

Regular Articles

Altering wettability to recover more oil from tight formations

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ABSTRACT

We describe here a method for modifying the bulk composition (pH, salinity, hardness) of fracturing fluids and overflushes to modify wettability and increase oil recovery from tight formations. Oil wetting of tight formations is usually controlled by adhesion to illite, kerogen, or both; adhesion to carbonate minerals may also play a role when clays are minor. Oil-illite adhesion is sensitive to salinity, dissolved divalent cation content, and pH. We measure adhesion between middle Bakken formation oil and core to verify a surface complexation model of reservoir wettability. The agreement between the model and experiments suggests that wettability trends in tight formations can be quantitatively predicted and that the bulk compositions of fracturing fluid and overflush compositions might be individually tailored to increase oil recovery.

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Introduction

There is an enormous incentive to improve tight formation oil recoveries from their typically low value of 6–8% of the original oil in place. Increased oil recovery would boost reserves while lowering unit operating costs. Here we examine how oil recovery might be increased by designing the bulk composition of the fracturing fluid (salinity, pH, hardness), or that of “squeeze job” overflushes, to make the formation more water-wet. We focus on chemically enhancing oil recovery, but expect that new physical procedures such as high intensity fracturing, refracturing, and choking will aid the chemical enhancement as well since the chemical and physical processes are largely independent. To do this, we identify the primary wettability-affecting reactions in tight formations and build a geochemical surface complexation model to predict how these reactions respond to changes in fluid chemistry. We then test the model by measuring the effect of salinity and pH on adhesion of middle Bakken oil to core. These measurements are combined to describe how fracturing or squeeze job fluids might be altered to increase water wetting and recover more oil from the middle Bakken. The general approach should apply to other tight formations as well.

Tight formations such as the Bakken and the Eagle Ford are source-reservoir systems primarily made up of quartz, feldspar,

calcite, dolomite, illite clay, and as much as 5–10% percent kerogen. **Table 1** shows approximate mineralogies of the three largest tight formations/basins in the US.

Oil tends to be associated with illite, or kerogen when present, or both (Bryndzia and Braunsdorf, 2014). A thin layer of water separates oil from the surface of reservoir minerals (Buckley et al., 1989; Dubey and Doe, 1993) as shown schematically in Fig. 1. Illite, the solid in Fig. 1, possesses a negative charge on its basal plane because of heterovalent substitution in the lattice. Illite negative surface charge is balanced by cations from solution, such as Na⁺, and by positively charged groups present at the oil-water interface, e.g. nitrogen bases, –NH⁺, and calcium-terminated carboxyls, –COOCa⁺. Electrostatic attraction between positively charged oil surface groups and negatively charged clay basal planes, is likely to control oil adhesion in sandstones containing clays such as illite or smectite (Brady and Krumhansl, 2013). While the number of negatively charged basal plane clay groups is fixed by lattice composition, the abundance of charged oil surface groups depends upon the history of the oil and chemistry of the oil and the connate fluid (e.g. pH, salinity, Ca + Mg). High numbers of –NH⁺ and –COOCa⁺ groups should favor formation of electrostatic “bridges” and oil adhesion – that is increase the degree of oil wetting. Decreasing the numbers of –NH⁺ and –COOCa⁺ groups should reduce oil adhesion, making the formation more water wet, resulting in greater oil mobility and recovery.

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Table 1
Lithology of three largest tight oil formations/basins in the USA.^a

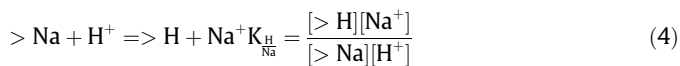
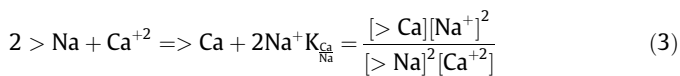
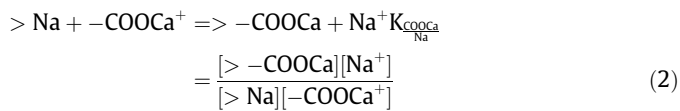
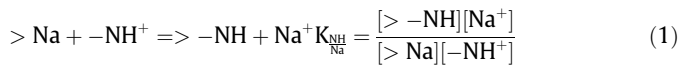
Basin	Production (mmbbl/d)	% Carbonate	% Clay	% Kerogen	Dominant clay
Permian	2040	5–40	10–40	1–10	Illite
Bakken	1220	10–15	10–20	2–8	Illite
Eagle Ford	900	40–60	10–40	5–10	Smec/illite/kaol

^a Balance is mostly quartz or biogenic silica.

