LABORATORY ANALYSIS OF A NEW SAND CONSOLIDATION MATERIAL FOR OILFIELD APPLICATIONS

A Thesis

by

JOSEPH DANIEL FILBRANDT

Submitted to the Office of Graduate Studies of
Texas A&M University
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

December 2010

Major Subject: Petroleum Engineering

Laboratory Analysis of a New Sand Consolidation

Material for Oilfield Applications

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Approved by:

Chair of Committee,	Stephen A. Holditch
Committee Members,	Ding Zhu
	John A. Gladysz
Head of Department,	Stephen A. Holditch

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ABSTRACT

Laboratory Analysis of a New Sand Consolidation
Material for Oilfield Applications. (December 2010)
Joseph Daniel Filbrandt, B.S., Purdue University
Chair of Advisory Committee: Dr. Stephen A. Holditch

The production of sand can be a major issue in many young, unconsolidated sandstone formations where there is little to no cement holding the individual sand grains together. When such reservoirs are produced, quite often operators face problems with reduced well productivity and equipment failure. Because of these issues, the industry has developed numerous techniques in its effort to control formation sand production. Sand consolidation is one technology that has been studied and used since the 1940s. The theory behind sand consolidation technology is to place a liquid material which will create a grain to grain contact that will bind individual sand grains together. Most consolidation treatments contain a preflush to clean and wet the surface, the consolidating system to bind the sand grains and give residual strength, and, finally, an overflush to ensure the formation is still able to produce fluids. With the successful placement of this fluid, the sand grains will be locked in placed so that they will not be produced. The technology has gone through many phases of conception since the 1940s; however, most consolidation material that is pumped in the past has been based upon an epoxy or furan backbone.

While there are many technologies available, for the purpose of my research, the epoxy technology was experimentally investigated. The testing of the fluid involved investigating numerous additives to obtain the correct residual strength of the sample, as well as the necessary retained permeability. For the epoxy fluid, the optimal preflush, epoxy system and overflush formulations were determined after 250 checkout tests.

Based upon these tests, the fluid was optimized to its working time and UCS results. The optimal system included the addition of PA2 to the preflush, along with PA1 and an aromatic amine curing agent to the epoxy system. PA1 and PA2 are adhesion promoter additives which were deemed necessary as a result of the testing. This system was then tested further in a HP/HT cell. While there is still room for improvement with respect to retained permeability, the system still performs very well in terms of UCS.

DEDICATION

To my parents, Chris and John Filbrandt, I thank you for the great support you have always given me, and a model for how to live my life.

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I would also like to thank my friends and colleagues at Texas A&M University for the memories and experiences.

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Finally, but by no means least, thanks to my mother and father, Chris and John, and siblings Lilah, John, Jake, and Jordan, for their encouragement and love. Quad jacks, queen high.

NOMENCLATURE

cp	centipoise
BHT	Bottomhole Temperature
CA	Catalyst Amount
DSC	Differential Scanning Calorimetry
HEC	Hydroxyethyl Cellulose
HP/HT	High Pressure/High Temperature (Cell)
IR	Infrared (Scans)
MIC	(Texas A&M) Microscopy and Imaging Center
PA	Proprietary Additive
PV	Pore Volume(s)
SEM	Scanning Electron Microscope
UCS	Unconfined Compressive Strength
VES	Viscoelastic Surfactant

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I. INTRODUCTION: SAND CONTROL AND PARTICLE CONSOLIDATION AS A MEANS THEREOF

The production of sand can be a major issue in many young, unconsolidated sandstone formations where there is little to no cement holding the individual sand grains together. When such reservoirs are produced, quite often operators face problems with reduced well productivity and equipment failure because of formation sand production. Unlike the hydrocarbons being produced, the formation sand has no economic value. This sand is detrimental to the operation, as it has the ability to negatively affect the reliability of not only downhole completion components, but also surface production equipment. The production of sand can erode, settle inside, and damage the surface equipment. Some examples of downhole components include valves, tubular, and pumps. Conversely, examples of surface equipment include separators, surface lines, and the wellhead. Such damage may require the production to be shut down, so that maintenance can be performed to repair the damaged component. A reduction in production may adversely impact the economics of a project. (Arukhe et al. 2005)

In the case of offshore operations, the cost of formation sand disposal is another issue for an operating company. There are high disposal costs with this produced sand, not just in transportation, but also in the cleaning of this sand so that it can be transported and disposed of properly. One final adverse effect of sand production is the wellbore restrictions created when heavy formation sand deposits at the bottom of the well, rather than be produced. It is possible the sand in the wellbore can cause production to stop completely due to sand bridges forming in the tubulars across or above the producing interval. (Arukhe et al. 2005)

Because of these issues, the industry has developed numerous techniques in its effort to control formation sand production. Some of the options available for sand

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This thesis follows the style of *SPE Journal*.

control are a gravel pack treatment, frac-pack treatment, or sand consolidation treatment. Multiple tests are typically completed prior to selecting which sand retention method to use. Sand production prediction tests are often the first set of tests ran on a formation core sample. Formation properties obtained from these tests can be extensive as Young's Modulus, Poisson's Ratio, Unconfined Compressive Stress (UCS), Horizontal and Vertical Stresses, and Reservoir Pressure. (Qiu et al. 2006) As the name implies, these tests are utilized to predict whether sand production is expected to occur. One of the most basic of these tests is the use of existing data from offset wells, or on the basis of analogy. Both utilize experience as the basis for the likelihood of sand production. Some other tests, which are more complex, include the use of a Scanning Electron Microscope (SEM), Uniaxial and Triaxial Strength Tests, extended leak off tests (XLOT), and Sand Influx Tests (SIT). (Slayter et al. 2008) Each test varies in complexity, and as such, its results are given a greater degree of certainty. Surprisingly, Qiu et al. observed that sufficient data is quite often available to utilize a less complex analytical model, such as the information from offset wells. These models avoid the need for extensive laboratory data or input to obtain results, and are quite often just as reliable. (Qiu et al. 2006)

Gravel packing operations are necessary in formations where there is high heterogeneity, which invokes the need for an additional filter in the wellbore to prevent sand production. This additional filter is typically gravel/sand, or other man-made proppants. (Tiffin et al. 1998) The fundamentals behind placing gravel between the screen and formation is that the gravel is designed to have a smaller pore throat diameter, than the grain size diameter of the formation sand. This allows for a porous medium which would facilitate the production of hydrocarbons, while controlling that of formation sand. (Martins et al. 2009) For a frac-pack treatment, a fracture stimulation treatment is completed simultaneously with the gravel pack treatment explained previously. The fracture treatment is pumped to bypass any near wellbore damage which may be present in the well.

Sand consolidation is one technology that has been studied and used since the 1940s. (Harrisberger et al. 1971) The theory behind sand consolidation technology is to place a liquid material which will create a grain to grain contact that will bind individual sand grains together. With the successful placement of this fluid, the sand grains will be locked in placed so that they will not be produced. The technology has gone through many phases of conception since the 1940s; however, most consolidation material that was pumped in the past has been based upon an epoxy or furan backbone. (Parlar et al. 1998)

A closely related subject is the use of consolidating materials while placing proppant during a fracturing treatment, to restrict proppant flow back. The process is similar in that the proppant which has been placed inside an induced fracture should not be produced, and should stay inside the fracture to 'prop' the fracture open. For the purpose of my research, a new material to consolidate formation sand grains has been investigated. However, due to the similar nature of this new method to the more traditional methods, lessons learned in the past can be applied to this new method. (Villesca et al. 2010)

A new chemical consolidation treatment based upon sol-gel chemistry has recently been introduced to the oilfield community. (Genolet and Schmidt 2009) The chemistry is unlike the epoxy or furan resin previously mentioned. The application of sol-gel in the oilfield is still so new that very little data is available. The sol-gel chemistry has been utilized extensively in the preparation of glasses and ceramics. (Schmidt 1994) For this specific technology, the appropriate organic and inorganic molecular compounds have been studied extensively to obtain the desired material properties. (Genolet and Schmidt 2009) Initial systems documented by Schmidt et al. showed promising results, but lacked long term stability under hydrothermal conditions, which was attributed to the system being a silane derived sol-gel materials. (Genolet and Schmidt 2009; Schmidt 1994) Alterations to this technology were then made by Schmidt and Akarsu. These alterations included the introduction of titania into the organic sol-gel backbone. (Genolet and Schmidt 2009) After this change, the system can now be tailored to specific reservoir conditions by changing the fluid's viscosity, curing time, binder strength, downhole chemical stability, flexibility, permeability/porosity retention, as well as flexibility on flushing material (nitrogen, brine, or organic liquids). (Genolet and Schmidt 2009)

Genolet et al. describe the system as having three types of precursors:

1) Metal alkoxides – Sol-gel reacted to the oxides for mechanical strength and hydrolytic stability.
2) Polymerizable organic monomers – Provide sufficient flexibility by the formation of polymeric chains.
3) Organoalkoxy silanes with polymerizable groupings – Provide a link between the inorganic backbone and the polymeric chain, as well as adhesive forces to the sand/proppant surface. (Genolet and Schmidt 2009)

Unlike most epoxy or furan systems whose cure is a gradual progression to a hardened state once exposed to an increased temperature, the sol-gel fluid is a rather quick process once initiated by thermal radical initiators (peroxides and/or azo-compounds). When mixed in the appropriate type and concentration, the curing time can be adjusted accordingly to ensure proper time is allotted to mix and pump the fluid on location without fear of a pre-mature cure in equipment or the tubulars, which would be a very costly mistake. Finally, all other additives for this fluid have been developed and checked to avoid any adverse affect on the thermal initiator activity. These interactions include possible catalytic effects of both the well, and formation materials or fluids. (Genolet and Schmidt 2009) The sol-gel chemistry appears to be a fluid system which has the capability to perform very well in oilfield applications, but due to the lack of data available it was not investigated further in my research.

Recently, consolidation treatments have seen an increased application in low productivity, brown-field wells. Most operators use a consolidation treatment as a low cost method, to ensure maximum drainage of the reservoir while maintaining sand control measures. While there has been much improvement with understanding the technology and chemical interactions of the fluids, similar issues observed more than 10 years ago are still being seen today. These issues include the effective placement of the consolidating fluid, fluid stability at increased temperatures, and removal of the fluid from the pore spaces. (Nguyen and Jaripatke 2009; Parlar et al. 1998)

The stability and placement issues are inter-related. Epoxy resin systems work well at temperatures <250^{o}F, while the temperature application of furan resins is slightly higher, <300^{o}F. Most epoxy resin systems are internally catalyzed, and the nature of these catalysts is that their activity increases with temperature. So the perfect balance of catalyst level and bottomhole temperature (BHT) is necessary. The most common issues with the furan resin system are associated with the mixing of fluid stages. This system is externally catalyzed; normally by some sort of acid. A sufficient amount of fluid must separate these two stages to ensure there is no mixing of these chemicals prior to placement, as its reaction rate is extremely fast once contacted by the initiating fluid. For this reason, if either system starts to develop viscosity by either mechanism, the fluid will not be placed effectively. (Nguyen and Jaripatke 2009; Parlar et al. 1998)

Directly related to fluid placement is the insurance that the entire interval is treated. Due to ineffective placement mainly due to reservoir heterogeneity, most consolidation treatments are limited to less than ~20-25 feet. Aqueous based systems have been developed to assist with this issue. (Villesca et al. 2010) Aqueous based systems have the advantage over solvent based systems, in that nitrogen foam can be added to the treatment fluid and this will assist diversion.

If successfully placed, the fluid must also be removed from the pore spaces so as to not restrict production later on. The optimal treatment would be where the fluid left in the rock matrix is only at the grain to grain contact points. (Villesca et al. 2010) This is a very delicate balance between increased rock strength, measured by its unconfined compressive strength (UCS), and retained permeability. Too much flushing of the consolidating fluid may leave a comparatively weaker consolidated sample, while not enough flushing will lead to a more consolidated sample, but less retained permeability.

As mentioned, there has been much work to target these deficiencies, but there is still room for improvement on the fluids currently offered. As early as 1998, advancements in treatment design and a better knowledge of rock/fluid interactions have been documented, which have led to an understanding of the necessary steps for a successful consolidation treatment. Parlar et al. stated a minimum of 6 steps must be followed for a successful consolidation treatment. These same 6 steps continue to be the basis of consolidation treatments, although occasionally slight variations have been made in the application of the technology. The six steps are as follows: 1) acid pre-treatment, 2) surfactant preflush, 3) resin, 4) displacement fluid, 5) acid catalyst overflush, and 6) shut-in. (Parlar et al. 1998) These six steps are for a specific system which is externally catalyzed. For internally catalyzed systems, step 5, the acid catalyst, is not necessary. Other slight modifications can be made depending on the wettability of the reservoir, and if certain stages are not pumped. For example, if an acid pre-treatment is pumped, it is recommended to pump a two stage surfactant preflush. One of these stages is designed to be compatible with the preceding acid treatment, and a second which is compatible with the consolidation treatment to follow. Both of the treatments would contain the same amount of surfactant, on a per volume basis. If the acid treatment was not pumped for the case when the formation was clean sandstone, it would be possible to remove the first part of the surfactant preflush stage.

Other variations include adding an additional preflush stage in the event that the acid pretreatment was not pumped and the formation is oil wet. In this case, it has been proven useful to add a mutual solvent to the preflush to ensure the formation is water wet. Work was done as early as 1974 to show this use of a solvent is effective. (Brooks 1974) At the end of the day, the conditions vary for every reservoir, but the basic steps have been investigated extensively. The purpose of my research will not be to challenge these already established results; instead, I will be investigating to determine the best suitable chemicals to achieve predefined constraints. These constraints are outlined in Section II. While determining the most suitable chemical, it will also be determined the point within the treatment when a specific chemical additive should be introduced to the consolidation system.

II. EXPERIMENTAL APPROACH

2.1 Background

The scope of my research involves the testing of a new epoxy resin fluid as a consolidation fluid. The fluid will be investigated in two separate steps. The epoxy technology will be investigated as the fluid system is developed. Although there are numerous similar products on the market today, as my research a new epoxy resin system will created to remediate some of the deficiencies mentioned earlier which still today plague many of the systems offered. My research will involve the testing of multiple curing agents, solvents, and other additives of the system. There will be a learning curve involved in this process. The 3 main constraints of both treatment systems are the following:

1) Ensure a working time of 6-8 hrs at a viscosity of 10 cp or less,
2) Optimize the UCS to a minimum value of 1000 psi, and
3) Obtain a minimum retained permeability of 75% with respect to the pretreatment permeability value.

An additional constraint is placed on the latter two, in that the requirements must be obtained while working in a low temperature environment (130^{o}F) after 24 hours.

The working time is directly related to the amount of catalyst used in the system. The amount of catalyst must be optimized to ensure not only the proper working time, but also ensure the required UCS is obtained within 24 hours. The UCS will be a function of the increased strength that the system shows after being applied to an unconsolidated formation sample. The retained permeability is equally as important because sufficient permeability must remain after the treatment in order to produce hydrocarbons at economic flow rates.

Historically, treatment designs have been based upon pore volumes (PV), which then can be used to compute the volume of treatment fluids to pump. Pore volume is directly related to the porosity of a sample. Porosity is defined as the area within a sample which is able to contribute to flow of fluids. With this in mind, when treatments are designed, the amount of free space for a sample of a given length and depth of invasion into the formation with a specific porosity, the void space in the sample can be calculated. The volume calculated from these dimensions is referred to as 1 pore volume. For this reason, typically to completely saturate a sample, terminology such as pore volumes are used. The fluid will undergo testing to find its application over a wide range of temperatures (130-200^{o}F), so as to maximize UCS and retained permeability, while determining the optimal ratio of pore volumes to pump over the temperature range.

As previously mentioned, for the epoxy system there will be a learning curve. To differentiate between different epoxy systems, a naming system was developed. The first system tested will be called System A. Each time a major component such as the curing agent was changed, the next alphabetical letter was used as a name for the system. As an example, if the fluid changes dramatically, the next fluid system will be called System B. The initial screening will be conducted to determine the proper fluid stage sequence, curing agent, solvent system, and catalyst amounts. These tests will be carried out via syringe tests. Fig. 1 depicts the setup of these syringe tests. The usefulness of this test is to quantitatively determine the UCS value after the epoxy system has cured. As well, a qualitative test of flowability is possible to give an idea how easily fluids can be injected through the sample after its cured. Once the correct balance of additives are known, the system will be further analyzed in a High Pressure/High Temperature (HP/HT) cell. The testing in the HP/HT Cell is also known as a coreflood test. These terms can be used interchangeably. By completing the tests in the HP/HT cell, the retained permeability of the sample, which is as useful as a UCS measurement, can be recorded.

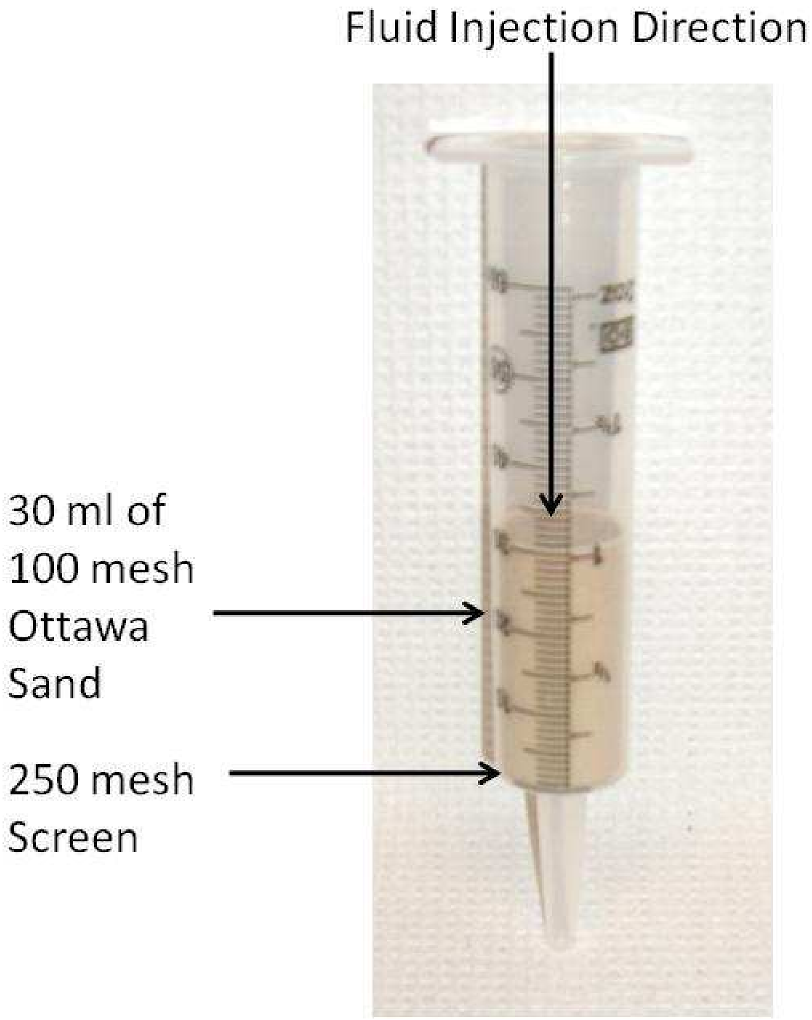


Fig. 1–Syringe Test Setup.

2.2 Syringe Test Setup

The following procedure details the steps that were followed for the 'preliminary' syringe tests. Due to ease and simplistic nature of the test, multiple samples could be prepared and investigated simultaneously. As shown in Fig. 1, a screen was placed at the bottom of the syringe, and then sand was packed on top of the screen. The sand utilized for these tests is the same as what was used in subsequent tests; 100 mesh Ottawa Sand.

A 3 inch sample was the standard sample size in the syringes. In the standard 60cc syringe shown in Fig. 1, this equates to approximately 30 cc of sand. With this in mind, the bulk density for 100 mesh sand is used to calculate the weight of 30 cc of sand.

$$\text{Bulk Density}_{\text{100 mesh sand}} = 100\, {}^{\text{lb}}\!/\!_{\text{ft}^3}\ x\ 453.592\, {}^{\text{grams}}\!/\!_{\text{lb}}\ x\ 1\, {}^{\text{ft}^3}\!/\!_{28316.8cc} = 1.60185\, {}^{\text{g}}\!/\!_{\text{cc}}$$

$$\text{Required Amount}_{\text{100 mesh sand}} = 1.60185\, {}^{\text{g}}\!/\!_{\text{cc}}\ x\ 30\text{cc} = 48.06\text{ g}$$

Following this calculation, since most resin systems are pumped as a function of the pore space/volume of the sample, it is necessary to calculate 1 pore volume.

$$\text{Porosity} = \left(1 - \frac{\text{Bulk Density}}{S.G.}\right) \text{x}\, 100$$

$$\text{S}.G._{\text{100 mesh sand}} = 2.65$$

$$\text{Porosity} = \left(1 - \frac{1.60185}{2.65}\right) x\, 100 = 39.6\%$$

$$1\,\text{pore volume of sample} = \frac{48.06\text{ g}}{1.60185\, {}^{\text{g}}\!/\!_{\text{cc}}}\ x\ 0.396 = 11.87\text{cc}$$

After the sand has been placed in the syringe, it was necessary to saturate the sample with either oil or brine fluid. For the oil environment, it was decided to use mineral oil. Mineral oil will demonstrate the consolidation fluid's performance in an oil environment. It is noted though that when this fluid is applied in an actual well, and if tested in a similar manner, produced fluids of the well should be used for saturating the sample to give as closely representative results as possible. For the case of the brine saturated sample, the fluid used was 3% KCl. For this step it was decided to use 5 PV of fluid to completely saturate the sample with the desired fluid. Using the above calculation, 59.3 cc of fluid was pumped through the sample.

Next, the consolidation fluid stages were then injected through the sample. While the composition of the preflush and curing system were varied throughout the investigation, it was decided that their volumes would be held constant at 3 PV. By being fixed, specific additives could be adjusted to yield the desired results without having to change more than one more at a time. Fixing the pore volume allowed for the iterations to be decreased substantially. On the other hand for the overflush stage, both its composition and volume were changed quite often to see the effect of its variability. As mentioned previously, the hopes of the investigation were to show as little reliance on overflush volumes.

Following the injection of the treatment stages, the samples were then placed in a convection oven for a given time at a specified temperature. For the samples the temperatures were either 130°F or 200°F, while the time was either 24 or 48 hours. As will be discussed later, it was quickly realized the conditions of 130°F and 24 hour curing time were the most demanding on the fluid system. At this point, it was decided to optimize the fluid for these conditions, and the system could be altered at a later time, since the fluid's cure profile would only be enhanced by a longer curing time or higher temperature application.

Once the predetermined cure time was reached, the sample was removed from the temperature bath to perform a qualitative 'flow-through test' with 3 PV of brine to obtain a preliminary estimate of regained permeability. The result was based strictly on the ease of injection of the flow-through test fluid, compared to the injection of the consolidation system. There were four categories of these injections:

1) E – The injection is as easy, if not easier than that for the consolidation treatment.
2) S/D – The injection is slightly more difficult than that for the consolidation treatment.
3) D – The injection is noticeably more difficult than the consolidation treatment.
4) N/A – The fluid was not able to be injected through the sample.

Again, these classifications are strictly qualitative, and an actual regained permeability test would be completed once the system appeared to give the required UCS values, as well as a post injection which was given an E or S/D rating for the flow-through test.

Following the flow-through test, the UCS of the sample was measured using a Tinius Olsen hydraulic press. Fig. 2 depicts the machine that was used for these tests. All syringe test samples were subjected to this tests, however as will later be discussed, not all samples were consolidated enough to be able to tests the samples. In most cases, these samples were still so unconsolidated that they appeared to simply be wet sand, with no apparent consolidation. In the results, samples which were too unconsolidated to test for its UCS will be represented as N/T, No Test.

Fig. 2–Tinius Olsen Hydraulic Press for Compressive Strength Test.

2.3 HP/HT Test Setup

Pictures of the setup are shown in Figs. 3 and 4. The diameter of the sand pack sample prepared in Figure 4 was 1 inch, and its length 3.25 inches. A 250 mesh screen was placed on the inlet and the outlet of the sample to ensure there was no loss of sand prior to consolidation. Following its preparation, the sample was sealed and secured in place. Next, the sample was loaded in the HP/HT Cell. The system allows for a confining pressure up to 10,000 psi once the sample is in place. A confining pressure of 1400 psi was applied to the sample. Sufficient time, normally 1 hour, was given to allow the sample to reach equilibrium with the applied confining pressure. While the sample was reaching equilibrium, the sample was saturated with 3% KCl brine fluid. The saturation stage also ensured that there was no residual air in the sample nor the flow lines.

The equipment setup for the testing was connected to a computer where the data was collected electronically. The pressure drop across the sample and the sample's temperature were recorded which allowed for the calculation of an average permeability over the entire injection period. Prior to measuring the sample's pre-treatment (baseline) permeability brine was pumped through the sample at 30 ml/min. This was observed to help clean the sand pack by removing out any residual fines in the system. Next, the rate was gradually increased from 2.5 ml/min to 30 ml/min so as to avoid irregular pressure responses. Multiple rate increases and decreases were pumped to gather as much data as possible, to give a good understanding of the initial permeability. Once the pressure drop leveled out and substantial data was obtained, the sample was heated to the test temperature.

Once at temperature, the fluid stages were injected through the sample. To avoid contamination of the pump with epoxy, a separate pump was utilized to pump the epoxy system. This system was primed and setup ahead of time to ensure accurate metering of the epoxy system. Once the treatment injection was complete, the sample was allowed to cure for 24 hours or the designated time.

After 24 hours the sample was removed, and all the lines cleansed to ensure a clean system for the post-treatment permeability test. While cleaning the system, the HP/HT cell was cooled to room temperature. Once the lines were cleaned, the sample was placed back in the HP/HT cell, and the confining pressure reapplied. Following the application of the confining pressure, the post-treatment permeability injection was performed. The same ramp as described earlier of multiple pumping rates was completed on the cores for multiple permeability measurements. The sample was then removed, and another core prepared to test. Lastly, the sample was tested for its UCS.

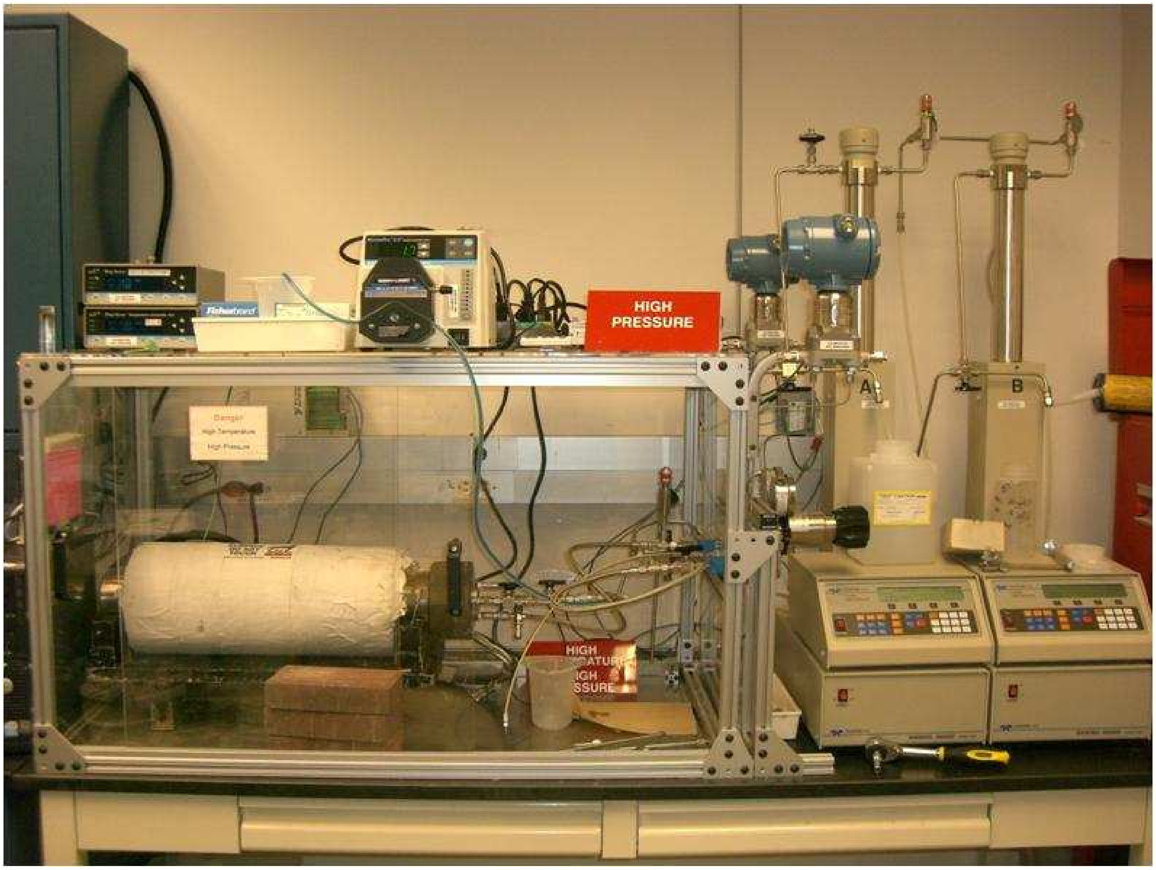


Fig. 3–Coreflood (HP/HT Cell) testing configuration Example 1.

Fig. 4– Coreflood (HP/HT Cell) testing configuration Example 2.

III. EXPERIMENTAL RESULTS

3.1 Syringe Test Results

For my research, the syringe test procedure describe above was used as an initial screening for the optimal fluid for the epoxy system. The system’s curing agent is an epoxy resin. The purpose of this testing was to determine the proper fluid stage sequence, curing agent, solvent system, and catalyst amounts. The naming of the system was previously discussed.

Prior to starting the syringe tests, the number of iterations necessary to find the optimum fluid was unknown. Based upon the results of each round, conclusions were made, and a way forward with the investigation was determined. By the end of the testing of this fluid, 250 individual syringe tests were completed over 14 separate rounds of syringe tests; each round ranging from 6 to 48 individual tests. Table 1 describes the main observations of each round of tests, as well as the path forward from these conclusions.

Table 1. Compiled Syringe Test Rounds Purposes.

Syringe Test Round #	Fluid System(s) Tested	Purpose
1	A	Test system’s performance at multiple overflush volumes, multiple temps, and multiple times.
2	A	Test 3 new catalysts to try to get a more rapid cure (24 hr), and not affected by brine overflush.
3	B	Test the new system with several overflush volumes, multiple temps, and multiple times.
4	B, C	Test 2 Hydrophobic & 2 Hydrophilic solvents to find system with best results.
5	B	Investigate high end of catalyst amounts to find upper limit.
6	B	Further investigate the low end of catalyst amounts to find the optimum. For fingering issues, change the overflush fluid to help.
7	B	Test the addition of an oil-wetting surfactant to the preflush and overflush fluids. Try another viscous overflush fluid.
8	B	Test 6 different flush fluids, including different combinations of brines, oil-wetting surfactant, and chelating agent.
9	B, C	Test effect of new additive (Proprietary Additive (PA) – PA1) in a new hydrophobic and current best hydrophilic systems.

Table 1 Continued.

Syringe Test Round #	Fluid System(s) Tested	Purpose
10	B, D	Further optimize the concentration of PA1 in the hydrophilic system, and in another new hydrophobic system.
11	B	Test influence of using mineral oil as preflush and overflush fluid.
12	B	Investigate effects of 4 new preflush systems, and additives (PA3 & PA4) in the epoxy system.
13	D	Test the effects of 4 new preflush systems, and additives (PA3 & PA4) in the previous best hydrophobic epoxy system.
14	B	Optimize the concentration of PA2 in the preflush, with respect to PA1 and PA4 in the hydrophilic epoxy system.

