PERFORMANCE OF FLUOROCHEMICALS ON WETTABILITY ALTERATION OF CARBONATE ROCKS TO ALLEVIATE CONDENSATE/WATER BANKING

J.Fahimpour, M.Jamiolahmady, Heriot-Watt University, R.Severac, DuPont de Nemours,
M.Sohrabi, Heriot-Watt University

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ABSTRACT

Accumulation of condensate and water around gas-condensate wells, which is attributed to strong liquid wetting characteristics of reservoir rocks, can significantly reduce the well productivity. Altering rock wettability around the wellbore to intermediate gas-wetting using appropriate chemical solutions has been proposed as a remedial work to alleviate the condensate/water blockages on a permanent basis.

There are a number of experimental studies reported on the impact of such chemical treatment processes for sandstone rocks. However, there are limited studies for carbonate rocks, which have different minerals on the rock surface. In this work, the performance of two anionic and nonionic fluorochemicals on positively charged carbonate substrates were investigated. The impact of chemical concentration, water and alcohol-based carrier solvents at low and high temperatures ranging from 65°C to 190°C and brine were investigated by measuring the pre- and post-treatment contact angles. The performance of chemicals were then evaluated further by conducting a series of spontaneous imbibition and unsteady-state displacement tests on treated carbonate core samples at 130°C by chemical solutions with optimized concentration. In displacement tests, impact of the chemical delivery method, rate and volume of injected chemical and chemical concentration were also studied.

The results of contact angle measurements highlighted the relative effectiveness of both anionic and nonionic fluorinated chemicals in altering the substrates wettability when delivered by a water-based solvent. A precipitated layer of anionic chemical was observed on the rock surface when the treatment temperature increased to 160°C and above. Using various alcohol-based solvents reduced the performance of both chemicals to some extent. Unlike the nonionic chemical, the anionic surfactant became unstable in solutions contaminated with brine.

The optimized method of continuous injection of anionic chemical solution through the core resulted in a significant increase in oil mobility by a factor of 1.7. The results of conducted spontaneous imbibition tests were also in close agreement with those of the unsteady state displacement experiments. The nonionic chemical, on the other hand, was

ineffective to alter the rock wettability as some level of plugging was observed at the core inlet face.

These findings highlight the importance of various parameters affecting the wettability alteration process and the special care, which should be taken in design and implementation of such costly operations for field applications.

INTRODUCTION

Well productivity of gas-condensate reservoirs can significantly decline when the well bottom-hole pressure falls below the dew-point pressure and a condensate buildup region is formed around the wellbore. The presence of water in addition to condensate can also adversely reduce the gas mobility around the wellbore. These condensate/water blockage issues are effectively related to the strongly liquid-wetting characteristic of the reservoir rock. Several field studies have verified the reduction of well productivity by more than 50% due to the liquid blockage of the near wellbore region. [1, 2].

For few decades, there have been a number of practical techniques proposed to prevent, delay or remove the condensate accumulation around the wellbore. Dry-gas recycling [3], hydraulic fracturing [4], drilling of horizontal and deviated wells [5, 6] and solvent injection [7] are a number of these methods. However, many of these techniques have only temporary success, due to reformation of condensate phase after the treatment, and some other are not economical, e.g. gas recycling.

Over the last decade, wettability alteration (chemical treatment) of the reservoir rock near the wellbore region, by using repellent oil and water surfactants aiming at making the rock surface intermediate gas-wet, has been proposed as a more permanent and cost-effective treatment compared to other conventional techniques [8]. Accordingly, a large number of experimental studies have been dedicated to the chemical treatment of sandstone rocks [9, 10], while there are only a few cases corresponding to carbonate rocks [11, 12].

In this paper, we have investigated the performance of two types of fluorosurfactants, with different ionic functionalities, on the limestone carbonate substrates and addressed the important factors controlling the wettability alteration process on these rock types.

