

Claim Elements:	United States Patent No. 9,410,405 B2 to Ayers et al.	U.S. Patent No. 6,436,880 B1 (Frenier)	Borchardt, <i>Chemicals Used in Oil Field Operations</i> , 1989
1. A method for remediating a subterranean-formed metal-polymer complex in a pre-existing well in a subterranean shale formation, the method comprising:	We claim: 1. A method for enhancing hydrocarbon recovery in a subterranean formation having a blockage or accumulation of material....	Frenier discloses "methods to enhance the productivity of hydrocarbon wells (e.g., oil wells) by...removing (by dissolution) near-wellbore formation damage." [a]n acid fluid that is useful in stimulation and workover operations, and in particular for the control of iron in acidizing operations..." Id. at Abstract	Chapter 1, Page 3 Chemicals of various types are used in every stage of drilling, completing, and producing oil and gas wells. This review describes these chemicals, why they are used, and recent developments. These chemicals include common inorganic salts, transition metal compounds, common organic chemicals and solvents, water-soluble and oil-soluble polymers, and surfactants. As existing fields become depleted, use of chemistry to maintain production via well stimulation, more efficient secondary recovery operations, and enhanced oil recovery become ever more important. Page 7 Formation damage control chemicals are added to reduce the permeability damage that occurs when drilling fluid enters the formation.
providing a metal-polymer complex remediation mixture comprising between 0.1% and 95% by weight metal complexing agent;	Claim 1. comprising: introducing components comprising a) a peroxygen, b) a surfactant, c) an alkali metal chelate, and d) a co solvent into a hydrocarbon bearing subterranean formation with a blockage or accumulation of material 3. The method of claim 1, wherein the peroxygen is introduced into the subterranean formation at a concentration between about 0.01 and about 20 percent.	Frenier discloses a "well treatment fluid," i.e., a remediation mixture, comprising: "a first acid; water; and a hydroxyethylaminocarboxylic acid." 4:12–13. Frenier further explains that "[e]xamples of first acids include non-oxidizing mineral acids...Other examples include non-oxidizing organic acids such as formic acid, acetic acid, and mixtures thereof." Id. at 5:27–31. 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. 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 falls squarely within the claimed range set forth in Element 1[a]. 6:25–47. Frenier further discloses treatment mixtures having between 15% and 28% and between 10% and 30% by weight metal complexing agent. See claims 1, 9, and 28 at id., 11:27–34; 11:54–63; 13:3–20.	Page 12 Encapsulation of crosslinkers and the use of ligands to complex with the transition metal atom have been used to delay crosslinking (82). Page 29 Other means of improving xanthan solution injectivity include... solid polymer hydration in solutions of metal complexing agents such as sodium citrate Page 31 the concentration of divalent metal ions has an effect on viscosity retention at high temperature. Chelating and sequestering agents have been used to reduce the adverse effect of divalent and multivalent metal ions on polyacrylamide solution viscosity.
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;	We claim: 1. A method for enhancing hydrocarbon recovery in a subterranean formation having a blockage or accumulation of material, comprising: introducing components comprising a) a peroxygen, b) a surfactant, c) an alkali metal chelate, and d) a co solvent into a hydrocarbon bearing subterranean formation with a blockage or accumulation of material... 5.27 Inevitably, introduced fluids or changes of formation pressures as a result of drilling, bore hole and well completion, stimulation, fracturing, treating, or production typically results in some level of formation 'damage' or 'blockage' as the fluids intrude the natural formation and have negative interactions in the natural or absolute fluids present in a formation. ... 5.41 The 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. 6.37 Additional blockages often referred to as filter cake (e.g., polymer containing gels and/or silicate residuals), blocking gels, biological cake, or face plugs can also form during various wellbore procedures.	"The method includes the step of injecting a well treatment fluid composition via a wellbore into a subterranean formation." "the injection step is preferably performed at a fluid pressure that is less than the minimum in situ rock stress..." Id. at 4:49–53	Page 7 Formation damage control chemicals are added to reduce the permeability damage that occurs when drilling fluid enters the formation.... Scale inhibitors are used to prevent the formation of insoluble calcium salts when the drilling fluid contacts formation minerals and saline formation waters. Commonly used scale inhibitors include ... polyacrylates, polyphosphates, and phosphonates. Page 18 Reduced injectivity due to formation damage can be a significant problem in injection wells. Precipitate formation due to ions present in the injection water contacting counterions in formation fluids ... can all reduce permeability near the wellbore.... compatibility of ions in the injection water and formation fluids, and filtration can all alleviate formation damage. Page 29 Compared to partially hydrolyzed polyacrylamide, xanthan gum is more expensive, more susceptible to bacterial degradation, and less stable at elevated temperatures. However, xanthan gum is more soluble in saline waters, particularly those containing divalent metal ions... Page 30-31 [Under the section on Polymer Flooding, showing the interaction of injected fluids with in situ formation fluid ions] Thus, the concentration of divalent metal ions has an effect on viscosity retention at high temperature. Chelating and sequestering agents have been used to reduce the adverse effect of divalent and multivalent metal ions on polyacrylamide solution viscosity. Page 38 While the injected chemicals are relatively inexpensive, large quantities must be injected due to reaction of the high pH agents with reservoir clays and by precipitation by divalent metal ions present in formation waters. (This precipitation has been used to reduce adverse surfactant and polymer interactions with divalent metal ions by injecting a caustic preflush prior to a micellar polymer flood).

