

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

PHARAOH ENERGY SERVICES, LLC

Petitioner

v.

FLEX-CHEM HOLDING COMPANY, LLC

Patent Owner

Case No. Unassigned
Patent No. 9,944,843
Issued: April 17, 2018
Application No.: 14/686,237
Filed: April 14, 2015

Title: STIMULATION OF WELLS IN NANO-DARCY SHALE FORMATIONS

**DECLARATION OF JEFFREY H. HARWELL, PH.D., UNDER 37
C.F.R. § 1.68 IN SUPPORT OF PETITION FOR *INTER PARTES* REVIEW
OF U.S. PATENT NO. 9,944,843**

I. INTRODUCTION AND SUMMARY OF OPINIONS

1. My name is Jeffrey H. Harwell. I submit this declaration on behalf of Petitioner Pharaoh Energy Services, LLC, which I understand is challenging the validity of claims 1–13 (Challenged Claims) of U.S. Patent No. 9,944,843 (the '843 Patent) in a petition for *inter partes* review (IPR).

2. I have been asked to provide an opinion on the validity of the Challenged Claims.

3. My opinions are based, at least in part, on the following references, as well as my understanding as to the general knowledge of a person of skill in the art at the time of the earliest provisional application (i.e., April 14, 2014) to which the '843 Patent claims priority:

<u>REFERENCE</u>	<u>RELEVANT DATE</u>
United States Patent No. 6,436,880 B1 to Frenier ("Frenier") (Ex. 1004).	May 3, 2000
United States Patent No. 10,072,205 B2 to Reyes et al. ("Reyes") (Ex. 1005).	June 25, 2013

4. As detailed below, I conclude that the Challenged Claims are anticipated and/or rendered obvious by Frenier and Reyes.

5. Additionally, to support my statements regarding the knowledge and

understanding of POSITAs, I rely on the following references:

<u>REFERENCE</u>
Excerpt from Borchardt, <i>Chemicals Used in Oil Field Operations</i> , February 21, 1989 (Ex. 1015) (clean copy downloaded April 16, 2024)
Schlumberger Online Glossary, https://glossary.slb.com/en/ (Ex. 1016) (screenshots assembled April 16, 2014)

6. The '843 Patent claims nothing more than well-known methods of acid-treating or matrix acidizing a subterranean formation using a chelating or sequestering agent to increase the permeability of the formation, thus making it easier to extract hydrocarbons from the well. Frenier discloses all of the limitations found in the claims of the '843 Patent.

7. As is generally disclosed in Borchardt, "Chemicals Used in Oil Field Operations," 1989, matrix and fracture acidizing and the use of acids, chelating agents, and many other common oil field chemicals in well stimulations and oil field water treatment have been known for decades. Note especially that Borchardt is a review paper, not a research paper, and it contains all of the chemicals described in '843 Patent.

8. At a minimum, it would have been obvious for a person having ordinary skill in the art to combine the described and disclosed treatment methods and compositions described by Frenier and the similar treatment methods and

compositions described and disclosed by Reyes to form those described and claimed in the '843 Patent. A person having ordinary skill in the art would have been motivated to combine these concepts and applications because they are clearly explained in both Frenier and Reyes to be used at least for stimulating and remediating oil and gas wells.

9. Beyond this, many of the operations and chemicals described in the '843 Patent have been commonly used and applied in well treatment and well stimulation operations for at least four decades.

II. QUALIFICATIONS AND BACKGROUND

7. My qualifications are detailed in my attached curriculum vitae, attached hereto as Exhibit A.

8. I hold the position of Professor Emeritus of Chemical Engineering at the University of Oklahoma ("OU").

9. I hold a doctoral degree in chemical engineering from the University of Texas at Austin, which I obtained in May 1983.

10. I have extensive knowledge in the area of stimulating, remediating, or otherwise treating oil and gas wells, including by the methods and use of treatment compositions such as those described in the prior art and the '843 Patent.

11. During my tenure at OU, I held the AGC Chair of Chemical Engineering at the University of Oklahoma, was a founding member of the Institute

for Applied Surfactant Science (IASR) industrial consortium, and was a founder and president of Surfactant Associates, Inc., a privately held for-profit corporation which provides consulting, industrial training, and R&D support for industry.

12. Generally, my research, teaching, and consulting are in the areas of colloid chemistry, nanotechnology, surfactant science, and their applications.

13. Applications of my research include in situ ground water remediation, oil production, consumer products, colloidal gels, microemulsions, nanoparticles, and pharmaceutical formulations.

14. During my 42 years at the University of Oklahoma, I have been an author on over 200 technical publications.

15. I have a Google Scholar h-index of 61 and an i10-index of 198.

16. My consulting activities have included companies such as ExxonMobil Chemical, Janssen Pharmaceuticals, Shell Chemicals, SC Johnson, Dow Chemical Company, PPG Industries, Church & Dwight, Proctor & Gamble, Clorox, ConocoPhillips, GSC plc, Lubrizol Corporation, Baker-Hughes, SASOL North America, and Stepan Chemical, among many.

17. I am a Fellow of the US National Academy of Inventors and a Fellow of the American Institute of Chemical Engineers.

18. During my career at OU, I served as Director of the School of Chemical, Biological and Materials Engineering, as Associate Dean of Engineering,

and as the Program Director of the Interfacial, Transport, and Separation Processes Program at the National Science Foundation.

19. I have chaired the 65th ACS Colloid and Surface Science Symposium and received the Victor K. LaMer Award from the Colloid and Surface Science division of the American Chemical Society, the Outstanding Remediation Project award from the National Ground Water Association, and Outstanding Paper awards from the AIChE and the American Oil Chemists Society.

20. My current activities include working on completion of my 5th book, "Formulation Engineering with HLD/NAC," (together with co-editors E.A. Acosta and D.A. Sabatini), and industrial training, consulting, and R&D through Surfactant Associates, Inc.

21. I am a named inventor on more than thirty issued U.S. patents, which are listed in my attached C.V.

III. UNDERSTANDING OF PATENT LAW

22. I am not an attorney. I have been provided with an understanding of the patent law relevant to conducting the analysis given in this report. The following represents a summary of my understanding of these issues.

23. I understand the patent-at-issue is the '843 Patent and that prior art to the '843 Patent includes patents and printed publications in the relevant art that predate the alleged priority date of the '843 Patent, which I have been told to assume

is April 14, 2014.

24. I understand that a patent claim is “anticipated,” and thus invalid, if each and every element of a claim be disclosed expressly, implicitly, and/or inherently in a single prior art reference, arranged in the prior art reference as arranged in the claim. I have also been told that even if a reference does not expressly spell out all the limitations arranged or combined as in the claim, the reference may still anticipate the claim if a person having ordinary skill in the art (POSITA), reading the reference, would “at once envisage” the claimed arrangement or combination.

25. I understand that anticipation only requires disclosure of the claimed compound or composition, however, not its properties or use. If the claimed composition is identical to a composition disclosed by a prior art reference, it does not matter that the applicant recognized additional properties of that composition.

26. I also understand that when, as by a recitation of ranges or otherwise, a claim covers several compositions, the claim is anticipated if one of them is in the prior art. I have also been told that when the prior art discloses a range which touches or overlaps the claimed range, but no specific examples falling within the claimed range are disclosed, the reference anticipates the claims if the claimed subject matter is disclosed in the reference with "sufficient specificity.”

27. I further understand for a disclosure to be implicit or inherent it must

be a necessary feature or result of an embodiment sufficiently described and enabled in the prior art reference. (In other words, that a certain result or characteristic *may occur or be present* in the prior art reference is not enough; that certain result or characteristic must always occur (or be present) for the result or characteristic to be implicit or inherently disclosed.) However, it is not necessary that POSITA would have recognized that implicit or inherent disclosure at the time of invention.

28. I understand the effect of explicitly incorporated references. Specifically, I understand that when a reference or material is explicitly incorporated by reference into a host document, it is effectively part of the host document as if it was explicitly contained therein.

29. I understand that a patent claim is invalid as “obvious” if the differences between the prior art and the claimed invention are such that they would have been “obvious” to POSITA before the effective filing date of the claimed invention (i.e., April 14, 2014).

30. I also understand that a patent can be obvious in light of a single prior art reference if it would have been obvious to modify that reference to arrive at the patented invention.

31. I have been further informed that obviousness does not require that a single reference disclose all of the requirements of the claim. Rather, two or more references can be combined if POSITA would have combined such references. In

reaching a conclusion on whether references would have been combined by POSITAs at the time of the invention I have been informed that I may consider, among other things: 1) whether the change was merely the predictable result of using prior art elements according to their own functions, 2) whether there is some teaching or suggestion in the prior art to make the modification or combination of elements claimed in the patent, 3) whether the innovation applies unknown technique that had been used to improve a similar device or method in a similar way, and 4) whether the claimed invention would have been obvious to try, meaning that the claimed and innovation was one of a relatively small number of possible approaches to the problem with a reasonable expectation of success by those having ordinary skill in the art.

32. I further understand if a POSITA can implement a predictable variation, obviousness likely bars its patentability. For the same reason, if a technique has been used to improve one device and a POSITA would recognize that it would improve similar devices in the same way, using the technique is obvious.

33. I understand that a claim may be obvious if common sense directs one to combine multiple prior art references or add missing features to reproduce the alleged invention recited in the claims.

34. Furthermore, I also have been informed that factors, such as a long felt need for the solution provided by the claimed invention, unsuccessful attempts

by others to find the solution provider provided by the claimed invention, unexpected and superior results from the claimed invention, and other evidence tending to show obviousness or nonobviousness, are a consideration.

35. I understand that a POSITA is determined by considering: 1) the education level of the inventor; 2) the type of problems encountered in the art; 3) prior art solutions to these problems; 4) the rapidity with which innovations are made; 5) the sophistication of the technology; and 6) education level of active workers in the field.

36. I understand that Patent Owner has described a POSITA at the time of the alleged invention of the '843 Patent had at least a bachelor's degree in chemistry, chemical engineering, petroleum engineering, geology, or the equivalent, and at least two years of experience in oil and natural gas well treatments, or an equivalent amount of relevant work and/or educational experience. I considered Patent Owner's description of a POSITA in performing the analysis provided below.

37. Based on my level of education and experience, I am familiar with the level of knowledge that a POSITA would have possessed at the time of the invention, i.e., April 14, 2014, and by then, I was a POSITA.

IV. DESCRIPTION OF THE TECHNOLOGY AND OVERVIEW OF THE '843 Patent

38. The '843 Patent is entitled "STIMULATION OF WELLS IN NANO-DARCY SHALE FORMATIONS." Ex. 1001, front cover.

39. The application for the '843 Patent (U.S. Patent Application No. 14,686,237) was filed April 14, 2015. Ex. 1001, front cover.

40. I also understand that the '843 Patent relates to U.S. Provisional Patent Application No. 61/979,210, filed on April 14, 2014.

41. Therefore, the '843 Patent has a "priority date" of April 14, 2014.

42. The specification of the '843 Patent describes "formulations and methods for stimulating the production from wells in nano-darcy shale formations" generally including the following steps:

(i) providing a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent at a pH of between about 0 and 10;

(ii) injecting the treatment mixture into the well at a pressure less than a fracture pressure of the nano-darcy shale formation until at least some of the treatment mixture contacts the nano-darcy shale formation;

(iii) maintaining the treatment mixture in contact with the nano-darcy shale formation for a contact time of between about 1 minute to 100 days, thereby allowing the metal complexing agent to bind with at least some naturally-occurring metals contained within the nano-darcy shale formation; and

(iv) removing the treatment mixture from the well after the contact time, thereby removing the bound naturally-occurring metals from the non-darcy shale formation and thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method.

Ex. 1001, 2:35–53.

43. The specification of the '843 Patent further defines what chemicals may be a “metal complexing agent”:

For the purposes of this disclosure, a metal-complexing agent may be any chemical that can bind with a metal regardless of the binding mechanism and includes **sequestration agents**, reducing agents, chelating agents, ligands porphyrins, pigments, peptides, saccharides and/or nucleic acids. In some embodiments, the metal-complexing agent is a **chelating agent**, an alkali metal salt thereof, a non-alkali metal salt thereof, or any combination thereof may be included in the treatment fluids described herein.

Id. at 4:54–63 (emphasis added).

44. The most notable of these are chelating and sequestering agents because, as shown below, they have been used (and described for use) for the methods claimed in the '843 Patent prior to its earliest effective filing date.

45. The specification of the '843 Patent further states that “the well stimulation mixture can contain the metal complexing agent as well as multiple chemical additives as desired.” *Id.* at 6:26–28. Namely, the “additives may include biocide, scale inhibitor, clay control additive, oxygen scavenger and surfactant that assist fluid recovery” as well as “corrosion inhibitors,” “dispersing agents,” “a colloidal silica deposition inhibitor,” “mutual solvents,” and/or “Acid ... in order to control the pH of the treatment mixture.” *Id.* at 6:28–30; 6:42; 7:3, 7:21; 7:36; 7:49–

50.

46. These additives too were used and described for use for the claimed methods of the '843 Patent prior to April 14, 2014.

V. CLAIM CONSTRUCTIONS

47. I have been told in the related district court litigation that Patent Owner has proposed construing the various claim terms of the '843 Patent using their plain and ordinary meaning to a POSITA.

48. For purposes of the present declaration I adopt the plain and ordinary meaning in offering my opinions below.

VI. OVERVIEW OF THE PRIMARY PRIOR ART

A. Frenier

49. I was provided with a copy of United States Patent No. 6,436,880 B1 to Frenier (Ex. 1004). I understand that Frenier was provided to the patent examiner during the prosecution of the '843 Patent.

50. Frenier discloses several methods of “acid-treating a subterranean formation.” *Id.* at 4:45. Generally, the methods include “the step of injecting a well treatment fluid composition via a wellbore into a subterranean formation... at a fluid pressure that is less than the minimum in situ rock stress (i.e., a matrix acidizing method), [but] can also be performed at a higher pressure (i.e., an acid fracturing method).” *Id.* at 4:49–53.

51. Frenier explains that the practice of using treatment fluids to improve the productivity of oil wells was disclosed over a century ago. *See id.* at 1:66–2:3.

52. Frenier identifies the need for “an acid well treatment fluid that is relatively inexpensive and can readily control precipitation of iron.” *Id.* at 3:54–56. Moreover, Frenier states it would be “desirable” if an “acid well treatment fluid could lead to improved radial penetration than is commonly seen for acid well treatment fluids known in the art,” “that the acid well treatment fluid could be used in either matrix acidizing or fracture acidizing treatments,” and “for the acid well treatment fluid to be useful in control of scale, such as alkaline earth carbonate scales in a wellbore in a carbonate formation.” *Id.* at 3:57–65.

53. Frenier addresses these needs by disclosing “the use of acidic fluids in stimulation and workover operations, and in particular, in matrix acidizing or fracture acidizing treatments.” *Id.* at 4:8–12.

54. Specifically, Frenier describes an embodiment that is a “well treatment fluid composition that comprises: a first acid; water; and hydroxyethylaminocarboxylic acid,” where the “first acid can be ... a non-oxidizing mineral acid, such as hydrochloric acid, hydrofluoric acid, or a mixture thereof,” or “[a]lternatively, the acid can be a non-oxidizing organic acid, such as formic acid, acetic acid, or a mixture thereof.” *Id.* at 4:12–22.

55. Of particular relevance to this Petition, Frenier discloses another aspect

of its invention, which is a “method of acid-treating a subterranean formation,” including the “step of injecting a well treatment fluid composition via a wellbore into a subterranean formation,” where, in one embodiment, the well treatment composition comprises the components described above, *e.g.* a first acid, water, and hydroxyethylaminocarboxylic acid, or simply hydroxyethylaminocarboxylic acids **“without the use of a first acid.”** *Id.* at 4:44–49; 6:31–33 (emphasis added).

56. Frenier details that, while the “injection step is preferably performed at a fluid pressure that is less than the minimum in situ rock stress (*i.e.*, a matrix acidizing method), the method can also be performed at a higher pressure (*i.e.*, an acid fracturing method).” *Id.* at 4:49–53. Frenier further describes that “[t]he method can also be used to remove deposits from the wellbore.” *Id.* at 4:53–55. Frenier also touts as another aspect of its invention a method of removing alkaline earth carbonate scale from a wellbore. *Id.* at 4:61–62.

57. In a more detailed discussion of the preferred embodiments, Frenier discloses that “a small amount of mineral acid, an organic acid, or a base can be added to provide pH control of the composition,” and that typical concentration ranges, by percent weight, are: hydroxyethylaminocarboxylic acid (1–30%); pH controller (0.1–15%); other additives (0.1–5%); and water (50–95%). *Id.* at 6:38–45.’

58. Frenier then more specifically describes the process of using the “well

treatment fluid compositions” in a matrix acidizing treatment. *Id.* at 7:20–37.

59. Frenier describes such “matrix acidizing methods” as “generally involv[ing] pumping the acid-containing well treatment composition down the wellbore and out through perforations into the target formation.” *Id.* at 7:24–27. Further, “[a]fter the composition has been injected into the formation, optionally the well can be shut in for a period of time to allow more complete reaction between the acid and the formation material.” *Id.* at 7:30–33. Frenier explicitly discloses that “[t]he desired result of the treatment is an increase in the permeability of the formation, for example by the creation or enlargement of passageways through the formation, and therefore an increase in the rate of production of formation fluids such as oil and gas.” *Id.* at 7:34–38.

B. Reyes

60. I was provided with a copy of United States Patent No. 10,072,205 B2 to Reyes et al. (Ex. 1005).

61. Reyes is directed to “acidizing subterranean formations, and, more specifically, to methods for acidizing subterranean formations in the presence of a chelating agent that is initially unable to complex a metal ion.” Ex. 1005 at 1:8–12. When able, “chelating agents may be utilized in various acidizing operations or in other subterranean treatment operations to complex a metal ion and sequester it in the subterranean environment.” *Id.* at 3:49–52.

62. Reyes further discloses methods and systems for acidizing subterranean formations using treatment fluids containing “dual-functioning” chelating agents. That is, the “chelating agents may play a dual role of slowing dissolution rates and performing a delayed metal complexation reaction.” *Id.* at 5:2–4.

63. Reyes discloses that “[a]cidizing operations may be used to stimulate a subterranean formation to increase production of a hydrocarbon resource therefrom.” *Id.* at 1:29–31. “Introduction of the acidizing fluid to the subterranean formation may take place at matrix flow rates without fracturing of the formation matrix, or at higher injection rates and pressures to fracture the formation (i.e., an acid-fracturing operation).” *Id.* at 1:31–35.

64. “During an acidizing operation, an acid-soluble material in the subterranean formation can be dissolved by one or more acids to expand existing flow pathways in the subterranean formation, to create new flow pathways in the subterranean formation, and/or to remove acid-soluble precipitation damage in the subterranean formation.” *Id.* at 1:35–41. “The acid-soluble material being dissolved by the acid(s) can be part of or formed from the native formation matrix or can have been deliberately introduced into the subterranean formation in conjunction with a stimulation or like treatment operation (e.g., proppant or gravel particulates).” *Id.* at 1:41–46.

65. Similarly to Frenier, Reyes also covers the formulation of treatment fluids with various additives commonly used in downhole operations to optimize the acidizing process. *Id.* at 11:34–54.

C. Motivation to Combine Frenier and Reyes

66. To the extent needed, a POSITA would have been motivated to combine the teachings of Frenier and Reyes to produce obvious, beneficial, and predictable results. Frenier and Reyes are analogous prior art and both pertain to the same field of endeavor. Specifically, both seek to improve generally known stimulation, matrix acidizing, or acid-treating operations on subterranean formations using treatment mixtures that include chelating and/or sequestering agents (*e.g.*, EDTA or HEDTA).

67. As discussed in both Frenier and Reyes, the beneficial results of the disclosed operations include increasing the permeability of subterranean formations of presently low permeability. Increasing the permeability of the formations provides the benefit of allowing more hydrocarbons to be recovered from the well. The optimized performance of hydrocarbon-bearing wells would have been desirable to a POSITA.

68. Both Frenier and Reyes identify significant limitations of traditional acid stimulation treatments. For instance, both patents acknowledge the risk of precipitation damage resulting from interactions with the treatment fluid and metal

ions within the formation. Additionally, they both recognize concerns regarding the rapid dissolution of injected acids and their rapid reaction with formation materials. While both patents express concerns about the rapid reactions between acids and formation materials, Frenier emphasizes the limited radial penetration depth of acid treatments resulting from these reactions, whereas Reyes focuses on the broader issue of formation matrix erosion due to the same rapid reactions.

69. A POSITA, aware of these challenges, would recognize the potential benefit of combining these disclosures to address them simultaneously. Here, both patents propose using acids and chelating agents, albeit with potentially different formulations or specific functionalities. A POSITA would recognize that combining these approaches offers enhanced effectiveness, particularly with regards to mitigating corrosion and precipitation damage using chelating agents. Further, a POSITA would recognize that combining these acid formulations and chelating agents might lead to a synergistic effect. The particular acid formulations could potentially enhance the overall effectiveness of the chelating agents and minimize unwanted reactions or interactions within the formation.

70. Additionally, both inventions involve wellbore stimulation using a combination of acids and chelating agents. This shared technological base would make the combination readily apparent to a POSITA. A POSITA would identify no technical obstacles likely to impede this combination, as both approaches involve

readily available wellbore treatment components. Moreover, the oil and gas industry has been constantly seeking to improve stimulation treatment efficiency while minimizing formation damage and equipment corrosion. A POSITA would recognize that combining these disclosures aligns with this trend toward more targeted and effective wellbore treatments.

71. Overall, the shared problem recognition, complementary solutions, potential for synergistic effects, and common technological foundation strongly suggest that a POSITA would have been motivated to combine the teachings of Frenier and Reyes. This combination offers a solution that addresses limitations identified in both disclosures, leading to potentially more effective and comprehensive wellbore stimulation treatments.

72. Further, given the similarity of the disclosures, a POSITA would have a reasonable expectation of success in combining the teachings of Frenier and Reyes. Specifically, for any treatment mixture compositions or methods of using those compositions not disclosed or rendered obvious by Frenier alone, a POSITA would have wanted to include the more-explicit teachings of Reyes (*e.g.*, biodegradable chelating agents or adding biocides) to improve the treatment operation, thus improving hydrocarbon production, and would have had a reasonable expectation of doing so.

VII. SPECIFIC GROUNDS FOR PETITION

73. Below I provide my analysis of the prior art as compared to various claims of the '843 Patent.

74. In particular, I have concluded there are two bases for invalidating the Challenged Claims of the '843 Patent. Ground I: Claims 1, 2, 4, 5, 7, and 13 are anticipated by Frenier. Ground II: Claims 1–13 are obvious over Frenier alone or in combination with Reyes.

