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Inhibición de la corrosión y las incrustaciones de CaSO₄ mediante la aplicación de polielectrolitos

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This research contributes to the field of enhanced hydrocarbon recovery. The polyelectrolytes evaluated in this work can be used for the control of corrosion and mineral fouling in oil reservoirs. The high temperature and salinity conditions of subway formations hinder the extraction of hydrocarbons due to the formation of mineral precipitates. In this regard, it is important to develop chemicals that prevent the formation of mineral scale in production wells and extraction equipment. Most of the products used in the oil industry come from foreign companies and are sometimes not effective enough. Therefore, it is necessary to develop proprietary technology to ensure domestic production and achieve energy dependence. The chemicals used in scale control must be effective in low pH conditions and must not promote corrosion of metallic materials. It has been found that performance of scale inhibitors is always influenced by the chemical characteristics and functional groups of the molecules. Therefore, it is necessary to perform precipitation and corrosion tests to select the appropriate inhibitor. The results showed that PEs inhibit 80% of CaSO₄ precipitation when added at low concentrations. PEs were found to inhibit the corrosion of carbon steel, generating efficiencies above 60%. HP_ITA was more effective in corrosion inhibition.

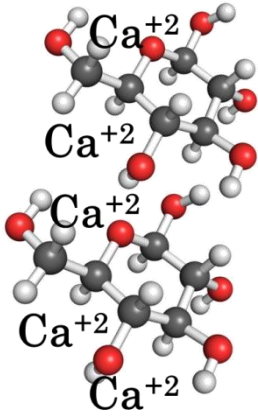
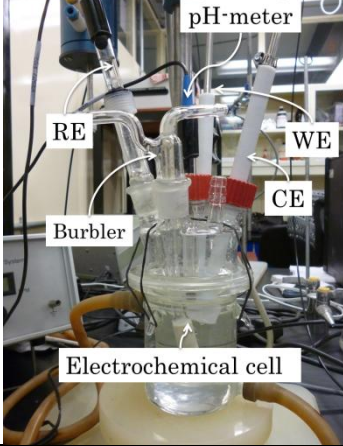

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Abstract

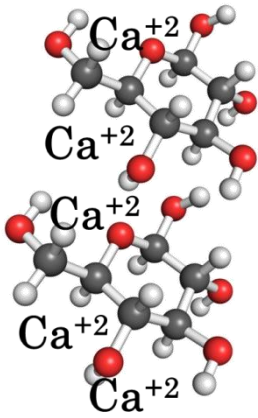
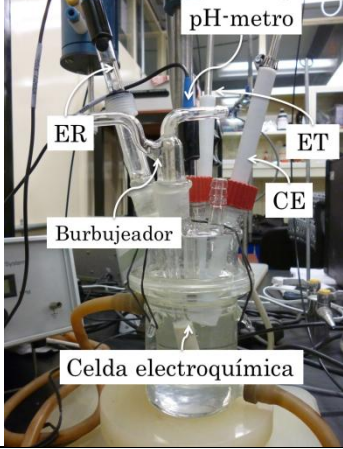

This study aimed to inhibit both the precipitation of CaSO_4 and the corrosion of carbon steel by applying polyelectrolytes (PEs). A homopolymer of itaconic acid (HP_ITA) and a copolymer of itaconic acid and sodium vinyl sulfonate (HP_ITA-VS) were synthesized. In addition, static precipitation methods were used to induce the formation of CaSO_4 and evaluate the inhibition capacity of the PEs. Also, electrochemical techniques were used to analyze the corrosion processes and evaluate the effectiveness of the PEs. The results showed that the PEs inhibit the precipitation of CaSO_4 by up to 80% at low concentrations. On the other hand, it was found that the PEs inhibit the corrosion of carbon steel, generating efficiencies higher than 58%. In conclusion, the efficiency in inhibiting corrosion was found to be influenced by the specific functional groups present in the PEs.