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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	26.62 b. Allowing the fluid sufficient mixing and contact, before or after the introduction to the well bore, to stabilize, extend the duration of activity, delay the decomposition, and/or control the decomposition rate of the peroxygen, hydrogen peroxide, sodium persulfate, and/or sources of hydrogen peroxide once applied to the wellbore or subterranean formation; US 9,410,405 B2 c. Wherein the composition can react and/or treat over a longer period of time than possible with the use of a peroxygen alone; Claim 6. The method of claim 5, further comprising sealing the well bore after introducing the components. Claim 7. The method of claim 5, further comprising removing the components from the well bore and then extracting hydrocarbon materials after a time delay. Claim 16. The method of claim 1, further comprising: allowing the components to contact blockage or damage in the subterranean formation, thereby altering, removing, degrading or dissolving the damage or blockage to increase a permeability, a relative permeability, or an absolute permeability of the subterranean formation causing an increase in the flow, production, or recovery of hydrocarbons. 8.27 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,	“[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.” “[p]arameters such as . . . shut-in time . . . 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 in the examples provided in Frenier, the treatment solutions were maintained in contact with the formation samples for 24 and 72 hours. 9:27–66.	Page 12 Surfactants are used to stabilize water-in-oil emulsions and to promote rapid return of injected fluid s and a faster return of the well to hydrocarbon production. Page 19 A 6-12 hour well shut-in period i s required to allow polymerization of hydroxylalumi num on formation surfaces to occur. Page 19 Zirconyl chloride can be used to stabilize swelling clays in both acidic environments and in the presence of 600 F steam. No well shut-in time is required for polymerization to occur so zirconyl chloride may be used in conjunction with hydraulic fracturing treatments. Page 19 Quaternary ammonium salt polymers are more versatile and have been used in drilling fluids , completion fluids , acidizing treatments, and hydraulic fracturing. No well shut-in period is required. Page 25 The most commonly used crosslinkers are Al(III) and Cr(III)compounds. The injected fluid preferentially enters the thief zone. The well is shut in to allow crosslinking to occur. After 1-7 days depending on the treatment, normal injection operations are resumed.
removing the low viscosity flow back fluid from the well after the contact time, thereby improving hydrocarbon production of the well.	Claim 7. The method of claim 5, further comprising removing the components from the well bore and then extracting hydrocarbon materials after a time delay.	“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.” 1:55–60. “[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.” Grebe, lines 59–74	Page 18 Scale inhibitors may also be used in acidizing. These include alcohol ethoxysulfonic acids (152). Scale inhibitors are also used in water and enhanced oil recovery injection wells and include low molecular weight poly(vinylsulfonate), polymethylmethacrylate-co ethylenediamine) (153), bis(phosphonomethylene)aminomethylene carboxylic acid, and poly(acrylic acid-co-3-acrylamido-3-methylbutanoic acid). Ethylenediaminetetraacetic acid and similar complexing agents have been used to remove scale from formation surfaces near wellbores. Washing with organic solvents such as xylene or toluene has been used to remove paraffins from perforations and the formation.