3.1.1 Syringe Test Round 1 Results

Based upon the given requirements of the fluid system, System A had a similar curing agent to some products on the market, but had a slightly different catalyst. The goal of the first round was to identify how the system's performance would change with respect to different overflush volumes. Both oil and brine saturated samples were tested in this round, as well as 4 separate overflush volumes. Each test was repeated 3 times to test the reproducibility of the results, 2 were brine saturated and the other oil saturated. The test grid for this round of testing can be seen in Table 2.

The results from the first round of testing are displayed in Table 3. A few different trends were observed from this first round of tests. Despite having a few samples which appeared to be slightly difficult or even very difficult to flow through after the treatment, 23 out of the 24 samples that were shut-in for 24 hours had a UCS value of 372 psi or lower. The results for the 48 hour shut-in samples looked more promising. For the 130 F - 48 hr, the lowest UCS value was 357 psi, while the highest was 4012 psi and the average for this group was 1177 psi. Despite being within the desired 1000 - 2000 psi range, there was not a clear consistent distribution. Likewise for the 200 F – 48 hr samples, the average was 1367 psi, with the lowest value of 501 psi, and the highest of 3691 psi. Again a good average, but the goal is to have all the samples above 1000 psi. These results were promising though, as there were quite a few samples in 48 hr cure time that had an easy or slightly difficult flow through tests. One trend that was observed was that the average UCS of oil saturated samples appeared to be slightly higher than that for the brine samples. The average of the brine saturated samples was 438 psi, while that of the oil samples was 1594 psi. There were a few that were extremely high, but for comparison purposes, it was clear the oil saturated samples performed better. Upon further analysis of the data, no clear trend could be developed for overflush volume or saturation vs. UCS results. For this reason, as well as the fact it was the first round of tests, it was decided to complete further analysis on the samples.

Differential scanning calorimetry (DSC) was performed on a few of the samples from this round. The results were consistent for the majority of the samples; the glass transition temperature was found to be in the range of 87°C to 95°C. These values were too low for the system used in this round, and it determined that the system was curing too slowly. Along with DSC, infrared (IR) scans were performed on the samples. These results showed that there was no evidence of the catalyst left in the sample. Based upon the results of the DSC tests and IR scans of the samples, the main conclusion was there was a strong possibility the catalyst was being lost during the overflush stage. As a result of the conclusion, for the next round of tests it was decided to slightly increase the catalyst level, and to switch to a higher activity/less water soluble alternative. The idea of changing the curing agent/system was a possibility, but it was decided to alter this one component at a time, in hopes of obtaining an optimum and then refining, if need be.

Table 2. Syringe Test Round 1 Test Grid.

Syringe Test Round 1 – 48 Total Samples			
24 hr Shut-In		**48 hr Shut-In**	
Temperature		**Temperature**	
130F	200F	130F	200F
3 PV of Brine Preflush 3 PV of System A 1 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 1 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 1 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 1 PV of Brine Overflush (2 Brine & 1 Oil Saturated)
3 PV of Brine Preflush 3 PV of System A 2 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 2 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 2 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 2 PV of Brine Overflush (2 Brine & 1 Oil Saturated)
3 PV of Brine Preflush 3 PV of System A 3 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 3 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 3 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 3 PV of Brine Overflush (2 Brine & 1 Oil Saturated)
3 PV of Brine Preflush 3 PV of System A 10 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 10 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 10 PV of Brine Overflush (2 Brine & 1 Oil Saturated)	3 PV of Brine Preflush 3 PV of System A 10 PV of Brine Overflush (2 Brine & 1 Oil Saturated)

Table 3. Syringe Test Round 1 Test Post Cure Injection Test and UCS Results.

		UCS (psi)				**Post Cure Flow Through Rating**			
Saturating Fluid	**Overflush**	**24 hr Shut-In**		**48 hr Shut-In**		**24 hr Shut-In**		**48 hr Shut-In**	
	(# of PV)	130°F	200°F	130°F	200°F	130°F	200°F	130°F	200°F
Brine	1 PV	N/T	90	830	501	S/D	D	D	E
Brine	1 PV	147	36	777	1009	D	S/D	S/D	E
Oil	1 PV	N/T	334	850	3691	D	E	S/D	D
Brine	2 PV	54	2	944	784	S/D	D	S/D	E
Brine	2 PV	87	29	1138	828	E	D	D	E
Oil	2 PV	N/T	372	1982	3468	D	E	D	E
Brine	3 PV	16	29	795	562	S/D	S/D	S/D	E
Brine	3 PV	98	16	959	606	S/D	D	S/D	E
Oil	3 PV	N/T	66	4012	2378	D	D	D	E
Brine	10 PV	267	73	558	753	D	S/D	S/D	E
Brine	10 PV	142	87	357	998	E	S/D	S/D	E
Oil	10 PV	109	1720	922	821	D	E	S/D	E

3.1.2 Syringe Test Round 2 Results

The tests for this round involved examining the effect of the glass transition temperature of the catalyst on the cure mechanism. The glass transition temperature is directly related to cure profile of the epoxy resin. Three different catalyst types were examined for this round; low (System 1), mid (System 2), and high glass transition temperature (System 3). Another system with the high glass transition temperature but with double the catalyst amount was also tested (System 4). Two samples were prepared with System 3 to once again examine reproducibility. Because of the fact that a 2 PV overflush gave the best UCS results on the previous round, it was decided to keep the overflush volume constant for this next round of tests at 2 PV. The preflush remained at 3 PV and both flush fluids were 3% KCl. The temperature and curing times for these samples were at 130°F for 24 hours, and 200°F for 48 hours. These two conditions represent both ends of the spectrum. It should be noted, that from this point on, unless otherwise noted, both brine and oil saturated samples were prepared for each test round. Finally, to compare the new system's performance to one system that is currently available on the market, two samples at each condition/saturation were prepared with the Schlumberger consolidation system (SandLOK®). The fluid formulation of the Schlumberger fluid is given in Table 4, and the test grid for this round is in Table 5.

The post cure flow through test results are shown in Table 6. The results improved greatly from the previous round in that only 2 of the 20 samples of the new system tested gave a difficult injection test. Comparatively, 2 of the 8 samples of the SandLOK® were difficult to inject through. Next, the UCS results from the second round are given in Table 6. The most striking observation in the results is the fact that none of the 24 hr samples of the new system were competent enough to test the UCS. Upon removal from the syringe, the samples were still entirely too unconsolidated to test. The lack of consolidation explains the improvement on the flow analysis for these samples. Upon inspection of the samples which were cured for 48 hours at 200°F, the samples were cured, however the average for both saturations were below the requirement; 236 psi for the brine saturated samples and 737 psi for the oil saturated samples. The results for the SandLOK® system showed very consistent UCS values, while at the same time meeting the criteria for the UCS value in all but one of the tests. Comparing these results and considering the lack of cure at the lower temperature, it was obvious there was room and need for improvement with the new system.

Based upon the results observed in this second round of tests, it was decided to change from the phenolic curing agent to a high functional amine curing agent. Although System A performed decent at the 200ºF and 48 hours cure time, it did not appear the system was able to provide the curing mechanics required at the lower temperature (130ºF) and low cure time of 24 hours. The amine curing agent provides a much faster curing chemistry as modeled in Fig. 5.

As shown in the fluid formulation for the SandLOK® system, a chelating agent and oil-wetting surfactant were introduced in the preflush and overflush stages. The addition of such chemicals is in line with conclusions reached by Parlar et al. and the effect that such additives may have on the curing performance. The superior UCS results of the commercial system were noted, but due to the possibility of masking effects from the curing agent or preflush or overflush stage, it was decided to first optimize the curing agent, and then focus on finding the best suitable additives for the two flushes.

Table 4. SandLOK® Fluid Formulation.

SandLOK ® Fluid Stages Formulation		
Preflush	Epoxy System	Overflush
Base Brine Oil-Wetting Surfactant Chelating Agent	Epoxy Resin Curing Agent Catalyst Diluent/Solvent	Base Brine Oil-Wetting Surfactant Chelating Agent

Table 5. Syringe Test Round 2 Test Grid.

Syringe Test Round 2 – 28 Total Samples			
Brine Saturated Samples		Oil Saturated Samples	
24 hr Shut-In (130°F)	48 hr Shut-In (200°F)	24 hr Shut-In (130°F)	48 hr Shut-In (200°F)
3 PV of Brine Preflush 3 PV of Low Tg System 2 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Low Tg System 2 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Low Tg System 2 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Low Tg System 2 PV of Brine Overflush
3 PV of Brine Preflush 3 PV of High Tg System 2 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of High Tg System 2 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of High Tg System 2 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of High Tg System 2 PV of Brine Overflush
3 PV of Brine Preflush 3 PV of Mid Tg System 2 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of Mid Tg System 2 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of Mid Tg System 2 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of Mid Tg System 2 PV of Brine Overflush (Repeat Twice)
3 PV of Brine Preflush 3 PV of High Tg – Double Catalyst System 2 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of High Tg – Double Catalyst System 2 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of High Tg – Double Catalyst System 2 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of High Tg – Double Catalyst System 2 PV of Brine Overflush
SandLOK System 3 PV of Preflush 3 PV of Epoxy System 3 PV of Overflush (Repeat Twice)	SandLOK System 3 PV of Preflush 3 PV of Epoxy System 3 PV of Overflush (Repeat Twice)	SandLOK System 3 PV of Preflush 3 PV of Epoxy System 3 PV of Overflush (Repeat Twice)	SandLOK System 3 PV of Preflush 3 PV of Epoxy System 3 PV of Overflush (Repeat Twice)

Table 6. Syringe Test Round 2 Post Cure Injection Test and UCS Results.

	UCS (psi)				**Post Cure Flow Through Rating**			
	24 hr Shut-In (130°F)		**48 hr Shut-In (200°F)**		**24 hr Shut-In (130°F)**		**48 hr Shut-In (200°F)**	
Binding Fluid	Brine	Oil	Brine	Oil	Brine	Oil	Brine	Oil
System 1	N/T	N/T	396	1365	S/D	S/D	S/D	S/D
System 2	N/T	N/T	137	716	E	S/D	E	S/D
System 3	N/T	N/T	71	526	S/D	E	S/D	S/D
System 3	N/T	N/T	404	861	E	S/D	S/D	D
System 4	N/T	N/T	172	368	S/D	S/D	E	D
SandLOCK	1388	798	1571	1328	E	D	S/D	S/D
SandLOCk	1471	1468	1747	1621	E	E	S/D	D

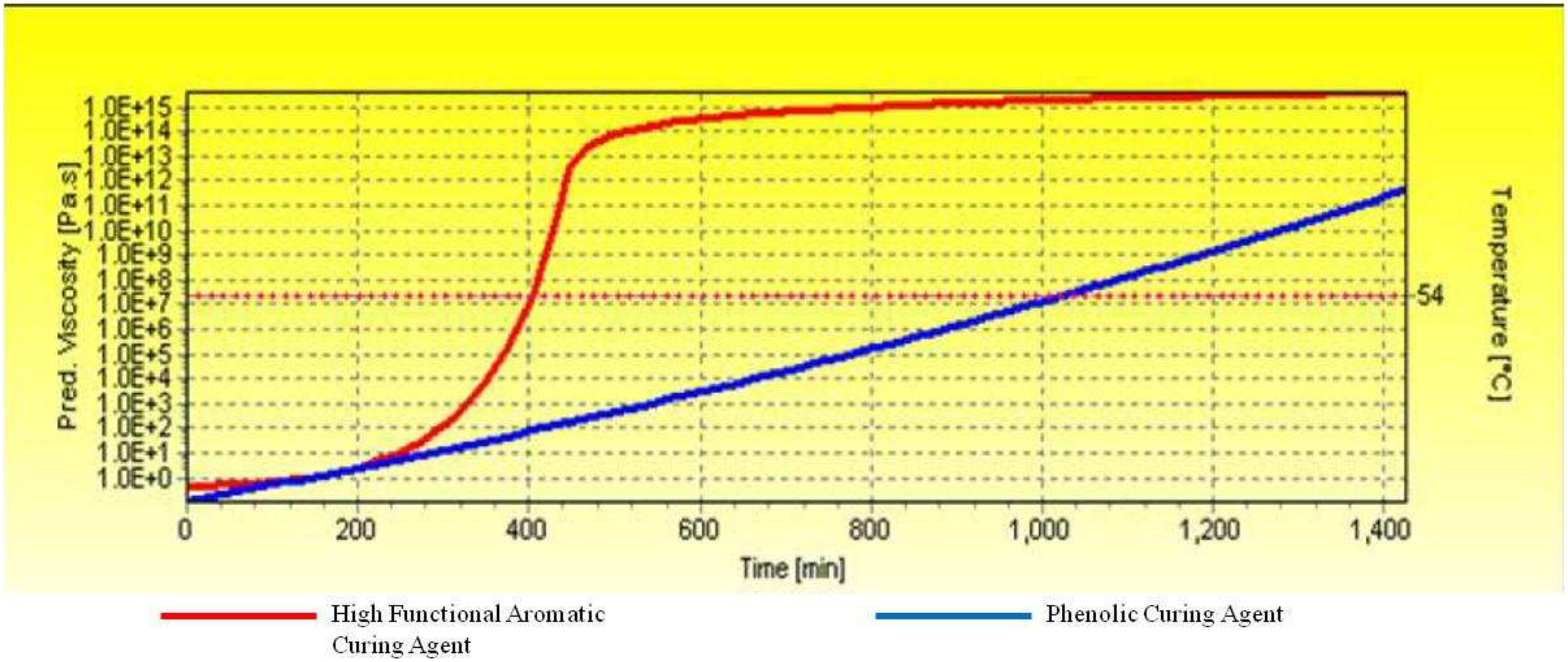


Fig. 5–Comparison of cure profile – Amine v. Phenolic.

3.1.3 Syringe Test Round 3 Results

In this round, the curing agent was changed, so the fluid system tested in this round was named System B. Due to the fact that a new curing agent in System B was being used for this round of tests, it was necessary to again investigate the effect of the amount of overflush volume. This round tested 3 different overflush volumes; 2, 3, and 10 pore volumes. Each test was completed twice at each of the given conditions. Also it had been observed in the tests to this point that the 24 hour cure time was the most demanding on the fluid with respect to curing time. For this reason, both the 130°F and 200°F samples were both cured for 24 hours. One final adjustment was the addition on a non-reactive dye to the fluid system. All the fluids to this point had been clear, and it was not possible to see what type of fluid movement occurred through the sand pack. The addition of this dye would allow for one more type of analysis to be completed on the sample, and give direction to improvements necessary for the system. From this point forward all of the fluid systems will have dye added to help with this analysis. The test grid for this round of tests is shown in Table 7.

The post cure injection tests results for this round of test are given in Table 8. The samples performed inadequately in this round. 22 out of the 24 samples had a difficult rating for flowing brine through the samples. Seeing these results it was expected the UCS values were going to be rather high, however this was not the case. Once again, there was trouble getting a stable/solid enough sample to be able to test its UCS at the low temperature and 24 hr cure time. The samples cured at 200°F did show that the new chemistry had the ability to obtain reasonable UCS results after a 24 hour cure. The brine saturated samples were below the desired UCS value, but the average of each of the oil samples was over 1000 psi. The conclusion was that the results were due to the incomplete removal of the original saturating fluid within the sample by the preflush, and the system having a greater affinity for oil rather than water. The results were nonetheless surprising considering the system considered is slightly hydrophilic. The addition of the dye proved to be useful in that different levels of fingering were observed in some of the samples while injecting the fluid, as well as when examining the samples once the UCS was performed and the samples were broken apart. The UCS results for this round are shown in Table 8.

Considering the results at the low temperature, but the fact there was a much better cure at the higher temperature, the conclusion was that the new curing agent was giving a good cure, but needed to be changed slightly. So it was decided to try other curing agents of the same chemical family prior to completing a large scale test for the next round. These samples were cured at 130°F for 24 hours. A successful formulation was found, and UCS values of 1886 and 50 psi were obtained for the brine and oil saturated samples, respectively. After seeing positive results, it was decided to continue to develop the aromatic amine chemistry of curing agents. For the next round, it was decided to compare several different solvent systems and their affinity for water; hydrophilic and hydrophobic solvents.

Table 7. Syringe Test Round 3 Test Grid.

Syringe Test Round 3 – 24 Total Samples			
Brine Saturated Samples		Oil Saturated Samples	
24 hr Shut-In (130°F)	48 hr Shut-In (200°F)	24 hr Shut-In (130°F)	48 hr Shut-In (200°F)
3 PV of Brine Preflush 3 PV of System B 2 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of System B 2 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of System B 2 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of System B 2 PV of Brine Overflush (Repeat Twice)
3 PV of Brine Preflush 3 PV of System B 3 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of System B 3 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of System B 3 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of System B 3 PV of Brine Overflush (Repeat Twice)
3 PV of Brine Preflush 3 PV of System B 10 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of System B 10 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of System B 10 PV of Brine Overflush (Repeat Twice)	3 PV of Brine Preflush 3 PV of System B 10 PV of Brine Overflush (Repeat Twice)

Table 8. Syringe Test Round 3 Post Cure Injection Test and UCS Results.

	UCS (psi)				**Post Cure Flow Through Rating**			
Overflush	**24 hr Shut-In (130°F)**		**24 hr Shut-In (200°F)**		**24 hr Shut-In (130°F)**		**24 hr Shut-In (200°F)**	
(# of PV)	Brine	Oil	Brine	Oil	Brine	Oil	Brine	Oil
2 PV	N/T	N/T	375	2750	D	D	D	D
2 PV	N/T	N/T	375	2909	D	D	D	D
3 PV	N/T	N/T	309	1410	D	D	D	D
3 PV	N/T	N/T	296	2032	D	D	D	D
10 PV	N/T	N/T	3	922	E	D	D	D
10 PV	N/T	N/T	10	1512	E	D	D	D

3.1.4 Syringe Test Round 4 Results

As mentioned previously, the goal of the testing for this round was to find a base solvent system that would yield the best results with System B. Based upon polarity, the solvent will show a greater affinity for oil or water. Non-polar solvents act hydrophobic, while polar solvent will behave more hydrophilic. Two of the systems tested in this round were hydrophobic, and the other two were more hydrophilic. The hydrophobic solvents compared in the round are Solvent Systems #3 & #4. The hydrophilic solvents examined are Solvent Systems #1 and #2. For this round of tests, the overflush volumes used were 1 and 3 pore volumes. The goal again was to observe the change in UCS and post cure injection flow based upon the amount of flush pumped. Also due to fact that the low temperature and 24 hr cure conditions were deemed as the hardest constraint for the system, as shown by the previous 3 rounds of testing, it was decided to only run tests at these conditions for this round. If the system performed as desired at the low temperature 24 hr cure conditions, previous results have shown that it will perform as good, if not better at the higher temperatures. Keeping in mind the system may eventually call for optimization of other components for the higher temperature, the more demanding curing conditions were applied unless otherwise noted. The test grid for this round of test can be seen in Table 9.

The results of the post flow through test appeared promising as the testing resulted in only 2 out of the 16 samples tested being given a difficult rating. Solvent system #1 performed very well, with 3 out of the 4 samples being given an easy rating. This was positive to see, especially for the case when 1 PV of overflush was pumped which is considered a small amount of fluid and still rating so low. Solvent system #3 also showed good results while having both of its 3 PV samples given an easy rating. The compiled results for the post cure injection tests and UCS results are given in Table 10. While none of the cores met the UCS requirement, a few conclusions were made from the results. Solvent system #1 appeared to give the most consistent results between the oil and brine systems. In fact it had the highest UCS value for all of the brine cases. While the 3 PV sample of system #1 was rather low, it was unclear if this was a representative UCS. While performing the tests, a small portion of the sample broke off, which the system called failure. This indeed is considered a failure, but the remaining portion of sample appeared to be much more consolidated as indicated by a much darker coloring left from the dye. However, due to how small the remaining portion of the sample left was, a second test was not able to be completed, as was done in other samples. This same event occurred on a sample from system #3, and when tested a second time the UCS values were noticeable larger. The two hydrophobic systems performed well in the oil saturated samples which makes sense due to their affinity.

Considering that solvent #1 gave the most consistent results for both oil and brine saturated samples, and the fact that it flowed easily after curing, it was decided for the next round of tests that it would be the solvent system of choice. There was still an obvious room for improvement on the UCS results, and for this reason it was decided to examine the effect of the amount of catalyst in the system. With an increased catalyst, the reaction may happen faster, and thus giving a better cure in the same amount of time. For the next round, it was decided to try to optimize the catalyst amount in the system.

Table 9. Syringe Test Round 4 Test Grid.

Syringe Test Round 4 – 16 Total Samples			
24 hr Shut-In (130°F)		24 hr Shut-In (130°F)	
Brine Saturated	Oil Saturated	Brine Saturated	Oil Saturated
3 PV of Brine Preflush 3 PV of Solvent System #1 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #1 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #1 1 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #1 1 PV of Brine Overflush
3 PV of Brine Preflush 3 PV of Solvent System #2 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #2 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #2 1 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #2 1 PV of Brine Overflush
3 PV of Brine Preflush 3 PV of Solvent System #3 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #3 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #3 1 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #3 1 PV of Brine Overflush
3 PV of Brine Preflush 3 PV of Solvent System #4 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #4 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #4 1 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Solvent System #4 1 PV of Brine Overflush

Table 10. Syringe Test Round 4 Post Cure Injection Test and UCS Results.

		UCS (psi)		**Post Cure Flow Through Rating**	
Solvent System	**Overflush**	**24 hr Shut-In (130°F)**		**24 hr Shut-In (130°F)**	
	(# of PV)	Brine	Oil	Brine	Oil
System #1	3 PV	48	247	E	S/D
System #1	1 PV	365	462	E	E
System #2	3 PV	44	83	S/D	E
System #2	1 PV	30	54	S/D	D
System #3	3 PV	63	277*	E	E
System #3	1 PV	101	547*	S/D	S/D
System #4	3 PV	N/T	614	S/D	D
System #4	1 PV	193	684	S/D	S/D

* Average of two tests on the sample

3.1.5 Syringe Test Round 5 Results

The purpose of the fifth round was to establish if additional catalyst increases the effectiveness of the cure. For this round of tests, two different amounts of catalysts were added to the system; CA1, which was the same amount as in the previous round, and two times this amount, CA2. It was decided to run the CA2 conditions at both 130°F and 200°F to see if an increased temperature would alter the curing agent's effectiveness. The test grid for this round of testing can be seen in Table 11.

The results of the post cure flow through tests are presented in Table 12. The CA1 system showed the best results with a slightly difficult rating for both the oil and brine samples. The other two systems performed better in the oil samples than the brine. The results were comparable for the UCS testing as well, with the CA1 system performing the best in both saturations. The oil saturated sample was within the desired range, but the water sample was once again well below the desired minimum. The UCS results are compiled in Table 12. The general trend in this round of samples was that most of the dye in the brine saturated samples was concentrated on the outer portion of the sample, and for the oil saturated samples the dye placement was pretty homogenous throughout the entire samples. Two possible explanations were given for these results, but the influence of each was not measured directly. The observations of the fluid's placement with in the sample were determined as a result of the rate at which the systems are pumped through the sample, as well as the viscosity of the original saturating fluid. The mineral oil used is more viscous than brine and is noticeably harder to displace while injecting the fluid system through the sample. Because of the increased viscosity, the displacement is more likely to be in a laminar flow regime, thus allowing increased contact with the entire sand pack, leading to the more homogeneous consolidated packs results. In an opposite manner, the brine is less viscous and fingers through the pack at a higher rate. The fingering of the fluid would lead to the results observed; namely a heterogeneous displacement with an outer layer of epoxy left behind.

Given the observed results, a few changes in the testing format were implemented for the next round of tests. With respect to the fingering observed in some of the samples, the conclusion was that this was most directly related to the rate at which the fluid is pushed through the sand pack. Up to this point, the fluid was just pushed through the sand pack without attempting to hold a constant rate for all the samples; normally as fast as possible. All indications are that injecting in such a manner resulted in fingering. To eliminate the rate as a possible cause of fingering, it was decided to try to inject all of the fluid stages at a consistent/constant rate. While the tests are being completed in a syringe and an exact rate measurement is not possible, it was decided to try to inject the samples at a rate as close to ~10 ml/min as possible. Secondly, the chemistry of the cure appeared to be working, and when the catalyst amount was increased, the UCS or ease of injection did not. So for the next round of tests, the low end amounts of catalyst were investigated to see if the CA1 catalyst amount was indeed the optimum. Finally, for the next round of tests, the viscosity effects of the displacing fluids and wettability modifying agents were investigated. When the preflush and overflush fluids of the SandLOK® fluid system were used in a previous round, the results and the fluid's appearance were more consistent. To see if the same consistent results were obtainable with System B, the SandLOK® flush systems were used in the next round of tests.

Table 11. Syringe Test Round 5 Test Grid.

Syringe Test Round 5 – 6 Total Samples			
24 hr Shut-In (130°F)		24 hr Shut-In (200°F)	
Brine Saturated	Oil Saturated	Brine Saturated	Oil Saturated
3 PV of Brine Preflush 3 PV of CA1 System 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of CA1 System 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of CA2System 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of CA2System 3 PV of Brine Overflush
3 PV of Brine Preflush 3 PV of CA2 System 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of CA2System 3 PV of Brine Overflush		

Table 12. Syringe Test Round 5 Post Cure Injection Test and UCS Results.

		UCS (psi)		**Post Cure Flow Through Rating**	
Catalyst System	**Temperature**	**24 hr Shut-In**		**24 hr Shut-In**	
	(°F)	Brine	Oil	Brine	Oil
CA1	130	358	1117	S/D	S/D
CA2	130	266	641	D	S/D
CA2	200	279	751	D	S/D

3.1.6 Syringe Test Round 6 Results

The influence of the overflush fluid’s composition and viscosity was tested in this round due to the fingering observed in the previous rounds. The first overflush fluid was the same as used for the current SandLOK® system. The formulation was given previously in Table 4. The overflush fluid has very little viscosity to it, but it incorporates a chelating agent and oil-wetting surfactant within the system. The other overflush fluid was selected because of its viscosity. The fluid was formulated using a viscoelastic surfactant (VES). The viscosity target for this system was 25 cp, which was more than double that of the epoxy system. The VES overflush fluid’s formulation is given in Table 13. Finally, for each flush system, 3 separate epoxy systems were tested where the catalyst amounts examined were CA1 (from previous rounds), CA3 and CA4. The test grid for this round is given in Table 14. The curing agent, an aromatic amine, as well as the 3% KCl preflush were kept the same as the previous two tests.

During the injection of the overflush stages, the fluid appeared to flow through the samples much more evenly. The samples flushed with the SandLOK® system appeared to have more residual epoxy left in the matrix than those flushed with the VES system. After observing the residual epoxy in the system while injecting the fluids prior to cure, the results of the post cure injection analysis were very surprising. As can be seen in Table 15, all of the brine saturated samples were completely plugged and were not able to be injected through. The oil saturated samples yielded better results. While fluids could be injected through the oil saturated samples, all of them received a difficult rating. The dye placement appeared to be as noticed previously, more evenly distributed in the samples with the SandLOK® flush system. These observations were also supported by the UCS results shown in Table 15. For both the brine and oil saturated samples, the UCS results were considerably higher in all of the cases where the SandLOK® flush system was utilized. While the CA3 system yielded very similar results, it was decided the CA1 catalyst system once again was the system of choice. This system had a consistently higher oil saturated sample UCS, as well as a UCS result for the brine sample which was very close to that for the CA3 system.

Since the desired UCS results were still not being observed consistently, the decision was to have another round of tests to investigate the effects of the preflush and overflush fluids on the UCS and post cure injection. The fluids utilized for the next round of tests would be very closely related to the suggestions/improvements listed from Parlar et al. More specifically, the introduction of an oil-wetting surfactant in the preflush stages and the use of a mutual solvent for oil saturated samples. Previously, DSC and IR analysis was completed on the samples to try to explain results. Some of the results obtained to date appear to be able to be explained by observation of the sample with the naked eye. However, it was decided to add one more analysis to ensure the diagnosis/explanations being given were indeed correct. For this reason, it was decided to examine as many as possible of the cured samples of any subsequent tests utilizing a Scanning Electron Microscope (SEM). All SEM analysis was completed at the Texas A&M Microscopy and Imaging Center (MIC).

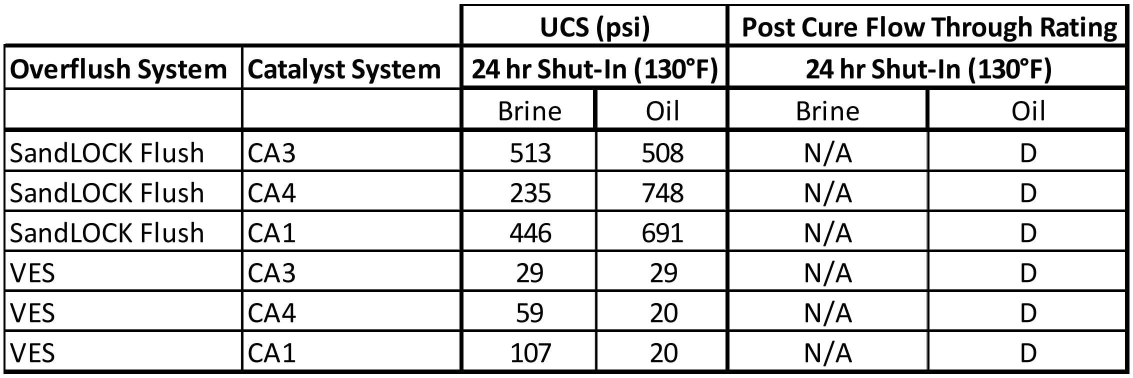
Table 13. Syringe Test Round 6 Viscoelastic Surfactant Fluid Formulation.

VES Overflush Formulation
Base Brine VES Chemical

Table 14. Syringe Test Round 6 Test Grid.

Syringe Test Round 6 – 12 Total Samples	
24 hr Shut-In (130°F)	
Brine Saturated	Oil Saturated
3 PV of Brine Preflush 3 PV of CA3 System 3 PV of SandLOK ® Overflush	3 PV of Brine Preflush 3 PV of CA3 System 3 PV of SandLOK ® Overflush
3 PV of Brine Preflush 3 PV of CA4 System 3 PV of SandLOK ® Overflush	3 PV of Brine Preflush 3 PV of CA4 System 3 PV of SandLOK ® Overflush
3 PV of Brine Preflush 3 PV of CA1 System 3 PV of SandLOK ® Overflush	3 PV of Brine Preflush 3 PV of CA1 System 3 PV of SandLOK ® Overflush
3 PV of Brine Preflush 3 PV of CA3 System 3 PV of VES Overflush	3 PV of Brine Preflush 3 PV of CA3 System 3 PV of VES Overflush
3 PV of Brine Preflush 3 PV of CA4 System 3 PV of VES Overflush	3 PV of Brine Preflush 3 PV of CA4 System 3 PV of VES Overflush
3 PV of Brine Preflush 3 PV of CA1 System 3 PV of VES Overflush	3 PV of Brine Preflush 3 PV of CA1 System 3 PV of VES Overflush

Table 15. Syringe Test Round 6 Post Cure Injection Test and UCS Results.