Methods

Thermodynamic Modeling

Sorption of $-\text{NH}^+$ and $-\text{COOCa}^+$, and competing Na^+ and Ca^{+2} , onto illite basal planes can be described by the following chemical reactions (Brady and Krumhansl, 2013):



where “>” is a negatively charged site on the clay basal plane and brackets denote aqueous activities and surface concentrations.

Reactions (1) and (2) promote oil-illite linking. Reactions (3) and (4) indirectly affect oil-illite attraction by affecting the amounts of competing exchangeable Na and Ca on the basal planes, and pH. Oil linking through sorption of $-\text{NH}^+$ oil surface groups to illite (Reaction (1)) will be most important at pH values where oil $-\text{N}$ groups are protonated and positively charged ($\text{pH} < 5-6$ at 25°C); and when Na^+ and Ca^{+2} concentrations are low, limiting competition with $-\text{NH}^+$ groups for illite basal plane sites. Electrostatic linking of $-\text{NH}^+$ groups can be decreased, and oil recovery enhanced, by raising the pH (Fig. 1a→b), and/or by increasing the salinity (Fig. 1a→c). Oil linking through $-\text{COOCa}^+$ exchange onto basal planes (Reaction (2)) will be most important at $\text{pH} > 5$ where some oil carboxylate groups are deprotonated and can become Ca-terminated. Low Na^+ will favor oil-illite linking through $-\text{COOCa}^+$ sorption (Reaction (2)) because there will be less competition for illite surface sites. Increased pore water Ca^{+2} will have a mixed effect on oil-illite adhesion; while Ca^{+2} promotes formation of the potentially bridging $>-\text{COOCa}^+$ complex on the oil surface, Ca^{+2} also effectively competes with $-\text{COOCa}^+$ for exchange sites on the illite surface.

Fig. 2 illustrates general oil-illite adhesion trends by showing the number of electrostatic bridges calculated to form between oil and illite as a function of pH and salinity; calculated values are listed in Table 2. The calculation relies on a diffuse layer model

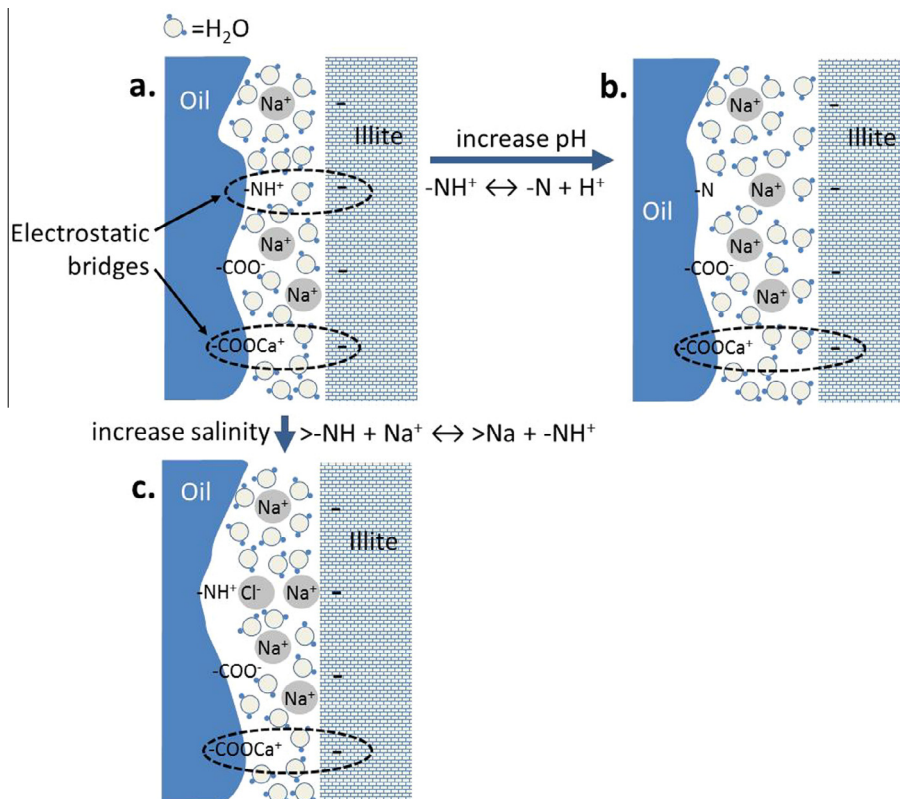


Fig. 1. Schematic of the oil-illite interface showing effects of increased pH (a→b) and increased salinity (a→c) on electrostatic bridging, and the relevant reactions for each change.