EXPERIMENTAL PROCEDURE

Contact Angle Measurements

The carbonate substrates of Texas Cream limestone rock (TC), with a strong liquid-wet surface ($\theta_{gas/liquid} = 0$), were used in these studies to investigate the effect of several parameters including chemical concentration, solvent composition, temperature and brine on the wettability alteration process. The post-treatment liquid/gas contact angles on the treated rock surface were measured. These tests were conducted by aging a small substrate of the rock in about 30 cc chemical solution, pressurized at 400 psi in a piston cell at the desired temperature for 18 hrs. Samples were then dried inside an oven for 2 hrs. A drop shape analysis apparatus was used to measure the contact angle of the sessile drop of the liquid, i.e. deionized water (DIW) and decane (representing oil), deposited on the rock surface at atmospheric conditions. It should be noted that all chemical concentrations mentioned in this paper are based on the active ingredient (solid percent) of the chemical agent inside the solution.

Brine Compatibility Tests

Compatibility of the wettability modifier agents with the resident fluids around the wellbore, especially brine, at reservoir conditions is one of the crucial factors influencing the effective adsorption of chemical molecules on the rock minerals. This was initially examined through a series of visual observations on the phase behavior of the chemical solutions after mixing with brines with different salinities (e.g. 1% and 10% $NaCl/CaCl_2$, with a weight ratio of 1:1) at room temperature. The impact of the brine on the wettability alteration process was subsequently investigated further by performing a number of contact angle measurements on the rocks treated at 130°C using chemical solutions contaminated with brine.

Spontaneous Imbibition Tests

The optimized chemical solution, obtained through the contact angle measurements, was selected to treat a number of carbonate cores to be used in spontaneous imbibition tests. The tests were conducted by hanging the dry core plug vertically at its top end from a support connecting to a digital balance and immersing the other end by 3 cm inside the liquid phase. The increase in the weight of the rock versus time owing to the liquid imbibed into the porous medium provided the spontaneous imbibition curve. The comparison between the curves before and after treatment determined the effectiveness of the chemicals on altering the rock wettability.

Unsteady-state Displacement Tests

The ultimate effect of a wettability modifier, i.e. the improvement of the liquid mobility, was evaluated by comparing the pressure differences, between the inlet and outlet of the core, before and after treatment during the unsteady-state displacement of the resident gas by the injected liquid phase. Such tests also reveal any undesired damage of the rock permeability due to excessive chemical particles adsorbed on the rock surface.

To simplify the experimental procedure, non-equilibrated liquids, e.g. C_{10} with N_2, were injected at a constant flow rate of 20 or 100 cc/hr into the core 100% saturated with N_2 at room temperature and atmospheric pressure. However, in some cases, equilibrated C_{10}/N_2 at 1000 psi was used to confirm the improvement of liquid mobility as a result of wettability alteration at more realistic conditions.

RESULTS AND DISCUSSION

After careful investigations of various groups of fluorinated chemicals, sourced from DuPont de Nemours, on carbonate substrates, two fluorosurfactants, C (nonionic) and Z (anionic), were selected for further studies. The main criteria in selection of an appropriate wettability modifier are to: i) make the rock surface intermediate gas-wet, ii) be stable and effective at elevated temperatures, iii) be compatible with brine, iv) have the minimal adverse effect corresponding to chemical solution back-flow on the post-treatment production period, for instance by using an alcohol-based carrier fluid and v) minimize the rock permeability damage due to possible excessive deposition of chemicals on the rock surface. The results of the experiments conducted with these two chemical agents will be discussed in the following sections:

Performance of Nonionic Chemical

Chemical C is a water-soluble, environmentally friendly fluorosurfactant. It has mainly non-ionic functionality with a slightly anionic structure as a part of its formulation, whereby the non-ionic functionality would provide a good stability in brine and the anionic head, on the other hand, can provide a good interaction with positively charged carbonate minerals.