		UCS (psi)		**Post Cure Flow Through Rating**	
Overflush System	**Catalyst System**	**24 hr Shut-In (130°F)**		**24 hr Shut-In (130°F)**	
		Brine	Oil	Brine	Oil
SandLOCK Flush	CA3	513	508	N/A	D
SandLOCK Flush	CA4	235	748	N/A	D
SandLOCK Flush	CA1	446	691	N/A	D
VES	CA3	29	29	N/A	D
VES	CA4	59	20	N/A	D
VES	CA1	107	20	N/A	D

3.1.7 Syringe Test Round 7 Results

For the case of the sixth round, the surfactant and VES components were only introduced in the overflush stage. In the seventh round, it was decided to follow the suggestions of Parlar et al, and introduce these chemicals in the preflush stage as well. The effects of a viscous flush would be investigated once again, except using a slightly different fluid system. The composition of the preflush and overflush systems are given in Table 16, while the test grid for the seventh round is given in Table 17. The baseline of a brine preflush and overflush had been previously established in the fifth round, with results of 358 and 1117 psi for brine and oil saturated samples respectively. To observe the effect of the addition of an oil-wetting surfactant, two sets of tests were conducted where the only chemical additive to the base brine was the oil-wetting surfactant. Certain resin systems on the market incorporate pumping a mutual solvent to change the wettability back to water-wet for production purposes if an oil-wetting surfactant is used in the preflush stage. For this reason, the second set of tests kept the oil-wetting surfactant preflush, but the overflush was a viscous overflush comprised of hydroxyethyl cellulose (HEC) and a mutual solvent. By introducing a viscous component to the overflush, this would also allow the examination of how viscosity is affecting the curing mechanisms. Like the previous round of tests, the viscosity target for this flush stage was 25 cp at 100 sec^{-1}. For the last pair of tests, the preflush was changed back to brine, and the post flush fluid was the same as the previous test; HEC with mutual solvent. This test would help test to see the effect of the addition of the oil-wetting surfactant to the preflush. For the third set of tests, one additional pre-preflush stage was injected for only the oil saturated samples. Parlar et al. suggested the introduction of a mutual solvent to change the wettability of an oil saturated reservoir prior to treatment. As a result of these conclusions, a pre-preflush stage was pumped in the last two oil saturated samples which composed of brine and a mutual solvent.

The post cure flow through test once again gave suboptimal results. None of the samples received a rating of easy and there was an even distribution of slightly difficult and difficult rated samples. These results are given in Table 18. The UCS results were also once again lower than expected, considering the post cure flow test results. Two trends were observed. It appeared the introduction of the oil-wetting surfactant had no significant increase or decrease with respect to the simple KCl preflush and overflush; 358 without surfactant, 323 and 355 psi with surfactant. The oil saturated case had a significant decrease for the same set of tests. For the same conditions, the oil saturated sample dropped from 1117 psi to 18 and 0 psi. The lack of improvement from the new fluid systems were very surprising considering the fact that once the core was tested and investigated, a very homogeneous amount of dye was observed in the samples. In previous samples, good tests results were normally observed for these cases. The other trend which appeared was that no matter the preflush, the systems with the viscous flush which included a mutual solvent, the UCS values recorded were minimal or nothing. The low UCS results coupled with the fact that very little dye appeared in the majority of these samples lead to the conclusion that the mutual solvent was stripping away the curing agent.

The results of this round were very surprising considering the results given in previous rounds. The reason behind the drastic change in results was investigated further. The first and probably the most important observation was that while the samples were put in the convection oven, the heating system had tripped and shut off. The lack of heating of the samples explains a few of the lower UCS values. To further analyze the samples, SEM was utilized to help explain the results. Fig. 6a is a SEM photograph of Sample 1, and Figs. 6b-g of Samples 4, 7, 9, 10, 11, 12, respectively As hoped, there were two very distinctive observations made from these pictures from the SEM. Samples 1 through 8 all had an oil-wetting surfactant in the preflush, while 9 through 12 did not. In most of the pictures from the first group, there appears to be a presence of an emulsion on the surface of the sand grains. It was concluded, the emulsion was the result of the oil-wetting surfactant since these 8 samples only shared that one component in common. Furthermore, the emulsion tendency/amount observed of the brine saturated samples appeared to be greater. In some photos, the emulsion seemed to completely cover the sand grains of the brine saturated samples, while for the oil samples the emulsion appeared to be small droplets on the sand grains. To support the conclusions reached in the SEM photos, a bottle test was conducted to test for an emulsion with the epoxy system. The bottle test of the epoxy system plus the oil-wetting surfactant in brine is shown in Fig. 7. The oil-wetting surfactant in 3% KCl appeared to form an emulsion. The cloudiness observed in the bottle tests, combined with the pictures from the SEM helped conclude the emulsion in the SEM photos was indeed due to the oil-wetting surfactant and KCl preflush. The second observation made from the SEM photos was that while there was evidence of the epoxy in the SEM photos in the samples, in many cases the amount was minimal. In some cases there was evidence of epoxy throughout the sample, but it appears as small amounts, not binding sand grains together.

Because of the fact that the heating oven had shut off, it was decided to complete another round of tests in which the effect of an oil-wetting surfactant was examined. Although there was evidence of an emulsion, the extent to which it contributed the low UCS values was unknown. It was also concluded that the addition of a viscosity promoting additive to the overflush did not lead to additional strength of the sample. This observation does not mean the viscosity effect is detrimental, as previous studies have shown its obvious positive influence; it simply shows that the additives utilized in these tests gave no additional strength.

Table 16. Syringe Test Round 7 Fluid Formulations.

Preflush Systems	Overflush Systems
System 1 Base Brine Oil-wetting Surfactant	System 1 Base Brine Oil-wetting Surfactant
System 2 Base Brine	System 2 Base Brine Mutual Solvent Gelling Agent (HEC)

Table 17. Syringe Test Round 7 Test Grid.

Syringe Test Round 7 – 12 Total Samples	
24 hr Shut-In (130°F)	
Brine Saturated	Oil Saturated
3 PV of Preflush System #1 3 PV of Epoxy System 3 PV of Overflush System #1	3 PV of Preflush System #1 3 PV of Epoxy System 3 PV of Overflush System #1
3 PV of Preflush System #1 3 PV of Epoxy System 3 PV of Overflush System #1	3 PV of Preflush System #1 3 PV of Epoxy System 3 PV of Overflush System #1
3 PV of Preflush System #1 3 PV of Epoxy System 3 PV of Overflush System #2	3 PV of Preflush System #1 3 PV of Epoxy System 3 PV of Overflush System #2
3 PV of Preflush System #1 3 PV of Epoxy System 3 PV of Overflush System #2	3 PV of Preflush System #1 3 PV of Epoxy System 3 PV of Overflush System #2
3 PV of Preflush System #2 3 PV of Epoxy System 3 PV of Overflush System #2	3 PV of Preflush System #2 3 PV of Epoxy System 3 PV of Overflush System #2
3 PV of Preflush System #2 3 PV of Epoxy System 3 PV of Overflush System #2	3 PV of Preflush System #2 3 PV of Epoxy System 3 PV of Overflush System #2

Table 18. Syringe Test Round 7 Post Cure Injection Test and UCS Results.

		UCS (psi)		**Post Cure Flow Through Rating**	
Preflush System	**Overflush System**	**24 hr Shut-In (130°F)**		**24 hr Shut-In (130°F)**	
		Brine	Oil	Brine	Oil
System 1	System 1	323	18	D	S/D
System 1	System 1	355	0	S/D	S/D
System 1	System 2	N/A	37	S/D	D
System 1	System 2	N/A	21	S/D	D
System 2	System 2	0	108	D	D
System 2	System 2	21	137	S/D	D

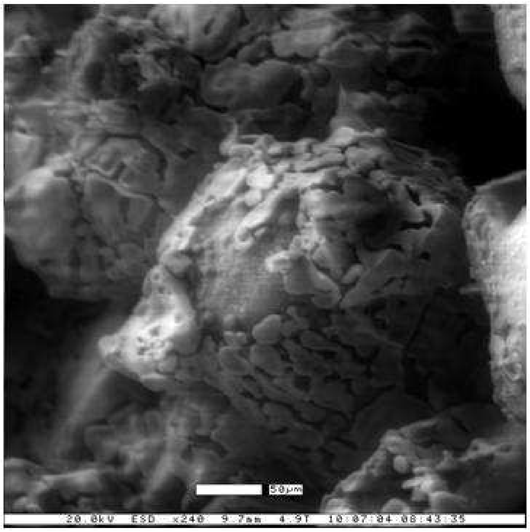


Fig. 6a–Round 7 Sample 1.

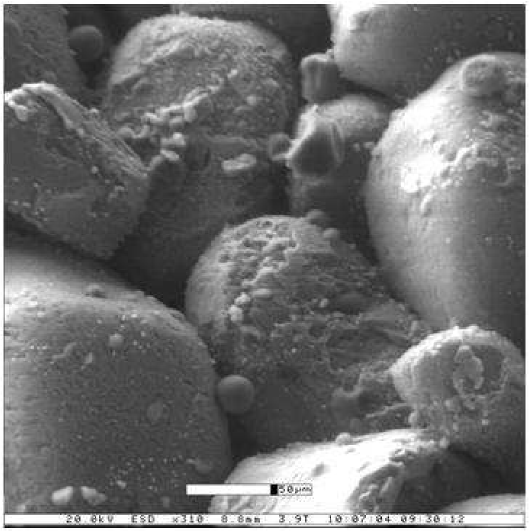


Fig. 6b–Round 7 Sample 4.

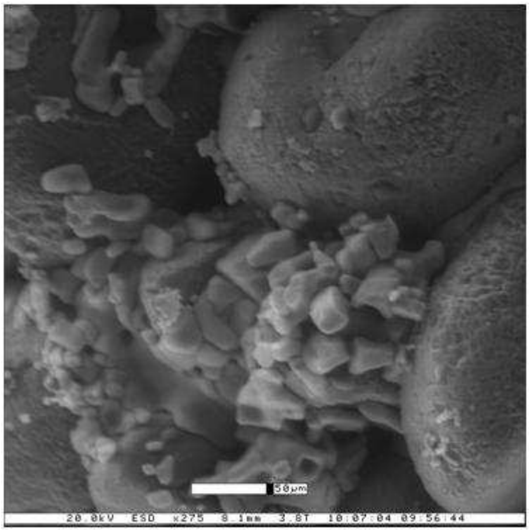


Fig. 6c–Round 7 Sample 7.

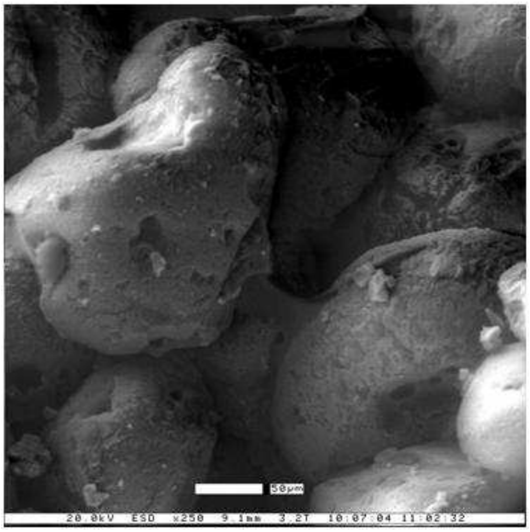


Fig. 6d–Round 7 Sample 9.

Figs. 6–Round 7 Syringe Test SEM Photos.

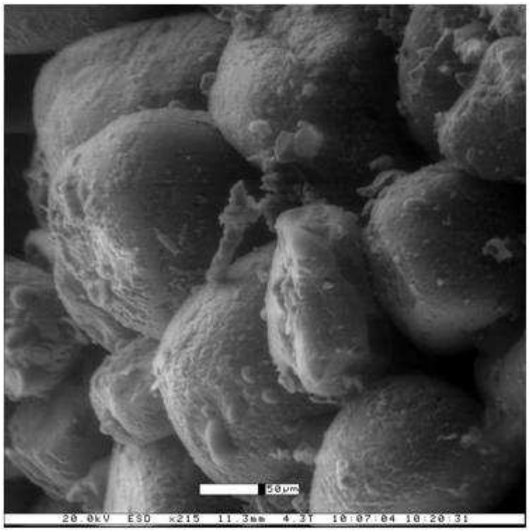


Fig. 6e–Round 7 Sample 10.

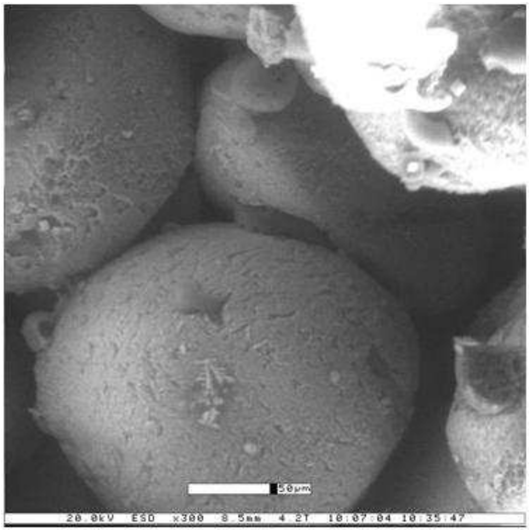


Fig. 6f–Round 7 Sample 11.

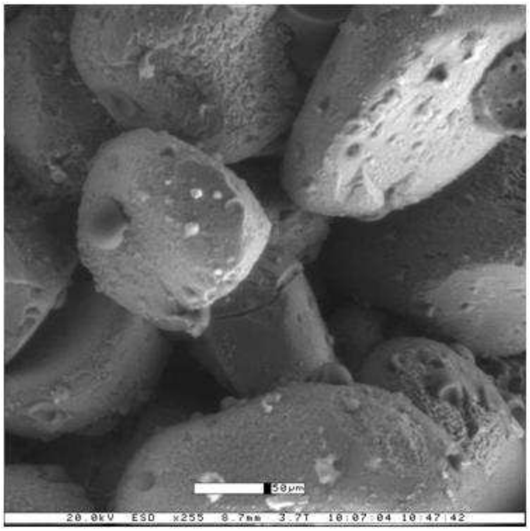


Fig. 6g–Round 7 Sample 12.

Figs. 6–Continued.

Fig. 7–Oil-Wetting Surfactant and Brine Emulsion Test.

3.1.1 Syringe Test Round 8 Results

The fluid formulations and test grid for this round of testing are given in Table 19 and 20, respectively. The purpose of this round was to investigate the effect of the base brine used when mixed with the oil-wetting surfactant, chelating agent, or the combination of the two in the preflush and overflush fluids. Two common base brines in this type of oilfield chemistry are potassium chloride and ammonium chloride, the latter which was used previously as the base brine for the SandLOK® system preflush and postflush. Since previously acceptable results were obtained with System B in an oil saturated sample with the brine preflush and overflush, this 'standard' was added to this round of tests. Besides being a standard, by testing the sample again it would also show if the results are reproducible.

The flow results improved slightly from previous rounds with more than half of the samples receiving a slightly difficult rating. Similar to the previous round, all the samples when the oil-wetting surfactant was in the flush package, the flow was slightly difficult. While the system may be forming an emulsion as established in the previous round, this emulsion does not appear to hurt the permeability of the core after cure. Unlike in previous tests, the 'standard' sample was given a difficult rating. The injection through the sample was much more difficult than previously observed. The results of the post cure injection tests are listed in Table 21. With respect to the flow through tests, it appeared the ability to flow through the sample in the case of the oil-wetting surfactant didn't appear to depend on the base brine. Conversely, when the chelating agent was used by itself in the base brine, the post flow results were much favorable with the ammonium chloride base brine. The results of the UCS tests were all slightly different, but they were all very close in magnitude. Although the UCS results were not the highest for the round, the potassium chloride with the oil-wetting surfactant and chelating agent gave the most consistent results the oil and brine saturated samples. Much like previous rounds, the oil saturated samples yielded much higher UCS values. While the post cure permeability retention wasn't the same as previous results for the 'standard,' the UCS results were very close to those obtained previously. For the examination of the dye placement within the samples after the UCS tests, in general as a set of tests, they all appeared to have a much more homogenous appearance than previous rounds of testing. A few samples were striated, but overall very good. It was also noted that the samples that did have internal striation, appeared to fail closer to where the striations occurred, rather than the homogeneous portion of the rock. It's assumed with less dye placement there is less of the binding agent in place, which would result in a weaker sample.

As a result of the analysis completed on the samples from the eighth round, the following conditions were determined for the next round of testing. From the sample's UCS results and the fact that the KCl brine sample repeatedly obtained a higher UCS value, it was decided with the current cure chemistry of System B, there was no additional benefit of adding a chelating agents, oil-wetting surfactant, or a combination of the two to either of the brines tested. Again, this is specific for the two chemicals of each tested, but it appeared the UCS values only decreased when these chemicals were introduced into the system. Secondly, as noted in the discussion of the SEM photos in the previous round, there still appeared to be an issue with the sand grains adhering to each other. For this reason, it was decided to investigate the effects of the concentration of different additives to increase the adhesion of the epoxy to the sand grains. Finally, the effects of the hydrophobic and hydrophilic solvents had been previously investigated, however not a hydrophobic or hydrophilic curing agent. For this reason, this would be examined in the next round of tests.

Table 19. Syringe Test Round 8 Fluid Formulations.

Preflush/Overflush Systems
System 1 Base Brine #1 Oil-wetting Surfactant
System 2 Base Brine #2 Oil-wetting Surfactant
System 3 Base Brine #1 Oil-wetting Surfactant Chelating Agent
System 4 Base Brine #2 Chelating Agent
System 5 Base Brine #1 Chelating Agent
System 6 Base Brine #1

Table 20. Syringe Test Round 8 Test Grid.

Syringe Test Round 8 – 11 Total Samples	
24 hr Shut-In (130°F)	
Brine Saturated	Oil Saturated
3 PV of Flush System #1 3 PV of Epoxy System 3 PV of Flush System #1	3 PV of Flush System #1 3 PV of Epoxy System 3 PV of Flush System #1
3 PV of Flush System #2 3 PV of Epoxy System 3 PV of Flush System #2	3 PV of Flush System #2 3 PV of Epoxy System 3 PV of Flush System #2
3 PV of Flush System #3 3 PV of Epoxy System 3 PV of Flush System #3	3 PV of Flush System #3 3 PV of Epoxy System 3 PV of Flush System #3
3 PV of Flush System #4 3 PV of Epoxy System 3 PV of Flush System #4	3 PV of Flush System #4 3 PV of Epoxy System 3 PV of Flush System #4
3 PV of Flush System #5 3 PV of Epoxy System 3 PV of Flush System #5	3 PV of Flush System #5 3 PV of Epoxy System 3 PV of Flush System #5
	3 PV of Flush System #6 3 PV of Epoxy System 3 PV of Flush System #6

Table 21. Syringe Test Round 8 Post Cure Injection Test and UCS Results.

		UCS (psi)		**Post Cure Flow Through Rating**	
Preflush System	**Overflush System**	**24 hr Shut-In (130°F)**		**24 hr Shut-In (130°F)**	
		Brine	Oil	Brine	Oil
System 1	System 1	387	463	S/D	S/D
System 2	System 2	473	541	S/D	S/D
System 3	System 3	423	453	S/D	S/D
System 4	System 4	485	571	E	S/D
System 5	System 5	400	783	D	D
System 6	System 6		995		VD

3.1.9 Syringe Test Round 9 Results

As previously mentioned, the main purpose for the ninth round of tests was to investigate the effects of an introducing a proprietary additive (PA1) for the epoxy adhesion issue noted by the SEM photos and the use of a hydrophobic curing agent in the epoxy system. Much like the tests completed to optimize the amount of catalyst; multiple concentrations of the additive were added to the system to determine if there was a maximum amount where the additive was non-beneficial. While the injection tests have seen gradual improvement, the desire is to hopefully have a system that will allow for a very easy flow once the system has cured. Although the actual retained permeability has not been measured yet, it's unlikely a good retained permeability would be observed in a sample where it's difficult to flow through after the cure. With this in mind, a hydrophobic curing agent system, C, was suggested; hoping because of its lack of affinity for water it would allow for a good post cure flow. Also, since deciding on the 3% KCl flush package, the effects of the mutual solvent pre-preflush had not been investigated. For the samples mentioned in this round so far, an oil saturated sample was repeated, as to examine the mutual solvent's benefit, or lack thereof. Finally, for all samples, a duplicate was completed in which an overflush was not pumped. The purpose of not injecting an overflush was to determine the maximum UCS obtainable from the system. The the fluid formulations and test grid for this ninth round are listed in Tables 22 and 23, respectively.

Similar flow results were observed for the hydrophilic solvent system, even at different concentrations of PA1. As desired, they hydrophobic did have a few samples that appeared to flow very easily in the post cure injection test. As suspected, in the systems where an overflush stage was not pumped it was not possible to pump any fluid through the sample after it had been cured. This was true for almost all the samples without an overflush. One of the systems with the hydrophobic curing agent without an overflush flowed very easily. The mutual solvent pre-preflush did not seem to benefit the post cure flow, as for all applicable samples the rating was the same as the sample without the mutual solvent preflush. For one of the hydrophobic system samples, the flow did switch from easy (without mutual solvent) to difficult (with mutual solvent). See Table 24 for the complete list of results.

There was an obvious correlation between the concentrations PA1.1 and PA1.2 of the new additive to the UCS results obtained for the samples. The nomenclature of the naming of the additive is described by the PA (Proprietary Additive), the first number stating the number of this type of additive used, and the second number being related to the concentration of that additive. Eight pairs of samples were prepared in which the only difference between the two samples was the concentration of the new additive in the binding system. For 7 out of the 8 pairs, there was an increased UCS value with increasing additive concentration. In one case the UCS value was doubled. However, in most cases the increase was still a noticeable improvement. For the samples with the hydrophobic curing agent, System C, and no overflush pumped, the UCS increased as expected. However, for the case of the hydrophilic system, the UCS was very low. Inspecting the samples after failure, the samples appeared to be one large piece but easily deformed. The lack of strength and ability to deform was attributed to possibly too much residual resin left in the pore spaces, which didn’t allow for a complete cure in these samples. With respect to the addition of a mutual solvent pre-preflush stage, there appeared to be no benefit of adding it to the hydrophilic system; it actually lowered the UCS in most cases. The increases were not substantial for the cases of the hydrophobic system, but it did not decrease the results in any of the samples. The compiled results from this round are given in Table 24. Finally, since there was a change of curing agents, two additional tests for only oil saturated samples was completed. This involved pumping the combination of an oil-wetting surfactant and chelating agent flush system. Both samples flowed very easily, but this was simply due to the fact there was no type of consolidation of the sample.

SEM photos were taken of only a few samples from this round of testing. Some of those photos are given in Figs. 8a-d. These pictures are of the hydrophilic system samples where there was not an overflush stage pumped. It’s quite clear to see just how plugged the pore throats are if an overflush stage isn’t pumped. While this does not explain the UCS results, it most certainly explains the reasoning for the samples not being able to be injected through after the samples cured. SEM photos to observe the effect of the new additive PA1 were taken in the next round, and will be discussed during its results.

Although promising results were obtained from the hydrophobic system in this round, there were issues keeping the system as one continuous phase while it was formulated. For this reason, it was decided to alter the formulation slightly for the next round of tests. Similar tests to this round would be necessary in the next round, since the system would once again be changed. With respect to the hydrophilic system, the next round of tests would once again examine the influence of the amount of PA1.

Table 22. Syringe Test Round 9 Fluid Formulations.

Pre-Preflush System	Preflush Systems	Epoxy Systems	Overflush Systems
Base Brine Mutual Solvent	System 1 Base Brine Oil-wetting Surfactant	System 1 Solvent #1 PA1.1	System 1 Base Brine
	System 2 Base Brine Oil-wetting Surfactant Chelating Agent	System 2 Solvent #1 PA1.2	System 2 Base Brine Oil-wetting Surfactant Chelating Agent
		System 3 Solvent #2 PA1.1	
		System 4 Solvent #2 PA1.2	

Table 23. Syringe Test Round 9 Test Grid.

Syringe Test Round 9 – 26 Total Samples		
24 hr Shut-In (130°F)		
Brine Saturated	Oil Saturated	Oil Saturated
3 PV of Preflush System #1 3 PV of Epoxy System #1 3 PV of Overflush System #1	3 PV of Preflush System #1 3 PV of Epoxy System #1 3 PV of Overflush System #1	3 PV of Pre-preflush System 3 PV of Preflush System #1 3 PV of Epoxy System #1 3 PV of Overflush System #1
3 PV of Preflush System #1 3 PV of Epoxy System #2 3 PV of Overflush System #1	3 PV of Preflush System #1 3 PV of Epoxy System #2 3 PV of Overflush System #1	3 PV of Pre-preflush System 3 PV of Preflush System #1 3 PV of Epoxy System #2 3 PV of Overflush System #1
3 PV of Preflush System #1 3 PV of Epoxy System #3 3 PV of Overflush System #1	3 PV of Preflush System #1 3 PV of Epoxy System #3 3 PV of Overflush System #1	3 PV of Pre-preflush System 3 PV of Preflush System #1 3 PV of Epoxy System #3 3 PV of Overflush System #1
3 PV of Preflush System #1 3 PV of Epoxy System #4 3 PV of Overflush System #1	3 PV of Preflush System #1 3 PV of Epoxy System #4 3 PV of Overflush System #1	3 PV of Pre-preflush System 3 PV of Preflush System #1 3 PV of Epoxy System #4 3 PV of Overflush System #1
3 PV of Preflush System #1 3 PV of Epoxy System #1 No Overflush	3 PV of Preflush System #1 3 PV of Epoxy System #1 No Overflush	3 PV of Pre-preflush System 3 PV of Preflush System #1 3 PV of Epoxy System #1 No Overflush
3 PV of Preflush System #1 3 PV of Epoxy System #2 No Overflush	3 PV of Preflush System #1 3 PV of Epoxy System #2 No Overflush	3 PV of Pre-preflush System 3 PV of Preflush System #1 3 PV of Epoxy System #2 No Overflush
3 PV of Preflush System #1 3 PV of Epoxy System #3 No Overflush	3 PV of Preflush System #1 3 PV of Epoxy System #3 No Overflush	3 PV of Pre-preflush System 3 PV of Preflush System #1 3 PV of Epoxy System #3 No Overflush
3 PV of Preflush System #1 3 PV of Epoxy System #4 No Overflush	3 PV of Preflush System #1 3 PV of Epoxy System #4 No Overflush	3 PV of Pre-preflush System 3 PV of Preflush System #1 3 PV of Epoxy System #4 No Overflush
	3 PV of Preflush System #2 3 PV of Epoxy System #3 3 PV of Overflush System #2	3 PV of Preflush System #2 3 PV of Epoxy System #4 3 PV of Overflush System #2

Table 24. Syringe Test Round 9 Post Cure Injection Test and UCS Results.

				UCS (psi)		Post Cure Flow Through Rating	
Mut. Solvent Pre-flush	**Preflush System**	**Epoxy System**	**Overflush System**	**24 hr Shut-In (130°F)**		**24 hr Shut-In (130°F)**	
Yes/No				Brine	Oil	Brine	Oil
No	System 1	System 1	System 1	448	696	S/D	S/D
No	System 1	System 2	System 1	722	1692	S/D	S/D
No	System 1	System 3	System 1	N/A	427	E	E
No	System 1	System 4	System 1	338	609	N/A	S/D
No	System 1	System 1	None	102	68	N/A	N/A
No	System 1	System 2	None	103	84	N/A	N/A
No	System 1	System 3	None	1001	977	N/A	N/A
No	System 1	System 4	None	1241	1224	N/A	VD
Yes	System 1	System 1	System 1		1057		S/D
Yes	System 1	System 2	System 1		107		S/D
Yes	System 1	System 3	System 1		603		D
Yes	System 1	System 4	System 1		712		S/D
Yes	System 1	System 1	None		60		N/A
Yes	System 1	System 2	None		110		N/A
Yes	System 1	System 3	None		942		N/A
Yes	System 1	System 4	None		1094		E
No	System 2	System 3	System 2		N/A		E
No	System 2	System 4	System 2		N/A		E

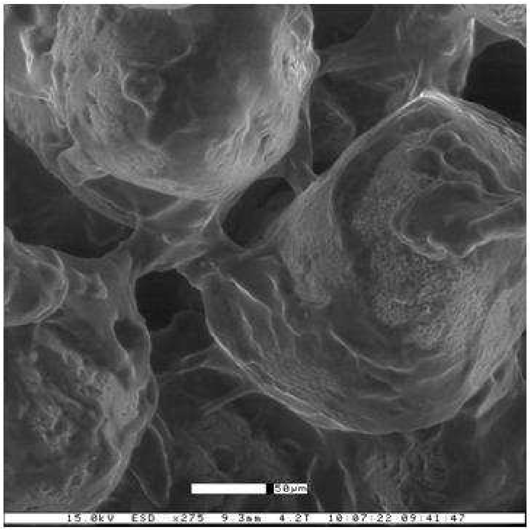


Fig. 8a–Round 9 Sample 9.

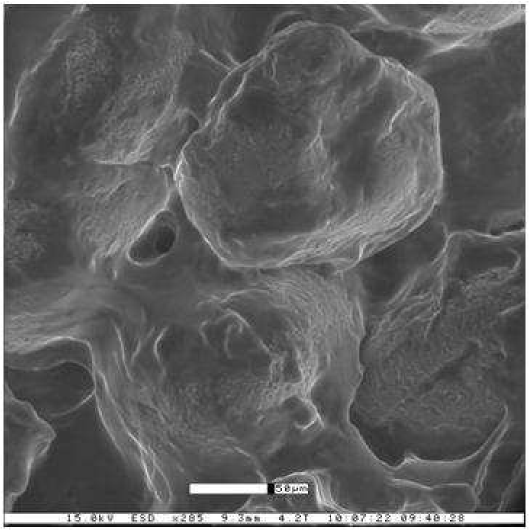


Fig. 8b–Round 9 Sample 10.

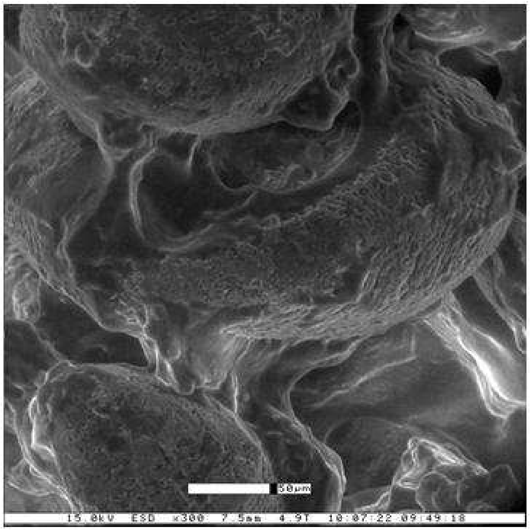


Fig. 8c–Round 9 Sample 11.

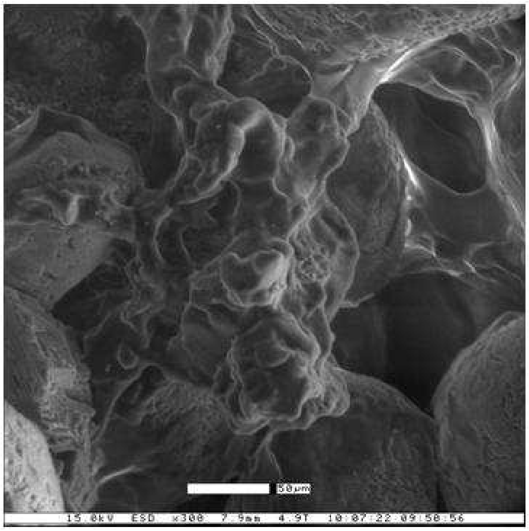


Fig. 8d–Round 9 Sample 12.

Fig. 8–Round 9 Syringe Test SEM Photos.

