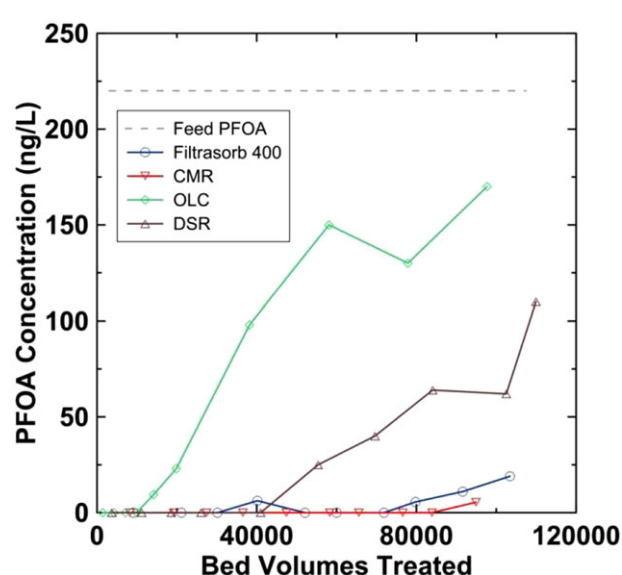
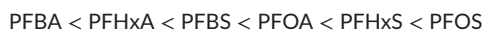


EXHIBIT 8 Removal of perfluorooctanoic acid using granular activated carbon at 15 min empty-bed contact time

illustrates the performance of the four carbons for a specific PFAS compound. The graphs display the concentrations of each PFAS compound as a function of BVs of water treated through the column. The exhibits are presented in order of molecule size: PFBA < PFBS < PFHxA < PFHxS < PFOA < PFOS. The adsorbability of the compounds differs from this order, because the sulfonic acids were more easily adsorbed than their carboxylic acid counterparts. The compounds are listed in order of adsorbability next:



The data predict that GAC can remove the short- and long-chain PFAS included in this study to below detection for nearly 54,000 BVs, or the equivalent of 373 days at a flow rate of gallons per

minute 530 gpm and nearly 285 million gallons of treatment in a full scale, single vessel system. PFBA is the exception, as it exhibited initial breakthrough after approximately 9,200 BVs or 64 days and 49 million gallons of treatment. Of the four activated carbon types tested, the re-agglomerated, bituminous coal-based GACs, both virgin (Filtrisorb 400) and dedicated reactivated (CMR), proved to have the highest removal capacities for all six PFAS compounds. Comparatively, the DSR's performance ranged between 43% and 57% of the Filtrasorb 400, while the OLC's performance ranged between 9% and 18% of the Filtrasorb 400. The poorer performance of the DSR relative to the CMR may be due to both a lower iodine value (representing less activity, i.e., fewer adsorption sites) and a larger particle size (causing slower kinetics) combined with the heterogeneity of the source material prior to reactivation, some of which is not optimized for PFAS removal. The

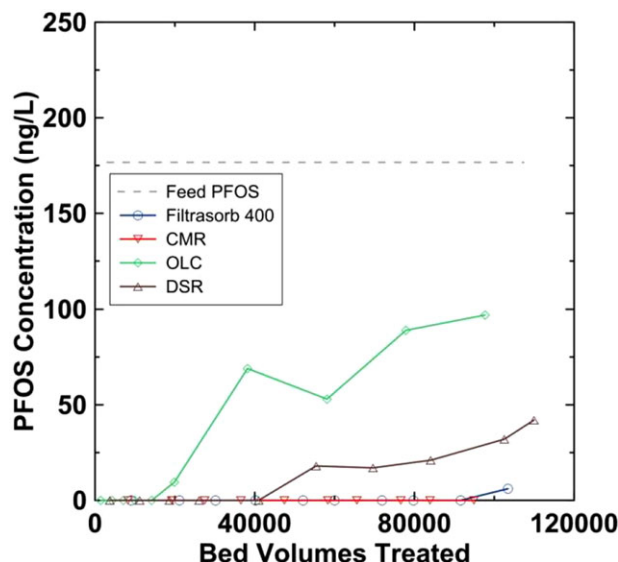


EXHIBIT 9 Removal of perfluorooctane sulfonic acid using granular activated carbon at 15 min empty-bed contact time

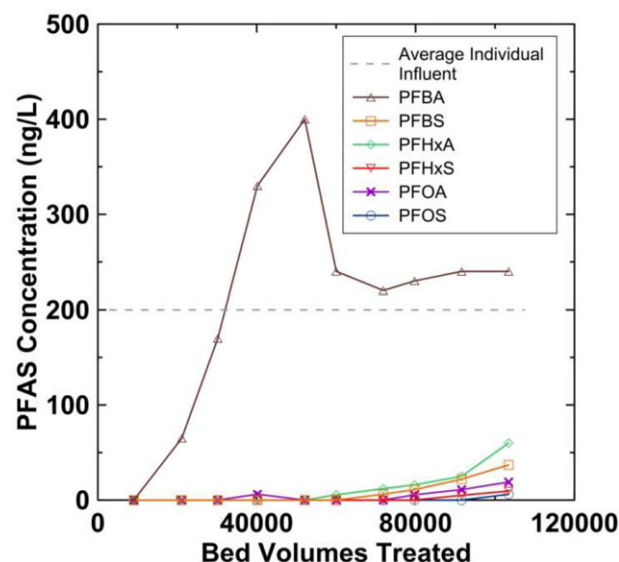


EXHIBIT 10 Removal of various PFAS using Filtrisorb 400 granular activated carbon at 15 min empty-bed contact time

poor performance of the coconut-based GAC relative to the coal-based GACs is consistent with earlier reports of comparative GAC tests with PFOA and PFOS (McNamara, Franco, Mimna, & Zappa, 2018; Qiu, Fujii, & Tanaka, 2007); its cause is not well understood.

