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Article

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Evaluation of the Impact of Ferrous Species on the Performance of a Poly(carboxylic acid)-Based Scale Inhibitor

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This work aims to evaluate, from a static and dynamic perspective, the performance of a polycarboxylic acid-based scale inhibitor in the presence of iron ions (Fe^{III}). The static (jar test) and dynamic (tube blocking test) tests were performed according to NACE TM0197-2010 and NACE TM31105-2005 standards, respectively. The lowest inhibition concentration (LIC) was determined under flow conditions of oil wells. In addition, the influence of the concentration of Fe^{III} ions on the precipitation process was also evaluated. The scale deposits were analyzed by X-ray diffraction (XRD), infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). The results demonstrated that the scale inhibitor is chemically compatible and efficient with the selected brines, possessing a LIC of 30 mg L^{-1} in the absence of Fe^{III} ions. In the presence of Fe^{III} ions, the inhibitor proved to be inefficient and incompatible, and it was not possible to determine the LIC. The combined XRD, FTIR, and SEM analyses allowed us to identify the inhibitor's mechanism of action as being one of complexation, poly(carboxylic acid)-Ca^{2+}. Furthermore, analyses in the presence of Fe^{III} ions demonstrated a significant change in the morphology of the incrustation of $CaCO_3$ crystals. Additionally, it has been proven that Fe^{III} ions significantly affect the performance of the inhibitor. Finally, the results indicated that in the absence of high concentrations of Fe^{III} ions, the poly(carboxylic acid) scale inhibitors can be an option to mitigate operating costs resulting from the deposition of inorganic scale in oil wells.

Keywords: scale inhibitor, polymer, poly(carboxylic acid), tube blocking tests, oil well, calcium carbonate

Introduction

One of the main technical-economic problems encountered in the oil industry, alongside corrosion and gas hydrates, is the formation of inorganic scales. This phenomenon occurs due to the deposition or adhesion of poorly soluble inorganic salts mainly inside pipes, reducing the diameter of ducts that transport oil, which results in damages to the production system, falling in the structures, loss of productivity, and expansion of expenses with interventions of repair and maintenance.[1] Inorganic scales are formed from reactions between anions (SO_4^{-II} and CO_3^{-II}) and divalent cations (Ca^{II}, Mg^{II}, Ba^{II}, and Sr^{II}) present in injection water (water introduced into the reservoir to stimulate oil production) and formation (water existing inside the rocks), respectively, under particular conditions of pressure, pH, and temperature.[2]

The most viable method in technical and economic terms to avert or delay the formation of scale is the use of chemical inhibitors (scale inhibitors). In recent years, these inhibitors have become important due to their simplicity in handling and efficiency in concentrations at the mg L^{-1} level. Furthermore, these inhibitors have minimal problems with corrosion and low cost.[3] In general, for a compound to work as a scale inhibitor, it must be soluble in brine, stable under operating conditions, and its chemical structure needs to have functional groups capable of interacting with the fouling ions.[4] The scale inhibitors may be organic or inorganic, containing functional groups in their structure, such as carboxylate, sulfonate, phosphate, and phosphonate, capable of sequestering (or blocking) divalent cations and, consequently, inhibiting the formation of insoluble crystals.[5,6] When applied correctly, these inhibitors are

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efficient at concentrations below 10 mg L^{-1}. However, these inorganic chemicals are involved as nutrients in the eutrophication process, which favor the increase of biological species and, consequently, the death of organisms that live in aquatic environments due to the deoxygenation of the water.[7] In recent years, polymer-based materials with higher biodegradability and lower toxicity compared to phosphate and phosphonate-based inhibitors, have been presented in the literature as the most viable alternative to inhibit or delay scale formation of $CaCO_3$.[8-12]