3.1.10 Syringe Test Round 10 Results

This round of tests once again investigated the effectiveness of a system with a hydrophobic curing agent, System D. A stable combination of the hydrophobic curing agent with a hydrophobic solvent was formulated and tested in this round. Due to the fact that there was a noticeable increase in UCS for the hydrophobic systems where a mutual solvent pre-preflush was pumped in the last round, this portion of the test was repeated for this round. For the hydrophilic system, the concentration of PA1 was increased slightly as compared to the tests of last round. Tests were once again completed to determine the optimal concentration of the adhesion promoter PA1 for this system. The two adhesion promoter concentrations from the previous round, PA1.1 and PA1.2 were repeated to examine reproducibility. The fluid formulations and test grid for this round are given in Tables 25 and 26, respectively.

Once again, promising post cure injection results were obtained for all the samples tested in this round. Most of the samples received a slightly difficult rating, and a few of the hydrophobic systems even received an easy rating. The results of the post cure flow through tests are shown in Table 27. The UCS results of the hydrophobic system performed as well as previous tests, however it was observed that the previous best system still was the optimum. The increase in concentration of PA1 for this round did not show any added benefit. While the hydrophobic system did not do as well with respect to the desired UCS value, it did show an increased UCS results with increased concentrations of PA1. The hydrophobic system once again also showed increased UCS results when a pre-preflush was pumped for the oil saturated samples in 2 out of the 3 samples. Finally, the samples were once again examined using SEM technology. The influence of PA1 for both systems was very obvious. For all the samples, there was increased adhesion between individual sand grains from previous samples without the additive. For the case of the hydrophilic system, while there was an increased cohesion between grains, there still appeared to be residual epoxy left on the sand grain face as shown in Figs. 9a-c. However, for the case of the hydrophobic systems, all signs of the epoxy appeared at the grain to grain contact points, as seen in Figs. 9d-f.

After such positive and consistent results for the hydrophilic system, it was decided that the system was ready to be ran in the coreflood apparatus. The system which would be used for the first tests was that described in this round as System B with PA1 in the epoxy system. For the next round of syringe tests, mineral oil was investigated as the preflush and overflush fluid for System B. With respect to the hydrophobic system, it did not perform as well as expected, but the images shown by the SEM show that if optimized, the system has potential to perform as well, if not better than the hydrophilic system. It was decided that one more set of syringe tests to optimize the hydrophobic system was required prior if it would indeed be necessary to test in the coreflood apparatus. Much like the testing of the hydrophilic system, the effects of surfactants and possibly even a different adhesion promoter needed to be tested.

Table 25. Syringe Test Round 10 Fluid Formulations.

Pre-Preflush System	Epoxy Systems
Base Brine Mutual Solvent	System 1 Solvent #1 Epoxy System B PA1.1
	System 2 Solvent #1 Epoxy System B PA1.2
	System 3 Solvent #1 Epoxy System B PA1.3
	System 4 Solvent #1 Epoxy System D PA1.2
	System 5 Solvent #2 Epoxy System D PA1.1
	System 6 Solvent #2 Epoxy System D PA1.2
	System 7 Solvent #2 Epoxy System D PA1.3

Table 26. Syringe Test Round 10 Test Grid.

Syringe Test Round 10 – 17 Total Samples		
24 hr Shut-In (130°F)		
Brine Saturated	Oil Saturated	Oil Saturated
3 PV of Brine Preflush 3 PV of Epoxy System #1 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Epoxy System #1 3 PV of Brine Overflush	
3 PV of Brine Preflush 3 PV of Epoxy System #2 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Epoxy System #2 3 PV of Brine Overflush	
3 PV of Brine Preflush 3 PV of Epoxy System #3 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Epoxy System #3 3 PV of Brine Overflush	
3 PV of Brine Preflush 3 PV of Epoxy System #4 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Epoxy System #4 3 PV of Brine Overflush	
3 PV of Brine Preflush 3 PV of Epoxy System #5 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Epoxy System #5 3 PV of Brine Overflush	3 PV of Pre-preflush 3 PV of Brine Preflush 3 PV of Epoxy System #5 3 PV of Brine Overflush
3 PV of Brine Preflush 3 PV of Epoxy System #6 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Epoxy System #6 3 PV of Brine Overflush	3 PV of Pre-preflush 3 PV of Brine Preflush 3 PV of Epoxy System #6 3 PV of Brine Overflush
3 PV of Brine Preflush 3 PV of Epoxy System #7 3 PV of Brine Overflush	3 PV of Brine Preflush 3 PV of Epoxy System #7 3 PV of Brine Overflush	3 PV of Pre-preflush 3 PV of Brine Preflush 3 PV of Epoxy System #7 3 PV of Brine Overflush

Table 27. Syringe Test Round 10 Post Cure Injection Test and UCS Results.

			UCS (psi)		**Post Cure Flow Through Rating**	
Mut. Solvent Pre-flush	**Epoxy System**	**Epoxy Additive/Concentration**	**24 hr Shut-In (130°F)**		**24 hr Shut-In (130°F)**	
Yes/No			Brine	Oil	Brine	Oil
No	2B	PA1.1	619	706	S/D	S/D
No	2B	PA1.2	920	1380	S/D	S/D
No	2B	PA1.3	689	1242	S/D	D
No	2C	PA1.2	37	68	S/D	S/D
No	2C	PA1.1	0	126	S/D	S/D
No	2C	PA1.2	165	193	E	S/D
No	2C	PA1.3	126	522	E	S/D
Yes	2C	PA1.1		204		S/D
Yes	2C	PA1.2		229		S/D
Yes	2C	PA1.3		163		E
No	2C	PA1.2		428*		S/D

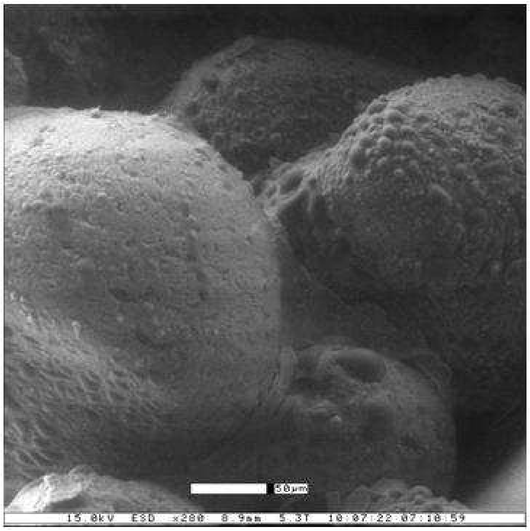


Fig. 9a–Round 10 Sample 1.

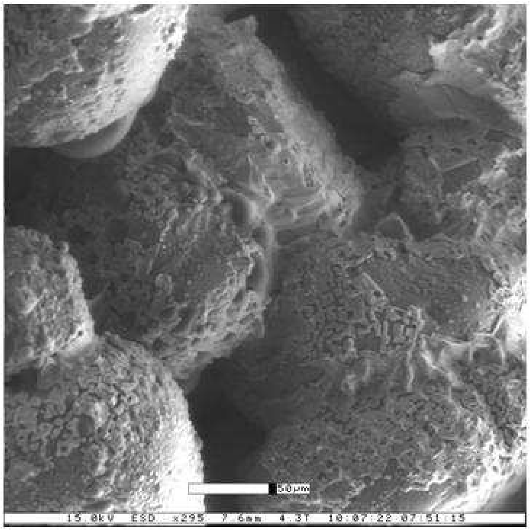


Fig. 9b–Round 10 Sample 4.

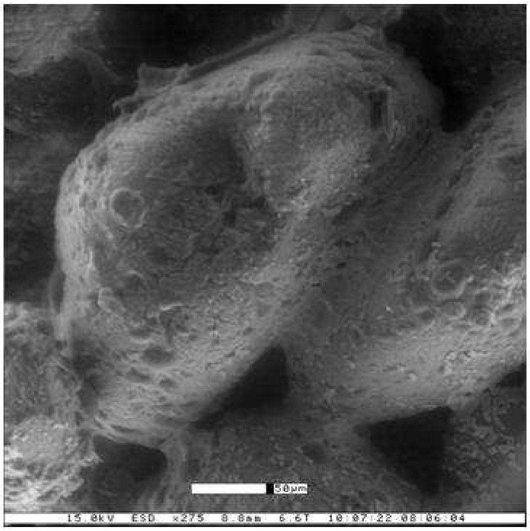


Fig. 9c–Round 10 Sample 5.

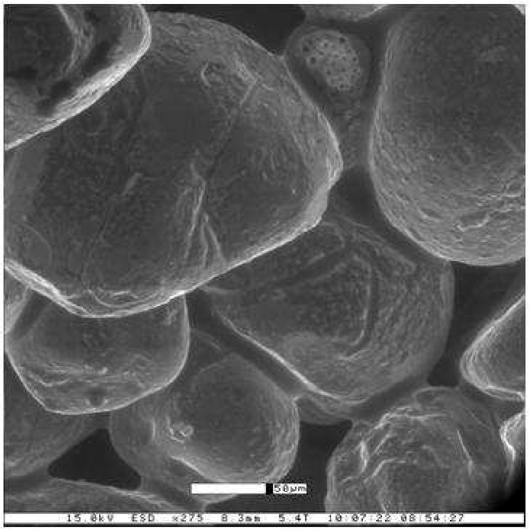


Fig. 9d–Round 10 Sample 9.

Fig. 9–Round 10 Syringe Test SEM Photos.

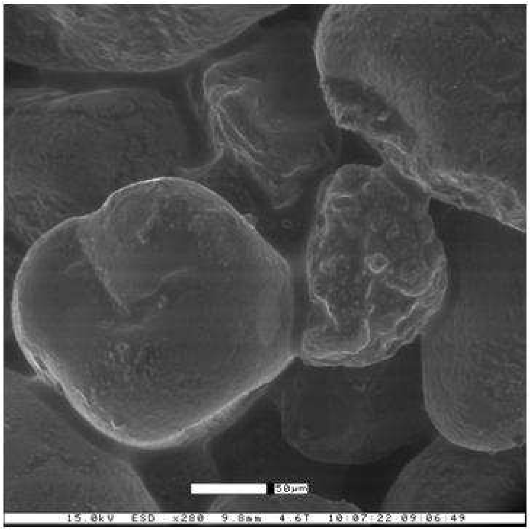


Fig. 9e–Round 10 Sample 10.

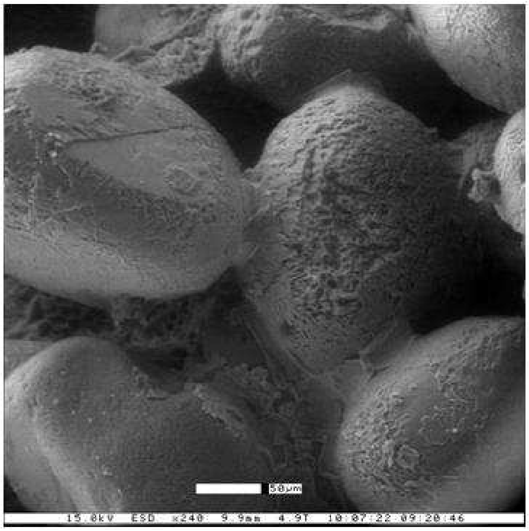


Fig. 9f–Round 10 Sample 12.

Fig. 9–Continued.

3.1.11 Syringe Test Round 11 Results

Because of the slight emulsion when the KCl brine was mixed with the hydrophilic system as referenced previously in the results of the in Round 7, it was decided to complete a bottle test of the system with the mineral oil used for the testing. After completing these tests, it appeared the hydrophilic system and the mineral oil did not create an emulsion, suggesting it could also be used as a possible overflush fluid. For the eleventh round of tests, samples were once again saturated with brine and oil and the effect of using mineral oil as the preflush and overflush fluid examined. Two samples would have the normal 3 PV overflush, while another would have double the overflush, 6 PV. The test grid for this round of tests is given in Table 28.

After the samples cured, the post cure injection tests were completed. The results of the injection test along with the UCS results are given in Table 29. All the samples appeared to have relatively good flow, receiving slightly difficult in all but one case. The exception was a sample which was received an easy rating for its injection test. The UCS results obtained were consistent between all the samples, ranging from 497 to 735 psi for the four similar samples. However, the effect of double the flush was quite evident, resulting in 308 and 38 for the brine and oil saturated samples, respectively. When the samples were examined for color and material properties, they were very similar to the samples completed in Round 9 when an overflush was not injected through the sample. While the samples were homogeneous with respect to fluid placement, the appearance was that a large amount of the curing agent system was left in the samples. The conclusion from the observation was that while there is a lack of interaction between the epoxy system and the flush fluid, the mineral oil does not effectively remove the epoxy fluid from the pore spaces at the conditions tested. Comparing the results of this round to the results obtained when the brine preflush and overflush for both saturations, the average UCS values decreased by 266 for the brine and 682 for oil saturated samples.

Because of the results observed in this round, as far as my research is concerned, the effects of a hydrocarbon based preflush and overflush will not be further investigated as it does not appear to improve the results in the syringe tests.

Table 28. Syringe Test Round 11 Test Grid.

Syringe Test Round 11 – 6 Total Samples	
24 hr Shut-In (130°F)	
Brine Saturated	Oil Saturated
3 PV of Mineral Oil Preflush 3 PV of System B 3 PV of Mineral Oil Overflush (Repeated Twice)	3 PV of Mineral Oil Preflush 3 PV of System B 3 PV of Mineral Oil Overflush (Repeated Twice)
3 PV of Mineral Oil Preflush 3 PV of Epoxy System 6 PV of Mineral Oil Overflush	3 PV of Mineral Oil Preflush 3 PV of Epoxy System 6 PV of Mineral Oil Overflush

Table 29. Syringe Test Round 11 Post Cure Injection Test and UCS Results.

		UCS (psi)		**Post Cure Flow Through Rating**	
Preflush System	**Overflush System**	**24 hr Shut-In (130°F)**		**24 hr Shut-In (130°F)**	
		Brine	Oil	Brine	Oil
Mineral Oil	Mineral Oil	614	735	S/D	S/D
Mineral Oil	Mineral Oil	497	613	S/D	S/D
Mineral Oil	2X Mineral Oil	303	38	S/D	E

3.1.12 Syringe Test Round 12 Results

For the twelfth round of tests several different variables were manipulated. While a hydrophilic system has been developed and consistently shows good results, this round investigated the effect of different additives in the preflush, as well as to test other additives in the epoxy system. For a few of the tests, an oil-wetting surfactant of anionic, cationic, and nonionic nature was added to the preflush. These tests were completed to observe how the different surfactants and the resulting change of wettability would affect the results. For these cases, the epoxy system was left the same as tested in previous rounds. For the same reason as the surfactants, a new additive, PA2, was added to preflush to see if would yield improved results. Finally, two new additives in the epoxy system were tested, PA3 and PA4. PA3 and PA4 are similar in nature to PA1 and PA2 in that they are used to improve the adhesion of the epoxy to the sand grain. The fluid systems used for this round are identified in Table 30, and the respective test grid is given in Table 31. As a reminder, the second number after the reference of which proprietary additive is being used, refers to a specific concentration of that additive used in the system.

While the post cure injection yielded positive results for a few of the samples, there was no clear correlation between the addition of the surfactant and the results. The nonionic surfactant preflush resulted in a system which was difficult to inject through for both the brine and oil saturated samples. Other than this, the rest of the samples were given a mix of easy and slightly difficult injection ratings. The results from the flow tests are given in Table 32. The effect of the surfactant for the UCS was a little more evident. The anionic surfactant gave similar results to the system when it is not used. While it did not improve results, it did not appear to hinder the sample’s performance. The cationic and nonionic resulted in a noticeable decrease in the UCS results for both samples. Coupled with the flow results, it does not appear the addition of a surfactant improves the current epoxy system’s performance. The addition of PA2 to the preflush showed very positive results. As can be seen in the UCS results in Table 32, both the brine and oil saturated samples were ~300 psi higher than the previous best results of the current optimized system.

The last variable changed in this round was the use of different adhesion promoting additives in the epoxy system. PA3 showed no improvements over the current system, PA1. However, PA4 showed a slight improvement over PA1; ~200 psi higher on the brine sample and about same results for the oil saturated sample.

The results of this round showed that the addition of the new additive PA2 to the preflush significantly improved the UCS results. For this reason, a future round of tests was necessary to examine the effect of its concentration. Similarly, seeing such positive effects of PA4 on the base epoxy system, the effect of the PA2 preflush with this system needed to be investigated. Lastly, the effects of a hydrophobic curing agent system still needed to be tested as well with these new additives. For this reason, the next round would be very similar to the current, except that the curing agent will be hydrophobic. Following this, Round 14 would optimize the adhesion promoter amount in the preflush stage.

Table 30. Syringe Test Round 12 Fluid Formulations.

Preflush System	Epoxy Systems
System 1 Base Brine	System 1 Epoxy System B PA1
System 2 Base Brine Anionic Oil-Wetting Surfactant	System 2 Epoxy System B PA3
System 3 Base Brine Cationic Oil-Wetting Surfactant	System 3 Epoxy System B PA1 PA3
System 4 Base Brine Nonionic Oil-Wetting Surfactant	System 4 Epoxy System B PA4
System 5 Base Brine PA2.1	

Table 31. Syringe Test Round 12 Test Grid.

Syringe Test Round 12 – 14 Total Samples		
24 hr Shut-In (130°F)		
Brine Saturated	Brine Saturated	Oil Saturated
3 PV of Preflush System 1 3 PV of System 1 – PA1.2 3 PV of Brine Overflush	3 PV of Preflush System 2 3 PV of System 1 – PA1.2 3 PV of Brine Overflush	3 PV of Preflush System 2 3 PV of System 1 – PA1.2 3 PV of Brine Overflush
3 PV of Preflush System 1 3 PV of System 2 – PA3.1 3 PV of Brine Overflush	3 PV of Preflush System 3 3 PV of System 1 – PA1.2 3 PV of Brine Overflush	3 PV of Preflush System 3 3 PV of System 1 – PA1.2 3 PV of Brine Overflush
3 PV of Preflush System 1 3 PV of System 3 – PA1.1/PA3.1 3 PV of Brine Overflush	3 PV of Preflush System 4 3 PV of System 1 – PA1.2 3 PV of Brine Overflush	3 PV of Preflush System 4 3 PV of System 1 – PA1.2 3 PV of Brine Overflush
3 PV of Preflush System 1 3 PV of System 2 – PA3.2 3 PV of Brine Overflush	3 PV of Preflush System 5 3 PV of System 1 – PA1.2 3 PV of Brine Overflush	3 PV of Preflush System 5 3 PV of System 1 – PA1.2 3 PV of Brine Overflush
	3 PV of Preflush System 1 3 PV of System 4 – PA4.2 3 PV of Brine Overflush	3 PV of Preflush System 1 3 PV of System 4 – PA4.2 3 PV of Brine Overflush

Table 32. Syringe Test Round 12 Post Cure Injection Test and UCS Results.

		UCS (psi)		**Post Cure Flow Through Rating**	
Preflush	**Adh. Promoter**	**24 hr Shut-In (130°F)**		**24 hr Shut-In (130°F)**	
		Brine	Oil	Brine	Oil
System 1	PA1.2	781		S/D	
System 1	PA3.1	563		S/D	
System 1	PA1.1 + PA3.1	782		E	
System 1	PA3.2	802		S/D	
System 2	PA1.2	723	1558	E	S/D
System 3	PA1.2	494	1225*	E	S/D
System 4	PA1.2	660	1108	D	D
System 5	PA1.2	1301	2031	S/D	S/D
System 1	PA4.2	1181	1637	E	S/D

* Average of two tests on the sample

3.1.13 Syringe Test Round 13 Results

As previously mentioned, the setup of the tests in the thirteenth round was extremely similar to that of the twelfth round, except that a hydrophobic curing agent was being tested instead of the hydrophilic one. The purpose of all the different fluid systems combinations was explained in the previous round. For completeness, the fluid formulations and test grid are shown in Tables 33 and 34, respectively.

The results of this round are shown in Table 35. As observed with the hydrophobic systems previously tested, the post cure injection results were favorable. While there were more 'slightly difficult' rated samples, there were quite a few which were 'easy' to flow through. For both the brine and oil saturated samples which had a cationic surfactant in the preflush, the injection was relatively easy. These samples were also very strong; yielding two of the highest UCS results for this round. PA3 did not improve results as compared to PA1 and PA4. The same was true for the anionic surfactant which decreased the UCS results slightly for the brine sample, but increased the result slightly for the oil sample. PA4 gave the most consolidated brine sample, and also flowed very easily. Finally, when PA2 was put in the preflush, this did not improve the system's performance like it had with the hydrophilic system. Overall the system performed well, but not as well as the current hydrophilic system.

Considering the results, it was decided that the hydrophobic system would not further be investigated. However, considering the results of the previous round, one final round of syringe tests was necessary to optimize the concentration of PA2 in the preflush.

Table 33. Syringe Test Round 13 Fluid Formulations.

Preflush System	Epoxy Systems
System 1 Base Brine	System 1 Epoxy System D PA1
System 2 Base Brine Anionic Oil-Wetting Surfactant	System 2 Epoxy System D PA3
System 3 Base Brine Cationic Oil-Wetting Surfactant	System 3 Epoxy System D PA4
System 4 Base Brine Nonionic Oil-Wetting Surfactant	
System 5 Base Brine PA2.1	

Table 34. Syringe Test Round 13 Test Grid.

Syringe Test Round 13 – 14 Total Samples	
24 hr Shut-In (130°F)	
Brine Saturated	Oil Saturated
3 PV of Preflush System 1 3 PV of System 1 – PA1.3 3 PV of Brine Overflush	3 PV of Preflush System 1 3 PV of System 1 – PA1.3 3 PV of Brine Overflush
3 PV of Preflush System 1 3 PV of System 2 – PA3.3 3 PV of Brine Overflush	3 PV of Preflush System 1 3 PV of System 2 – PA3.3 3 PV of Brine Overflush
3 PV of Preflush System 2 3 PV of System 1 – PA1.3 3 PV of Brine Overflush	3 PV of Preflush System 2 3 PV of System 1 – PA1.3 3 PV of Brine Overflush
3 PV of Preflush System 3 3 PV of System 1 – PA1.3 3 PV of Brine Overflush	3 PV of Preflush System 3 3 PV of System 1 – PA1.3 3 PV of Brine Overflush
3 PV of Preflush System 4 3 PV of System 1 – PA1.3 3 PV of Brine Overflush	3 PV of Preflush System 4 3 PV of System 1 – PA1.3 3 PV of Brine Overflush
3 PV of Preflush System 5 3 PV of System 1 – PA1.3 3 PV of Brine Overflush	3 PV of Preflush System 5 3 PV of System 1 – PA1.3 3 PV of Brine Overflush
3 PV of Preflush System 1 3 PV of System 3 – PA4.3 3 PV of Brine Overflush	3 PV of Preflush System 1 3 PV of System 3 – PA4.3 3 PV of Brine Overflush

Table 35. Syringe Test Round 13 Post Cure Injection Test and UCS Results.

		UCS (psi)		**Post Cure Flow Through Rating**	
Preflush	**Epoxy Additive/Concentration**	**24 hr Shut-In (130°F)**		**24 hr Shut-In (130°F)**	
		Brine	Oil	Brine	Oil
System 1	PA1.3	218	529	S/D	S/D
System 1	PA3.3	82	329	S/D	S/D
System 2	PA1.3	92	584	S/D	E
System 3	PA1.3	639	1605	E	E
System 4	PA1.3	518	263	S/D	S/D
System 5	PA1.3	263	386	S/D	S/D
System 1	PA4.3	668	217	E	E

3.1.14 Syringe Test Round 14 Results

The addition of PA2 to the preflush resulted in noticeable improvements in the performance of the hydrophilic fluid system in Round 12. Because of the improvements observed, the purpose of this round was to optimize its concentration. Also, the addition of PA4 to the base system showed promising results in Round 12 without the addition of PA2 to the preflush. To see if it would show any better results with PA2 in the preflush, it was also tested in this round. The fluid formulations for this round are given in Table 36, while the test grid for this round of tests is given in Table 37.

The UCS and post cure injection results are given in Table 38. While there were several samples which were quite easy to inject through, there did not appear to be a correlation to the amount of PA2 in the preflush. However, two of the concentrations, PA2.2 and PA2.3, both systems were rated easy in 7 out of the 8 combinations. With respect to the UCS results, when PA1 was in the epoxy system the optimal concentration of PA2 was that which was used in Round 12, PA2.1. A decrease or increase in concentration of the adhesion promoter did not further improve the results. While the trend was not as noticeable for the case of PA4, the best results for this system was with PA2.4 system as the preflush fluid.

For the sake of my research and the chemistry being tested, it appears as if the optimal concentrations of additives have been discovered. Syringe tests have given reproducible results for both the brine and oil saturated samples. For this reason, System B was then further tested in the HP/HT Cell described in Section 3.2.

Table 36. Syringe Test Round 14 Fluid Formulations.

Preflush System	Epoxy Systems
System 1 Base Brine PA2.2	System 1 Epoxy System B PA1.2
System 2 Base Brine PA2.1	System 2 Epoxy System B PA4.2
System 3 Base Brine PA2.3	
System 4 Base Brine PA2.4	

Table 37. Syringe Test Round 14 Test Grid.

Syringe Test Round 14 – 16 Total Samples	
24 hr Shut-In (130°F)	
Brine Saturated	Oil Saturated
3 PV of Preflush System 1 3 PV of System 1 3 PV of Brine Overflush	3 PV of Preflush System 1 3 PV of System 1 3 PV of Brine Overflush
3 PV of Preflush System 2 3 PV of System 1 3 PV of Brine Overflush	3 PV of Preflush System 2 3 PV of System 1 3 PV of Brine Overflush
3 PV of Preflush System 3 3 PV of System 1 3 PV of Brine Overflush	3 PV of Preflush System 3 3 PV of System 1 3 PV of Brine Overflush
3 PV of Preflush System 4 3 PV of System 1 3 PV of Brine Overflush	3 PV of Preflush System 4 3 PV of System 1 3 PV of Brine Overflush
3 PV of Preflush System 1 3 PV of System 2 3 PV of Brine Overflush	3 PV of Preflush System 1 3 PV of System 2 3 PV of Brine Overflush
3 PV of Preflush System 2 3 PV of System 2 3 PV of Brine Overflush	3 PV of Preflush System 2 3 PV of System 2 3 PV of Brine Overflush
3 PV of Preflush System 3 3 PV of System 2 3 PV of Brine Overflush	3 PV of Preflush System 3 3 PV of System 2 3 PV of Brine Overflush
3 PV of Preflush System 4 3 PV of System 2 3 PV of Brine Overflush	3 PV of Preflush System 4 3 PV of System 2 3 PV of Brine Overflush

Table 38. Syringe Test Round 14 Post Cure Injection Test and UCS Results.

		UCS (psi)		**Post Cure Flow Through Rating**	
Preflush	**Epoxy System**	**24 hr Shut-In (130°F)**		**24 hr Shut-In (130°F)**	
		Brine	Oil	Brine	Oil
System 1	System 1	1183	1534	E	S/D
System 2	System 1	1482	1918	S/D	S/D
System 3	System 1	1383	1828	E	E
System 4	System 1	1190	1619	S/D	S/D
System 1	System 2	1234*	1254	E	E
System 2	System 2	1303	1855	S/D	S/D
System 3	System 2	1335	1276	E	E
System 4	System 2	1421	1917	S/D	S/D

3.2 Coreflood Results

After the tenth round of syringe tests, fluid system B was consistently performing well with respect to its strength. While the post cure flow injections completed on every sample were useful, an actual retained permeability measurement was necessary to accurately access the performance of the fluid.

After each sample was prepared to be 1” diameter x 3.25” length, it was loaded into the HP/HT cell and a confining pressure of 1400 psi was applied. For each sample, the pre-treatment permeability was measured at a minimum of 5 different injection rates. While switching in between rates, for the first few seconds the sample takes a while to reach equilibrium with respect to pressure drop across the sample at the new injection rate. For this reason, all samples will be presented with the permeability from all recorded values (raw data), and a second (smoothed data) in which the values when the sample is reaching equilibrium are removed from the data set. Following this, the sample was heated to the test temperature and the treatment fluids were injected. Each sample’s respective conditions are discussed in their respective section. Most of the fluid stages were designed to be 3 PV which was 49.6 ml for the given sample size.

3.2.1 Coreflood Sample 1 Results

The pre-treatment raw and smoothed permeability data for this sample is plotted in Figs. 10 and 11. The test temperature for Sample 1 was 130°F. For this sample, the volume of the preflush, epoxy system, and overflush was 3 PV, and the stages were injected at 5 ml/min. Following the injection of these stages, the system was left for 24 hours to allow the sample to cure.

After 24 hours, the sample was tested for retained permeability. As shown for the pre-treatment data, Figs. 12 and 13 show the raw and smoothed post-treatment permeability data, respectively. The average permeability for each injection rate is given in Table 39, where it is compared to the pre-treatment permeability. In this table, the average permeabilities of the smoothed and raw data are also given. Considering that some data was removed, there is very good agreement in the retained permeability numbers. The average retained permeability of the smoothed and raw data was 66.3%. While removing the sample to test its UCS, the sample broke into a few pieces. For this reason, the average of the three UCS results was taken as the sample's UCS; 112 psi.

While the retained permeability measurement was satisfactory, the UCS wasn't as high as desired considering how well the system was performing in the syringe tests. From these results, it was determined that further testing was still needed to optimize the system, as well as show reproducible results.

Table 39. Coreflood Sample 1 Permeability Data.

Rate (ml/min)	**Post-Perm** (md)	**Pre-Perm** (md)	**Retained Perm** (%)
30	2952	4742	62.3%
20	2848	4591	62.0%
10	2796	4479	62.4%
5	3031	4407	68.8%
2.5	3648	4245	86.0%
Smoothed	2926	4441	**65.9%**
Raw	2977	4470	**66.6%**

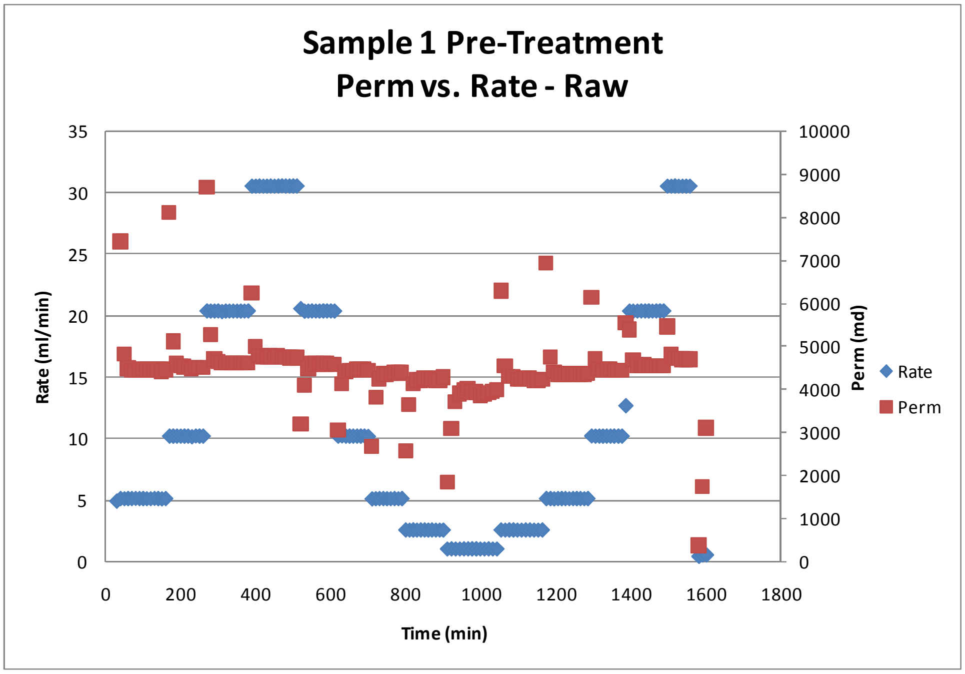


Fig. 10–Coreflood Sample 1 Pre-Treatment Permeability Plot – Raw Data.

