Impacts of EOR Chemicals on Calcium Carbonate Scale Formation and Inhibition

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ABSTRACT

A field trial has been planned for a chemically enhanced oil recovery (EOR) project in Saudi Arabia. The injection chemicals will be a combination of surfactants and polymers. There is a concern that the breakthrough of these chemicals in produced water could affect the scale formation process and so interfere with the scale treatment program. The objective of this study is to investigate the potential impacts of the injected EOR chemicals on calcium carbonate (CaCO₃) scale inhibitor performance.

Laboratory tests were carried out using both static bottle and dynamic tube blocking methods. CaCO₃ scale inhibition was studied in the absence and presence of the EOR chemicals at the estimated peak breakthrough concentrations of 200 mg/L polymers and 150 mg/L surfactants. Three different types of inhibitors were evaluated: tri-phosphonate, penta-phosphonate and polyacrylate. Inhibition (%) from the bottle test and the minimum effective dose (MED), based on the tube blocking method, were determined for each inhibitor at 160 °F. Scale precipitates from the bottle tests were also characterized for morphology and polymorphs using environmental scanning electron and X-ray diffraction (XRD) techniques.

Obtained results showed significant reductions in scale inhibitor performance caused by the EOR chemicals. In dynamic tube blocking tests, the MED values of the inhibitors were increased roughly 10 times with the EOR chemicals. The static bottle tests also showed considerable changes under the test conditions. For example, the inhibition (%) of the penta-phosphonate-based inhibitor at 20 mg/L was diminished from 100% to 20%, and the percentage of the tri-phosphonate inhibitor was decreased from 100% to below 50% and 70% at 10 mg/L and 20 mg/L, respectively. The polymeric inhibitor completely lost its inhibition efficiency over the tested concentration range of 2 mg/L to 20 mg/L. The impact of EOR chemicals were also demonstrated by the remarkable ranges of crystal morphologies, changing from simple aragonite columns to nanorod, distorted spheroid and a flower-like superstructure in the presence of EOR chemicals and inhibitors.

This article presents the first reported comprehensive study on the impacts of the EOR chemicals on CaCO₃ scale formation and prevention. Results suggest that the performance of scale inhibitors could be substantially affected by the EOR chemicals. Such potential impact should be determined and a new scale treatment program should be developed for the success of EOR projects.

INTRODUCTION

Calcium carbonate (CaCO₃) is the primary scale type occurring in the oil production systems in Saudi Arabia. Scale formation largely results from the loss of dissolved carbon dioxide (CO₂) gas from produced water that occurs with pressure drops, although temperature increase and turbulent flow are considered as the main causes in some systems, such as electric submersible pumps. Over the years, cost-effective mitigation programs have been developed and optimized⁴. The squeeze treatment was first implemented in the giant Ghawar field in late 1981 and is widely used nowadays⁵⁶. Long squeeze lives have been achieved in many cases⁷⁸. An encapsulated scale inhibitor has also been used for wells with low scaling tendencies and low reservoir pressures⁹. Scale inhibitors are applied by continuous injection at oil-water separation plants.

Laboratory studies and field observations have shown that the effectiveness of a scale inhibitor product is affected by many factors, such as water composition, system pressure, oil-water ratio, temperature, etc. Therefore, treatment programs have been adjusted to account for any changes in production conditions. With the upcoming field trial of a chemically enhanced oil recovery (EOR) technique, a detailed study has been conducted to understand the potential effects of EOR chemicals on production chemistry and to evaluate the effectiveness of the incumbent scale treatment programs at EOR production conditions. This article presents part of the study to illustrate the performance of scale inhibitors.

The field trial is planned to test the oil displacement ability of surfactant and polymer flooding in a small depleted area of a carbonate reservoir. The surfactant and polymer products were selected after extensive coreflooding evaluations. The mechanisms of these chemicals are well documented¹⁰⁻¹⁴. The addition of polymers is to increase the viscosity of the water phase, thereby reducing the permeability of water to the porous rock, which creates a more efficient and uniform front to displace oil from the reservoir. The addition of surfactants is to decrease
the oil-water interfacial tension, mobilize the remaining oil and improve the wettability of porous rocks, thereby allowing better displacement of the oil by injected water.