2. The method of claim 1, wherein the metal complexing agent is citric acid, formic acid, or mixtures thereof.	11.35 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.	Frenier cites U.S. Pat. No. 4,888,121 to Dill, entitled “Compositions and Method for Controlling Precipitation When Acidizing Sour Wells” (“Dill”). According to Frenier, Dill “discloses an acidizing composition that includes an acid such as CHI; an iron sequestering agent such as citric acid, EDTA, or nitrilotriacetic acid (NTA); and a sulfide modifier such as formaldehyde.” 3:39–45	Page 6 pH control aids in reducing corrosion and scaling and in controlling interaction of the drilling fluid with formation minerals. Sodium hydroxide, calcium carbonate, sodium bicarbonate, sodium carbonate, potassium hydroxide, magnesium oxide, calcium oxide, fumaric acid, and formic acid have all been used commercially in this application. Page 13 Buffers also are used to maintain the proper pH for the crosslinking reaction to occur at an optimum rate. Sodium bicarbonate and sodium carbonate are used to attain basic pH while weak acids: acetic, fumaric, formic, and adipic, are generally used to obtain acidic pH values. Page 14 Acids may be used as breakers to reduce the viscosity of acid sensitive fracturing gels. Precipitation of Fe(III) compounds from acid solutions as the pH increases above 2.2 is a particular problem. Complexing agents that have been used include 5-sulfosalicylic acid and citric acid (136); dihydroxymaleic acid (137); ethylenediaminetetraacetic acid (138); lactic acid (138); blends of hydroxylamine hydrochloride, citric acid, and glucono-delta-lactone (139); nitriloacetic acid; blends of citric acid and acetic acid; lactic acid; and gluconic acid.Precipitation of Fe(III) compounds from acid solutions as the pH increases above 2.2 is a particular problem. Complexing agents that have been used include 5-sulfosalicylic acid and citric acid; dihydroxymaleic acid (137); ethylenediaminetetraacetic acid; lactic acid; blends of hydroxylamine hydrochloride, citric acid, and glucono-delta-lactone; nitriloacetic acid; blends of citric acid and acetic acid; lactic acid; and gluconic acid. Page 25 Adjustment of the pH may allow deeper penetration of the fluid s in an aluminate crosslinking system prior to gelation. Page 26 While successful applications of this technology in waterfloods and in surfactant polymer floods have been reported, temperature and pH stability limitations of the polymer and the

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3. 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.	21.27 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.	3.39 U.S. Pat. No. 4,888,121, Compositions and Method for Controlling Precipitation When Acidizing Sour Wells, discloses an acidizing composition that includes an acid Such as CHI; an iron Sequestering agent Such as citric acid, EDTA, or nitrilotriacetic acid (NTA); and a sulfide modifier such as formaldehyde. This composition is Stated to inhibit precipi- tation of ferric hydroxide, ferrous sulfide, and free sulfur, during the well acidizing treatment.	Page 17 Precipitation of Fe(III) compounds from acid solutions as the pH increases above 2.2 is a particular problem. Complexing agents that have been used include 5-sulfosalicylic acid and citric acid; dihydroxymaleic acid; ethylenediaminetetraacetic [EDTA] acid; lactic acid; blends of hydroxylamine hydrochloride, citric acid, and glucono-delta-lactone; nitriloacetic acid; blends of citric acid and acetic acid; lactic acid; and gluconic acid.
4. 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.	see Claim element 3, above 34.32 Chelates The alkali metal chelate is a chelate of an alkali metal. Alkali metals include lithium (Li), sodium (Na), potassium (K), rubidium (Rb), and cesium (Cs). The alkali metal chelate may be, for example, a mono-, di-, tri- or tetra-sodium ethylenediaminetetraacetic acid (EDTA), a mono-, di-, tri- ortetrapotassium ethylenediaminetetraacetic acid (EDTA), sodium ethylenediamine-N,N'-disuccinic acid (EDDS) or combination thereof. In some embodiments, the alkali metal chelate is sodium EDTA.		See claim element 3, above.
5. 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, nitrilotriacetic acid (NTA), and mixtures thereof.	See 3 and 4, above.	See claim element 3, above.	See claim element 3, above.
6. 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.		Frenier discloses treatment mixtures having between 15% and 28% and between 10% and 30% by weight metal complexing agent. 11:27–34; 11:54–63; 13:3–20.	