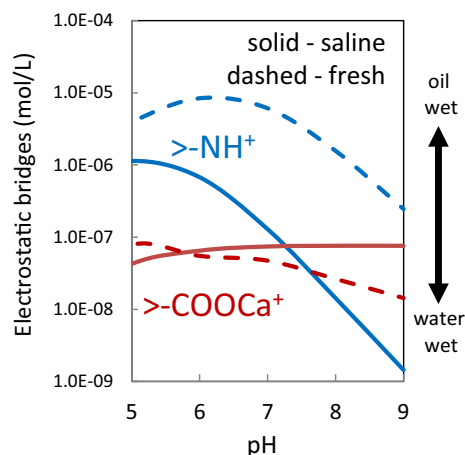


Fig. 2. Estimated effect of salinity and pH on oil-illite binding at 25 °C. The vertical axis is the calculated number of charged oil surface groups attached to the illite basal plane. Blue lines are for oil $-NH^+$ groups attached to illite. Red lines are for oil $-COOCa^+$ groups attached to illite. Solid lines are for saline water (4 M NaCl, 0.5 M $CaCl_2$, 1 mmol C_{tot}); dashed lines are for fresh water (0.02 M NaCl, 0.25 mmol $CaCl_2$, 1 mmol C_{tot}). Each calculation maintained calcite saturation. Calculated aqueous and surface concentrations are listed in Table 2. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

of the oil surface and used the geochemical speciation code PHREEQC (Parkhurst and Appelo, 1999). The calculation was done for 25 °C so that the results can be compared against 25 °C oil-illite adhesion measurements presented later, but is easily done for the ambient temperatures of tight reservoirs which routinely exceed 100 °C, or for other clays, such as kaolinite (Brady et al., 2015). The calculation in Fig. 2 isolates oil-illite interaction, but has also been applied to reservoirs in which the oil is linked to calcite and dolomite (Brady et al., 2012) since oil adhesion to calcite and dolomite depends on surface charge reactions as well (Hirasaki and Zhang, 2004). Oil adhesion to kerogen is probably hydrophobic with some electrostatic linking of charged oil groups to polar functional groups present at the kerogen-water interface; van der Waals forces will also be important.

The calculation in Fig. 2 and Table 2 assumed a base number (BN) acid number (AN) ratio of BN/AN = 10/1. The oil acid and base numbers are thought to roughly correlate with the total number of surface carboxylate and nitrogen base groups present at the oil surface (Dubey and Doe, 1993). Kurtoglu (2014) reported middle Bakken AN's of 0.09 mg KOH/g and "not detect". The single BN

measurement reported by Kurtoglu (2014) was 1.16 mg KOH/g. A rough average BN/AN from oils in conventional reservoirs is 3/1. The rock cation exchange capacity used in the calculation came from Kurtoglu's (2014) measured value of 0.504 meq/100 g for a portion of the middle Bakken.

Experimental verification

Oil-illite adhesion trends measured on middle Bakken oil and cores (Table 2 and Fig. 3) support the theoretical model above. 0.2 ± 0.002 g of sieved core material was added to a set of 40 ml polypropylene centrifuge tubes. Then, 0.01 to 0.4 M NaCl brines were added. Dilute HCl or NaOH was added to each tube to create a range of final pH values. Samples were placed on a shaker table to equilibrate for 24 h, and opened periodically to limit buildup of CO_2 as calcite in the rock dissolved. After 24 h, 2 ml of middle Bakken crude oil was added, the tubes sealed and placed on the shaker table for 2 h, then centrifuged at high speed for 1 h, to separate the oil in solution from the oil that remained on the rock; the crushed rock + oil went to the bottom of the tube. The tubes were then frozen, and a Dremel™ tool used to cut the frozen pellet of crushed rock from the bottom of the tube. The frozen pellet was placed in a 15 ml glass vial and allowed to melt. 10 ml of hexane was added to leach adhering oil from the crushed rock. The hexane + oil was extracted and analyzed by UV-Visible spectrometry to determine the amount of oil present. Absorbance varied directly with the amount of oil present, and was quantified by comparing the samples with standards made using precisely known amounts of the middle Bakken oil.