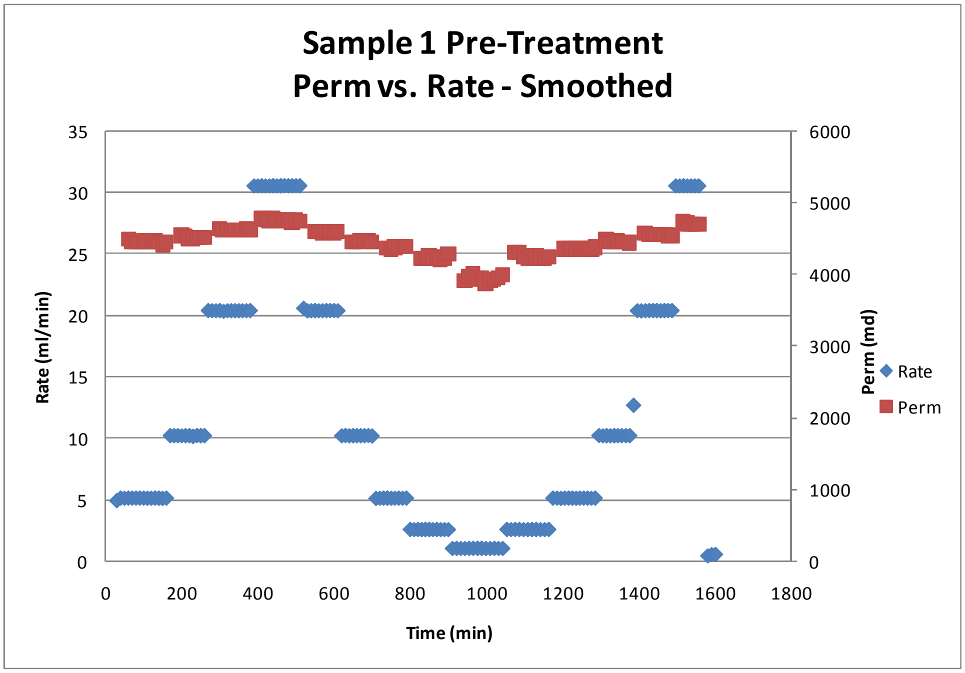


Fig. 11–Coreflood Sample 1 Pre-Treatment Permeability Plot – Smoothed Data.

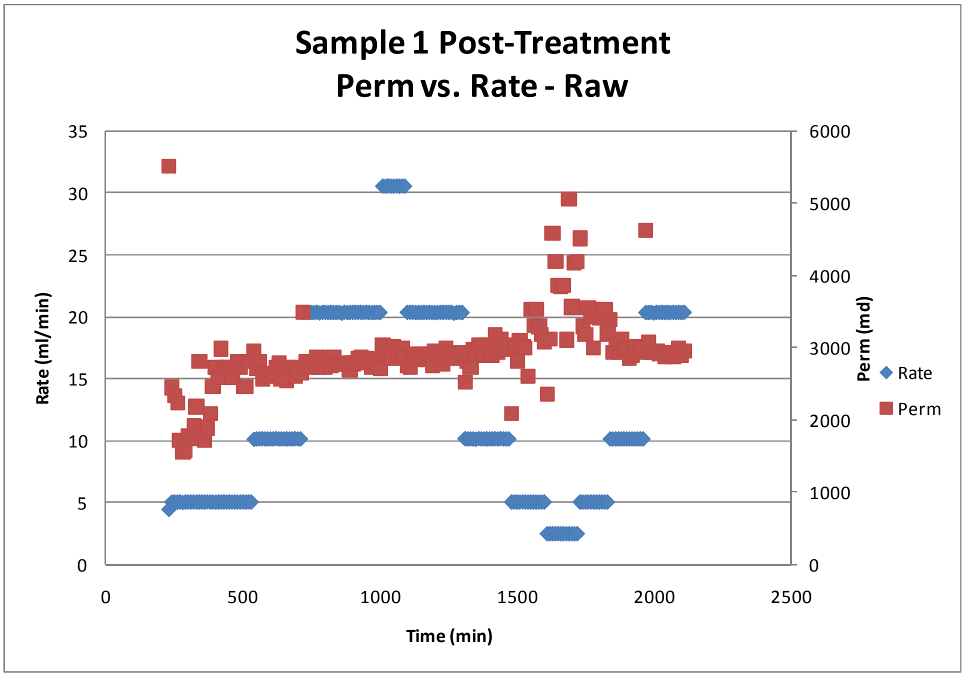


Fig. 12–Coreflood Sample 1 Post-Treatment Permeability Plot – Raw Data.

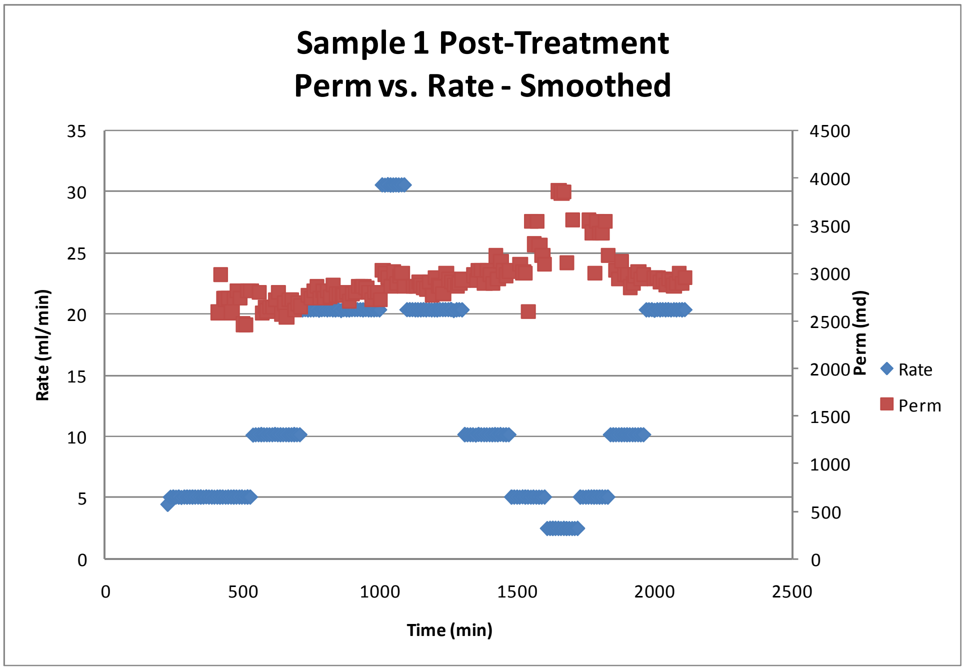


Fig. 13–Coreflood Sample 1 Post-Treatment Permeability Plot – Smoothed Data.

3.2.2 Coreflood Sample 2 Results

Following the testing of the first sample, it was decided to run the exact same system, the exact same constraints, to see if the results were reproducible. The pre-treatment raw and smoothed permeability data are given in Figs. 14 and 15. The average permeability for each rate is given in Table 40.

Following the 24 hour cure time, the sample was removed to allow for the cleaning of the injection lines, as it appeared a small amount of epoxy plugged the flow loop injection line. While removing the line and preparing the sample for the new injection line, a small amount of the sample broke off from the sample. The broken portion was removed and the sample was fixed so that the post-treatment permeability could be measured. The change in length of the sample was noted, as this is critical in the permeability calculation. The average permeability from Figs. 16 and 17 is given in Table 40. The average of the smoothed and raw data permeability's was slightly lower than the previous round, with a value of 52.5%. As expected with the lower retained permeability, a higher UCS value was obtained on the sample. This sample had a UCS value of 331 psi.

While the retained permeability and UCS measurements were not extremely close for the two samples, the system appears to be show the same correlation of UCS and retained permeability as most current systems. As one increases, the other will typically decrease.

Table 40. Coreflood Sample 2 Permeability Data.

Rate	**Post-Perm**	**Pre-Perm**	**Retained Perm**
(ml/min)	(md)	(md)	(%)
20	2826	5680	49.7%
10	2862	5895	48.6%
5	3100	5851	53.0%
2.5	3169	5453	58.1%
Smoothed	2957	5665	**52.2%**
Raw	3010	5710	**52.7%**

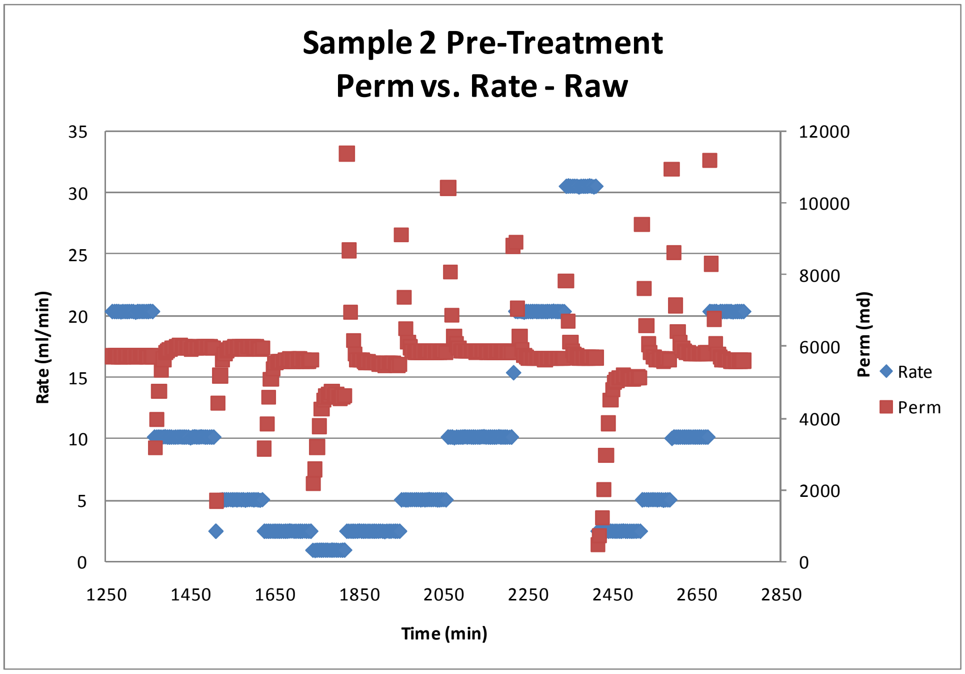


Fig. 14–Coreflood Sample 2 Pre-Treatment Permeability Plot – Raw Data.

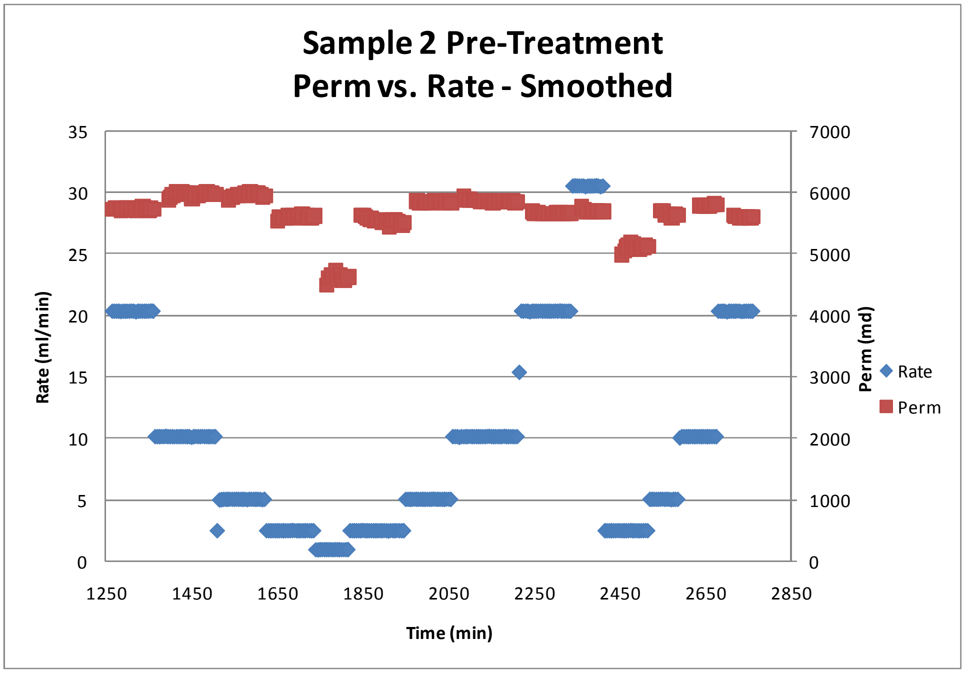


Fig. 15–Coreflood Sample 2 Pre-Treatment Permeability Plot – Smoothed Data.

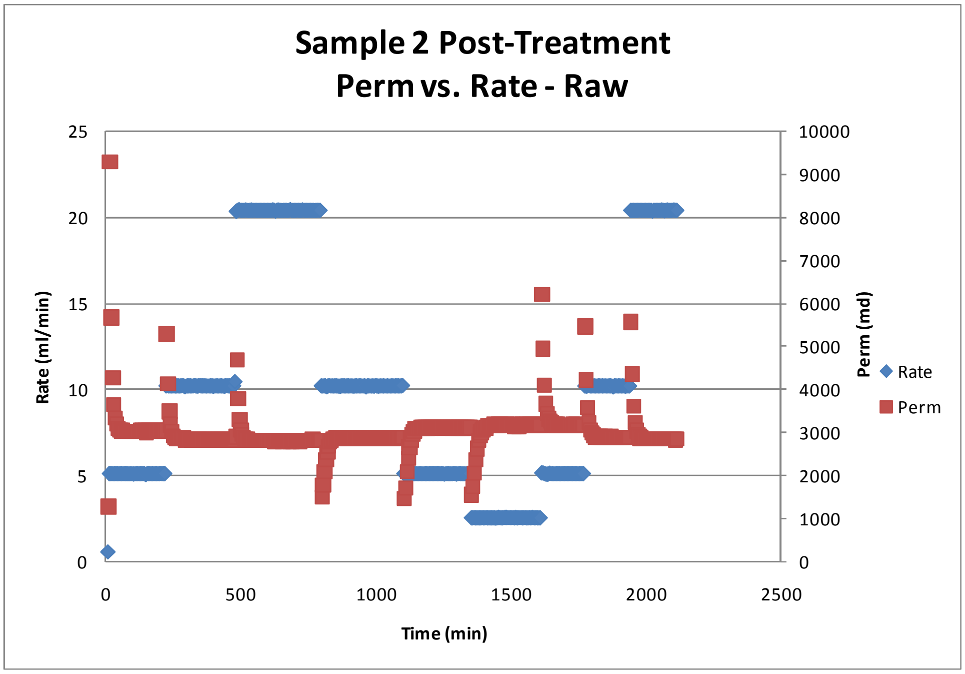


Fig. 16–Coreflood Sample 2 Post-Treatment Permeability Plot – Raw Data.

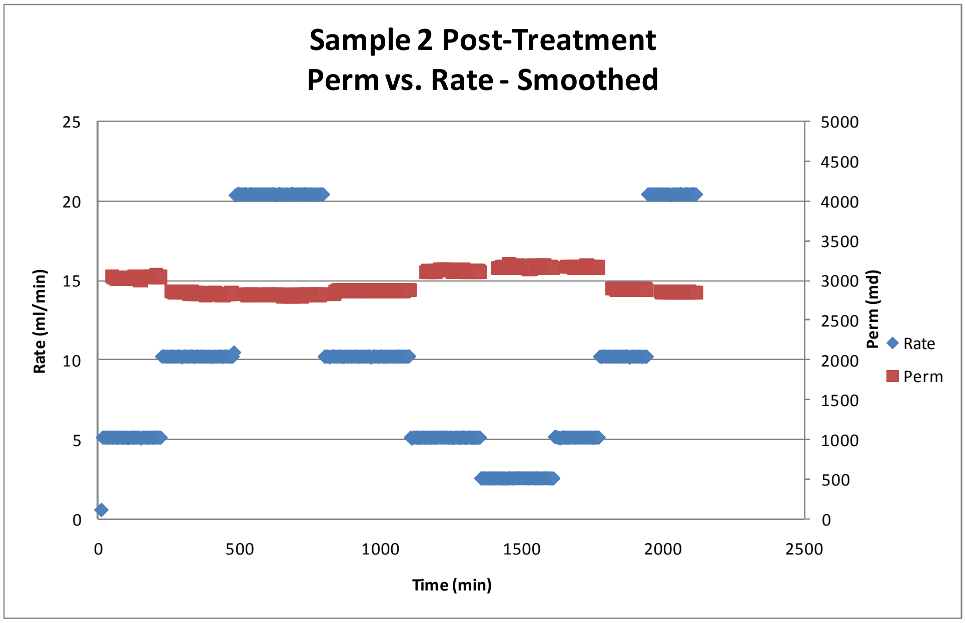


Fig. 17–Coreflood Sample 2 Post-Treatment Permeability Plot – Smoothed Data.

3.2.3 Coreflood Sample 3 Results

For Sample 3, the effect of injection rate was investigated. It was decided to decrease the injection rate of the fluid stages. The idea behind the decrease in rate was that a lower rate will allow for increased contact time between the epoxy system and the sand grains. Because the UCS results were not close to the average of the same system in the syringe tests (821 psi), it was suspected that the epoxy was not effectively adhering to the sand grains at the higher rate. At the same time, while injecting the flush, at this rate it is more like to obtain a more consistent removal of the epoxy from the pore spaces. The amount of fluid injected was once again held constant to the same amounts as Samples 1 and 2, as well as all other test conditions. The pre-treatment raw and smoothed permeability data is given in Figs. 18 and 19. The average permeability using the smoothed data for each rate is given in Table 41.

Following the 24 hour cure period, the sample was removed to clean the injection lines. Following this, the post-treatment permeability measurements were taken. The average permeabilities calculated in Table 41 were from Figs. 20 and 21. The average retained permeability from this round was much lower, with a value of 25.0%. The same UCS versus retained permeability relationship observed in the previous two tests appeared to happen with Sample 3. With a lower retained permeability, Sample 3 had a higher UCS value than previous rounds, 753 psi. Unfortunately, while the decrease in rate helped increase the residual strength of the sample, it decreased the permeability of the sample more than expected.

Table 41. Coreflood Sample 3 Permeability Data.

Rate	**Post-Perm**	**Pre-Perm**	**Retained Perm**
(ml/min)	(md)	(md)	(%)
20	1725	5805	29.7%
10	1548	5595	27.7%
5	1216	5249	23.2%
2.5	1101	4649	23.7%
Smoothed	1397	5665	**24.7%**
Raw	1397	5511	**25.3%**

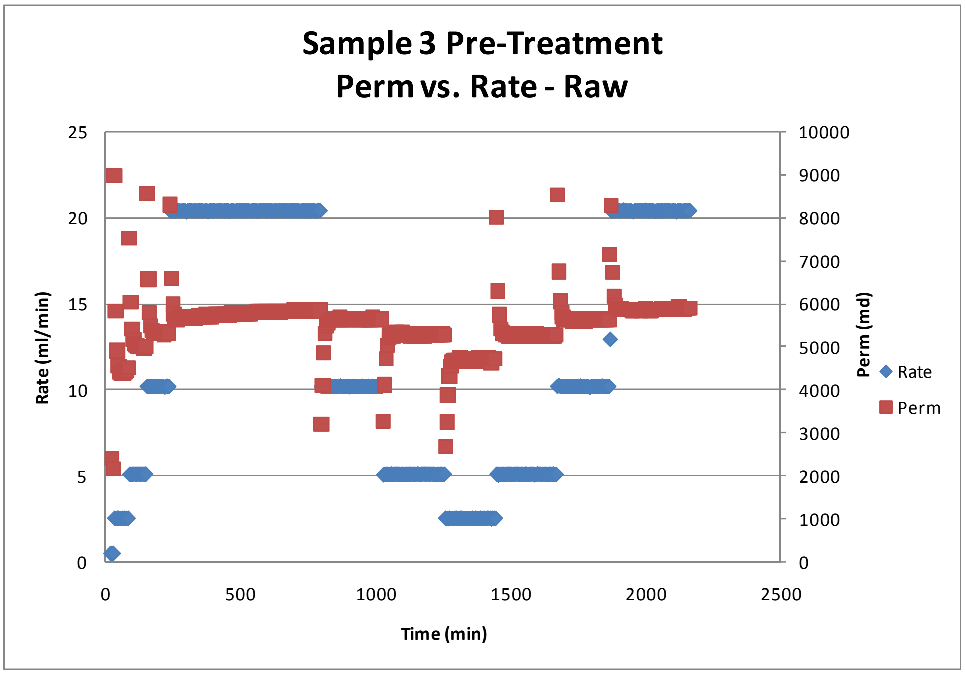


Fig. 18–Coreflood Sample 3 Pre-Treatment Permeability Plot – Raw Data.

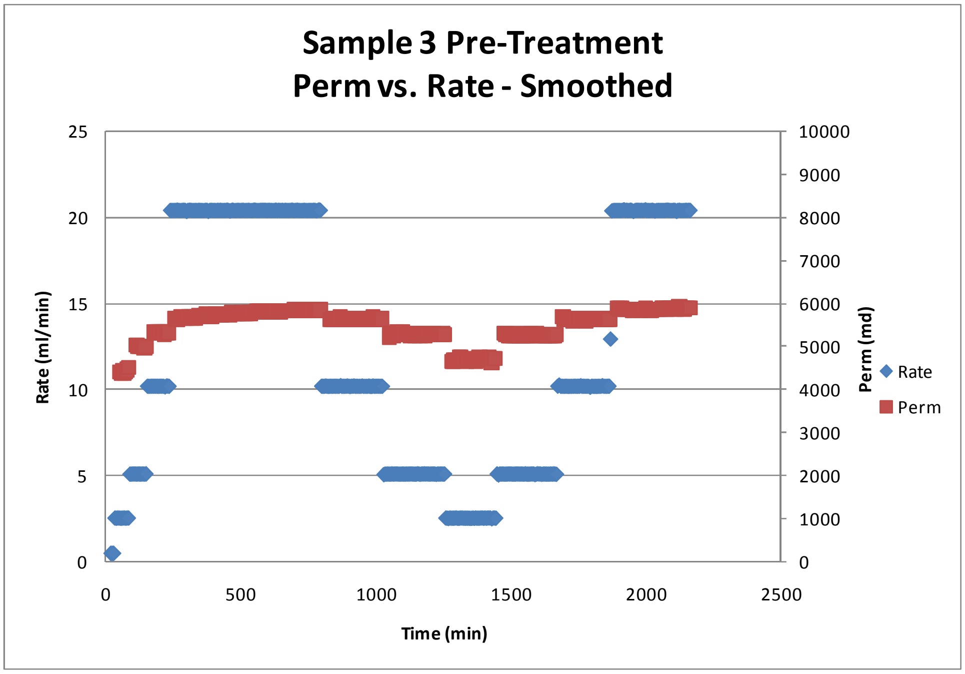


Fig. 19–Coreflood Sample 3 Pre-Treatment Permeability Plot – Smoothed Data.

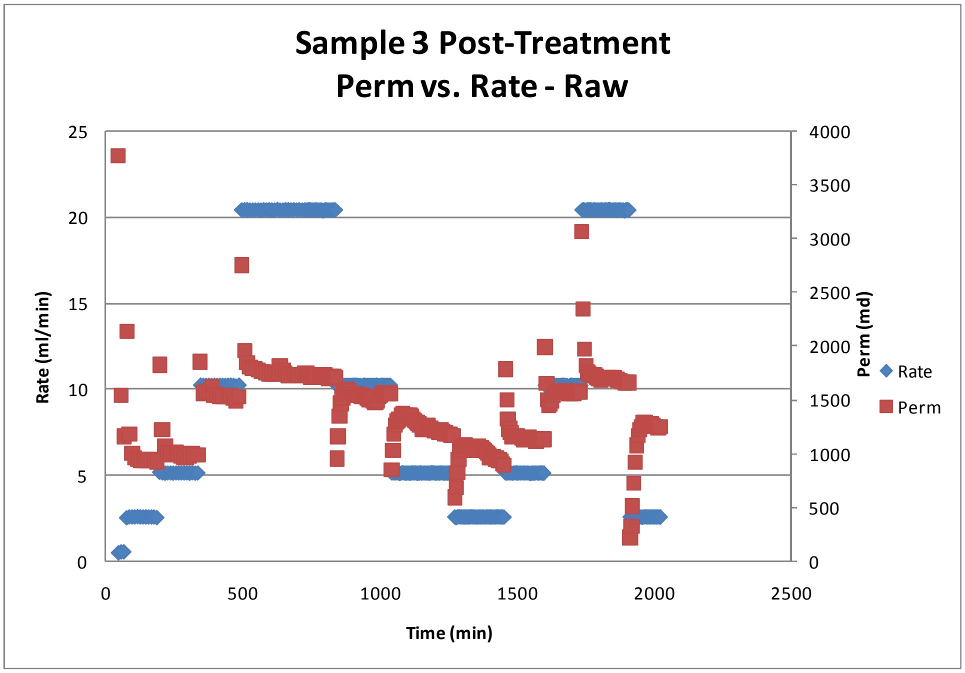


Fig. 20–Coreflood Sample 3 Post-Treatment Permeability Plot – Raw Data.

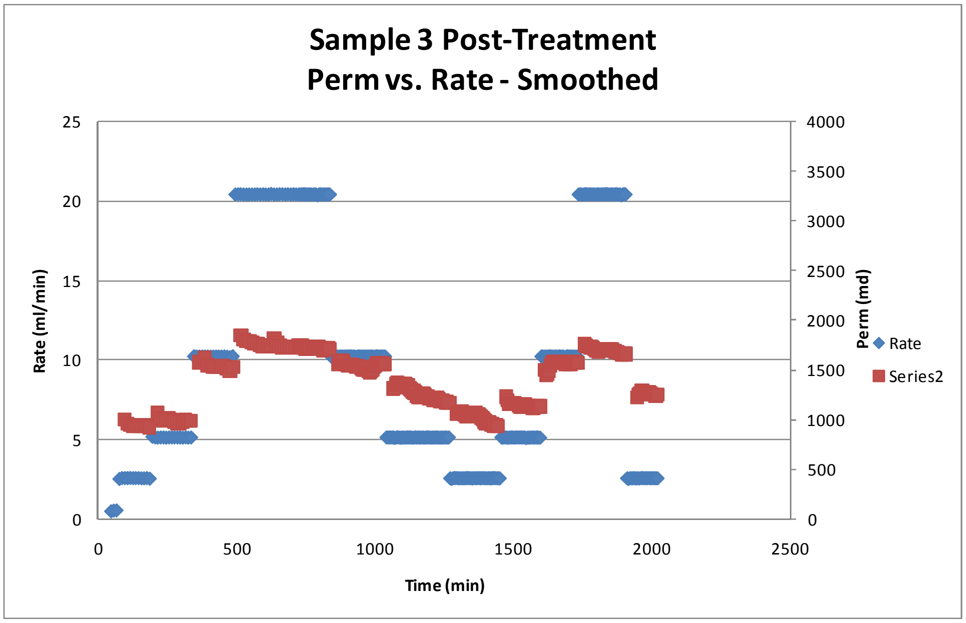


Fig. 21–Coreflood Sample 3 Post-Treatment Permeability Plot – Smoothed Data.

3.2.4 Coreflood Sample 4 Results

For Sample 4, the volume of the overflush fluid was increased in attempt to increase the retained permeability observed in Sample 3. The goal of pumping a larger amount of overflush was that residual epoxy left in pore spaces would be more likely to be removed. The volumes of the preflush and epoxy system and the injection rate of the fluid stages were the same as the previous sample. The pre-injection permeabilities were measured and are shown in Figs. 22 and 23. While injecting the fluid stages into the sample, all stages were pumped as planned.

Once the sample was allowed to cure for 24 hours, its retained permeability tested much lower than expected. The sample flowed slightly at a low rate with a very large pressure drop across the sample. For all intents and purposes, the retained permeability was 0%, but the actual value was calculated to be 0.3%. Considering the overflush was doubled, this was surprising. The most likely explanation was determined after examination of the sample. One complete half of the upper portion of the sample was a very dark blue throughout the sample, indicating residual dye from the epoxy system. This color gradually tapered off towards the bottom of the sample. After observing this and in conjunction with the retained permeability result, the conclusion was made that at the lower rate the overflush stage fingered through the lower portion of the sample. Despite pumping double the overflush, it appeared fingering dominated the retained permeability. The sample did however perform very well with respect to its UCS, giving a higher value of 977 psi. This was not surprising considering the very low retained permeability.

Considering the results of the four samples of the system to this point, there did not appear to be an issue placing the preflush and epoxy system in the sample, rather the removal of the overflush at the lower rate. To support this, at the higher overflush rate, the two samples gave 66.3% and 52.5%, and after lowering the rate, 25.0% and 0.3% retained permeabilities. For this reason, it was decided to place the preflush and epoxy systems at the lower rate, while increasing the rate in the overflush stage to its original value, for the next round of tests.

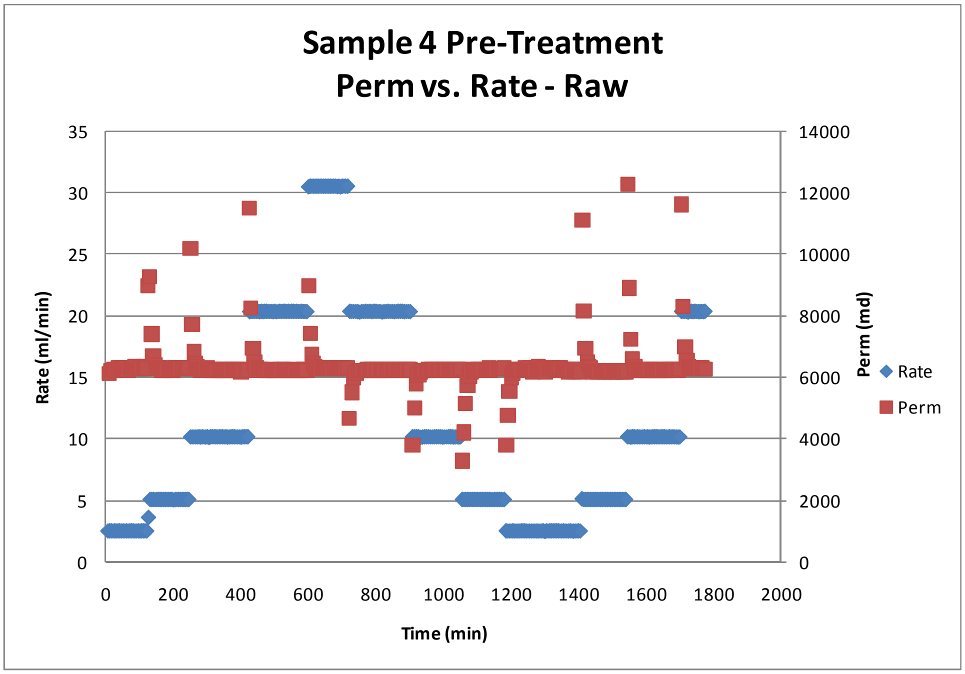


Fig. 22–Coreflood Sample 4 Pre-Treatment Permeability Plot – Raw Data.