A. Ground I: Claims 1, 2, 4, 5, 7, and 13 are Anticipated by Frenier

1. Independent Claim 1

a. Element 1[Preamble]: “A method for stimulating an existing well in a nano-darcy shale formation comprising:”

75. Frenier states, “[t]his invention relates to the stimulation of hydrocarbon wells and in particular to acid fluids and methods of using such fluids in **treating a subterranean formation having low permeability.**” Ex. 1004 at 1:6-9 (emphasis added).

76. Frenier further explains that “[t]echniques performed by hydrocarbon producers to increase the net permeability of the reservoir are referred to as ‘stimulation.’ Essentially, one can perform a stimulation technique by... (2) injecting chemicals through the wellbore and into the formation to react with and dissolve small portions of the formation to create alternative flowpaths for the hydrocarbon....” *Id.* at 1:39–54.

77. A POSITA would have known and understood the methods and compositions of Frenier to apply to “subterranean formation[s] having low permeability,” (Ex. 1004 at 1:6-9) which would include shale formations having nano-darcy permeability.

78. Frenier discloses that its “invention relates to the use of acidic fluids in stimulation and **workover operations**, and in particular, in matrix acidizing or fracture acidizing treatments.” Ex. 1004 at 4:8–12 (emphasis added).

79. Given this disclosure by Frenier, a POSITA would have understood the methods and compositions of Frenier to apply to stimulating or otherwise treating pre-existing wells, in addition to new wells.

80. Specifically, it has been well known in the industry and to POSITAs prior to April 14, 2014 that a “workover operation” has generally meant “[t]he repair or **stimulation of an existing production well** for the purpose of restoring, prolonging or enhancing the production of hydrocarbons.” Ex. 1016 at p. 1.

81. Frenier discloses “method[s] for stimulating an existing well in a nano-darcy shale formation.”

82. It is my opinion that each limitation of the preamble of claim 1, as described above, was disclosed by Frenier prior to April 14, 2014.

b. Element 1[a]: “providing a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent at a pH of between about 0 and 10;”

83. As mentioned above, the '843 Patent defines what chemicals may be a “metal complexing agent”:

For the purposes of this disclosure, a metal-complexing agent may be any chemical that can bind with a metal regardless of the binding mechanism and includes **sequestration agents**, reducing agents, **chelating agents**, ligands porphyrins, pigments, peptides, saccharides and/or nucleic acids. In some embodiments, the metal-complexing agent is a **chelating agent**, an alkali metal salt thereof, a non-alkali metal salt thereof, or any combination thereof may be included in the treatment fluids described herein.

Id. at 4:54–63 (emphasis added).

84. Frenier discloses the use of chelating agents and sequestering agents, among other things, in well treatment operations and mixtures.

85. Frenier discloses a “well treatment fluid,” i.e., a treatment mixture, comprising: “a first acid; water; and a hydroxyethylaminocarboxylic acid.” Ex. 1004 at 4:12–13.

86. Frenier further explains that “[e]xamples of first acids include non-oxidizing mineral acids such as hydrochloric acid, sulfuric acid, phosphoric acid, hydrofluoric acid, and mixtures thereof. Other examples include non-oxidizing organic acids such as formic acid, acetic acid, and mixtures thereof.” *Id.* at 5:27–31.

87. However, Frenier also discloses that “hydroxyethylaminocarboxylic acids have the advantage of being effective in acidizing treatment of a formation

without the use of a first acid.” *Id.* at 6:31–33 (emphasis added).

88. Moreover, Frenier states, “[p]referably, the hydroxyethylaminocarboxylic acid is selected from hydroxyethylethylenediaminetriacetic acid (HEDTA), hydroxyethyliminodiacetic acid (HEIDA), or a mixture thereof.” *Id.* at 4:13–17.

89. HEDTA and citric acid are two common chelating agents that were used in well treatment operations prior to April 14, 2014 and continue to be used today. See Ex. 1016 at p. 3.

90. Thus, Frenier discloses various treatment mixtures wherein the metal complexing agent may be a traditional chelating or sequestering agent such as HEDTA or HEIDA, or may be a lower pH mineral or organic acid such as HCL, HF, or acetic acid. A POSITA would have known and understood the same.

91. Frenier provides examples of “typical concentration ranges” for its treatment mixture with and without a first acid, including between 1% and 30% by weight metal complexing agent, which covers much of the claimed range set forth in claim 1. Ex. 1004 at 6:25–47.

92. Frenier further discloses treatment mixtures having between 15% and 28% by weight metal complexing agent and between 10% and 30% by weight metal complexing agent. See claims 1, 9, and 28 at Ex. 1004, 11:27–34; 11:54–63; 13:3–20. However, Frenier includes that “[t]he proportion of the various components of a

composition of the present invention will vary depending on the characteristics of the formation to be treated, the acid to be used, and other factors well known in the art.” *Id.* at 6:16–19.

93. Given the disclosures of Frenier, a POSITA would have known and understood to provide a treatment mixture comprising between 0.1% and 95% by weight metal complexing agent, or chelating agent, such as between 15% and 28% by weight HEDTA.

94. Frenier further discloses the treatment mixture having “a pH of between about 0 and 10” as required by Element 1[a].

95. Specifically, claim 28 of Frenier discloses the treatment mixture consisting of “a pH controller agent to adjust the pH of the treatment fluid between to about 1 to about 12.” *Id.* at 13:9–10.

96. Frenier also discloses an embodiment containing water and a hydroxyethylaminocarboxylic acid, where “the pH of the well treatment fluid is less than about 12, **more preferably from about 1 to about 4**, and can be controlled by an organic acid, a mineral acid, or a base.” *Id.* at 4:57–60 (emphasis added).

97. It is clear from the above that Frenier discloses using a treatment mixture comprising HEDTA as a chelating agent at between 0.1% and 95% by weight and the treatment mixture preferable having a pH between 1 and about 4.

98. The disclosures of Frenier entirely encompass the pH range required

by claim 1, but include a preferred pH range within the pH range of claim 1.

99. The disclosures of Frenier do not significantly deviate, if at all, from the pH range required by claim 1.

100. Additionally, Frenier cites Athey et al., U.S. Pat. No. 5,972,868, entitled “Method for Controlling Alkaline Earth and Transition Metal Scaling with 2-Hydroxyethyl Iminodiacetic Acid,” which, according to Frenier, discloses “compositions comprising 2-hydroxyethyliminodiacetic acid as a chelant for the removal of alkaline earth scale in downhole equipment. The compositions can be at any pH from about 2 to about 13.” *Id.* at 3:66-67; 4:1-5.

101. Frenier also provides examples from which the “present invention can be further understood.” *Id.* at 8:37. Those examples include treatment mixtures having 20% by weight metal complexing agent at pH values of 4, 2.5, and 3.5. *Id.* at 10:61–64.

102. In light of the above, a POSITA would have known and understood to provide the treatment mixture comprising a chelating agent, such as HEDTA or acetic acetic, at a pH between about 0 and 10.

103. A POSITA would have known to provide HEDTA, a chelating agent, as a “metal complexing agent” both at a pH between 0 and 10 and between 0.1% and 95% by weight of the treatment solution.

104. A POSITA would have known to use the above composition to treat

an existing subterranean formation having nano-darcy permeability.

105. Thus, it is my opinion that Frenier discloses all of the limitations of this claim element.

c. Element 1[b]: “injecting the treatment mixture into the well at a pressure less than a fracture pressure of the nano-darcy shale formation until at least some of the treatment mixture exits the well and contacts the nano-darcy shale formation;”

106. Frenier discloses “a method of acid-treating a subterranean formation.” Ex. 1004 at 4:44–45. “The method includes the step of injecting a well treatment fluid composition via a wellbore **into a subterranean formation.**” *Id.* at 4:45–47 (emphasis added). Indeed, “the well treatment fluid composition comprises the components described above.” *Id.* at 4:47–49.

107. Frenier further states, “the injection step is **preferably performed at a fluid pressure that is less than the minimum in situ rock stress (i.e., a matrix acidizing method)**, [but] the method can also be performed at a higher pressure (i.e., an acid fracturing method).” *Id.* at 4:49–53 (emphasis added).

108. Claims 9 and 28 of Frenier are also directed to these “acid-treatment” and “matrix acidizing” methods. *Id.* at 11:54–62.

109. Frenier discloses that “[s]uch matrix acidizing methods generally involve pumping the acid-containing well treatment composition down the wellbore and **out through perforations into the target formation.**” *Id.* at 7:24–

27 (emphasis added).

110. In light of the above, a POSITA would have known and understood to “inject[] the treatment mixture into the well at a pressure less than a fracture pressure of the nano-darcy shale formation until at least some of the treatment mixture exits the well and contacts the nano-darcy shale formation.”

111. Thus, it is my opinion that all of the limitations of this claim element are disclosed by Frenier.

d. Element 1[c]: “maintaining the treatment mixture in contact with the nano-darcy shale formation for a contact time of between about 1 minute to 100 days, thereby allowing the metal complexing agent to bind with at least some naturally-occurring metals contained within the nano-darcy shale formation;”

112. Frenier discloses that “[a]fter the composition has been injected into the formation, optionally the well can be shut in for a period of time to allow more complete reaction between the acid and the formation material.” Ex. 1004 at 7:30–34.

113. Frenier also explains that “[p]arameters such as pumping rate, pumping time, **shut-in time**, acid content, and additive package, must be determined for each particular treatment since each of these parameters depends upon the extent of the damage, formation geology (e.g., permeability), formation temperature, depth of the producing zone, etc. A well-treatment designer of ordinary skill is familiar with the essential features of matrix acidizing treatments.” *Id.* at 7:39–46 (emphasis

added).

114. Notably, Frenier explicitly provides examples wherein the treatment solutions were maintained in contact with the formation samples for 24 and 72 hours. *Id.* at 9:27–66.

115. Thus, a contact time between about 1 minute to 100 days would have been known and understood by a POSITA as a period to allow for a proper reaction between the metal complexing agent and the metals of the formation.

116. A POSITA also would have known that the contact time should be between about 1 minute to 100 days so as to avoid precipitation of the metals, thereby further congesting the well.

117. Frenier also discloses the limitation that the metal complexing agent binds “with at least some naturally-occurring metals contained within the nano-darcy shale formation.” Specifically, claim 28 of Frenier includes the element: “said matrix-acidizing resulting in the formation of wormholes due to the dissolution of minerals **naturally-present in the formation.**” Ex. 1004 at 13:17–19 (emphasis added).

118. Frenier also provides that “[u]se of the well treatment compositions of the present invention in a matrix acidizing process provides for the ready control of iron liberated from the formation, thus helping to minimize the precipitation of iron in the formation or in the wellbore.” *Id.* at 8:12–16.

119. Additionally, Frenier states, “types of scales treatable by the fluids and techniques of the present invention include chloride scales (e.g., NaCl), iron scales (e.g., FeS, Fe₂O₃), silica scales (e.g., SiO₂), sulfate scales (e.g. CaSO₄), and hydroxide scales (e.g., Mg(OH)₂)” in addition to carbonate scales such as CaCO₃ and FeCO₃. *Id.* at 7:1–11.

120. Frenier also provides examples of its inventive concepts wherein “[i]t was observed that substantially all the Fe and Ca present in the sample comprising Na₃HEDTA remained in solution (i.e. both metals were fully sequestered by Na₃HEDTA) at both room temperature and 150° F...” and the results “indicate[d] HEDTA and HEIDA were effective at sequestering iron and minimizing its precipitation.” *Id.* at 8:62–65; 9:54–55.

121. Thus, Frenier discloses “allowing the metal complexing agent to bind with at least some naturally-occurring metals contained within the nano-darcy shale formation.”

122. A POSITA would have known and understood, in light of Frenier, to maintain contact between the treatment mixture for some time between 1 minute and 100 days so that that metal complexing agent can react with at least some naturally occurring metals in the nano-darcy shale formation.

123. It is my opinion that Frenier discloses all of the limitations of this claim element.

- e. **Element 1[d]: “removing the treatment mixture from the well after the contact time, thereby removing the bound naturally-occurring metals from the non-darcy shale formation and thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method.”**

124. Frenier discloses removal of the treatment mixture from the well, thereby removing the metals from the formation and improving the hydrocarbon production from the well in several different instances.

125. First, Frenier plainly states, “the present invention relates to methods to **enhance the productivity of hydrocarbon wells** (e.g., oil wells) by creating alternate flowpaths by removing portions of a wellbore coating, dissolving small portions of the formation, **or removing (by dissolution) near-wellbore formation damage.**” Ex. 1004 at 1:55–60. “The efficiency of a matrix treatment depends primarily upon **removing** or bypassing regions of low permeability that restrict productivity.” *Id.* at 6:49–51 (emphasis added).

126. Second, Frenier states, “[a]nother aspect of the present invention is a method of acid-treating a subterranean formation. The method includes the step of injecting a well treatment fluid composition via a wellbore into a subterranean formation... **The method can also be used to remove deposits from the wellbore.**” Ex. 1004 at 4:44–54.

127. Third, Frenier also teaches using the well treatment fluid to remove

drilling mud that contains naturally-occurring metals:

The well treatment fluid compositions of the present invention can be used to remove drilling mud from the wellbore. Removal of drilling mud occurs especially readily if the mud contains carbonates, especially calcium carbonate. Removal of drilling mud can be performed by any technique know [*sic*] in the art, and involves the steps of injecting a well treatment fluid composition of the present invention into the wellbore.

Id. at 7:13–20.

128. Fourth, Frenier incorporates by reference, U.S. Patent. No. 1,877,504A to Grebe, entitled “Treatment of Deep Wells,” issued September 13, 1932 (“Grebe”) (Ex. 1010). Grebe is directed to “increasing or restoring” the flow of an oil well in a limestone formation “by treating with a quantity of an acid, such as hydrochloric acid. The acid has the effect of attacking and dissolving the rock, thereby enlarging the cavity at the bottom of the well, or the channels and pores in the rock through which oil flows to the well.” Ex. 1010 at page 1, lines 45–52.

129. Grebe further discloses, “[i]n carrying out our improved method we employ a mineral acid, preferably hydrochloric acid, inasmuch as the latter **upon reacting with the calcareous rock forms water soluble salts which remain in solution and are removed from the well with the spent acid.**” *Id.* at lines 59–74 (emphasis added).

130. Fifth, Frenier also incorporates by reference U.S. Patent. No. 2,094,479 to Vandergrift, entitled “Treatment of Wells,” issued September 28, 1937

(“Vandergrift”). Vandergrift is directed to a “method of increasing the flow of fluid mineral products from a well which comprises introducing into the well a chemical reagent comprising hydrofluoric acid and a mineral acid and permitting it to act upon the formations adjacent to the well cavity.” Ex. 1011 at 4:14–19.

131. Vandergrift discloses, “[a]fter the mixed acid reagent has had sufficient time to act upon the formation, **the spent acid may be removed by pumping or bailing out.** It may sometimes be desirable to flush out the well with water or other neutral liquid after the spent acid has been removed and before the well is put back into production.” *Id.* at 4:34–40 (emphasis added).

132. Sixth, Frenier incorporates by reference, U.S. Patent. No. 4,695,389 to Kubala entitled “AQUEOUS GELLING AND/OR FOAMING AGENTS FOR AQUEOUS ACIDS AND METHODS OF USING THE SAME,” issued September 22, 1987 (“Kubala”) (Ex. 1012). Kubala is directed to:

[A] novel acid gelling agent that can be combined with aqueous acids at higher concentrations of the acids to produce highly retarded gelled acid systems that exhibit the unique characteristic of building viscosity during the carbonate rock dissolving process, reaching a peak viscosity when the acid is partially spent (e.g., approximately 12% HCl and in the presence of calcium ion) with virtually complete breaking of the gel when the acid is totally spent.

Ex. 12 at 2:6–14. Kubala further discloses, “after the reaction is completed, the viscosity again drops to a very low value insuring **ease of removal and backflushing of the formation.**” *Id.* at 10:43–46 (emphasis added).

133. A POSITA would have known and understood the removal step of claim 1 in light of any of the above disclosures of Frenier, and certainly in light of a combination of the same.

134. Specifically, a POSITA would have known and understood, in light of Frenier and the references incorporated therein, to “remov[e] the treatment mixture from the well after the contact time, thereby removing the bound naturally-occurring metals from the non-darcy shale formation and thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method.”

135. Accordingly, it is my opinion that all of the limitations of this claim element are disclosed by Frenier and its incorporated references.

136. Frenier fully disclosed each and every element of Claim 1 of the '843 Patent to a POSITA prior to April 14, 2014.

2. Dependent Claim 2

a. “The method of claim 1 wherein the metal complexing agent is citric acid.”

137. Frenier cites to U.S. Pat. No. 4,888,121 to Dill, entitled “Compositions and Method for Controlling Precipitation When Acidizing Sour Wells” (“Dill”).

138. According to Frenier, Dill “discloses an acidizing composition that includes an acid such as CHI; **an iron sequestering agent such as citric acid,**

EDTA, or nitrilotriacetic acid (NTA); and a sulfide modifier such as formaldehyde."

Id. at 3:39–45 (emphasis added).

139. Thus, a POSITA would have known and understood to use citric acid as the metal complexing/sequestering agent prior to April 2014.

140. Additionally, Frenier discloses, "[i]t has been discovered that hydroxyethylaminocarboxylic acids are very soluble in mineral acids or **organic acids**, and can be mixed with mineral acids or organic acids to provide a wide range of useful formulations." Ex. 1004 at 5:16–20 (emphasis added). A POSITA would have known and understood Frenier's disclosure of "organic acids" as metal complexing agents to include citric acid.

141. Frenier also incorporates by reference U.S. Patent No. 4,574,050 to Crowe et al., entitled "METHOD FOR PREVENTING THE PRECIPITATION OF FERRIC COMPOUNDS DURING THE ACID TREATMENT OF WELLS," issued March 4, 1986 ("Crowe") (Ex. 1013).

142. Crowe is directed to "methods of substantially eliminating problems arising from the presence of ferric ions which occur during the acidization of wells." Ex. 1013 at 1:15–17.

143. Crowe states, "[t]he most common iron sequestering agents in commercial practice are **citric acid**, ethylenediaminetetra acetic acid (EDTA), nitrilotriacetic acid (NTA), acetic acid, lactic acid, and **citric acid/acetic acid**

mixtures.” *Id.* at 1:58–64 (emphasis added).

144. Frenier’s disclosure of “organic acids” as metal complexing agents would include citric acid. *Id.*

145. It was also well-within the knowledge of a POSITA prior to april 14, 2014 to select citric acid as a chelating agent for well treatment operations and compositions. Ex. 1016 at p. 3; Ex. 1015 at p. 15.

146. Thus, Frenier discloses every element of dependent claim 2.

3. Dependent Claim 4

a. “The method of claim 1 wherein the metal complexing agent is acetic acid.”

147. As set forth above, Frenier discloses that “[e]xamples of first acids include non-oxidizing mineral acids such as hydrochloric acid, sulfuric acid, phosphoric acid, hydrofluoric acid, and mixtures thereof. Other examples include non-oxidizing organic acids such as formic acid, **acetic acid**, and mixtures thereof.” *Id.* at 5:27–31 (emphasis added).

148. A POSITA would have known in light of Frenier and its incorporated references to use acetic acid as a metal complexing agent.

149. It was also well-within the knowledge of a POSITA prior to April 14, 2014 to use acetic acid as a complexing agent. Ex. 1015 at p. 15.

150. Frenier discloses every element of dependent claim 4.

4. Dependent Claim 5

- a. “The method of claim 1 wherein the metal complexing agent includes at least one of ethylenediaminetetraacetic acid (EDTA), propylenediaminetetraacetic acid (PDTA), nitrilotriacetic acid (NTA), N-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), diethylenetriaminepentaacetic acid (DTPA), hydroxyethyliminodiacetic acid (HEIDA), cyclohexylenediaminetetraacetic acid (CDTA), diphenylaminesulfonic acid (DPAS), ethylenediaminedi(o-hydroxyphenylacetic) acid (EDDHA), glucoheptonic acid, gluconic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, phthalic acid, terephthalic acid, aconitic acid, carballylic acid, trimesic acid, isocitric acid, citric acid, or any salt or derivative of any of the previously listed compounds.”

151. As set forth above, Frenier discloses the metal complexing agent including several, if not all, of the acids listed in claim 5.

152. For example, Frenier discloses that “hydroxyethylaminocarboxylic acids have the advantage of being effective in acidizing treatment of a formation without the use of a first acid.” *Id.* at 6:31–33; *see also* claim 28, *id.* at 13:3–19.

153. Frenier states, “[p]referably, the hydroxyethylaminocarboxylic acid is selected from hydroxyethylethylenediaminetriacetic acid (**HEDTA**), hydroxyethyliminodiacetic acid (**HEIDA**), or a mixture thereof.” *Id.* at 4:13–17. (emphasis added).

154. Additionally, I incorporate herein my statement and conclusions above for Dependent Claims 2 with respect to the metal complexing agent including citric acid. *See* Ex. 1016 at p. 3; Ex. 1015 at p. 15.

155. Accordingly, it is my opinion that Frenier discloses every element of Dependent Claim 5.

5. Dependent Claim 7

a. **“The method of claim 1 wherein the treatment mixture further comprises between 0.1 and 95% of acid(s) as pH modifier.”**

156. Frenier discloses a treatment mixture including the use of additives, explicitly including pH modifiers: “[t]he composition can also include one or more additives that are compatible with the acid composition, such as... **pH control agents**. Many such additives are well known in the art.” *Id.* at 5:56–63 (emphasis added).

157. Indeed, the use of pH modifiers to control the pH of the treatment mixtures, and the performance of the treatment mixture, was well known and understood by POSITAs prior to April 14, 2014.

158. Frenier provides examples of treatment mixtures comprising 0.1% to 15% by weight pH modifier (*id.* at 6:40–45), and claims 28, 31, 49 are directed to treatment mixtures that include pH modifiers. *Id.* at 13:3–19; 13:25–27; 14:44–45.

159. Frenier provides, in Example 4, data concerning the testing of five hydroxyethylaminocarboxylic acid aqueous solutions, the pH of all but one of which was adjusted using either hydrochloric acid or formic acid. For example, two aqueous solution of 20% Na₃HEDTA was adjusted with hydrochloric acid to

maintain a pH of 4 and 2.5, respectively. *See id.* at 10:60-67; Table 3. Because Frenier explicitly discloses the use of an acid as a pH modifier, a POSITA would inherently recognize the need for a sufficient amount of acid to achieve the desired pH change. A concentration of 0.1% by weight represents a minimal amount for an acid solution. An pH modifier at a concentration of 0.1% by weight represents a starting point that is unlikely to have any unexpected or unobvious effects.

