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A Novel Method to Evaluate Ability of Surfactants in Slickwater Fluids to Minimize Formation Damage and Optimize Hydrocarbon Recovery after Hydraulic Fracturing

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Abstract

In recent years, use of slickwater fracturing fluids has emerged as a standard practice in fracturing U.S. shale reservoirs. Using less proppant and fewer components than traditional fracturing fluid systems, slickwater systems have greater fracture-conductivity than similar guar-based systems. With the adoption of this new technique, a key area of interest is in assessing the impact of surfactant additives on improving permeability and maximizing hydrocarbon recovery. This study examines the ability of surfactants to mitigate permeability impairment in a slickwater system.

Although the effects of various surfactants on regain permeability have been investigated in the past, approaches in the literature have not accounted for key petrophysical parameters that affect flow properties through porous media. The new method described here uses a coreflooding rig to both accurately simulate the formation during hydraulic fracturing, and to categorize the effectiveness of flowback aids in different mineralogies. By using a fracturing fluid system to treat a core sample, field dosages of different surfactant and microemulsion packages were injected until differential pressures stabilized. Oil regain permeability was compared to initial oil permeability to determine the improvement factor for each. This approach can distinguish between various classes of additives and can assist operators in selecting the right chemistry for their wells.

Introduction

The use of polymeric additives in hydraulic fracturing fluids is ubiquitous and has historically included the use of guar and other viscosifying polymers as well as friction reducing polyacrylamides and related copolymers and derivatives. In most cases, chemical additives called breakers are included in the fracturing fluid to break down the polymer into lower molecular weight fragments once the treatment has been completed. Incomplete guar degradation has been shown to result in reduced proppant pack conductivity and lower hydrocarbon production due to the presence of polymer residue in the fracture and as a filter cake along the fracture walls.¹⁻⁴ Slickwater fluids contain lower polymer concentration than traditional guar fluids and the use of polyacrylamide friction reducers has been reported to cause less damage to proppant

pack conductivity.² The massive volume of these treatments, however, means that there is still a large quantity of polymer pumped into the formation so permeability impairment by polymer residue remains a concern, especially in tight rock and shale formations with already low permeability.⁵⁻⁷

Incorporation of surfactant additives into fracturing fluid formulations with the intention of enhancing hydrocarbon production has become common. Such products are often labeled as flowback aids or interfacial tension and wettability modifiers, and are expected to assist with recovery of stimulation fluid from the formation after treatment and prevent or remediate damage like water blockage. Several investigators have used proppant packed columns to evaluate the influence of surfactant and microemulsion formulations on fluid recovery, but the results of such studies are often contradictory and do not provide a reliable method for correlating surfactant solution properties with fluid recovery performance.⁸⁻¹⁰ Additionally, fracturing fluids that have been displaced into the formation during treatment will interact with the rock matrix in ways that are not represented in the proppant packed column tests.¹¹

Core flow evaluation to measure oil and/or water permeability of a rock sample has been used to overcome some of the limitations of the proppant pack test but no standard test methods have been defined in the literature, making it difficult to draw comparisons between reported test results. Aging the rock cores at elevated temperatures during the coreflood procedure results in unpredictable (and sometimes unintended) changes to the rock wettability, particularly with the wide range of aqueous and hydrocarbon fluids used. In many cases, regain permeability tests are not performed at various flow rates to measure the endpoint of permeability and thus fail to account for the influence of petrophysical parameters such as the capillary end effect which can affect fluid distribution and flow in porous media.

In our work, we have found that injection of broken polyacrylamide friction reducer solutions is a useful method for simulating fracturing fluid leakoff of slickwater treatments to reproducibly damage rock cores by reducing their permeability to oil. The damaged cores can be used to study the effectiveness of surfactant or microemulsion additives for remedial cleanup

of the induced damage, and the inclusion of a surfactant/microemulsion additive in the broken polymer fluid prior to pumping the fluid through the core provides information about the ability of additives to prevent polymer damage.

This paper examines the ability of surfactant and microemulsion formulations to remediate and prevent permeability impairment caused by broken polyacrylamide friction reducer residue. Limestone and sandstone cores that had been damaged by injection of broken friction reducer were treated with surfactant or microemulsion formulations and the change in oil regain permeability measured with either kerosene or shale crude oil.