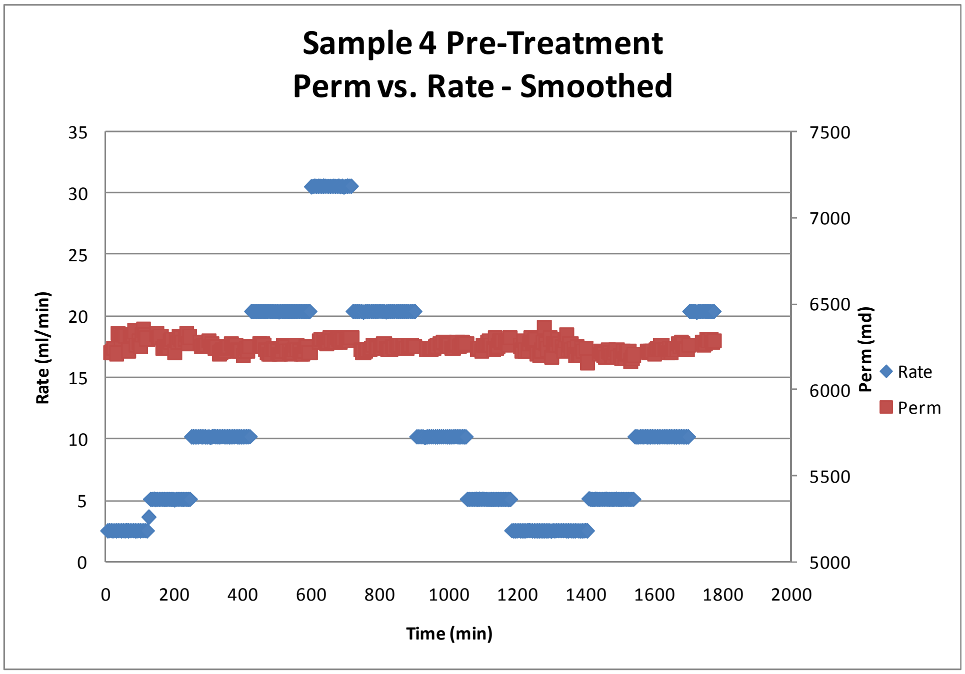


Fig. 23–Coreflood Sample 4 Pre-Treatment Permeability Plot – Smoothed Data.

3.2.5 Coreflood Sample 5 Results

The first adjustment for Sample 5 was previously mentioned; an increased overflush displacement rate. Also directly related to the results observed for the 12th round of syringe tests, Sample 5 incorporated the use of the additive PA2 in the preflush stage. As observed in the syringe tests, the addition of the new chemical additive to the preflush significantly improved the UCS results. So far in the coreflood tests, the correct combination of UCS and retained permeability had not been attained. The UCS results weren't particularly close to the results to the syringe tests, and had very low retained permeability. For this reason, PA2 was added to the preflush to obtain similar UCS values for the syringe and tests in the HP/HT Cell.

For Sample 5, the preflush and epoxy system were pumped at 2 ml/min, and then the rate was increased to 5 ml/min while pumping the overflush stage. The same relation of sample volume to stage volumes as previous rounds was kept constant. The pre-injection permeability measurements of the raw and smoothed data are given in Figs. 24 and 25. Following the measurement of the pre-treatment permeability, the chemical stages were injected into the sample.

Following the sample's 24 hour cure time, the post-injection permeability was measured and is given in Figs. 26 and 27. The retained permeability comparison at each rate and overall average is given in Table 42. The sample's average retained permeability was 47.2% was decent, but still not as high as desired. The sample performed very well with respect to its UCS value; 2401 psi. Like the syringe tests, the influence of the additive PA2 to the preflush was quite significant. Because this was the first test of this system with the new preflush additive in the coreflood apparatus, it was decided to run the same system again to see if the same results were reproducible.

Table 42. Coreflood Sample 5 Permeability Data.

Rate	**Post-Perm**	**Pre-Perm**	**Retained Perm**
(ml/min)	(md)	(md)	(%)
30	3190	6817	46.8%
20	3045	6685	45.6%
10	2985	6512	45.8%
5	3042	6263	48.6%
2.5	3134	5962	52.6%
Smoothed	3041	6466	**47.0%**
Raw	3062	6460	**47.4%**

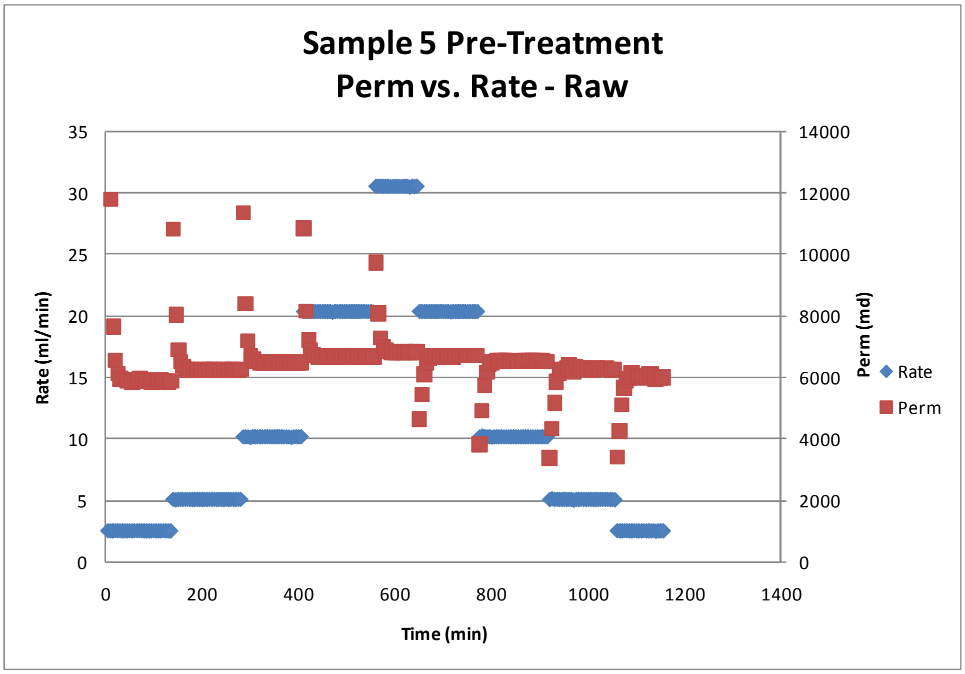


Fig. 24–Coreflood Sample 5 Pre-Treatment Permeability Plot – Raw Data.

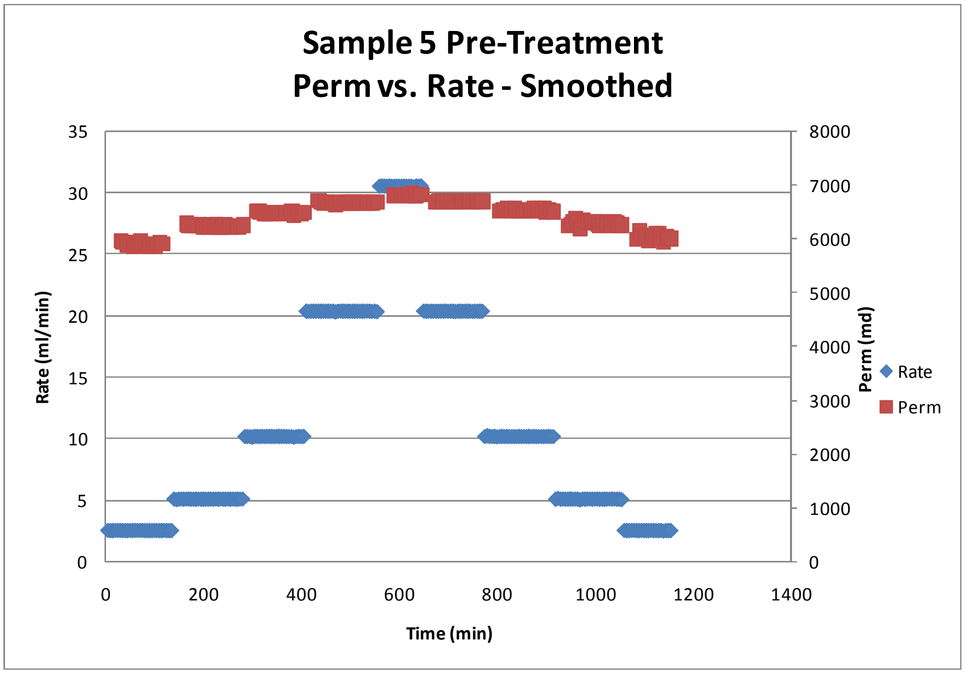


Fig. 25–Coreflood Sample 5 Pre-Treatment Permeability Plot – Smoothed Data.

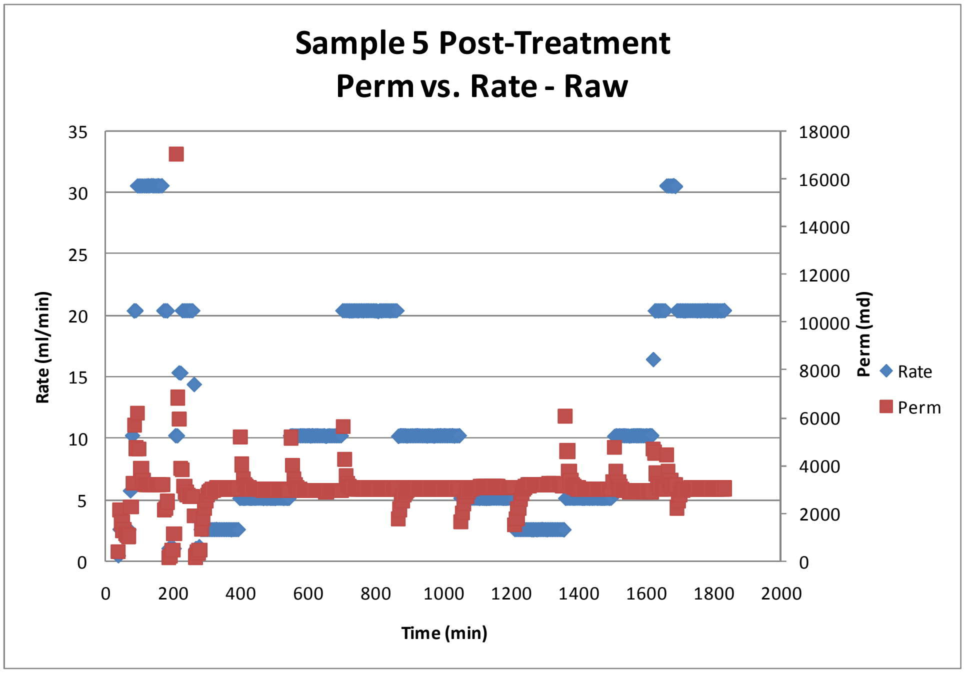


Fig. 26–Coreflood Sample 5 Post-Treatment Permeability Plot – Raw Data.

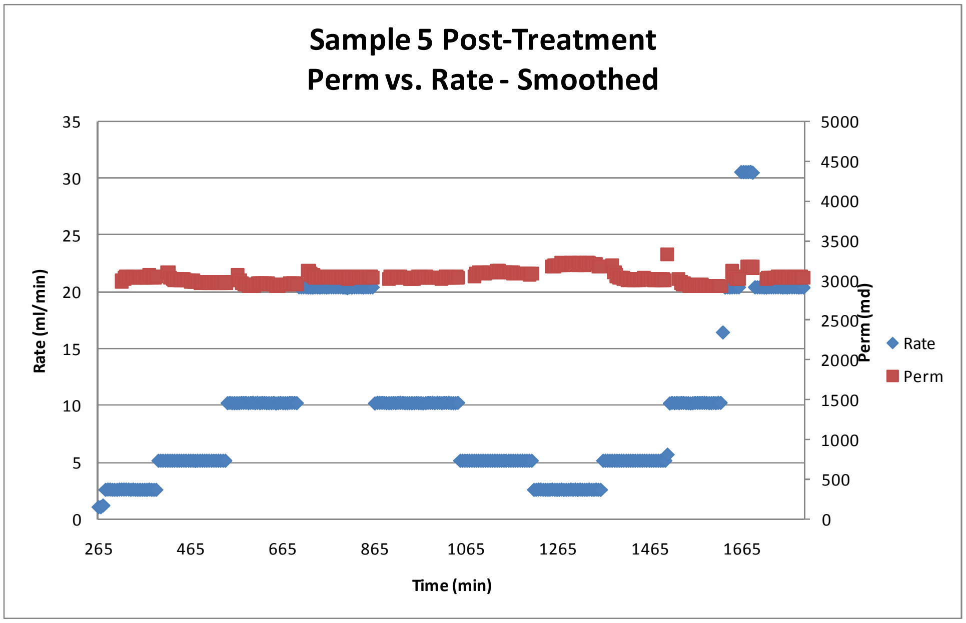


Fig. 27–Coreflood Sample 5 Post-Treatment Permeability Plot – Smoothed Data.

3.2.6 Coreflood Sample 6 Results

As mentioned in the previous section, the purpose of Sample 6 was to attempt to duplicate the results observed with Sample 5. All conditions were the same as described in the previous section. Figs. 28 and 29 show the raw and smoothed pre-injection permeability measurements. All fluid volumes were injected as planned, but a pressure increase was observed while injecting the overflush of this sample. For the previous stage, a slight pressure increase (1 psi) was noticed while injecting the overflush. This was normal, as the epoxy is slightly more viscous that than overflush, and eventually dropped to zero psi. However, while injecting the overflush for Sample 6 the pressure read 2 psi, and gradually decreased to 1 psi, then zero right before the end of the overflush stage.

The retained permeability utilizing the data from Figs. 30 and 31 and Table 43 was 26%. Although there was no visual evidence within the sample to explain the decrease in permeability, the explanation came from the pressure observations while pumping the overflush stage. The pressures observed during the overflush stage did show that there was difficulty removing the epoxy from the pore throats. Although there was no clear explanation why this happened on this sample and not the previous, it does explain the decrease in retained permeability.

The UCS for this sample was 1444 psi. Comparing the retained permeability of Samples 5 and 6, the expectation was that the UCS for Sample 6 would have been higher than that of Sample 5. The most likely explanation is the timing of testing of the UCS of the respective samples. Meaning, all samples to this point when removed, its UCS value was tested within a few hours. However, due to logistical issues, Sample 5 was not tested for an additional 60 hrs after it was removed. While Sample 5 was refrigerated to negate any additional curing, it appears the sample continued to cure despite refrigeration. While the two samples with the new preflush system yielded slightly different results, the reasoning behind these differences appeared to be explained by the observations/actions mentioned.

Table 43. Coreflood Sample 6 Permeability Data.

Rate	**Post-Perm**	**Pre-Perm**	**Retained Perm**
(ml/min)	(md)	(md)	(%)
30	1527	5315	28.7%
20	1487	5184	28.7%
10	1447	5073	28.5%
5	1296	4900	26.4%
2.5	1176	4619	25.5%
Smoothed	1300	5003	**26.0%**
Raw	1317	5076	**25.9%**

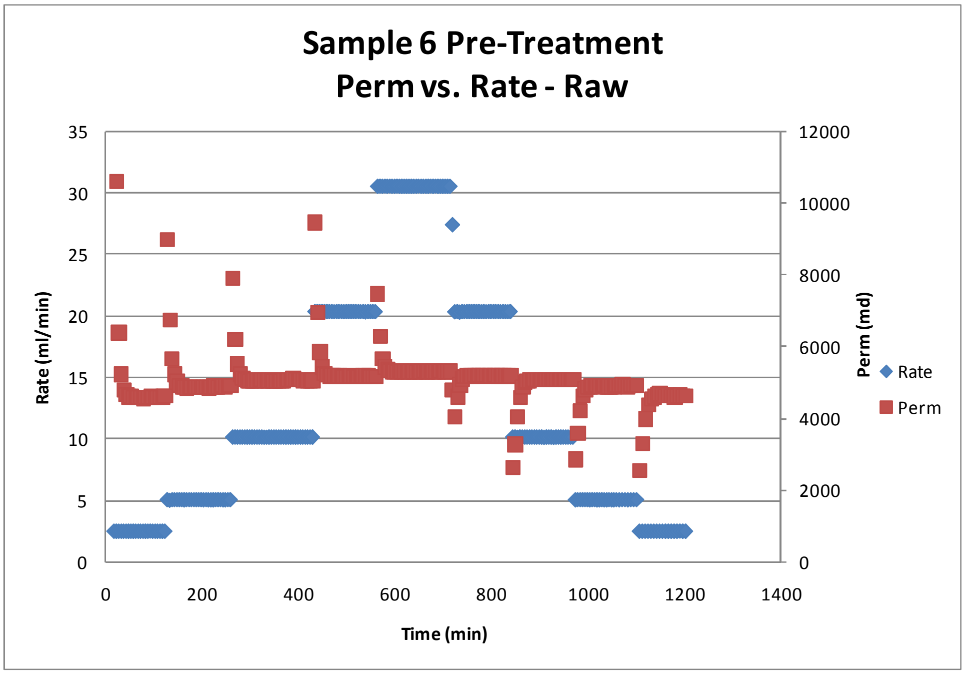


Fig. 28–Coreflood Sample 6 Pre-Treatment Permeability Plot – Raw Data.

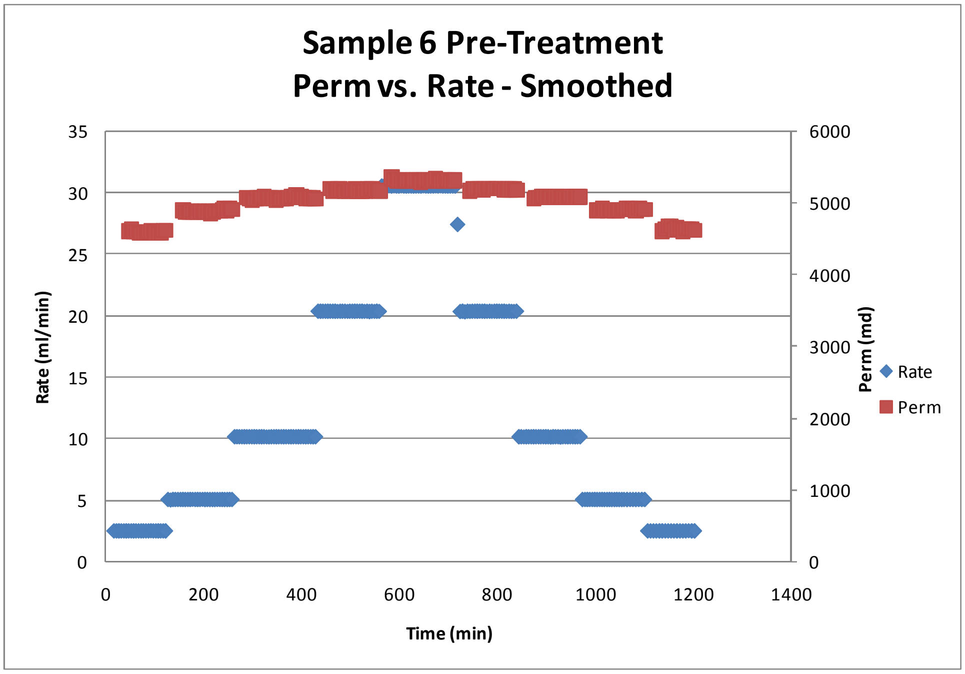


Fig. 29–Coreflood Sample 6 Pre-Treatment Permeability Plot – Smoothed Data.

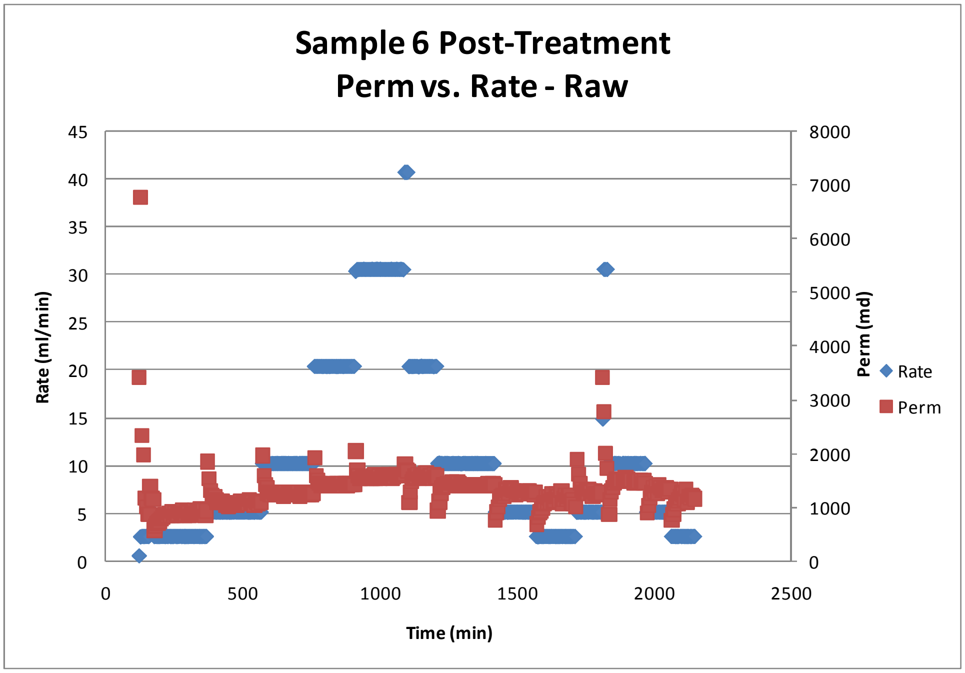


Fig. 30–Coreflood Sample 6 Post-Treatment Permeability Plot – Raw Data.

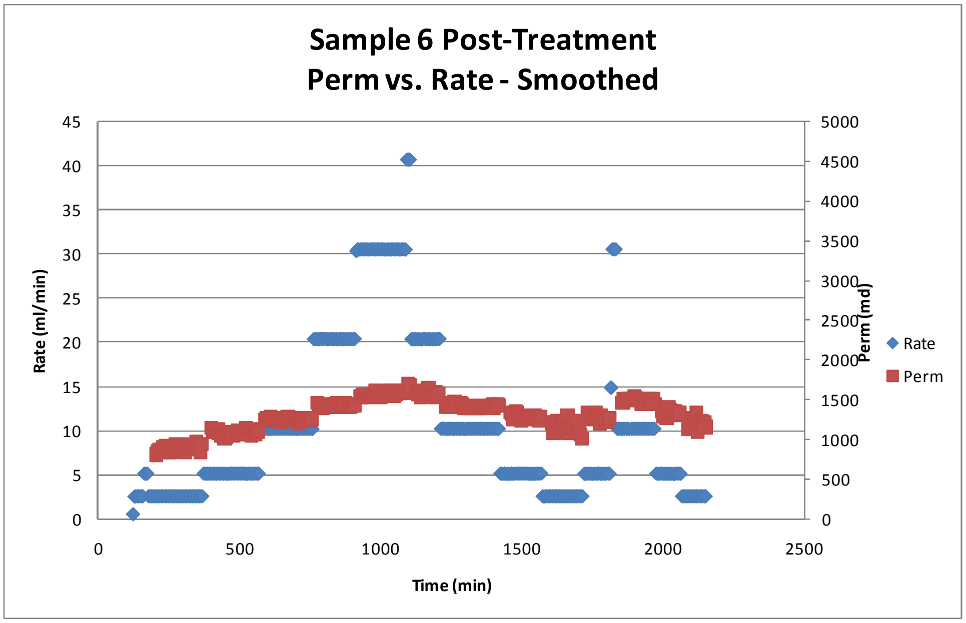


Fig. 31–Coreflood Sample 6 Post-Treatment Permeability Plot – Smoothed Data.

3.2.7 Coreflood Sample 7 Results

While in Sample 4 the additional overflush volume did not increase the retained permeability, it was pumped at the lower rate. Since the overflush stage's rate had been increased, the purpose of Sample 7 was to investigate the effect of doubled overflush at the higher injection rate. All other variables, preflush and epoxy system volumes and rates, were the same as the Samples 5 and 6. The raw and smoothed pre-injection permeability measurements for this sample are shown in Figs. 32 and 33. Because of the pressure observations made on the two previous samples, the coreflood system was modified slightly so that the injection pressure of the overflush fluid could be monitored and recorded more accurately. The pressure as a function of injection time of the overflush fluid is shown in Fig. 34. While the overflush volume was chosen as twice the previous, after approximately 5 PV of overflush, the pressure differential leveled off, as can be seen on the figure.

After the sample was allowed to cure, the post cure injection of the sample was completed. The retained permeabilities are shown in Table 44, as calculated from Figs. 35 and 36. The retained permeability of the sample was 55.9%. Compared to the two previous samples, this was a noticeable increase in retained permeability. When the sample was taken to test the UCS value, one more additional test was completed. The sample was cut in half to see if there is a clear difference in UCS values of the portion closest to the inlet or outlet. The UCS value for the inlet portion was 1266 psi and 1360 psi for the outlet portion, giving an average of 1313 psi. While there is a slight difference between the two, it was not very substantial. Both visually and by UCS results, it appears the sample is homogenously treated. Finally, while the retained permeability increased compared to the average of the two previous samples, the decrease in the UCS measurement with the doubled overflush was not as substantial. The decrease in UCS was approximately 31.7% comparing the average of the previous two samples, 1922.5 psi, to the UCS of this sample, 1313 psi. Comparatively, the retained permeability of Sample 7 was 55.9%, compared to the 36.6% average of the previous samples; a 52.7% increase over the average of the other samples. This observation shows that for these conditions, the system is slightly affected by the overflush volume, keeping its strength while yielding a better retained permeability.

Table 44. Coreflood Sample 7 Permeability Data.

Rate (ml/min)	**Post-Perm** (md)	**Pre-Perm** (md)	**Retained Perm** (%)
30	3197	6101	52.4%
20	3124	5985	52.2%
10	3109	5872	52.9%
5	3340	5672	58.9%
2.5	3427	5355	64.0%
Smoothed	3205	5838	**54.9%**
Raw	3288	5779	**56.9%**

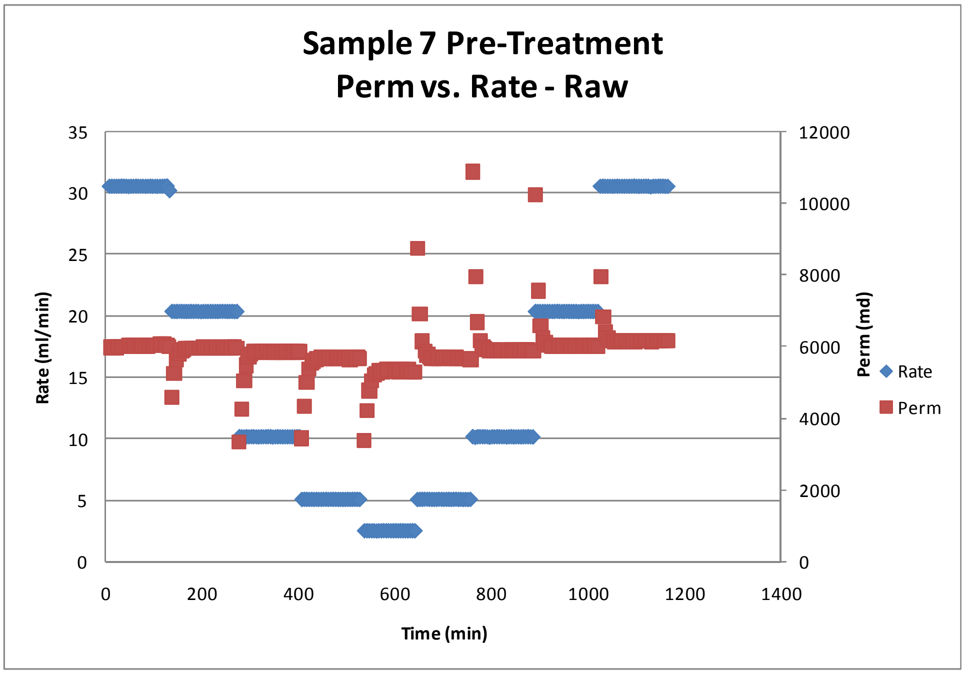


Fig. 32–Coreflood Sample 7 Pre-Treatment Permeability Plot – Raw Data.

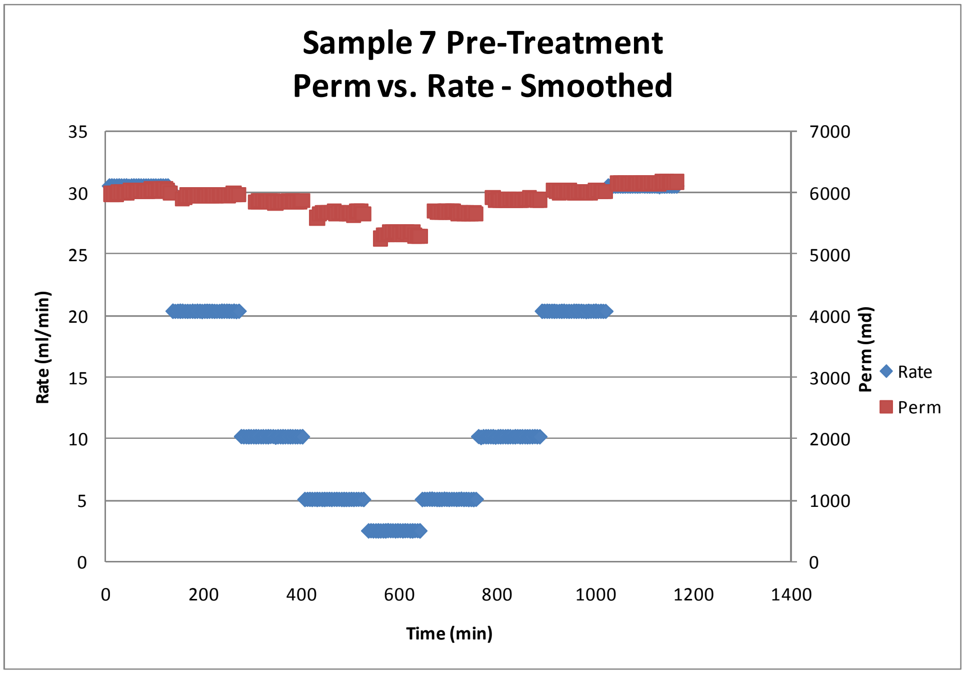


Fig. 33–Coreflood Sample 7 Pre-Treatment Permeability Plot – Smoothed Data.

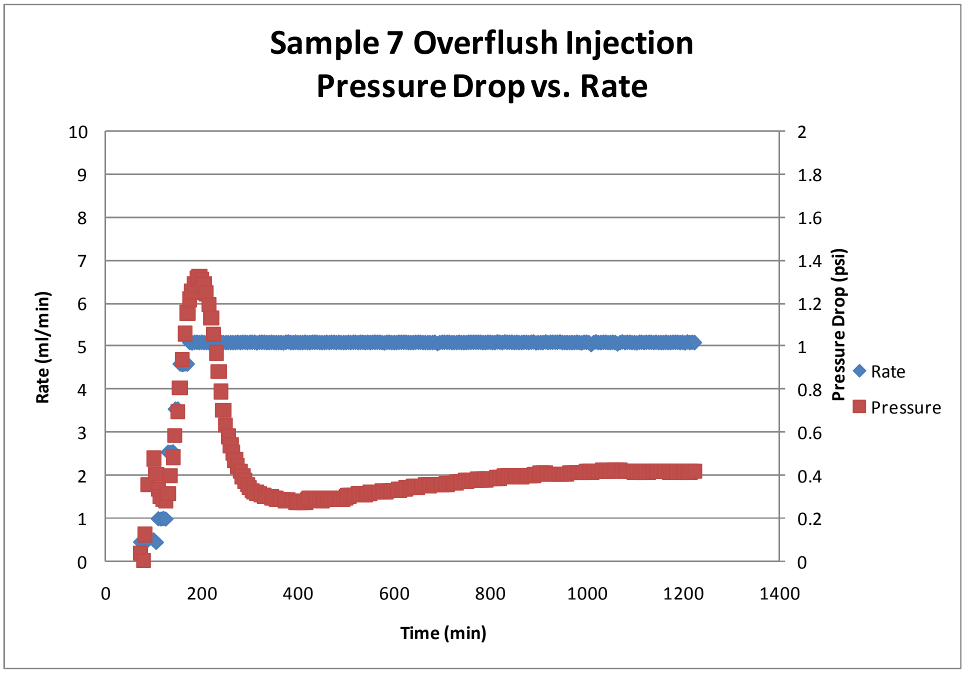


Fig. 34–Coreflood Sample 7 Overflush Injection.