Much of the previous scale studies related to chemical EOR were focused on the pH effects in alkaline-surfactant-polymer (ASP) flooding. With the addition of sodium hydroxide or sodium carbonate, the pH of the injected aqueous chemical solution is significantly elevated. The scaling tendencies of pH sensitive compounds such as CaCO₃ and magnesium carbonate are thereby considerably exacerbated\(^{15-17}\). Water could even become supersaturated to magnesium hydroxide\(^{18, 19}\). Alkaline pH also leads to the dissolution of quartz and clay minerals in the reservoir, thereby increasing the dissolved silicate concentration in the produced water. Changes in water composition, pH or temperature could result in the formation of amorphous silica or magnesium/calcium silicate scale\(^{20-22}\). On scale control, Karazincir et al. (2011)\(^{15}\) examined the feasibility of using complexing agents and scale inhibitors to prevent carbonate scale. They found that a high concentration of complexing agent (11,000 ppm) was effective up to a pH value of 10.5 in waters with 450 ppm bicarbonate alkalinity and 1,000 ppm divalent cations. With the addition of 200 ppm to 500 ppm of phosphonate or polyvinyl sulfonate-based scale inhibitor, the required complexing agent concentration was reduced to 5,500 ppm. Lo et al. (2011)\(^{19}\) studied the scale control when ASP was injected into a reservoir containing low salinity/low divalent ion (Ca\(^{2+}\) < 100 mg/L) formation water. They found that a penta-phosphonate-based inhibitor could provide effective control under this particular field condition; however, when the previously injected seawater was also co-produced, scale control was only achieved at limited formation water-seawater mixing ratios\(^{23}\). Recently, Alwi et al. (2016)\(^{24}\) evaluated alternative inhibitor chemistries and examined the synergistic effects of different additives to improve CaCO₃ scale control. The new formulations showed a much improved performance in produced waters with high seawater content. It should be noted that the EOR surfactants or polymers were seldom considered in the previous studies due to the overwhelming effect of high pH levels.

Although the majority of the injected surfactants and polymers are consumed by reservoir rocks, they still can reach the production wells at concentrations much higher than the applied scale inhibitor dosage. Unfortunately, there is little knowledge on the effect of these chemicals on scale formation and prevention. In this article, some recent works are summarized to show their significant impacts on CaCO₃ inhibitor performance. In addition, changes in CaCO₃ morphology and mineralogy are also discussed.

**EXPERIMENTAL**

**Materials**

The test brine composition is listed in Table 1. Synthetic brines were prepared with analytical grade chemicals and double de-ionized (DI) water, and filtrated through the 0.45 μm membrane filters for all tests. The EOR chemicals were tested at the estimated peak breakthrough concentrations, i.e., 200 mg/L for polymers and 150 mg/L for surfactants. The three scale inhibitors evaluated were the most common products used in the company’s upstream operations. They are tri-phosphonate-based inhibitor K, penta-phosphonate-based product S and polyacrylate-based inhibitor T. All tests were conducted at 160 °F.

### Dynamic Tube Blocking Tests

In the dynamic tube blocking tests, blank runs — without the scale inhibitor and EOR chemicals — were performed first to determine the brine scaling time. Bicarbonate concentration would be adjusted if the scaling rate was too slow. Test duration for subsequent inhibitor runs was circa three times the blank scaling time. All inhibitors were initially tested at 15 mg/L, and then changed for the determination of the minimum effective dose (MED). The system was cleaned thoroughly with acetic acid and DI water after each run with the given inhibitor concentration. Table 2 summarizes the test conditions.

### Static Bottle Test Method

In the static bottle tests, sample preparations were carried out at room temperature (\(~72\ ^\circ\)F). The test bottle was first filled with 50 mL cation brine and then dosed with a pre-calculated amount of inhibitor. After adding 50 mL of anion brine, the test bottle was quickly capped, sealed and shaken to mix thor-
oughly. Before placing the bottle into a preheated oven at 160 °F, ~15 mL brine was withdrawn using a needle syringe for chemical analysis. After 24 hours, test bottles were visually examined for precipitates, and then ~15 mL solid-free brine was collected. The exact weights of brine samples were measured to the second decimal (0.01 g) before analysis.

Oil field brines often contain a much higher calcium concentration than bicarbonate alkalinity. The amount of CaCO₃ scale formed in such brines will be limited by the available bicarbonate ions. The percent change in Ca²⁺ concentration due to CaCO₃ precipitation would be much smaller than that of bicarbonate, according to the reaction stoichiometry: Ca²⁺ + 2HCO₃⁻ = CaCO₃ + H₂O + CO₂. For example, if 50% HCO₃⁻ ions were converted into CaCO₃ in the test brine, the decrease in Ca²⁺ concentration would be only 2.1%. Such a change is too small to be determined accurately. Quantitation of inhibitor performance based on Ca²⁺ concentrations, a method often used in the industry, could result in significant errors.