Chemical Concentration vs. Temperature

Contact angle measurements were conducted at various chemical concentrations at two low and high temperature limits. Figures 1 and 2 show the relevant contact angles measured on the treated substrates at 65 and 130°C, respectively. In all these treatments the chemical agent was diluted in DIW, as its basic aqueous solvent. It should be noted that the treatments were unpromising when the chemical was delivered in alcohol or combined water/alcohol solvents. It is noted from Figure 1 that at 65°C, the treatment provides acceptable $\theta_{air/water}$ of 140° at concentrations from 0.05 to 0.5 wt%. However, at higher concentrations of 1wt% the chemical becomes ineffective to repel the water phase. On the other hand, the chemical is ineffective to repel the oil phase at its low concentration range (0.05 and 0.1 wt%), but at higher concentrations (0.5 and 1 wt%) results in promising $\theta_{air/decane}$ of 100°. Almost the same trends are observed at elevated temperature of 130°C as shown in Figure 2. That is, the chemical loses its water repellency characteristic at concentrations above 0.1 wt%. However, it shows acceptable oil repellency at all five levels of concentrations. The $\theta_{air/decane}$ results suggest that as the chemical concentration, or the number of chemical molecules, inside the solution increases, the level and strength of the bond between the chemical and mineral surface increases. This is in favour of improving the chemical's oil repellency. However, it should be noted that possible formation of bilayers, at higher concentrations on the rock surface, can deteriorate the performance of the chemical to repel the water phase. Furthermore increasing the temperature can accelerate and boost the reaction between chemical molecules and the rock surface and therefore cause the formation of bilayers to happen at lower concentrations.

Effect of Brine on Nonionic Chemical Performance

Chemical C because of its nonionic functionality showed an acceptable level of stability in low and high-salinity brines over a wide range of its concentrations. To examine the impact of brine on wettability alteration process, two treatment tests with 0.2 wt% of chemical C diluted in DIW/brine (1:1) were conducted at 65 and 130°C. High-salinity brine (10 wt%) was used in these tests. Post-treatment contact angle measurements revealed that the chemical preserves its effectiveness with respect to the oil phase in the presence of brine and approximately $\theta_{air/decane}$ of 100° was obtained at both temperatures. On the other hand, an unpromising wettability alteration level was observed with respect to the water phase, e.g. $\theta_{air/water}$=0°. Comparing these with the results of tests performed by free-brine solutions at 65°C (Figure 1), in which $\theta_{air/water}$ of 140° was attained at the 0.05 to 0.5 wt% chemical concentration range, reveals that contaminating the chemical solution with brine during the treatment process reduces the solubility of chemical molecules inside the solution and consequently increases their reaction rate with the charged surface of the rock and as mentioned previously, any excessive adsorbed layer of chemicals reduces the water repellency of the chemical agent.

Treatment of Carbonate Cores with Nonionic Chemical

Based on the contact angle measurements, 0.1 wt% surfactant C diluted in DIW was selected as the optimum treatment solution, providing satisfactory levels of oil and water repellency at 130°C. Accordingly, this treatment solution was used to treat TC1 carbonate core (Table1) by injecting 10 pore volumes (PV) of chemical solution at a flow rate of 20 cc/hr into the core at 130°C. The post-treatment pressure drops during the unsteady-state displacement tests were to some extent above those of the pre-treatment case. This revealed that the rock permeability was damaged due to the adsorption of chemical particles during the treatment process. Careful examination of the rock surface showed that the damage has occurred only at the core inlet face as a precipitated layer of chemicals was observed at the inlet face. After trimming about 3 mm of the core inlet face, the pre- and post-treatment trends became similar. In order to investigate the impact of injection rate and contact time between chemical and rock surface, in another similar test on TC2 carbonate core, the injection rate was increased by a factor of 10 to 200 cc/hr. The same plugging issue at the TC1 core inlet face was also observed (Figure 3). These results increased the possibility of existence of large chemical micelles or aggregates (the collection of surfactant molecules) dispersed inside the solution that can be potentially filtered at the core inlet face. This was investigated by measuring the particle size of chemical C in DIW using a light scattering technique. Table 2 tabulates these measurements. It is noted that the average size of aggregates for 0.1 wt% of chemical C in DIW is 243 nm. Based on mercury porosimetry and centrifuge Pc measurements, it was noted that about 15-30% of total pore volume in Texas Cream rock consists of pores with a radius less than 250 nm. Comparing the pore size distribution of Texas cream rock and particle size distribution of chemical C can simply explain the plugging issue observed at the core inlet face during the injection of chemical solution.

Performance of Anionic Chemical

Chemical Z is an anionic phosphate fluorosurfactant. It is primarily a water-based chemical but can also be used in water/alcohol mixtures.