Exhibit 10 is a summary plot of the capability of re-agglomerated bituminous coal GAC (Filtrisorb 400) for removing all six PFAS tested.

3.2 | System design

Single vessels operated in parallel and dual vessels operated in series (lead/lag) are the two operating designs typically used for treating contaminated waters with fixed bed granular carbon. In single vessel design, the carbon bed runs until the effluent contaminant concentration achieves a target treatment objective or discharge concentration. This arrangement is preferred when the contaminant adsorption has rapid kinetics, as seen in a very short mass transfer zone (MTZ). The resulting acceptable carbon utilization is due to the MTZ occupation of a very small percentage of the carbon bed. This mode of operation would also be favorable if the PFAS treatment objective equals 50% or more of the inlet PFAS concentration.

In a dual vessel system, the first vessel, called the lead vessel, typically is run until its effluent contaminant concentration achieves at

least 50% of the influent contaminant concentration, at which time it is removed from service. The second vessel in series, called the lag vessel, has been adsorbing contaminants that may not have been removed in the lead vessel during the operation of the lead bed. When the lead bed is taken out of service, the lag bed is moved into the lead position. A fresh supply of carbon is installed in the former lead vessel, which is then repositioned in the lag position. This sequence is repeated every time the lead bed effluent concentration exceeds 50% breakthrough.

Exhibits 11 and 12 summarize the capability of re-agglomerated bituminous coal GAC for removing all six PFAS tested for single vessel and dual vessel design. With the exception of PFBA removal, over 550 million gallons of water can be treated in a lead-lag system for both short- and long-chain PFAS before seeing breakthrough in the effluent. The data for a single vessel design present greater scatter and operating days are largely dependent on the chain length and functional group. Based on the length of the MTZs formed with the poorer performing carbons, it is likely the lead/lag operation will result in lower carbon use rates and lower overall costs for the stronger performing carbons. Unfortunately, the best performing carbons were still removing a large percentage of most of the PFAS at the point the study was ended, meaning their full capacities for the tested PFAS could not be computed.

EXHIBIT 11 Relative single vessel performance predictions of PFAS adsorption on Filtrisorb 400

Re-agglomerated bituminous coal GAC—Single vessel design			
PFAS	Bed volumes treated	Simulated days	Gallons treated
PFBA (1 ppt breakthrough)	9,250	64	49,200,000
PFHxA (1 ppt breakthrough)	53,500	373	285,000,000
PFBS (1 ppt breakthrough)	62,000	432	329,000,000
PFOA (1 ppt breakthrough)	73,300	511	390,000,000
PFHxS (1 ppt breakthrough)	82,200	573	437,000,000
PFOS (1 ppt breakthrough)	93,600	652	497,000,000

EXHIBIT 12 Relative dual vessel performance predictions of PFAS adsorption on Filtrasorb 400

Re-agglomerated bituminous coal GAC—Dual vessel (lead/lag) design			
PFAS	Bed volumes treated	Simulated days	Gallons treated
PFBA (@ 50% breakthrough)	19,000	130	100,000,000
PFHxA (@ 32% breakthrough)	>103,000	>721	>550,000,000
PFBS (@ 20% breakthrough)	>103,000	>721	>550,000,000
PFOA (@ 9% breakthrough)	>103,000	>721	>550,000,000
PFHxS (@ 19% breakthrough)	>103,000	>721	>550,000,000
PFOS (@ 3.5% breakthrough)	>103,000	>721	>550,000,000

4 | CONCLUSIONS

The study results show that both long- and short-chain PFAS are adsorbable onto GAC. The performance data suggest that for five out of six PFAS compounds included in this study, GAC can provide treatment for one to two years before carbon exchange is required based on the flow rates simulated and concentrations tested. The study shows a positive correlation between molecular weight and a compound's adsorbability onto GAC, within each type of PFAS studied. Additionally, the sulfonate compounds are more readily adsorbed onto GAC compared to the carboxylates. While the BVs achieved for PFBA are relatively low compared to the other compounds, GAC did exhibit adsorptive capacity for the compound.

The type of activated carbon used and system design (series vs. single vessel operation) for PFAS treatment will impact the performance and economics of a given system. The data presented in this study demonstrate that re-agglomerated, bituminous coal-based GAC product, whether virgin or dedicated reactivated, provides significantly greater capacity for PFAS compounds of all chain lengths compared to coconut-based GAC and a pool reactivated GAC.

The process of selecting any remediation technology for PFAS removal is complicated and highly dependent upon water quality, specifically the level of PFAS and background contaminants. While this study reaffirms GAC effectiveness for various PFAS compounds and chain lengths, column testing is always recommended to optimize system design, as the background contamination (e.g., naturally occurring organic matter) will impact operational life and carbon usage rates.

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How to cite this article: Westreich P, Mimna R, Brewer J, Forrester F. The removal of short-chain and long-chain perfluoroalkyl acids and sulfonates via granular activated carbons: A comparative column study. *Remediation*. 2018;29:19–26. <https://doi.org/10.1002/rem.21579>