

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. IPR2024-00822

Patent No. 10,697,282

Issued: June 30, 2020

Application No.: 16/226,464

Filed: December 19, 2018

Title: METHOD FOR REMEDIATION OF SUBTERRANEAN FORMED
METAL-POLYMER COMPLEXES USING A METAL-COMPLEXING
AGENT

**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. 10,697,282**

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 1–22 (Challenged Claims) of U.S. Patent No. 10,697,282 B2 (the '282 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., October 25, 2013) to which the '282 Patent claims priority:

<u>REFERENCE</u>	<u>RELEVANT DATE</u>
United States Patent No. 9,410,405 B2 to Ayers et al. (“ Ayers ”) (Ex. 1004).	November 30, 2011
United States Patent No. 6,436,880 B1 to Frenier (“ Frenier ”) (Ex. 1005).	May 3, 2000

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

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. 1014) (clean copy downloaded April 16, 2024)
Schlumberger Online Glossary, https://glossary.slb.com/en/ (Ex. 1015) (screenshots assembled April 16, 2014)

6. 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 ’282 Patent.

7. 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 Ayers and the similar treatment methods and compositions described and disclosed by Frenier to form those described and claimed in the ’282 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 Ayers and Frenier to be used at least for remediating oil and gas wells. Additionally, as is clear from Borchardt, the interaction of polymers with naturally occurring ions in oil reservoirs has been known for over three decades, as

has the use of a wide variety of chelating agents for sequestering these ions within a reservoir. Consequently, it has long been obvious to a POSITA that chelates could be used to dissolve in situ metal-polymer complexes.

8. Beyond this, many of the operations and chemicals described in the '282 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 '282 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 '282 Patent and that prior art to the '282 Patent includes patents and printed publications in the relevant art that predate the alleged priority date of the '282 Patent, which I have been told to assume is October 25, 2013.

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., October 25, 2013).

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 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 '282 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., October 25, 2013, and by then, I was a POSITA.

IV. DESCRIPTION OF THE TECHNOLOGY AND OVERVIEW OF THE '282 Patent

38. The '282 Patent is entitled "METHOD FOR REMEDIATION OF SUBTERRANEAN FORMED METAL-POLYMER COMPLEXES USING A METAL-COMPLEXING AGENT." Ex. 1001, front cover.

39. The application for the '282 Patent (U.S. Patent Application No.

16/226,464) was filed December 19, 2018. Ex. 1001, front cover.

40. I also understand that the '282 Patent relates to U.S. Provisional Patent Application No. 61/895,873, filed on October 25, 2013.

41. Therefore, the '282 Patent has a “priority date” of October 25, 2013.

42. The specification of the '282 Patent describes “[f]ormulations and methods for remediating subterranean-formed metal-polymer complexes, such as polyacrylamide or other gelable polymers that crosslink with metal ions, from wells in subterranean formations, including wells in nano-darcy shale formations.” Ex. 1001, at 2:20–24¹.

43. The disclosed method includes injecting a metal-polymer complex remediation mixture containing a metal complexing agent into a subterranean formation adjacent to a well a pressure below the fracture pressure of the formation. *Id.* at 2:25–29. After allowing sufficient contact time, the mixture is pumped from the subsurface. *Id.* at 2:29–30.

44. According to the '282 Patent, application of this method results in the remediation of polymer-containing, subterranean-formed metal-polymer complexes in shale formations. *Id.* at 2:30–33..

45. Although the '282 Patent fails to conclusively set forth the

¹ The notation N:XX–YY refers to column (or page) number N, and lines XX to YY of a reference.

mechanism of action underlying its purportedly inventive concept, it suggests that the metal complexing agent is likely forming complexes with the metals in the metal-polymer complexes, and particularly divalent metal ions. *Id.* at 2:33–37.

46. The specification of the '282 Patent describes a “method for remediation a subterranean-formed metal-polymer complex in a pre-existing well in a subterranean shale formation” generally including the following steps:

- (i) providing a metal-polymer complex remediation mixture comprising between 0.1% and 95% by weight metal complexing agent;
- (ii) injecting the metal-polymer complex remediation mixture into the well at a pressure less than a fracture pressure of the subterranean formation until at least some of the metal-polymer complex remediation mixture contacts the subterranean-formed metal-polymer complex, wherein the subterranean-formed metal polymer complex forms from a previously injected fracturing fluid and the metal of the subterranean-formed metal-polymer complex includes metal naturally present within the subterranean formation;
- (iii) maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex for a contact time of between about 1 minute and about 100 days, thereby allowing the metal complexing agent to cause the subterranean-formed metal-polymer complex to dissociate and dissolve but not precipitate the metal and thereby creating a low viscosity flow back fluid comprising the spent metal-polymer complex remediation mixture and the metal-polymer complex components; and
- (iv) removing the low viscosity flow back fluid from the well after the contact time, thereby improving hydrocarbon production of the well.

Ex. 1001, 2:35–53.

47. With regards to the metal complexing agent, the '282 Patent provides an expansive definition: “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 acid.” *Id.* at 5:16–21 (emphasis added).

48. Notably, the use of chelating agents in well treatment solutions has been well-known prior to the effective filing date of the '282 Patent, as will be discussed below. The specification of the '282 Patent further provides that “[t]he metal-polymer complex remediation mixture can contain the metal complexing agent as well as multiple chemical additives as desired. The additives may include biocide, scale inhibitor, clay control additive, oxygen scavenger and surfactant that assist fluid recovery.” *Id.* at 6:56–60.

49. Additionally, the '282 Patent discloses that the additives may include “a corrosion inhibitor”; “a surfactant”; “a biocide”; “a dispersing agent”; “a colloidal silica deposition inhibitor”; “a mutual solvent”; and “[a]cid...in order to control the pH of the remediation mixture.” *See id.* at 7:5–8:27. The use of these additives in well treatment mixtures was also well-established prior to the effective filing date of the '282 Patent.

50. These additives too were used and described for use for the claimed

methods of the '282 Patent prior to October 25, 2013.

V. CLAIM CONSTRUCTIONS

51. I have been told in the related district court litigation that Patent Owner has proposed construing the various claim terms of the '282 Patent using their plain and ordinary meaning to a POSITA.

52. 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. Ayers

53. I was provided with a copy of United States Patent No. 9,410,405 B2 to Ayers et al (Ex. 1004).

54. Ayers, entitled “Compositions and Methods for Enhanced Hydrocarbon Recovery,” discloses “a method of using a fluid formulation for increasing flow, production, or recovery of oil and gas hydrocarbons from a subterranean formation.” Ex. 1004, Abstract. Ayers discloses a fluid formulation for a composition including a peroxygen, surfactant, alkali metal chelate, and a cosolvent. *Id.* at 1:51–53.

55. Ayers describes “[a] method for enhancing hydrocarbon recovery in a subterranean formation having a blockage or accumulation of material” generally including the following steps:

- (i) introducing components comprising a) a peroxygen, b) a surfactant, c) an alkali metal chelate, and d) a cosolvent into a hydrocarbon bearing subterranean formation with a blockage or accumulation of material,
- (ii) generating at least one of oxygen gas and carbon dioxide gas via decomposition of the peroxygen to create gas pressure in the subterranean formation, and
- (iii) mobilizing, degrading, removing, releasing, realigning or redistributing the material creating the blockage or accumulation in the subterranean formation physically via generated gas pressure and solubilization by the surfactant,
- (iv) wherein the aggregate pH of the components is less than about 10 and/or a concentration of alkali metal chelate is greater than about 0.2% and less than about 5% by weight of non-water components.

Id. at 49:10–29.

56. With regards to the alkali metal chelate, Ayers explains that “[t]he alkali metal chelate may be, for example, a mono-, di-, tri- or tetra-sodium ethylenediaminetetraacetic acid (EDTA), a mono-, di-, tri- or tetra-potassium ethylenediaminetetraacetic acid (EDTA), sodium ethylenediamine-N,N'-disuccinic acid (EDDS) or combination thereof. In some embodiments, the alkali metal chelate is sodium EDTA.” *Id.* at 21:27–36.

B. Frenier

57. I was provided with a copy of United States Patent No. 6,436,880 B1 to Frenier (Ex. 1005).

58. 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).” Ex. 1005 at 4:49–53.

59. 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.

60. 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.

61. 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.

62. 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.

63. 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).

64. 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.

65. 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.’

66. Frenier then more specifically describes the process of using the “well treatment fluid compositions” in a matrix acidizing treatment. *Id.* at 7:20–37.

67. 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.

C. Motivation to Combine Ayers and Frenier

68. To the extent that it is needed, a POSITA would have been motivated to combine the teachings of Ayers and Frenier to produce obvious, beneficial, and predictable results. Ayers and Frenier are analogous prior art and both pertain to the

same field of endeavor. Specifically, both seek to enhance hydrocarbon production using treatment mixtures that include chelating agents.

69. As discussed in both Ayers and Frenier, 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.

70. Ayers discloses a composition for enhanced hydrocarbon recovery, acknowledging its potential use as a stimulation fluid. Ex. 1004 at 27:14–14 (“The composition according to an embodiment of the present invention can be for a drilling fluid, treatment fluid, stimulation fluid, fracturing fluid, and/or a fluid used in an enhanced oil recovery technique.”).

71. Notably, acid treatments are recognized as a well-established stimulation technique within the industry. *Id.* at 11:35–37 (“Matrix stimulation treatments using acids, or acids in combination with water, surfactants, or other chemicals, are very common and also referred to as ‘acid treatments’ or ‘acidizing’.”).

72. Frenier, on the other hand, specifically discloses a fluid formulation for an acid treatment for use in stimulation activities. Specifically, Frenier

discloses an “acid fluid that is useful in stimulation and workover operations, and in particular, for the control of iron in acidizing operations.” Ex. 1005 at Abstract.

73. A POSITA at the time of filing would have been highly motivated to combine the teachings of Ayers and Frenier. First, a POSITA would have recognized that Ayers’ composition, aimed at enhancing hydrocarbon recovery, possesses properties that could be beneficial for stimulation fluids. Combining it with Frenier’s dedicated matrix acidizing fluid could create a synergistic effect, potentially leading to a more effective stimulation treatment. Second, as Ayers acknowledges acid treatments as a stimulation technique, a POSITA would have been aware of the established benefits of incorporating components of an acid treatment mixture into a stimulation fluid. Frenier’s matrix acidizing fluid specifically addresses this need, and combining it with Ayers’ composition offers a potential pathway to an improved stimulation fluid.

74. Finally, a POSITA would have recognized that it was possible that Ayers’ composition, while beneficial for enhancing hydrocarbon recovery, might lack some of the specific properties desired for optimal stimulation. A POSITA, familiar with Frenier’s treatment fluid, would be motivated to combine the technologies to address these potential shortcomings and create a more versatile stimulation fluid. Moreover, a POSITA would have a reasonable expectation of success in combining the teachings of Ayers and Frenier.

VII. SPECIFIC GROUNDS FOR PETITION

75. Below I provide my analysis of the prior art as compared to various claims of the '282 Patent.

76. In particular, I have concluded there are two bases for invalidating the Challenged Claims of the '282 Patent. Ground I: Claims 1–3, 11–14, and 19 are anticipated by Ayers. Ground II: Claims 1–22 are obvious over Ayers alone or in combination with Frenier.

A. Ground I: Claims 1–3, 11–14, and 19 are Anticipated under 35 U.S.C. § 102 by Ayers

1. Independent Claim 1

a. Element 1[Preamble]: “A method for remediating a subterranean-formed metal-polymer complex in a pre-existing well in a subterranean shale formation, the method comprising:”

77. To the extent the preamble is limiting, Ayers discloses this limitation.

78. Ayers discloses “a method of using a fluid formulation for increasing flow, production, or recovery of oil and gas hydrocarbons from a subterranean formation.” Ex. 1004 at Abstract.

79. The method in Ayers aims to alleviate reductions in the productivity of pre-existing oil and gas wells in subterranean formations.

80. Ayers identifies the following, and other similar factors, as potential causes of reduced flow, production, or recovery of oil and gas hydrocarbons from

a subterranean formation: “[t]he formation of immobile emulsions, gels or polymers, the incomplete recovery of stimulation or fracking liquids, and the formation of larger particle blockages that may include precipitates, flocculants and/or colloids with clays, minerals, paraffins, asphaltenes, and condensates also can lead to clogging and reduction of oil and gas recovery from subterranean formations.” *Id.* at 5:41–47.

81. Ayers further identifies that “damage or blockage causing polymers can include polysaccharide polymers, cross-linked polymers, mannosidic and/or galactomannosidic linkages of guar, cellulose, cellulose derived, guar, guar derived, starches, xanthans, and/or other viscosity increasing polymers.” *Id.* at 24:1–6.

82. Accordingly, to the extent the preamble is limiting, Ayers discloses this limitation.

b. Element 1[a]: “providing a metal-polymer complex remediation mixture comprising between 0.1% and 95% by weight metal complexing agent;”

83. Ayers discloses a “fluid formulation” or “composition,” i.e., a metal-polymer complex remediation mixture, including “a peroxygen, a surfactant, a alkali metal chelate, and a cosolvent.” Ex. 1004 at 1:51–53.

84. Ayers explains that “an alkaline chelate can be added to the formation

to **chelate metals associated with the formation matrix** or pore liquids.” *Id.* at 29:61–62 (emphasis added).

85. According to the ’282 Patent, “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.” Ex. 1001 at 5:16–21 (emphasis added).

86. Here, the alkali metal chelate component of the fluid formulation in Ayers discloses a metal complexing agent.

87. Ayers provides that the chelate component of its fluid formulation may be, *inter alia*, EDTA, EDDS, or salts thereof. *See* Ex. 1004 at 21:27–36.

88. The specification of ’282 Patent includes EDTA as a “suitable complexing agent” and “any salt thereof, any derivative thereof, and the like.” *See* Ex. 1001 at 5:60–6:9.