Solvent Composition

Contact angle measurements, like those mentioned for chemical C, were performed using various concentrations of chemical Z, diluted in its basic solvent, i.e. DIW. The results suggested that 0.5 wt% of this fluorosurfactant is able to provide an acceptable level of wettability alteration at 130°C, i.e. $\theta_{air/decane}$=115° and $\theta_{air/water}$=139° were obtained. Because alcohol-based solvents are more favourable carrier fluids than water-based ones, their potential in delivering this anionic chemical was also investigated. Figure 4 shows a number of these tests conducted using DIW/IPA or DIW/Ethanol solvents. Overall, the anionic chemical showed poor performance with respect to the oil phase when delivered by a water/alcohol mixture, whilst its good water-repellency characteristic was preserved in many cases similar to that of water-based solvent. As a matter of fact, alcohols hypothetically tend to decrease the CMC (critical micelle concentration) of the surfactants, which in turn could increase the solubility of the surfactant molecules inside the solution. This can reduce the interaction between the chemical molecules and the charged minerals and subsequently, causes lesser extent of liquid repellency. The alcohol type and its proportion inside the solution affects the extent of this behaviour. Following these results we decided to concentrate our studies on water-based solutions as alcohol-based solvents showed poor performance especially to repel the oil phase, which is the main concern in gas-condensate reservoirs, i.e. condensate banking.

Treatments at Elevated Temperatures

As the stability and effectiveness of any wettability modifier at realistic reservoir conditions is the most crucial part of a chemical treatment process, two treatments, using 0.5 wt% chemical Z + DIW, at elevated temperatures of 160 and 190°C, were conducted. Promising air/water and air/decane contact angles like those obtained at 130°C were obtained. However, as Figure 5 depicts, an undesirable layer of chemical was precipitated on the rock surface at both cases. It is likely that increasing the temperature has increased the reactions between the anionic head group of the chemical and the positively charged surface of the carbonate mineral.

Effect of Brine on Anionic Chemical Performance

The chemical's compatibility with brine was examined using two different types of salts, i.e. NaCl and $CaCl_2$, at two limits of low (1%) and high (10%) salinities. Accordingly, 1 wt% chemical Z diluted in DIW was contaminated with brine with a weight ratio of (1:1) at room temperature. It is noted that at this mixing ratio, the final chemical concentration in solution becomes 0.5 wt%. Figure 6 depicts the phase behaviour of the chemical agent inside the solution for different cases. It was evident that the anionic chemical is only stable in 1% NaCl brine as a transparent solution was obtained after adding brine into the chemical solution. However, increasing the NaCl concentration to 10% caused the instability and coagulation of chemical particles inside the solution. $CaCl_2$ brine showed more adverse effect as the chemical severely coagulated even at low concentration of 1 wt%. These observations underline the importance of the type of salt components and moreover their relevant concentrations in the reservoir brine composition on the stability and therefore performance of such fluorinated chemicals.

In order to evaluate the impact of brine on performance of this anionic chemical further, the carbonate substrate was treated with the only stable solution discussed above, i.e. 0.5 wt% chemical Z+DIW/1% NaCl (1:1), at 130°C. Acceptable contact angles of $\theta_{air/decane}$=87° and $\theta_{air/water}$=134° were obtained, showing the preserved level of chemical effectiveness after contamination with brine.

Treatment of Carbonate Cores with Anionic Chemical

The optimum treatment solution of 0.5 wt% chemical Z + DIW was used to treat TC3 carbonate core at 130°C. Here to examine the effect of chemical delivery method and volume of injected chemical on treatment performance, two different methods were investigated: 1- the evacuated core was saturated with 1 PV chemical solution and then aged in such conditions for 18 hrs at 130°C, 2- approximately 10 PVs of chemical solution were injected through the core with a flow rate of 20 cc/hr at 130°C and then was aged for extra 18 hrs with the solution. The remaining chemicals in both cases were displaced by N_2 at the end of treatment and the core was dried for subsequent imbibition and displacement tests. It should be noted that TC3 was initially treated by the first method and following an unsuccessful treatment, as will be discussed later, retreated with the second procedure.