Objectives	Methodology	Contribution
<ul style="list-style-type: none">• Synthesize polyelectrolytes• Inhibit corrosion and CaSO_4 precipitation• Found the best concentration	<ul style="list-style-type: none">• Infrared spectroscopy• Static precipitation test (NACE-TM0374).• Electrochemical measures	Polyelectrolytes inhibit both corrosion and CaSO_4 precipitation
		

Inhibition, Polyelectrolyte, Efficiency

Resumen

El objetivo de este trabajo es inhibir la precipitación del CaSO_4 y la corrosión del acero al carbono mediante la aplicación de polielectrolitos (PEs). Se sintetizo un homopolímero de ácido itacónico (HP_ITA) y un copolímero de ácido itacónico y vinil sulfonato de sodio (HP_ITA-VS). Además, se utilizaron métodos de precipitación estática para inducir la formación de CaSO_4 y evaluar la capacidad de inhibición de los PEs. También, se utilizaron técnicas electroquímicas para analizar los procesos de corrosión y evaluar la efectividad de los PEs. Los resultados mostraron que los PEs inhiben en un 80% la precipitación del CaSO_4 cuando se adicionan en bajas concentraciones. Por otra parte, se encontró que los PEs inhiben la corrosión del acero al carbono, generando eficiencias superiores al 58%. En conclusión, se tiene que la eficiencia en la inhibición de la corrosión es influenciada por los grupos funcionales que conforman a los PEs evaluados.

Objetivo	Metodología	Contribución
<ul style="list-style-type: none">• Sintetizar polielectrolitos• Inhibir la corrosión y la precipitación de CaSO_4• Determinar la concentración adecuada	<ul style="list-style-type: none">• Espectroscopia de infrarrojo.• Precipitación mineral (NACE-TM0374).• Mediciones electroquímicas	Los polielectrolitos son capaces de inhibir la corrosión y la precipitación de CaSO_4
		

Inhibición, Polielectrolito, Eficiencia

Introduction

Corrosion and mineral scaling are common issues in the oil industry, especially in processes or operations involving the handling of water streams with high ionic content (Olajire, 2015). Mineral scales are typically composed of calcium carbonate (CaCO_3), calcium sulfate (CaSO_4), barium sulfate (BaSO_4), strontium sulfate (SrSO_4), calcium phosphate ($\text{Ca}_3(\text{PO}_4)_2$), or aluminosilicates. In some cases, scale deposits hinder oil and gas extraction operations by obstructing the pores that make up the formation. This issue is known as "formation damage" and arises from the incompatibility of waters within the formation (Peretomode et al., 2022). Additionally, mineral scaling can damage surface equipment (pipelines, boilers, pumps, heat exchangers, cooling towers, etc.), leading to production shutdowns or economic costs (Visser & Jeurink, 1997; Walker et al., 2012).

Scale inhibitors are chemical substances that prevent the formation of mineral scales (Kamal et al., 2018). Ethylenediamine tetra(methylenephosphonate) (EDTMP), butylenediamine tetra(methylenephosphonate) (BDTMP), pentylenediamine tetra(methylenephosphonate) (PDTMP), hexamethylenediamine tetra(methylenephosphonate) (HDTMP), and ethylenediaminetetraacetic acid (EDTA) are examples of substances used as scale inhibitors (Mpelwa & Tang, 2019). However, it has been shown that these substances inhibit scaling only when used at high concentrations and generally precipitate or degrade under high-temperature or highly acidic conditions (Martinod et al., 2008). Additionally, there are reports indicating that phosphate-based inhibitors can cause eutrophication problems when discharged into water bodies (Nowack, 2003).

Polyelectrolytes are polymeric macromolecules that contain ionic groups in their structures (carboxylates, sulfonates, phosphates, etc.). They are used in water purification and treatment for the flotation of solid particles (Bolto & Gregory, 2007). The ionic groups in polyelectrolytes interact with charged particles and can form stable complexes to prevent the sedimentation of minerals (Meka et al., 2017). Additionally, they are used at low concentrations, and some are biodegradable. Polyelectrolytes can be a good option to prevent scale formation, and their ionic groups can also interact with charged surfaces to prevent the corrosion of metallic materials (Chen et al., 2019).

The objective of this work is to synthesize polyelectrolytes derived from itaconic acid and sodium vinyl sulfonate to analyze their effect on the inhibition of corrosion and the precipitation of CaSO_4 . Additionally, it aims to determine the appropriate inhibitor concentration and analyze the effect of chemical structure on the inhibition percentage.

Methodology

Polyelectrolyte synthesis

In this study, poly(itaconic acid) (HP_ITA) and poly(itaconic acid-co-sodium vinyl sulfonate) (CP_ITAC-VS) were synthesized. The synthesis of the inhibitors was based on the procedure described in patent US 11046605 B2. Subsequently, Fourier Transform Infrared Spectroscopy (FTIR) analyses were conducted to determine the functionality of the synthesized polyelectrolytes (Gao et al., 2015).