7. 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.	Claim 4 4. The method of claim 1, wherein the chelate concentration in the components introduced into the subterranean formation is between about 0.00001 and about 5.0 percent by weight.	2.51 In response to this “radial penetration' problem, organic acids (e.g., formic acid, acetic acid) are sometimes used, since they react more slowly than mineral acids such as HCl. Id. at 5:56–63 (“pH control agents”)	
8. The method of claim 1, wherein the metal-polymer complex remediation mixture comprises between 0.1% and about 60% by weight metal complexing agent.	See Claim element 7, above	See Claims 6 and 7, above	

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9. 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.	Claim 1 A method for enhancing hydrocarbon recovery in a subterranean formation having a blockage or accumulation of material, comprising: introducing components comprising a) a peroxygen, b) a surfactant, c) an alkali metal chelate, and d) a co solvent into a hydrocarbon bearing subterranean formation with a blockage or accumulation of material....	5.56 The composition can also include one or more additives that are compatible with the acid composition, Such as surfactants, corrosion inhibitors (Such as the alkylphenones described in U.S. Pat. Nos. 5,013,483 and 5,096,618), stabilizers, solvents, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. Many such additives are well known in the art.	Page 3 Chemicals of various types are used in every stage of drilling, completing, and producing oil and gas wells. This review describes these chemicals, why they are used, and recent developments. These chemicals include common inorganic salts, transition metal compounds, common organic chemicals and solvents, water-soluble and oil-soluble polymers, and surfactants. As existing fields become depleted, use of chemistry to maintain production via well stimulation, more efficient secondary recovery operations, and enhanced oil recovery become ever more important. Page 20 Adsorption of corrosion inhibitors or cationic surfactants can reduce sandstone formation permeability. Alcohols can be used to remove corrosion inhibitors from rock surfaces. 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.
10. The method of claim 1, wherein the metal-polymer complex remediation mixture comprises: between 1% and 95% by weight metal complexing agent;	Claim 1. ...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.	See Claims 6 and 7, above	
between 1 and 10,000 parts per million (ppm) of a corrosion inhibitor;	see above	5.56 The composition can also include one or more additives that are compatible with the acid composition, Such as surfactants, corrosion inhibitors (Such as the alkylphenones described in U.S. Pat. Nos. 5,013,483 and 5,096,618), stabilizers, solvents, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. Many such additives are well known in the art.	
between 1 and 10,000 ppm of a biocide;	see above	5.56 The composition can also include one or more additives that are compatible with the acid composition, Such as surfactants, corrosion inhibitors (Such as the alkylphenones described in U.S. Pat. Nos. 5,013,483 and 5,096,618), stabilizers, solvents, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. Many such additives are well known in the art.	
between 1 and 10,000 ppm of-a colloidal silica deposition inhibitor; and		5.56 The composition can also include one or more additives that are compatible with the acid composition, Such as surfactants, corrosion inhibitors (Such as the alkylphenones described in U.S. Pat. Nos. 5,013,483 and 5,096,618), stabilizers, solvents, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. Many such additives are well known in the art.	
between 1 and 500 gallons mutual solvent per thousand gallons (gpt) of water.	See Claim element 1	5.56 The composition can also include one or more additives that are compatible with the acid composition, Such as surfactants, corrosion inhibitors (Such as the alkylphenones described in U.S. Pat. Nos. 5,013,483 and 5,096,618), stabilizers, solvents, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. Many such additives are well known in the art.	
11. The method of claim 1, wherein the metal-polymer complex remediation mixture further comprises a pH modifier.	See Claim element 1	5.56 The composition can also include one or more additives that are compatible with the acid composition, Such as surfactants, corrosion inhibitors (Such as the alkylphenones described in U.S. Pat. Nos. 5,013,483 and 5,096,618), stabilizers, solvents, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. Many such additives are well known in the art.	Page 6 pH control aids in reducing corrosion and scaling and in controlling interaction of the drilling fluid with formation minerals. Sodium hydroxide, calcium carbonate, sodium bicarbonate, sodium carbonate, potassium hydroxide, magnesium oxide, calcium oxide, fumaric acid, and formic acid have all been used commercially in this application.
12. 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.	See Claim element 1	"5.56 The composition can also include one or more additives that are compatible with the acid composition, Such as surfactants, corrosion inhibitors (Such as the alkylphenones described in U.S. Pat. Nos. 5,013,483 and 5,096,618), stabilizers, solvents, iron reducing agents, viscosity modifying agents, wetting agents, emulsifiers, non-emulsifiers, and pH control agents. Many such additives are well known in the art. " See example at 6:40--45 6.34 In such an aqueous composition, a small amount of a mineral acid, an organic acid, or a base can be added to provide pH control of the composition. Typical concentration ranges for such an aqueous composition are as follows (percentages are by weight): pH controller 0.1 - 15%	Page 14 Acids can sometimes break emulsions within the formation either by reducing the pH or by dissolving fine particles which can stabilize emulsions. Breaking the emulsion reduces fluid viscosity thus increases the fluid carrying capacity of the flow channel. Acids may be used as breakers to reduce the viscosity of acid-sensitive fracturing gels.