Discussion

Fig. 3 shows that measured oil-illite adhesion decreases with increasing NaCl content and with increasing pH, consistent with thermodynamic model predictions from Fig. 2. The pH effect is clearest for the high salinity fluids that are most characteristic of middle Bakken formation waters. There is likely to be an optimal combination of pH and salinity that best leverages wettability alteration. Oil that adheres less strongly to the reservoir solids is more likely to be swept to the wellbore along the fractures by the ambient pressure field.

Low salinity increases oil adhesion in illite-containing reservoirs, but apparently decreases oil adhesion in kaolinite-containing reservoirs (Lager et al., 2006; Jerauld et al., 2008; Morrow and Buckley, 2011; Sandengen et al., 2011; Skrettingland et al., 2011; Nasralla and Nasr-El-Din, 2012; Suijkerbuijk et al.,

Table 2
Calculated aqueous and surface concentrations (all except pH in mol/L).^a

	pH	Ca	Na	$-NH^+$	$-COOCa^+$	$>H$	$>Na$	$>_2Ca$	$>-NH$	$>-COOCa$
Saline	4	1.47E+00	3.97E+00	5.93E-05	1.05E-06	6.02E-02	4.04E-02	5.26E-03	6.03E-07	1.07E-08
	5	6.22E-01	3.99E+00	5.42E-05	2.04E-06	1.13E-02	8.33E-02	9.10E-03	1.13E-06	4.25E-08
	6	5.03E-01	3.98E+00	2.88E-05	2.78E-06	1.24E-03	9.33E-02	9.23E-03	6.75E-07	6.50E-08
	7	4.97E-01	3.98E+00	5.43E-06	3.14E-06	1.25E-04	9.42E-02	9.31E-03	1.29E-07	7.43E-08
	8	4.96E-01	3.98E+00	6.02E-07	3.21E-06	1.26E-05	9.43E-02	9.32E-03	1.42E-08	7.60E-08
	9	4.96E-01	4.00E+00	6.08E-08	3.21E-06	1.25E-06	9.44E-02	9.28E-03	1.44E-09	7.59E-08
Fresh	4	9.77E-01	1.02E-01	5.92E-05	9.14E-07	8.66E-02	1.94E-03	1.13E-02	1.12E-06	1.73E-08
	5	2.46E-01	2.05E-02	5.24E-05	1.04E-06	3.14E-02	1.55E-03	3.98E-02	3.95E-06	7.84E-08
	6	1.16E-02	1.60E-02	2.19E-05	1.43E-07	1.56E-02	6.15E-03	4.56E-02	8.43E-06	5.51E-08
	7	1.01E-03	4.12E-02	5.59E-06	4.33E-08	4.45E-03	4.51E-02	3.16E-02	6.12E-06	4.74E-08
	8	1.79E-04	3.40E-02	7.50E-07	1.26E-08	8.44E-04	7.13E-02	2.04E-02	1.57E-06	2.64E-08
	9	5.92E-05	2.78E-02	8.01E-08	4.68E-09	1.23E-04	8.53E-02	1.37E-02	2.46E-07	1.44E-08

^a For the calculation: exchange constants for Eqs. (1) and (2) = 1; acidity constants for $-NH^+$ and $-COOH = 10^{-6}$ and 10^{-5} ; log K for the reaction $-COOH + Ca^{2+} \leftrightarrow -COOCa^+ + H^+ = 10^{-3.8}$ (Brady and Krumhansl, 2013); illite ion exchange capacity = 0.313 mol/L. The thermodynamic database used was thermo.com.V8.R6.230 from Lawrence Livermore National Laboratory. Illite exchange constants were the default PHREEQC thermo.com.V8.R6.230 ion exchange values. The overall calculation was done with PHREEQC version 2.15.06. Rock porosity was assumed to be 20%.

Table 3
Oil adsorption measurement results.