Precipitation test

The NACE-TM0374 methodology was used to analyze the effect of polyelectrolytes on the precipitation of CaSO_4 (Sanni et al., 2019). The test involves mixing (1:1) two brines (one containing calcium ions (Ca^{2+}) and the other containing sulfate ions (SO_4^{2-})) in different vials and adding a specific amount of polyelectrolyte (0, 50, 100, 150, and 200 ppm) to the mixture. The mixtures (brine + polyelectrolyte) are then heated to 80°C for 6 hours. After the heating period, the contents of each vial are filtered, and the amount of dissolved Ca^{2+} is determined. To evaluate the efficiency of CaSO_4 precipitation inhibition, the initial and final Ca^{2+} concentrations are compared using Equation 1.

$$\%EIP = \frac{C_A - C_B}{C_C - C_B} \quad [1]$$

Where C_A is the amount of Ca^{2+} in the solutions prepared with polyelectrolyte after heating, C_B is the amount of Ca^{2+} in the reference solution (without polyelectrolyte) after heating, and C_C is the amount of Ca^{2+} in the reference solution before heating.

Electrochemical measures

Electrochemical tests were conducted to analyze the effect of polyelectrolytes on the corrosion of AISI-1018 steel (Zou et al., 2011). For each experiment, fresh electrolyte solutions were prepared using analytical-grade reagents and deionized water. The corrosive medium consisted of a 0.01 M H_2SO_4 + 1.0 M NaCl solution. Additionally, to promote the formation of mineral scales, the corrosive solution also contained 0.01 M $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$. Measurements were performed at various polyelectrolyte concentrations (0, 100, 120, 150, 180, 200 ppm) to determine the optimal inhibitor concentration.

The electrochemical tests were conducted in a 250 mL glass cell under static conditions, atmospheric pressure, and a constant temperature of 40°C. The cell contained three electrodes: a piece of AISI-1018 steel coated with epoxy resin (0.71 cm² exposed area) as the working electrode (WE), a graphite rod as the counter electrode (CE), and a saturated calomel electrode (SCE) placed in a Luggin capillary as the reference electrode (RE). Potentiodynamic scans were performed to determine the corrosion rate. The WE was polarized in the cathodic direction to -300 mV vs OCP, followed by polarization in the anodic direction to +200 mV vs OCP.

Results

Spectroscopic analysis

Figure 1 shows the FTIR spectra of HP_ITA and CP_ITA-VS. In both spectra, a broad band is observed in the 3750–3400 cm⁻¹ region, representing the stretching of the O–H bond. Additionally, broad adsorption bands are seen in the 3400–3250 cm⁻¹ region, indicating the stretching of the –CH₂– bonds due to the vibration of the bonds linking the repeating units, and a band in the 1410–1380 cm⁻¹ interval indicating the bending of the O–H bond. Specifically, in the HP_ITA spectrum, a band at 1701 cm⁻¹ corresponds to the stretching of the C=O bond, and the band at 1176 cm⁻¹ represents the stretching of the C–O bond. On the other hand, in the CP_ITA-VS spectrum, a band at 1713 cm⁻¹ corresponds to the stretching of the C=O bond, and two adsorption bands at 1153 cm⁻¹ and 1036 cm⁻¹ indicate the asymmetric and symmetric stretching of the S=O bonds (Laschewsky, 2012).