160. A POSITA would have known to include between .0 and 95% of acid(s) as pH modifier.

161. It is my opinion that Frenier discloses every limitation of dependent claim 7.

6. Dependent Claim 13

- a. **“The method of claim 1 further wherein the metal complexing agent is selected from acetic acid, acrylates, dihydroxymaleic acid, salts of dihydroxymaleic acid, EDTA (ethylenediamine tetraacetic acid), salts of EDTA, erythorbic acid, erythroboric acid, formic acid, gluconodeltalactone, GLDA (glutamic acid N,N-diacetic acid), salts of GLDA, HEDTA (hydroxyethylenediamine triacetic acid), salts of HEDTA, HEIDA (disodium ethanoldiglycine), salts of HEIDA, MGDA (methylglycine N,N-diacetic acid), salts of MGDA, NTA (nitriolotriacetic acid), organic metal complexers, ligands, porphyrins, pigments, peptides, saccharides, nucleic acids, phosphonic acid, polyacrylic acid and citric acid in an amount sufficient to sequester at least a portion of a metal compound.”**

162. As set forth above, Frenier discloses the metal complexing agent selected from several, if not all, of the acids listed in claim 13.

163. For example, Frenier discloses that “hydroxyethylaminocarboxylic acids have the advantage of being effective in acidizing treatment of a formation without the use of a first acid.” *Id.* at 6:31–33; *see also* claim 28, *id.* at 13:3–19. “Preferably, the hydroxyethylaminocarboxylic acid is selected from hydroxyethylethylenediaminetriacetic acid (HEDTA), hydroxyethyliminodiacetic acid (HEIDA), or a mixture thereof.” *Id.* at 4:13–17.

164. Additionally, I incorporate herein my statements and conclusions above for Dependent Claims 2 and 5.

165. Accordingly, it is my opinion that Frenier discloses every element of Dependent Claim 13.

B. Ground II: Claims 1–13 are obvious under 35 U.S.C. § 103(a) over Frenier alone and/or in combination with Reyes

166. With respect to claims 1, 2, 4, 5, 7, and 13, I incorporate herein my statements and conclusions above. At a minimum, every element of claim 1 would have been obvious to a POSITA in light of the aforementioned disclosures of Frenier.

1. Independent Claim 1

a. Element 1[Preamble]: “A method for stimulating an existing well in a nano-darcy shale formation comprising:”

167. Frenier states “[t]his invention relates to the stimulation of

hydrocarbon wells and in particular to acid fluids and methods of using such fluids in **treating a subterranean formation having low permeability.**" Ex. 1004 at 1:6-9 (emphasis added).

168. Frenier further explains that "[t]echniques performed by hydrocarbon producers to increase the net permeability of the reservoir are referred to as 'stimulation.' Essentially, one can perform a stimulation technique by... (2) injecting chemicals through the wellbore and into the formation to react with and dissolve small portions of the formation to create alternative flowpaths for the hydrocarbon...." *Id.* at 1:39–54.

169. It would have been obvious to a POSITA in light of Frenier to apply the methods and compositions disclosed therein to "subterranean formation[s] having low permeability," including nano-darcy shale formations.

170. Moreover, Frenier discloses that its "invention relates to the use of acidic fluids in stimulation and **workover operations**, and in particular, in matrix acidizing or fracture acidizing treatments." Ex. 1004 at Abstract; 4:8–12.

171. It would have been obvious to a POSITA, in light of Frenier, to apply the methods and compositions disclosed in Frenier to stimulating or otherwise treating pre-existing wells, in addition to new wells.

172. Specifically, it has been well known in the industry that a "workover operation" has generally meant "[t]he repair or **stimulation of an existing**

production well for the purpose of restoring, prolonging or enhancing the production of hydrocarbons.” Ex. 1016 at p. 1.

173. Moreover, Reyes states, “[t]he present disclosure generally relates to acidizing subterranean formations.” Ex. 1005 at 3:44–45. “Acidizing operations may be used to stimulate a subterranean formation to increase production of a hydrocarbon resource therefrom.” *Id.* at 1:29–31.

174. Reyes further explains that “[i]n some embodiments, the treatment fluids may be used to treat an **existing fracture** in a subterranean formation in order to enhance a flow pathway therein.” *Id.* at 13:40–42 (emphasis added). *Id.* at 1:39–54.

175. Thus, it would have been obvious to a POSITA prior to the effective filing date of the ’843 Patent to apply the stimulation methods of Frenier and Reyes to an existing well in a nano-darcy shale formation.

176. It would have been obvious for a POSITA to apply the stimulation methods of Frenier and Reyes to an existing well in a nano-darcy shale formation for the predictable benefit of increasing hydrocarbon production from the well.

b. Element 1[a]: “providing a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent at a pH of between about 0 and 10;”

177. As mentioned above, the ’843 Patent defines what chemicals may be a “metal complexing agent”:

For the purposes of this disclosure, a metal-complexing agent may be any chemical that can bind with a metal regardless of the binding mechanism and includes **sequestration agents**, reducing agents, **chelating agents**, ligands porphyrins, pigments, peptides, saccharides and/or nucleic acids. In some embodiments, the metal-complexing agent is a **chelating agent**, an alkali metal salt thereof, a non-alkali metal salt thereof, or any combination thereof may be included in the treatment fluids described herein.

Id. at 4:54–63 (emphasis added).

178. Frenier discloses the use of chelating agents and sequestering agents, among other things, in well treatment operations and the treatment mixtures used therefore.

179. Frenier discloses a “well treatment fluid,” i.e., a treatment mixture, comprising: “a first acid; water; and a hydroxyethylaminocarboxylic acid.” Ex. 1004 at 4:12–13.

180. Frenier further explains that “[e]xamples of first acids include non-oxidizing mineral acids such as hydrochloric acid, sulfuric acid, phosphoric acid, hydrofluoric acid, and mixtures thereof. Other examples include non-oxidizing organic acids such as formic acid, acetic acid, and mixtures thereof.” *Id.* at 5:27–31.

181. However, Frenier also discloses that “hydroxyethylaminocarboxylic acids have the advantage of being effective in acidizing treatment of a formation **without the use of a first acid.**” *Id.* at 6:31–33 (emphasis added).

182. Frenier states, “[p]referably, the hydroxyethylaminocarboxylic acid is selected from hydroxyethylethylenediaminetriacetic acid (HEDTA), hydroxyethyliminodiacetic acid (HEIDA), or a mixture thereof.” *Id.* at 4:13–17.

183. Thus, Frenier discloses various treatment mixtures wherein the metal complexing agent may be a traditional chelating or sequestering agent such as HEDTA or HEIDA, or may be a lower pH mineral or organic acid such as HCL, HF, or acetic acid.

184. It is my opinion that it would have been at least obvious to select HEDTA or HEIDA as a metal complexing agent, i.e., a chelating agent, when providing a treatment composition for stimulating or remediating a subterranean formation having nano-darcy permeability.

185. Additionally, Frenier provides examples of “typical concentration ranges” for its treatment mixture with and without a first acid, including between 1% and 30% by weight metal complexing agent, which covers much of the claimed ranges set forth in claim 1. *Id.* at 6:25–47.

186. Frenier further discloses treatment mixtures having between 15% and 28% by weight metal complexing agent and between 10% and 30% by weight metal complexing agent. *See* claims 1, 9, and 28 at Ex. 1004, 11:27–34; 11:54–63; 13:3–20. However, Frenier includes that “[t]he proportion of the various components of a composition of the present invention will vary depending on the characteristics of

the formation to be treated, the acid to be used, and other factors well known in the art.” *Id.* at 6:16–19.

187. Frenier further discloses the treatment mixture having “a pH of between about 0 and 10” as required by Element 1[a]. Specifically, claim 28 of Frenier discloses the treatment mixture consisting of “a pH controller agent to adjust the pH of the treatment fluid between to about 1 to about 12.” *Id.* at 13:9–10.

188. Frenier also discloses an embodiment containing water and a hydroxyethylaminocarboxylic acid, where “the pH of the well treatment fluid is less than about 12, more preferably from about 1 to about 4, and can be controlled by an organic acid, a mineral acid, or a base.” *Id.* at 4:57–60.

189. Additionally, Frenier cites Athey et al., U.S. Pat. No. 5,972,868, entitled “Method for Controlling Alkaline Earth and Transition Metal Scaling with 2-Hydroxyethyl Iminodiacetic Acid,” which, according to Frenier, discloses “compositions comprising 2-hydroxyethyliminodiacetic acid as a chelant for the removal of alkaline earth scale in downhole equipment. The compositions can be at any pH from about 2 to about 13.” *Id.* at 3:66-67; 4:1-5.

190. Frenier also provides examples from which the “present invention can be further understood.” *Id.* at 8:37. Those examples include treatment mixtures having 20% by weight metal complexing agent at pH values of 4, 2.5, and 3.5. *Id.* at 10:61–64.

191. Indeed, to the extent the ranges of the '843 Patent are not anticipated by Frenier, it would have been obvious to a POSITA prior to the effective filing date of the '843 Patent to provide a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent at a pH of between about 0 and 10.

192. I agree with the Examiner's conclusion that "discovering the optimum or workable ranges involves only routine skill in the art." Ex. 1002 at 246 (citing *In re Aller*, 105 USPQ 233).

193. Additionally, Reyes discloses a "treatment fluid," i.e., a treatment mixture, comprising: "an aqueous carrier fluid, an acid, and a chelating agent...." Ex. 1005 at 6:6–8.

194. Similarly to Frenier, either the chelating agent or the acid disclosed by Reyes teaches the metal complexing agent of the '843 Patent.

195. Specifically with respect to chelating agents, Reyes states, "the concentration of the chelating agent in the treatment fluid may range between about 0.1% to about 40% of the treatment fluid by weight." *Id.* at 11:21–23. Reyes also predominantly provides the example of using aminopolycarboxylic acid chelating agents but also states that, alternatively, "traditional chelating agents such as" EDTA, HEDTA, HEIDA, and citric acid may be used. *Id.* at 10:59–67; 11:1–4.

196. Moreover, Reyes states:

One of ordinary skill in the art will recognize that there are fairly well established pH ranges over which a chelating agent

can be expected to effectively complex a metal ion. The pH range over which metal ion complexation may effectively take place can be dependent on a number of factors including, but not limited to, the chemical structure of the chelating agent, the functional groups present in the chelating agent, and the particular metal ion being complexed. Nevertheless, one of ordinary skill in the art will understand these issues and be able to determine if a particular metal ion is being effectively complexed by a given chelating agent.”

Id. at 4:5–16.

197. Thus, to the extent the limitation “providing a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent at a pH of between about 0 and 10” is not anticipated or rendered obvious by Frenier alone, such a limitation would have been obvious to a POSITA before the effective filing date of the ’843 Patent in light of the combination of Frenier and Reyes.

c. Element 1[b]: “injecting the treatment mixture into the well at a pressure less than a fracture pressure of the nano-darcy shale formation until at least some of the treatment mixture exits the well and contacts the nano-darcy shale formation;”

198. Frenier discloses “a method of acid-treating a subterranean formation.” Ex. 1004 at 4:44–45. “The method includes the step of injecting a well treatment fluid composition via a wellbore **into a subterranean formation.**” *Id.* at 4:45–47 (emphasis added). Indeed, “the well treatment fluid composition comprises the components described above.” *Id.* at 4:47–49.

199. Moreover, “the injection step is **preferably performed at a fluid pressure that is less than the minimum in situ rock stress (i.e., a matrix acidizing**

method), [but] the method can also be performed at a higher pressure (i.e., an acid fracturing method).” *Id.* at 4:49–53 (emphasis added).

200. Claims 9 and 28 of Frenier are also directed to these “acid-treatment” and “matrix acidizing” methods. *Id.* at 11:54–62. Frenier discloses that “[s]uch matrix acidizing methods generally involve pumping the acid-containing well treatment composition down the wellbore and **out through perforations into the target formation.**” *Id.* at 7:24–27 (emphasis added).

201. Thus, it would have been obvious to a POSITA prior to the effective filing date of the ’843 Patent to “inject[] the treatment mixture into the well at a pressure less than a fracture pressure of the nano-darcy shale formation until at least some of the treatment mixture exits the well and contacts the nano-darcy shale formation.”

202. Additionally, Reyes discloses, “in some embodiments, the treatment fluids described herein can be introduced to a subterranean formation **below a fracture gradient pressure of the subterranean formation.**” Ex. 1005 at 4:44–45 (emphasis added). Reyes additionally states, “[i]ntroduction of the acidizing fluid to the subterranean formation may take place at matrix flow rates without fracturing of the formation matrix....” *Id.* at 1:31–33.

203. Reyes also discloses that “[t]he treatment fluid may be conveyed via line 12 to wellhead 14, where the treatment fluid enters tubular 16, tubular 16

extending from wellhead 14 into subterranean formation 18. **Upon being ejected from tubular 16, the treatment fluid may subsequently penetrate into subterranean formation 18.**” *Id.* at 12:46–50 (emphasis added).

204. Thus, Reyes explicitly discloses all of the limitations of this claim element.

205. However, at a minimum, the element “injecting the treatment mixture into the well at a pressure less than a fracture pressure of the nano-darcy shale formation until at least some of the treatment mixture exits the well and contacts the nano-darcy shale formation” would have been obvious to a POSITA before the effective filing date of the ’843 Patent in light of Frenier and Reyes.

d. Element 1[c]: “maintaining the treatment mixture in contact with the nano-darcy shale formation for a contact time of between about 1 minute to 100 days, thereby allowing the metal complexing agent to bind with at least some naturally-occurring metals contained within the nano-darcy shale formation;”

206. Frenier discloses that “[a]fter the composition has been injected into the formation, optionally the well can be shut in for a period of time to allow more complete reaction between the acid and the formation material.” Ex. 1004 at 7:30–34.

207. Frenier also explains that “[p]arameters such as pumping rate, pumping time, **shut-in time**, acid content, and additive package, must be determined for each particular treatment since each of these parameters depends upon the extent

of the damage, formation geology (e.g., permeability), formation temperature, depth of the producing zone, etc. A well-treatment designer of ordinary skill is familiar with the essential features of matrix acidizing treatments.” *Id.* at 7:39–46 (emphasis added).

208. Indeed, to the extent the range of 1 minute to 100 days is not explicitly or inherently disclosed by Frenier, such a contact time would have been obvious to a POSITA as a period to allow for a proper reaction between the metal complexing agent and the metals of the formation.

209. Notably, in the examples provided in Frenier, the treatment solutions were maintained in contact with the formation samples for 24 and 72 hours. Ex. 1004 at 9:27–66.

210. Frenier discloses the limitation that the metal complexing agent binds “with at least some naturally-occurring metals contained within the nano-darcy shale formation.”

211. Specifically, claim 28 of Frenier includes the element: “said matrix-acidizing resulting in the formation of wormholes due to the dissolution of minerals **naturally-present in the formation.**” *Id.* at 13:17–19 (emphasis added).

212. Frenier also provides that “[u]se of the well treatment compositions of the present invention in a matrix acidizing process provides for the ready control of iron liberated from the formation, thus helping to minimize the precipitation of

iron in the formation or in the wellbore.” *Id.* at 8:12–16.

213. Additionally, Frenier states, “types of scales treatable by the fluids and techniques of the present invention include chloride scales (e.g., NaCl), iron scales (e.g., FeS, Fe₂O₃), silica scales (e.g., SiO₂), sulfate scales (e.g. CaSO₄), and hydroxide scales (e.g., Mg(OH)₂)” in addition to carbonate scales such as CaCO₃ and FeCO₃. *Id.* at 7:1–11.

214. Frenier also provides examples of its inventive concepts wherein “[i]t was observed that substantially all the Fe and Ca present in the sample comprising Na₃HEDTA remained in solution (i.e. both metals were fully sequestered by Na₃HEDTA) at both room temperature and 150° F...” and the results “indicate[d] HEDTA and HEIDA were effective at sequestering iron and minimizing its precipitation.” *Id.* at 8:62–65; 9:54–55.

215. Therefore, Frenier discloses “allowing the metal complexing agent to bind with at least some naturally-occurring metals contained within the nano-darcy shale formation.”

216. Thus, it would have been obvious to a POSITA in light of Frenier to maintain contact between the treatment mixture for some time between 1 minute and 100 days so that that metal complexing agent can react with at least some naturally occurring metals in the nano-darcy shale formation.

217. Additionally, Reyes states as follows:

In various embodiments, the methods described herein may comprise complexing a metal ion with the chelating agent, once the pH of the treatment fluid rises and the chelating agent again becomes active for complexing a metal ion in the subterranean formation. The metal ion being complexed by the chelating agent is not believed to be particularly limited. **Illustrative sources of the metal ion may include, for example, a native carbonate mineral present in the subterranean formation,** a non-native carbonate material that was previously introduced to the subterranean formation, and/or metal ions being leached into the subterranean formation through corrosion of a drilling tool or wellbore pipe, for example. Illustrative metal ions that may be present in a subterranean formation due to dissolution of a carbonate mineral include, for example, **calcium ions and magnesium ions.** Illustrative metal ions that may be present in a subterranean formation due to corrosion include iron ions, or any other metal ion resulting from the dissolution of steel by an acid. **In various embodiments, the metal ion being complexed by the chelating agent may include, for example, a calcium ion, a magnesium ion, an iron ion, or any combination thereof.** The metal ion may be complexed with the chelating agent through a direct reaction of the chelating agent with a surface in the subterranean formation (i.e., a carbonate mineral surface), or the metal ion may be complexed by the chelating agent after the metal ion has undergone an initial dissolution by the acid."

Ex. 1005 at 9:54-67; 10:1-13 (emphasis added).

218. Thus, Reyes also discloses "allowing the metal complexing agent to bind with at least some naturally-occurring metals contained within the nano-darcy shale formation."

219. Therefore, this claim element would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier and Reyes.

e. Element 1[d]: "removing the treatment mixture from the well

after the contact time, thereby removing the bound naturally-occurring metals from the non-darcy shale formation and thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method.”

220. Frenier teaches removal of the treatment mixture from the well, thereby removing the metals from the formation and improving the hydrocarbon production from the well in several different instances.

221. First, Frenier plainly discloses, “the present invention relates to methods to **enhance the productivity of hydrocarbon wells** (e.g., oil wells) by creating alternate flowpaths by removing portions of a wellbore coating, dissolving small portions of the formation, **or removing (by dissolution) near-wellbore formation damage.**” Ex. 1004 at 1:55–60. “The efficiency of a matrix treatment depends primarily upon **removing** or bypassing regions of low permeability that restrict productivity.” *Id.* at 6:49–51 (emphasis added).

222. Second, Frenier states, “[a]nother aspect of the present invention is a method of acid-treating a subterranean formation. The method includes the step of injecting a well treatment fluid composition via a wellbore into a subterranean formation... **The method can also be used to remove deposits from the wellbore.**” *Id.* at 4:53–55.

223. Third, Frenier also teaches using the well treatment fluid to remove drilling mud that contains naturally-occurring metals:

The well treatment fluid compositions of the present invention can be used to remove drilling mud from the wellbore. Removal of drilling mud occurs especially readily if the mud contains carbonates, especially calcium carbonate. Removal of drilling mud can be performed by any technique know [*sic*] in the art, and involves the steps of injecting a well treatment fluid composition of the present invention into the wellbore.

Id. at 7:13–20.

224. Fourth, Frenier incorporates Grebe (provided above, Ex. 1010) by reference. Grebe is directed to “increasing or restoring” the flow of an oil well in a limestone formation “by treating with a quantity of an acid, such as hydrochloric acid. The acid has the effect of attacking and dissolving the rock, thereby enlarging the cavity at the bottom of the well, or the channels and pores in the rock through which oil flows to the well.” (Ex. 1010, page 1, lines 45–52). Grebe further discloses, “[i]n carrying out our improved method we employ a mineral acid, preferably hydrochloric acid, inasmuch as the latter **upon reacting with the calcareous rock forms water soluble salts which remain in solution and are removed from the well with the spent acid.**” *Id.* at lines 59–74 (emphasis added).

225. Fifth, Frenier also incorporates Vandergrift (provided above, Ex. 1011) by reference. Vandergrift is directed to a “method of increasing the flow of fluid mineral products from a well which comprises introducing into the well a chemical reagent comprising hydrofluoric acid and a mineral acid and permitting it to act upon the formations adjacent to the well cavity.” Ex. 1011 at 4:14–19.

Vandergrift discloses, “[a]fter the mixed acid reagent has had sufficient time to act upon the formation, **the spent acid may be removed by pumping or bailing out.** It may sometimes be desirable to flush out the well with water or other neutral liquid after the spent acid has been removed and before the well is put back into production.” *Id.* at 4:29–40 (emphasis added).

226. Sixth, Frenier incorporates Kubala (provided above, Ex. 1012) by reference. Kubala is directed to:

[A] novel acid gelling agent that can be combined with aqueous acids at higher concentrations of the acids to produce highly retarded gelled acid systems that exhibit the unique characteristic of building viscosity during the carbonate rock dissolving process, reaching a peak viscosity when the acid is partially spent (e.g., approximately 12% HCl and in the presence of calcium ion) with virtually complete breaking of the gel when the acid is totally spent.

Kubala further discloses, “after the reaction is completed, the viscosity again drops to a very low value insuring **ease of removal and backflushing of the formation.**” *Id.* at 10:43–46 (emphasis added).

227. Additionally, it would have been obvious to a POSITA prior to the effective filing date of the ’843 Patent to “remov[e] the treatment mixture from the well after the contact time, thereby removing the bound naturally-occurring metals from the non-darcy shale formation and thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method.”

228. A POSITA would have been motivated to remove the bound metals from the formation during this period to avoid precipitation of the metals, thereby congesting the well.

229. Thus, each and every element of Claims 1 of the '843 Patent would have been obvious to a POSITA prior to its effective filing date.

230. Additionally, Reyes states, “[a]cidizing operations may be used to **stimulate a subterranean formation to increase production of a hydrocarbon resource therefrom.**” Ex. 1005 at 1:29–31 (emphasis added).

231. Reyes further discloses that “the treatment fluid may, in some embodiments, flow back to wellhead 14 and exit subterranean formation 18. In some embodiments, **the treatment fluid that has flowed back to wellhead 14 may subsequently be recovered** and recirculated to subterranean formation 18.” *Id.* at 12:62-67 (emphasis added).

232. Therefore, this claim element would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier and Reyes.

2. Dependent Claim 2

a. “The method of claim 1 wherein the metal complexing agent is citric acid.”

233. Frenier cites to U.S. Pat. No. 4,888,121 to Dill, entitled “Compositions and Method for Controlling Precipitation When Acidizing Sour Wells” (“Dill”).

234. According to Frenier, Dill “discloses an acidizing composition that includes an acid such as CHI; **an iron sequestering agent such as citric acid**, EDTA, or nitrilotriacetic acid (NTA); and a sulfide modifier such as formaldehyde.” *Id.* at 3:39–45 (emphasis added).

235. Thus, a POSITA would have known and understood, or at a minimum it would have been obvious, to use citric acid as the metal complexing/sequestering agent prior to April 2014.