89. Additionally the ’282 Patent also identifies EDDS and any salt or derivative thereof, as a suitable complexing agent. *See* Ex. 1004 at 6:10–38.

90. Ayers further discloses that the chelate concentration “may be between about **0.2 and about 5 percent by weight**,” which is a specific range within the claimed range (i.e., between 0.1% and 95% by weight). Ex. 1004 at 36:27–29.

91. As such, Ayers anticipates this limitation of Element 1[a]

c. **Element 1[b]: “injecting the metal-polymer complex remediation mixture into the well at a pressure less than a fracture pressure of the subterranean formation until at least some of the metal-polymer complex remediation mixture contacts the subterranean-formed metal-polymer complex, wherein the subterranean-formed metal polymer complex forms from a previously injected fracturing fluid and the metal of the subterranean-formed metal-polymer complex includes metal naturally present within the subterranean formation;”**

92. Ayers discloses injecting a metal-polymer complex remediation mixture into the well at a pressure less than the fracture pressure of the subterranean formation.

93. Specifically, Ayers discloses that “[t]he composition may be introduced into the wellbore or subterranean formation by, for example, pumping, displacing, or locating the composition at a desired location...**at a rate and pressure less than the reservoir hydraulic fracture pressure.**” Ex. 1004 at 36:65–37:3 (emphasis added).

94. Moreover, Ayers discloses that components of the fluid formulation may be “**injected** separately or in subcombinations” or “**injected** together as a single composition.” *See id.* at 42:43–49 (emphasis added).

95. Accordingly, Ayers discloses injecting the metal-polymer complex remediation mixture into the well at a pressure less than a fracture pressure of the

subterranean formation, as claimed in Element 1[b] of the '282 Patent.

96. Ayers also discloses that “[t]he introduced **composition may be brought into contact with, for example, blockage or damage in the wellbore or subterranean formation.**” *Id.* at 38:29–31.

97. Ayers further explains that said blockage or damage may result from previously injected fracturing fluid:

Inevitably, hydraulic fracturing also typically results in some level of formation damage and blockage as the **fracturing fluids intrude the natural formation and have negative interactions** in the way of swelling certain clays, migrating fines, introducing microbial growth and biofouling, **forming immobile emulsions or gels**, and forming larger particle blockages that may include precipitates, flocculants and/or colloids with clays, minerals, paraffins, asphaltenes, and/or condensates.

Id. at 8:27–35.

98. As such, Ayers discloses that subterranean formations of metal-polymer complexes may result due to previous injections of fracturing fluid. For example, fracturing fluid containing polyacrylamide, a common polymer additive to fracturing fluid, may intrude the natural formation and have negative interactions in the way of forming a metal-polymer complex with metal ions naturally present in the formation.

99. Ayers discloses the presence of transition metals in the formation. *See id.* at 28:20–21 (“[t]he presence of inorganic transition metals, either naturally

present in pore liquids (oil or water) or the formation”); *Id.* at 29:59–60 (“solid phase transition metals associated with the formation matrix”).

100. Moreover, Ayers recognizes that, “[o]ver time **stranded polymer** in the well bore, filter cake, and **in adjacent subterranean formations can form matrices that block oil flow** to and in a well including polymers, oil, asphaltenes, paraffins, clays, inorganic precipitates, and biological cells and biofilms.” *Id.* at 13:28–32 (emphasis added).

101. Further, Ayers discloses that, in some embodiments, “the damage or blockage may include viscosity increasing polymers. **The polymer may have been introduced as a separate treatment fluid.** The polymer may include, for example, polysaccharide polymer, **cross-linked polymers**, mannosidic and galacomannosidic linkages of guar, cellulose, cellulose derived guar, starch es, xanthans, other viscosity increasing polymer, or combinations thereof.” *Id.* at 38:46–53 (emphasis added).

102. Accordingly, Ayers discloses the limitations of Element 1[d].

d. Element 1[c]: “maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex for a contact time of between about 1 minute and about 100 days, thereby allowing the metal complexing agent to cause the subterranean-formed metal-polymer complex to dissociate and dissolve but not precipitate the metal and thereby creating a low viscosity flow back fluid comprising the spent metal-polymer complex remediation mixture and the

metal-polymer complex components; and”

103. Ayers discloses maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex. *Id.* at 22:20–24 (“As part of the methods for application the **composition can be allowed to contact** the wellbore or a portion of a well bore or **subterranean formation or hydrocarbon-bearing subterranean formation** for a sufficient period of time to increase flow, production, and/or recovery of hydrocarbons.”).

104. Ayers, as established above, discloses the existence of subterranean-formed metal-polymer complexes occurring within the subterranean formation. Accordingly, those in-situ formed metal-polymer complexes would necessarily be encountered by the composition disclosed in Ayers.

105. Further, Ayers discloses contacting the metal-polymer complex remediation mixture with the subterranean-formed metal-polymer complex for a contact time between about 1 minute and 100 days. For instance, Ayers discloses the following regarding the contact time for the disclosed composition:

Such a sufficient or adequate time can be, for example, from about 1 minute, 2 minutes, 5 minutes, 15 minutes, 30 minutes, 1 hour, 2 hours, 6 hours, 12 hours, 1 day, 2 days, 4 days, 1 week, 2 weeks, 1 month, 2 months, 3 months, or 6 months to about 2 minutes, 5 minutes, 15 minutes, 30 minutes, 1 hour, 2 hours, 6 hours, 12 hours, 1 day, 2 days, 4 days, 1 week, 2 weeks, 1 month, 2 months, 3 months, 6 months, or 12 months.

Id. at 22:32–39.

106. In one Example, Ayers discloses a contact time of 8 to 16 hours, which is a specific range within the claimed range (i.e., between about 1 minute and about 100 days). As such, Ayers anticipates this limitation of Element 1[a].

107. Further, Ayers makes clear that the disclosed composition can be “[a]llowed to contact blockage or damage in a portion of the wellbore or a portion of a subterranean formation for **a sufficient time** at the ambient temperature of the well bore or the subterranean formation to sufficiently alter, remove, **degrade, and/or dissolve the permeability damage or blockage...**” *Id.* at 23:30–36 (emphasis added).

108. Accordingly, any contact time between the composition in Ayers, which includes a chelating agent (i.e., a metal complexing agent), and a subterranean-formed metal-polymer complex (i.e., the permeability blockage or damage), for a time sufficient to degrade and/or dissolve the damage and or blockage would inherently seem to meet the limitation of at least “about” 1 minute of contact time.

109. Furthermore, as previously discussed, Ayers explains that the disclosed composition induces the dissociation and dissolution of subterranean-formed metal-polymer complexes. *See id.* at 23:30–36. Ayers discloses a composition including a chelating agent, i.e., a metal complexing agent. Such chelating agents would prevent metal ion precipitation by forming complexes that

keep the metals in solution. *See* Ex. 1004 at 1:63–65 (“The alkali metal chelate can serve the purpose of scavenging ionic or **bound phases of metals in a formation**[.]”) (emphasis added).

110. Ayers further discloses that the dissociation and dissolution of the subterranean-formed metal-polymer complexes generate a low viscosity flowback fluid comprising the spent metal-polymer complex remediation mixture and metal-polymer complex components. Ayers explains that the disclosed composition will “liquefy” encountered inorganic and organic materials, the “liquified material” then displaced through the formation and into the well for subsequent removal. *See id.* at 28:25–35; *see also id.* at 42:36–38 (“In a method according to the invention, components or a composition that have been injected into a subterranean formation can be **removed** from the wellbore.”) (emphasis added).

111. Additionally, Figure 3 of Ayers shows an example of materials evacuated from a well subsequent to treatment with the disclosed composition. Ayers notes that the evacuated materials from Figure 3 depict “flowable characteristics that readily enable pumping as a liquid.” *Id.* at 43:40–41.

112. A POSITA at the time of filing would understand that to mean that to disclose a low viscosity flow back fluid.

113. Accordingly, Ayers discloses the limitations of Element 1[c].

e. Element 1[d]: “**removing the low viscosity flow back fluid from the well after the contact time,**

thereby improving hydrocarbon production of the well.”

114. Ayers discloses that “the composition is removed from the wellbore after introduction. **The composition may be removed from the wellbore**, for example, by pumping or flushing.” *Id.* at 37:41–44 (emphasis added).

115. Ayers further explains that “after the composition is introduced into the wellbore or subterranean formation, the wellbore or **subterranean formation is contacted** for a period of time sufficient **to increase flow, production, or recovery of hydrocarbons.**” *Id.* at 36:60–64 (emphasis added); *see also id.* at 22:20–24.

116. Accordingly, Ayers discloses removing the low viscosity flow back fluid after the contact time, thereby improving hydrocarbon production of the well. *See also id.* at 42:36–38.

117. Accordingly, Ayers discloses the limitations of Element 1[d].

2. Dependent Claim 2

a. “The method of claim 1, wherein the metal complexing agent is citric acid, formic acid, or mixtures thereof.”

118. First, Ayers discloses that “[i]n some embodiments, an organic acid, mineral acid, or a base is added to the composition to adjust the pH. The acid may be, for example, hydrochloric, hydrofluoric, **formic, citric** or acetic acid.” *Id.* at 35:66–36:2 (emphasis added).

119. While Ayers highlights the pH-adjusting function of these acids, the ability of these acids to perform other functions due to their inherent properties remain unrestricted.

120. Crucially, a POSITA, prior to the effective date of the '282 Patent, would have recognized as inherent citric and formic acid's well-established chelating properties, rendering them suitable metal complexing agents within the disclosed subterranean treatment process.

121. Additionally, Ayers discloses that the use of citric acid, formic acid, or mixtures thereof, within fluid formulations for treating subterranean formations to achieve increased permeability and, consequently, enhance hydrocarbon production: "Matrix stimulation treatments using acids, or acids in combination with water, surfactants, or other chemicals, are **very common** and also referred to as 'acid treatments' or 'acidizing'. Hydrochloric acid (HCl), acetic acid, **citric, formic** and hydrofluoric acid **are commonly used** acids for matrix treatment." *Id.* at 11:35–40 (emphasis added).

122. It is common knowledge within the relevant industry that citric acid, formic acid, and mixtures thereof, possess effective chelating properties. Moreover, given the acknowledged purpose of the claimed invention—to enhance hydrocarbon recovery—it would be inherent to select a material capable of binding with metal ions, which are often present in subterranean formations and can operate

to impede fluid flow. Ex. 1015 at p. 3

123. Thus, Ayers discloses the limitations of Dependent Claim 2.

3. Dependent Claim 3

- a. **“The method of claim 1, wherein the metal complexing agent is selected from the group consisting 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, salts thereof, and mixtures thereof.”**

124. Ayers discloses that “[c]ompositions and methods of embodiments according to the present invention include or require the use of a chelate, such as a mono-, di-, tri-, or tetra-sodium ethylenediaminetetraacetic acid (EDTA), a mono-, di-, tri-, or tetra-potassium ethylenediaminetetraacetic acid (EDTA)...For example, the chelate can be sodium EDTA.” Ex. 1004 at 21:27–36.

125. As such, Ayers discloses wherein the metal complexing agent is selected from a group including EDTA and salts thereof, such as mono-, di-, tri-, or tetra- sodium and/or potassium EDTA.

126. Accordingly, Ayers discloses the limitations of Dependent Claim 3.

4. Dependent Claim 11

a. “The method of claim 1, wherein the metal-polymer complex remediation mixture further comprises a pH modifier.”

127. Ayers discloses that “pH control of the composition can be enabled by use of alkalinity increasing pH buffers, such as caustic soda, sodium carbonate, sodium bicarbonate, lime or hydrated lime or with organic or inorganic acids in the case of lowering pH.” *Id.* at 28:1–4; *see also id.* at 20:53–54.

128. Accordingly, Ayers disclose the limitations of Dependent Claim 11.

5. Dependent Claim 12

a. “The method of claim 11, wherein the metal-polymer complex remediation mixture further comprises between 0.1 and 30% of one or more acids as a pH modifier.”

129. I incorporate herein my statements and conclusions above concerning Dependent Claim 11. Ayers discloses that the pH modifier may be one or more acids: “[i]n some embodiments, an **organic acid, mineral acid**, or a base is added to the composition to adjust the pH.” Ex. 1004 at 35:66–67 (emphasis added).

130. Additionally, Ayers discloses that the composition described therein may “be augmented with less than 1% acids.” *Id.* at 32:28–29. This concentration falls squarely within the claimed range.

131. Further, since Ayers discloses that “the pH may be adjusted by adding

an acid or base to the composition,” and discloses, for example, that “[t]he composition may have a pH less than about 10.0.” *Id.* at 41:60–61; *id.* at 35:18–19.

132. Consequently, Ayers inherently anticipates the inclusion of an acid pH modifier in an amount falling within the claimed range, as adjusting the pH requires the presence of an acid pH modifier in an amount sufficient to attain the desired pH level.

133. Accordingly, Ayers discloses the limitations of Dependent Claim 12.

6. Dependent Claim 13

- a. “The method of claim 12, wherein the acid is selected from the group consisting of hydrochloric acid, aqua regia, arsenic acid, boric acid, carbonic acid, chloric acid, chromic acid, fluoroantimonic acid, fluoroboric acid, fluorosulfuric acid, fulminic acid, hexafluorophosphoric acid, hexafluorosilicic acid, hydrobromic acid, hydrofluoric acid, hydrogen iodide, hypochlorous acid, hypofluorous acid, hypophosphoric acid, iodic acid, nitric acid, nitrosyl-O-hydroxide, nitrous acid, orthocarbonic acid, perchloric acid, permanganic acid, perrhenic acid, pertechnetetic acid, phosphoric acid, silicic acid, sulfuric acid, thiocyanic acid, titanilic acid, tungstic acid, xenic acid, and mixtures thereof.”**

134. I incorporate herein my statements and conclusions above concerning Dependent Claim 11 and 12. Ayers discloses that “[t]he acid may be, for example,

hydrochloric, hydrofluoric, formic, citric or acetic acid.” Ex. 1004 at 35:67–36:4 (emphasis added).