Experimental

Materials

Indiana Limestone cores having a length of 2 inches and diameter of 1.5 inches were extracted from a single limestone outcrop block with absolute permeability between 2 – 4 mD. Initial wettability of the limestone was water-wet with a porosity of 0.16 – 0.17. Gray Sandstone cores with a length of 6 inches and a diameter of 1.5 inches were cut from a single sandstone block. Absolute permeability of the sandstone was between 56 – 60 mD and the rock was initially water-wet with a porosity of 0.19 – 0.20. Sandstone and limestone cores were dried at 200°F to determine dry weight. The cores were then saturated with 2% KCl using a vacuum pump, and weighed again to determine pore volume.

2 wt% KCl brine was used as aqueous phase for all the coreflood experiments. Kerosene and Shale Crude were used as the hydrocarbon phases for coreflood tests. Several studies have reported using surrogate oil, such as kerosene, isoparaffinic oil, mineral oil, and *n*-pentane, to represent a light hydrocarbon from low permeability unconventional reservoirs^{8-10, 12-14}, so a commercial laboratory grade of kerosene was obtained. Shale Crude was obtained from an unconventional reservoir and centrifuged to remove base sediment and water, followed by filtration through 0.45 μm nitrocellulose filter paper under pressure to remove remaining solids prior to use. API gravity of the Shale Crude was 33° and the viscosity was 2.2 at 65°C (144°F).

Seven flowback aid formulations (A – G) were evaluated for their ability to affect regained oil permeability in coreflood tests. Five of the formulations were identified as microemulsions, while Formulation E and F were surfactant blends. Surfactant Formulations A – G were added to 2 wt % KCl brine at a concentration of 0.2% by volume (2 gal/1,000 gal, or 2 gpt), which falls within the recommended field loading range for typical flowback aids or surfactant additives.

To simulate a broken slickwater fracturing fluid, a commercially available dry polymer consisting of partially hydrolyzed polyacrylamide (HPAM) was dissolved in aqueous 2 wt % KCl to give a 1000 ppm HPAM solution. Once the solution was visually homogenous, it was sheared at 10,000 s⁻¹ for two hours using an IKA T50 laboratory homogenizer. Over the course of the shearing, the temperature rose from ambient

to 210°F. The solution viscosity dropped from 3.5 cP to 1.5 cP at 100 s⁻¹ indicating that the polymer was mechanically degraded or “broken”. The broken HPAM solution was filtered through a 350 mesh screen to remove any insoluble matter prior to use.

Calculations

The oil permeability of the core was calculated according to Darcy’s law.

$$k = \frac{Q \times l \times \mu}{\Delta P \times A}$$

where

k = permeability

Q = flow rate

l = core sample length

μ = the oil viscosity

ΔP = pressure drop

A = core cross sectional area

The improvement factor (IF) is defined by:

$$IF = \frac{k_{final}}{k_{initial}}$$

where

k_{final} = final permeability

$k_{initial}$ = initial permeability

Methodologies

Current methods used to describe the effects of surfactant additives in a fracturing fluid are limited. Two common tests, the sand pack column test and regain permeability test, are widely used to measure fluid recovery and return permeability.^{13, 15-20}

In the carry-over test, a glass column is filled with a mixture of fracturing fluid and solid proppant and connected to a flowmeter at the lower end and weighing balance to measure collected effluent at the upper end. Using a set flow rate of gas or displacing solvent, the percent recovery of the initial fluid can be calculated and the effect of added surfactant can be compared. Himes, et al.¹⁸ used the same packing procedure in the gravity drainage column test and showed that one of difficulties found with the test was reproducibility; differences in column packing and operating procedures can cause significant deviations in reproducibility. Furthermore, one possible explanation for the conflicting results in the literature could be the gas which can cause foaming of surfactant-containing fluids in sand pack. The packed column method also assumes that all fracturing fluid to be recovered is located in the proppant pack, which means that key petrophysical parameters affecting fluid flow through the formation rock are omitted in this test method.

An alternate method to investigate the effect of surfactants on fluid recovery and regain permeability utilizes a coreflooding rig. For this set of experiments, a Chandler Model 6100 Formation Response Tester was used to simulate downhole conditions. These coreflood tests focus on the reservoir-matrix region near the fracture face in a hydraulically fractured reservoir. As shown in Figure 1, high pressure fracturing fluids enter into the reservoir-matrix and flow perpendicular to fracture faces. During the fracturing treatment, fluid will leak off into the formation leaving polymer residue as a filter cake on the formation face or trapped in the near-wellbore region. This leak-off can generate formation damage due to phase trapping and residual fracturing fluids. Longoria, et al.¹⁴ explained schematic of field geometry and laboratory scaling in detail. No attempt was made to evaluate fluid recovery in these tests due to the small pore volume of the selected rock cores relative to the total system volume; instead, these studies concentrated on reproducibly evaluating the regain permeability of core samples. Several replicate control tests (without surfactant) were performed to determine the IF precision and it was found to be ± 0.02 . This number was used for the IF determinations in this work.