The efficiency of polymeric inhibitors is related to several factors, such as concentration, pH, salinity, temperature, and pressure.[13,14] More recently, some articles have shown that iron ions (Fe^{III}), originating from the corrosion of tubes and the dissolution of iron-based minerals, such as siderite, can promote changes in the performance of several inhibitors. For example, Graham *et al.*[5] described that the scale inhibitors tested showed less efficiency in the presence of 10 mg L^{-1} Fe^{III} and the lowest inhibition concentration (LIC) increased 100 times for phosphonate-based inhibitors.[5] In another study,[15] Fe^{III} showed an adverse impact on both phosphonates and polymeric inhibitors. Shen *et al.*[15] made a similar observation that the performance of inhibitors, including polycarboxylic acid, aminotri(methylene phosphonic acid), and carboxymethyl-inulin, dropped considerably in the presence of 5 mg L^{-1} Fe^{III}.[15] It is important to emphasize that all studies described in the literature to date have been limited to the evaluation of the influence of iron ions on the performance of inhibitors under static conditions (jar test). There are no reports of this evaluation under dynamic conditions (tube blocking tests).[16] Still, on the effect of iron ions, the literature presents contradictory results. For example, in another study by Zeino *et al.*,[17] it was found that the performance of inhibitors based on phosphonate and polyphosphinocarboxylic acid was reduced, while the performance of poly(vinyl sulfonate) and sulfonated copolymer was not significantly affected by the presence of Fe^{III} ions.[17] Additionally, in addition to the contradictions, there are no reports on the mechanism by which iron ions reduce the performance of inhibitors under dynamic conditions - formation of Fe-inhibitor complex or formation of ferrous crystals ($Fe(OH)_3$ or $FeCO_3$). Therefore, this work aims to evaluate, from a static and dynamic perspective, the performance of the polycarboxylic acid-based scale inhibitor in the presence of iron (Fe^{III}) ions. The static (jar test) and dynamic (tube blocking test) tests were performed according to NACE TM0197-2010[18] and NACE TM31105-2005[19] standards, respectively. The inhibitor LIC was determined under conditions similar to flow, temperature, and pressure found in oil wells. Furthermore, the effects caused by the presence of Fe^{III} ion on the precipitation process were also evaluated. The scale deposits were analyzed by X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM).

Experimental

Materials

Acetic acid (CH_3COOH), sodium acetate ($NaCH_3COO$), ethylenediaminetetraacetic acid (EDTA), sodium bicarbonate ($NaHCO_3$), and potassium bromide (KBr) were purchased from Synth (Diadema, Brazil). Barium chloride ($BaCl_2.2H_2O$), calcium chloride ($CaCl_2.2H_2O$), strontium chloride ($SrCl_2.6H_2O$), iron chloride ($FeCl_3.6H_2O$), and magnesium chloride ($MgCl_2.6H_2O$) were purchased from Sigma-Aldrich (Taufkirchen, Germany). Hydrochloric acid (HCl), sodium chloride (NaCl), sodium formate (NaCHOO), and sodium hydroxide (NaOH) were purchased from Chemco (Hortolândia, Brazil). The poly(carboxylic acid)-based scale inhibitor was kindly donated by Petrobras. Deionized water (≤ 18.2 MΩ cm) was produced using a Milli-Q water purification system. All chemicals were used as received without further purification.

Synthetic brines

The evaluation of the scale inhibitors require the preparation of two different brines: one rich in cations (formation water (FW)) and the other rich in anions (injection water (IW)), in line with the compositions shown in Table 1. The cations and anions were dissolved in deionized water under mechanical stirring for 24 h. Afterward, the resulting brines were vacuum filtered (0.45 µm) and stored in airtight vials at 4 °C. A fraction of the filtered anionic brine was used to solubilize the scale inhibitor at different concentrations. The inhibitors were solubilized under stirring for 24 h, without pH adjustment, and then stored under the same conditions as the brines without inhibitor.