In this study, total alkalinity was used as the indicator for inhibitor performance. Total alkalinity is defined²⁵ as:

\[
[\text{Alk.}] = [\text{HCO}_3^-] + [\text{CO}_3^{2-}] + [\text{OH}^-] - [\text{H}^+] \quad (1)
\]

For each mole of CaCO₃ formed, one mole of Ca²⁺ and two equivalent moles of alkalinity are consumed:

\[
\Delta[\text{Alk.}] = 2\Delta[\text{Ca}^2⁺] \quad (2)
\]

The above relationship is based on charge balance, Fig. 1. [Alk.] is the difference between the base cations (Na⁺, K⁺, Ca²⁺ and Mg²⁺) and the conservative anions (Cl⁻, SO₄²⁻):

\[
\Sigma \text{Cations (in mole/L)} = 2[\text{Ca}^2⁺] + 2[\text{Mg}^2⁺] + [\text{H}^+] + [\text{Na}^+] + [\text{K}⁺] \quad (3)
\]

\[
\Sigma \text{Anions (in mole/L)} = [\text{HCO}_3^-] + 2[\text{CO}_3^{2-}] + [\text{OH}^-] + [\text{Cl}^-] + 2[\text{SO}_4^{2-}] + [\text{H}^+] + [\text{OH}^-] - [\text{H}^+] - [\text{OH}^-] \quad (4)
\]

The charge balance leads to:

\[
[\text{Alk.}] = 2[\text{Ca}^2⁺] + 2[\text{Mg}^2⁺] + [\text{Na}^+] + [\text{K}⁺] - [\text{Cl}^-] - 2[\text{SO}_4^{2-}] \quad (5)
\]

Total alkalinity can be easily determined by the titration method. In this study, it was measured by titrating the samples with a 0.020 N sulfuric acid standard solution to the end point of pH 4.5. The percent inhibition was then calculated using the following equation:

\[
\text{Inhibition (\%)} = \frac{[\text{Alk.}]_\text{sample} - [\text{Alk.}]_\text{blank}}{[\text{Alk.}]_\text{blank} - [\text{Alk.}]_\text{blank}} \times 100 \quad (6)
\]

where \([\text{Alk.}]_\text{sample}\) = total alkalinity in the treated sample at \(t = 24\) hours, \([\text{Alk.}]_\text{blank}\) = total alkalinity in the blank sample (no additives) at \(t = 24\) hours, and \([\text{Alk.}]_\text{blank}\) = total alkalinity in the blank sample at \(t = 0\).

### CaCO₃ Precipitate Characterizations

At the end of the bottle test, the brine solution was filtrated through a 0.45 mm filter. The retained solids were washed with DI water to remove residual dissolved salts and dried at 100 °F overnight for instrumental characterizations. The shape and surface morphology of precipitates were examined using an environmental scanning electron microscope (ESEM) operating at 15 kV in 0.15 torr water vapor pressure. Energy dispersive...
spectroscopy (EDS) analyses were performed for chemical compositions. Samples with similar precipitate shapes were combined to obtain an adequate quantity for phase identification by X-ray diffraction (XRD). The XRD patterns were collected on a Rikagu powder X-ray diffractometer with Cu-Kα radiation (40 kV, 40 mA). Using the search-match capabilities of XRD software JADE 9.1+, all diffraction peaks were identified. For samples with more than one mineral phase, the relative weight% of each phase was quantified using the Rietveld refinement method.

RESULTS AND DISCUSSION

Chemical analysis results showed that the additives at the tested concentrations had little effect on brine pH and alkalinity. Therefore, the changes in scale inhibitor performances reported in this section should be caused by the interference of the EOR chemicals with reaction kinetics, rather than alteration in the thermodynamic driving force.

Effects on CaCO₃ Deposition in Dynamic Tube Blocking Tests

In the dynamic tube blocking test, no DP changes were observed over 90 min with the original test brine in repeated runs. The HCO₃⁻ concentration was increased gradually to accelerate the scaling process. At 750 mg/L HCO₃⁻, the blank scaling time was shortened to ~40 min.