	Acid added (meq)	pH final	Oil extracted (g/g hexane)	Clay (g)	Hexane (g)	Sorbed oil (g/g clay)
0.1 M NaCl	0.00E+00	9.025	0.001774654	0.2032	6.4926	0.05670332
	2.50E-06	9.113	0.001215888	0.2056	7.009	0.04145019
	5.00E-06	9.175	0.001208712	0.2049	6.9346	0.04090743
	7.50E-06	9.021	0.001913739	0.2047	6.8489	0.06403034
	1.00E-05	8.893	0.001866836	0.2023	6.7787	0.06255424
	1.25E-05	8.662	0.001626952	0.2042	6.7174	0.05352049
	1.50E-05	8.725	0.001538915	0.2058	6.7031	0.05012392
	1.75E-05	8.364	0.00163357	0.2047	6.6277	0.05289111
	2.00E-05	8.555	0.001628296	0.2083	6.6963	0.05234547
	2.25E-05	8.472	0.001795011	0.2046	6.6159	0.05804307
	2.50E-05	8.718	0.001823493	0.206	6.6094	0.05850581
	2.75E-05	8.332	0.001695808	0.2046	6.633	0.054977
	0.4 M NaCl	-2.50E-06	8.534	0.000643715	0.2001	6.4854
0.00E+00		8.626	0.000646437	0.2009	6.3847	0.02054408
2.50E-06		8.594	0.000369508	0.2004	6.7852	0.01251091
5.00E-06		8.552	0.000584112	0.2006	6.7535	0.01966502
7.50E-06		8.362	0.000575909	0.2004	6.5991	0.01896449
7.50E-06		8.362	0.000625815	0.2004	6.5991	0.02060786
1.00E-05		8.308	0.000505915	0.2007	6.6822	0.01684417
1.50E-05		8.338	0.00050979	0.2009	6.6387	0.01684591
2.00E-05		8.427	0.000475712	0.2004	6.6638	0.01581862
2.50E-05		8.283	0.000758866	0.2009	6.6431	0.02509318
3.00E-05		8.307	0.000755669	0.1998	6.6257	0.02505925
3.50E-05		8.136	0.000615774	0.1998	6.6076	0.0203643
4.00E-05		8.435	0.000512	0.2002	8.4063	0.02149863
4.0 M NaCl	0.00E+00	8.539	0.000150168	0.2002	6.5223	0.00489232
	1.00E-05	8.228	0.000159217	0.2000	7.0296	0.00559617
	2.00E-05	7.97	0.000153062	0.2016	6.9079	0.00524471
	3.00E-05	7.891	0.00015135	0.2013	6.8579	0.0051562
	4.00E-05	7.699	0.000138269	0.2007	5.7698	0.00397502
	5.00E-05	7.663	0.000210748	0.2002	6.3441	0.00667836
	6.00E-05	7.496	0.000238805	0.2003	6.8554	0.00817325
	7.00E-05	7.402	0.000326024	0.2001	7.0552	0.01149506
	8.00E-05	7.236	0.000408136	0.2001	6.1153	0.01247313
	9.00E-05	7.184	0.000373848	0.2008	7.1427	0.01329823
	1.00E-04	7.353	0.000428803	0.2009	6.8276	0.01457291

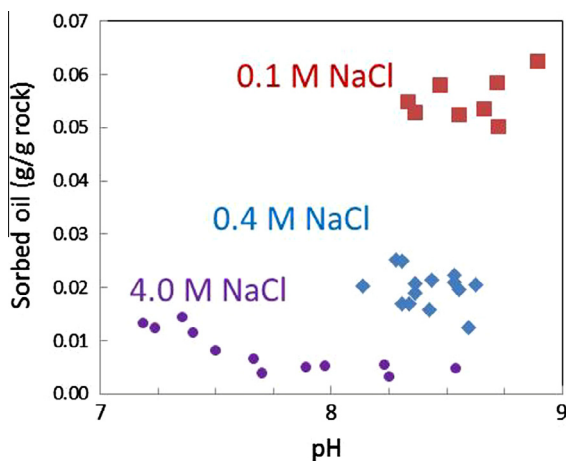


Fig. 3. Oil adsorption in the middle Bakken as a function of pH and NaCl. Values are given in Table 3.

2012). The tendency for low salinity solutions to decrease oil recovery from clay basal planes has been observed experimentally before (Rezaeidoust et al., 2010; Lebedeva and Fogden, 2011), and appears to be the direct result of the different oil sorption characteristics of clay basal planes vs. clay edges (Brady and Krumhansl, 2013). Illite surface charge is mostly basal planes; reservoir kaolinite is mostly edges.