135. Accordingly, Ayers discloses the limitations of Dependent Claim 13.

7. Dependent Claim 14

a. “The method of claim 12, wherein the acid is hydrochloric acid.”

136. I incorporate herein my statements and conclusions above concerning Dependent Claim 11, 12, and 13. As discussed above, Ayers discloses wherein the acid is hydrochloric acid. *See id.* at 35:66–36:4.

8. Dependent Claim 19

a. “The method of claim 1, wherein the contact time is between about 4 hours and about 4 days.”

137. With regards to contact time, Ayers discloses that “[i]n some embodiments, the composition is allowed to remain in the wellbore or subterranean formation for a period of time before hydrocarbon extraction resumes.” Ex. 1004 at 8:59–61.

138. Specifically, Ayers discloses that the contact time may be within a range that is “more than 4 hours” and “less than...4 days.” This range falls squarely within the range claimed by the ’282 Patent.

In some embodiments, the composition is allowed to remain in the wellbore or subterranean formation for a period of time before hydrocarbon extraction resumes. **The time may be more than, for example, more than 4**

hours, 6 hours, 8 hours, 10 hours, 12, hours, 16 hours, 20 hours, 24 hours, 30 hours, 36 hours, 42 hours, 48 hours, 3 days, 4 days, 5 days, 1 week. The time may be, for example, less than 30 days, 4 weeks, 3 weeks, 20 days, 2 weeks, 10 days, 1 week, 5 days, 4 days, 3 days.

Id. at 38:59–67.

139. Ayers makes clear that the disclosed composition can be “[a]llowed to contact blockage or damage in a portion of the wellbore or a portion of a subterranean formation for **a sufficient time** at the ambient temperature of the well bore or the subterranean formation to sufficiently alter, remove, **degrade, and/or dissolve the permeability damage or blockage...**” *Id.* at 23:30–36 (emphasis added).

140. Accordingly, any contact time between the composition in Ayers, which includes a chelating agent (i.e., a metal complexing agent), and a subterranean-formed metal-polymer complex (i.e., the permeability blockage or damage), for a time sufficient to degrade and/or dissolve the damage and or blockage would seem to meet the limitation of at least “about” 4 hours of contact time.

141. As such, Ayers discloses the limitation wherein the contact time is between four (4) hours and about four (4) days.

B. Ground II: Claims 1–22 are Obvious under 35 U.S.C. § 103 Over Ayers alone and/or in View of Frenier

1. Independent Claim 1

a. Element 1[Preamble]: “A method for remediating a subterranean-formed metal-polymer complex in a pre-existing well in a subterranean shale formation, the method comprising:”

142. To the extent the preamble is limiting, Ayers discloses this limitation.

143. Ayers discloses “a method of using a fluid formulation for increasing flow, production, or recovery of oil and gas hydrocarbons from a subterranean formation.” Ex. 1004 at Abstract.

144. The method in Ayers aims to alleviate reductions in the productivity of pre-existing oil and gas wells in subterranean formations.

145. Ayers identifies the following, and other similar factors, as potential causes of reduced flow, production, or recovery of oil and gas hydrocarbons from a subterranean formation: “[t]he formation of immobile emulsions, gels or polymers, the incomplete recovery of stimulation or fracking liquids, and the formation of larger particle blockages that may include precipitates, flocculants and/or colloids with clays, minerals, paraffins, asphaltenes, and condensates also can lead to clogging and reduction of oil and gas recovery from subterranean formations.” *Id.* at 5:41–47.

146. Ayers further identifies that “damage or blockage causing polymers can include polysaccharide polymers, cross-linked polymers, mannosidic and/or galactomannosidic linkages of guar, cellulose, cellulose derived, guar, guar

derived, starches, xanthans, and/or other viscosity increasing polymers.” *Id.* at 24:1–6.

147. Additionally, Frenier also discloses this limitation, to the extent the preamble is limiting. Frenier discloses “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. 1005 at 1:55–60 (emphasis added).

148. It would have been obvious to a POSITA to apply the disclosure of Ayers to a method for remediating a subterranean formed metal-polymer complex in a pre-existing well in a subterranean formation.

149. Moreover, Frenier discloses this limitation, to the extent the preamble is limiting. Frenier discloses “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. 1005 at 1:55–60 (emphasis added).

150. A POSITA would have understood Frenier’s disclosure to apply to remediating a subterranean-formed metal-polymer complex.

151. Further, Frenier discloses [a]n acid fluid that is useful in stimulation

and **workover operations**, and in particular for the control of iron in acidizing operations, the removal of alkaline earth carbonate scale in scale removal operations, and matrix or fracture acidizing operations...” *Id.* at Abstract (emphasis added).

152. 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. 1015 at p. 1.

153. Accordingly, to the extent the preamble is limiting, Ayers, either alone or in combination with Frenier, discloses this limitation.

7. It is my opinion that each limitation of the preamble of claim 1, as described above, was disclosed by Ayers, either alone or in combination with Frenier, prior to October 25, 2013.

b. Element 1[a]: “providing a metal-polymer complex remediation mixture comprising between 0.1% and 95% by weight metal complexing agent;”

154. Ayers discloses a “fluid formulation” or “composition,” i.e., a metal-polymer complex remediation mixture, including “a peroxygen, a surfactant, a alkali metal chelate, and a cosolvent.” Ex. 1004 at 1:51–53. Ayers explains that “an alkaline chelate can be added to the formation to **chelate metals associated**

with the formation matrix or pore liquids.” *Id.* at 29:61–62 (emphasis added).

155. According to the ’282 Patent, “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.” Ex. 1004 at 5:16–21 (emphasis added). Here, the alkali metal chelate component of the fluid formulation in Ayers discloses a metal complexing agent.

156. With regards to the chelate component, Ayers provides:

Compositions and methods of embodiments according to the present invention include or require the use of a chelate, such as a mono-, di-, tri-, or tetra-sodium ethylenediaminetetraacetic acid (EDTA), a mono-, di-, tri-, or tetra-potassium ethylenediaminetetraacetic acid (EDTA) or sodium ethylenediamine-N,N-disuccinic acid (EDDS), or combinations. The selected chelate can be present in a final concentration as applied to the wellbore or subterranean formation that varies from 0.0001 to 5.0 percent by weight. For example, the chelate can be sodium EDTA.

Id. at 21:27–36.

157. Ayers provides that the chelate component of its fluid formulation may be, *inter alia*, salts of EDTA. *Id.*

158. The specification of ’282 Patent includes EDTA as a “suitable complexing agent” and “any salt thereof, any derivative thereof, and the like.” *See* Ex. 1004 at 5:60–6:9.

159. Additionally the '282 Patent also identifies EDDS and any salt or derivative thereof, as a suitable complexing agent. *See id.* at 6:10–38.

160. Ayers further discloses that the chelate concentration “may be between about **0.2 to about 5.0 percent by weight**,” which is a specific range within the claimed range (i.e., between 0.1% and 95% by weight). Ex. 1004 at 36:20–22 (emphasis added).

161. Moreover, Frenier discloses a “well treatment fluid,” i.e., a remediation mixture, comprising: “a first acid; water; and a hydroxyethylaminocarboxylic acid.” Ex. 1005 at 4:12–13.

162. 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.

163. 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). “Preferably, the hydroxyethylaminocarboxylic acid is selected from hydroxyethylethylenediaminetriacetic acid (HEDTA), hydroxyethyliminodiacetic acid (HEIDA), or a mixture thereof.” *Id.* at 4:13–17.

164. 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 an organic acid such as citric, formic, , or acetic acid. *Id.*

165. A POSITA would have known and understood the same.

166. 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 Independent Claim 1[a]. Ex. 1005 at 6:25–47. 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. 1005, 11:27–34; 11:54–63; 13:3–20.

167. 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.

168. Moreover, a POSITA, prior to the effective date of the '282 Patent, would have recognized that the specific proportion of metal complexing agent within the claimed remediation mixture is likely to vary depending on several factors, including the unique characteristics of the subterranean formation being

treated. *See* Ex. 1005 at 6:16–19 (“The 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.”); *see also* Ex. 1004 at 19:27–33 (“Factors affecting concentrations of the components of the composition to be applied include the magnitude or extent of the deposits or blockages to be removed, and the composition of the deposits...or blockages.”).

169. The broad ranges disclosed in Ayers and Frenier provide ample room for a POSITA to make adjustments. Given the vastness of these concentration ranges and the well-understood functionality of metal complexing agents, a POSITA could readily arrive at successful formulations through routine experimentation. The established functions of metal complexing agents provide a predictable framework for optimizing the composition for a particular application. A POSITA, familiar with these functionalities and the specific needs of a treatment project, could readily adjust the ratios within the claimed broad ranges to achieve the desired outcome.

170. This level of optimization would not require inventive leaps but rather routine calculations and experimentation based on established scientific principles. Overall, the broadness of the claimed ranges and the predictability associated with optimizing the composition based on well-known functionalities

render the invention obvious to a POSITA.

171. Thus, it is my opinion that Ayers, either alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses Element 1[a].

- c. **Element 1[b]: “injecting the metal-polymer complex remediation mixture into the well at a pressure less than a fracture pressure of the subterranean formation until at least some of the metal-polymer complex remediation mixture contacts the subterranean-formed metal-polymer complex, wherein the subterranean-formed metal polymer complex forms from a previously injected fracturing fluid and the metal of the subterranean-formed metal-polymer complex includes metal naturally present within the subterranean formation;”**

172. Ayers discloses injecting a metal-polymer complex remediation mixture into the well at a pressure less than the fracture pressure of the subterranean formation.

173. Specifically, Ayers discloses that “[t]he composition may be introduced into the wellbore or subterranean formation by, for example, pumping, displacing, or locating the composition at a desired location. In some embodiments, the pumping, displacing, or locating the composition is **at a rate and pressure less than the reservoir hydraulic fracture pressure.**” Ex. 1004 at 36:65–37:3 (emphasis added).

174. Moreover, Ayers discloses that components of the fluid formulation may be **“injected** separately or in subcombinations” or **“injected** together as a

single composition.” *See id.* at 42:43–49 (emphasis added). Accordingly, Ayers discloses injecting the metal-polymer complex remediation mixture into the well at a pressure less than a fracture pressure of the subterranean formation, as claimed in Element 1[b] of the ’282 Patent.

175. Ayers also discloses that “[t]he introduced **composition may be brought into contact with, for example, blockage or damage in the wellbore or subterranean formation.**” *Id.* at 38:29–31.

176. Ayers explains that said blockage or damage may result from previously injected fracturing fluid:

Inevitably, hydraulic fracturing also typically results in some level of formation damage and blockage as the **fracturing fluids intrude the natural formation and have negative interactions** in the way of swelling certain clays, migrating fines, introducing microbial growth and biofouling, **forming immobile emulsions or gels**, and forming larger particle blockages that may include precipitates, flocculants and/or colloids with clays, minerals, paraffins, asphaltenes, and/or condensates.

Id. at 8:27–35.

177. As such, Ayers discloses that subterraneous formations of metal-polymer complexes may result due to previous injections of fracturing fluid. For example, fracturing fluid containing polyacrylamide, a common polymer additive to fracturing fluid, may intrude the natural formation and have negative interactions in the way of forming a metal-polymer complex with metal ions

naturally present in the formation.

178. Ayers discloses the presence of transition metals in the formation. *See id.* at 28:20–21 (“[t]he presence of inorganic transition metals, either naturally present in pore liquids (oil or water) or the formation”); *Id.* at 29:59–60 (“solid phase transition metals associated with the formation matrix”).

179. Moreover, Ayers recognizes that, “[o]ver time **stranded polymer** in the well bore, filter cake, and **in adjacent subterranean formations can form matrices that block oil flow** to and in a well including polymers, oil, asphaltenes, paraffins, clays, inorganic precipitates, and biological cells and biofilms.” *Id.* at 13:28–32 (emphasis added).

180. Further, Ayers discloses that, in some embodiments, “the damage or blockage may include viscosity increasing polymers. **The polymer may have been introduced as a separate treatment fluid.** The polymer may include, for example, polysaccharide polymer, **cross-linked polymers**, mannosidic and galacomannosidic linkages of guar, cellulose, cellulose derived guar, starch es, xanthans, other viscosity increasing polymer, or combinations thereof.” *Id.* at 38:46–53 (emphasis added).

181. Additionally, Frenier discloses “a method of acid-treating a subterranean formation.” Ex. 1005 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). Frenier further explains 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)**...” *Id.* at 4:49–53 (emphasis added).

182. Thus, it is my opinion that Ayers, either alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses Element 1[b].

d. Element 1[c]: “maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex for a contact time of between about 1 minute and about 100 days, thereby allowing the metal complexing agent to cause the subterranean-formed metal-polymer complex to dissociate and dissolve but not precipitate the metal and thereby creating a low viscosity flow back fluid comprising the spent metal-polymer complex remediation mixture and the metal-polymer complex components; and”

183. Ayers discloses maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex. Ex. 1004 at 22:20–24 (“As part of the methods for application the **composition can be allowed to contact** the wellbore or a portion of a well bore or **subterranean formation or hydrocarbon-bearing subterranean formation** for a sufficient period of time to increase flow, production, and/or recovery of hydrocarbons.”).

184. Ayers, as established above, discloses the existence of subterranean-formed metal-polymer complexes occurring within the subterranean formation.