Inhibitor evaluation - chemical compatibility test and dynamic scale loop test

The chemical compatibility between the scale inhibitor and the self-precipitating brine was evaluated through the NACE TM0197-2010 standard test.[18] In this work, the cationic and anionic brines were added separately in airtight vials in a volume ratio of 1:1. The anionic brine was used to prepare various doses of poly(carboxylic acid) inhibitor. Then, all airtight vials were preheated in an oven

Table 1. Synthetic brine compositions prepared

Ion	Formation water (FW) / (mg L^{-1})	Injection water (FW) / (mg L^{-1})	50:50 Brine mixture / (mg L^{-1})
CH_3COO^-	358	175	533
Ba^{2+}	245	< 0,1	245
HCO_3^-	320	315	635
Br^-	277	95	372
Ca^{2+}	5080	958	6038
Cl^-	50126	24018	74144
Sr^{2+}	146	22	168
$CHOO^-$	1	< 1	< 2
Mg^{2+}	687	844	1531
K^+	355	445	800
Na^+	22986	12785	35771
SO_4^{2-}	16	2011	2027

at 65 °C for 1 h. After preheating, the brines were mixed, photographed, and returned to the oven under the same conditions. Finally, new photographs were taken at 1, 2, and 24 h after mixing.

The dynamic scale loop test, which was intended to measure inhibitor efficiency under dynamic settings, was carried out using a Dynamic Scale Rig 4000 from Scaled Solutions, Livingston, Scotland, UK, following the NACE TM31105-2005 standard (capillary precipitation test).[19] In this apparatus, the brines are concurrently injected at a 1:1 ratio into a metallic capillary that is 1 meter long and 0.5 mm in diameter using high-performance liquid chromatography (HPLC) pumps (total flow rate of 10 mL min^{-1}). The capillary's temperature (65 ºC) and pressure (145 psi) remained unchanged throughout the test. The dynamic test displays the differential pressure as a function of time while the brines are flowing, which shows whether the capillary is partially or completely blocked. In this study, inhibitors were considered effective if the differential pressure did not rise over 1 psi for 60 min, or three times the blank duration (inhibitor-free mixture), whichever was greater.

Characterization of $CaCO_3$

To evaluate the effect of inhibitor and iron ions (17 mg L^{-1}) on the structure of $CaCO_3$ crystals, the composition of the precipitates was determined by the infrared-attenuated total reflectance (FTIR-ATR) spectroscopy studies using a Shimadzu IRAffinity-1 spectrophotometer (Shimadzu, Tokyo, Japan). All spectra (averaging 16 scans) were acquired in the 400 to 4000 cm^{-1} spectral region with a spectral resolution of 1 cm^{-1}. Scanning electron microscopy (SEM) was used to analyze the morphology of precipitates using a Hitachi TM3000 tabletop microscope (California, US) with a 15 kV accelerating voltage. The XRD patterns were obtained using a Shimadzu XRD-7000 (Tokyo, Japan) with Cu Kα radiation (λ = 1.5406, −40 kV, 30 mA), 2θ from 10 to 80°, a sample pitch of 0.02, and a scan speed of 5° min^{-1}. The distinct phases contained in the $CaCO_3$ precipitates were quantified (relative weight percentage) using Rietveld refinement. All diffraction peaks were discovered using the Materials Analysis Using the Diffraction (MAUD) program's search-match capabilities.[20]

Results and Discussion

Scale inhibitors are one of the most effective means of preventing or delaying the production of $CaCO_3$ inorganic scale. Because the performance of these inhibitors has been shown to vary greatly depending on the application settings, it is critical to investigate the many elements that significantly influence their performance. The tests used to assess these inhibitors, whether dynamic or static, are designed to imitate field circumstances and, as a result, estimate the true efficiency as well as the actuation mechanism. In this study, we employed static and dynamic methodologies to assess the efficacy of a polycarboxylic acid-based polymer as a scaling inhibitor under various situations. Furthermore, the inhibitor's effectiveness was evaluated in the presence and absence of iron ions (Fe^{III}).

Compatibility test

To determine the concentration limits at which inhibitor and fouling cations (Ca^{2+}) interact without producing byproducts, a chemical compatibility test was conducted. The compatibility studies demonstrated that, at inhibitor concentrations below 200 mg L^{-1}, a change in the mixture's visual appearance (the precipitation of $CaCO_3$ crystals) occurred at intervals of 0, 1, 2, and 24 h (Figure 1).

The visual appearance of the mixture (precipitation of $CaCO_3$ crystals) did not alter for up to 24 h for combinations containing inhibitors at concentrations over 200 mg L^{-1}.