Independent of fluid chemistry, fracturing of tight formations produces oil because it increases permeability and exposes fractured rock surface area. But how much oil comes out will depend

on oil-rock adhesion as well as the pressure drive and effects of fracturing. Figs. 2 and 3 suggest that fresh, slickwater fracturing fluid probably increases oil adhesion to the middle Bakken in areas contacted by the slickwater. But several processes are at work. Baseline oil adhesion is low because of the high salinity of the reservoir waters. Injection of low salinity fracturing fluid increases oil adhesion because it decreases salinity. This oil adhesion increase is somewhat moderated by the rise in pH that low salinity fluids cause: low salinity shifts Reaction (4) to the right raising local pH (Vaidya and Fogler, 1990) thereby decreasing oil adhesion. Low salinity fluids will also imbibe into the rock more forcefully than high salinity brines, pushing oil out of the rock and into the wellbore (Schembre et al., 2006; Sandengen and Arntzen, 2013). Connate brine seeping and diffusing back into the rock after fracturing will increase salinity from that of slickwater, and consequently lower local pH (Reaction 4 going to the left). Increased salinity favors oil desorption; decreased pH works against it.

To better understand oil production in reservoirs where the salinity difference between connate water and slickwater is very large, a process model that explicitly treats salinity-driven imbibition may be required. Some tight formations strongly imbibe aqueous solutions through capillary and osmotic effects (Dehghanpour et al., 2012; Kathel and Mohanty, 2013; Engelder et al., 2014; Nguyen et al., 2014, 2015). Imbibed water displaces oil into fractures and interbeds with larger pores and lower capillary pressures, through which it may return to the hydrofracture network via capillary/osmotic counterflow. One benefit in such reservoirs is that imbibition may be utilized to pull wettability-altering chemicals into the rock, increasing hydrocarbon yield (Dehghanpour et al., 2012; Kathel and Mohanty, 2013; Nguyen

et al., 2015). Dilute solutions should be more strongly imbibed into hypersaline reservoirs because of the greater osmotic pressure difference. Thus, use of dilute fracture or reworking fluids in these reservoirs will potentially enhance production by driving capillary/osmotic counterflow, but will result in greater oil adhesion, reducing oil mobility. Conversely, use of more concentrated fluids will reduce counterflow but enhance oil mobility by decreasing adhesion. An optimized water composition might promote the effects of both imbibition and decreased adhesion while decreasing hydrofracturing demand for freshwater.

Parallel reactions between injectate and the reservoir must also be considered when optimizing injectate chemistry, in particular, scale formation and rock-buffering of fluids. The high salt content of waters in many unconventional reservoirs could make them sensitive to scale formation caused by injectates. For example, increases in subsurface pH would favor calcite formation, as would increases in Ca^{+2} . Note also that the rock itself will alter injectate chemistry and cause the composition of the fluid that comes into contact with the oil to be different from the composition of the fluid originally injected. In addition to simple mixing with the connate fluids, the most important of these rock-buffering reactions are likely to be ion exchange reactions on the clays and calcite dissolution/growth. Obviously the net effect will be reservoir-specific.

Conclusions

Oil-illite adhesion at 25 °C depends on both salinity and pH. For high base number oils, increasing pH decreases adhesion by deprotonating adhering oil $-\text{NH}^+$ groups thus decreasing their electrostatic attraction for negatively charged illite basal planes. Increased salinity tends to decrease adhesion by increasing the number of cations at the clay basal plane that positively charged oil groups, $-\text{NH}^+$ and $-\text{COOCa}^+$, must compete with. For high acid number oils, added Ca^{+2} can in some cases increase the number of $-\text{COOCa}^+$ groups and thereby increase oil adhesion to illite. Other important reactions that might potentially occur in parallel to the above wettability-altering reaction include: pH shifts due to ion exchange, scale formation, and fluid-mixing. Salinity-dependent imbibition will in many cases control the latter.

The oil adhesion model presented above lends itself to optimizing the wettability-enhancing properties of fracturing fluids and that of squeeze jobs designed to alter wettability to recover more oil from previously fractured rock. Again, technology improvements that physically increase the extent and surface area of the fractures, or control reservoir pressure losses, should increase the amount of extra oil recovered through wettability alteration.

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