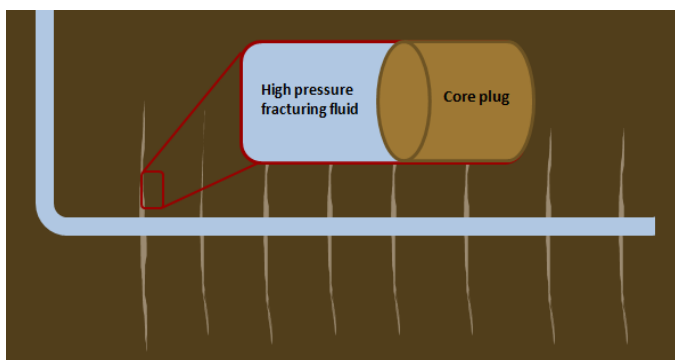


Figure 1. Schematic of field geometry and core testing

Replication of Literature Coreflood Conditions

Initial coreflood tests were performed using a procedure similar to that described by Mahmoudkhani, et al.¹³ to examine the effect of different surfactant formulations on core permeability. Six inch length sandstone cores were loaded into the coreflood holder, heated to 150°F, and then injected with 2% KCl in the forward direction. Initial water permeability was determined after several pore volumes of 2% KCl solution had been passed through the core at an injection rate of 4 mL/min. Next, Shale Crude was passed through the core in the reverse direction and the oil permeability was determined. To avoid wettability alteration of the core in this study, an aging step was not included during coreflood experiments. Five pore volumes of a 2% KCl test solution was then injected in the forward direction. The tests solutions were either 2% KCl only as control or 2% KCl with 2 gpt Formulation A. Following the test solution, a final Shale Crude injection was done in the reverse direction to determine the IF.

One-Phase and Two-Phase Coreflood Procedures

In order to create damage in the core and evaluate surfactant formulations, a modification was made to the coreflood procedure above and broken polymer was injected into the core to simulate formation damage caused by the leakoff and potential flowback of a slickwater fracturing fluid following a stimulation treatment.

A one-phase coreflood using only an aqueous fluid phase was designed to create permeability impairment due to the inefficient cleanup of broken friction reducer polymer in both tight limestone core and gray sandstone core. Two inch length sandstone and limestone cores were prepared, then individual cores were loaded into a coreflood holder at 77°F and injected with 2% KCl in the forward direction to measure absolute permeability. Then, 50 – 70 pore volumes (PV) of broken polymer solution was injected in the forward direction until the pressure stabilized. 2% KCl was re-injected in the forward direction at various flow rates and the permeability measurements were compared before and after broken polymer injection to calculate the permeability reduction caused by the polymer.

A two-phase coreflood procedure using both aqueous and oil phases was designed to understand the effect of residual polymer on relative permeability and chemical performance in the presence of oil. Two inch length limestone cores were loaded into a coreflood holder and heated to 150°F before injection with 2% KCl in the forward direction to measure absolute permeability. Oil (kerosene or Shale Crude) was then injected in the reverse direction at various flow rates and initial oil permeability was measured. 2% KCl was injected in the forward direction after the initial oil flood and the initial water permeability was measured. Around 50 – 70 PV of broken polymer solution (with and without surfactant formulation) was injected in the forward direction until the pressure stabilized. Oil was re-injected at various flow rates in the reverse direction to measure the final oil permeability for comparison with initial oil permeability and calculation of the overall improvement factor, IF.

Results

Literature Replication Study

Table 1 lists data from the literature replication experiment. For the initial surfactant injection, a microemulsion flowback aid was selected. It was observed that a treatment of several pore volumes of a 2 gpt dilution of Formulation A resulted in an IF of approximately 1.05 (Coreflood 2) while the control experiment containing no added surfactants had an IF of 1.02 (Coreflood 1).