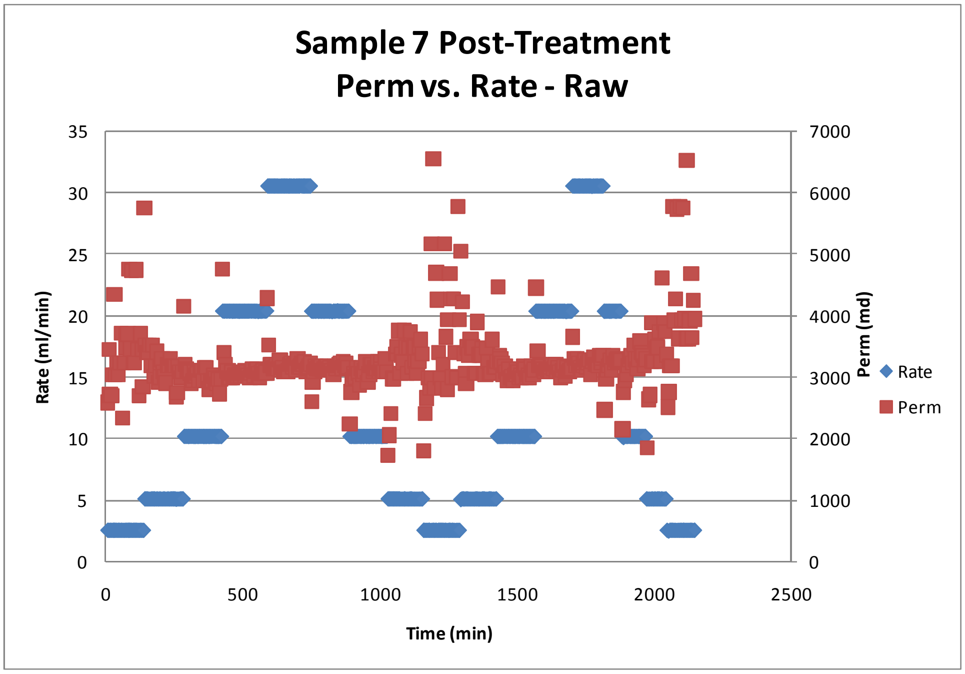


Fig. 35–Coreflood Sample 7 Post-Treatment Permeability Plot – Raw Data.

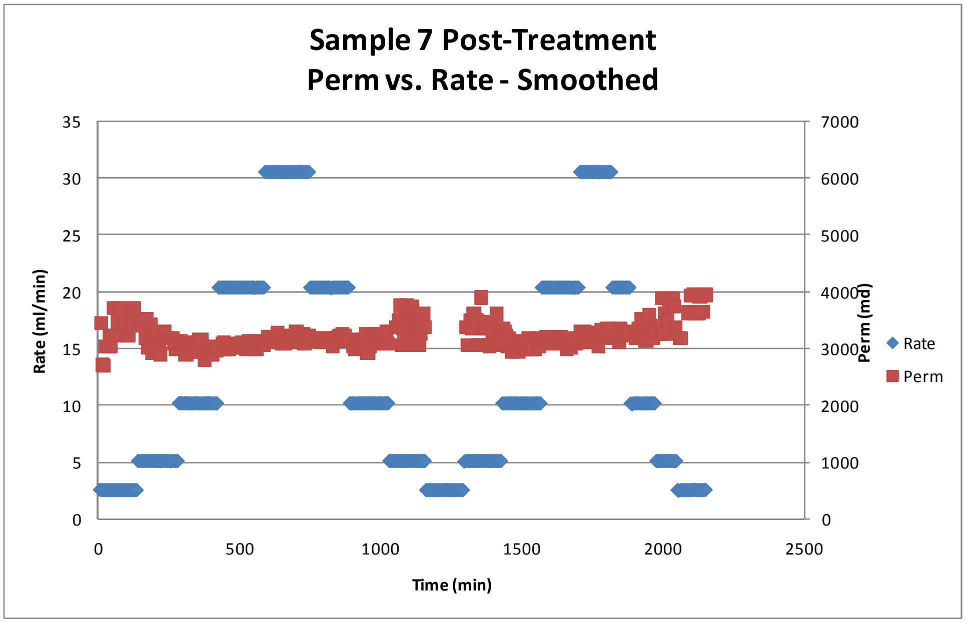


Fig. 36–Coreflood Sample 7 Post-Treatment Permeability Plot – Smoothed Data.

3.2.8 Coreflood Sample 8 Results

While positive results were observed from Sample 7, the desire was still to try to increase the retained permeability. Because of this, the objective of Sample 8 was to investigate the effect of injecting all the fluid stages at the high injection rate, 5 ml/min. Samples 1 and 2 were completed at the same conditions, however they did not have the additive PA2 in it, and since being added, very positives UCS results have been observed.

The retained permeability for this sample is given in Table 45. For Sample 8, the average retained permeability was 55.4% with a UCS value of 1683 psi. The respective raw and smoothed pre-injection plots are Figs. 37 and 38, and the raw and smoothed post-injection plots are Figs. 39 and 40. Compared to the 59.4% average of Samples 1 and 2, the retained permeability is of the same magnitude, while the UCS increased significantly. The results of the UCS and retained permeability values for Sample 8 were very promising, but again the goal was to have a sample with a slightly higher retained permeability. The plot of the overflush injection pressures is given in Fig. 41. For this reason, the next few rounds of tests were completed to see if an increased overflush volume could yield a positive increase in the retained permeability, while not significantly decreasing the UCS; as observed in Round 7. Keeping in mind, the goal of the system was to not to be greatly affected by the overflush volume. Finally, while there is a desire to increase the retained permeability, this system does yield good results currently.

Table 45. Coreflood Sample 8 Permeability Data.

Rate	**Post-Perm**	**Pre-Perm**	**Retained Perm**
(ml/min)	(md)	(md)	(%)
30	2919	5659	51.6%
20	2907	5570	52.2%
10	2926	5464	53.6%
5	2715	5273	51.5%
2.5	3577	4917	72.7%
Smoothed	2922	5421	**53.9%**
Raw	2944	5201	**56.6%**

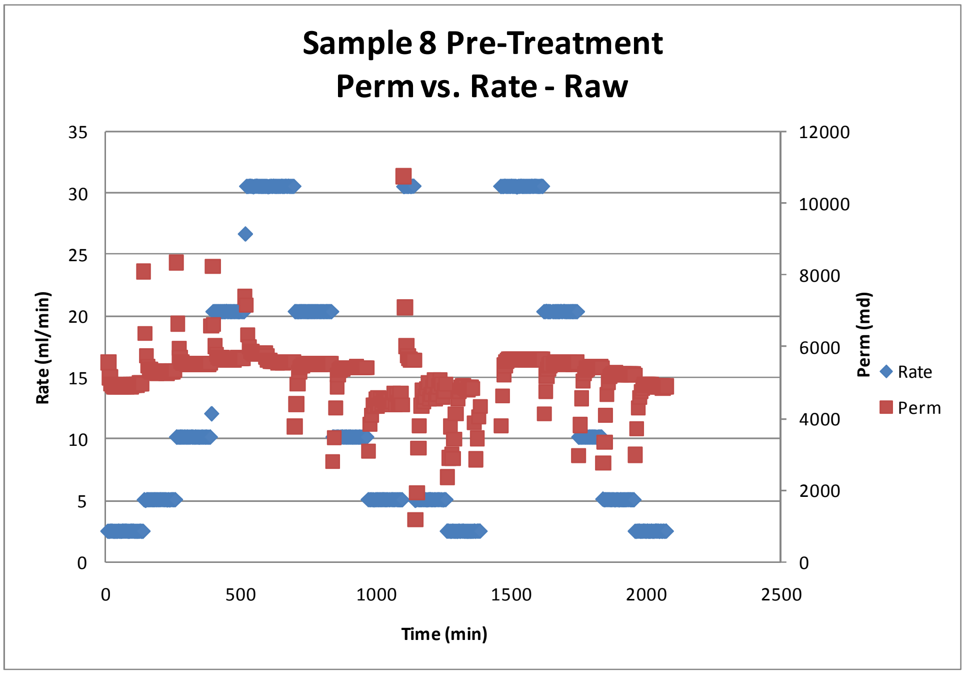


Fig. 37–Coreflood Sample 8 Pre-Treatment Permeability Plot – Raw Data.

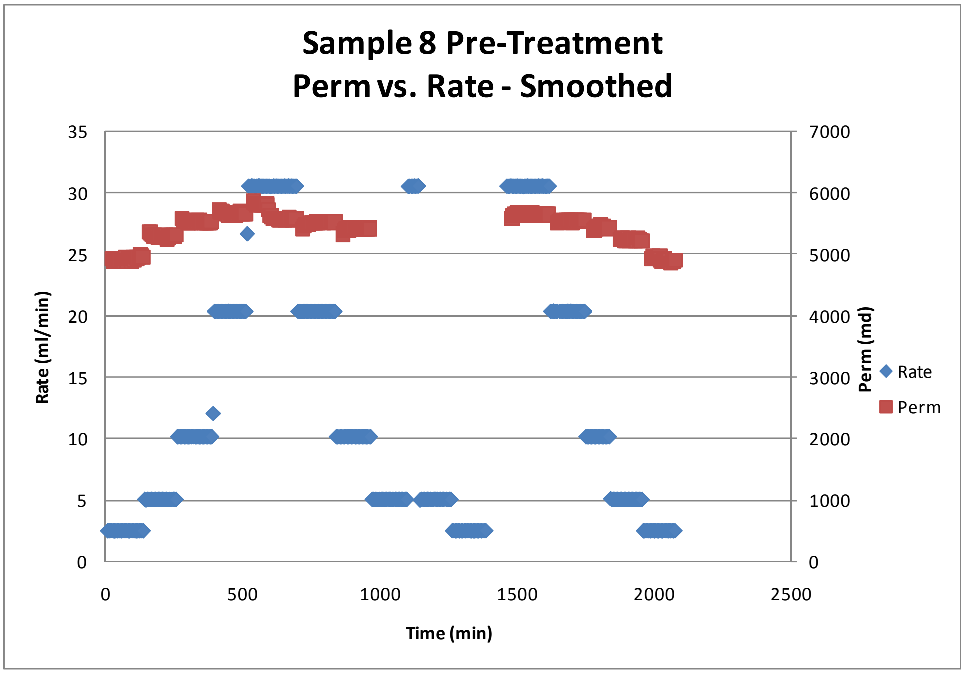


Fig. 38–Coreflood Sample 8 Pre-Treatment Permeability Plot – Smoothed Data.

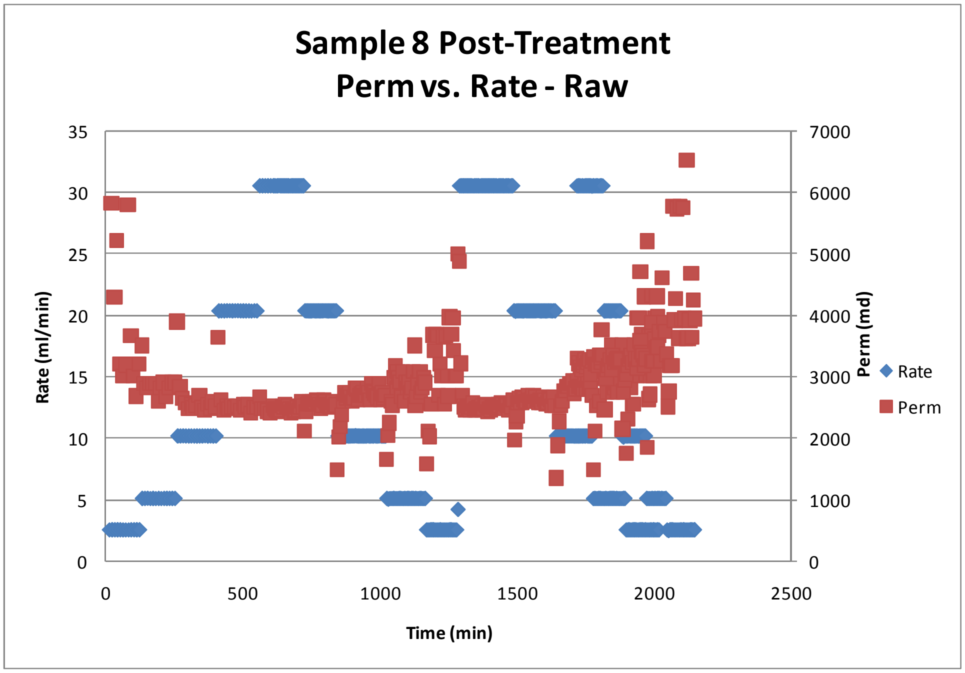


Fig. 39–Coreflood Sample 8 Post-Treatment Permeability Plot – Raw Data.

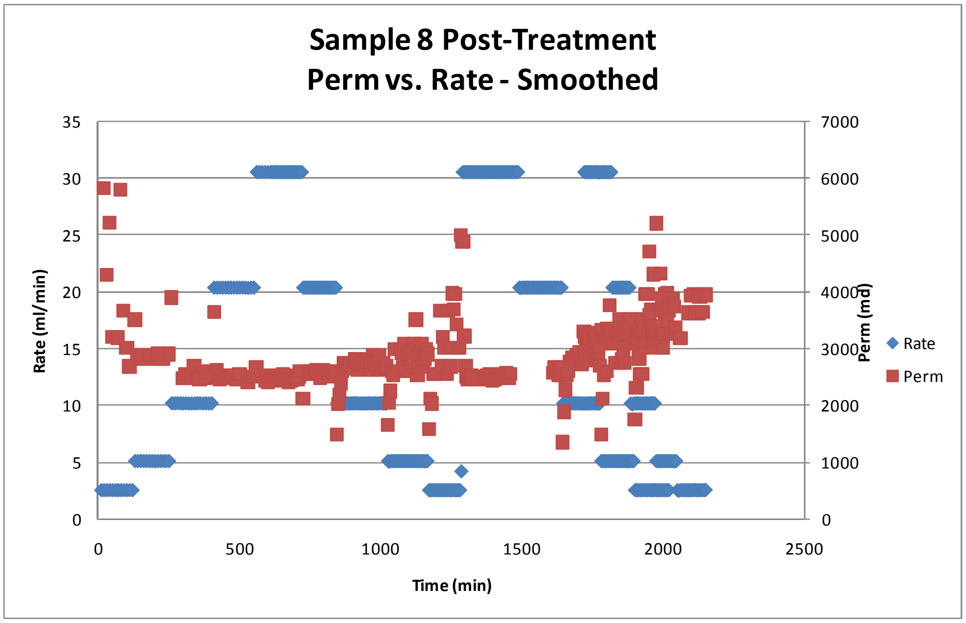


Fig. 40–Coreflood Sample 8 Post-Treatment Permeability Plot – Smoothed Data.

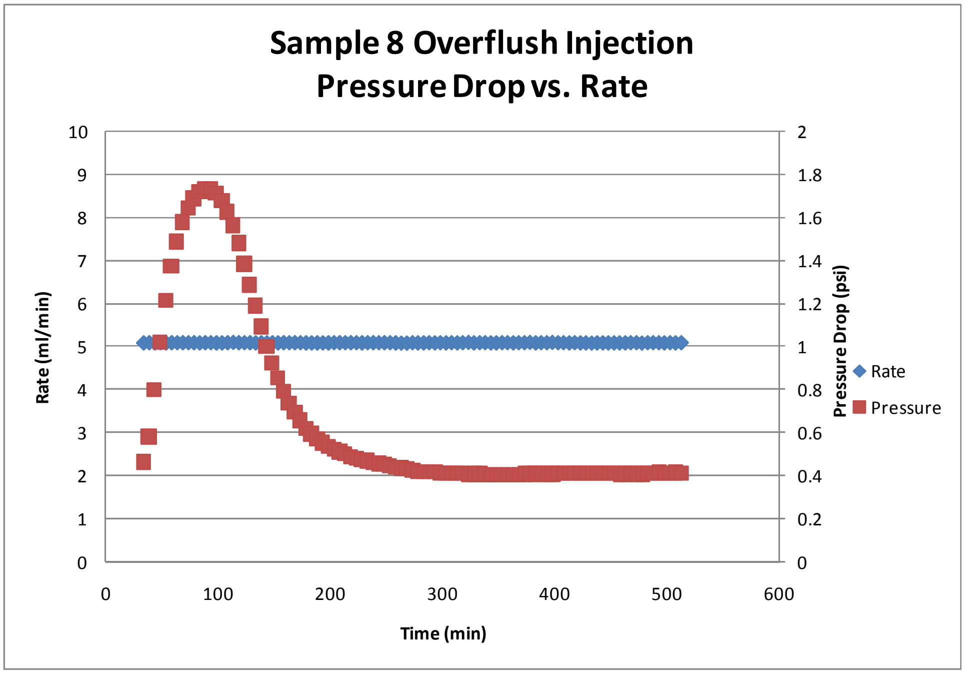


Fig. 41–Coreflood Sample 8 Overflush Injection.

3.2.9 Coreflood Sample 9 Results

Sample 9 was prepared in the same manner as Sample 8, except that the overflush volume was doubled from the previous round. While a higher retained permeability was desired, a more robust system in which overflush volume does not greatly affect performance of the fluid was equally as important. The pre-injection permeability plots and the respective data is given in Figs. 42 and 43, and Table 46. The overflush injection is also given in Fig. 44.

The average retained permeability for this sample was 50.8%. The raw and smoothed post-injection permeability plots are given in Figs. 45 and 46. Considering double the amount of overflush was pumped, this was surprising, as Sample 8 yielded 55.4%. At the same time, the UCS value decreased from 1683 psi to 1484 psi. While the decrease in UCS is the same behavior observed with most systems on the market, this normally would correspond to an increase in retained permeability. After examining the sample, there was no information to explain the decrease in the retained permeability. Despite this decrease, both the UCS and retained permeability are of the same magnitude. Because there was no obvious information available to explain, it was assumed this difference was due to inherent experimental error.

Table 46. Coreflood Sample 9 Permeability Data.

Rate	**Post-Perm**	**Pre-Perm**	**Retained Perm**
(ml/min)	(md)	(md)	(%)
30	2605	4900	53.2%
20	2503	4796	52.2%
10	2489	4729	52.6%
5	2545	4620	55.1%
2.5	2588	4451	58.1%
Smoothed	2540	5000	**50.8%**
Raw	2541	5009	**50.7%**

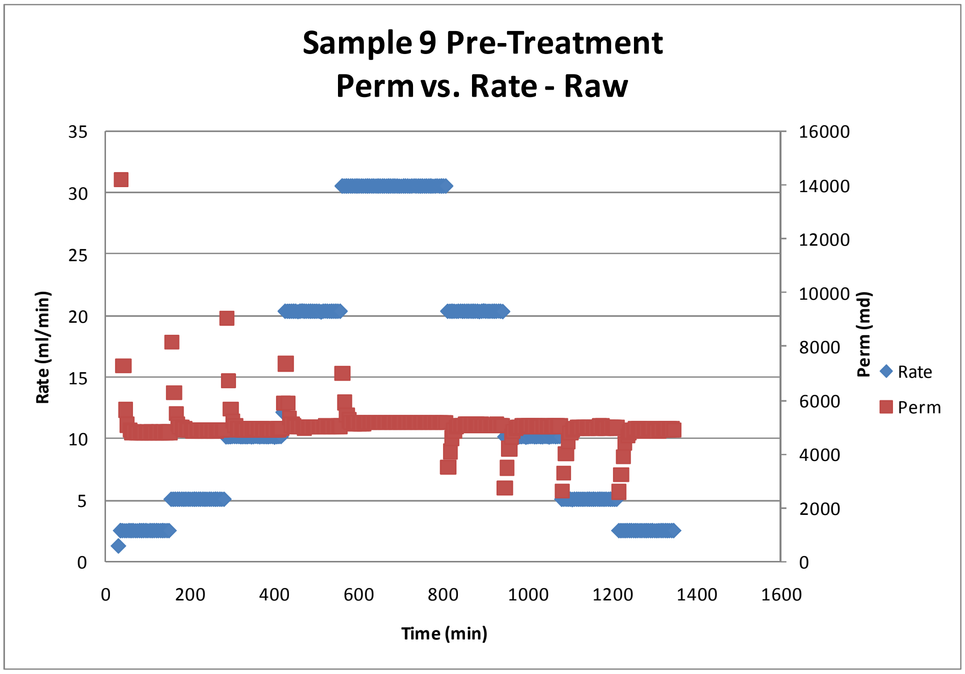


Fig. 42–Coreflood Sample 9 Pre-Treatment Permeability Plot – Raw Data.

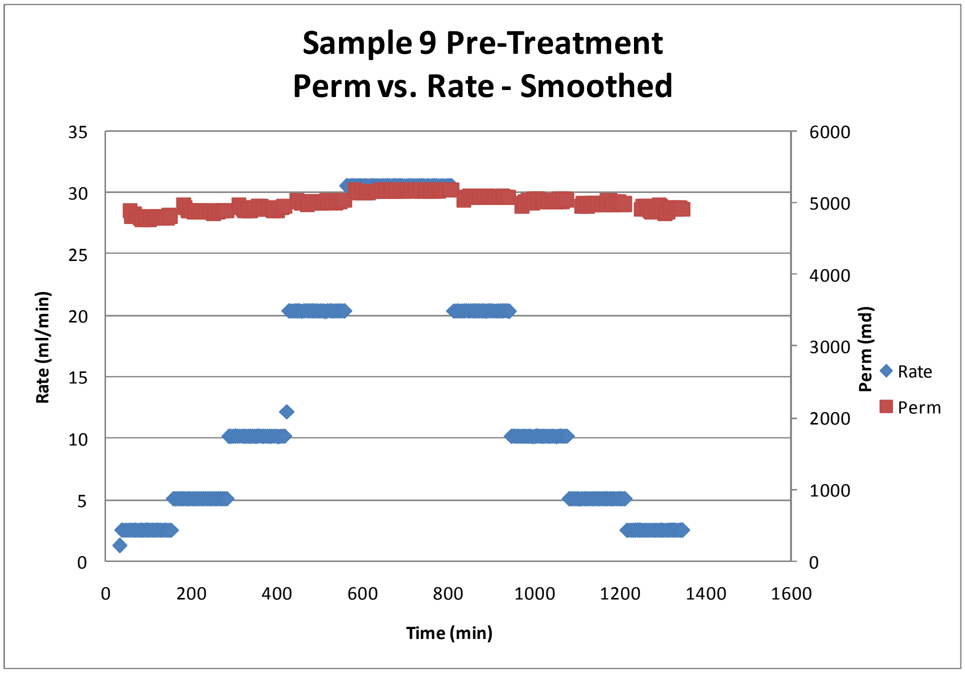


Fig. 43–Coreflood Sample 9 Pre-Treatment Permeability Plot – Smoothed Data.

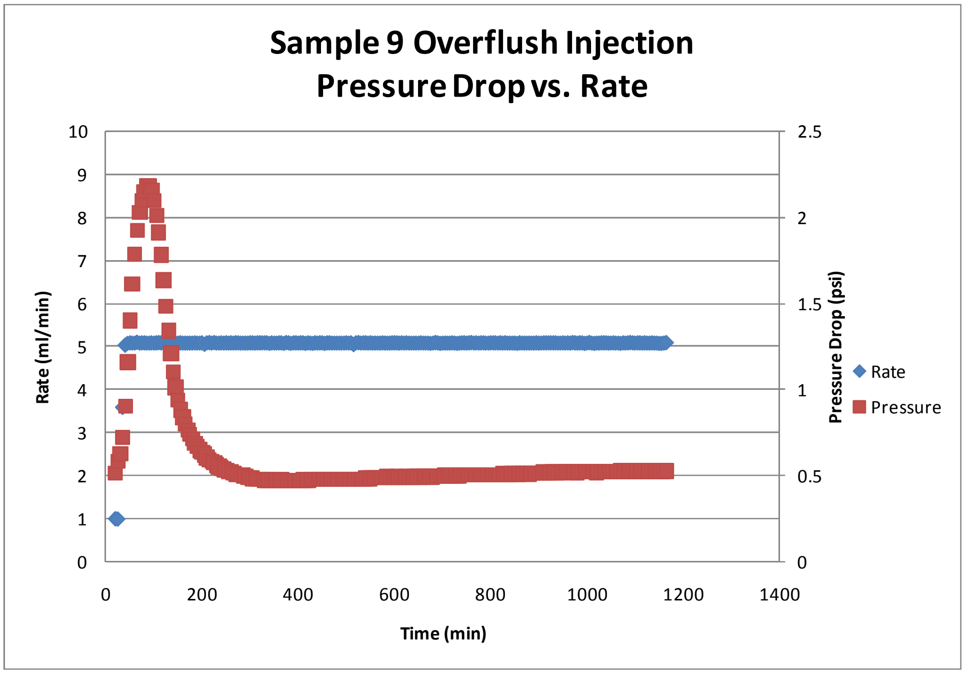


Fig. 44–Coreflood Sample 9 Overflush Injection.

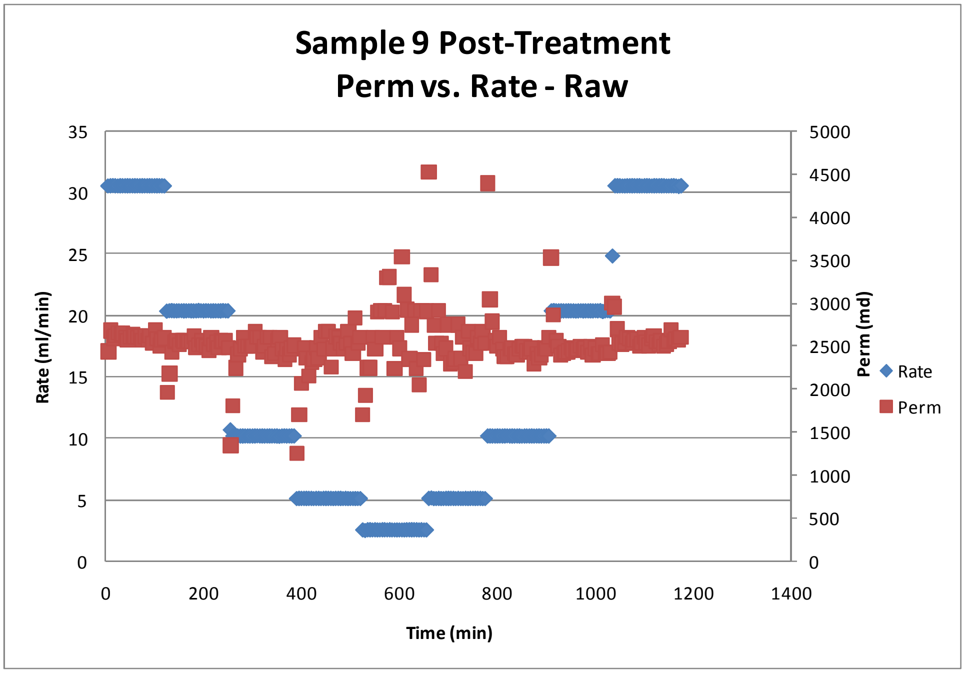


Fig. 45–Coreflood Sample 9 Post-Treatment Permeability Plot – Raw Data.

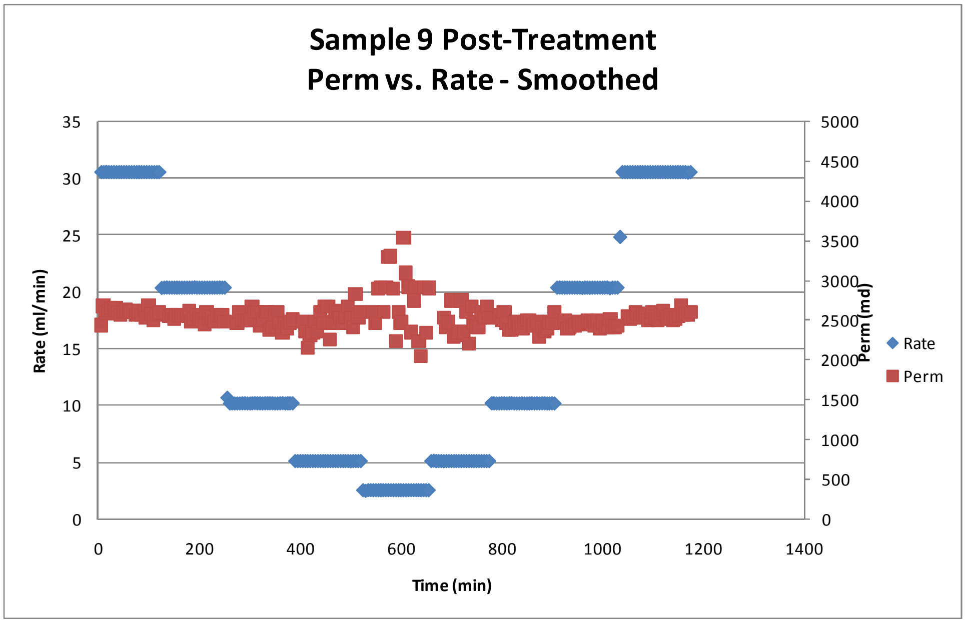


Fig. 46–Coreflood Sample 9 Post-Treatment Permeability Plot – Smoothed Data.

3.2.10 Coreflood Sample 10 Results

While a trend was seen between Samples 8 and 9, an additional data point was required to see the system's overall performance with the volume of overflush. For this reason, Sample 10 had the same conditions as the previous two tests except that the overflush was once again doubled from the previous round. This corresponds to four times the initial overflush volume. The sample's pre-treatment data is given in Table 47, as calculated from Figs. 47 and 48. Once again, while injecting the overflush the pressure appeared to level off and became constant prior to reaching the end of the overflush stage. This is shown in Fig. 49.

After the sample cured, it's UCS and retained permeability were tested. The retained permeability was 35.8% with a UCS of 1089 psi. The same relationship from Sample 8 to 9 was observed for this sample; namely both the UCS and retained permeability decreased with an increased overflush volume. The sample's post-injection permeability plots are given in Figs. 50 and 51. This was not the first time when all stages were injected at the same conditions, and the retained permeability decreased with increased overflush volume. The same trend was observed previously with Samples 3 and 4. It was not clear why this trend was observed in Samples 3, 4, 8, 9, or 10. To gain a better understanding of this system, it was decided to examine the samples using SEM.

Based upon these photos and the fact that the epoxy system is hydrophilic, the conclusion was made that as additional overflush was pumped through the sample the hydrophilic components were preferentially removed. By this happening, the stable system which was initially created was disturbed and the epoxy's adhesion to the sand grains was altered. This can be seen in the SEM photos in Figs. 52a-d, in which 52a and b are Sample 8 and 52c and d are Sample 10. There is noticeably more epoxy left in the pore spaces in the latter two photos. One final supporting piece of information to this conclusion lies in the pressure drop data from the overflush injections of the three samples. When the overflush injection starts, the pressure gradually increased until it eventually decreased. As previously explained, this is due to the difference in viscosity, as well as the overflush finding the preferential path within the pack. Next, this pressure drop leveled off for a short period, and then as observed in all the samples increased very slightly for all three samples. This shows that the pressure drop across the sample increases with an increased overflush volumes which decreases retained permeability.