Figure 7 and Figure 8 show the spontaneous imbibition and unsteady state displacement tests, respectively, carried out on TC3 before and after each treatment. The water imbibition tests (in Figure 7) demonstrate that the rock wettability has been significantly altered with respect to the water phase following the first treatment procedure (method 1). However, the decane imbibition curves, before and after first treatment, show minimal difference following the same trends. This implies that method 1 has delivered insufficient chemicals to the rock surface to render it effectively oil repellent. The trends of the pressure drops during the displacement tests by decane (Figure 8) are also consistent with this observation. On the other hand, exposing the rock surface to considerably greater amount of chemical molecules, i.e. method 2, shows that a promising level of oil repellency has occurred inside the core. Both imbibition and displacement tests after the second treatment, strongly confirm this fact (Figures 7 and 8).

To confirm our findings and evaluate the maximum performance of this anionic chemical on improving the oil mobility, the second method of treatment, explained above, was repeated on another carbonate core (TC4). In this test equilibrated C_{10}/N_2 at 1000 psi was used to conduct the unsteady-state displacement tests to eliminate the solubility between non-equilibrated C_{10} and N_2 used in the previous displacement tests and make the calculation of oil mobility more accurate. Figure 9 shows the results of pre- and post-treatment displacement tests at such conditions. After conducting material balance on the volume of the injected and recovered decane phase and applying Darcy's law using the stabilized pressure differences along the core, an average decane saturation of 0.79 at the stabilized conditions was obtained. The decane relative permeabilities before and after treatment were 0.16 and 0.27, respectively. This verifies the improvement of oil kr almost by a factor of 1.7 as a result of an effective wettability alteration using this anionic fluorosurfactant. Absolute permeability measurements also demonstrated no sign of permeability damage after the treatment, confirming the capability of this chemical to be used as a wettability modifier.

CONCLUSIONS

In this study the performance of two nonionic and anionic fluorosurfactants on the wettability alteration of a limestone carbonate rock was investigated. The impact of the chemical concentration, carrier solvent, temperature, brine and delivery method on the treatment process were investigated. The main conclusions drawn from this study are:

- Nonionic chemical delivered an acceptable level of oil and water repellency ($\theta_{air/decane}$=100° and $\theta_{air/water}$=140°) when diluted in deionized water as a solvent, at both low (65°C) and high (130°C) temperatures. It was demonstrated that at 65°C this fluorosurfactant is more effective to repel the water phase at lower concentrations (0.05-0.5 wt%), while at higher concentrations, e.g. 1 wt%, when its water repellency becomes poorer, as a results of formations of bilayers, its oil repellency improves. The same trend was observed at high temperature as well.
- Nonionic chemical showed a good compatibility with low and high salinity brines with an acceptable preserved level of oil repellency after contamination with brine. However, as brine reduced the solubility of chemical in the solution and consequently increased its reaction rate by the rock, the chemical water repellency deteriorated due to existence of bilayers.
- The existence of large chemical aggregates in the nonionic chemical solution caused the plugging of the core inlet face and therefore an ineffective treatment was obtained.
- Anionic chemical demonstrated effective oil and water repellency ($\theta_{air/decane}$=115° and $\theta_{air/water}$=139°) when 0.5 wt% chemical delivered by deionized water at 130°C. However, this chemical was not effective in alcohol-based solvents. Elevated temperatures, e.g. 160 and 190°C, caused deposition of a layer of chemical, due to excessive adsorption, on the rock surface.
- The type and concentration of the salts (NaCl and $CaCl_2$) present in brine are the key factors affecting the brine stability of chemical. $CaCl_2$ showed more adverse effect on the chemical stability compared to NaCl.
- A promising improvement in oil mobility by a factor of about 1.7 was obtained after injecting 10 PVs of anionic chemical solution into the carbonate core. This shows that the anionic chemical tested could effectively alter the carbonate core wettability without damaging the rock permeability. However, the volume of the chemical delivered into the core played a crucial role in obtaining the desired wettability level.

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Table 1: Rock properties of carbonate cores (1″ diameter) used for chemical treatments.

Core Type	Length (cm)	K (md)	Ø (%)
TC1	9.2	9.8	29.5
TC2	8.8	10.0	29.5
TC3	9.0	10.8	29.0
TC4	8.8	9.7	24.8

Table 2: Particle size and distribution, measured right after the preparation of the solution, for different concentrations of chemical C in DIW, conducted at DuPont laboratory.