Accordingly, those in-situ formed metal-polymer complexes would necessarily be encountered by the composition disclosed in Ayers. *Id.*

185. Further, Ayers discloses contacting the metal-polymer complex remediation mixture with the subterranean-formed metal-polymer complex for a contact time between about 1 minute and 100 days. For instance, Ayers discloses the following regarding the contact time for the disclosed composition:

Such a sufficient or adequate time can be, for example, from about 1 minute, 2 minutes, 5 minutes, 15 minutes, 30 minutes, 1 hour, 2 hours, 6 hours, 12 hours, 1 day, 2 days, 4 days, 1 week, 2 weeks, 1 month, 2 months, 3 months, or 6 months to about 2 minutes, 5 minutes, 15 minutes, 30 minutes, 1 hour, 2 hours, 6 hours, 12 hours, 1 day, 2 days, 4 days, 1 week, 2 weeks, 1 month, 2 months, 3 months, 6 months, or 12 months.

Id. at 22:32–39.

186. In one Example, Ayers discloses a contact time of 8 to 16 hours, which is a specific range within the claimed range (i.e., between about 1 minute and about 100 days). *Id.* at 43:27.

187. Ayers makes clear that the disclosed composition can be “[a]llowed to contact blockage or damage in a portion of the wellbore or a portion of a subterranean formation for **a sufficient time** at the ambient temperature of the well bore or the subterranean formation to sufficiently alter, remove, **degrade, and/or dissolve the permeability damage or blockage...**” *Id.* at 23:30–36 (emphasis added).

188. Accordingly, any contact time between the composition in Ayers, which includes a chelating agent (i.e., a metal complexing agent), and a subterranean-formed metal-polymer complex (i.e., the permeability blockage or damage), for a time sufficient to degrade and/or dissolve the damage and or blockage would seem to meet the limitation of at least “about” 1 minute of contact time.

189. Furthermore, given the vastness of time within the range, a POSITA would recognize the optimal contact time by experimentation and trial and error since it has been held, “[w]here the general conditions of a claim are disclosed in the prior art, it is not inventive to discover the optimum or workable ranges by routine experimentation.” *In re Aller*, 220 F.2d 454, 456, 105 USPQ 233, 235 (CCPA 1955).

190. Furthermore, as previously discussed, Ayers explains that the disclosed composition induces the dissociation and dissolution of subterranean-formed metal-polymer complexes. *See id.* at 23:30–36.

191. Ayers discloses a composition including a chelating agent, i.e., a metal complexing agent. A POSITA at the time of filing would recognize that such chelating agents would prevent metal ion precipitation by forming complexes that keep the metals dissolve in solution. *see also* Ex. 1004 at 1:63–65 (“The alkali metal chelate can serve the purpose of scavenging ionic or **bound phases of metals**

in a formation[.]”) (emphasis added).

192. Ayers further discloses that the dissociation and dissolution of the subterranean-formed metal-polymer complexes generate a low viscosity flowback fluid comprising the spent metal-polymer complex remediation mixture and metal-polymer complex components. Ayers explains that the disclosed composition will “liquefy” encountered inorganic and organic materials, the “liquified material” then displaced through the formation and into the well for subsequent removal. *See id.* at 28:25–35; *see also id.* at 42:36–38 (“In a method according to the invention, components or a composition that have been injected into a subterranean formation can be **removed** from the wellbore.”).

193. Additionally, Figure 3 of Ayers shows an example of materials evacuated from a well subsequent to treatment with the disclosed composition. Ayers notes that the evacuated materials depict “flowable characteristics that readily enable pumping as a liquid.” *Id.* at 42:36–38.

194. A POSITA at the time of filing would understand that to mean that to disclose a low viscosity flow back fluid.

195. Similarly, 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. 1005 at 7:30–34.

196. 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).

197. Notably, in the examples provided in Frenier, the treatment solutions were maintained in contact with the formation samples for 24 and 72 hours. Ex. 1005 at 9:27–66.

198. Here, as with Ayers above, a POSITA would recognize the optimal contact time by experimentation and trial and error since it has been held, “[w]here the general conditions of a claim are disclosed in the prior art, it is not inventive to discover the optimum or workable ranges by routine experimentation.” *In re Aller*, 220 F.2d at 456.

199. It is my opinion that Ayers, either alone or in combination with Frenier, discloses the limitations of Element 1[c].

e. Element 1[d]: “removing the low viscosity flow back fluid from the well after the contact time, thereby improving hydrocarbon production of the well.”

200. Ayers discloses that “the composition is removed from the wellbore

after introduction. **The composition may be removed from the wellbore**, for example, by pumping or flushing.” *Id.* at 37:41–44 (emphasis added). Ayers further explains that “after the composition is introduced into the wellbore or subterranean formation, the wellbore or **subterranean formation is contacted** for a period of time sufficient **to increase flow, production, or recovery of hydrocarbons.**” *Id.* at 36:60–64 (emphasis added); *see also id.* at 22:20–24.

201. Accordingly, Ayers discloses removing the low viscosity flow back fluid after the contact time, thereby improving hydrocarbon production of the well. *See also id.* at 42:36–38.

202. Similarly, Frenier discloses removal of the low viscosity flow back fluid from the well after the contact time, thereby improving hydrocarbon production of the well. 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. 1005 at 1:55–60.

203. Frenier also incorporates by reference, U.S. Patent. No. 1,877,504A to Grebe, entitled “Treatment of Deep Wells,” issued September 13, 1932 (“Grebe”) (Ex. 1013). 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. 1013 at page 1, lines 45–52.

204. 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).

205. It is my opinion that, Ayers, either alone or in combination with Frenier, as informed by the knowledge of a POSITA, discloses this limitation.

2. Dependent Claim 2

a. “The method of claim 1, wherein the metal complexing agent is citric acid, formic acid, or mixtures thereof.”

206. First, Ayers discloses that “[i]n some embodiments, an organic acid, mineral acid, or a base is added to the composition to adjust the pH. The acid may be, for example, hydrochloric, hydrofluoric, **formic, citric** or acetic acid.” *Id.* at 35:66–36:2 (emphasis added). While Ayers highlights the pH-adjusting function of these acids, the ability of these acids to perform other functions due to their inherent properties remain unrestricted.

207. Crucially, a POSITA at the time of filing would have recognized

citric and formic acid's well-established chelating properties, rendering them suitable metal complexing agents within the disclosed subterranean treatment process.

208. Additionally, Ayers discloses that the use of citric acid, formic acid, or mixtures thereof, within fluid formulations for treating subterranean formations to achieve increased permeability and, consequently, enhance hydrocarbon production: "Matrix stimulation treatments using acids, or acids in combination with water, surfactants, or other chemicals, are **very common** and also referred to as 'acid treatments' or 'acidizing'. Hydrochloric acid (HCl), acetic acid, **citric, formic** and hydrofluoric acid **are commonly used** acids for matrix treatment." *Id.* at 11:35–40 (emphasis added).

209. A POSITA, at the time of filing, would have recognized citric acid, formic acid, and mixtures thereof, as well-suited metal complexing agents for the disclosed treatment process. It is common knowledge within the relevant industry that citric acid, formic acid, and mixtures thereof, possess effective chelating properties.

210. Moreover, given the acknowledged purpose of the claimed invention—to enhance hydrocarbon recovery—a POSITA would have understood the importance of selecting a material capable of binding with metal ions, which are often present in subterranean formations and can operate to impede fluid flow.

211. Thus, it is my opinion that Ayers, as informed by the knowledge of a POSITA, discloses every limitation of this dependent claim.

3. Dependent Claim 3

- a. **“The method of claim 1, wherein the metal complexing agent is selected from the group consisting 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, salts thereof, and mixtures thereof.”**

212. Ayers discloses that “[c]ompositions and methods of embodiments according to the present invention include or require the use of a chelate, such as a mono-, di-, tri-, or tetra-sodium ethylenediaminetetraacetic acid (EDTA), a mono-, di-, tri-, or tetra-potassium ethylenediaminetetraacetic acid (EDTA)...For example, the chelate can be sodium EDTA.” *Id.* at 21:27–36.

213. As such, Ayers discloses wherein the metal complexing agent is selected from a group including EDTA and salts thereof, such as mono-, di-, tri-, or tetra- sodium and/or potassium EDTA.

214. It would have been obvious to a POSITA before the effective filing date of the '282 Patent to select one of the acids, salts, or mixtures listed in Dependent Claim 3 in light of the disclosure in Ayers.

215. Thus, it is my opinion that Ayers, as informed by the knowledge of a POSITA, discloses every limitation of this dependent claim.

4. Dependent Claim 4

- a. **“The method of claim 1, further wherein the metal complexing agent is selected from the group consisting of 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, salts thereof, and mixtures thereof."

216. Ayers discloses that "[c]ompositions and methods of embodiments according to the present invention include or require the use of a chelate, such as...sodium ethylenediamine-N,N-disuccinic acid (EDDS)." *See id.* at 21:27–36.

217. As such, Ayers discloses wherein the metal complexing agent is selected from a group including EDDS and salts thereof, such as sodium EDDS.

218. It would have been obvious to a POSITA before the effective filing date of the '282 Patent to select one of the acids, salts, or mixtures listed in Dependent Claim 4 in light of the disclosure in Ayers.

219. Thus, it is my opinion that Ayers, as informed by the knowledge of a POSITA, discloses every limitation of this dependent claim.

5. Dependent Claim 5

- a. "The method of claim 1, further wherein the metal complexing agent is selected from acetic acid, acrylates, dihydroxymaleic acid, salts of dihydroxymaleic acid, ethylenediamine tetraacetic acid (EDTA), salts of EDTA,**

erythorbic acid, erythroboric acid, formic acid, gluconodeltalactone, glutamic acid N,N-diacetic acid (GLDA), salts of GLDA, hydroxyethylenediamine triacetic acid (HEDTA), salts of HEDTA, disodium ethanoldiglycine (HEIDA), salts of HEIDA, methylglycine N,N-diacetic acid (MGDA), salts of MGDA, nitriolotriacetic acid (NTA), and mixtures thereof.”

220. Ayers discloses that “[c]ompositions and methods of embodiments according to the present invention include or require the use of a chelate, such as a mono-, di-, tri-, or tetra-sodium ethylenediaminetetraacetic acid (EDTA), a mono-, di-, tri-, or tetra-potassium ethylenediaminetetraacetic acid (EDTA)...For example, the chelate can be sodium EDTA.” *Id.* at 21:27–36.

221. As such, Ayers discloses wherein the metal complexing agent is selected from a group including EDTA and salts thereof, such as mono-, di-, tri-, or tetra- sodium and/or potassium EDTA.

222. It would have been obvious to a POSITA before the effective filing date of the '282 Patent to select one of the acids, salts, or mixtures listed in Dependent Claim 5 in light of the disclosure in Ayers.

223. Thus, it is my opinion that Ayers, as informed by the knowledge of a POSITA, discloses every limitation of this dependent claim.

6. Dependent Claim 6

a. “The method of claim 1, wherein the metal-polymer complex remediation mixture

comprises between 10% and 80% by weight metal complexing agent, one or more additives, and mutual solvent.”

224. Ayers discloses a remediation mixture that includes, *inter alia*, a metal complexing agent (i.e., a chelating agent) and additional components such a surfactant and a cosolvent. Ex. 1004 at 21:27–36.

225. Ayers explains that the composition may contain a chelating agent having a concentration between 0.00001 to 5.0 percent by weight (*id.* at 36:20–22); a surfactant at a concentration between 0.01 and about 50 percent by weight (*id.* at 36:5–7); and a cosolvent having a concentration between about 1 to 40 percent by weight (*id.* at 36:46–48).

226. Notably, Ayers incorporates by reference U.S. Patent No. 6,593,279 (Von Krosig et al.), which, according to Ayers, teaches a composition for cleaning oil sludges from oil well cuttings, well formations, and down hole wells. *See id.* at 12:18–20. The Von Krosig reference discloses a composition requiring a “co-solvent,” and discloses the following “co-solvents”: “n-butanol, iso-butanol, n-butoxyl propanol, ethers, di-propylene glycol, mono butyl ether, glycols including propylene glycol, ethylene glycol, butylene glycol, hexylene glycol, di-propylene glycol, di-ethylene glycol, tri-propylene glycol, triethylene glycol, poly glycols; ethers, including mono-methyl ether, and mixtures of such co-solvents.” Ex. 1011 at 6:7–14. Additionally, Borchardt includes a discussion of mutual solvents used

in well stimulation fluids: “Mutual solvents have been used to reduce surfactant adsorption on formation minerals, particularly oil-wetting surfactants. Ethylene glycol monobutyl ether is the most commonly used mutual solvent.” Ex. 1014 at p. 14. “Oil-soluble corrosion inhibitors may be dissolved by organic solvents such as xylene or toluene containing a mutual solvent, most often ethylene glycol monobutyl ether, EGMBE. Aqueous fluids containing 5-10% EGMBE can be used to dissolve cationic surfactants.” Ex. 1014 at p. 18.

227. A POSITA would understand that aforementioned listing of “co-solvents” disclose mutual solvents, i.e., a chemical additive for stimulation treatments that is soluble in water, oil, and acid-based treatment fluids.

228. Similarly, Frenier discloses a well treatment fluid composition, containing, *inter alia*, a first acid, a hydroxyethylaminocarboxylic acid, and one or more additives, including surfactants, corrosion inhibitors, stabilizers, solvents, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. *See* Ex. 1005 at 4:12–13; 5:56–62.

229. 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 an organic acid such as formic, citric , or acetic acid. *See id.* at 4:23–27; 5:27–3.

230. Frenier further discloses that, if a solvent is used in the disclosed

formulation, “the solvent typically comprises an alcohol or a glycol ether.” *Id.* at 6:14–15.

231. A POSITA at the time of filing would have understood glycol ether to disclose a mutual solvent.

232. 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 Dependent Claim 6. *Id.* at 6:25–47. 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. Ex. 1005 at 11:27–34; 11:54–63; 13:3–20. Frenier further discloses that composition additives, e.g., wetting agents, non-emulsifiers, or solvents, may comprise between 0.1–10% and 0.1–5%. *See id.* at 6:25–45.