Table 1. Surfactant injection in sandstone core

Coreflood #	Initial Permeability (mD)	Final Oil Permeability (mD)	IF (Shale Crude)
Coreflood 1	53.9	55.5	1.02
Coreflood 2	54.4	57.0	1.05

In assessing the results from these initial coreflood tests, the difference in IF for the two runs was within the observed standard error for the method. Thus, it was determined that this procedure does not effectively differentiate between surfactant formulations and the control. This is clearly different than the results expected based on previous studies. One probable reason for this is that damage in the form of water blockages or wettability alteration is never created in the core. It appears that some previous studies also did not assess for the capillary end effect, which may give incorrect values for the measured endpoint permeability. It was also observed that some cases in the literature used pressure drop values before pressure readings stabilized. All of these can lead to false assumptions on the state of the core as the system is still in a state of flux.

One-Phase Coreflood – Polymer Damage

One-phase coreflood with gray sandstone was performed at room temperature and absolute permeability using 2% KCl was measured to be 56.3 mD. Figure 2 illustrates the pressure response at flow rates between 4 to 0.5 mL/min. The absolute permeability was determined once the differential pressure showed a variability of less than 1% at each flow rate. During the test, 55 pore volumes (PV) of broken polymer fluid were injected into the gray sandstone core. As shown in Figure 3, the pressure stabilized after approximately 20 pore volumes of polymer had been injected. After the polymer injection, 2% KCl was again injected to calculate the IF of water. Averaged final water permeability was found to be 39.4 mD, and the precision of permeability was within 1%. Figure 4 shows the pressure response ramp during this stage. For the post-waterflood step, the permeability was determined using the data from the second ramp, since these showed better stabilization at the different flow rates. This injection shows that the addition of the polymer injection resulted in a 30% reduction to the overall permeability. It was observed that additional injection of brine during the post-water step did not affect the final permeability, this suggests that the mechanism of permeability impairment is independent of rate or volume of the injection fluid and indicates permanent permeability damage.

The same procedure was used for limestone coreflood testing. Tighter core (2.9 mD) was used and pressure stabilized after 40 PV of polymer injection. The observed pressure response during the polymer and subsequent post-waterflood injections was similar to those using the sandstone cores. The final permeability reduction in the limestone cores was observed to be 69%, which is significantly more than the damage observed in the sandstone cores. One reason could be that less permeable rock will see an increase in damage caused by the polymer injection due to pore size reduction. It was also observed that a longer equilibration time was required during the polymer injection stage. More studies are planned to examine the differences in mineralogy and permeability on polymer damage using this method.

As summarized in Table 2, these results demonstrated that residual polymer in both sandstone and limestone affected permeability even in the absence of oil. Given the reproducible polymer damage in one-phase coreflood, this polymer study

was expanded to two-phase corefloods.

Table 2. Summary of one-phase corefloods

Coreflood #	Mineralogy	Initial (mD)	Post (mD)	IF (water)
Coreflood 3	Gray sandstone	56.3	39.4	0.70
Coreflood 4	Indiana limestone	2.9	0.9	0.31

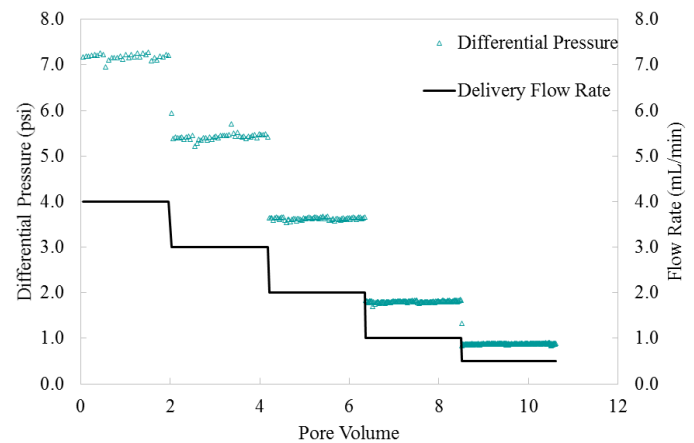


Figure 2. Brine injection in gray sandstone

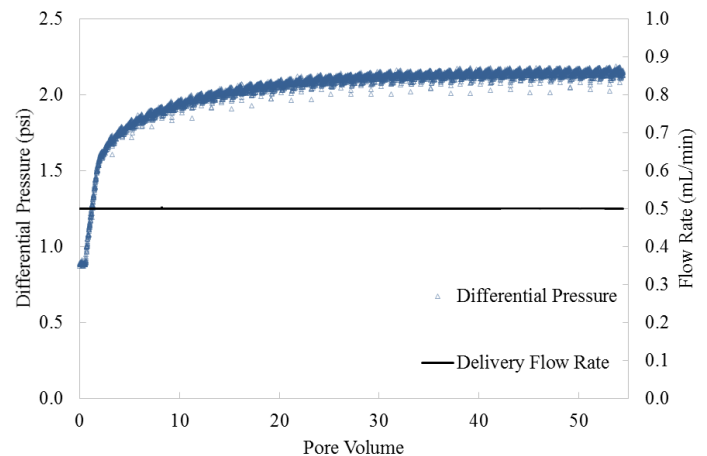


Figure 3. Injection of broken polymer into gray sandstone showing pressure response