Box 1

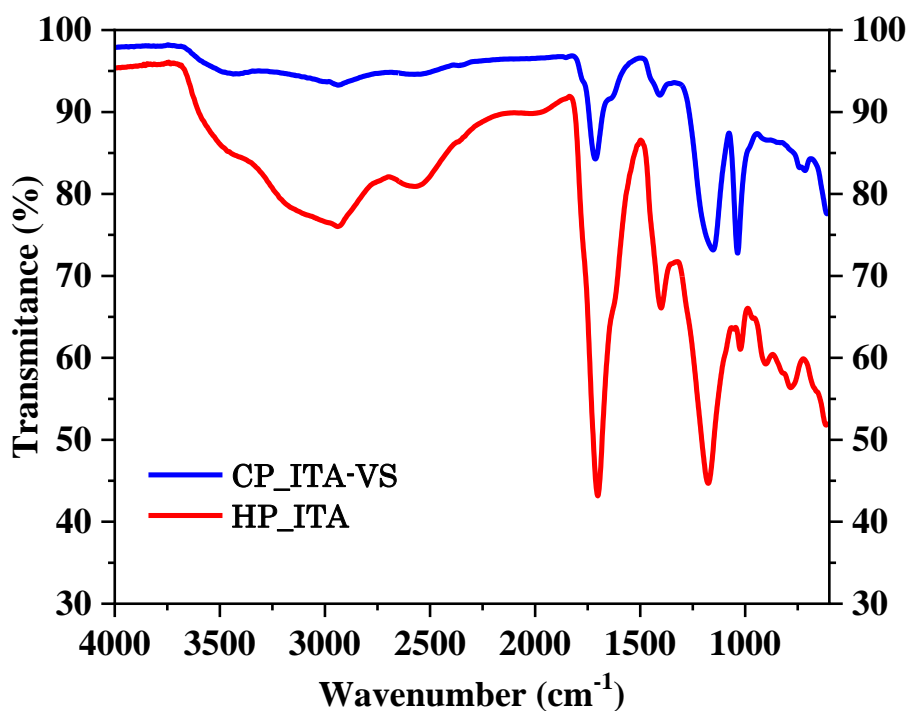


Figure 1

Infrared spectra of the synthesized polyelectrolytes

Source: Own construction

Inhibition of CaSO₄ precipitation

The results indicate that the polyelectrolytes can inhibit the precipitation of CaSO₄ at different concentrations. These polyelectrolytes have the ability to form stable complexes with the ionic species. Therefore, the inhibition can be attributed to the interaction of Ca²⁺ with the functional groups of the polyelectrolytes (Bisatto et al., 2022). Table 1 shows the results of the static precipitation tests. The synthesized polyelectrolytes exhibit high efficiency in inhibiting precipitation (EIP), with values greater than 80%. Additionally, it was found that the percentage of efficiency in inhibiting precipitation (EIP) increases as the concentration of polyelectrolytes increases.

Box 2

Table 1
The results of static precipitation tests at different polyelectrolyte concentrations

Concentration (ppm)	Efficiency in inhibiting precipitation	
	HP_ITA	HP_ITA
50	80.7 %	83.9%
100	89.1%	90.6%
150	97.8%	98.9%
200	>99%	>99%

Source: *Own construction*

Corrosion inhibition

Figure 2 shows the potentiodynamic scans of the working electrode (WE) in the presence and absence of HP_ITA. In the absence of polyelectrolyte (reference), the anodic current density (ACD) increases as the potential becomes more positive, indicating that anodic polarization promotes the corrosion of the metal surface. On the other hand, the cathodic current density (CCD) increases as the potential becomes more negative, indicating that cathodic polarization promotes the reduction of acidic species on the metal surface (Ramírez-Estrada et al., 2020).

Box 3

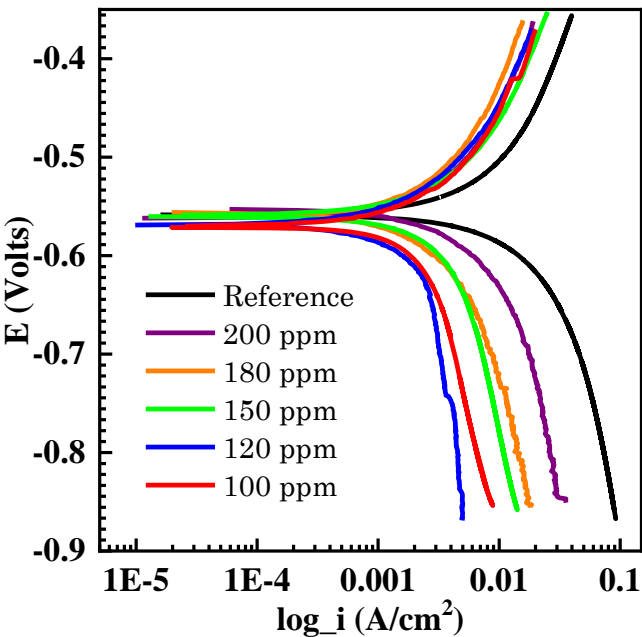


Figure 2
Potentiodynamic scans of AISI-1018 steel at different concentrations of HP_ITA

Source: *Own construction*

The ACD and CCD obtained in the presence of HP_ITA are lower than the current densities of the reference. The decrease in ACD in the presence of HP_ITA represents less corrosion of the material and suggests the formation of a protective film on the electrode surface (Tlili et al., 2008).