Concentration (Active ingredient) wt%	0.01		0.05		0.1		0.5	
Fluorosurfactant	Average Size (nm)	Distribution	Average Size (nm)	Distribution	Average Size (nm)	Distribution	Average Size (nm)	Distribution
Chemical C	158.2	0.227	256.2	0.024	243.0	0.097	246	0.045

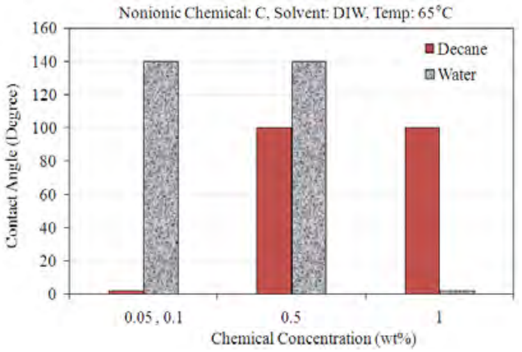

Figure 1: Effect of concentration of the nonionic chemical C on wettability alteration of carbonate rock at 65°C.

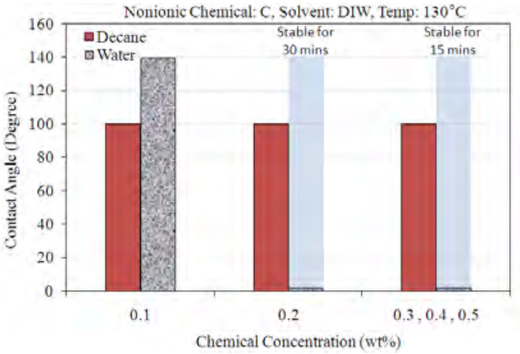

Figure 2: Effect of concentration of the nonionic chemical C on wettability alteration of carbonate rock at 130°C.

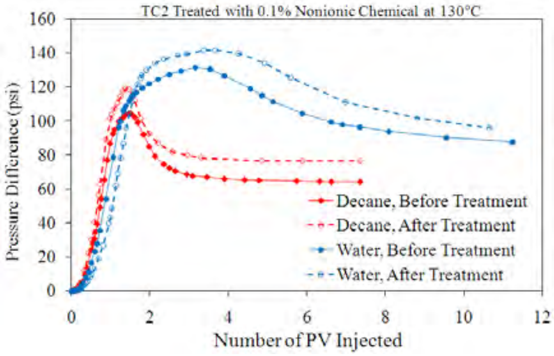

Figure 3: Pressure difference during unsteady-state injection of non-equilibrated C_{10}/N_2 with a flow rate of 100 cc/hr into the TC2 carbonate core (100% saturated with N_2, outlet pressure=14.7 psia) before and after treatment (by 0.1 wt% nonionic chemical+DIW at 130°C).

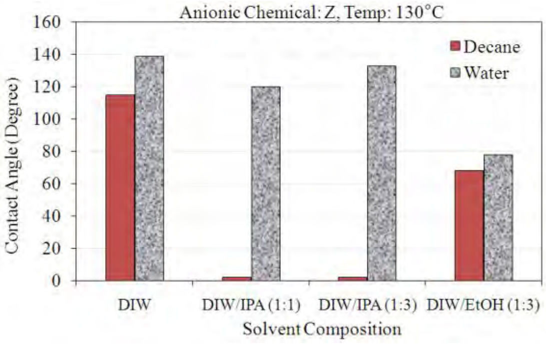

Figure 4: Effect of solvent composition of the anionic chemical Z on wettability alteration of carbonate rock at 130°C.

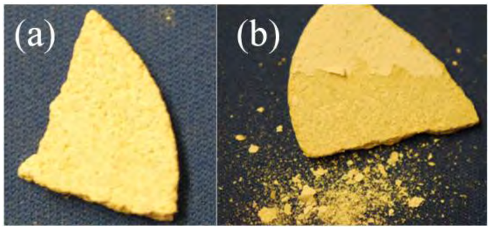

Figure 5: Carbonate substrates treated by 0.5%wt anionic chemical Z diluted in DIW at different temperatures of: a) 130°C and b) 160 and 190°C. A deposited layer of chemical is evident on the treated rocks at two higher temperatures (b).