236. Additionally, Frenier discloses, “[i]t has been discovered that hydroxyethylaminocarboxylic acids are very soluble in mineral acids or **organic acids**, and can be mixed with mineral acids or organic acids to provide a wide range of useful formulations.” Ex. 1004 at 5:16–20 (emphasis added). A POSITA would have known and understood Frenier’s disclosure of “organic acids” as metal complexing agents to include citric acid.

237. Frenier also incorporates by reference U.S. Patent No. 4,574,050 to Crowe et al., entitled “METHOD FOR PREVENTING THE PRECIPITATION OF FERRIC COMPOUNDS DURING THE ACID TREATMENT OF WELLS,” issued March 4, 1986 (“Crowe”) (Ex. 1013).

238. Crowe is directed to “methods of substantially eliminating problems arising from the presence of ferric ions which occur during the acidization of wells.” Ex. 1013 at 1:15–17.

239. Crowe states, “[t]he most common iron sequestering agents in commercial practice are **citric acid**, ethylenediaminetetra acetic acid (EDTA), nitrilotriacetic acid (NTA), acetic acid, lactic acid, and **citric acid/acetic acid mixtures**.” *Id.* at 1:58–64 (emphasis added).

240. Thus, selecting citric acid to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the ’843 Patent in light of Frenier and its incorporated references.

241. Additionally, Reyes explicitly discloses, “as an alternative to aminopolycarboxylic acid chelating agents, traditional chelating agents such as, for example, [EDTA, PDTA, NTA, HEDTA, DTPA, HEIDA, CDTA, DPAS, EDDHA], glucoheptonic acid, gluconic acid, **citric acid**, any salt thereof, any derivative thereof, or the like may be included in the treatment fluids described herein.” Ex. 1005 at 10:59–67; 11:1–4 (emphasis added) (shortened for brevity).

242. It was also well-within the knowledge of a POSITA prior to april 14, 2014 to select citric acid as a chelating agent for well treatment operations and compositions. Ex. 1016 at p. 3; Ex. 1015 at p. 15.

243. Thus, selecting citric acid to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the ’843 Patent in light of Frenier and Reyes.

3. Dependent Claim 3

a. “The method of claim 1 wherein the metal complexing agent is ethylenediaminetetraacetic acid (EDTA).”

244. I incorporate herein my statements and conclusions above with respect to Dependent Claim 2.

245. According to Frenier, Dill “discloses an acidizing composition that includes an acid such as CHI; **an iron sequestering agent such as** citric acid, **EDTA**, or nitrilotriacetic acid (NTA); and a sulfide modifier such as formaldehyde.” *Id.* at 3:39–45 (emphasis added).

246. Frenier also states, “acid treatment fluids often contain additives to minimize iron precipitation and H₂S evolution, for example **by sequestering the Fe ions in solution using chelating agents, such as ethylenediaminetetraacetic acid (EDTA).**” *Id.* at 3:35–38 (emphasis added).

247. Notably, although it found HEDTA to perform better, Frenier also provides examples of using EDTA to sequester iron and minimize precipitation. *See id.* at 8:40–11:25.

248. Frenier also incorporates by reference Crowe, which states, “[t]he most common iron sequestering agents in commercial practice are citric acid, **ethylenediaminetetra acetic acid (EDTA)**, nitrilotriacetic acid (NTA), acetic acid, lactic acid, and citric acid/acetic acid mixtures.” *Id.* at 1:58–64 (emphasis added).

249. Thus, selecting EDTA to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the ’843 Patent in

light of Frenier.

250. Additionally, Reyes explicitly discloses, “as an alternative to aminopolycarboxylic acid chelating agents, traditional chelating agents such as, for example, **ethylenediaminetetraacetic acid (EDTA)**, [PDTA, NTA, HEDTA, DTPA, HEIDA, CDTA, DPAS, EDDHA], glucoheptonic acid, gluconic acid, citric acid, any salt thereof, any derivative thereof, or the like may be included in the treatment fluids described herein.” Ex. 1005 at 10:59–67; 11:1–4 (emphasis added).

251. Thus, selecting EDTA to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the ’843 Patent in light of Frenier and Reyes.

4. Dependent Claim 4

a. “The method of claim 1 wherein the metal complexing agent is acetic acid.”

252. As set forth above, Frenier discloses that “[e]xamples of first acids include non-oxidizing mineral acids such as hydrochloric acid, sulfuric acid, phosphoric acid, hydrofluoric acid, and mixtures thereof. Other examples include non-oxidizing organic acids such as formic acid, **acetic acid**, and mixtures thereof.” Ex. 1004 at 5:27–31 (emphasis added).

253. Thus, selecting acetic acid to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the ’843 Patent in light of Frenier.

254. Additionally, Reyes explicitly discloses that “the treatment fluids described herein may include an acid.... Suitable organic acids may include, for example, formic acid, **acetic acid**, chloroacetic acid, dichloroacetic acid, trichloroacetic acid, methanesulfonic acid and the like.” Ex. 1005 at 7:34–46 (emphasis added).

255. Reyes further states, “[b]oth mineral acids (e.g., hydrochloric acid) and organic acids (e.g., **acetic** and formic acids) can be used to treat a carbonate formation, often with similar degrees of success.” *Id.* at 1:59–62 (emphasis added).

256. Thus, selecting acetic acid to use as the metal complexing agent would have been obvious to a POSITA before the effective filing date of the ’843 Patent in light of Frenier and Reyes.

5. Dependent Claim 5

- a. “The method of claim 1 wherein the metal complexing agent includes at least one of ethylenediaminetetraacetic acid (EDTA), propylenediaminetetraacetic acid (PDTA), nitrilotriacetic acid (NTA), N-(2-hydroxyethyl)ethylenediaminetriacetic acid (HEDTA), diethylenetriaminepentaacetic acid (DTPA), hydroxyethyliminodiacetic acid (HEIDA), cyclohexylenediaminetetraacetic acid (CDTA), diphenylaminesulfonic acid (DPAS), ethylenediaminedi(o-hydroxyphenylacetic) acid (EDDHA), glucoheptonic acid, gluconic acid, oxalic acid, malonic acid, succinic acid, glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, phthalic acid, terephthalic acid, aconitic acid, carballylic acid, trimesic acid, isocitric acid, citric acid, or any salt or derivative of any of the previously listed compounds.”

257. As set forth above, Frenier discloses the metal complexing agent including several, if not all, of the acids listed in claim 5. For example, Frenier discloses that “hydroxyethylaminocarboxylic acids have the advantage of being effective in acidizing treatment of a formation without the use of a first acid.” Ex. 1004 at 6:31–33; *see also* claim 28, *id.* at 13:3–19. “Preferably, the hydroxyethylaminocarboxylic acid is selected from hydroxyethylethylenediaminetriacetic acid (**HEDTA**), hydroxyethyliminodiacetic acid (**HEIDA**), or a mixture thereof.” *Id.* at 4:13–17. (emphasis added).

258. Reyes also discloses, notably in the same order as the '843 Patent, the metal complexing agent including several, if not all, of the acids listed in claim 5. Reyes states, “as an alternative to aminopolycarboxylic acid chelating agents, traditional chelating agents such as, for example, [**EDTA, PDTA, NTA, HEDTA, DTPA, HEIDA, CDTA, DPAS, EDDHA**], **glucoheptonic acid, gluconic acid, citric acid, any salt thereof, any derivative thereof**, or the like may be included in the treatment fluids described herein.” Ex. 1005 at 10:59–67; 11:1–4 (emphasis added).

259. Additionally, I incorporate herein my statements and conclusions above with respect to Dependent Claims 2–3 regarding the metal complexing agent including citric acid and EDTA.

260. Thus, selecting one of the acids listed in claim 5 to use as the metal

complexing agent would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier alone or in combination with Reyes.

6. Dependent Claim 6

- a. **“The method of claim 1 wherein the treatment mixture comprises: between about 0.1% and 95% by weight metal complexing agent;”**

261. Frenier provides examples of “typical concentration ranges” for its treatment mixture with and without a first acid, including between 1% and 30% by weight metal complexing agent, which covers much of the claimed ranges set forth in claim 1. Ex. 1004 at 6:25–47.

262. Frenier further discloses treatment mixtures having between 15% and 28% by weight metal complexing agent and between 10% and 30% by weight metal complexing agent. *See* claims 1, 9, and 28 at Ex. 1004, 11:27–34; 11:54–63; 13:3–20.

263. However, Frenier includes that “[t]he proportion of the various components of a composition of the present invention will vary depending on the characteristics of the formation to be treated, the acid to be used, and other factors well known in the art.” *Id.* at 6:16–19. Thus, the limitation, “wherein the treatment mixture comprises: between about 0.1% and 95% by weight metal complexing agent,” would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier.

264. Additionally, Reyes states, “the concentration of the chelating agent in the treatment fluid may range between about 0.1% to about 40% of the treatment fluid by weight.” Ex. 1005 at 11:21–23.

265. Reyes similarly discloses the ranges between about 0.1% to about 5%, between about 1% to about 10%, between about 5% to about 15%, between about 0.5% to about 5%, between about 10% to about 25%, between about 10% to about 40%, and between about 30% to about 40% of the treatment fluid by weight. *Id.* at 11:23–33.

266. To the extent the ranges of the ’843 Patent are not anticipated by Frenier, it would have been obvious to a POSITA prior to the effective filing date of the ’843 Patent, given Frenier alone or in combination with Reyes, to provide a treatment mixture containing between about 0.1% and 95% by weight metal complexing agent.

267. I agree with the Examiner’s statement that “discovering the optimum or workable ranges involves only routine skill in the art.” Ex. 1002 at 249 (citing *In re Aller*, 105 USPQ 233).

b. “between 1 and 10,000 parts per million (ppm) of a corrosion inhibitor;”

268. Frenier discloses a treatment mixture including the use of additives, explicitly including corrosion inhibitors and examples thereof.

269. Frenier states, “[t]he composition can also include one or more

additives that are compatible with the acid composition, such as surfactants, **corrosion inhibitors** (such as the alkylphenones described in U.S. Pat. Nos. 5,013,483 and 5,096,618), stabilizers, solvents, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. Many such additives are well known in the art. Corrosion inhibitors in particular should be selected with reference to the particular acid used in the composition.” Ex. 1004 at 5:56–65 (emphasis added).

270. Reyes also provides a non-exclusive list of additives that are beneficial and well known in the art:

[T]he treatment fluids described herein may further comprise any number of additives that are commonly used in downhole operations including, for example, silica scale control additives, surfactants... scale inhibitors, **corrosion inhibitors**..., particulate diverters..., pH control agents (e.g., buffers), hydrate inhibitors, consolidating agents, bactericides, catalysts, clay stabilizers, breakers, delayed release breakers, and the like. Combinations of these additives can be used as well. Given the benefit of the present disclosure, one of ordinary skill in the art will be able to formulate a treatment fluid having properties suitable for a given application.”

Ex. 1005 at 11:34-54 (emphasis added).

271. Thus, it would have been obvious to a POSITA prior to the effective filing date of the '843 Patent to provide a treatment mixture including between 1 and 10,000 parts per million (ppm) of a corrosion inhibitor.

272. Additionally, it would have been obvious to a POSITA to add a

corrosion inhibitor to the compositions of Frenier to inhibit downhole corrosion. Such additives were well known and understood in the art for their predictable and beneficial results.

c. “between 1 and 10,000 ppm of a biocide;”

273. Frenier discloses a treatment mixture including the use of additives, such as a biocide. Specifically, Frenier states, “[t]he composition can also include one or more additives... [and many] such additives are well known in the art.” Ex. 1004 at 5:56–63.

274. Indeed, the use of biocides in matrix acidizing treatment solutions was well known in the art prior to April 14, 2014, and a POSITA would have known, or at least it would have been obvious, to include such additives into the treatment mixture to control microbial growth.

275. Additionally, Reyes discloses a treatment mixture including the use of additives, such as a bactericide, i.e., a “biocide.” Reyes states, “the treatment fluids described herein may further comprise any number of additives that are commonly used in downhole operations including, for example, ... **bactericides** ... [O]ne of ordinary skill in the art will be able to formulate a treatment fluid having properties suitable for a given application.” *Id.* at 11:34-54 (emphasis added).

276. It would have been known and understood, or at least obvious, to a POSITA prior to the effective filing date of the '843 Patent to provide a treatment

mixture including between 1 and 10,000 parts per million (ppm) of a biocide.

d. “between 1 and 10,000 ppm of a colloidal silica deposition inhibitor;”

277. Frenier discloses a treatment mixture including the use of additives, such as a colloidal silica deposition inhibitor: “[t]he composition can also include one or more additives... [and many] such additives are well known in the art.” Ex. 1004 at 5:56–63.

278. Indeed, the use of colloidal silica deposition inhibitors in well treatment solutions was well known in the art prior to April 14, 2014, and a POSITA would have known, or at least it would have been obvious, to include such additives into the treatment mixture to inhibit the creation of colloidal silica deposits.

279. Reyes also discloses a treatment mixture including the use of additives, such as silica scale control additives, i.e., “a colloidal silica deposition inhibitor.” Reyes states, “the treatment fluids described herein may further comprise any number of additives that are commonly used in downhole operations including, for example, **silica scale control additives** ... [O]ne of ordinary skill in the art will be able to formulate a treatment fluid having properties suitable for a given application.” Ex. 1005 at 11:34-54 (emphasis added).

280. Thus, it would have been obvious to a POSITA prior to the effective filing date of the ’843 Patent to provide a treatment mixture including between 1 and 10,000 parts per million (ppm) of a colloidal silica deposition inhibitor.

e. “between 1 and 500 gallons per thousand gallons (gpt) of a mutual solvent; ”

281. Frenier discloses a treatment mixture including the use of additives, explicitly including solvents: “[t]he composition can also include one or more additives that are compatible with the acid composition, such as... **solvents**, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. Many such additives are well known in the art.” Ex. 1004 at 5:56–63 (emphasis added).

282. In light of the above disclosure of Frenier, a POSITA would have known, or at a minimum it would have been obvious, to include mutual solvents into the treatment mixture prior to April 14, 2014.

283. Reyes discloses a treatment mixture including the use of additives, explicitly including various types of mutual solvents, such as “hydrate inhibitors.” Reyes states, “the treatment fluids described herein may further comprise any number of additives that are commonly used in downhole operations including, for example, ... **hydrate inhibitors** ... [O]ne of ordinary skill in the art will be able to formulate a treatment fluid having properties suitable for a given application.” Ex. 1005 at 11:34-54 (emphasis added).

284. It would have been obvious to a POSITA prior to the effective filing date of the ’843 Patent to provide a treatment mixture including between 1 and 500 gallons per thousand gallons (gpt) of a mutual solvent.

f. “and between 1 and 2000 parts per million of a surfactant.”

285. Frenier discloses a treatment mixture including the use of additives, explicitly including surfactants: “[t]he composition can also include one or more additives that are compatible with the acid composition, such as **surfactants**... and pH control agents. Many such additives are well known in the art.” Ex. 1004 at 5:56–63 (emphasis added).

286. Reyes also discloses a treatment mixture including the use of additives, explicitly including surfactants. Reyes states, “the treatment fluids described herein may further comprise any number of additives that are commonly used in downhole operations including, ... **surfactants** ... [O]ne of ordinary skill in the art will be able to formulate a treatment fluid having properties suitable for a given application.” Ex. 1005 at 11:34-54 (emphasis added).

287. Thus, it would have been obvious to a POSITA prior to the effective filing date of the ’843 Patent to provide a treatment mixture including between 1 and 2000 parts per million of a surfactant in light of Frenier alone or in combination with Reyes.

7. Dependent Claim 7

a. “The method of claim 1 wherein the treatment mixture further comprises between 0.1 and 95% of acid(s) as pH modifier.”

288. As set forth above in my statements and conclusions regarding dependent claim 6, Frenier discloses a treatment mixture including the use of

additives, explicitly including pH modifiers: “[t]he composition can also include one or more additives that are compatible with the acid composition, such as... **pH control agents**. Many such additives are well known in the art.” Ex. 1004 at 5:56–63 (emphasis added).

289. Additionally, Frenier provides examples of treatment mixtures comprising 0.1% to 15% by weight pH modifier (*id.* at 6:40–45), and claims 28, 31, and 49 are directed to treatment mixtures that include pH modifiers. *Id.* at 13:3–19; 13:25–27; 14:44–45.

290. Reyes also discloses a treatment mixture including the use of additives, explicitly including pH modifiers: “the treatment fluids described herein may further comprise any number of additives that are commonly used in downhole operations including, ... **pH control agents (e.g., buffers)** ... [O]ne of ordinary skill in the art will be able to formulate a treatment fluid having properties suitable for a given application.” Ex. 1005 at 11:34–54 (emphasis added).

291. It would have been obvious to a POSITA prior to the effective filing date of the ’843 Patent to provide a treatment mixture further comprising between 0.1 and 95% of acid(s) as pH modifier in light of Frenier alone or in combination with Reyes.

8. Dependent Claim 8

- a. “The method of claim 1 wherein the injecting operation further comprises: alternately injecting a first amount of

treatment mixture and a second amount of a diverting mixture into the well.”

292. Frenier discloses the use of diverting mixture during the injecting operation. Specifically, Frenier states that, during the matrix acidizing process, “[p]ackers can be used in the wellbore to control the formation zones into which the treatment fluid is injected from the wellbore.” Ex. 1004 at 7:27–29.

293. It would have been obvious to a POSITA to alternately inject the treatment mixture and a diverting mixture to better control the placement of the treatment fluid into the formation zones and avoid so-called “thief zones.”

294. The practice of acid-treating a well in conjunction with the use of a diverting mixtures was well known in the art prior to April 14, 2014.

295. For example, Borchardt notes that “[d]iverting agents assist in distributing acid more uniformly through the perforated formation interval (141). These are usually oil soluble hydrocarbon resin particles. They may be dissolved by post-acid injection of xylene or similar solvents. Oil-soluble waxes, naphthalene, and solid organic acids such as benzoic acid have also been used (142). Best results are obtained using a broad range of particle sizes.” Ex. 1015 at p. 15. “These precipitates plug flow channels and the cumulative effect is to reduce permeability in the most flooded portions of the reservoir diverting injectant to rock zones containing higher oil saturations. These precipitates plug flow channels and the cumulative effect is to reduce permeability in the most flooded portions of the

reservoir diverting injectant to rock zones containing higher oil saturations.” *Id.*

296. Moreover, Frenier incorporates by reference in its entirety U.S. Pat. No. 5,203,413 to Zerhboub, entitled “Product and Process for Acid Diversion in the Treatment of Subterranean Formations,” issued April 20, 1993 (“Zerhboub”) (Ex. 1014).

297. Zerhboub discloses alternately injecting a first amount of treatment mixture and a second amount of a diverting mixture into the well. Specifically, Zerhboub claims the invention of “[a] process for treating a relatively low permeability first zone of a subterranean formation....” Ex. 1014 at 8:44–45. The claimed invention comprising the steps of:

- (a) injecting a foam comprising an inert fluid, a gas and at least one surfactant into the subterranean formation followed by the step of

- (b) injecting a treatment fluid into the subterranean formation, the treatment fluid comprising an active fluid and at least one surfactant contained in the foam and

- (c) repeating said sequential foam and treatment fluid injecting steps (a) and (b) using an increasing volume of foam in each subsequent foam injecting step whereby the treatment fluid is diverted toward the first zone.

Ex. 1014 at 8:44–60. Zerhboub further clarifies that the injected foam of step (a) is a diverting mixture. Ex. 1014 at 4:1–5; 4:36–37 (“The principle of diversion by means of a foam is also known.”).

298. Moreover, Reyes states, “the treatment fluids described herein may

further comprise any number of additives that are commonly used in downhole operations including ... **particulate diverters**... [O]ne of ordinary skill in the art will be able to formulate a treatment fluid having properties suitable for a given application.” *Id.* at 11:34-54 (emphasis added).

299. It would have been obvious to a POSITA in light of Frenier and Reyes to alternately inject a first amount of treatment mixture and a second amount of a diverting mixture into the well to better control the placement of the treatment fluid into the formation zones and avoid so-called “thief zones.”

9. Dependent Claim 9

- a. **“The method of claim 1 wherein the injecting operation further comprises: injecting the treatment mixture until the pressure within the well reaches a predetermined target pressure calculated based on the fracture pressure of the nano-darcy shale formation;”**

300. Frenier discloses injecting the treatment mixture until the pressure within the well reaches a predetermined target pressure calculated based on the fracture pressure of the nano-darcy shale formation.

301. Notably, the ’843 Patent provides that “the target pressure may be the fracture pressure for the formation....” Ex. 1001 at 8:65–66.

302. Frenier states, “[o]ne of ordinary skill in the art will recognize that the well treatment compositions of the present invention can be used in the fracture acidizing of a formation. **By increasing the pumping pressure (to above the**

minimum in situ rock stress), a matrix acidizing treatment becomes an acid fracturing treatment.” *Id.* at 4:49–53 (emphasis added).

303. Thus, Frenier describes injecting the treatment mixture until the pressure within the well reaches fracture pressure, i.e., “a predetermined target pressure calculated based on the fracture pressure of the nano-darcy shale formation.”

304. Likewise, Frenier’s disclosure that “the injection step is preferably performed at a fluid pressure that is less than the minimum in situ rock stress (i.e., a matrix acidizing method)” provides that the predetermined target pressure calculated based on the fracture pressure of the nano-darcy shale formation is simply any predetermined pressure less than that of the fracture pressure. Ex. 1004 at 4:49–53.

305. Similarly, Reyes more clearly states, “[o]ne of ordinary skill in the art will understand how to measure the fracture gradient of a subterranean formation and **how to introduce a treatment fluid to a subterranean formation above or below this pressure.**” Ex. 1005 at 9:44–53 (emphasis added).

306. It would have been obvious to a POSITA to “inject[] the treatment mixture until the pressure within the well reaches a predetermined target pressure calculated based on the fracture pressure of the nano-darcy shale formation” because it would have been desired by those performing the method to predetermine a pressure that is less than the fracture pressure but still sufficient to cause the

treatment fluid to disperse into the subterranean formation and then inject the treatment mixture until the well reaches that predetermined pressure.

b. “and upon reaching the target pressure, shutting in the well.”

307. Frenier explicitly discloses shutting in the well upon reaching the target pressure. Specifically, Frenier states, “[a]fter the composition has been injected into the formation, optionally the well can be shut in for a period of time to allow more complete reaction between the acid and the formation material.” Ex. 1004 at 7:30–33.

308. This method would have also been obvious to POSITA, as stated by the Examiner, to “have more control over the contact time between the mixture and the well.” Ex. 1002 at p. 252; Ex. 1014 at 7:25–29 (“Stage 3: Shut-in of the well. The decompression destabilizes the foam primarily in the low permeability cell. As indicated above, the advantage of a shut-in is that diversion is accelerated.”).