The findings of the chemical compatibility test demonstrated that there was no production of insoluble by-products due to the interaction of the incrusting cation with the polymers across the whole period examined. In addition, the crystals of $CaCO_3$ precipitated in low inhibitor concentrations indicated that the scale inhibitor was efficient only in concentrations over 200 mg L^{-1}. The compatibility test performed in the presence of Fe^{III} ions revealed that immediately after mixing there was a visual change (formation of a yellowish precipitate, Figure 2) for inhibitor concentrations below 500 mg L^{-1}. After the

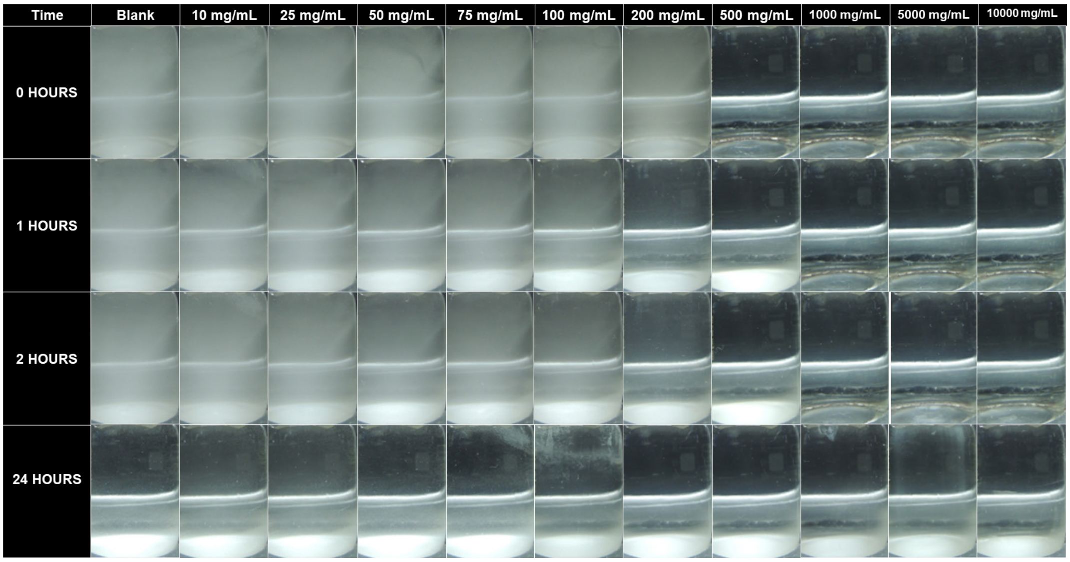

Figure 1. The visual appearance of the self-precipitating mixed brines and scale inhibitor brines in the chemical compatibility test at 65 °C, without Fe^{III} ion.

1 h period, all samples showed visual changes (again indicating the formation of precipitate). As a preliminary observation, it is possible to confirm that the presence of Fe^{III} ions significantly alters the action of the scale inhibitor. Only with the result of the compatibility test, it is imprecise to define the mechanism. However, the presence of Fe^{III} ions may be interfering in two ways: (*i*) interacting chemically with the inhibitor (chemical bonding); or (*ii*) precipitating in the form of $Fe(OH)_3$ (considering pH = 7.4 and Kps = 2.80×10^{-39}) and acting as a seed, favoring the growth of $CaCO_3$ crystals on its surface. Iron ions hydrolyze to create iron hydroxide colloid when added to water, as seen by the pale-yellow hue of the solution. Because iron hydroxide colloid has a positive charge and poly(carboxylic acid)-based scale inhibitor has a negative charge, electrostatic adsorption is favored. As a flocculant, the iron hydroxide colloid exhibits flocculation capabilities that can expedite the flocculation settling of calcium carbonate microcrystals distributed by scale inhibitor.