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13. 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, titanilic acid, tungstic acid, xenic acid, and mixtures thereof.	11.34 Acid Treatments 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.	6.34 In such an aqueous composition, a small amount of a mineral acid, an organic acid, or a base can be added to provide pH control of the composition. Typical concentration ranges for such an aqueous composition are as follows (percentages are by weight): pH controller 0.1 - 15%	Page 14 Acids are selected based on the nature of the well treatment and the mineralogy of the formation. The critical chemical factors in properly selecting an acid are: stoichiometry (how much formation material is dissolved by a given amount of acid), the equilibrium constant (complete reaction of the acid is desired), and reaction rate between the acid and the formation material.... Mineral acids include hydrochloric acid and blends of hydrochloric and hydrofluoric acid (usually 12% HCl/3% HF). Hydrochloric acid is used to acidize carbonate formations. Its advantages are relatively low cost, high carbonate mineral dissolving power, and the formation of soluble reaction products (which minimizes formation damage). The primary disadvantage of hydrochloric acid is its corrosive nature. Organic acids used in carbonate rock acidizing include formic, acetic, sulfamic, and chloroacetic acids. These have the advantage of being less corrosive than the mineral acids. This permits use in applications requiring a long contact time with pipe (as perforating fluids) or with aluminum- or chrome-plated pump parts. It is also easier to retard (inhibit) reaction of organic acids with carbonates at elevated temperatures.
14. The method of claim 12, wherein the acid is hydrochloric acid.	11.34 Acid Treatments 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.	6.34 In such an aqueous composition, a small amount of a mineral acid, an organic acid, or a base can be added to provide pH control of the composition. Typical concentration ranges for such an aqueous composition are as follows (percentages are by weight): pH controller 0.1 - 15% See claims 10-13	See claim element 13 above.
15. 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.	6.11 Using downhole packers or plugs within a cased well allows for isolation of vertical levels of the production zone, or isolation of the production zone. This isolation can be important in directing various treatment fluids, or for supporting different production schemes.	"[p]ackers can be used in the wellbore to control the formation zones into which the treatment fluid is injected from the wellbore." 7:27–29. Zerhoub at 1:52--59 [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...	Page 13 Page 25 The most commonly used crosslinkers are Al(III) and Cr(III) compounds. The injected fluid preferentially enters the thief zone. The well is shut in to allow crosslinking to occur. After 1-7 days depending on the treatment, normal injection operations are resumed. Diverting agents assist in distributing acid more uniformly through the perforated formation interval (141). These are usually oil-soluble hydrocarbon resin particles. They may be dissolved by post-acid injection of xylene or similar solvents. Oil-soluble waxes, naphthalene, and solid organic acids such as benzoic acid have also been used. Best results are obtained using a broad range of particle sizes.
16. 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 that does not cause substantial fracturing of the formation.	See claim element 1.	"performed at a fluid pressure that is less than the minimum in situ rock stress...."	Page 13 Page 25 The most commonly used crosslinkers are Al(III) and Cr(III) compounds. The injected fluid preferentially enters the thief zone. The well is shut in to allow crosslinking to occur. After 1-7 days depending on the treatment, normal injection operations are resumed. Page 13 Matrix acidizing is the injection of acids into the formation at a pressure below the formation parting pressure (the pressure at which natural fractures are forced open by injected fluids). Properly designed, the injected acid enters the flow channels of the formation and flows radially outward from the wellbore dissolving mineral fine particles in the flow channels. Minerals forming the flow channel walls also react with the acids. These processes increase formation permeability near the wellbore. The end result is to increase well productivity without increasing the produced water:oil or gas:oil ratios.