On the other hand, the decrease in CCD in the presence of HP_ITA indicates that there are fewer active sites on the metal surface for the reduction of acidic species. This behavior can be attributed to the adsorption of HP_ITA molecules on the surface of the working electrode (Morizot et al., 1999).

Box 4

Table 2
Electrochemical parameters obtained from the potentiodynamic scans of AISI-1018 steel at different concentrations of HP_ITA

Concentration (ppm)	E_{corr} (Volts)	i_{corr} ($A \cdot cm^{-2}$)	β_a ($mV \cdot dec^{-1}$)	$-\beta_c$ ($mV \cdot dec^{-1}$)	CIE (%)
Reference	-0.564	7.95×10^{-3}	68	70	—
100	-0.570	2.83×10^{-3}	40	80	64.4
120	-0.565	2.17×10^{-3}	75	80	72.7
150	-0.560	2.48×10^{-3}	75	75	68.8
180	-0.560	2.91×10^{-3}	80	75	63.4
200	-0.560	3.28×10^{-3}	60	50	58.7

E_{corr} = corrosion potential; i_{corr} = current density of corrosion; β_a = anodic Tafel slope; β_c = cathodic Tafel slope; ICE = corrosion inhibition efficiency.

Source: Own construction

The electrochemical parameters from the potentiodynamic sweeps (Figure 2) are presented in Table 2. The results indicate that the corrosion inhibition efficiency (CIE) is greater than 58% and that at 120 ppm the efficiency is 72.7%. Additionally, it was found that the CIE decreases as the HP_ITA concentration increases (>120 ppm). PEs are soluble species that interact with ionic species and water molecules present on the metal surface. As the concentration of HP_ITA increases, more water molecules are attracted to the surface of the electrode (ET), which leads to an increase in the current density of corrosion (i_{corr}) and a decrease in the CIE. The appearance of the electrodes (images not shown) matches with the results of the electrochemical tests. In the absence of PEs (reference), the accumulation of $CaSO_4$ crystals on the electrode surface is observed. At low concentrations of HP_ITA (<180 ppm), the DCA values are lower than the reference, and the amount of scale deposits decreases. With 150 ppm of HP_ITA, very small crystals are observed on the electrode surface. However, at high concentrations (>150 ppm), the CIE values decrease, indicating an increase in the corrosion of the material. The images obtained at 180 ppm and 200 ppm show an increase in the number of crystals, indicating a rise in the corrosion of the metal surface.

Box 5

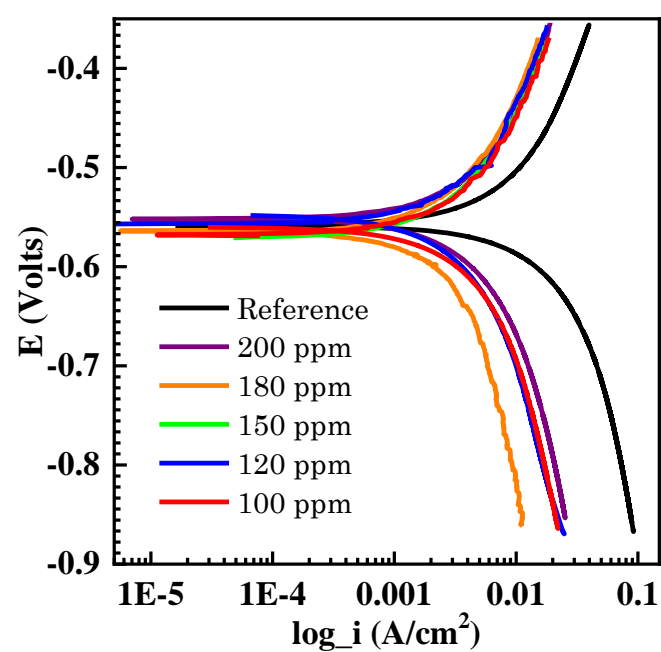


Figure 3
Potentiodynamic scans of AISI-1018 steel at different concentrations of CP_ITA-VS

Source: Own construction

Figure 3 shows the potentiodynamic sweeps of the WE in the presence and absence of CP_ITA-VS. The DCA and DCC obtained in the presence of CP_ITA-VS are lower than the current densities of the reference (Rehim et al., 2008). The decrease in the DCA by CP_ITA-VS represents less corrosion of the material and suggests the formation of a protective film on the electrode surface. Additionally, no changes are observed in the shape of the anodic curves when different concentrations of CP_ITA-VS are used, indicating that increasing the concentration does not alter the kinetics of the anodic reaction.