Dynamic scale loop test

The dynamic efficiency test uses capillaries to model oil well conditions in order to forecast the development of precipitate from a mixture of brines (cationic and anionic in volumetric ratios of 1:1) under certain temperature and pressure parameters. Figures 3a and 3b present the results of the scale inhibitor at 65 ºC in the absence and presence of Fe^{III} ion, respectively. The outcomes showed that the inhibitor administered without the presence Fe^{III}

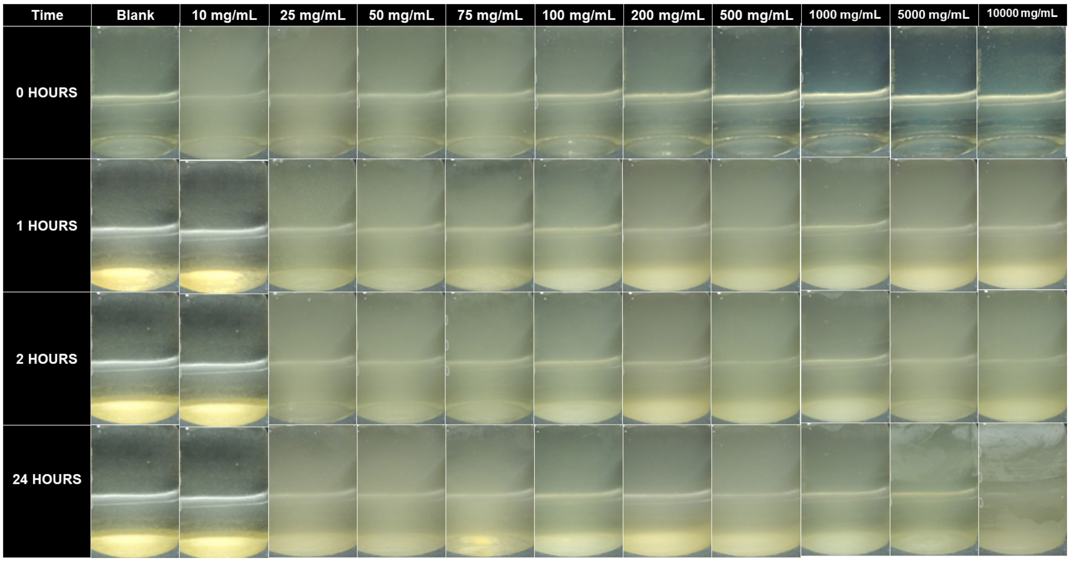

Figure 2. The visual appearance of the self-precipitating mixed brines and scale inhibitor brines in the chemical compatibility test at 65 °C, with Fe^{III} ion.

ions was effective and chemically compatible (between 30 and 60 mg L^{-1}), displaying a LIC of 30 mg L^{-1} (Figure 3a). Note that the 20 mg L^{-1} inhibitor was sufficient to delay fouling (increase in blank time, the time at which the pressure suddenly increased for the mixed brine without inhibitor), but not inhibit it. The application of 20 mg L^{-1}, although it did not prevent the formation of CaCO$_3$ crystals, significantly altered the kinetics of the formation. It was observed that even with the pressure differential increasing, it did not exceed the 5 psi of the blank test. As the increase in the inhibitor concentration did not change the pressure differential, it can be concluded, similarly to the compatibility test, that there was no chemical incompatibility. In the dynamic test, incompatibility is identified when the fouling time reduces with increasing inhibitor concentration, indicating the formation of insoluble by-products from inhibitor interactions.

In Figure 3b, which shows the performance results of the inhibitor in the presence of Fe$^{\text{III}}$ ions, it is possible to observe that the blank solution exceeded 1 psi in 10 min. For the solution containing 30 mg L^{-1} of inhibitor (LIC of the inhibitor in the absence of Fe$^{\text{III}}$ ions), a behavior similar to the blank was observed. It was preliminarily noticed that under dynamic conditions the performance of the inhibitor is altered in the presence of Fe$^{\text{III}}$ ions. As the concentration increased to 60 mg L^{-1}, there was a delay in the precipitation time. However, total capillary obstruction was also observed after 35 min of testing. This indicates that the polymer acted as an inhibitor against the precipitation of CaCO$_3$, however, the concentration tested was not sufficient to prevent their growth. By increasing the concentration to 100 and 120 mg L^{-1}, greater effectiveness in the inhibition mechanism was observed, although the differential pressure reached approximately 1.5 psi in the 60 min of testing. For the concentration of 160 mg L^{-1}, the behavior observed was characteristic of incompatibility. The increase in the pressure differential observed after 60 min of testing, of 2.5 psi, was greater than that observed for the solution with 120 mg L^{-1}. Therefore, it was not possible to determine the minimum effective concentration to prevent the formation of crystals under these conditions.