With the addition of EOR chemicals, the scaling time was extended to over 50 min, Fig. 2. This could be caused by either the postponed CaCO₃ precipitation or the changes in the precipitate’s shape and surface property, which delayed the CaCO₃ deposition or led to slow growth rates.

Figures 3 to 6 show the test results on the scale inhibitor performance. The test duration for each inhibitor concentration was fixed at 120 min. In the absence of EOR chemicals, the tri-phosphonate-based inhibitor K was effective at concentrations down to 2 mg/L, Fig. 3. The polycrylate-based inhibitor T was much less effective. At 15 mg/L, ∆P started to increase after 80 min and reached 0.2 psi at the end of the test run.

Table 3. MED values determined from dynamic tube blocking tests

<table>
<thead>
<tr>
<th>Scale Inhibitor</th>
<th>MED Values (mg/L)</th>
<th>Without EOR Chemicals</th>
<th>With EOR Chemicals</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inhibitor K (Tri-phosphonate)</td>
<td>≤ 2 mg/L</td>
<td>15 to 20 mg/L</td>
<td></td>
</tr>
<tr>
<td>Inhibitor S (Penta-phosphonate)</td>
<td>2 to 5 mg/L</td>
<td>40 to 50 mg/L</td>
<td></td>
</tr>
<tr>
<td>Inhibitor T (Polycrylate)</td>
<td>10 to 15 mg/L</td>
<td>&gt; 100 mg/L</td>
<td></td>
</tr>
</tbody>
</table>

Fig. 8. Summary of static bottle test results based on alkalinity measurements.
10 mg/L, it failed after 104 min. For tests with penta-phosphonate-based inhibitor S, no ∆P increases were observed at 15 mg/L, 10 mg/L and 5 mg/L. At 2 mg/L, ∆P started to increase after 50 min and exceeded the failure criteria of 1 psi after 100 min. The MED values were ≤ 2 mg/L for inhibitor K, 2 mg/L to 5 mg/L for inhibitor S and 10 mg/L to 15 mg/L for inhibitor T.

Scale inhibitors became much less effective in the presence of EOR chemicals. Inhibitor K failed at 15 mg/L after 90 min and passed at 20 mg/L and 30 mg/L. There was no deposition in the scaling coil at 30 mg/L, as indicated by the ∆P profile, but a significant increase of ∆P was recorded at 20 mg/L, Fig. 4. Similarly, the MED value for inhibitor S was increased to 50 mg/L, Fig. 5. The increases in ∆P exceeded 1 psi after 84 min at 15 mg/L, 90 min at 30 mg/L, and 107 min at 40 mg/L. At 50 mg/L, ∆P was 0.2 psi after 120 min. Inhibitor T failed at all tested concentrations up to 100 mg/L, Fig. 6, although the scaling times were gradually increased with inhibitor concentration, Fig. 7.

Table 3 summarizes the MED values determined from the dynamic tube blocking tests. It can be seen that for all three inhibitors tested, their scale inhibition efficacies were decreased roughly 10 times by the EOR chemicals under these test conditions.

**Effects on CaCO₃ Inhibition in Static Bottle Tests**

Static bottle test results also showed the adverse effects of the EOR chemicals on scale inhibitor performances. The calculated inhibition percentage results based on alkalinity measurements are shown in Fig. 8, which were in good agreement with the visual observations.

For inhibitors K and S, precipitates were only observed in the 2 mg/L samples when no EOR chemicals were added; with the presence of the EOR chemicals, precipitates were found in all samples. With inhibitor K, the attained inhibition percentage was > 80% at 2 mg/L and 100% at 10 mg/L and 20 mg/L without the EOR chemicals, but it was reduced to < 10% at 2 mg/L, < 20% at 5 mg/L, < 50% at 10 mg/L and < 70% at 20 mg/L with the EOR chemicals added.

Inhibitors S and T had even larger declines in the inhibition percentage. The effectiveness of inhibitor T was entirely erased by the EOR chemicals. Actually, there was more CaCO₃ formed at low inhibitor concentrations — 2 mg/L to 10
mg/L — than in the blank samples. The inhibition percentage of inhibitor S became negligible at 2 mg/L and 5 mg/L, compared to ~50% at 2 mg/L and > 95% at 5 mg/L without EOR chemicals; at 10 mg/L and 20 mg/L inhibitor S concentrations, inhibition percentage was decreased, respectively, from 98% to ~10% and from 100% to ~20%. For all three inhibitors tested, they were more effective to CaCO₃ inhibition at 2 mg/L in solutions without the EOR chemicals than at 20 mg/L with the EOR chemicals.