309. Thus, it would have been obvious to a POSITA in light of Frenier and Reyes prior to the effective filing date of the ’843 Patent to shut in the well upon reaching the target pressure.

10. Dependent Claim 10

a. “The method of claim 1, further comprising: injecting the treatment mixture containing a metal complexing agent in combination with hydraulic fracturing and injecting into the formation in conjunction with propagation of induced fractures,”

310. Frenier states that “one can perform a stimulation technique by...

injecting chemicals through the wellbore and into the formation at pressures sufficient to fracture the formation, thereby creating a large flow channel though [sic] which hydrocarbon can more readily move from the formation and into the wellbore.” Ex. 1004 at 1:40–54.

311. Thus, it would have been obvious to a POSITA to combine the step of injecting the treatment mixture with hydraulic fracturing and injecting the treatment mixture into the formation in conjunction with propagation of induced fractures. *See* Ex. 1015 at p. 1 (“Chemicals ofv arious types are used in every stage of drilling, completing, and producing oil and gas wells.”).

312. However, Reyes explicitly discloses that treatment fluids may be used in conjunction with treatment operations such as “drilling operations, **stimulation operations, production operations, remediation operations**, sand control treatments, and the like.” Ex. 1005 at 1:13–17 (emphasis added).

313. Thus, it would have been obvious to a POSITA in light of Frenier and Reyes prior to the effective filing date of the ’843 Patent to “inject[] the treatment mixture containing a metal complexing agent in combination with hydraulic fracturing and injecting into the formation in conjunction with propagation of induced fractures.”

b. “the metal complexing agent including citric acid or EDTA.”

314. I incorporate herein my statement and conclusions with respect to

dependent claims 2, 3, and 5. Both Frenier and Reyes teach selecting either citric acid or EDTA to use as the metal complexing agent.

315. Thus, such would have been obvious to a POSITA before the effective filing date of the '843 Patent in light of Frenier alone or in combination with Reyes.

11. Dependent Claim 11

- a. **“The method of claim 1 further comprising: monitoring at least one of pH and well pressure during the maintaining operation;”**

316. Frenier explicitly discloses the use of “pressure transient analysis” during the maintaining operation: “[t]he efficiency of a matrix treatment depends primarily upon removing or bypassing regions of low permeability that restrict productivity.... **Pressure transient analysis** is a common technique for estimating the extent of damage.” Ex. 1004 at 6:49–64 (emphasis added).

317. As a POSITA would have known and understood prior to April 14, 2014, pressure transient analysis includes monitoring well pressure over time while the matrix acidizing operation is taking place.

318. Additionally, a POSITA would have known and understood, or at least it would have been obvious, to monitor at least one of the pH and well pressure during the maintaining operation to determine when the well stimulation operation had either removed the blockage in the well or the reactants in the injected fluids had

been spent.

319. There are also numerous safety practices that have been common in the industry since prior to April 14, 2014 that include monitoring at least one of the pH and well pressure during well treatment operations.

320. Monitoring at least one of the pH and well pressure while the treatment mixture contacts the subterranean formation has been common practice in the industry since well-before the effective filing date of the '843 Patent.

b. “and initiating the removing operation based on results of the monitoring operation.”

321. As mentioned above, Frenier discloses the use of “pressure transient analysis” during the maintaining operation: “[p]ressure transient analysis is a common technique for estimating the extent of damage.” Ex. 1004 at 6:49–64. Monitoring the time required for the pressurization of a well to drop back to the reservoir pressure is a direct indication of the presence or absence of a blockage or other flow impediment.

322. Thus, it would have been obvious to a POSITA before the effective date of the '843 Patent to remove the treatment mixture from the well after the contact time based on the results of the pressure transient analysis.

323. Additionally, a POSITA would have known, or at a minimum it would have been obvious to a POSITA, to perform this step so as to remove the metals from the formation before they begin to precipitate and congest the well.

324. Frenier discusses the need for avoiding precipitation after contact with the formation. Ex. 1004 at 3:23–47; 8:12–16.

12. Dependent Claim 12

- a. “The method of claim 1 further wherein the metal complexing agent is selected from glutamic acid diacetic acid (GLDA), methylglycine diacetic acid (MGDA), β -alanine diacetic acid (β -ADA), ethylenediaminedisuccinic acid, S,S-ethylenediaminedi succinic acid (EDDS), iminodisuccinic acid (IDS), hydroxyiminodisuccinic acid (HIDS), polyamino disuccinic acids, N-bis[2-(1,2-dicarboxyethoxy)ethyl]glycine (BCA6), N-bis[2-(1,2-dicarboxyethoxy)ethyl]aspartic acid (BCA5), N-bis[2-(1,2-dicarboxyethoxy)ethyl]methylglycine (MCBA5), N-tris[(1,2-dicarboxyethoxy)ethyl]amine (TCA6), N-methyliminodiacetic acid (MIDA), iminodiacetic acid (IDA), N-(2-acetamido)iminodiacetic acid (ADA), hydroxymethyl-iminodiacetic acid, 2-(2-carboxyethylamino) succinic acid (CEAA), 2-(2-carboxymethylamino) succinic acid (CMAA), diethylenetriamine-N,N''-disuccinic acid, triethylenetetramine-N,N'''-disuccinic acid, 1,6-hexamethylenediamine-N,N'-disuccinic acid, tetraethylenepentamine-N,N''''-disuccinic acid, 2-hydroxypropylene-1,3-diamine-N,N'-disuccinic acid, 1,2-propylenediamine-N,N'-disuccinic acid, 1,3-propylenediamine-N,N'-disuccinic acid, cis-cyclohexanediamine-N,N'-disuccinic acid, trans-cyclohexanediamine-N,N'-disuccinic acid, ethylenebis(oxyethylenenitrilo)-N,N'-disuccinic acid, glucoheptanoic acid, cysteic acid-N,N-diacetic acid, cysteic acid-N-monoacetic acid, alanine-N-monoacetic acid, N-(3-hydroxysuccinyl) aspartic acid, N-[2-(3-hydroxysuccinyl)]-L-serine, aspartic acid-N,N-diacetic acid, aspartic acid-N-monoacetic acid, any salt thereof, any derivative thereof, or any combination thereof.”

325. As set forth above, Frenier discloses the metal complexing agent selected from several, if not all, of the acids listed in claim 12.

326. Frenier discloses that “[i]t has been discovered that hydroxyethylaminocarboxylic acids are very soluble in mineral acids or **organic acids**, and can be mixed with mineral acids or organic acids to provide a wide range of useful formulations.” Ex. 1004 st 5:16–20 (emphasis added).

327. A POSITA would have known and understood Frenier’s disclosure of “organic acids” as metal complexing agents to include the listed biodegradable acids of claim 12.

328. Additionally, Reyes provides a nearly identical list of biodegradable chelating agents:

In this regard, suitable aminopolycarboxylic acid chelating agents may include, for example, glutamic acid diacetic acid (GLDA), methylglycine diacetic acid (MGDA), β -alanine diacetic acid (β -ADA), ethylenediaminedisuccinic acid, S,S-ethylenediaminedisuccinic acid (EDDS), iminodisuccinic acid (IDS), hydroxyiminodisuccinic acid (HIDS), polyamino disuccinic acids, N-bis[2-(1,2-dicarboxyethoxy)ethyl]glycine (BCA6), N-bis[2-(1,2-dicarboxyethoxy)ethyl]aspartic acid (BCA5), N-bis[2-(1,2-dicarboxyethoxy)ethyl]methylglycine (MCBA5), N-tris[(1,2-dicarboxyethoxy)ethyl]amine (TCA6), N-bis[2-(carboxymethoxy)ethyl]glycine (BCA3), N-bis[2-(methylcarboxymethoxy)ethyl]glycine (MCBA3), N-methyliminodiacetic acid (MIDA), iminodiacetic acid (IDA), N-(2-acetamido)iminodiacetic acid (ADA), hydroxymethyliminodiacetic acid, 2-(2-carboxyethylamino) succinic acid (CEAA), 2-(2-carboxymethylamino) succinic acid (CMAA), diethylenetriamine-N,N'-disuccinic acid, triethylenetetramine-N,N'''-disuccinic acid, 1,6-hexamethylenediamine-N,N'-disuccinic acid, tetraethylenepentamine-N,N'''-disuccinic acid, 2-hydroxypropylene-1,3-diamine-N,N'-disuccinic acid, 1,2-propylenediamine-N,N'-disuccinic acid, 1,3-propylenediamine-N,N'-disuccinic acid, cis-

cyclohexanediamine-N,N'-disuccinic acid, trans-
cyclohexanediamine-N,N'-disuccinic acid,
ethylenebis(oxyethylenenitrilo)-N,N'-disuccinic acid,
glucoheptanoic acid, cysteic acid-N,N-diacetic acid, cysteic
acid-N-monoacetic acid, alanine-N-monoacetic acid, N-(3-
hydroxysuccinyl) aspartic acid, N-[2-(3-hydroxysuccinyl)]-L-
serine, aspartic acid-N,N-diacetic acid, aspartic acid-N-
monoacetic acid, any salt thereof, any derivative thereof, or any
combination thereof. Particularly suitable biodegradable
chelating agents that may be used in the treatment fluids
described herein include, for example, MGDA, GLDA, EDDS,
 β -ADA, IDS, TCA6, BCA3, BCA5, BCA6, MCBA3, and
MCBA5.

Ex. 1005 at 10:24–58.

329. Thus, Reyes discloses the metal complexing agent selected from several, if not all, of the acids listed in claim 12. At a minimum, it would have been obvious to a POSITA in light of Frenier and Reyes prior to the effective filing date of the '843 Patent to select any of the biodegradable chelating agents listed in claim 12 as the metal complexing agent.

13. Dependent Claim 13

- a. **“The method of claim 1 further wherein the metal complexing agent is selected from acetic acid, acrylates, dihydroxymaleic acid, salts of dihydroxymaleic acid, EDTA (ethylenediamine tetraacetic acid), salts of EDTA, erythorbic acid, erythroboric acid, formic acid, gluconodeltalactone, GLDA (glutamic acid N,N-diacetic acid), salts of GLDA, HEDTA (hydroxyethylenediamine triacetic acid), salts of HEDTA, HEIDA (disodium ethanoldiglycine), salts of HEIDA, MGDA (methylglycine N,N-diacetic acid), salts of MGDA, NTA (nitriolotriacetic acid), organic metal complexers, ligands, porphyrins, pigments, peptides, saccharides, nucleic acids, phosphonic acid, polyacrylic acid**

and citric acid in an amount sufficient to sequester at least a portion of a metal compound.”

330. As set forth above, Frenier discloses the metal complexing agent selected from hydroxyethylaminocarboxylic acids, such as “hydroxyethylethylenediaminetriacetic acid (HEDTA), hydroxyethyliminodiacetic acid (HEIDA), or a mixture thereof.” Ex. 1004 at 4:13–17.

331. Moreover, I incorporate herein my statements and conclusions directed to Dependent Claims 2, 3, and 5 with respect to the metal complexing agent including citric acid and EDTA.

332. Additionally, Reyes discloses several, if not all, of the acids listed in claim 12. Reyes explicitly discloses, “as an alternative to aminopolycarboxylic acid chelating agents, traditional chelating agents such as, for example, **[EDTA, PDTA, NTA, HEDTA, DTPA, HEIDA, CDTA, DPAS, EDDHA]**, glucoheptonic acid, gluconic acid, **citric acid**, any salt thereof, any derivative thereof, or the like may be included in the treatment fluids described herein.” Ex. 1005 at 10:59–67; 11:1–4 (emphasis added) (shortened for brevity).

333. It would have been obvious to a POSITA in light of Frenier and Reyes prior to the effective filing date of the '843 Patent to select any of the chelating agents listed in claim 13 as the metal complexing agent “in an amount sufficient to sequester at least a portion of a metal compound.”

VIII. CONCLUSION

334. I hereby declare under penalty of perjury under the law of the United States of America that the foregoing is true and correct, and that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true. I understand that willful false statements are punishable by fine or imprisonment or both. 18 U.S.C. § 1001.

Dated: April 16, 2024

By: Dr. Jeffrey H. Harwell
Dr. Jeffrey H. Harwell

EXHIBIT A

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	All	Since 2019
Citations	14737	2934
h-index	61	28
i10-index	198	83

EDUCATION:

- Ph.D. (Chemical Engineering), The University of Texas at Austin, May 1983. Dissertation: "Surfactant Adsorption and Chromatographic Movement with Applications in Enhanced Oil Recovery." Supervising Professor: R.S. Schechter
- M.S. (Chemical Engineering), Texas A&M University, M.S., August 1979. Thesis: "Non-Equilibrium Model for Fixed Bed Multicomponent Adiabatic Adsorption." Supervising Professor: A.I. Liapis
- M.Div. Western Conservative Baptist Seminary, June 1977.
- B.A. (Major: Chemistry; Minor: Philosophy), Texas A&M University, May 1974.

AWARDS and DISTINCTIONS:

- Professor Emeritus, The University of Oklahoma, 2022
- National Academy of Inventors Fellow, 2014

- University of Oklahoma Intellectual Property Management Office 2013 Innovator Award for EOR Surfactant Commercialization
- Asahi Glass Chair of Chemical Engineering, The University of Oklahoma, January 2010.
- American Institute of Chemical Engineers, Fellow, 2008
- Outstanding Ground Water Remediation Project, National Ground Water Association, for Surbec Environmental, LLC, 2006.
- Distinguished Graduates Society, Honorary Member, University of Oklahoma College of Engineering, 2005.
- Soap and Detergent Association, Distinguished Paper Award, ***J. Surfactants and Detergents***, 2003, American Oil Chemists Society.
- George Lynn Cross Research Professor, The University of Oklahoma, 1999.
- Visiting Scholar, Science University of Tokyo, (Hosted by Professor Masahiko Abe), October 29 to November 18, 1996.
- Outstanding Paper Award (co-author), Spring National AIChE Meeting, New Orleans, LA, March 1996.
- Conoco/DuPont Professor of Chemical Engineering, The University of Oklahoma, July 1995.
- Outstanding Paper Award, Summer National AIChE Meeting, Minneapolis, Minnesota, August 1992.
- Co-Chairman of the 65th ACS Colloid and Surface Science Symposium, June 16-19, 1991.
- Outstanding Paper Award (co-author), American Oil Chemists Society Annual Meeting, Chicago, Illinois, May 1991.
- Principal Invited Lectures/Keynote Addresses: *Consumer Specialty Products Association, New Horizon Plenary Lecture, 2014; Japan Research Institute of Material Technology Lectureship Award, Tokyo, Japan, December 4-5, 1998; Interfaces Against Pollution, Wageningen, The Netherlands, August 10-13, 1997; Structured Fluids and Interfaces, Engineering Foundation Conference, Lucca, Italy, January 12-17, 1997; Plenary Lecture, Japanese Oil Chemists Society, Tsukuba, Japan, October 31-November 1, 1996; Separation Technology VI: Advances and Opportunities in Environmental Separations, Engineering Foundation Conference, Snowbird, Utah, July 22-27, 1995; Kodak Symposium on Surfactant Adsorption and Surface Solubilization, 208th ACS National Meeting, Washington, D.C., August 21-25, 1994; NATO Advanced Studies Institute: Migration and Fate of Pollutants in Soils and Subsoils, Maratea, Italy, May 24-June 6, 1992; EUCHEM Workshop on Adsorption of Surfactants and Macromolecules from Solution, Abo (Turku), Finland, June 12-14, 1989; 2nd International Conference on The Industrial Applications of Surfactants, Royal Society of Chemistry, The University of Salford, Salford, England, April 18-20, 1989; Victor K. LaMer Award Lecture, 58th Colloid and Surface Science*

Symposium, Carnegie-Mellon University, Pittsburgh, PA, June 11-13, 1984.

- Victor K. LaMer Award of the Colloid and Surface Science Division of the American Chemical Society, 1984.
- Welch Foundation Scholar (1970-74).
- National Merit Finalist (1970).

EXPERIENCE:

- President, Surfactant Associates, Inc., Norman, Oklahoma, 1987 to Present.
- Asahi Glass Chair in Chemical Engineering, 2010 to 2021.
- Founder, Chemical Flooding Technologies, LLC.
- Director of Engineering, Surbec Environmental, LLC, May 2007 to 2008.
- ConocoDuPont/ConocoPhillips Professor of Chemical Engineering, 1992 to December 2010.
- Associate Dean of Engineering, The University of Oklahoma, 1998 to June 2005.
- Director, School of Chemical Engineering and Materials Science, The University of Oklahoma, 1991 to 1998.
- Professor of Chemical Engineering, School of Chemical Engineering and Materials Science, University of Oklahoma; June 1992 to Present.
- Program Director, Interfacial, Transport and Separation Processes Program, The National Science Foundation, Washington, D.C.; July 1988 to July 1989.
- Principal, SouthWest NanoTechnologies, LLC, Norman, Oklahoma, January 2003 to present.
- Principal, Surbec Environmental, LLC., Norman, Oklahoma; December 1996 to present.
- Associate Professor of Chemical Engineering, School of Chemical Engineering and Materials Science, University of Oklahoma; August 1987 to 1992.
- Assistant Professor of Chemical Engineering, School of Chemical Engineering and Materials Science, University of Oklahoma; November 1982 to July 1987.

INDUSTRIAL CONSULTING/R&D CONTRACTS:

ExxonMobil, Janssen Pharmaceuticals, Shell Chemicals, DeepStar Consortium, InVia Paving Technologies, MWV Chemicals, SC Johnson, Ductil, Elk Petroleum, EA Engineering, Sinotech Engineering, Castrol, Schlumberger-Dowell, Dow Chemical Company, DuPont Specialty Chemicals, DuPont Environmental Remediation Services, Boerringer-Manheim, Weyerhaeuser, PPG Industries,

Church & Dwight, Proctor & Gamble, Clorox, Alpha Metals, Pickering EnviroRem, Inc., Kerr McGee, Arco Oil and Gas, Shell Development Company, Shell Chemicals, ExxonMobil Chemicals, Mobil Oil Company, Phillips Petroleum, Glaxo/Welcome, IBAH, Inc. EIC Laboratories, Inc., Hitachi Research, Kiwi Brands, James River Corporation, Eastman Kodak, Hart Chemical, Sandoz Chemicals, Lubrizol Corporation, Nease Corporation, Baker-Hughes, SASOL North America, Stepan Chemical.

ON-SITE PRESENTATIONS OF *APPLIED SURFACTANT SCIENCE AND TECHNOLOGY* SHORT COURSE:

Baker-Hughes, Church & Dwight, GSK, Ecolab, Novozymes, Genentech, Unilever Europe, Unliever North America, Henkel North America, Dial Corporation, Parker/AmChem, Proctor & Gamble Co./Cincinnati, Proctor & Gamble Co./Kobe, Japan, Dow Chemical Company, Monsanto, DuPont, The Clorox Company, 3M Company, Cytec, Kimberly Clark, Varn Products, Reckitt Benckiser, Sinotech Engineering, SASOL North America, Stepan Chemical.

PROFESSIONAL SOCIETIES:

- American Institute of Chemical Engineers (AIChE)
- American Chemical Society (ACS)
- American Oil Chemists Society (AOCS)
- Society of Petroleum Engineers (SPE)

RESEARCH CONTRACTS:

“In situ CO₂ Generation,” \$375,000, DeepStar Consortium, Chevron Technology Corporation, co-PI Ben Shiau, Petroleum Engineering, April 2015 to March 2017.

“Properties of Nanotube Fluoropolymer Composites,” Asahi Glass Corporation; co-PI Brian P. Grady, Chemical Engineering, Univ. Oklahoma; January 2012 to December 2016.

“Interfacially Active SWNT/Silica Nanohybrids,” Advanced Energy Consortium, \$1,700,000; PI Ben Shiau, Petroleum Engineering, Co-PI Steve Crossly, Chemical Engineering, Univ. Oklahoma; May 2009 to October 2016.

”Center for Interfacial Reaction Engineering,” \$6,022,000, PI Daniel E. Resasco; co-PIs Friederike C. Jentoft, Khaled A.M. Gasem (OSU), and Sanwu Wang (U Tulsa); DOE/EPSCoR, September 2010 to July 2017.

"Investigation of Asphaltene Compounds as Surface Active Moieties," \$272,755,

PI Brian Grady; Phillips 66, January 2011 to October 2016.

"Exploration of Surfactant Properties after Chemical Modification of a New Molecule," \$494,114, PI Brian Grady; Novus International, January 2011 to June 2016.

"Properties of Evotherm and Asphalt Affecting Adhesion for Various Aggregate Sources," InVia Pavement Technologies, LLC, \$137,000, co-PI Brian Grady, CBME, September 2013 to August 2014.

"EAGER: Effects of Thermodynamic Phase Changes at Reservoir Conditions on the Interfacial Properties of Chemicals Used in Hydraulic Fracturing," National Science Foundation, \$95,183; co-PI B. Shiau, MPGE, B. Grady, CBME, University of Oklahoma, October 2012 to September 2014.

"Reduction of Uncertainty in Surfactant-Flooding Pilot Design using Multiple Single Well Tests, Fingerprinting, and Modeling," Research Partnership to Secure Energy for America (RPSEA), \$1,036,205; PI Ben Shiau, Petroleum Engineering, Univ. Oklahoma; co-PI A. Jamili, Petroleum Engineering, Univ. Oklahoma, January 2013 to December 2014.

"Development and Optimization of New Surfactant Formulations for Environmental Remediation," \$177,000, PI Ben Shiau; Surbec Environmental, LLC, September 2010 to January 2013.

"Boosting Oklahoma Oil Production with Next Generation Surfactant Technology," EDGE, \$2,000,000; co-PI Ben Shiau, Petroleum Engineering; January 2010 to December 2012.

"Development of Carbon Dioxide Infused Fire Fighting Foam for Euthanasia for Layer Chicken," \$49,500, PI J.H. Harwell; co-PIs Ben Shiau, Bruce Roberts; USDA/Texas A&M University, November 2010 to October 2011.

"Boosting Domestic Oil Production with Next Generation Surfactant Technology," US Department of Energy, \$489,000, co-PI Ben Shiau, Petroleum Engineering; April 2010 to March 2012.

"Chemical EOR Feasibility Study Proposal for Grieve Field, Natrona County, Wyoming," Elk Petroleum Corporation, \$120,000; co-PI Ben Shiau, Dept. Petrol. Eng., Univ. Oklahoma, August 2008 to July 2009.

"Nanostructured Microemulsions as Alternative Solvents to VOCs in Cleaning

Technologies and Vegetable Oil Extraction," Environmental Protection Agency, \$314,966, July 2003 to June 2007. Investigators: D.A. Sabatini, J. Harwell.