233. Accordingly, Ayers, either alone or in combination with Frenier, discloses a remediation mixture containing a metal complexing agent, one or more additives, and a mutual solvent, with the combined weight percentage of those components falling between 10% and 80%.

234. Moreover, a POSITA at the time of filing would have recognized that the specific proportions of various components within the claimed remediation mixture are likely to vary depending on several factors, including the unique

characteristics of the subterranean formation being treated. *See id.* at 6:16–19 (“The 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.”); *see also* Ex. 1004 at 19:27–33 (“Factors affecting concentrations of the components of the composition to be applied include the magnitude or extent of the deposits or blockages to be removed, and the composition of the deposits...or blockages.”).

235. The broad ranges disclosed in Ayers and Frenier provide ample room for a POSITA to necessary make adjustments. Given the vastness of these concentration ranges and the well-understood functionalities of the disclosed components (metal complexing agents, surfactants, solvents), a POSITA could readily arrive at successful formulations through routine experimentation. The established functions of these components provide a predictable framework for optimizing the composition for a particular application.

236. A POSITA, familiar with these functionalities and the specific needs of a treatment project, could readily adjust the component ratios within the claimed broad ranges to achieve the desired outcome. This level of optimization would not require inventive leaps but rather routine experimentation based on established scientific principles.

237. Overall, the broadness of the claimed ranges and the predictability

associated with optimizing the composition based on well-known functionalities render the invention obvious to a POSITA.

7. Dependent Claim 7

- a. “The method of claim 6, wherein the metal complexing agent comprises between 10% and 80% by weight citric acid, formic acid, or mixtures thereof, between 0.1 and 30% of one or more acids as a pH modifier, and mutual solvent.”**

238. As discussed above, Ayers discloses a remediation mixture that includes, *inter alia*, a metal complexing agent (i.e., a chelating agent) and additional components such a surfactant and a cosolvent. Ex. 1004 at 21:27–36.

239. Ayers explains that the composition may contain a chelating agent having a concentration between 0.00001 to 5.0 percent by weight [*Id.* at 36:20–22]; a surfactant at a concentration between 0.01 and about 50 percent by weight [*Id.* at 36:5–7]; and a cosolvent having a concentration between about 1 to 40 percent by weight [*Id.* at 36:46–48].

240. Notably, Ayers incorporates by reference U.S. Patent No. 6,593,279 (Von Krosig et al.), which, according to Ayers, teaches a composition for cleaning oil sludges from oil well cuttings, well formations, and down hole wells. *See id.* at 12:18–20. The Von Krosig reference discloses a composition requiring a “co-solvent,” and discloses the following “co-solvents”: “n-butanol, iso-butanol, n-butoxyl propanol, ethers, di-propylene glycol, mono butyl ether, glycols including

propylene glycol, ethylene glycol, butylene glycol, hexylene glycol, di-propylene glycol, di-ethylene glycol, tri-propylene glycol, triethylene glycol, poly glycols; ethers, including mono-methyl ether, and mixtures of such co-solvents.” Ex. 1011 at 6:7–14.

241. A POSITA would understand that aforementioned listing of “co-solvents” disclose mutual solvents, i.e., a chemical additive for stimulation treatments that is soluble in water, oil, and acid-based treatment fluids.

242. Additionally, Ayers discloses that “[t]he pH of the composition can be adjusted if need to avoid pH changes in the formation.” Ex. 1004 at 20:53–54. Specifically, Ayers discloses that “[i]n some embodiments, an organic acid, mineral acid, or a base is added to the composition to adjust the pH. The acid may be, for example, hydrochloric, hydrofluoric, formic, citric or acetic acid.” *Id.* at 35:66–36:2.

243. Accordingly, it would have been obvious to a POSITA at the time of filing that one or more acids may be used as a pH modifier.

244. Further, Frenier discloses a treatment fluid comprising, *inter alia*, a first acid: “Examples 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. 1005 at 5:27–31 (emphasis added).

245. Although Frenier refers to formic acid as a possible “first acid,” formic acid’s inherent properties as a metal complexing agent is not diminished. It would have been obvious to a POSITA at the time of filing to select formic acid for use as a metal complexing agent.

246. Frenier discloses that its treatment mixtures, without and without a first acid, may contain metal complexing agents in a concentration between 1% and 30% by weight, which covers much of the claimed ranges set forth in Dependent Claim 7. Ex. 1005 at 6:25–47.

247. 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. Ex. 1005 at 11:27–34; 11:54–63; 13:3–20.

248. Frenier further discloses a treatment mixture including, *inter alia*, 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). Frenier explains that pH of the treatment fluid “can be controlled by can **organic acid, a mineral acid**, or a base.” *Id.* at 4:57–60 (emphasis added).

249. Accordingly, Frenier discloses the use of one or more acids as a pH modifier. Frenier provides examples of treatment mixtures comprising 0.1% to 15% by weight pH modifier. *Id.* at 6:40–45, and composition additives, such as

solvents, comprising between 0.1–10% and 0.1–5% by weight. *See id.* at 6:25–45.

250. Accordingly, Ayers and Frenier, informed by the knowledge of a POSITA, discloses a remediation mixture containing citric acid, formic acid, or mixtures thereof at a concentration between 10% and 80% by weight; and one or more acids as a pH modifier and a mutual solvent, with a combined weight percentage falling between 0.1% ad 30% by weight.

8. Dependent Claim 8

- a. “The method of claim 1, wherein the metal-polymer complex remediation mixture comprises between 0.1% and about 60% by weight metal complexing agent.”**

251. Ayers discloses a formulation containing a metal complexing agent having a concentration of 0.00001 to 5.0 percent by weight. Ex. 1004 at 36:20–22.

252. Similarly, Frenier discloses a formulation containing a metal complexing agent having a concentration between 1% and 30% by weight, which covers much of the claimed ranges set forth in Dependent Claim 8. Ex. 1005 at 6:25–47.

9. Dependent Claim 9

- a. “The method of claim 1, wherein the metal-polymer complex remediation mixture further comprises a mutual solvent and an additive selected from the group consisting of acid inhibitor, corrosion inhibitor, scale inhibitor, clay control additive, surfactant, biocide, and mixtures thereof.”**

253. As discussed above, Frenier discloses a treatment mixture that may include a mutual solvent, such as one comprising a glycol ether, (Ex. 1005 at 6:14–15) and one or more additives, including surfactants, corrosion inhibitors, stabilizers, solvents, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. *See* Ex. 1005 at 5:56–62. Additionally, Borchardt includes a discussion of mutual solvents used in well stimulation fluids: “Mutual solvents have been used to reduce surfactant adsorption on formation minerals, particularly oil-wetting surfactants. Ethylene glycol monobutyl ether is the most commonly used mutual solvent.” Ex. 1014 at p. 14. “Oil-soluble corrosion inhibitors may be dissolved by organic solvents such as xylene or toluene containing a mutual solvent, most often ethylene glycol monobutyl ether, EGMBE. Aqueous fluids containing 5-10% EGMBE can be used to dissolve cationic surfactants.” Ex. 1014 at p. 18.

254. Accordingly, Frenier discloses a remediation mixture further comprising a mutual solvent and an additive selected from a group including surfactants and corrosion inhibitors.

10. Dependent Claim 10

- a. “The method of claim 1, wherein the metal-polymer complex remediation mixture comprises: between 1% and 95% by weight metal complexing agent;”**

255. As discussed above, Ayers discloses a formulation containing a metal

complex agent having a concentration of 0.00001 to 5.0 percent by weight. *See* Ex. 1004 at 36:20–22.

256. Similarly, Frenier discloses a formulation containing a metal complexing agent having a concentration between 1% and 30% by weight, which covers much of the claimed ranges set forth in Dependent Claim 10. Ex. 1005 at 6:25–47.

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

257. Frenier discloses a treatment mixture having one or more additives: “[t]he composition can also include one or more additives that are compatible with the acid composition, such as...**corrosion inhibitors** (such as the alkylphenones described in U.S. Pat. Nos. 5,013,483 and 5,096,618)...Corrosion inhibitors in particular should be selected with reference to the particular acid used in the composition.” *See* Ex. 1005 at at 5:56–65.

258. Frenier provides examples of treatment mixtures containing additives, such as corrosion inhibitors, at a concentration between 0.1–10% and 0.1–5% by weight. *See id.* at 6:25–45. A POSITA at the time of filing would understand that a treatment mixture containing additives, including a corrosion inhibitor, at a concentration between 0.1–10% and 0.1–5% by weight discloses a treatment mixture containing between 1 and 10,000 ppm of a corrosion inhibitor.

259. Even if Frenier doesn't explicitly mention 1-10,000 ppm for the

corrosion inhibitor, disclosing a broader range that includes it suggests that such concentrations are encompassed by the invention.

c. “between 1 and 10,000 parts per million (ppm) of a biocide;”

260. Frenier discloses a treatment mixture including the use of various additives: “The composition can also include one or more additives... [and many] such additives are well known in the art.” Ex. 1005 at 5:56–63.

261. Indeed, the use of biocides in matrix acidizing treatment solutions was well known in the art prior to the effective date of the ’282 Patent, 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.

262. For instance, Ayers provides that the composition disclosed therein “can be added to a separate **treatment fluid** including acids, polymers, proppants, solvents, surfactants, **biocides**, enzymes, hydroxides, and/or other treatment or fracturing fluids.” Ex. 1004 at 24:36–39 (emphasis added).

263. Moreover, due to the absence of evidence of the criticality of the use of biocide within the amounts as claimed, as well as the small amounts thereof that may be included in order to fall within the range as claimed (1 and 10,000 parts per million), it would have been obvious to a POSITA before the effective filing date of the ’282 Patent to include an amount of biocide as desired and when need to prevent bacterial growth within the well and/or formation as based on the actual

conditions encountered in the formation and desired treatments to provide thereto.

d. “between 1 and 10,000 parts per million (ppm) of a colloidal silica deposition inhibitor;”

264. Frenier discloses a treatment mixture including the use of various additives: “The composition can also include one or more additives... [and many] such additives are well known in the art.” Ex. 1005 at 5:56–63.

265. Indeed, the use of colloidal silica deposition inhibitors in matrix acidizing treatment solutions was well known in the art prior to the effective date of the '282 Patent, and a POSITA would have known, or at least it would have been obvious, to include such additives into the treatment mixture to control colloidal silica deposition.

266. For instance, Ayers explains that “[t]he formation of immobile emulsions, gels or polymers, the incomplete recovery of stimulation or fracking liquids, and the formation of larger particle blockages that may include precipitates, flocculants and/or **colloids** with clays, minerals, paraffins, asphaltenes, and condensates also can lead to clogging and reduction of oil and gas recovery from subterranean formations.” Ex. 1004 at 5:41–47 (emphasis added).

267. Moreover, due to the absence of evidence of the criticality of the use of colloidal silica deposition inhibitors within the amounts as claimed, as well as the small amounts thereof that may be included in order to fall within the range as

claimed (1 and 10,000 parts per million), it would have been obvious to a POSITA before the effective filing date of the '282 Patent to include an amount of colloidal silica deposition inhibitor as desired and when need to prevent colloidal silica buildup within the well and/or formation as based on the actual conditions encountered in the formation and desired treatments to provide thereto.

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

268. As discussed above, Frenier discloses a treatment mixture including various additives, including a mutual solvent. Frenier provides examples of treatment mixtures containing additives, such as mutual solvents, at a concentration between 0.1–10% and 0.1–5% by weight. *See* Ex. 1005 at 6:25–45.

269. A POSITA at the time of filing would understand that a treatment mixture containing additives, including a mutual solvent, at a concentration between 0.1–10% and 0.1–5% by weight discloses a treatment mixture containing between 1 and 500 gallons of mutual solvent per thousand gallons of water.

270. Even if Frenier doesn't explicitly mention said range, disclosing a broader range that includes it suggests that such concentrations are encompassed by the invention.

11. Dependent Claim 11

a. “The method of claim 1, wherein the metal-polymer complex remediation mixture further comprises a pH modifier.”

271. Ayers discloses that “[t]he pH of the composition can be adjusted if need to avoid pH changes in the formation.” *Id.* at 20:53–54. It would have been obvious to a POSITA at the time of filing that one or more acids may be used as a pH modifier.

272. Further, Frenier discloses a treatment mixture having additives, including a pH modifier: “[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). 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, 49 are directed to treatment mixtures that include pH modifiers. *Id.* at 13:3–19; 13:25–27; 14:44–45.

273. Accordingly, Ayers, alone or in combination with Frenier, disclose wherein the metal polymer complex remediation mixture further comprises a pH modifier.

12. Dependent Claim 12

- a. **“The method of claim 11, wherein the metal-polymer complex remediation mixture further comprises between 0.1 and 30% of one or more acids as a pH modifier.”**

274. Ayers discloses that “[t]he pH of the composition can be adjusted if need to avoid pH changes in the formation.” Ex. 1004 at 20:53–54.

275. Ayers further explains that “[i]n some embodiments, an organic acid, mineral acid, or a base is added to the composition to adjust the pH. The acid may be, for example, hydrochloric, hydrofluoric, formic, citric or acetic acid.” *Id.* at 35:66–36:4 (emphasis added).

276. It would have been obvious to a POSITA at the time of filing to include an acid pH modifier in an amount encompassing the range claimed.

277. Frenier further discloses a treatment mixture including, *inter alia*, 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. 1005 at 5:56–63 (emphasis added). Frenier explains that pH of the treatment fluid “can be controlled by can **organic acid, a mineral acid**, or a base.” *Id.* at 4:57–60 (emphasis added).