17. 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	11. The method of claim 1, wherein the components are introduced into the subterranean formation by at least one from the group consisting of pumping, displacing, and locating the components at a desired location and at a rate and pressure equal to or greater than the reservoir hydraulic fracture pressure. 12. The method of claim 1, further comprising fracturing the subterranean formation.	Zerhoub (incorporated into Frenier) provides that the process disclosed therein "includes a shut-in step between each sequential foam injecting and treatment fluid injecting step." 1:63–65. "performed at a fluid pressure that is less than the minimum in situ rock stress...."	Page 13 Page 25 The most commonly used crosslinkers are Al(III) and Cr(III) compounds. The injected fluid preferentially enters the thief zone. The well is shut in to allow crosslinking to occur. After 1-7 days depending on the treatment, normal injection operations are resumed. Page 13 Matrix acidizing is the injection of acids into the formation at a pressure below the formation parting pressure (the pressure at which natural fractures are forced open by injected fluids). Properly designed, the injected acid enters the flow channels of the formation and flows radially outward from the wellbore dissolving mineral fine particles in the flow channels. Minerals forming the flow channel walls also react with the acids. These processes increase formation permeability near the wellbore. The end result is to increase well productivity without increasing the produced water:oil or gas:oil ratios.

Claim Elements: upon reaching the target pressure, shutting in the well.	United States Patent No. 9,410,405 B2 to Ayers et al. 37:38 The composition may be introduced, for example, into the wellbore. The wellbore may be sealed after introducing the composition. In some embodiments, the wellbore may be left open to the atmosphere. In some embodiments, the composition is removed from the wellbore after introduction. The composition may be removed from the well bore, for example, by pumping or flushing.	U.S. Patent No. 6,436,880 B1 (Frenier) Zerhoub (incorporated into Frenier) provides that the process disclosed therein “includes a shut-in step between each sequential foam injecting and treatment fluid injecting step.” 1:63–65. “performed at a fluid pressure that is less than the minimum in situ rock stress....”	Borchardt, <i>Chemicals Used in Oil Field Operations</i> , 1989 Page 13 Page 25 The most commonly used crosslinkers are Al(III) and Cr(III)compounds. The injected fluid preferentially enters the thief zone. The well is shut in to allow crosslinking to occur. After 1-7 days depending on the treatment, normal injection operations are resumed. Page 13 Matrix acidizing is the injection of acids into the formation at a pressure below the formation parting pressure (the pressure at which natural fractures are forced open by injected fluids) . Properly designed, the injected acid enters the flow channels of the formation and flows radially outward from the wellbore dissolv ing mineral fine particles in the flow channels. Minerals forming the flow channel walls also react with the acids. These processes increase formation permeability near the wellbore. The end result is to increase well productivity without increasing the produced water:oil or gas:oil ratios.Page 13 Matrix acidizing is the injection of acids into the formation at a pressure below the formation parting pressure (the pressure at which natural fractures are forced open by injected fluids) . Properly designed, the injected acid enters the flow channels of the formation and flows radially outward from the wellbore dissolv ing mineral fine particles in the flow channels. Minerals forming the flow channel walls also react with the acids. These processes increase formation permeability near the wellbore. The end result is to increase well productivity without increasing the produced water:oil or gas:oil ratios.
18. The method of claim 1 further comprising: monitoring at least one of pH and well pressure during the maintaining operation; and		Pressure transient analysis is a common technique for estimating the extent of damage.” 6:49–54.	Page 13 Matrix acidizing is the injection of acids into the formation at a pressure below the formation parting pressure (the pressure at which natural fractures are forced open by injected fluids) . Properly designed, the injected acid enters the flow channels of the formation and flows radially outward from the wellbore dissolv ing mineral fine particles in the flow channels. Minerals forming the flow channel walls also react with the acids. These processes increase formation permeability near the wellbore. The end result is to increase well productivity without increasing the produced water:oil or gas:oil ratios. Page 24 Seismic, geotomographic, controlled-source audio magnetotelluric, and pressure analyses as well as tracer chemical injection and analyses are all used to understand flui d movement within the reservoir and monitor EOR processes (191).
initiating the removing operation based on results of the monitoring operation.	See Claim element 17, above	Pressure transient analysis is a common technique for estimating the extent of damage.” Ex. 1005 at 6:49–54.	Page 13 Matrix acidizing is the injection of acids into the formation at a pressure below the formation parting pressure (the pressure at which natural fractures are forced open by injected fluids) . Properly designed, the injected acid enters the flow channels of the formation and flows radially outward from the wellbore dissolv ing mineral fine particles in the flow channels. Minerals forming the flow channel walls also react with the acids. These processes increase formation permeability near the wellbore. The end result is to increase well productivity without increasing the produced water:oil or gas:oil ratios.