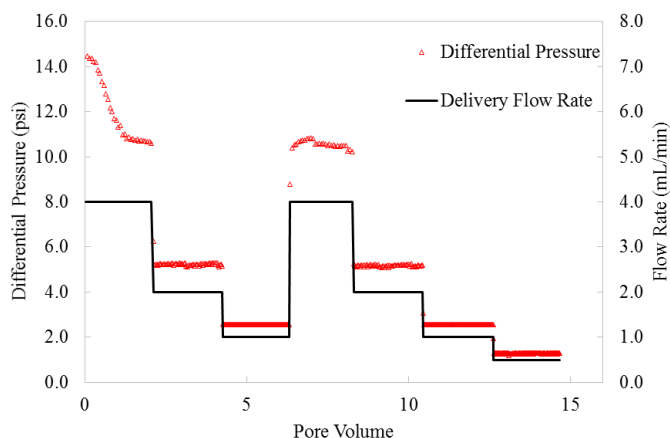


Figure 4. Post-waterflood in gray sandstone

Two-Phase Coreflood

Control – Polymer Injection

First, a two-phase coreflood without surfactant was carried out in limestone to establish a baseline for evaluating the performance of microemulsion and surfactant formulations. The control test using kerosene gave an IF of 0.77, while the control with Shale Crude was measured to be 0.69. Figure 5 shows oil permeability before and after Shale Crude injection. Various flow rates were used to confirm end point permeability and eliminate capillary end effect. Trailing tails shown in Figure 5 illustrates the pressure equilibration delay once flow rate was changed. No emulsion was observed in the effluent with both kerosene and Shale Crude. Based on these observations, it is believed that the permeability impairment was truly caused by broken polymer.

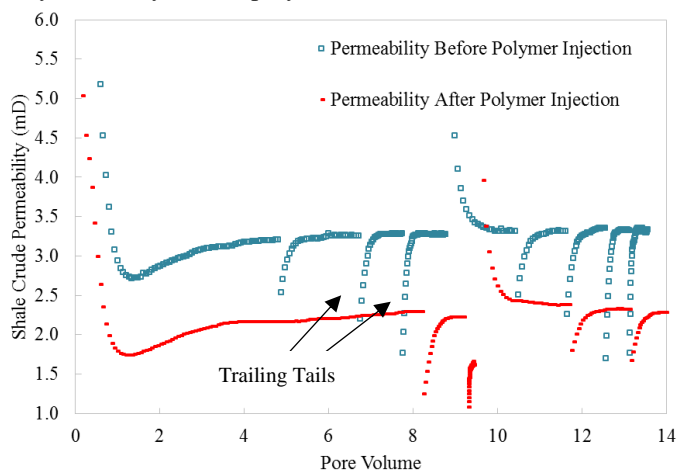


Figure 5. Shale Crude permeability in limestone core before and after broken polymer injection

Performance Evaluation – Polymer/Formulation Co-Injection

As described in the methods section, the purpose of this testing was to examine the effects of surfactant formulations when co-injected with polymer. A field strength (2 gpt) dosage was used

as the standard for each injection. Table 3 summarizes the results of the two-phase corefloods with the different injected surfactant blends and corresponding measured IF with oil. Results show that when using kerosene as the surrogate oil, all of the microemulsion formulations showed poorer to the same performance as the control test. One possible explanation is that surfactant formulations adversely affected the IF due to emulsion formation in the core. Effluent from tests conducted with Formulations B, C, and D all showed visible emulsions that did not immediately break after injection. To test the impact of emulsion formation on the IF, a demulsifier (Formulation E) was injected into the core in the place of a flowback aid. This test resulted in a nearly clear effluent; however, the IF for this was approximately equivalent to the IF in the control test and added no significant improvement.

In order to better describe the ability of the polymer injection method to effectively differentiate between different formulations, Coreflood 8 containing Formulation D was duplicated using the exact same test conditions. As shown in Table 3, the observed IF was 0.58 for the first run and 0.59 for the second. These results agree within the previously estimated precision of the coreflood tests. While the IF for Formulation D was observed to be lower than other formulations, this test clearly highlights the reproducibility of the method.