"Center for Application of Single-Walled Carbon Nanotubes," U.S. Department of Energy, \$962,000 May 2006 to May 2008. Investigators: Resasco, Daniel E.; Schmidtke, David W.; Harwell, Jeffrey H.; Scamehorn, John F.; McFetridge, Peter S.; Rennaker, Robert L. II.; Olson, Brandon W.; Striolo, Alberto

"Drag Reduction Using Surface-Attached Polymer Chains and Nanotubes," Oklahoma State Regents for Higher Education – EPSCoR, \$39,150, July 2004 to June 2005. Investigators: K. Parthasarathy, D. Papavassiliou, J. Newman, J. Harwell, L. Lee

"Catalytic Production of Carbon Nanotubes," Conoco, \$156,000, February 15, 1999 to February 15, 2001. Principal Investigator: D.E. Resasco.

"Large-Scale Production of Single-Wall Carbon Nanotubes", Oklahoma Center for the Advancement of Science and Technology, \$62,000, September 01, 2000 to August 30, 2001. Principal Investigator: D.E. Resasco.

"A Field Demonstration of Surfactant-Enhanced DNAPL Remediation," U.S. EPA (CR 825686-01-0), \$300,020, October 1, 1997 to September 30, 2000. Co-Principal Investigators: R.C. Knox and D.A. Sabatini.

"Surfactant-Enhanced Treatability Study at Alameda Point." Surbec Environmental, LLC, \$40,000, 3/99 to 2/00, CO-PI (33%), R.C. Knox, D.A. Sabatini and J.H. Harwell

"Surfactant-Enhanced Subsurface Remediation of Petroleum Hydrocarbons – McLelland." Surbec Environmental, LLC, \$50,000, 1/99 to 12/99, CO-PI (30%), R.C. Knox, D.A. Sabatini and J.H. Harwell

"Fundamentals of Admicellar Polymerization," National Science Foundation (CTS-9521985), \$265,000, April 15, 1996 to March 31, 1999. Co-Principal Investigators: B.P. Grady and E.A. O'Rear.

"Graduate Research Traineeships in Environmentally Friendly Natural Gas Technologies," National Science Foundation, \$555,500, September 15, 1994 to August 31, 1999. Co-Principal Investigator: L.L. Lobban.

"Applications of Photocatalysis in Air Conditioning," OCAST, \$97,000. July 1, 1998 to June 30, 1999. Co-principal investigators: L.L. Lobban and G.K.

Newman.

“Applications of Photocatalysis in Air Conditioning,” International Environmental Corporation, \$25,000. July 1, 1998 to June 30, 1999. Co-principal investigators: L.L. Lobban and G.K. Newman.

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INVITED OR KEYNOTE PRESENTATIONS:

49. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Air Force Center for Environmental Excellence, San Antonio, Texas, January 24, 2006.

48. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Iowa DEQ, Des Moines, IO, January 31, 2006.

47. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Oklahoma Ground Water Association Annual Meeting, Oklahoma City, OK, February 23, 2006.

46. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," EPA National Tanks Conference, Memphis, TN, March 20-22, 2006.

45. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Altus AFB, Altus, OK, 29-Mar, 2006.

44. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," EPA Region 3 Annual Meeting, Reston, VA, 3-Apr, 2006.

43. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Army Corps of Engineers, Brocken Arrow, OK, 18-Apr, 2006.

42. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Flying J Corporate Environmental Group, Salt Lake City, NV, 2-May, 2006.

41. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Colorado Department of Environmental Protection, Petroleum Storage Tank Division, Denver, CO, 18-May, 2006.
40. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Inter Tribal Environmental Consortium, Oklahoma Convention, Oklahoma City, OK, June 20-21, 2006.
39. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Nebraska DEQ, Lincoln, NE, July 25-26, 2006.
38. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," ExxonMobil Greenpoint Remediation Group, Brooklyn, NY, 31-Jul, 2006.
37. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," New York Department of Environmental Conservation, Albany, NY, 16-Aug, 2006.
36. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," MWH Engineering, Environmental Division, Des Moines, IA, August 31, 2006.
35. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Senaca Engineering, Environmental Division, Des Moines, IA, August 31, 2006.
34. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Illinois Department of Environmental Quality, Springfield, IL, September 13, 2006.
33. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," New Mexico Inter Tribal Environmental Consortium Annual Meeting, Albuquerque, NM, October 26, 2006.
32. B. Shiau, M. O'Kelley, J. Harwell, "Surfactant Enhanced Aquifer Remediation: Theory and Field Applications," Environmental Professionals of Iowa Annual Meeting, Des Moines, IA, November 15-16, 2006.
31. B. Shiau, M. O'Kelley, J. Harwell, D. Sabatini, B. Knox, T. Jones, "Surfactant

Enhanced Aquifer Remediation: Theory and Field Applications,” National Ground Water Association Annual Meeting, Las Vegas, NV, December 5-8, 2006.

30. J.H. Harwell, D.A. Sabatini, and R. Knox, “Remediation of soils and aquifers with surfactants,” Symposium on Surfactants, Polymers, and Colloids in the Aquatic Environment, 220th ACS National Meeting, Washington, D.C., August 20-24, 2000.

29. Gerard Newman, Robert Shambaugh, J.H. Harwell, Newman Technologies, “Aligned, Carbon Nanotube/Polymer Composite Fabrics,” NanoSpace 2000 – Advancing Human Frontier, The Third annual International Conference, Integrated Nano/Microtechnology for Space Applications, Houston, Texas, January 2000.

28. J. H. Harwell, “Oil, Dirt, and Linkers: The True Story of My Research Career,” Environmental Sciences, Engineering, and Policy in the 21st Century Seminar Series, University of Michigan, April 2000.

27. J.H. Harwell and D.A. Sabatini, “The Role of Surfactants in Environmental Remediation,” Interfaces Against Pollution, Wageningen, The Netherlands, August 10-13, 1997.

26. J.H. Harwell and D.A. Sabatini, “Surfactants in Cleaning Our Soil and Water”, Structured Fluids and Interfaces, Engineering Foundation Conference, Lucca, Italy, January 12-17, 1997

25. J.H. Harwell, “Using Surfactants to Clean on Soil and Water,” Plenary Lecture, Japanese Oil Chemists Society, Tsukuba, Japan, October 31-November 1, 1996.

OTHER PRESENTATIONS AT NATIONAL OR INTERNATIONAL SYMPOSIA:

179. Ion specific effects in Winsor III microemulsions, B. Trotter, M. Kadhum, B. Shiau, J. Harwell, 253rd ACS National Meeting in San Francisco, California, April 2-6, 2017.

178. An Application of the Hydrophilic-Lipophilic Difference Concept in Surfactant Formulations for Enhanced Oil Recovery. P. Lohateeraparp, B. Shiau, J. Harwell, 103rd AOCS Annual Meeting, Long Beach, CA, April 29 to May 2, 2012.

7. Designing Surfactant Formulations for Oil Recovery from a High Salinity Reservoir: Single-well Test. T-P. Hsu, B-J. Shiau, P. Lohateeraparp, J. H. Harwell,

B. L. Roberts, 103rd AOCS Annual Meeting, Long Beach, CA, April 29 to May 2, 2012.

176. Surfactant Flushing for Aquifer Remediation at Ft. Drum, New York. J.H. Harwell, B.J. Shiau, P. Lohateeraparp, T.P. Hsu, 102nd AOCS Annual Meeting, Cincinnati, Ohio, May 1-4, 2011.

175. Advanced Microemulsion Systems for Subsurface Remediation: Laboratory and Field Results. D.A. Sabatini, J.H. Harwell, B.J. Shiau, R.C. Knox, 102nd AOCS Annual Meeting, Cincinnati, Ohio, May 1-4, 2011.

174. Surfactant Formulations for Chemical Flooding under High Salinity Reservoir Conditions. B.J. Ben Shiau, Tzu-Ping Hsu, Prapas Lohateeraparp, Bruce L. Roberts, Jeffrey H. Harwell, 102nd AOCS Annual Meeting, Cincinnati, Ohio, May 1-4, 2011.

173. Thermodynamics of Mixed Anionic/Nonionic Surfactant Adsorption on Alumina, J.J. Lopata, K.M. Werts, J.F. Scamehorn, J.H. Harwell, B.P. Grady, 100th AOCS Annual Meeting, Orlando, Florida, May 3-6, 2009.

172. Microemulsion-based Detergency: Cost-effective and Environmentally-friendly Formulations Optimized for High-unsaturated-content Vegetable Oils. T. Phan, J. Harwell, D. Sabatini, 100th AOCS Annual Meeting, Orlando, Florida, May 3-6, 2009.

171. Microemulsion Properties and Cleaning Efficiency of Extended Surfactant-Based Mixtures., A. Witthayapanyanon^{1,3}, T. Phan^{2,3}, J.H. Harwell^{1,3}, D.A. Sabatini^{2,3}, 99th AOCS Annual Meeting, Seattle, Washington, May 18-21, 2008.

170. Tri T. Phan, Jeffrey H. Harwell, and David A. Sabatini, "A Systematic Study of Triglyceride Molecular Structure Effects on Surfactant-oil Interactions," The 81st ACS Colloid & Surface Science Symposium, June 24-27, 2007.

169. A. Witthayapanyanon, J.H. Harwell, D.A. Sabatini, "Robust Microemulsion Systems Using Extended Surfactants," 98th AOCS Annual Meeting, Quebec City, Canada, May 13-16, 2007.

168. T.T. Phan, J.H. Harwell, D.A. Sabatini, "A Systematic Study of Triglyceride

Molecular Structure Effects on Microemulsion Formation,” 98th AOCS Annual Meeting, Quebec City, Canada, May 13-16, 2007.

167. Sukhwan Soontravanich, John F. Scamehorn, Jeffrey H. Harwell, "Monomer-Micelle Equilibrium Compositions in an Anionic/Amphoteric Surfactant Mixture," 97th AOCS Annual Meeting, St. Louis, Missouri, April 30-May 3, 2006.

166. Sukhwan Soontravanich, John F. Scamehorn, Jeffrey H. Harwell, "Precipitation of a Mixture of an Anionic and an Amphoteric Surfactant: Experimental Phase Boundaries and Theoretical Predictions," 97th AOCS Annual Meeting, St. Louis, Missouri, April 30-May 3, 2006.

165. L. Do, A. Witthayapanyanon, E. Acosta, J.H. Harwell, and D.A. Sabatini, "Aqueous-Based Microemulsions as an Alternative to Solvent Replacements," 97th AOCS Annual Meeting, St. Louis, Missouri, April 30-May 3, 2006.

164. L.D. Do, A. Witthayapanyanon, J.H. Harwell, D.A. Sabatini, and E. Acosta, "Aqueous Extended-Surfactant-Based Technology for Edible Oil Extraction," 97th AOCS Annual Meeting, St. Louis, Missouri, April 30-May 3, 2006.

163. S. Soontravanich, J.F. Scamehorn, and J.H. Harwell, "Monomer-Micelle Equilibrium Compositions in an Anionic/Amphoteric Surfactant Mixture," 97th AOCS Annual Meeting, St. Louis, Missouri, April 30-May 3, 2006.

162. S. Soontravanich, J.F. Scamehorn, and J.H. Harwell, "Precipitation of a Mixture of an Anionic and an Amphoteric Surfactant: Experimental Phase Boundaries and Theoretical Predictions," 97th AOCS Annual Meeting, St. Louis, Missouri, April 30-May 3, 2006.

161. E. Acosta, Sh. Yuan, D. Sabatini, J. Harwell, "Formulation of Lecithin-Based Non-Toxic Microemulsions for Drug Delivery Applications," 97th AOCS Annual Meeting, St. Louis, Missouri, April 30-May 3, 2006.

160. Linh D. Do, Anuradee Witthayapanyanon, Jeffrey H. Harwell, David A. Sabatini, "Self-Assembling of Linker-based Microemulsions," 80th ACS Colloid and Surface Science Symposium, Boulder, CO, June 18-21, 2006.

159. A. Witthayapanyanon, T. Phan, E.J. Acosta, J.H. Harwell, D.A. Sabatini, "Self

Assembly of Linker-based Microemulsions,' 229th ACS National Meeting, San Diego, CA, March 13-17, 2005.

158. A. Witthayapanyanon, L. Do, E.J. Acosta, J.H. Harwell, D.A. Sabatini, "Advanced Microemulsion for Solvent Replacement," 229th ACS National Meeting, San Diego, CA, March 13-17, 2005.

157. E. Szekeres, E. Acosta, D. Sabatini, J. Harwell, "How to predict enhanced-solubilization in linker-modified limonene microemulsions," 95th AOCS Annual Meeting, May 9-12, 2004, Cincinnati, Ohio.

156. E. Acosta, D. Sabatini, J. Harwell, "Lecithin-based non-toxic microemulsions for food and drug delivery applications," 95th AOCS Annual Meeting, May 9-12, 2004, Cincinnati, Ohio.

155. David A. Sabatini, Anuradee Witthayapanyanon, Edgar J. Acosta, Jeffrey H. Harwell, "Formulation And Characterization Of Nanostructured Microemulsions As Solvent Replacement Systems," 227th ACS National Meeting, March 28-April 1, 2004, Anaheim, California.

154. A. Witthayapanyanon¹, L. Do¹, T. Phan, J.H. Harwell, D.A. Sabatini, "Microemulsion Formation with Triglyceride Oils," 95th AOCS Annual Meeting, May 9-12, 2004, Cincinnati, Ohio.

153. E. Acosta¹, D. Sabatini¹, J. Harwell¹, J. Scamehorn¹, A. Witthayapanyanon, "Surfactant-Based Alternative Solvents: Phase Behavior and Performance Studies," 95th AOCS Annual Meeting, May 9-12, 2004, Cincinnati, Ohio.

152. E. Acosta*¹, D. Sabatini¹, J. Harwell¹, E. Szekeres, "Use of the Net-Average Curvature Model for Predicting Mixed Surfactant Microemulsion Performance in Cleaning Formulations," 95th AOCS Annual Meeting, May 9-12, 2004, Cincinnati, Ohio.

151. E. Acosta, D.A. Sabatini and J.H. Harwell, "Predicting Microemulsion Properties with the Net-Average Curvature Model," 226th ACS National Meeting, September 7-11, 2003, New York, NY.

150. E. Acosta, D.A. Sabatini and J.H. Harwell, "Linker-Based Non-Toxic Microemulsions," 226th ACS National Meeting, September 7-11, 2003, New York, NY.
149. E. Szekeres, D.A. Sabatini and J.H. Harwell, "A Two-State Model for Selective Solubilization in Water – Surfactant – Mixed Oil System," AIChE 2002 Annual Meeting, November 3-8, Indianapolis, IN.
148. E. Acosta, D.A. Sabatini and J.H. Harwell, "Modeling Microemulsion Solubilization and Interfacial Tension: The Net-Average Curvature Model," AIChE 2002 Annual Meeting, November 3-8, Indianapolis, IN.
147. E. Acosta, D.A. Sabatini and J.H. Harwell, "Combined Linker Effect: A Novel Type of Self-Assembly in Microemulsions," 224th ACS National Meeting, Boston, MA, August 18-22, 2002.
146. M. Dreyer, L.L. Lobban, G. Newman, and J.H. Harwell, "Photocatalysis on high-porosity, low-density UV titanium dioxide aerogels for indoor air decontamination," 220th ACS National Meeting, Washington, D.C., August 20-24, 2000.
145. J.H. Harwell, B. Shiau, M. Hasegawa, D. Sabatini, R. Knox, R. Lago, L. Yeh and R. Sillan, "Surfactant-Enhanced Subsurface Remediation of DNAPLs at the Former Naval Air Station Alameda, California," The Second International Conference on Remediation of Chlorinated and Recalcitrant Compounds, Monterey, California, May 22-25, 2000.
144. R.C. Knox, D.A. Sabatini, J.H. Harwell, and B.J. Shiau, "Surfactant-Enhanced Subsurface Remediation at Hill AFB", University of Tubingen, Tubingen, DE, March 14, 1998.
143. R.C. Knox, D.A. Sabatini, and J.H. Harwell, "Enhanced Contaminant Extraction", Surfactant Workshop, University of Stuttgart, Stuttgart, DE, March 11, 1998.
142. D.A. Sabatini, J.H. Harwell, and R.C. Knox, "Surfactant Enhanced Subsurface Remediation: Laboratory and Field Observations." The University of Strasbourg, Strasbourg, France, December 19, 1997.
141. D.A. Sabatini, J.H. Harwell, and R.C. Knox, "Surfactant-Enhanced In Situ Ground Water Remediation." Separations Technology VII: Separations for Clean Production, Engineering Foundation Conference, Davos, Switzerland, October 26-

31, 1997.

140. D.A. Sabatini, J.H. Harwell, R.C. Knox, and B.J. Shiau, "Surfactant Selection Criteria for Enhanced Subsurface Remediation: Laboratory and Field Observations." German Colloid Chemistry Society Meeting, University Essen, Essen, DE. October 2, 1997.

139. D.A. Sabatini, J.H. Harwell, R.C. Knox, and B.J. Shiau, "Surfactant Selection Criteria for Enhanced Subsurface Remediation: Laboratory and Field Observations." German Colloid Chemistry Society Meeting, University Essen, Essen, DE. October 2, 1997.

138. B.J. Shiau, H.K. Karapanagioti, R.C. Knox, D.A. Sabatini, and J.H. Harwell, "Solubilization and Mobilization of Mixed NAPL Wastes Using Surfactants", Emerging Technologies in Hazardous Waste Management IX, Pittsburgh, PA, Sept. 15-17, 1997.

137. D.A. Sabatini, R.C. Knox, and J.H. Harwell, "Surfactant-Enhanced Subsurface Remediation: Laboratory and Field Observations." The R. S. Kerr Environmental Research Laboratory, Ada, OK, July 31, 1997.

136. R.C. Knox, D. A. Sabatini and J.H. Harwell, "Long-Term Implications of Chemical Extractants Used for Subsurface Remediation", 17th Annual Meeting of the International Association for Impact Assessment, New Orleans, LA, May 28-31, 1997.

135. J.H. Harwell, D.A. Sabatini, J. O'Haver, and R.C. Knox, "ATDF Demonstrations: Surfactant Recovery", Research Technologies Development Forum, In Situ Flushing Action Team, Hill AFB, UT, May 8-9, 1997.

134. R.C. Knox, D.A. Sabatini, J.H. Harwell, and B.J. Shiau, "SERDP Enhanced Source Removal: Surfactant Solubilization", RTDF In-Situ Flushing Action Team Meeting, Hill Air Force Base, UT, May 8-9, 1997.

133. D.A. Sabatini, J.H. Harwell, R.C. Knox, and B.J. Shiau, "SERDP Enhanced Source Removal: Surfactant Mobilization", RTDF In-Situ Flushing Action Team Meeting, Hill Air Force Base, UT, May 8-9, 1997.

132. P.M. Hunt, B.P. Grady, E.A. O'Rear, and J.H. Harwell, "Kinetics of Admicellar Polymerization: Adsolubilized Styrene Initiated by AIBN," 215th ACS National Meeting, Dallas, Texas, March 29 - April 2, 1998.

131. J.H. O'Haver, B. Kitiyanan, J.H. Harwell, and D.A. Sabatini, "The Use of Liquid-Liquid Extraction for the Separation of Organic Contaminants from Groundwater-Surfactant Streams," 215th ACS National Meeting, Dallas, Texas, March 29 - April 2, 1998.
130. G.K. Newman, B. Gunturi, C. Crowe, G. Fillippi, M. Powers, J. Goldberg and J.H. Harwell, "Particle Removal from Surfaces via a Surfactant Modified Fluorocarbon: Application to Oxygen Delivery Lines," 215th ACS National Meeting, Dallas, Texas, March 29 - April 2, 1998.
129. B. Wu, D.A. Sabatini, J.H. Harwell, and B.J. Shiau, "Formulating >Environmentally Acceptable= Middle Phase Microemulsion Systems with Petroleum Hydrocarbons," 215th ACS National Meeting, Dallas, Texas, March 29 - April 2, 1998.
128. G.K. Newman, P.D. Newman, and J.H. Harwell, "A New Nano-Thin Photoresist Technology Based on Surfactant Templating for Use in Semiconductor Lithography," National AIChE Annual Meeting, Los Angeles, CA., Nov. 16-21, 1997.
127. G.K. Newman, B. Fillipi, J.H. Harwell, S.J. Kersey, "Enhanced Gas Storage in High Density, Monolithic Carbon Aerogels," National AIChE Annual Meeting, Los Angeles, CA., Nov. 16-21, 1997.
126. B.V.S. Gunturi, G.K. Newman, A. Tabatabai, J.S. Goldberg, A.C. Hepp, M.R. Powers, and J.H. Harwell, "Simulation of a System Using Near and Supercritical Fluids for Cleaning of Process Lines," National AIChE Annual Meeting, Los Angeles, CA., Nov. 16-21, 1997.
125. S.J. Kersey, G.K. Newman, L.L. Lobban, and J.H. Harwell, "Enhanced Photocatalysis: Adjusting the Ratio of Silicon and Titanium in a Binary $\text{TiO}_2/\text{SiO}_2$ Aerogel to Increase Catalyst Activity," National AIChE Annual Meeting, Los Angeles, CA., Nov. 16-21, 1997.
124. G.K. Newman and J.H. Harwell, "Gas Adsorption on Fullerene Adsorbents," National AIChE Annual Meeting, Los Angeles, CA., Nov. 16-21, 1997.
123. G.K. Newman, L.L. Lobban, J.H. Harwell, and M. Dreyer, "Ultra Low Density, Semi-Transparent Photocatalytic Aerogels: Their synthesis, Characterization and Commercial Potential," National AIChE Annual Meeting, Los Angeles, CA., Nov. 16-21, 1997.

122. G.K. Newman, S.J. Kersey, and J.H. Harwell, "Enhanced Oxidation of VOCs in a Novel TiO₂/Fluorocarbon Reactor," National AIChE Annual Meeting, Los Angeles, CA., Nov. 16-21, 1997.

121. B.J. Shiau, B. Wu, H.K. Karapangioti, R.C. Knox, D.A. Sabatini, and J.H. Harwell, "Solubilization and Mobilization of Mixed NAPL Wastes Using Surfactants," Emerging Technologies in Hazardous Waste Management IX, Pittsburgh, Pennsylvania, September 15-17, 1997.

120. B.J. Shiau, D.A. Sabatini, R.C. Knox, and J.H. Harwell, "Surfactant-Enhanced Mobilization of Mixed NAPL Wastes at Hill AFB: A Case Study," 213th American Chemical Society National Meeting and Exposition, San Francisco, CA., April 13-17, 1997.

119. B.J. Shiau, D.A. Sabatini, R.C. Knox, and J.H. Harwell, "Surfactant-Enhanced Solubilization of Mixed NAPL Wastes at Hill AFB: A Case Study," 213th American Chemical Society National Meeting and Exposition, San Francisco, CA., April 13-17, 1997.