19. The method of claim 1, wherein the contact time is between about 4 hours and about 4 days.	Claim 7. The method of claim 5, further comprising removing the components from the well bore and then extracting hydrocarbon materials after a time delay.	“[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.” 7:30–34. Frenier also explains that “shut-in time... must be determined for each particular treatment [but] a well-treatment designer of ordinary skill is familiar with the essential features of matrix acidizing treatments.” Id. at 7:39–46.	Page 13 Page 25 The most commonly used crosslinkers are Al(III) and Cr(III)compounds. The injected fluid preferentially enters the thief zone. The well is shut in to allow crosslinking to occur. After 1-7 days depending on the treatment, normal injection operations are resumed.
20. The method of claim 19, wherein the contact time is between 1 to 4 days.	Claim 7. The method of claim 5, further comprising removing the components from the well bore and then extracting hydrocarbon materials after a time delay.	“[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.” 7:30–34. Frenier also explains that “shut-in time... must be determined for each particular treatment [but] a well-treatment designer of ordinary skill is familiar with the essential features of matrix acidizing treatments.” Id. at 7:39–46.	Page 13 Page 25 The most commonly used crosslinkers are Al(III) and Cr(III)compounds. The injected fluid preferentially enters the thief zone. The well is shut in to allow crosslinking to occur. After 1-7 days depending on the treatment, normal injection operations are resumed.
21. A method for remediating a subterranean-formed metal-polymer complex in an existing, low-producing well in a hydrocarbon-bearing subterranean shale formation, comprising:	We claim: 1. A method for enhancing hydrocarbon recovery in a subterranean formation having a blockage or accumulation of material....	Frenier discloses “methods to enhance the productivity of hydrocarbon wells (e.g., oil wells) by...removing (by dissolution) near-wellbore formation damage.” [a]n acid fluid that is useful in stimulation and workover operations, and in particular for the control of iron in acidizing operations...” Id. at Abstract	
observing a well production decrease;	“[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.” 47:17–22 <i>see also id.</i> at 47:25–28 (“It 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.”)	Frenier discloses “methods to enhance the productivity of hydrocarbon wells (e.g., oil wells) by...removing (by dissolution) near-wellbore formation damage.” [a]n acid fluid that is useful in stimulation and workover operations, and in particular for the control of iron in acidizing operations...” Id. at Abstract	Page 18 Reduced injectivit y due to formation damage can be a significant problem in injection wells. Precipitate formation due to ions present in the injection water contacting counterions in formation fluids , solids initial y present in the injection fluid (scaling), bacterial corrosion products, and corrosion products from metal surfaces in the injection system can al l reduce permeability near the wellbore (153). The consequent reduced injection rate can result in a lower rate of oi l production at offset wells. Dealing with corrosion and bacterial problems, compatibility of ions in the injection water and formation fluids , and filtration can all alleviate formation damage.

Claim Elements:	United States Patent No. 9,410,405 B2 to Ayers et al.	U.S. Patent No. 6,436,880 B1 (Frenier)	Borchardt, <i>Chemicals Used in Oil Field Operations</i> , 1989
providing a metal-polymer complex remediation mixture containing between 1% and 95% by weight metal complexing agent;	Claim 1. comprising: introducing components comprising a) a peroxygen, b) a surfactant, c) an alkali metal chelate, and d) a co solvent into a hydrocarbon bearing subterranean formation with a blockage or accumulation of material 3. The method of claim 1, wherein the peroxygen is introduced into the subterranean formation at a concentration between about 0.01 and about 20 percent.	Frenier discloses a “well treatment fluid,” i.e., a remediation mixture, comprising: “a first acid; water; and a hydroxyethylaminocarboxylic acid.” 4:12–13. Frenier further explains that “[e]xamples of first acids include non-oxidizing mineral acids. . . Other examples include non-oxidizing organic acids such as formic acid, acetic acid, and mixtures thereof.” Id. at 5:27–31. 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. 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 falls squarely within the claimed range set forth in Element 1[a]. 6:25–47. Frenier further discloses treatment mixtures having between 15% and 28% and between 10% and 30% by weight metal complexing agent. See claims 1, 9, and 28 at id., 11:27–34; 11:54–63; 13:3–20.	