Box 6

Table 3
Electrochemical parameters obtained from the potentiodynamic scans of AISI-1018 steel at different concentrations of CP_ITA-VS

Concentration (ppm)	E_{corr} (Volts)	i_{corr} (A•cm ⁻²)	β_a (mV•dec ⁻¹)	$-\beta_c$ (mV•dec ⁻¹)	CIE (%)
Reference	-0.564	7.95×10^{-3}	68	70	—
100	-0.560	3.14×10^{-3}	75	85	60.5
120	-0.550	2.51×10^{-3}	70	80	68.4
150	-0.571	2.31×10^{-3}	40	60	70.9
180	-0.570	2.68×10^{-3}	65	55	66.3
200	-0.550	2.81×10^{-3}	55	50	64.7

E_{corr} = corrosion potential; i_{corr} = current density of corrosion; β_a = anodic Tafel slope; β_c = cathodic Tafel slope; ICE = corrosion inhibition efficiency

Source: *Own construction*

The electrochemical parameters from the potentiodynamic sweeps (Figure 3) are presented in Table 3. The results indicate that at 150 ppm of CP_ITA-VS, the efficiency is 70.9%. However, it was found that as the concentration increases (>150 ppm), the corrosion inhibition efficiency (CIE) decreases. This behavior can be attributed to the increase in water molecules on the electrode surface; as the concentration of CP_ITA-VS increases, more water molecules are attracted to the electrode surface, which results in an increase in the corrosion current (i_{corr}) and a decrease in the CIE.

When comparing the results from Tables 2 and 3, it is found that the polyelectrolyte HP_ITA is more effective than CP_ITA-VS in corrosion inhibition. The CIE of HP_ITA is 72.7% at 120 ppm, while the CIE of CP_ITA-VS is 70.9% at 150 ppm. The difference in performance can be attributed to the functional groups that make up each polyelectrolyte (Saleh & Atia, 2006). The HP_ITA compound is composed solely of carboxyl groups, whereas the CP_ITA-VS compound contains both carboxyl and sulfonate groups.

Conclusions

The results showed that the PEs inhibit 80% of CaSO₄ precipitation when added at low concentrations. It was also found that the synthesized PEs inhibit carbon steel corrosion, achieving efficiencies greater than 60%. The HP_ITA compound was more effective in corrosion inhibition; the CIE of HP_ITA is 72.7% at 120 ppm, while the CIE of CP_ITA-VS is 70.9% at 150 ppm. Therefore, the sulfonate groups reduce the inhibition capacity of CP_ITA-VS.

Declarations

Conflict of interest

The authors declare that they have no conflicts of interest. They have no financial interests or personal relationships that could have influenced this book.

Authors' contributions

Ramírez-Estrada, Alejandro: Contributed to the conceptualization and design of the study, development of the methodology, data analysis, and manuscript writing.

Mena-Cervantes, Violeta Yasmín: Participated in project supervision, critical analysis of results, and manuscript review.

Aguilar-Aguilar, Fidel Alejandro: Collaborated in conducting laboratory experiments and in the collection and processing of experimental data.

Hernández-Altamirano, Raúl: Contributed to the technical analysis of results, data validation, and technical and academic review of the final document.

Availability of data and materials

The data and graphics shown in this study are available.

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Abbreviations

- ACD: Anodic current density
- CCD: Cathodic current density
- CE: Counter electrode
- CIE: Corrosion inhibition efficiency
- E_{corr} : Corrosion potential
- EIP: Efficiency in inhibiting precipitation
- FTIR: Fourier transform infrared spectroscopy
- HP_ITA: Poly(itaconic acid)
- HP_ITA-VS: Poly(itaconic acid-co-sodium vinyl sulfonate)
- I_{corr} : Current density of corrosion
- OCP: Open circuit potential
- PEs: Polyelectrolytes
- RE: Reference electrode
- WE: Working electrode

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