118. S.W. Horstkamp, K.E. Starling, J.H. Harwell, and R.G. Mallinson, "High Energy Density Storage of Natural Gas for Transportation," Second Joint China/US Chemical Engineering Conference, Beijing, China, May 19-22, 1997.

117. M. Ioneva, J.H. Harwell, R.G. Mallinson, "Methane Storage in Controlled Porosity Sorbents," National AIChE Meeting, Houston, TX, March 9-13, 1997.

116. N. Tharapiwattananon, J.F. Scamehorn, S. Osuwan, J.H. Harwell, and K.J. Haller, "Surfactant Recovery from Water Using Foam Fractionation," 88th American Oil Chemists Society Annual Meeting, Seattle, Washington, May 11-15, 1997.

115. S.W. Horstkamp, K.E. Starling, J.H. Harwell, and R.G. Mallinson, "High Energy Density Storage of Natural Gas for Transportation," Gas Processors Association 76th Annual convention, San Antonio, Texas, March 10-12, 1997.

114. M.A. Ioneva, R.G. Mallinson, J.H. Harwell, "Sorptive Storage and Separation of Light Hydrocarbon vapors by Capillary Condensation in MCM-41 Type Mesoporous Materials," Materials Research Society Fall Annual Meeting, Boston, Massachusetts, December 2-6, 1996

113. M Ioneva, J. Harwell, R. Mallinson, "Organic Guest Templated Sorbents

with Controlled Micro and Mesoporosity,” National AIChE Meeting, Chicago, Illinois, November 10-15, 1996

112. M Ioneva, J. Harwell, R. Mallinson, “Adsorptive Storage of Methane in Sorbents with Controlled Porosity,” National AIChE Meeting, Chicago, Illinois, November 10-15, 1996

111. B.P. Grady, J. Reynolds, E.A. O’Rear, and J.H. Harwell, “Admicellar Polymerization--A Novel Way to Improve Polymer-Filler Adhesion in Polymer-Matrix Composites,” National AIChE Meeting, Chicago, Illinois, November 10-15, 1996

110. B.P. Grady, J. Reynolds, E.A. O’Rear, and J.H. Harwell, “Admicellar Polymerization--A Novel Way to Improve Polymer-Filler Adhesion in Polymer-Matrix Composites,” National AIChE Meeting, Chicago, Illinois, November 10-15, 1996.

109. E.A. O’Rear, C. Lai, J.H. Harwell, “Perfluoroheptanoic Acid Reversed Micelle Formation in 1,1,2-Trichlorotrifluoroethane,” National AIChE Meeting, Chicago, Illinois, November 10-15, 1996.

108. M Ioneva, J. Harwell, R. Mallinson, “Storage of Light Hydrocarbon Vapors by Capillary Condensation in MCM-41 Type Mesoporous Sorbents with controlled Porosity,” National AIChE Meeting, Chicago, Illinois, November 10-15, 1996

107. R.C. Knox, D.A. Sabatini, A. Thal, and J.H. Harwell, “Surfactant-Enhanced Solubilization of Mixed NAPL Wastes: A Case Study,” Emerging Technologies in Hazardous Waste Management VI, Birmingham, Alabama, September 9-12, 1996.

106. B. Krebs-Yuill, J.H. Harwell, D.A. Sabatini, G.E. Quinton, and S.H. Shoemaker, “Economic Study of Surfactant-Enhanced Pump-and-Treat Remediation,” 69th Annual Water Environment Federation Conference, Dallas, Texas, October 5-9, 1996.

105. C.L. Lai, E.A. O’Rear, and J.H. Harwell, “Adsolubilization of Fluorocarbon Alcohols into Perfluoroheptanoate Admicelles Formed on Alumina,” Emerging Technologies in Hazardous Waste Management VI, Birmingham, Alabama, September 9-12, 1996.

104. B.J. Shiau, S. Deshpande, H.K. Karapanagioti, D. Wade, D.A. Sabatini, and J.H. Harwell, “Surfactant Selection Protocol for Optimizing Ex Situ Soil Washing,” Emerging Technologies in Hazardous Waste Management VI,

Birmingham, Alabama, September 9-12, 1996.

103. B.J. Shiau, B. Wu, H.K. Karapanagioti, R.C. Knox, D.A. Sabatini, and J.H. Harwell, "Use of Surfactants for Aquifer Remediation of Petroleum Wastes," Emerging Technologies in Hazardous Waste Management VI, Birmingham, Alabama, September 9-12, 1996.

102. D.A. Sabatini, R.C. Knox, J.H. Harwell, and B.J. Shiau, "Surfactant Enhanced NAPL Remediation: From the Laboratory to the Field," ***International Workshop on Soil and Aquifer Pollution--Non-Aqueous Phase Liquids---Contamination and Reclamation***. Technion-Israel Institute of Technology, Haifa, Israel, May 13-15, 1996.

101. B.J. Shiau, B. Wu, H.K. Karapanagioti, T. Carter, D.A. Sabatini, and J.H. Harwell, "Surfactant Selection for Subsurface Remediation of Petroleum Wastes," National ACS Meeting, New Orleans, LA, March 24-29, 1996.

100. J.F. Scamehorn, S.A. Smith, B.J. Shiau, J.H. Harwell, and D.A. Sabatini, "Performance of a New Class of Ethoxylated Sulfate Surfactants in a Subsurface Remediation Application," National ACS Meeting, New Orleans, LA, March 24-29, 1996.

99. W. Kimchuwanit, J.F. Scamehorn, S. Osuwan, K.J. Haller and J.H. Harwell, "Use of a Surfactant Coacervate Phase to Extract Trichloroethylene from Water," National ACS Meeting, New Orleans, LA, March 24-29, 1996.

98. J.L. Reynolds, B.P. Grady, J.H. Harwell, E.A. O'Rear, and W.H. Waddell, "Admicellar Polymerization on Precipitated Silica," National ACS Meeting, New Orleans, LA, March 24-29, 1996.

97. D.A. Sabatini, R.C. Knox, J.H. Harwell, C.C. West, B.J. Shiau, E. Brown, "Surfactant Enhanced Remediation of Subsurface Petroleum Contamination: Results of a Field Test," National ACS Meeting, New Orleans, LA, March 24-29, 1996.

96. M. Ioneva and J.H. Harwell, "Adsorption of Light Hydrocarbons in Sorbents with Controlled Porosity," National AIChE Meeting, New Orleans, LA, February 25-29, 1996.

95. S. Horstkamp, R. Mallinson, K. Starling, and J. Harwell, "Liquid Storage of Methane via Solubilization in Natural Gas Constituents," National AIChE Meeting, New Orleans, LA, February 25-29, 1996.

94. M.A. Ioneva and J.H. Harwell, "Introduction of Microporosity in MCM-41 Type of Mesoporous Compositions," Materials Research Society Fall Annual Meeting, Boston, Massachusetts, November 27-December 1, 1995.
93. A. Bhummasobhana, J.F. Scamehorn, J.H. Harwell, and S. Osuwan, "Use of Surfactant-Enhanced Carbon Regeneration for Wastewater Applications," AIChE Fall Annual Meeting, Miami Beach, Florida, November, 1995.
92. G.K. Newman, and J. Harwell, "Controlling Nanostructure in Monolithic Metal Aerogels," AIChE Fall Annual Meeting, Miami Beach, Florida, November, 1995.
91. G.K. Newman, and J. Harwell, "High Pressure Adsorption of Hydrocarbon Gases on Fullerene Soot and Modified Adsorbents," AIChE Fall Annual Meeting, Miami Beach, Florida, November, 1995.
90. G.K. Newman, and J. Harwell, "High Pressure Adsorption on Novel High Density, High Surface Area, Composite Adsorbents," AIChE Fall Annual Meeting, Miami Beach, Florida, November, 1995.
89. M. Ioneva, J.H. Harwell, R.G. Mallinson, and K.E. Starling, "Capillary Condensation of Gaseous Hydrocarbon Mixtures in MCM-41 Type Materials," AIChE Fall Annual Meeting, Miami Beach, Florida, November, 1995.
88. M. Ioneva, J.H. Harwell, R.G. Mallinson, and K. E. Starling, "Adsorption of Gaseous Hydrocarbon Mixtures in MCM-41 Type Materials," AIChE Fall Annual Meeting, Miami Beach, Florida, November, 1995.
87. M. Dreyer, G.K. Newman, and J. Harwell, "Aerogels as Adsorbents for Natural Gas Storage," AIChE Fall Annual Meeting, Miami Beach, Florida, November, 1995.
86. D.A. Sabatini, B.J. Shiau, and J.H. Harwell, "Surfactant Enhanced Subsurface Remediation of Petroleum Wastes," Second Annual International Petroleum Environmental Conference, New Orleans, September 25-27, 1995.
85. J. Van Duine, I.A. Legiec, D.A. Sabatini, and J.H. Harwell, "Surfactant Washing of Soils Contaminated with Hazardous Liquids II," Emerging Technologies in Hazardous Waste Management VII, Atlanta, Georgia, September 17-20, 1995.

84. C.J. Haskins, J.F. Scamehorn, and J.H. Harwell, "Kinetics of Precipitation of Surfactants," 86th American Oil Chemist's Society Annual Meeting, San Antonio Texas, May 7-11, 1995.
83. J.D. Rouse, D.A. Sabatini, J.H. Harwell, "Influence of Surfactants on Bioremediation of Petroleum Hydrocarbons," U.S. EPA, RSKERL Research Seminar, Oklahoma City, Oklahoma, June 1-3, 1994.
82. W. Xin, G.K. Newman, and J.H. Harwell, "Methane Adsorption on C60 Modified Adsorbents," AIChE Fall National Meeting, San Francisco, California, November 13-18, 1994.
81. M. Dreyer, G.K. Newman, and J.H. Harwell, "Aerogels as Adsorbents for Natural Gas Storage," AIChE Fall National Meeting, San Francisco, California, November 13-18, 1994.
80. M.A. Ioneva, G.K. Newman, and J.H. Harwell, "The Effects of Pore Size of MCM-41 on Adsorptive Storage of Natural Gas," AIChE Fall National Meeting, San Francisco, California, November 13-18, 1994.
79. J. Van Duine, M.A. Donato, I.A. Legiec, D.A. Sabatini, and J.H. Harwell, "Surfactant Washing of Soils Contaminated with Hazardous Liquids I," Emerging Technologies in Hazardous Waste Management VI, Atlanta, Georgia, September 19-21, 1994.
78. U.N. Choori, J.F. Scamehorn, and J.H. Harwell, "Surfactant Recovery from Remediation of Subsurface Contaminated Soils," Emerging Technologies in Hazardous Waste Management VI, Atlanta, Georgia, September 19-21, 1994.
77. D.A. Sabatini, and J.H. Harwell, "Surfactant Washing of Soils Contaminated with Hazardous Liquids II," Emerging Technologies in Hazardous Waste Management VI, Atlanta, Georgia, September 19-21, 1994.
76. B.J. Shiau, D.A. Sabatini, and J.H. Harwell, "Microemulsification of Single and Mixed Chlorinated Organic Solvents Using Edible Surfactants," Spring National ACS Meeting, San Diego, California, March 12-18, 1994.
75. T.S. Soerens, D.A. Sabatini, and J.H. Harwell, "Experimental and Modeling Studies of Surfactant-Enhanced DNAPL Remediation in 1D Columns," Spring National ACS Meeting, San Diego, California, March 12-18, 1994.
74. J.D. Rouse, D.A. Sabatini, and J.H. Harwell, "Influence of Surfactants on

Microbial Degradation of Organic Compounds," Spring National ACS Meeting, San Diego, California, March 12-18, 1994.

73. J.D. Rouse, D.A. Sabatini, and J.H. Harwell, "Use of Twin-Head and Ethoxylated High-Performance Anionic Surfactants in Subsurface Remediation," Spring National ACS Meeting, San Diego, California, March 12-18, 1994.

72. K.M. Lipe, M. Hasegawa, D.A. Sabatini, and J.H. Harwell, "Modeling and Optimization of Ultrafiltration and Air Stripping for Use in Surfactant Recycling," Spring National ACS Meeting, San Diego, California, March 12-18, 1994.

71. B. Krebs-Yuill, J.H. Harwell, D.A. Sabatini, and R.C. Knox, "Edible Hydrotropes in the Use of Edible Surfactants in Surfactant-Enhanced Soil Remediation," Spring National ACS Meeting, San Diego, California, March 12-18, 1994.

70. B. Krebs-Yuill, J.H. Harwell, D.A. Sabatini, and R.C. Knox, "Economic Considerations in Surfactant-Enhanced Pump-and-Treat," Spring National ACS Meeting, San Diego, California, March 12-18, 1994.

69. J.H. O'Haver, J.H. Harwell, E.A. O'Rear, and L.L. Lobban, "Adsolubilization," AIChE Fall National Meeting, St. Louis, Missouri, November 7-12, 1993.

68. B. Krebs-Yuill and J.H. Harwell, "Economic Optimization of Surfactant Enhanced Water Flooding," AIChE Summer National Meeting, Seattle, Washington, August 15-19, 1993.

67. B. Roberts, T. Sorens, J.H. Harwell, D.A. Sabatini, and R.C. Knox, "Model Aquifer Evaluation of Surfactants for Enhanced Water Flooding," AIChE Summer National Meeting, Seattle, Washington, August 15-19, 1993.

66. J.H. Harwell, Thomas Soerens, and J.H. Harwell, "Formation of Shock Waves in Surfactant Enhanced Remediation of DNAPL Contaminated Aquifers," AIChE Summer National Meeting, Seattle, Washington, August 15-19, 1993.

65. D.A. Sabatini, J.D. Rouse, B.J. Shiau, T.S. Soerens, and J.H. Harwell, "Role of Surfactant Selection in Design of Surfactant Enhanced Subsurface Remediation," AIChE Summer National Meeting, Seattle, Washington, August 15-19, 1993.

64. J.H. O'Haver, J.H. Harwell, E.A. O'Rear, and W.H. Waddell, "Modification of the Surface Properties of Silica by the Formation of Ultrathin Polystyrene Films in Adsorbed Surfactant Bilayers," 205th National ACS Meeting, Denver, Colorado,

March 28-April 2, 1993.

63. J.H. O'Haver, J.H. Harwell, E.A. O'Rear, and W.H. Waddell, "Formation of Ultrathin Polystyrene films in Adsorbed Surfactant Bilayers on Alumina," Materials Research Society Spring Meeting, San Francisco, April 12-16, 1993.

62. J. O'Haver, J.H. Harwell, and E.A. O'Rear, "Admicellar Copolymerization of Styrene/Butadiene in an adsorbed Surfactant Bilayer on Alumina," AIChE 1992 Fall National Meeting, Miami Beach, Florida, November 1-6, 1992.

61. B. Shiau, A. Gupta, J.H. Harwell, R.C. Knox, and D.A. Sabatini, "Edible surfactants in Enhanced Remediation Processes," AIChE 1992 Summer National Meeting, Minneapolis, Minnesota, August 9-12, 1992. *Selected as an Outstanding Paper at the Summer National Meeting.*

60. J. Rouse, D.A. Sabatini, J.H. Harwell, "Minimizing Surfactant Losses During Surfactant Enhanced NAPL Remediation," 3rd International Conference on Ground Water Quality Research--Subsurface Restoration, Dallas, Texas, June 21-24, 1992.

59. K. Micelle Lipe, D.A. Sabatini, and J.H. Harwell, "Surfactant Recovery Using MEUF in Subsurface NAPL Remediation," 3rd International Conference on Ground Water Quality Research--Subsurface Restoration, Dallas, Texas, June 21-24, 1992.

58. A. Gupta, B. Shiau, J.H. Harwell, and D.A. Sabatini, "Enhanced Solubilization and Mobilization of Subsurface DNAPL Contamination Using Edible Surfactant," 3rd International Conference on Ground Water Quality Research--Subsurface Restoration, Dallas, Texas, June 21-24, 1992.

57. T. S. Sorens, D.A. Sabatini, and J.H. Harwell, "Surfactant Enhanced solubilization of Residual DNAPLS--Column Studies," 3rd International Conference on Ground Water Quality Research--Subsurface Restoration, Dallas, Texas, June 21-24, 1992.

56. B. Krebs-Yuill and J.H. Harwell, "Economics of Surfactant Recovery in Surfactant Enhanced Ground Water Remediation," 3rd International Conference on Ground Water Quality Research--Subsurface Restoration, Dallas, Texas, June 21-24, 1992.

55. C-L. Lai, E.A. O'Rear, and J.H. Harwell, "Formation of Adsorbed Polytetrafluoroethylene Thin Films on Alumina Using Two-Dimensional

Solvents," 66th Colloid and Surface Science Symposium, Morgantown, West Virginia, June 14-17, 1992.

54. A. Gupta, B. Shiau, J.H. Harwell, and D.A. Sabatini, "Use of "Edible" Surfactants for Enhanced Solubilization and Mobilization of Subsurface DNAPL Contamination," 203rd National ACS Meeting, San Francisco, CA, April 5-10, 1992.

53. B. Black, D.A. Sabatini, and J.H. Harwell, "Surfactant Properties Affecting the Efficiency of Surfactant Enhanced Subsurface Remediation," 203rd National ACS Meeting, San Francisco, CA, April 5-10, 1992.

52. B. Shiau, D.A. Sabatini, and J.H. Harwell, "Influence of Molecular Structure of Rhodamine WT on Sorptive Transport in Subsurface Media," Fall Meeting of The American Geophysical Union, San Francisco, California, December, 1991.

51. B. Shiau, J.H. Harwell, and J.F. Scamehorn, "Precipitation in Anionic/Cationic/Nonionic Surfactant Mixtures," AIChE Annual Meeting, Los Angeles, California, November, 18-22, 1991.

50. G.K. Newman, J.H. Harwell, and K. E. Starling, "Methane Sorption in Admicelles," AIChE Annual Meeting, Los Angeles, California, November, 18-22, 1991.

49. J.J. Lopata, J.F. Scamehorn, and J.H. Harwell, "Thermodynamics of Adsorption of Binary Anionic/Nonionic Surfactant Mixtures on Alumina," 65th Annual ACS Colloid and Surface Science Symposium Norman, Oklahoma, June, 1991.

48. C. Palmer, D.A. Sabatini, and J.H. Harwell, "Sorption of Hydrophobic and Amphiphilic (Nonionic Surfactant) Organics with Subsurface Materials," 65th Annual ACS Colloid and Surface Science Symposium Norman, Oklahoma, June, 1991.

47. B. Shiau, J.H. Harwell, and J.F. Scamehorn, "Effect of Nonionic Surfactant on Precipitation of Mixtures of Anionic and Cationic Surfactants," 65th Annual ACS Colloid and Surface Science Symposium Norman, Oklahoma, June, 1991.

46. J.H. Harwell, "Factors Affecting Surfactant Selection in Ground Water Remediation Projects," 65th Annual ACS Colloid and Surface Science Symposium Norman, Oklahoma, June, 1991.

45. C.L. Lee, E.A. O'Rear and J.H. Harwell, "Separation of Racemates using Chiral Admicellar Chromatography," 65th Annual ACS Colloid and Surface Science Symposium Norman, Oklahoma, June, 1991.
44. B. Shiau, J.H. Harwell, and J.F. Scamehorn, "Effect of Nonionic Surfactant on Precipitation of Mixtures of Anionic and Cationic Surfactants," 82nd Annual AOCS Meeting, Chicago, Illinois, May, 1991. Selected as an Outstanding Paper at the 82nd Annual Meeting.
43. J.J. Lopata, J.F. Scamehorn, and J.H. Harwell, "Thermodynamics of Adsorption of Surfactant Mixtures on Alumina," AIChE Annual Meeting, Chicago, Illinois, November, 1990.
42. N.P. Hankins and J.H. Harwell, "Application of Coherence Theory to a Reservoir Enhanced Oil recovery Simulator," AIChE Annual Meeting, Chicago, Illinois, November, 1990.
41. A.S. Arshad and J.H. Harwell, "Experimental Verification of Surfactant Enhanced Volumetric Sweep efficiency," AIChE Annual Meeting, Chicago, Illinois, November, 1990.
40. A. Tabatabai, M.V. Gonzalez, J.H. Harwell, and J.F. Scamehorn, "Curious Aspects of Ionic Surfactant Adsorption on Carbonate Minerals," 64th Annual ACS Colloid and Surface Science Symposium, Lehigh University, Bethlehem, Pennsylvania, June, 1990.
39. A. Tabatabai, M.V. Gonzalez, J.H. Harwell, and J.F. Scamehorn, "Optimum Surfactant Selection for Carbonate Reservoirs," AIChE National Meeting, Orlando, Florida, March, 1990.
38. N.P. Hankins and J.H. Harwell, "Feasibility of Surfactant Enhanced Waterflooding," AIChE National Meeting, Orlando, Florida, March, 1990.
37. A. Arshad and J.H. Harwell, "Surfactant Precipitation in Porous Media and its Effect on formation Permeability," AIChE National Meeting, Orlando, Florida, March, 1990.
36. L.A. Hole, J.F. Scamehorn and J.H. Harwell, "Kinetics of Surfactant Precipitation," International Chemical Congress of Pacific Basin Societies, Honolulu, December, 1989.
35. L.A. Hole, J.F. Scamehorn and J.H. Harwell, "Kinetics of Surfactant

Precipitation," International Chemical Congress of Pacific Basin Societies, Honolulu, December, 1989.

34. J.H. Harwell, "Topology of the Precipitation Phase Boundary in an Anionic, Cationic, Nonionic Surfactant Mixture," 63rd ACS Colloid and Surface Science Symposium, University of Washington, Seattle, Washington, June 18-21, 1989.

33. L.A. Hole, J.F. Scamehorn and J.H. Harwell, "Kinetics of Surfactant Precipitation," 63rd ACS Colloid and Surface Science Symposium, University of Washington, Seattle, Washington, June 18-21, 1989.

32. L.A. Hole, J.F. Scamehorn and J.H. Harwell, "Surfactant Precipitation Kinetics," 80th Annual AOCS Meeting, Cincinnati, Ohio, May, 1989.

31. M. Yeskie, C. Lee, E.A. O'Rear, and J.H. Harwell, "Adsolubilization," 63rd ACS Colloid and Surface Science Symposium, University of Washington, Seattle, Washington, June 18-21, 1989.

30. M. Yeskie, C. Lee, E.A. O'Rear, and J.H. Harwell, "Incorporation of Alcohols and Alkanes into Adsorbed Surfactant Aggregates," National AIChE Meeting, Washington, D.C., November, 1988.

29. B.L. Roberts, J.F. Scamehorn, and J.H. Harwell, "Surfactant Enhanced Carbon Regeneration to Recover Volatile Organics," 196th National ACS Meeting, Los Angeles, September, 1988.

28. B.L. Roberts, J.F. Scamehorn and J.H. Harwell, "Surfactant-Enhanced Carbon Regeneration to Recover Volatile Organics," 62nd ACS Colloid and Surface Science Symposium, Penn State University, June, 1988.