According to the literature,[21] the results, particularly those related to the LIC, can be deemed adequate. For instance, when used at 80 ºC, the inhibitor 1-hydroxyethane-1,1-diphosphonic acid (HEDP) has a LIC of 25 mg L^{-1}.[19] In a different investigation, Wang *et al.*[13] found that when applied at room temperature, 15 mg L^{-1} of partly hydrolyzed polyacrylamide (HPAM) was able to prevent the production of CaCO$_3$. In addition, it should be emphasized that this article presents, in an unprecedented way, the influence of Fe$^{\text{III}}$ ions on the performance of polymeric inhibitors under dynamic conditions (tube blocking test).

Investigations into the scale-inhibition mechanism and crystal characterization

The CaCO$_3$ precipitated from the solutions with and without inhibitor (30 mg L^{-1}), in the presence and absence of Fe$^{\text{III}}$ ion, was characterized by measuring its chemical composition (FTIR), morphology (SEM), and crystalline forms (XRD). The chemical structures of the inhibitor polymer (carboxylic acid) and the CaCO$_3$ precipitate were studied using FTIR spectroscopy. Figure 4 shows the four vibrational modes in which the carbonate absorption bands may be seen: ν_1 (symmetric stretching) at 1081 cm^{-1}, ν_2 (out-of-plane bending) at 849 cm^{-1}, ν_3 (doubly planar asymmetric stretching) at 1441 cm^{-1}, and ν_4 (doubly degenerate planar bending) at 709 cm^{-1}. No significant difference was observed between the spectra of Figure 4. In addition, the characteristic bands of the inhibitor were also not observed.

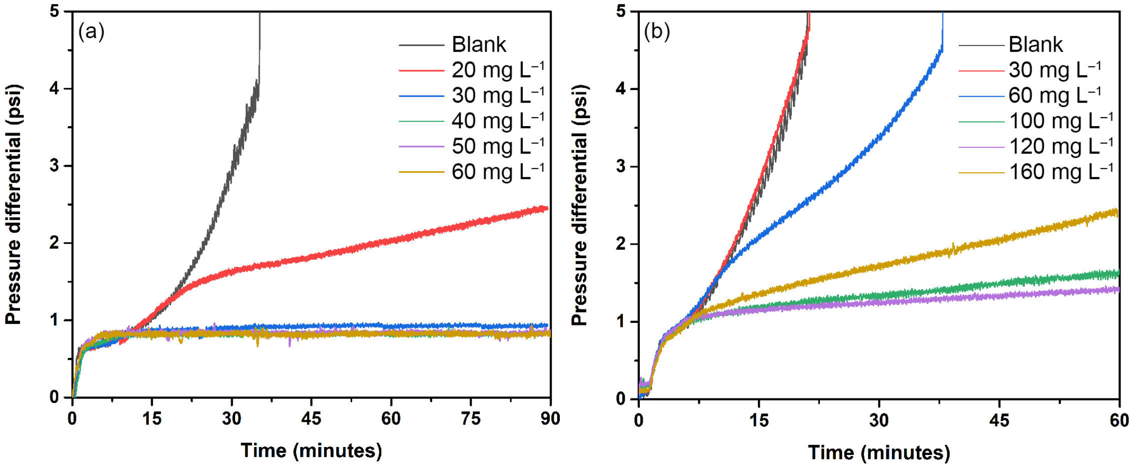

Figure 3. Scale inhibitor dynamic tube blocking test results at 145 psi and 65 ºC (a) without Fe$^{\text{III}}$ ion and (b) with Fe$^{\text{III}}$ ion. For all tests, a flow rate of 10 mL min^{-1} and a pH of 6.50 were used.