278. Accordingly, Frenier discloses the use of one or more acids as a pH modifier. Frenier provides examples of treatment mixtures comprising 0.1% to 15% by weight pH modifier. *Id.* at 6:40–45, and composition additives, such as solvents, comprising between 0.1–10% and 0.1–5% by weight. *See id.* at 6:25–45.

279. Accordingly, Ayers alone or in combination with Frenier, disclose wherein the metal-polymer complex remediation mixture further comprises between 0.1 and 30% of one or more acids as a pH modifier.

13. Dependent Claim 13

- a. “The method of claim 12, wherein the acid is selected from the group consisting of hydrochloric acid, aqua regia, arsenic acid, boric acid, carbonic acid, chloric acid, chromic acid, fluoroantimonic acid, fluoroboric acid, fluorosulfuric acid, fulminic acid, hexafluorophosphoric acid, hexafluorosilicic acid, hydrobromic acid, hydrofluoric acid, hydrogen iodide, hypochlorous acid, hypofluorous acid, hypophosphoric acid, iodic acid, nitric acid, nitrosyl-O-hydroxide, nitrous acid, orthocarbonic acid, perchloric acid, permanganic acid, perrhenic acid, pertechnetic acid, phosphoric acid, silicic acid, sulfuric acid, thiocyanic acid, titanic acid, tungstic acid, xenic acid, and mixtures thereof.”

280. Ayers discloses that “[i]n some embodiments, an organic acid, mineral acid, or a base is added to the composition to adjust the pH. The acid may be, for example, **hydrochloric, hydrofluoric**, formic, citric or acetic acid.” Ex. 1004 at 35:66–36:4 (emphasis added).

281. Frenier further discloses a treatment mixture including, *inter alia*, 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. 1005 at 5:56–63. Frenier explains that pH of the treatment fluid “can be controlled by can organic acid, a mineral acid, or a base.” *Id.* at 4:57–60. For instance, Frenier identifies “a non-oxidizing mineral acid, such as **hydrochloric acid, hydrofluoric acid**, or a mixture thereof.” *Id.* at

4:18–20 (emphasis added).

282. Accordingly, Ayers alone or in combination with Frenier, disclose wherein the acid is selected from a group including hydrochloric acid and hydrofluoric acid.

14. Dependent Claim 14

a. **“The method of claim 12, wherein the acid is hydrochloric acid.”**

283. As discussed above, Ayers discloses wherein the acid is hydrochloric acid. *See id.* at 35:66–36:4. Similarly, Frenier discloses the same.

15. Dependent Claim 15

a. **“The method of claim 1, wherein the injecting operation further comprises: alternately injecting a first amount of metal-polymer complex remediation mixture and a second amount of a diverting mixture into the well.”**

284. Ayers discloses that “[p]olymers such as xanthan gum, guar gum, and hydrolyzed polyacrylamide can be added to Component A or optionally can be injected in a polymer drive **following** the addition of the combined nonionic surfactant/cosolvent/hydrogen peroxide/sodium chelate/antioxidant components of a composition according to an embodiment of the present invention.” *Id.* at 30:42–48 (emphasis added).

285. A POSITA, prior to the effective date of the ’282 Patent, would find the use of polymers like xanthan gum, guar gum, and hydrolyzed polyacrylamide

as diverting materials to be obvious.

286. These polymers are well-established and documented materials used in the relevant industry for diversion purposes.

287. Their function of creating temporary downhole plugs or increasing fluid viscosity to influence flow paths is common knowledge within the field.

288. Moreover, a POSITA would recognize that the brief list of xanthan gum, guar gum, and hydrolyzed polyacrylamide are just a few examples within a broader category of known diverting materials.

289. Further, Frenier discloses 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. 1005 at 7:27–29.

290. 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.”

291. 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.” (“Zerhboub”). *See id.* at 7:46–48; Ex. 1012.

292. Zerhboub discloses alternately injecting a first amount of treatment mixture and a second amount of a diverting mixture into the well. Specifically,

Zerhboub discloses:

a treatment fluid injected into a subterranean formation is **diverted from a zone of relatively high permeability to a zone of relatively lower permeability** by a process of **first injecting a foam** comprising an inert fluid, a gas and at least one surfactant and **subsequently injecting a treatment fluid** comprising an active fluid and at least one surfactant used in the preceding foam injection step. This sequential treatment is repeated as necessary utilizing the increasing volumes of foam in each subsequent foam injecting step.

Ex. 1012 at 1:52–59 (emphasis added). Zerhboub explains that the injected foam is a diverting mixture. *Id.* at 4:1–5; 4:36–37 (“The principle of diversion by means of a foam is also known.”).

293. Accordingly, Ayers, either alone or in combination with Frenier, discloses the limitation, wherein the injecting operation further comprises: alternately injecting a first amount of metal-polymer complex remediation mixture and a second amount of a diverting mixture into the well.

16. Dependent Claim 16

- a. **“The method of claim 1, wherein the injecting operation further comprises: injecting the metal-polymer complex remediation mixture until pressure within the well reaches a predetermined target that does not cause substantial fracturing of the formation.”**

294. Ayers discloses that “[t]he composition may be introduced into the wellbore or subterranean formation by, for example, pumping, displacing, or

locating the composition at a desired location. In some embodiments, the pumping, displacing, or locating the composition is at a rate and **pressure less than the reservoir hydraulic fracture pressure.**” Ex. 1004 at 36:65–37:3 (emphasis added).

295. Further, in Example 6 of Ayers, where the disclosed composition was used to “remediate biological, clay and other blockages”—and not for the purpose of fracturing—the composition was introduced into the well bore and “[a]ll injection wells were shut in around 750–790 psi surface pressure.” *Id.* at 47:9–10; 47:43–45. In that example, the mixture was introduced into the wellbores until a predetermined target pressure was achieved, which was adequate for remediating blockages in the formation without causing substantial fracturing. While the injection of the treatment mixture in that example ceased upon reaching a predetermined surface pressure, a POSITA would recognize that surface pressure can provide information indicative of well pressure.

296. Moreover, in Example 6, it is disclosed that along with surface pressure, downhole reservoir pressure was also being monitored, noting that surface pressure eventually adjusted to be “approximately equal to downhole reservoir pressures.” *See* Ex. 1004 at 47:54–62.

297. Similarly, Frenier discloses that the injection step of the treatment mixture disclosed therein may be “performed at a fluid pressure that is **less than**

the minimum in situ rock stress (i.e., a matrix acidizing method).” Ex. 1005 at 4:49–52.

298. A POSITA, prior to the effective date of the ’282 Patent, would have found it obvious to inject a remediation mixture into a well until the pressure within the well reaches a predetermined target pressure that voids substantial fracturing of the formation.

299. A POSITA would understand the detrimental effects of exceeding the formation’s fracture pressure, including the damage it could do to the wellbore, creation of unwanted flow paths, and its negative impact on treatment effectiveness. Monitoring well pressure during treatment is a well-established practice in the relevant industry, and a POSITA would be aware of the importance of such monitoring, especially to prevent the detrimental effects mentioned earlier.

300. A POSITA would also recognize the established correlation between increasing wellbore pressure and the risk of fracturing the formation and, as such, it would have been obvious to a POSITA to select a target pressure that would not cause substantial fracturing of the formation.

301. Accordingly, Ayers, either alone or in combination with Frenier, discloses the limitation, wherein the injecting operation further comprises: injecting the metal-polymer complex remediation mixture until pressure within the well reaches a predetermined target that does not cause substantial fracturing of

the formation.

17. Dependent Claim 17

- a. **“The method of claim 1, wherein the injecting operation further comprises: injecting the metal-polymer complex remediation mixture until the pressure within the well reaches a predetermined target pressure calculated based on the fracture pressure of the subterranean formation; and upon reaching the target pressure, shutting in the well.”**

302. As discussed above, Ayers discloses that “[t]he composition may be introduced into the wellbore or subterranean formation by, for example, pumping, displacing, or locating the composition at a desired location. In some embodiments, the pumping, displacing, or locating the composition is at a rate and **pressure less than the reservoir hydraulic fracture pressure.**” Ex. 1004 at 36:65–37:3 (emphasis added).

303. Ayers also provides examples that disclose this limitation. For instance, Example 3 of Ayers discloses the use of a metal-polymer complex remediation mixture for the purpose of “penetrating shale layer in a hydrocarbon bearing subterranean formation...to fragment shale layer causing secondary porosity and increased permeability.” *Id.* at 44:62–65.

304. In that example, the treatment mixture was allowed to gravity flow into the wellbore before a water chase was pumped behind the mixture to displace the mixture into the targeted subterranean formation. *See id.* at 45:6–11. Ayers

explains that “[t]he water chase was pumped at a rate of 2 to 8 gpm at a pressure of 6 psi to 100 psi; the rates and **pressures remained well below reservoir fracturing pressures.**” *Id.* at 45:11–14 (emphasis added).

305. In another instance, as detailed in Example 6, a metal-polymer complex remediation mixture was injected into wells and into the subterranean formation for the purpose of “remediat[ing] biological, clay and other blockages,” *Id.* at 47:9–12. The injection process involved pumping the mixture into the wellbore and displacing the mixture into subterranean formation using a water chase, after which “[a]ll injection wells **were shut in at around 750–790 psi surface pressure.**” *Id.* at 47:43–45 (emphasis added).

306. Accordingly, Ayers discloses injecting a remediation mixture until the pressure within the well reaches a predetermined target pressure calculated based on the fracture pressure of the subterranean formation, and upon reaching the target pressure, shutting in the well.

307. Also, as discussed above, Frenier discloses that the injection step of the treatment mixture disclosed therein may be “performed at a fluid pressure that is **less than the minimum in situ rock stress** (i.e., a matrix acidizing method).” Ex. 1005 at 4:49–52. As such, Frenier discloses reaching a predetermined a target pressure calculated based on the fracture pressure of the formation, the calculation taking into account, for example, in situ rock stress, a parameter closely related to

formation fracture pressure.

308. Furthermore, as discussed previously in the arguments pertaining to Dependent Claim 15, Zerhoub, which Frenier incorporated by reference in its entirety, provides that the process disclosed therein “includes a shut-in step between each sequential foam injecting and treatment fluid injecting step.” Ex. 1012 at 1:63–65.

309. A POSITA, prior to the effective date of the ’282 Patent, would have found it obvious to inject a remediation mixture into a well until the pressure reaches a predetermined target pressure calculated based on the fracture pressure of the formation, and then shut-in the well, in view of Ayers and Frenier.

310. As mentioned previously, pressure monitoring is a standard practice during formation treatments, and a POSITA understands the importance of fracture prevention due to its detrimental effects.

311. A POSITA would recognize that the fracture pressure of a formation is a critical parameter to consider when injecting fluids downhole.

312. A POSITA with access to common wellbore data and knowledge of industry practices would find it obvious to calculate a target pressure based on the formation’s fracture pressure, and to shut-in the well after reaching the calculated target pressure.

18. Dependent Claim 18

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

313. Ayers discloses this limitation. In Example 6, Ayers notes that after the injection step, monitoring of, at least, well pressure occurred during the maintaining operation. Specifically, in that Example, Ayers provides that:

The morning following injector well treatments (12 to 24 hrs after injector well treatments, then shut in), surface pressures at the injector wells were 350 for injector #1, 380 for #2 and 300 for #3. **These pressures were approximately equal to downhole reservoir pressures,** and indicated that oxygen and carbon dioxide gas generated in-situ from the volumes of composition placed into the subterranean formation through the injector treatments had dispersed out into the formation toward production wells.

Id. at 47:54–62 (emphasis added). As such, Ayers discloses monitoring well pressure during the maintaining operation.

314. Further, Frenier 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. 1005 at 6:49–54.

315. A POSITA, prior to the effective date of the '282 Patent, would have known that pressure transient analysis includes monitoring well pressure over time

during the maintaining operation.

316. Monitoring both pH and well pressure during treatment and wellbore contact with the mixture is standard practice in the relevant industry. Monitoring pH allows for adjustments to the treatment mixture's composition if necessary, ensuring optimal performance in the formation.

317. Similarly, monitoring well pressure helps identify potential pressure buildups that could lead to unwanted fracturing. Finally, it is understood in the industry that monitoring both parameters allows for early detection of potential issues that could compromise wellbore integrity and worker safety.

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

318. Ayers discloses this limitation. More particularly, Ayers discloses, in Example 3, that:

The pressure in the test wellbore was observed to begin rising steadily after 2 to 3 hours following treatment injection, and continued to rise over the next 4 hours until blowing the surface packer from the wellbore. Gas was continually observed bubbling and escaping from the open wellbore over the next 30 hours, at which time poly tubing was lowered to the bottom of the wellbore and clean water was pumped into the wellbore at sufficient rate to flush solids from the wellbore and onto the ground surface surrounding the test wellbore.

Id. at 45:23–33. Accordingly, Ayers discloses initiating the removing operation based on the results of the monitoring operation.

319. Moreover, a POSITA, before the effective date of the '282 Patent, would have found it obvious to initiate the removing operation based on the results of a monitoring operation. In the relevant industry, monitoring pressure and pH during and after treatment is a well-established practice.

320. A POSITA would understand that fluctuations in pressure or pH observed during monitoring operations could indicate formational changes, prompting the POSITA to consider initiating removal procedures.

19. Dependent Claim 19

- a. **“The method of claim 1, wherein the contact time is between about 4 hours and about 4 days.”**

321. Ayers discloses this limitation. Ayers discloses that:

In some embodiments, the composition is allowed to remain in the wellbore or subterranean formation for a period of time before hydrocarbon extraction resumes. **The time may be more than, for example, more than 4 hours, 6 hours, 8 hours, 10 hours, 12, hours, 16 hours, 20 hours, 24 hours, 30 hours, 36 hours, 42 hours, 48 hours, 3 days, 4 days, 5 days, 1 week. The time may be, for example, less than 30 days, 4 weeks, 3 weeks, 20 days, 2 weeks, 10 days, 1 week, 5 days, 4 days, 3 days.**

Ex. 1004 at 38:59–67.