The next set of coreflood results used a filtered crude oil for the oil injection steps. The results with Shale Crude show that all of the tested formulations had higher IF compared to the control. Interestingly, formulations that resulted in substantially lower IF values when using kerosene showed the opposite response with the crude oil. Injection of Formulation B gave a 45% decrease in IF with kerosene, but had a nearly 6% increase in IF with Shale Crude. One possible explanation is that the interactions between surfactant and oil highly depends on the constituents of each oil. It is likely that the presence of natural organic acids, asphaltenes, and resins present in the crude oil affect the performance of the flowback aid.

Formulation G, a non-viscous oil-in-water microemulsion showed the best performance in remediating the damage from residual polymer in the corefloods with Shale Crude. The IF in oil was found to be 0.88 and this value represents a 28% improvement compared to the control value of 0.69. Analysis of the effluent from the two coreflood tests also highlighted differences in the selected surfactants. Figure 6 shows that a slightly milky emulsion was initially exhibited in both effluent samples. While the effluent from using Formulation B remained stable, the effluent containing Formulation G quickly separated. This may indicate that emulsion tendency may have had an effect on the lower IF observed using Formulation B.

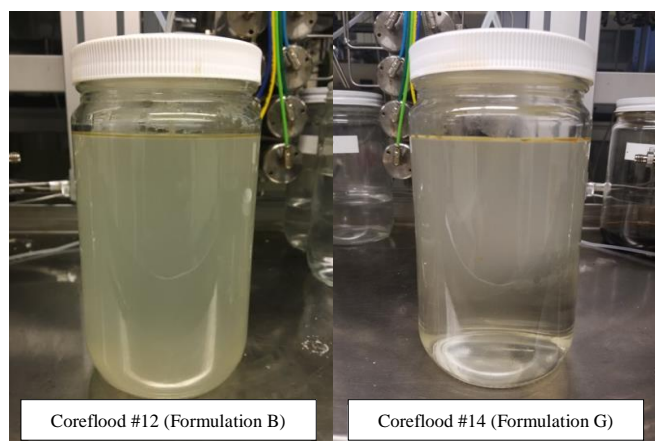


Figure 6. Effluent samples after injection of Formulation B (left) and Formulation G (right)

Table 3. Summary of two-phase corefloods. The estimated precision of the IF values is ± 0.02 .

Coreflood #	Oil	Formulation	IF (oil)
Coreflood 5	Kerosene	None (Control)	0.77
Coreflood 6	Kerosene	Formulation B	0.42
Coreflood 7	Kerosene	Formulation C	0.54
Coreflood 8	Kerosene	Formulation D	0.58
Coreflood 9	Kerosene	Formulation D	0.59
Coreflood 10	Kerosene	Formulation E	0.81
Coreflood 11	Shale Crude	None (Control)	0.69
Coreflood 12	Shale Crude	Formulation B	0.73
Coreflood 13	Shale Crude	Formulation F	0.75
Coreflood 14	Shale Crude	Formulation G	0.88

Conclusions

Using methods established in the literature, initial evaluations of flowback aids in brine and oil saturated cores showed few differences between initial and final injection permeabilities. It was noted that reproducibility was poor in these tests and that results were often misleading. The work from this initial survey led to the development of a test using a broken polymer to better simulate a slickwater fracturing fluid. Coreflood results from over a dozen tests show that using the polymer injection method described in this study resulted in a substantial drop in relative permeability in both one-phase and two-phase sandstone and limestone cores. This novel method was shown to be reproducible over separate coreflooding units and multiple runs.

After creating a baseline for describing damage, it was shown that flowback aids, including microemulsions and basic surfactant packages, could be evaluated and ranked based on their ability to remediate the polymer damage and improve oil regain permeabilities. One formulation in particular showed a 28% increase in the measured improvement factor used to determine an increase in oil permeability. Differences between field crude oil and surrogate oil were described, and these

results show the importance of using actual field oil to evaluate formulation performance. Future testing is planned to examine additional surfactant and microemulsion formulations and to correlate structure function properties with oil regain permeability.

Nomenclature

<i>FBA</i>	= Flowback Aid
<i>gpt</i>	= Gallons per thousand gallons
<i>HPAM</i>	= Hydrolyzed polyacrylamide
<i>IF</i>	= Improvement Factor
<i>PV</i>	= Pore Volume

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