The observed spectral equality in the formed $CaCO_3$ (precipitated) in the absence (Figures 4a and 4c) and in the presence of inhibitor (Figures 4b and 4d), with and without iron, demonstrates that the scale inhibitor was not adsorbed, because there are no poly(carboxylic acid) characteristic bands, such as $-CH_2-$ and –OH, in the $CaCO_3$ spectra. As a result, it is feasible to conclude that the inhibitor's mode of action involves the capture of fouling cations.[22]

The $CaCO_3$ morphology and structure were investigated concurrently. Figure 5a shows that non-uniform products with lengths ranging from 8 to 20 m were generated from $CaCO_3$ in the absence of inhibitor and Fe^{III} ions. The SEM images for $CaCO_3$ in Figure 5b, in the presence of the inhibitor, show an irregular to spherical morphological shape as well as size reduction to below 5 µm. Figures 5c and 5d show the SEM images for $CaCO_3$ in the presence of Fe^{III} ions, and in the simultaneous presence of inhibitor and Fe^{III} ions, respectively. It can be seen that the presence of Fe^{III} changed the morphology of the $CaCO_3$ crystals from non-uniform to aggregate, regardless of the presence of the inhibitor.

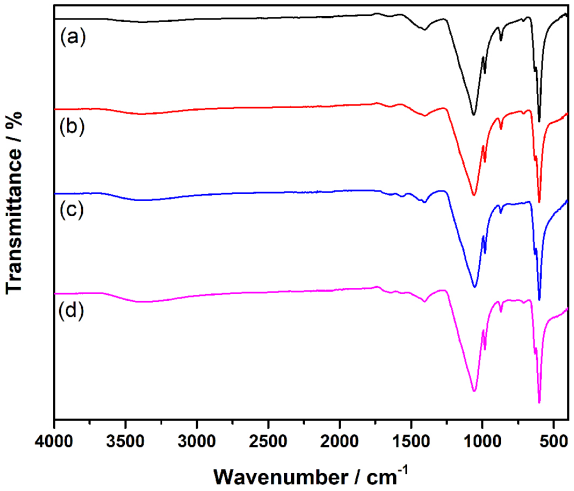

Figure 4. FTIR-ATR spectra of powder (a) $CaCO_3$; (b) $CaCO_3$ with inhibitor; (c) $CaCO_3$ with Fe^{III} ion; and (d) $CaCO_3$ with inhibitor and with Fe^{III} ion. The samples were collected after a compatibility test (at 65 °C) between self-precipitating brines for $CaCO_3$ formation.

XRD spectra of $CaCO_3$ precipitate without and with inhibitor, in the presence and absence of Fe^{III} ions, are shown in Figure 6. In Figures 6a and 6b, both without inhibitor, it can be observed that the precipitated crystal only includes one $CaCO_3$ polymorph, aragonite. The main diffraction peaks at 26.30, 27.15, 33.08, 36.06, 42.85, 44.80, 48.30, and 50.14° corresponding to the aragonite crystal faces are 111, 021, 012, 102, 220, 221, 202, and 113, respectively. Figures 6c and 6d, both with an inhibitor, show the diffraction peaks for $CaCO_3$ similar to those obtained in the absence of an inhibitor. However, the peaks between 45 and 50° disappeared. The quantitative study of the phases following the Rietveld refinement revealed that they were 62% aragonite, which means that in addition to a change in morphology, there was a phase change.[23,24]

In conclusion, the characterization techniques used enabled a deeper comprehension of the $CaCO_3$ scale generation as well as the inhibitor mode of action. The above-described shift in polymorphism is crucial because incidences of fouling development can occur even when an inhibitor is present (in inhibitor concentration lower than LIC, for example), however, because the crystals would be less resistant, reparative treatment would be possible (for example, through mechanical methods).