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;	We claim: 1. A method for enhancing hydrocarbon recovery in a subterranean formation having a blockage or accumulation of material, comprising: introducing components comprising a) a peroxygen, b) a surfactant, c) an alkali metal chelate, and d) a co solvent into a hydrocarbon bearing subterranean formation with a blockage or accumulation of material... 5.27 Inevitably, introduced fluids or changes of formation pressures as a result of drilling, bore hole and well completion, stimulation, fracturing, treating, or production typically results in some level of formation 'damage' or 'blockage' as the fluids intrude the natural formation and have negative interactions in the natural or absolute fluids present in a formation. ... 5.41 The 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. 6.37 Additional blockages often referred to as filter cake (e.g., polymer containing gels and/or silicate residuals), blocking gels, biological cake, or face plugs can also form during various wellbore procedures.	“The method includes the step of injecting a well treatment fluid composition via a wellbore into a subterranean formation.” “the injection step is preferably performed at a fluid pressure that is less than the minimum in situ rock stress...” Id. at 4:49–53	Page 13 Page 25 The most commonly used crosslinkers are Al(III) and Cr(III)compounds. The injected fluid preferentially enters the thief zone. The well is shut in to allow crosslinking to occur. After 1-7 days depending on the treatment, normal injection operations are resumed. Page 13 Matrix acidizing is the injection of acids into the formation at a pressure below the formation parting pressure (the pressure at which natural fractures are forced open by injected fluids) . Properly designed, the injected acid enters the flow channels of the formation and flows radially outward from the wellbore dissolving mineral fine particles in the flow channels. Minerals forming the flow channel walls also react with the acids. These processes increase formation permeability near the wellbore. The end result is to increase well productivity without increasing the produced water:oil or gas:oil ratios.
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	26.62 b. Allowing the fluid sufficient mixing and contact, before or after the introduction to the well bore, to stabilize, extend the duration of activity, delay the decomposition, and/or control the decomposition rate of the peroxygen, hydrogen peroxide, sodium persulfate, and/or sources of hydrogen peroxide once applied to the wellbore or subterranean formation; US 9,410,405 B2 c. Wherein the composition can react and/or treat over a longer period of time than possible with the use of a peroxygen alone; Claim 6. The method of claim 5, further comprising sealing the well bore after introducing the components. Claim 7. The method of claim 5, further comprising removing the components from the well bore and then extracting hydrocarbon materials after a time delay. Claim 16. The method of claim 1, further comprising: allowing the components to contact blockage or damage in the subterranean formation, thereby altering, removing, degrading or dissolving the damage or blockage to increase a permeability, a relative permeability, or an absolute permeability of the subterranean formation causing an increase in the flow, production, or recovery of hydrocarbons. 8.27 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,	“[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.” “[p]arameters such as...shut-in time...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 in the examples provided in Frenier, the treatment solutions were maintained in contact with the formation samples for 24 and 72 hours. 9:27–66.	Page 13 Page 25 The most commonly used crosslinkers are Al(III) and Cr(III)compounds. The injected fluid preferentially enters the thief zone. The well is shut in to allow crosslinking to occur. After 1-7 days depending on the treatment, normal injection operations are resumed.

Claim Elements:	United States Patent No. 9,410,405 B2 to Ayers et al.	U.S. Patent No. 6,436,880 B1 (Frenier)	Borchardt, <i>Chemicals Used in Oil Field Operations</i> , 1989
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.	Claim 7. The method of claim 5, further comprising removing the components from the well bore and then extracting hydrocarbon materials after a time delay.	“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.” 1:55–60. “[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.” Grebe, lines 59–74	Page 13 Page 25 The most commonly used crosslinkers are Al(III) and Cr(III)compounds. The injected fluid preferentially enters the thief zone. The well is shut in to allow crosslinking to occur. After 1-7 days depending on the treatment, normal injection operations are resumed.
22. 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.	6.11 Using downhole packers or plugs within a cased well allows for isolation of vertical levels of the production zone, or isolation of the production zone. This isolation can be important in directing various treatment fluids, or for supporting different production schemes.	“[p]ackers can be used in the wellbore to control the formation zones into which the treatment fluid is injected from the wellbore.” 7:27–29. Zerhboub at 1:52--59 [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...	Page 13 Page 25 The most commonly used crosslinkers are Al(III) and Cr(III)compounds. The injected fluid preferentially enters the thief zone. The well is shut in to allow crosslinking to occur. After 1-7 days depending on the treatment, normal injection operations are resumed. Page 17 Diverting agents assist in distributing acid more uniformly through the perforated formation interval (141). These are usually oil -soluble hydrocarbon resin particles. They may be dissolved by post-acid injection of xylene or similar solvents. Oil-soluble waxes, naphthalene, and solid organic acids such as benzoic acid have also been used (142). Best results are obtained using a broad range of particle sizes.