27. J.C. Amante, J.H. Harwell, and J.F. Scamehorn, "Precipitation of Mixtures of Anionic and Cationic Surfactants," 79th Annual AOCS Meeting, Phoenix, May 1988.

26. N.D. Gullickson, J.F. Scamehorn, and J.H. Harwell, "Liquid/Coacervate Extraction Using Nonionic Surfactant," 195th National ACS Meeting, Toronto, June 1988.

25. J.C. Amante, J.H. Harwell, and J.F. Scamehorn, "Precipitation of Anionic/Cationic Surfactant Mixtures in Surfactant-Enhanced Volumetric Sweep Efficiency," 195th National ACS Meeting, Toronto, June 1988.

24. B.L. Roberts, J.F. Scamehorn, and J.H. Harwell, "Surfactant-Enhanced Carbon

Regeneration," U.S. Department of Energy Industrial Waste Gas Utilization Program Review, Washington, D.C. May, 1988.

23. B.L. Roberts, J.F. Scamehorn, and J.H. Harwell, "Surfactant-Enhanced Carbon Regeneration," U.S. Department of Energy Separation Innovative Concepts Fair, Arlington, Virginia, May, 1988.

22. J.F. Scamehorn and J.H. Harwell, "Surfactant-Based Separation Techniques for Pollution Control," AIChE Annual Meeting, New York, November, 1987.

21. N.D. Gullickson, J.F. Scamehorn and J.H. Harwell, "Removal of Pollutants from Water using Liquid/Coacervate Extraction," AIChE Annual Meeting, New York, November, 1987.

20. J. Barton, T.P. Fitzgerald, J.H. Harwell, and E.A. O'Rear, "Batch and Chromatographic Separation of Isomers Using Two-Dimensional Solvents," Summer National Meeting of the American Institute of Chemical Engineers, Minneapolis, Minnesota, August 16-19, 1987.

19. J. Wu, E.A. O'Rear, and J.H. Harwell, "Application of Thin Films to Porous Mineral Oxides Using Two Dimensional Solvents," Summer National Meeting of the American Institute of Chemical Engineers, Minneapolis, Minnesota, August 16-19, 1987.

18. K.L. Stellner, J.C. Amante, J.F. Scamehorn, and J.H. Harwell, "Precipitation Phenomena in Surfactant-Enhanced Mobility Control," 61st Annual Colloid and Surface Science Symposium, Ann Arbor, Michigan, June 21-24, 1987.

17. J.C. Amante, K.L. Stellner, J.F. Scamehorn and J.H. Harwell, "Precipitation of Anionic/Cationic Surfactant mixture," 78th Annual AOCS Meeting, New Orleans, May, 1987.

16. C.R. Grout and J.H. Harwell, "A New Separation and Regeneration Process Using Fixed-Bed Adsorbubilization," 193rd National ACS Symposium, Denver, April 5-10, 1987.

15. T.P. Fitzgerald and J.H. Harwell, "The Effects of Process Conditions on the Removal of n-Heptanol from Aqueous Streams Using Admicellar Chromatography," 193rd National ACS Symposium, Denver, April 5-10, 1987.

14. J.H. Harwell and E.A. O'Rear, "Surfactant Bilayers in the Formation of Ultra-Thin Films," Council for Chemical Research, Workshop for Chemical

Science and Technology Transfer, Denver, April 4-5, 1987.

13. C.R. Grout and J.H. Harwell, "A New Wastewater Treatment Technology Using Admicellar Chromatography," Presented at the First International Congress on Recent Advances in The Management of Hazardous and Toxic Wastes in the Process Industries, Vienna, March 8-13, 1987.

12. J.F. Scamehorn and J.H. Harwell, "Surfactant Based Wastewater Treatment Technologies," Presented at the First International Congress on Recent Advances in The Management of Hazardous and Toxic Wastes in the Process Industries, Vienna, March 8-13, 1987.

11. E.A. O'Rear, J. Wu, and J.H. Harwell, "Adsorbed Surfactant Bilayers as Two-Dimensional Solvents for the Formation of Thin Films," Organic Thin Films Gordon Conference (Poster Session), Santa Barbara, February, 1986.

10. D. Bitting and J.H. Harwell, "Mechanisms of Counterion Association with Physically Adsorbed Surfactant Aggregates," 60th ACS Colloid and Surface Science Symposium, Atlanta, June 16-18, 1986.

9. J. Wu, J.H. Harwell, and E.A. O'Rear, "Adsorbed Sodium Dodecyl Sulfate Bilayers as a Two-Dimensional Polymerization Solvent for Styrene," 60th ACS Colloid and Surface Science Symposium, Atlanta, June 16-18, 1986.

8. J.H. Harwell and D. Bitting, "An Experimental Study of the Incorporation of Counterions into the Structure of Surfactant Aggregates Adsorbed on Aluminum Oxide from Aqueous Solutions," 191st National ACS Symposium, New York, New York, April 14-18, 1986.

7. J.H. Harwell, "Surfactant Aggregate Formation on Adsorption From Aqueous Solution onto Alumina," 191st National ACS Symposium, New York, New York, April 14-18, 1986.

6. J. Wu, J.H. Harwell, and E.A. O'Rear, "Polymerization of Styrene Inside a Physically Adsorbed Surfactant Bilayer," 191st National ACS Symposium, New York, New York, April 14-18, 1986.

5. J.H. Harwell, B.L. Roberts and J.F. Scamehorn, "Thermodynamics of Adsorption of Surfactant Mixtures on Minerals," Annual AIChE Meeting, New Orleans, Louisiana, April 7-10, 1986.

4. J. Wu, J.H. Harwell, and E.A. O'Rear, "Two-Dimensional Solvents: Kinetics of Styrene Polymerization with Interphase Mass Transfer Effects," National AIChE

Meeting, Miami Beach, Florida, November 2-7, 1986.

3. T.P. Fitzgerald and J.H. Harwell, "Admicellar Chromatography: A New Low Energy Separation Process," National AIChE Meeting, Miami Beach, Florida, November 2-7, 1986.

2. B.L. Roberts, J.F. Scamehorn, and J.H. Harwell, "Adsorption of Mixtures of Anionic Surfactants on Alumina," 59th ACS Colloid and Surfactant Science Symposium, Potsdam, New York, June 24-28, 1985.

1. J.H. Harwell, F.G. Helfferich, and R.S. Schechter, "Interactions of Dispersion and Mixed Micelle Formation in the Chromatographic Movement of Surfactant Mixtures," Spring National AIChE Meeting, Houston, Texas, April 1981.

OTHER PRESENTATIONS:

J. H. Harwell, "Surfactant Enhanced Aquifer Remediation: Background and Field Results," Department of Environmental Quality, State of New Mexico, November 18, 2005.

J. H. Harwell, "Surfactant Enhanced Aquifer Remediation: Background and Field Results," Environmental Consulting Services, Burlington, Vermont, July 28, 2005.

J. H. Harwell, B.J. Shiau, "Surfactant Enhanced Aquifer Remediation: Background and Field Results," Sinotech Engineering, Taiwan, Taipei, July 13, 2005.

J. H. Harwell, B.J. Shiau, "Surfactant Enhanced Aquifer Remediation: Background and Field Results," Greater Pacific Petrochemicals Corporation, Kaohsiung, Taiwan, July 11, 2005.

J. H. Harwell, B.J. Shiau, "Surfactant Enhanced Aquifer Remediation: Background and Field Results," Department of Environmental Quality, State of Maine, June 24, 2005.

J. H. Harwell, B.J. Shiau, "Surfactant Enhanced Aquifer Remediation: Background and Field Results," Department of Environmental Quality, State of Vermont, June 22, 2005.

J. H. Harwell, B.J. Shiau, "Surfactant Enhanced Aquifer Remediation: Background and Field Results," Department of Environmental Quality, State of New Hampshire, June 20, 2005.

J. H. Harwell, B.J. Shiau, "Surfactant Enhanced Aquifer Remediation: Background and Field Results," The Southern Company, Little Rock, Arkansas, June 2, 2005.

J. H. Harwell, B.J. Shiau, "Surfactant Enhanced Aquifer Remediation: Background and Field Results," The Southern Company, Little Rock, Arkansas, February 21, 2005.

J.H. O'Haver, E.A. O'Rear, and J.H. Harwell, "Co-Polymerization Within an Adsorbed Surfactant Bilayer on Alumina," ACS Pentasectional Meeting, Bartlesville, Oklahoma, February, 1992.

Chung-Li Lai, E.A. O'Rear, and J.H. Harwell, "Formation of Adsorbed Polytetrafluoroethylene Thin Films on Alumina Using Two Dimensional Solvents," ACS Pentasectional Meeting, Bartlesville, Oklahoma, February, 1992.

B.J. Shiau, D.A. Sabatini, and J.H. Harwell, "Influence of Molecular Structures of Rhodamine WT on Sorptive Transport in Subsurface Media," Oklahoma Academy of Science 80th Annual Meeting, Southeastern Oklahoma State University, November 8, 1991.

J. Wu, J.H. Harwell, and E.A. O'Rear, "Two Dimensional Solvents: Kinetics of Styrene Polymerization in Admicelles at or Near Saturation," 31st ACS Pentasectional Meeting, Lawton, Oklahoma, April, 1986.

T.P. Fitzgerald and J.H. Harwell, "Admicellar Chromatography: A New Low Energy Separation Process," 31st American Chemical society Pentasectional Meeting, Lawton, Oklahoma, April, 1986.

C.R. Grout and J.H. Harwell, "Fixed Bed Adsolubilization: A New Wastewater Treatment Technology," 42nd American Chemical Society Southwest Regional Meeting, Houston, Texas, November 19-21, 1986.

1987-88 MASUA HONOR LECTURES

"The New Surfactant-Based Separation Processes," Department of Chemical Engineering, The University of Missouri at Columbia, Columbia, Missouri, September 29, 1987.

"The New Surfactant-Based Separation Processes," Department of Chemical Engineering, Kansas State University, Manhattan, Kansas, October 13, 1987.

"The New Surfactant-Based Separation Processes," Department of Chemical Engineering, The University of Nebraska, Lincoln, Nebraska, November 5, 1987.

GRADUATE THESES AND DISSERTATIONS

COMPLETED: Ph.D. Dissertations Supervised—21
M.S. Theses Supervised—28

2016 Danielle Baker

2015 Javen Weston
Manish Budhathoki

2009 Thu T. Nguyen, Formulating Alcohol-Free Microemulsions Using Rhamnolipid Biosurfactant and Rhamnolipid Mixtures, Ph.D., December, 2009.

2008 Mohammed Merzouk, Alcohol-Free Microemulsions for Subsurface Remediation, M.S. Thesis, August, 2008.

Shahryar Gilani, Adsorption of Surfactant on Single Walled Carbon nanotubes, M.S. Thesis, May, 2008.

2004 Edgar Acosta, Theory and Applications of Lipophilic and Hydrophilic Linkers, Ph.D., May 2004.

2003 Erika Szekeres, Mixed-NAPL Microemulsions, Ph.D., May 2003.

2001 Laura Wesson, Mixing Rules for Forming Microemulsions from Disodium Diphenyloxide Disulfonates, Ph.D., August 2001.

2000 Boonyarak Kitiyanan, Single Wall Carbon Nanotubes Production by Heterogeneous Catalytic Reaction, Ph.D., May 2000.

Edgar J. Acosta, Use of Lipophilic and Hydrophilic Linkers in Trichloroethylene Microemulsions, M.S. Thesis, May 2000.

San Trong Tran, Enhancement of Tetrachlorethylene Solubilization in Water Using Linkers in Microemulsion, M.S. Thesis, May 2000.

1999 Karolina Oetman, Regeneration of Surfactant Process Streams, M.S. Thesis, May 1999.

1996 Justin Van Duine, Surfactant Soil Remediation, M.S. Thesis, December, 1996.

Mariana Ioneva (1994-1995 Link Foundation Fellowship recipient), Methane Storage on Novel Zeolitic Materials, Ph.D. Dissertation, August, 1996.

Steven Horstkamp, Storage of Methane in Hydrocarbon Mixtures, M.S. Thesis, May 1996. (Co-Advisor with R.G. Mallinson)

1995 Gerald K. Newman, Novel Adsorbents for Methane Storage, Ph.D. Dissertation, December 1995.

Chung-Li Lai, Fluoropolymer Thin Films, (Co-Advisor with E.A. O'Rear), Ph.D. Dissertation, August 1995.

John H. O'Haver (Materials Research Society Outstanding Graduate Research Award, 1993; DuPont Plunkett Award Honorable Mention, 1994), Surfactant Thin Film Modified Silicas, (Co-Advisor with E.A. O'Rear), Ph.D. Dissertation, May, 1995.

Barbara Krebs-Yuill, Surfactant Enhanced In-Situ Aquifer Remediation, Ph.D. Dissertation, May 1995.

Martina Dreyer, Methane Storage on Aerogels and Transition Metal Doped Aerogels, M.S. Thesis, August 1995.

Scott Smith, Performance and Chemical Stability of a New Class of Ethoxylated Sulfate Surfactants in a Subsurface Remediation Application, (with J.F. Scamehorn, co-advisor) M.S. Thesis, April 1995.

1994 Shurong Xin, Methane Storage on Surfactant Modified Silicas, M.S. Thesis, December 1994.

Nuriye Duman, Surface Modification of Titanium Dioxide by In Situ Polymerization, M.S. Thesis, August 1994.

Umesh Choori, Continuous Co-Current Vacuum Stripping of Surfactant Solutions, (with J.F. Scamehorn, co-advisor) M.S. Thesis, December 1994.

Mariana Ioneva, Methane Storage on Novel Zeolitic Materials, M.S. Thesis, May 1994.

- Weikang Xin, Methane Storage on Buckminsterfullerene Thin Films, M.S. Thesis, May 1994.
- 1993 Elias Bandari, Surfactant Modification of Activated Carbon for Methane Storage, M.S. Thesis (Co-Advisor with K. E. Starling).
- Anil Gupta, Technical Feasibility of Edible-Surfactants for Surfactant-Enhanced *In-Situ* Remediation of Subsurface Contamination with Tetrachloroethylene (PCE), M.S. Thesis.
- 1992 Chung-Li Lai, Polymerization of Tetrafluoroethylene by Using the Method of the Formation of Fluorocarbon Thin Films, M.S. Thesis (Co-Advisor with E.A. O'Rear).
- Jeffrey John Lopata, A Study of the Thermodynamic Properties of Surfactant Mixtures: Mixed Micelle Formation and Mixed Surfactant Adsorption, Ph.D. Dissertation (Co-Advisor with J.F. Scamehorn).
- Hoern-Yann Chen, Formation of Polystyrene Thin Films on Titania, M.S. Thesis (Co-Advisor with E.A. O'Rear).
- 1991 Aliarza Seyed Arshad, A Feasibility Study of Surfactant Enhanced Waterflooding, Ph.D. Dissertation.
- Timothy L. Fitzgerald, Admicellar Chromatography for the Alcohols and Metal Ions, Ph.D. Dissertation.
- Anup Mangaokar, Formation of Ultrathin Polystyrene Films on High Surface Area Silica, M.S. Thesis (Co-Advisor with E.A. O'Rear).
- 1990 Mohammad Arshad, A Study of a New Method of Measuring Monomer-Micelle Equilibrium for Mixed Surfactants in an Aqueous solution, M.S. Thesis (Co-Advisor with J.F. Scamehorn).
- Bor-Jier Shiau, Precipitation of Ternary Mixtures of Surfactants, M.S. Thesis (Co-Advisor with J.F. Scamehorn).
- Rong-Song Wu, Molecular Simulation and Modeling for the Thermodynamic Properties of Polyatomic Molecules and for Surfactant Adsorption, Ph.D. Dissertation (Co-Advisor with L.L. Lee).

- 1989 Nicholas P. Hankins, A Study of Surfactant Adsorption with Applications in Surfactant Assisted Enhanced Oil Recovery Processes, Ph.D. Dissertation.
- Chon-Lin Lee, Two Dimensional Solvents in the Separation of Isomers, Ph.D. Dissertation (Co-Advisor with E.A. O'Rear).
- David Steinhoff, Aspects of the Orientation of Adsorbed Surfactant Aggregates Studied by Flotation and In Situ Polymerization, M.S. Thesis.
- John Fagbemi, Anionic and Cationic Surfactant Adsorption on Berea, M.S. Thesis (Co-Advisor with J.F. Scaimehorn).
- Ahmadali Tabatabai, Adsorption of Anionic Surfactant on Carbonate Minerals, M.S. Thesis (Co-Advisor with J.F. Scaimehorn).
- Maria v. Gonzalez, Adsorption of Cationic surfactants on Carbonate Minerals, M.S. Thesis (Co-Advisor with J.F. Scaimehorn).
- 1988 Mark A. Yeskie, Various Aspects of Surfactant Aggregates Adsorbed on an Oxide Surface: The Hemimicelle/Admicelle Transition and the Interaction of Alcohols and Alkanes, Ph.D. Dissertation.
- Jeffrey J. Lopata, A Study of the Adsorption of Binary Anionic Surfactant Mixtures on Alpha Aluminum Oxide, M.S. Thesis (Co-Advisor with J.F. Scaimehorn).
- 1987 Jengyue Wu, The Admicelle as a Two Dimensional Solvent for Polymerization--Formation, Characterization and Application of Ultra-Thin Films, Ph.D. Dissertation (Co-Advisor with E.A. O'Rear)
- Chris R. Grout, The Use of Fixed-Bed Adsolubilization as a Separation Technique for Dilute Aqueous Solutions, M.S. Thesis
- Joel C. Amante, Precipitation of Anionic-Cationic Surfactant Mixtures, M.S. Thesis (Co-Advisor with J.F. Scaimehorn)
- 1985 Daryl Bitting, Specific Counterion Effects in Anionic Surfactant Adsorption of a Mineral Oxide Surface, M.S. Thesis
- Seyed Alireza Arshad, Improving Sweep Efficiency in Oil Reservoirs Using Surfactant Phase Behavior, M.S. Thesis

TEACHING INTERESTS: Applied Mathematics and Numerical Methods, Classical and Statistical Thermodynamics, Basic and Applied Surface and Colloid Science, Separations Science and Technology

MAJOR SERVICE ACTIVITIES

THE UNIVERSITY OF OKLAHOMA:

Engineering Dean Search Committee, University of Oklahoma, 2018/2019
Associate Dean of Engineering, OU/COE, 1998-2005
Chairman, CEMS, 1992-1998
Interim Chairman, CEMS, 1991-1992
Committee A, 1991
Freshman Advisor, 1990-1991
Asahi Glass Chair Committee, 1990-1991
Seminar Coordinator, 1989-1991
Departmental Strategic Planning Committee, 1987
Research Council, 1987-1988
CEMS Graduate Program Coordinator 1985-1988
Chair, Mobile Thermodynamics Laboratory Committee 1986-1991
Participated in Departmental Program Review, 1987
Energy Center Committee, 1986-1987
Graduate Program Committee, 1983-1985
College of Engineering Hand-Held Computer Committee
Editor, Three Issues of OkChE Magazine

PROFESSIONAL SERVICE:

Director, Norman Area, Central Oklahoma Section, AIChE, June 2000-May 2002

Co-Organizer and Co-Chair, Surfactant-Based Separation Process, 218th ACS National Meeting, Dallas, Texas, March 29-April 2, 1998.

Organizing Committee, Engineering Foundation Conference, Separation Technologies VII, Davos, Switzerland, October 26-31, 1997.

Session Chair, "Surfactants in Environmental Restoration," Engineering Foundation Conference, Separation Technologies VII, Davos, Switzerland, October 26-31, 1997.

Co-Organizer and Co-Chair, "Surfactant-Enhanced Remediation of Subsurface

Contamination: Emerging Technologies," 207th ACS National Meeting, San Diego, California, March 13-17, 1994.

ACS Awards Program Committee Member, 1989-1993.

Co-Chair, "Non-Linear Wave Propagation: Fixed Bed Operations," Session Number 89, AIChE 1992 Fall National Meeting, Miami Beach, Florida, November 1-6, 1992.

Co-Chair, "Applications of the Theory of Non-Linear Wave Propagation," Session Number 93, AIChE 1992 Fall National Meeting, Miami Beach, Florida, November 1-6, 1992.

Co-Chair, "Separation of Contaminants from Soil and Ground Water," Session Number 83, AIChE 1992 Summer National Meeting, Minneapolis, Minnesota, August 9-12, 1992.

Co-Chairman of the 65th ACS Colloid and Surface Science Symposium of the Colloid and Surface Science Division of the American Chemical Society, June 16-19, 1991.

Chair of the AIChE National Symposium on Waste Treatment and Management, Separations Technology Division, Washington, D.C., December, 1989.

Program Director, Interfacial, Transport and Separation Processes Program, The National Science Foundation, Washington, D.C.; July 1988 to July 1989.

REVIEWING:

National Science Foundation (Interfacial, Thermodynamic, and Separation Processes, Chemical and Thermal Systems Division), Petroleum Research Fund of the American Chemical Society, Gas Research Institute, Chemical Engineering, Journal Physical Chemistry, Chemical Engineering Communications, AIChE Journal, Langmuir, Journal of Colloid and Interface Science, Society of Petroleum Engineers Journal, ACS Symposium Series, Environmental Science and Technology, Colloids and Surfaces.

COMMUNITY:

Deacon, Wildwood Community Church, October 1983 to December 1985

Elder, Wildwood Community Church, December 1985 to December 1994;
December 1995 to 2003, December 2019 to present.
Chairman of the Board of Elders, Wildwood Community Church, December 1986
to December 1994; October 1999 to 2005

SUMMARY OF MAJOR RESEARCH ACCOMPLISHMENTS

1. Invented admicellar chromatography, a new adsorptive process for waste water, biological and enantiomeric separations.
2. Promotion of surfactant-based separation processes as an identifiable field of research.
3. Developed the concept of adsorbed surfactant bilayers as two-dimensional solvents (adsolubilization).
4. Developed a phase separation model of surfactant adsorption.
5. Discovered the dominant role of mixed micelle formation in the chromatographic movement of surfactant mixtures.
6. Demonstration of importance of counterion binding on admicelles.
7. Co-inventor of process for making ultrathin films by polymerization of monomer adsolubilized in adsorbed surfactant bilayers--Thin Film via a Surfactant Templates (TFST).
8. Co-invention of surfactant assisted water flooding as a new enhanced oil recovery technique.
9. Co-founder of the Institute for Applied Surfactant Research, the University of Oklahoma.
10. Co-Inventor of a new rubber reinforcing technology using in situ polymerization of a cross-linking polymer in a surfactant film on the reinforcer surface.
11. Lead developer of the use of surfactants for *in situ* ground water remediation and ex situ soil washing for environmental restoration.