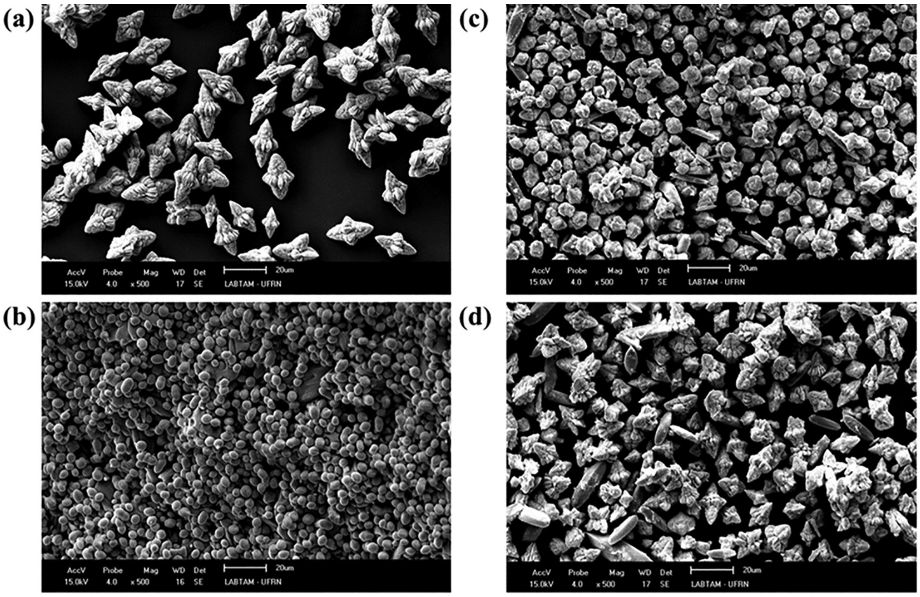

Figure 5. SEM images of (a) $CaCO_3$; (b) $CaCO_3$ with inhibitor; (c) $CaCO_3$ with Fe^{III} ion; and (d) $CaCO_3$ with inhibitor and with Fe^{III} ion.

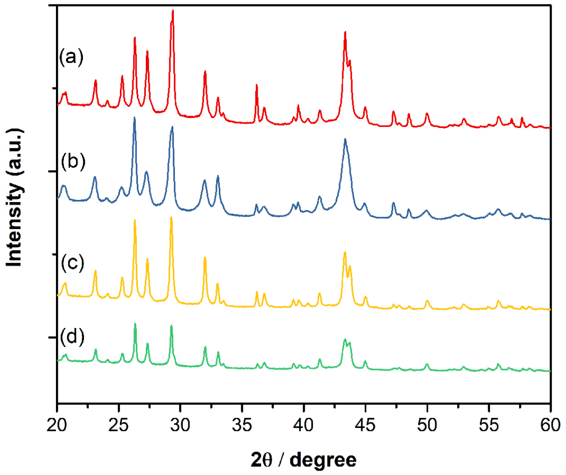

Figure 6. XRD pattern of $CaCO_3$ crystals: (a) $CaCO_3$; (b) $CaCO_3$ with inhibitor; (c) $CaCO_3$ with Fe^{III} ion; and (d) $CaCO_3$ with inhibitor and with Fe^{III} ion.

Conclusions

When used in oil well settings, poly(carboxylic acid) proved effective as a scale inhibitor at a minimal concentration (MEC) of 30 mg L^{-1}. Additionally, at 100 °C, it demonstrated chemical compatibility with fouling cations. The complexing of fouling cations was shown to be the inhibitory mechanism by the selected characterization techniques. Additionally, the $CaCO_3$ polymorphism changed as a result of the inhibitor's presence. The combined XRD, IR-ATR, and SEM analyses allowed us to identify the inhibitor's mechanism of action as being one of complexation, poly(carboxylic acid)-Ca^{2+}. Furthermore, analyses in the presence of Fe^{III} ions demonstrated a significant change in the morphology of the $CaCO_3$ crystals. Additionally, it has been proven that Fe^{III} ions significantly affect the performance of the inhibitor. Finally, the results indicated that in the absence of high concentrations of Fe^{III} ions, the poly(carboxylic acid) scale inhibitors can be used to reduce operating costs caused by inorganic scale deposition in oil wells.

Acknowledgments

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