322. Ayers makes clear that the disclosed composition can be “[a]llowed to contact blockage or damage in a portion of the wellbore or a portion of a subterranean formation for **a sufficient time** at the ambient temperature of the well

bore or the subterranean formation to sufficiently alter, remove, **degrade, and/or dissolve the permeability damage or blockage...**” *Id.* at 23:30–36 (emphasis added).

323. Accordingly, any contact time between the composition in Ayers, which includes a chelating agent (i.e., a metal complexing agent), and a subterranean-formed metal-polymer complex (i.e., the permeability blockage or damage), for a time sufficient to degrade and/or dissolve the damage and or blockage would seem to meet the limitation of at least “about” 4 hours of contact time.

324. Furthermore, given the vastness of time within the range, a POSITA would recognize the optimal contact time by experimentation and trial and error since it has been held, “[w]here the general conditions of a claim are disclosed in the prior art, it is not inventive to discover the optimum or workable ranges by routine experimentation.” *In re Aller*, 220 F.2d 454, 456, 105 USPQ 233, 235 (CCPA 1955).

325. Similarly, 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. 1005 at 7:30–34.

326. 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).

327. Here, as with Ayers above, a POSITA would recognize the optimal contact time by experimentation and trial and error since it has been held, “[w]here the general conditions of a claim are disclosed in the prior art, it is not inventive to discover the optimum or workable ranges by routine experimentation.” *In re Aller*, 220 F.2d at 456.

328. As such, Ayers, either alone or in combination with Frenier, discloses the limitation wherein the contact time is between four (4) hours and about four (4) days.

20. Dependent Claim 20

a. “The method of claim 19, wherein the contact time is between 1 to 4 days.”

329. I incorporate herein my statements and conclusions above concerning Dependent Claim 19. For the reasons discussed above, Ayers, either alone or in combination with Frenier, discloses the limitation wherein the contact time is between one (1) day to four (4) days.

21. Independent Claim 21

- a. **“Element 21[Preamble]: “A method for remediating a subterranean-formed metal-polymer complex in an existing, low-producing well in a hydrocarbon-bearing subterranean shale formation, comprising:”**

330. To the extent the preamble is limiting, Ayers discloses this limitation. Ayers discloses “a method of using a fluid formulation for increasing flow, production, or recovery of oil and gas hydrocarbons from a subterranean formation.” Ex. 1004 at Abstract.

331. The method in Ayers aims to alleviate reductions in the productivity of pre-existing oil and gas wells in subterranean formations. Ayers identifies the following, and other similar factors, as potential causes of reduced flow, production, or recovery of oil and gas hydrocarbons from a subterranean formation: “[t]he formation of immobile emulsions, gels or polymers, the incomplete recovery of stimulation or fracking liquids, and the formation of larger particle blockages that may include precipitates, flocculants and/or colloids with clays, minerals, paraffins, asphaltenes, and condensates also can lead to clogging and reduction of oil and gas recovery from subterranean formations.” *Id.* at 5:41–47.

332. Ayers further identifies that “damage or blockage causing polymers can include polysaccharide polymers, cross-linked polymers, mannosidic and/or

galactomannosidic linkages of guar, cellulose, cellulose derived, guar, guar derived, starches, xanthans, and/or other viscosity increasing polymers.” *Id.* at 24:1–6.

b. Element 21[a] “observing a well production decrease;”

333. Ayers discloses, with regards to Example 6, that “[p]rior to this **treatment** example, water injection/extraction wells were continuously operating, with a **reported** increase in injection well head pressure and decreased volumetric injection flowrates, with a **decreasing and low yield of oil production in the extraction wells over time.**” Ex. 1004 at 47:17–22 (emphasis added). More particularly, with regards to Example 6, “[i]t was **reported by the well operators** that there had been a **decrease in volumetric injection flowrates, with a decreasing and low yield of oil** at less than 1.3 BOPD.” *Id.* at 47:25–28 (emphasis added).

334. As such, Ayers discloses the limitation of observing a well production decrease, as claimed in Element 21[a].

c. Element 21[b] “providing a metal-polymer complex remediation mixture containing between 1% and 95% by weight metal complexing agent;”

335. Ayers discloses a “fluid formulation,” i.e., a metal-polymer complex remediation mixture, including “a peroxygen, a surfactant, **a alkali metal chelate,**

and a cosolvent.” *Id.* at 1:51–53 (emphasis added). Ayers explains that “an alkaline chelate can be added to the formation to **chelate metals associated with the formation matrix or pore liquids.**” *Id.* at 29:61–62 (emphasis added).

336. According to the ’282 Patent, “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.” Ex. 1001 at 5:16–21 (emphasis added). Here, the alkali metal chelate component of the fluid formulation in Ayers discloses a metal complexing agent.

337. With regards to the chelate component, Ayers provides:

Compositions and methods of embodiments according to the present invention include or require the use of a chelate, such as a mono-, di-, tri-, or tetra-sodium ethylenediaminetetraacetic acid (EDTA), a mono-, di-, tri-, or tetra-potassium ethylenediaminetetraacetic acid (EDTA) or sodium ethylenediamine-N,N-disuccinic acid (EDDS), or combinations. The selected chelate can be present in a final concentration as applied to the wellbore or subterranean formation that varies from 0.0001 to 5.0 percent by weight. For example, the chelate can be sodium EDTA.

Id. at 21:27–36.

338. Ayers provides that the chelate component of its fluid formulation may be, *inter alia*, salts of EDTA. The specification of ’282 Patent includes EDTA as a “suitable complexing agent” and “any salt thereof, any derivative thereof, and

the like.” *See* Ex. 1001 at 5:60–6:9. Additionally the ’282 Patent also identifies EDDS and any salt or derivative thereof, as a suitable complexing agent. *See id.* at 6:10–38.

339. Ayers further discloses that “[t]he chelate concentration in the composition when introduced into the wellbore or subterranean formation may be between about 0.00001 to 5.0 percent by weight.” Ex. 1004 at 36:20–22.

340. Moreover, Frenier discloses a “well treatment fluid,” i.e., a treatment mixture, comprising: “a first acid; water; and a hydroxyethylaminocarboxylic acid.” Ex. 1005 at 4:12–13. 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.

341. 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). “Preferably, the hydroxyethylaminocarboxylic acid is selected from hydroxyethylethylenediaminetriacetic acid (HEDTA), hydroxyethyliminodiacetic acid (HEIDA), or a mixture thereof.” *Id.* at 4:23–27. 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 an organic acid such as HCL, HF, citric, formic, acetic acid.

342. 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 Independent Claim 21[b]. *Id.* at 6:25–47.

343. 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. 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.

344. Accordingly, Ayers, either alone or in combination with Frenier, discloses a remediation mixture containing a metal complexing agent at a concentration between 1% and 95% by weight.

345. Moreover, a POSITA, before the effective date of the ’282 Patent, would have recognized that the specific proportion of the metal complexing agent within the claimed remediation mixture is likely to vary depending on several factors, including the unique characteristics of the subterranean formation being treated. *See* Ex. 1005 at 6:16–19 (“The 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.”); *see also* Ex. 1004 at 19:27–33 (“Factors affecting concentrations of the components of the composition to be applied include the magnitude or extent of the deposits or blockages to be removed, and the composition of the deposits...or blockages.”).

346. The broad ranges disclosed in Ayers and Frenier provide ample room for a POSITA to necessary make adjustments. Given the vastness of the concentration range and the well-understood functionalities of metal complexing agents, a POSITA could readily arrive at successful formulations through routine experimentation.

347. The established functions of metal complexing agents provide a predictable framework for optimizing the composition for a particular application. A POSITA, familiar with these functionalities and the specific needs of a treatment project, could readily adjust the metal complexing agent ratios within the claimed broad ranges to achieve the desired outcome.

348. This level of optimization would not require inventive leaps but rather routine experimentation based on established scientific principles. Overall, the broadness of the claimed ranges and the predictability associated with optimizing the composition based on well-known functionalities render the

invention obvious to a POSITA.

d. Element 21[c] “injecting the metal-polymer complex remediation mixture into the well at a pressure less than a fracture pressure of the subterranean shale formation until at least some of the metal-polymer complex remediation mixture contacts the subterranean-formed metal-polymer complex, wherein the subterranean-formed metal polymer complex forms from a previously injected fracturing fluid and the metal of the subterranean-formed metal-polymer complex includes metal naturally present within the subterranean formation;”

349. Ayers discloses injecting a metal-polymer complex remediation mixture into the well at a pressure less than the fracture pressure of the subterranean formation. Specifically, Ayers discloses that “[t]he composition may be introduced into the wellbore or subterranean formation by, for example, pumping, displacing, or locating the composition at a desired location. In some embodiments, the pumping, displacing, or locating the composition is **at a rate and pressure less than the reservoir hydraulic fracture pressure.**” Ex. 1004 at 36:65–37:3 (emphasis added).

350. Moreover, Ayers discloses that components of the fluid formulation may be “**injected** separately or in subcombinations” or “**injected** together as a single composition.” *See id.* at 42:43–49 (emphasis added).

351. Accordingly, Ayers discloses injecting the metal-polymer complex

remediation mixture into the well at a pressure less than a fracture pressure of the subterranean formation, as claimed in Element 1[b] of the '282 Patent.

352. Ayers also discloses that:

The introduced **composition may be brought into contact with, for example, blockage or damage in the wellbore or subterranean formation**. The contact may alter, remove, degrade, or dissolve the blockage or damage. The damage or blockage may include, for example, clay, biological material, inorganic fines, organic fines, precipitates, anhydrite, insoluble materials, asphaltenes, paraffins, or combinations thereof. In some embodiments, at least some of the blockage or damage materials are mobilized into the wellbore. In some embodiments, at least some of the blocking and/or damage materials are recovered from the wellbore.

Id. at 38:29–39 (emphasis added). Ayers explains that:

Inevitably, hydraulic fracturing also typically results in some level of formation damage and blockage as the **fracturing fluids intrude the natural formation and have negative interactions** in the way of swelling certain clays, migrating fines, introducing microbial growth and biofouling, **forming immobile emulsions or gels**, and forming larger particle blockages that may include precipitates, flocculants and/or colloids with clays, minerals, paraffins, asphaltenes, and/or condensates.

Id. at 8:27–35.

353. Ayers discloses that subterraneous formations of metal-polymer complexes may result due to previous injections of fracturing fluid. For example, fracturing fluid containing polyacrylamide, a common polymer additive to fracturing fluid, may intrude the natural formation and have negative interactions

in the way of forming a metal-polymer complex with metal ions naturally present in the formation.

354. Ayers discloses the presence of transition metals in the formation. *See id.* at 28:20–21 (“[t]he presence of inorganic transition metals, either naturally present in pore liquids (oil or water) or the formation”); *Id.* at 29:59–60 (“solid phase transition metals associated with the formation matrix”).

355. Moreover, Ayers recognizes that, “[o]ver time **stranded polymer** in the well bore, filter cake, and **in adjacent subterranean formations can form matrices that block oil flow** to and in a well including polymers, oil, asphaltenes, paraffins, clays, inorganic precipitates, and biological cells and biofilms.” *Id.* at 13:28–32 (emphasis added).

356. Further, Ayers discloses that, in some embodiments, “the damage or blockage may include viscosity increasing polymers. **The polymer may have been introduced as a separate treatment fluid.** The polymer may include, for example, polysaccharide polymer, **cross-linked polymers**, mannosidic and galacomannosidic linkages of guar, cellulose, cellulose derived guar, starch es, xanthans, other viscosity increasing polymer, or combinations thereof.” *Id.* at 38:46–53 (emphasis added).

- e. **Element 21[d] “maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex for a contact time of**

between about 1 minute and about 100 days, thereby allowing the metal complexing agent to cause the subterranean-formed metal-polymer complex to dissociate and dissolve, thereby creating a low viscosity flow back fluid comprising the spent metal-polymer complex remediation mixture and the metal-polymer complex components; and”

357. Ayers discloses maintaining the metal-polymer complex remediation mixture in contact with the subterranean-formed metal-polymer complex. *Id.* at 22:20–24 (“As part of the methods for application the **composition can be allowed to contact** the wellbore or a portion of a well bore or **subterranean formation or hydrocarbon-bearing subterranean formation** for a sufficient period of time to increase flow, production, and/or recovery of hydrocarbons.”). Ayers, as established above, discloses the existence of subterranean-formed metal-polymer complexes occurring within the subterranean formation. Accordingly, those in-situ formed metal-polymer complexes would necessarily be encountered by the composition disclosed in Ayers. *Id.*

358. Further, Ayers discloses contacting the metal-polymer complex remediation mixture with the subterranean-formed metal-polymer complex for a contact time between about 1 minute and 100 days.

359. For instance, Ayers discloses the following regarding the contact time for the disclosed composition:

Such a sufficient or adequate time can be, for example, from about 1 minute, 2 minutes, 5 minutes, 15 minutes, 30 minutes, 1 hour, 2 hours, 6 hours, 12 hours, 1 day, 2 days, 4 days, 1 week, 2 weeks, 1 month, 2 months, 3 months, or 6 months to about 2 minutes, 5 minutes, 15 minutes, 30 minutes, 1 hour, 2 hours, 6 hours, 12 hours, 1 day, 2 days, 4 days, 1 week, 2 weeks, 1 month, 2 months, 3 months, 6 months, or 12 months.

Id. at 22:32–39.

360. Ayers makes clear that the disclosed composition can be “[a]llowed to contact blockage or damage in a portion of the wellbore or a portion of a subterranean formation for **a sufficient time** at the ambient temperature of the well bore or the subterranean formation to sufficiently alter, remove, **degrade, and/or dissolve the permeability damage or blockage...**” *Id.* at 23:30–36 (emphasis added).