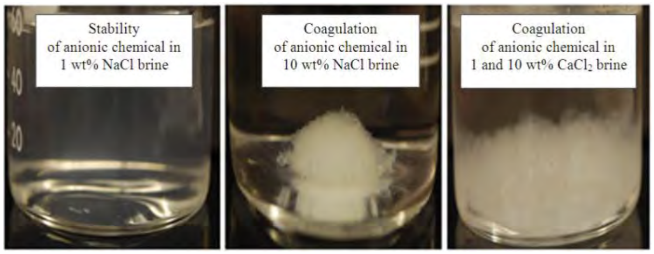

Figure 6: Compatibility tests of anionic chemical Z performed with two types of brine at different salinities.

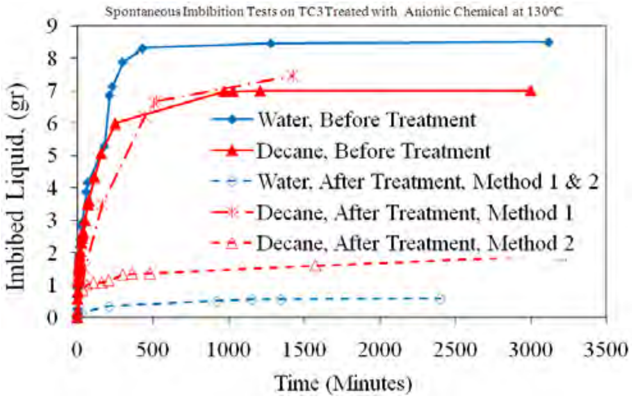

Figure 7: Spontaneous imbibition tests before and after treatment on the TC3 carbonate core treated by 0.5 wt% anionic chemical + DIW at 130°C using two treatment methods: 1-saturating core by 1 PV chemical solution and 2- injecting 10 PVs chemical solution through the core.

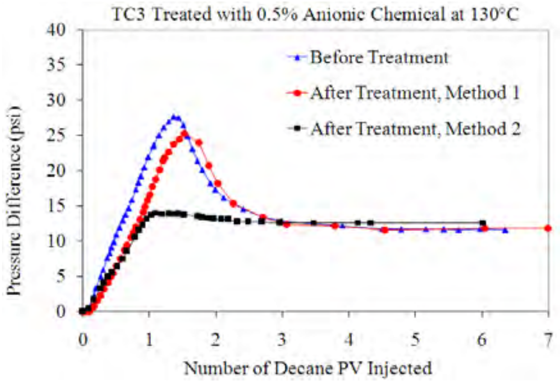

Figure 8: Pressure differences during unsteady-state injection of non-equilibrated C_{10} into the TC3 carbonate core (100% saturated core with N_2, outlet pressure=14.7 psi) before and after treatment by 0.5 wt% anionic chemical + DIW at 130°C using two treatment method: 1-saturating core by 1 PV chemical solution and 2- injecting 10 PVs chemical solution through

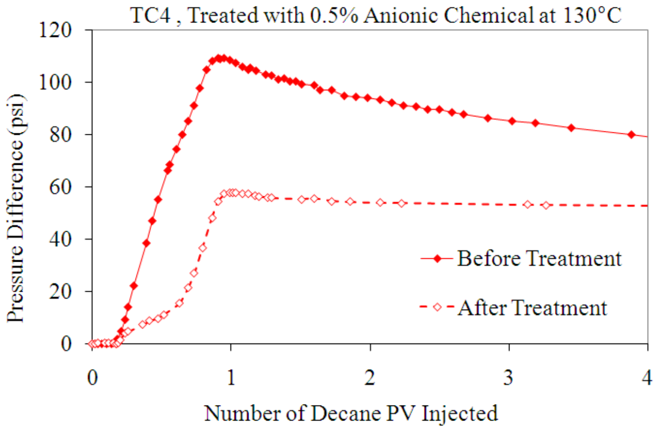

Figure 9: Pressure differences during unsteady-state injection of equilibrated C_{10} with a flow rate of 20 cc/hr into the TC4 core (100% saturated with N_2, outlet pressure=1000 psi) before and after treatment by 0.5 wt% anionic chemical + DIW at 130°C.