Table 47. Coreflood Sample 10 Permeability Data.

Rate	**Post-Perm**	**Pre-Perm**	**Retained Perm**
(ml/min)	(md)	(md)	(%)
30	1710	4888	35.0%
20	1707	4787	35.7%
10	1679	4739	35.4%
5	1662	4711	35.3%
2.5	1791	4691	38.2%
Smoothed	1709	4766	**35.8%**
Raw	1708	4791	**35.7%**

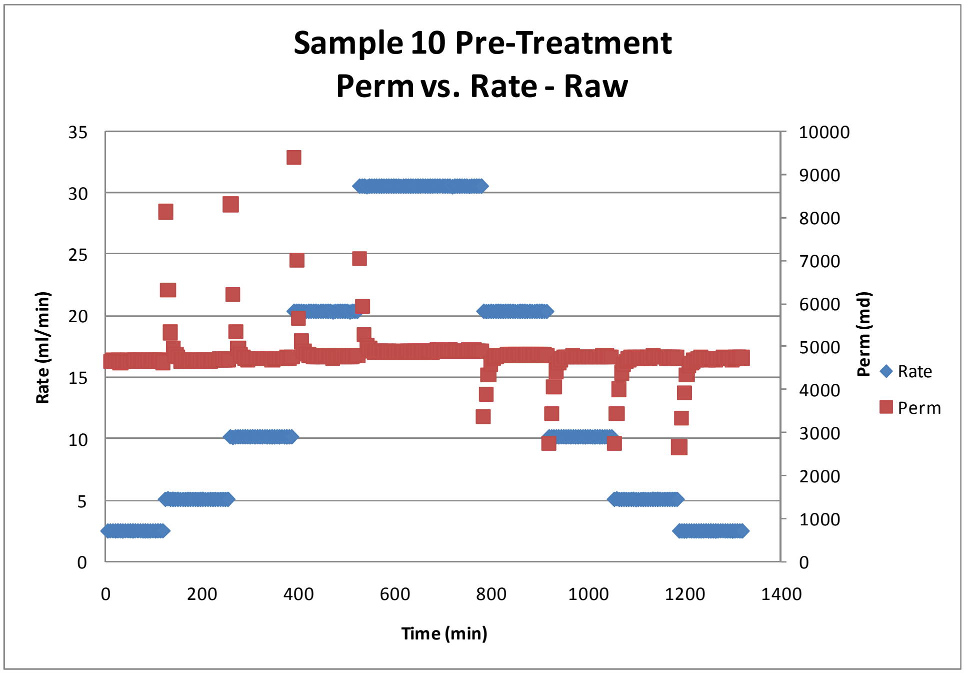


Fig. 47–Coreflood Sample 10 Pre-Treatment Permeability Plot – Raw Data.

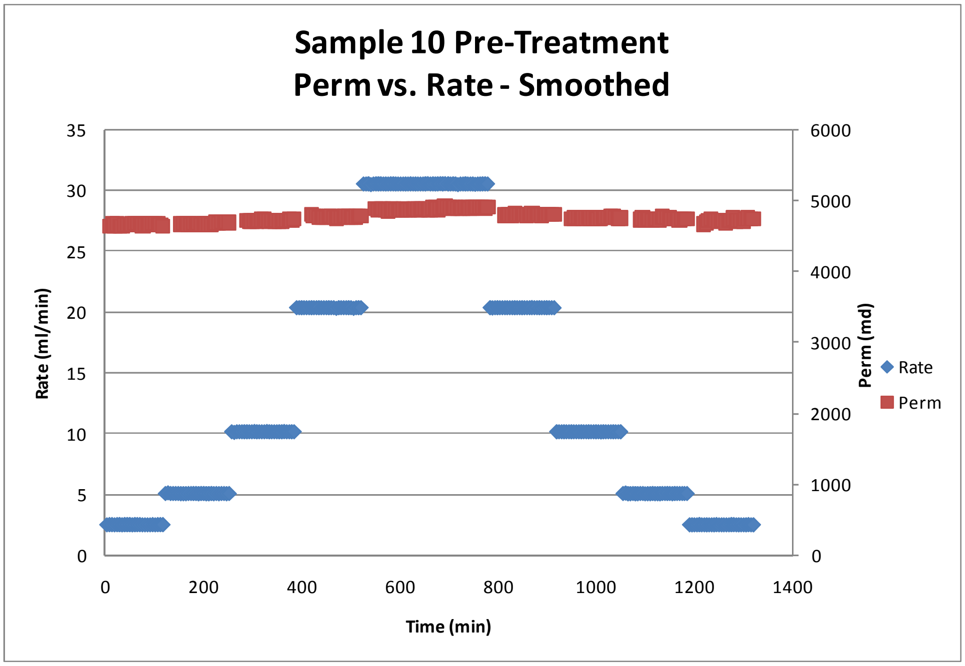


Fig. 48–Coreflood Sample 10 Pre-Treatment Permeability Plot – Smoothed Data.

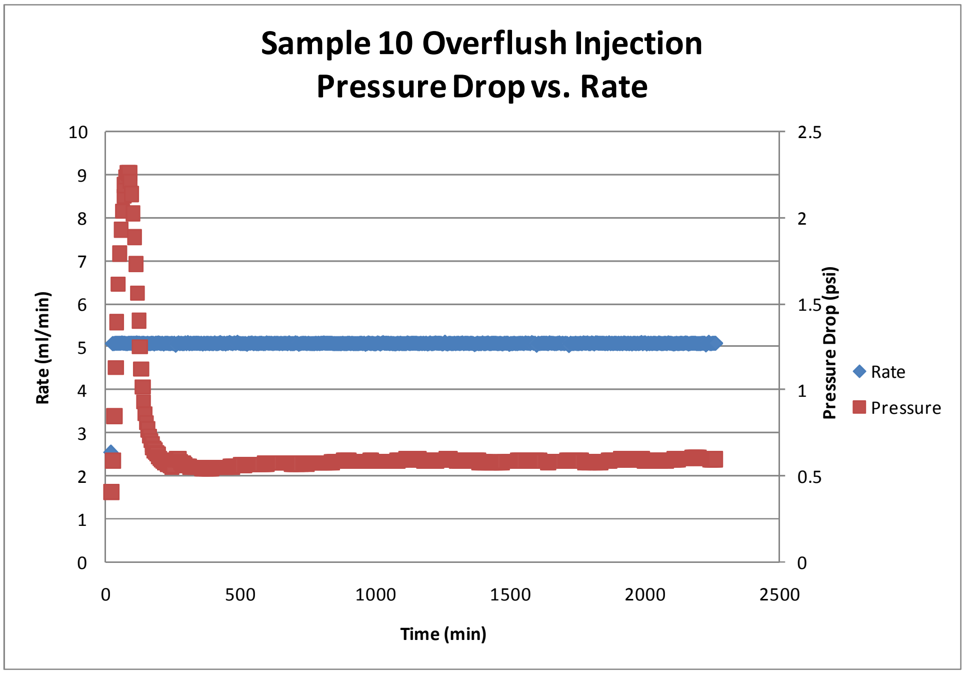


Fig. 49–Coreflood Sample 10 Overflush Injection.

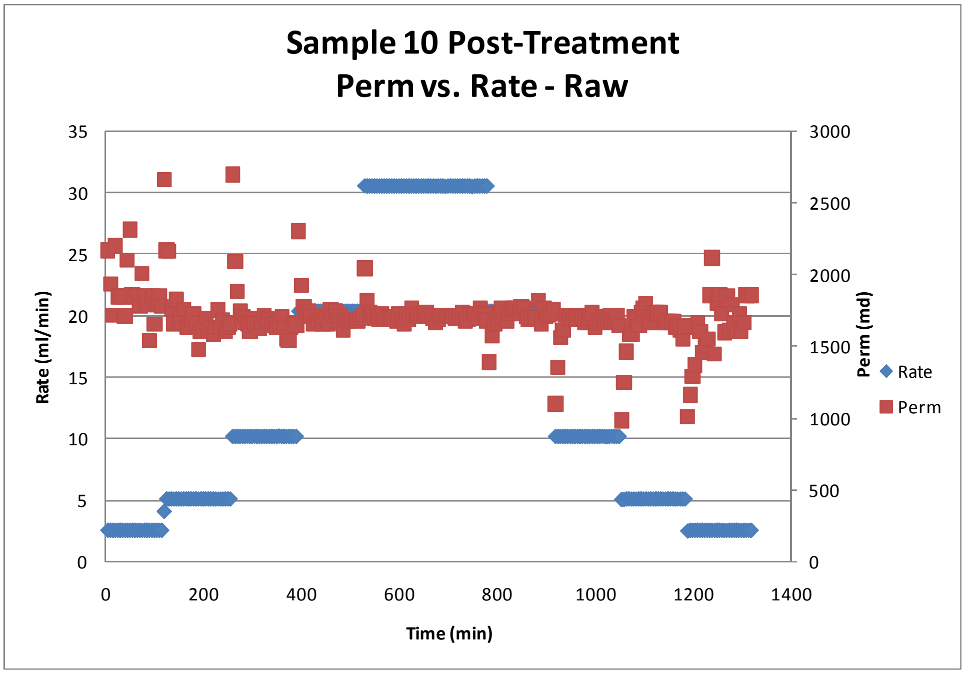


Fig. 50–Coreflood Sample 10 Post-Treatment Permeability Plot – Raw Data.

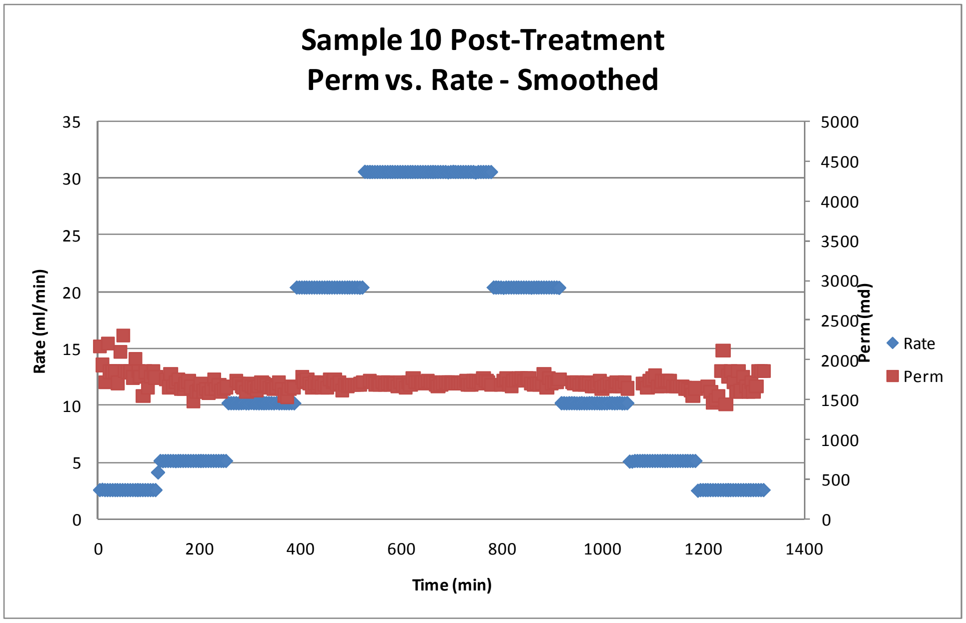


Fig. 51–Coreflood Sample 10 Post-Treatment Permeability Plot – Smoothed Data.

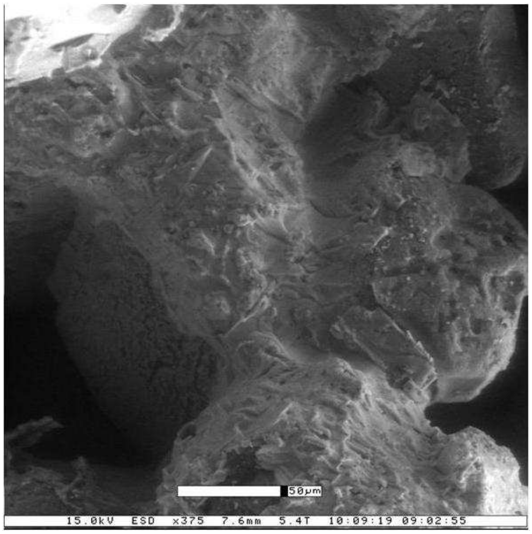


Fig. 52a–Sample 8 SEM Photo.

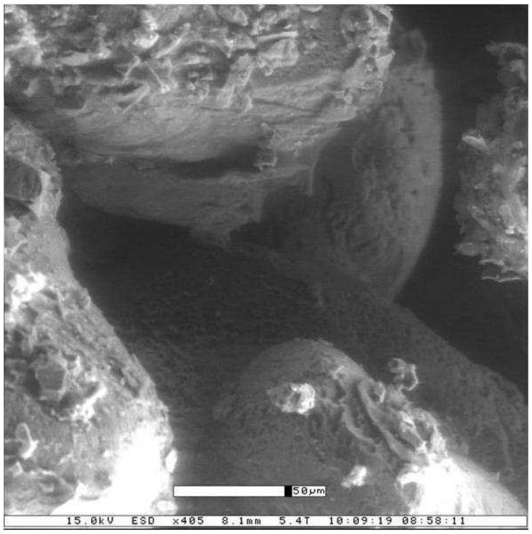


Fig. 52b–Sample 8 SEM Photo.

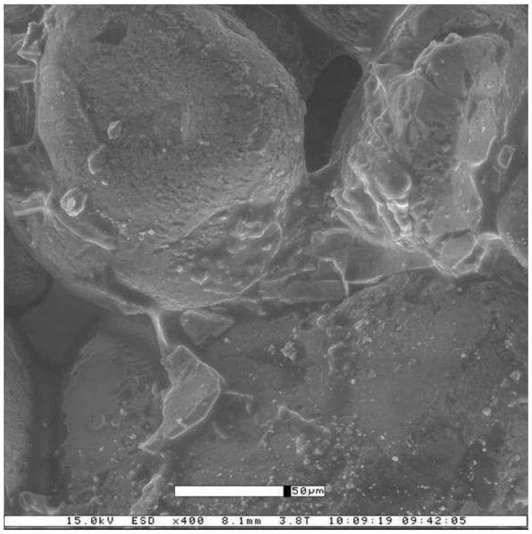


Fig. 52c–Sample 10 SEM Photo.

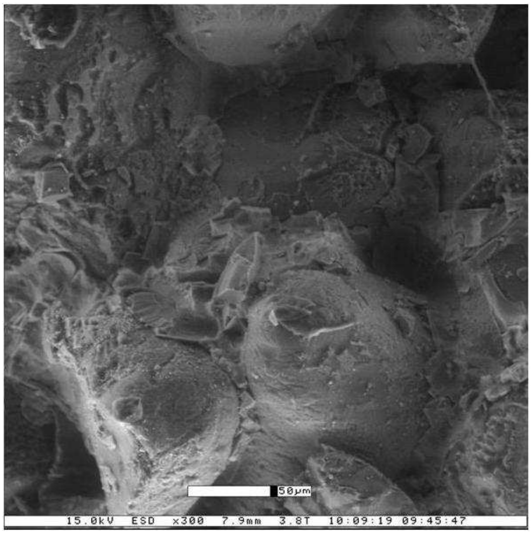


Fig. 52d–Sample 10 SEM Photo.

Fig. 52–Coreflood Samples SEM Photos.

3.2.11 Coreflood Sample 11 Results

Sample 11 was the last test completed on the system in the HP/HT cell. The goal for the eleventh round was to observe the system's performance at a higher temperature, 200°F. The other conditions for this sample were that the injections of all the stages were at the higher injection rate of 5 ml/min, and the overflush volume was doubled (6 PV). The pre-injection plots are Figs. 53 and 54, while the post-injection plots are Figs. 55 and 56.

The sample yielded very positive results after it was allowed to cure. The average UCS of the sample was 2122 psi, with a retained permeability of 59.5% as calculated in Table 48. The increase in UCS was expected as the system was at a higher temperature which allows the sample to cure much more rapidly. At the same time, as mentioned in the syringe test, the system performs well, but its working time for field applications at this higher temperature needs to be investigated further. The system has a working time of 8 hours before it develops any viscosity at 130°F, however at 200°F, the system was starting to show increased viscosity after 5 hours. Overall, the system performed very well at this increased temperature.

Table 48. Coreflood Sample 11 Permeability Data.

Rate	**Post-Perm**	**Pre-Perm**	**Retained Perm**
(ml/min)	(md)	(md)	(%)
30	2788	4900	56.9%
20	2721	4796	56.7%
10	2770	4729	58.6%
5	2832	4620	61.3%
2.5	2831	4451	63.6%
Smoothed	2785	4704	**59.2%**
Raw	2819	4709	**59.9%**

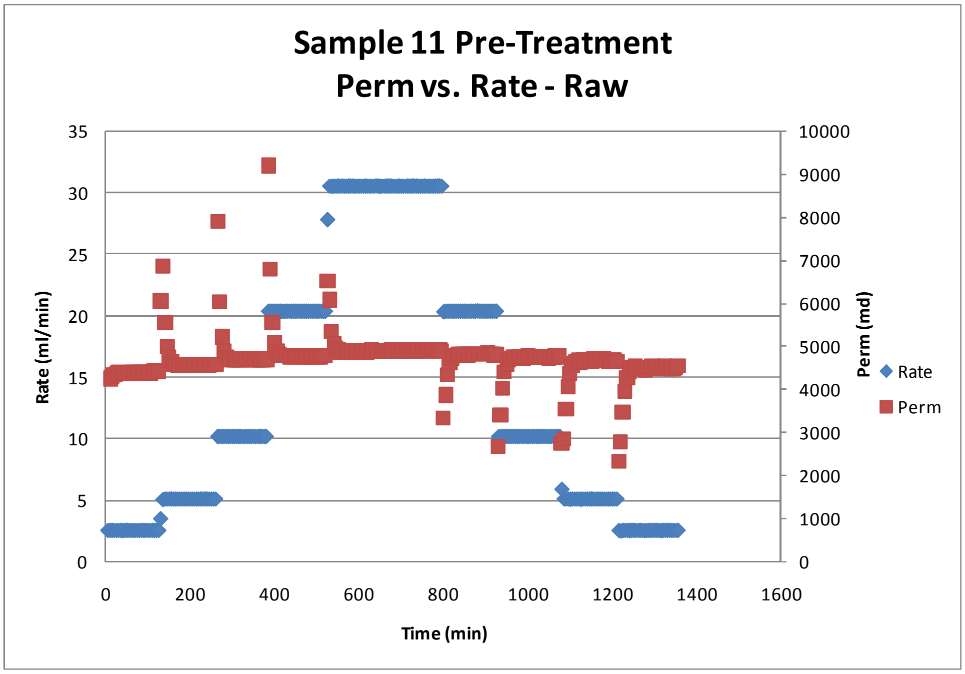


Fig. 53–Coreflood Sample 11 Pre-Treatment Permeability Plot – Raw Data.

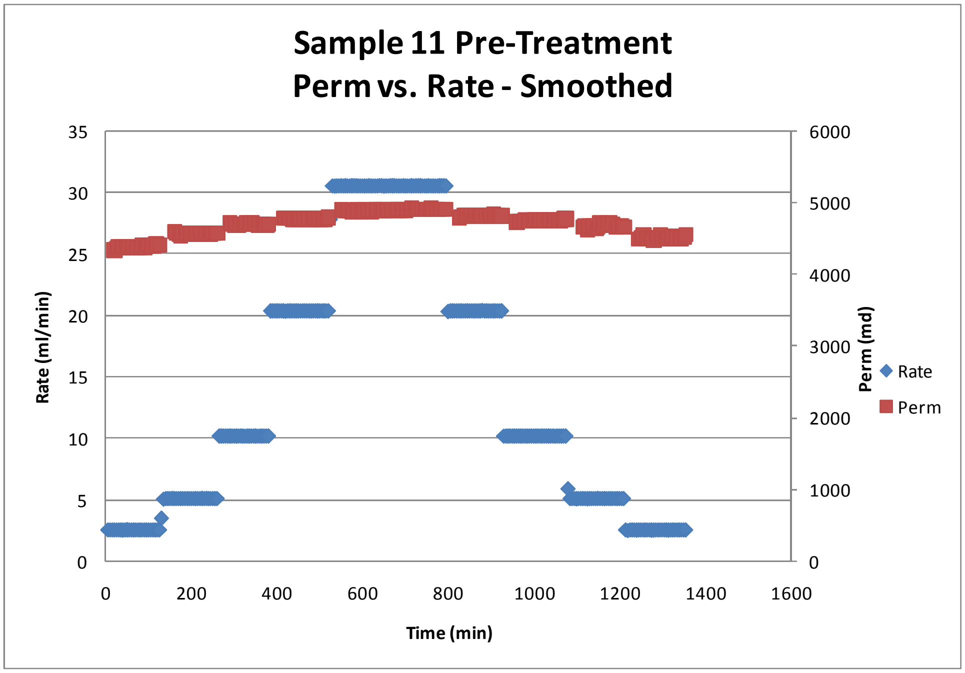


Fig. 54–Coreflood Sample 11 Pre-Treatment Permeability Plot – Smoothed Data.

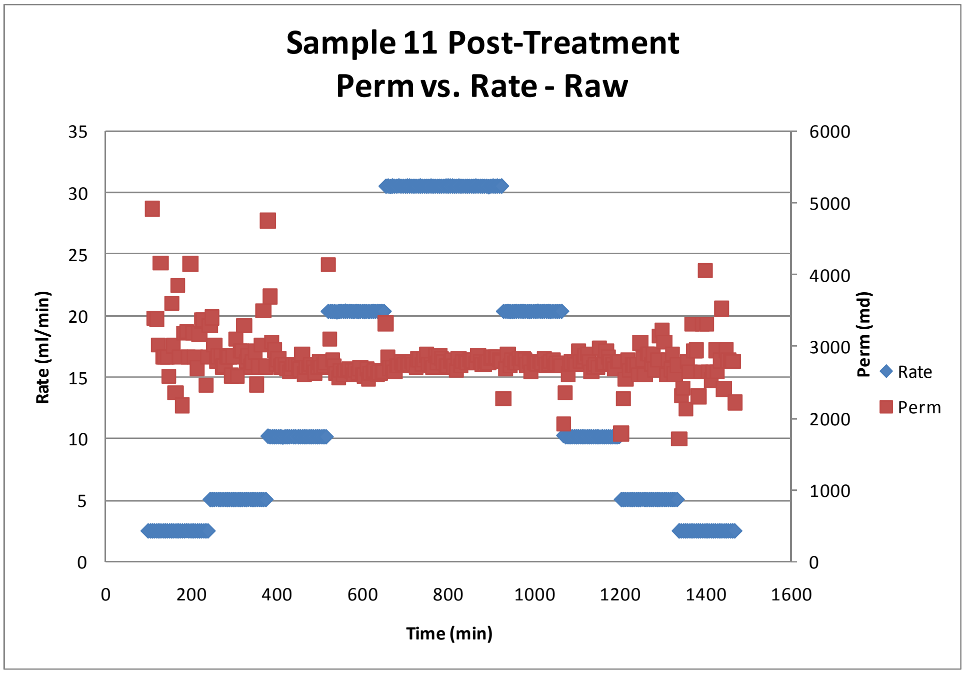


Fig. 55–Coreflood Sample 11 Post-Treatment Permeability Plot – Raw Data.

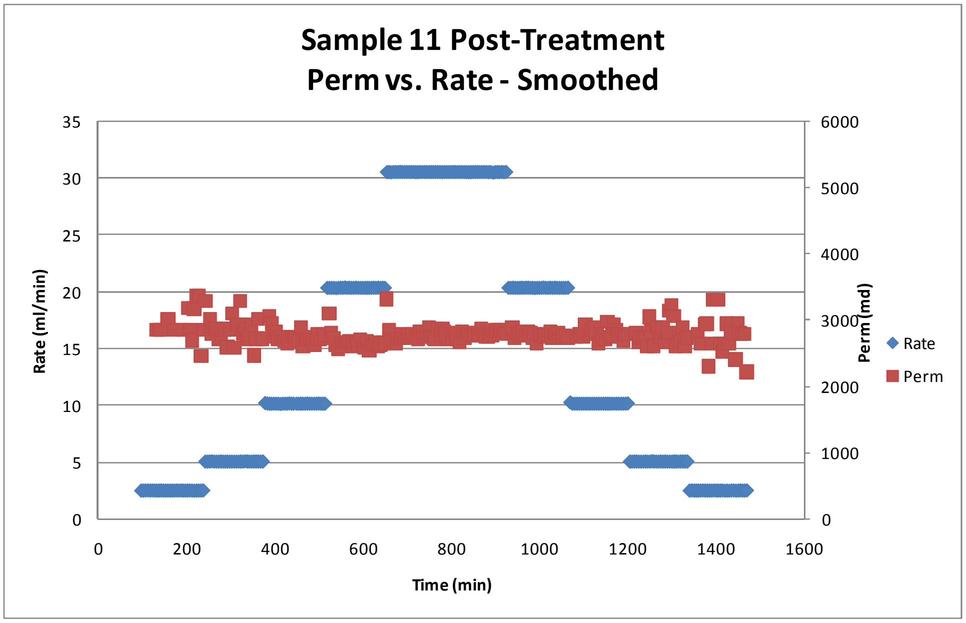


Fig. 56–Coreflood Sample 11 Post-Treatment Permeability Plot – Smoothed Data.

IV. SUMMARY AND CONCLUSIONS

A total of 250 syringe tests and 11 tests in the coreflood apparatus were completed on the epoxy system for the purpose of my research. The following three sections will summarize these results, make conclusions from these results, and mention areas of investigation necessary for future work.

4.1 Summary

The epoxy system went through numerous ‘bench-top’ trials through which the optimal curing agent, solvent type and concentration, preflush fluid formulations, catalyst amounts, and other additives and their concentrations were determined. Based upon these 250 syringe tests, the optimal system is given in the Table 49 below.

Table 49. Syringe Tests Optimal Fluid Formulations.

Preflush System	Epoxy System	Overflush
Base Brine PA2.1	Epoxy System B Amine Curing Agent Methyl Carbitol Solvent PA1.2	Base Brine

While reaching the latter stages of finding the optimal formulation for System B in the syringe tests, the testing of the fluid in the HP/HT cell was started. The addition of PA2 to the preflush gave the necessary strength to the sample after seeing subpar UCS values in Samples 1 through 4. Based upon the test results and SEM photos, the system appears to have a strong interaction with overflush fluid as the overflush volume directly influences the retained permeability and UCS values. The results of each of the tests in the HP/HT cell for the system are given in Table 50.

Table 50. UCS and Permeability Results.

Sample	UCS (psi)	Retained Perm. (%)
1	112	66.3%
2	331	52.5%
3	753	25.0%
4	977	0.3%
5	2401	47.2%
6	1444	26.0%
7	1313	55.9%
8	1684	59.4%
9	1484	50.8%
10	1089	35.8%
11	2122	59.5%

*See each section for specific test conditions.

The purpose of the 14 rounds of syringe tests was explained previously, and was given in Table 1. Table 51 states the purpose of each round, its outcome, and the conclusion based upon the results are given for each round.

Table 51. Compiled Syringe Test Results.

Syringe Test Round #	Fluid System(s) Tested	Purpose	Conclusions
1	A	Test system's performance at multiple overflush volumes, multiple temps, and multiple times.	Good results for a few 48 hr samples, inconsistent results for 24 hr samples. Possible catalyst removal during flush.
2	A	Test 3 new catalysts to try to get a more rapid cure (24 hr), and not affected by brine overflush.	Still issues with 24 hr cure of system. Switch to a new curing agent with a more rapid cure profile.
3	B	Test the new system with several overflush volumes, multiple temps, and multiple times.	Nothing substantial at 24 hr, but new system appears promising. Change base solvent to optimize the system further.
4	B, C	Test 2 Hydrophobic & 2 Hydrophilic solvents to find system with best results.	Hydrophilic system gave most promising results. Optimize the catalyst further.
5	B	Investigate high end of catalyst amounts to find upper limit.	Optimal value of the two found. Fingering observed in samples. For future samples add dye to diagnose fingering and inject at a more consistent rate.
6	B	Further investigate the low end of catalyst amounts to find the optimum. For fingering issues, change the overflush fluid to help.	Optimum catalyst amount was the same as the previous round. The change of overflush fluid did not improve the fingering as desired. Possible wettability issue, change the preflush fluid components as well.
7	B	Test the addition of an oil-wetting surfactant to the preflush and overflush fluids. Try another viscous overflush fluid.	Incomplete cure on most of the sample due to heating issues. SEM photos show emulsion with current system. Try more preflush and overflush fluids to still optimize the UCS values.
8	B	Test 6 different flush fluids, including different combinations of brines, oil-wetting surfactant, and chelating agent.	None of the flush fluid combinations showed improved UCS values over a brine preflush and overflush fluid. Keep brine as preflush and overflush fluid. Try new additive to help placement of the epoxy on the sand grains.
9	B, C	Test effect of new additive (Proprietary Additive (PA) – PA1) in a new hydrophobic and current best hydrophilic systems.	Current hydrophilic system's results improve, and still the best results. Further investigate a hydrophobic option b/c of emulsion of hydrophilic system with flush fluids.

Table 51 Continued.

Syringe Test Round #	Fluid System(s) Tested	Purpose	Conclusions
10	B, D	Further optimize the concentration of PA1 in the hydrophilic system, and in another new hydrophobic system.	Despite no emulsion, the hydrophilic system still gives the best UCS results. Try flush with a non-reactive preflush and overflush fluid.
11	B	Test influence of using mineral oil as preflush and overflush fluid.	Despite not reacting with the system, the system is greatly affected by increased overflush volumes, and UCS substantially lower than previous flush systems.
12	B	Investigate effects of 4 new preflush systems, and additives (PA3 & PA4) in the epoxy system.	PA2 in preflush shows great results, PA4 in the epoxy system shows improved results over current base system with brine flushes.
13	D	Test the effects of 4 new preflush systems, and additives (PA3 & PA4) in the previous best hydrophobic epoxy system.	Promising flow results, however UCS results were well below current optimums of hydrophilic system.
14	B	Optimize the concentration of PA2 in the preflush, with respect to PA1 and PA4 in the hydrophilic epoxy system.	Optimum fluid system obtained, and investigated further in the HP/HT cell.

4.2 Conclusions

Based upon the testing described to this point, the following conclusions have been made for the epoxy system. These conclusions are based upon the specific testing conditions applied to the fluid system. For any such fluid to be applied to an actual well in the field, I recommend that the tests be repeated under specific well conditions; preferably with an actual formation core and respective saturating fluids.

1) The hydrophilic system consistently showed better results than a hydrophobic system; in terms of both solvents and curing agents.
2) UCS results increase significantly with the addition of PA2 to the preflush.
3) Retained permeability isn't as high as desired, and is affected by the overflush volume. System still needs to be optimized with respect to this property.
4) The rate at which the system is injected appears to affect the results of the system.
5) System's results are affected when the overflush is pumped at a higher rate than that at which the preflush and epoxy system were injected.

4.3 Future Work

Based upon the results of the epoxy system to date, the following recommendations are made for areas of future investigation to further optimize the system.

1) The system’s performance at different permeability ranges, preferably with actual formation cores.
2) The effect of solvent concentration and injection rate on the results.
3) The system’s performance at a wider range of temperature applications.
4) Optimize the system’s catalyst level for the higher temperature applications with respect to working time.
5) The system’s performance when clays are introduced into the sand pack.
6) Run multiple tests at same conditions to determine system’s consistency.

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