361. Accordingly, any contact time between the composition in Ayers, which includes a chelating agent (i.e., a metal complexing agent), and a subterranean-formed metal-polymer complex (i.e., the permeability blockage or damage), for a time sufficient to degrade and/or dissolve the damage and or blockage would seem to meet the limitation of at least “about” 1 minute of contact time.

362. Furthermore, given the vastness of time within the range, a POSITA would recognize the optimal contact time by experimentation and trial and error since it has been held, “[w]here the general conditions of a claim are disclosed in

the prior art, it is not inventive to discover the optimum or workable ranges by routine experimentation.” *In re Aller*, 220 F.2d 454, 456, 105 USPQ 233, 235 (CCPA 1955).

363. Additionally, with regards to contact time, Frenier discloses 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. 1005 at 7:30–34. 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.” *Id.* at 7:39–46.

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

365. Furthermore, as previously discussed, Ayers explains that the disclosed composition induces the dissociation and dissolution of subterranean-formed metal-polymer complexes. *See id.* at 23:30–36. Ayers discloses a composition including a chelating agent, i.e., a metal complexing agent.

366. A POSITA at the time of filing would recognize that such chelating

agents would prevent metal ion precipitation by forming complexes that keep the metals dissolve in solution. *See also* Ex. 1004 at 1:63–65 (“The alkali metal chelate can serve the purpose of scavenging ionic or **bound phases of metals in a formation**[.]”) (emphasis added).

367. Ayers further discloses that the dissociation and dissolution of the subterranean-formed metal-polymer complexes generate a low viscosity flowback fluid comprising the spent metal-polymer complex remediation mixture and metal-polymer complex components. Ayers explains that the disclosed composition will “liquefy” encountered inorganic and organic materials, the “liquified material” then displaced through the formation and into the well for subsequent removal. *See id.* at 28:25–35; *see also id.* at 42:36–38 (“In a method according to the invention, components or a composition that have been injected into a subterranean formation can be removed from the wellbore.”); Ex. 1004 at 43:30–32 (“Liquids pumped from the treated wells contained clays, oils, asphaltene materials and iron precipitates, along with the treatment chemicals.”).

368. Figure 3 of Ayers shows an example of materials evacuated from a well subsequent to treatment with the disclosed composition. Ayers notes that the evacuated materials depict “flowable characteristics that readily enable pumping as a liquid.” *Id.* at 43:40–41. A POSITA at the time of filing would understand this to mean that to disclose a low viscosity flow back fluid.

f. Element 21[e] “removing the low viscosity flow back fluid from the well after the contact time, thereby improving the hydrocarbon production of the well relative to the hydrocarbon production immediately prior to performance of the method.”

369. Ayers discloses that “the composition is removed from the wellbore after introduction. **The composition may be removed from the wellbore**, for example, by pumping or flushing.” *Id.* at 37:41–44 (emphasis added).

370. Ayers further explains that “after the composition is introduced into the wellbore or subterranean formation, the wellbore or **subterranean formation is contacted** for a period of time sufficient **to increase flow, production, or recovery of hydrocarbons.**” *Id.* at 36:60–64 (emphasis added); *see also id.* at 22:20–24.

371. In Example 6 of Ayers, it was noted that, following treatment, “[o]nce injection and extraction wells were brought back online for normal operations, the existence of gas and **increased oil production** flow rates were observed at 5 of the 8 extraction wells.” *Id.* at 48:8–11 (emphasis added).

372. Similarly, in Example 1 of Ayers, it was noted that “[w]ithin 24 hours following the treatment of the well bore and perforations to remove blockages and restore flow to the well, oil can be seen entering the casing perforations in the oil bearing zone from the still photographic image obtained from the down hole video in FIG. 4.” *Id.* at 43:42–46. Example 1 of Ayers also states that following

treatment, the wells experienced “more than a **600% increase in flow and oil production** continuing 30 days after treatment.” *Id.* at 44: 27–29 (emphasis added).

373. The additional words “relative to the hydrocarbon production immediately prior to performance of the method” are superfluous given that “improvements” are always measured to prior performance.

374. Accordingly, Ayers discloses removing the low viscosity flow back fluid after the contact time, thereby improving hydrocarbon production of the well immediately prior to performance of the method. *See also id.* at 42:36–38.

22. Dependent Claim 22

- a. “The method of claim 21, wherein the injecting operation further comprises: alternately injecting a first amount of metal-polymer complex remediation mixture and a second amount of a diverting mixture into the well.”

375. Ayers discloses this limitation. Ayers discloses that “[p]olymers such as xanthan gum, guar gum, and hydrolyzed polyacrylamide can be added to Component A or optionally can be injected in a polymer drive **following** the addition of the combined nonionic surfactant/cosolvent/hydrogen peroxide/sodium chelate/antioxidant components of a composition according to an embodiment of the present invention.” Ex. 1004 at 30:42–48 (emphasis added).

376. A POSITA, prior to the effective date of the '282 Patent, would find

the use of polymers like xanthan gum, guar gum, and hydrolyzed polyacrylamide as diverting materials to be obvious.

377. These polymers are well-established and documented materials used in the relevant industry for diversion purposes.

378. Their function of creating temporary downhole plugs or increasing fluid viscosity to influence flow paths is common knowledge within the field.

379. Moreover, a POSITA would recognize that the brief list of xanthan gum, guar gum, and hydrolyzed polyacrylamide are just a few examples within a broader category of known diverting materials.

380. Further, Frenier discloses 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. 1005 at 7:27–29.

381. 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.”

382. 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.” (“Zerhboub”). *See id.* at 7:46–48; Ex. 1012. Zerhboub discloses alternately injecting a first amount of treatment mixture and a second amount of a diverting mixture into the well. Specifically, Zerhboub

discloses:

a treatment fluid injected into a subterranean formation is **diverted from a zone of relatively high permeability to a zone of relatively lower permeability** by a process of **first injecting a foam** comprising an inert fluid, a gas and at least one surfactant and **subsequently injecting a treatment fluid** comprising an active fluid and at least one surfactant used in the preceding foam injection step. This sequential treatment is repeated as necessary utilizing the increasing volumes of foam in each subsequent foam injecting step.

Ex. 1012 at 1:52–59 (emphasis added). Zerhboub explains that the injected foam is a diverting mixture. *Id.* at 4:1–5; 4:36–37 (“The principle of diversion by means of a foam is also known.”).

383. Accordingly, Ayers, either alone or in combination with Frenier, discloses the limitation, wherein the injecting operation further comprises: alternately injecting a first amount of metal-polymer complex remediation mixture and a second amount of a diverting mixture into the well.

VIII. CONCLUSION

384. 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 18, 2024

By: 

Dr. Jeffrey H. Harwell

EXHIBIT A

JEFFREY H. HARWELL

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[Google Scholar Profile](#)

[Cited by:](#)

	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.

“Surfactant/Cosolvent Treatability Study for DNAPL at WAG 6, Paducah Gaseous Diffusion Plant,” Surbec Environmental, LLC, \$100,000. 6-10-98 to 12-30-98. Co-Principal Investigator: Robert Knox.

“Surfactant-Enhanced Subsurface Remediation of Petroleum Hydrocarbons,” Surbec Environmental, LLC, \$100,000, May 1, 1998 to April 30, 1999. Co-Principal Investigator: Robert Knox.

"Photocatalytic Complete Oxidation of Indoor Air Contaminants," Center for Indoor Air Research, \$483,000. January 1, 1995-February 28, 1998. Co-Principal Investigator: Lance Lobban.

“Oxidation of VOCs via an Ozonated, Ultraviolet Irradiated Fluorocarbon/TiO₂ Dispersion,” Surfactant Associates, Inc., \$14,521. July 1, 1997 to June 30, 1998. Co-Principal Investigator: Jerry Newman.

“Design and Build Micellar-Enhanced Ultrafiltration (MEUF) Units,” U.S. Department of Defense--Navy, \$163,167, June 23, 1997 to February 28, 1998. Co-Principal Investigators: Ed Tucker and J.F. Scamehorn.

“Improved Dispersability of TiO₂ Particles via Surface Modification Using Admicellar Polymerization,” Nikko Chemicals Co., LTD, \$29,803, July 1, 1997 to June 30, 1998. Co-Principal Investigator: E.A. O’Rear.

"Surfactant Selection Protocol for Optimizing Ex Situ Soil Washing," Environmental Protection Agency Northeast Hazardous Substance Research Center, \$120,000 August 1, 1996-December 31, 1997. Co-Principal Investigator: D.A. Sabatini

"Surfactant-Enhanced Subsurface Remediation: Surfactant Recovery Options," U.S. Environmental Protection Agency Environmental Technology Initiative Program, \$395,000, October 1, 1995-September 30, 1997. Co-Principal Investigator: D.A. Sabatini.

“Field Demonstration of Surfactant Enhanced Subsurface Remediation (SESR),” DOD/”ATDF/Rice University (R1155B-241000), \$398,705, October 1, 1995 to

October 31, 1997. Co-Principal Investigator: D.A. Sabatini.

"Design Manual for the Extraction of Contaminants from Unconsolidated Subsurface Environments," U.S. Environmental Protection Agency, \$1,200,000., October 1, 1994 to September 30, 1997. Co-Principal Investigators: R. C. Knox and D.A. Sabatini.

"Liquid Natural Gas as a Transportation Fuel in the Heavy Trucking Industry," U.S. Department of Energy, DE-FG22-94BC14971 and DE-FG36-95GO10112, \$1,146,000, May 10, 1994-May 30, 1997. Co-Principal Investigators: W.H. Sutton, S. Gollahalli, F.C. Lai, A.K. Agrawal, K.E. Starling, L.L. Lobban, R.G. Mallinson, C.M. Sliepcevich.

"Surfactant Enhanced Soil Remediation", Oklahoma Center for the Advancement of Science and Technology, \$234,353, August 1, 1994 to July 31, 1997. Co-Principal Investigators: D.A. Sabatini and R.C. Knox.

"Organization and Implementation of Graduate Engineering Programs In Chulalongkorn University's Petroleum and Petrochemical College as a Joint Academic Venture with the Participating U.S. Universities," U.S. Agency for International Development, University Development Linkages Project, \$600,000., February 1, 1993 to May 15, 1997. Project Director: J.F. Scamehorn (University of Oklahoma); Co-Directors: J.H. Harwell (University of Oklahoma), J.W. Schwank (University of Michigan), and K. Ishida (Case Western Reserve).

"Surfactant-Enhanced Subsurface Remediation of Petroleum Hydrocarbons," Trust Environmental Services, LLC., \$14,400, 1/1/97-6/30/97. Co-Principal Investigators: D.A. Sabatini and R.C. Knox.

"Surfactant Recovery by Micellar-Enhanced Ultrafiltration," \$45,397, Lockheed-Martin, September 27 to October 25, 1996. Co-Principal Investigator: Bruce Roberts.

"Filler and Reinforcer Compatibilization," Surfactant Associates, Inc., \$32,720, July 1, 1992 to December 31, 1996. Co-Principal Investigator: E.A. O'Rear.

"A Pilot-Scale Demonstration of Surfactant-Enhanced Subsurface Remediation," U.S. Environmental Protection Agency, \$99,993, August 1, 1994 to July 1, 1996. Co-Principal Investigators: D.A. Sabatini and R.C. Knox.

"Surfactant Selection Protocol for Optimizing Ex Situ Soil Washing," Environmental Protection Agency Northeast Hazardous Substance Research

Center, \$219,906, August 1, 1994-July 31, 1996. Co-Principal Investigator: D.A. Sabatini

"Engineering Research Equipment: Calvet-Type Calorimeter for Determining Heats of Adsorption and Heats of Reaction," National Science Foundation, \$33,167, April 15, 1995-March 31, 1996. Co-Principal Investigators: Daniel Resasco, Brian Grady, and Lance Lobban.

"DowFax Components for Treating Soil Contaminated with Chlorinated Solvents and Heavy Metals," Dow Chemical Company, \$33,737, December 1, 1995-November 30, 1996. Co-Principal Investigator: D.A. Sabatini.

"Soil Washing Efficiency Using Individual DOWFAX Components," Dow Chemical U.S.A., \$31,778., September 1, 1994 to August 31, 1995. Co-Principal Investigator: D.A. Sabatini.

"Development of Technologies for High Energy Density Storage of Natural Gas at Moderate Conditions for Use as a Vehicular Transportation Fuel," Link Foundation, \$18,000, July 1, 1994 to June 30, 1995.

"Surfactant Enhanced Remediation of DNAPL Contamination with Edible Surfactants," U.S. Environmental Protection Agency, \$378,696., October 16, 1991 to October 15, 1994. Co-Principal Investigators: D.A. Sabatini and R.C. Knox

"Mixing Rule Constants for the Optimization of DowFax Blends for Soil Washing, *In Situ* Aquifer Remediation, Oil Spill Clean-up, and Solvent-Free Cleaning Systems," Dow Chemical Co., \$31,950., January 1, 1994 to December 31, 1994. Co-Principal Investigator: D.A. Sabatini.

"Research on New Ultrathin Film Formation: Polymerized Fluorinated Thin Film," Hitachi Research Laboratory, Hitachi-shi, Japan, \$120,000, October 1, 1989 to September 30, 1993. Co-Principal Investigator: E.A. O'Rear.

"New Sorptive-Storage Technology for Natural Gas," Oklahoma Center for the Advancement of Science and Technology, \$168,000, August 1, 1990 to July 31, 1993. Co-Principal Investigator: K.E. Starling.

"Surface Modification with Polymeric Thin Films," Oklahoma Center for the Advancement of Science and Technology, \$292,000, August 1, 1990 to July 32, 1993. Co-Principal Investigators: E.A. O'Rear, B.M. Fung.

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RECENT PRESENTATIONS

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.

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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.
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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.