**Effects on CaCO₃ Morphology and Polymorph**

The impacts of the EOR chemicals on the CaCO₃ formation and inhibition were also reflected in the ESEM and XRD analyses. Precipitates recovered from the samples free of the EOR chemicals were in the form of hexagonal columns, Fig. 9a, the typical aragonite CaCO₃ formed at elevated temperatures. Low concentrations of scale inhibitors showed no strong influence on the precipitate shape, Fig. 9b. The EOR chemicals, on the other hand, resulted in a large variety of unique morphologies and also the formation of calcite and vaterite crystals. With the EOR chemicals alone, precipitates were primarily in the forms of short cylinders and elongated rhombohedrons, Fig. 10a. Some short cylinder crystals had ridged sides and rough ends, Fig. 10b. XRD analyses showed that the CaCO₃ formed was about two-thirds aragonites and one-third calcites, Fig. 10c.

New shapes of CaCO₃ precipitates emerged when scale inhibitors were also present. With polyacrylate inhibitor T, elongated rhombohedrons were gradually replaced by nanorod-shaped crystallites, Fig. 11a. At 20 mg/L of inhibitor S, > 90% CaCO₃ crystals were in the form of disjunct nanorods, Fig. 11b. All precipitates had aragonite crystal structures, Fig. 11c.

The combination of the EOR chemicals with inhibitors K or S led to the formation of disk-shaped crystals, Fig. 12a. Trace amounts of Mg, P, S and Cl elements were detected by EDS in this type of crystal, Fig. 12b. Meanwhile, the short columns formed in the inhibitor-free solution, as previously seen in Fig. 9a, were developed into compact arrays of needle crystals with similar lengths at inhibitor concentrations of 2 mg/L and 5 mg/L. As inhibitor concentrations were increased to 10 mg/L and 20 mg/L, the disks were evolved to distorted spheroids, and the compact needles were changed into loosely packed needles, Fig. 12c.

At 20 mg/L concentrations of inhibitor K, a flower-shaped superstructure was also developed, Fig. 12d. All three CaCO₃ polymorphs — aragonite, calcite and vaterite — were identified by XRD analysis in these samples, Fig. 12e, suggesting that the EOR chemicals promoted the formation of calcite and vaterite.

**CONCLUSIONS**

A comprehensive study was performed to investigate the effects of EOR polymers and surfactants on CaCO₃ scale formation and scale inhibitor performance. The data presented in this work shows that the effectiveness of common scale inhibitor chemistries, such as tri-phosphonate, penta-phosphonate and polyacrylate, can be significantly deteriorated by the EOR chemicals. Under the specific test conditions in this study, their efficacies were reduced on the order of 10 times. This could be caused by the decrease in scale inhibitor interaction with the growing CaCO₃ crystals. It is possible that the EOR chemicals, in particular surfactants, adsorb on the CaCO₃ surface and then prevent scale inhibitor molecules from reaching the active growth sites. The EOR chemicals may also combine with the scale inhibitor to form complexes, which are less effective for scale inhibition. The strong interactions of the EOR chemicals with CaCO₃ scale and the scale inhibitors are also evident in the significant changes in CaCO₃ morphology and polymorphs.

Results presented in this article suggest that the selected EOR surfactant and polymer combination could negatively affect the
scale inhibitor performance. To safeguard the production system with adequate scale protection, the potential interference of the EOR chemicals on incumbent scale treatment programs should be evaluated, and a new inhibitor treatment program should be proactively developed.

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REFERENCES


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He studied the seismic facies, sedimentary facies and sequence stratigraphy of Jurassic formations in the Turpan-Hami basin of China. Shouwen used piezoelectric transducers to measure the sonic velocity of various dolomites from the Madison formation of Wyoming and Montana, and he developed an empirical formula to predict the sonic velocity of dolomite according to thin section description. He also developed new XRD methods in-house for quantitative mineral analysis of sandstone and successfully solved the problem caused by the Rietveld method limitation. Shouwen’s specialties include sequence stratigraphy, clastics diagenesis, clay mineralogy and formation damage assessment, thin section description, XRF elemental analysis, XRD phase identification and quantification, crystallite size determination, texture and residual stress analyses.

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