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Green inhibitor for corrosion protection of ASTM A1011 steel in saline medium using a dynamic system

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Abstract

Currently, inhibitors represent an economical option for protecting metals against corrosion. However, most inhibitors are synthetic and harmful to the environment. The present study focuses on estimating the anticorrosive effect of *Oreocallis grandiflora* extract (OGE) on ASTM A1011 steel immersed in a 3.5 wt.% NaCl solution. The investigation was carried out using open circuit potential (OCP) and potentiodynamic polarization curves to different concentrations of OGE in saline solution using a dynamic system. The results of potentiodynamic polarization curves indicated that the corrosion rate of ASTM A1011 steel decreased as the OGE concentration was increased. This inhibitor demonstrated an efficiency of 64 to 88 % in enhancing the corrosion resistance of the steel substrates. The value of the free energy of adsorption was $-23.59 \text{ kJ mol}^{-1}$, indicating that the OGE molecules are spontaneously adsorbed onto the surface of the substrate. The results of this study showed that *Oreocallis grandiflora* extract has high potential as a green corrosion inhibitor, as it forms a barrier between the substrate and the corrosive solution.

Keywords

Oreocallis grandiflora extract, eco-friendly inhibitor, steel sheets, corrosion resistance, adsorption isotherm

Introduction

Metal corrosion is a problem that affects all types of industries. This problem causes significant economic losses, damage to human health, and adverse effects on ecosystems [1]. Therefore, it is important to protect metals from corrosion to avoid any loss [2]. There are various ways to protect metals from corrosion, such as cathodic protection, sacrificial anodes, anticorrosive paints, corrosion inhibitors, etc. [3].

In the industrial sector, cooling water pipes, boiler pipes, drainage pipes, etc., suffer extensive damage from corrosion, which is why some technology must be used to mitigate this phenomenon,

one of these solutions is corrosion inhibitors, although the vast majority are synthetic, whether organic or inorganic, although it is true that these inhibitors extend the useful life of the pipes, they are not environmentally friendly [1,4]. Currently, inhibitors represent an economical option to protect metals from corrosion, which is why they are widely used in industry. However, most inhibitors are of synthetic origin, which are harmful to the environment. Recent research has shown that corrosion inhibitors derived from natural products exhibit strong adsorption on the surfaces of metallic substrates, providing a degree of protection against corrosive agents. In turn, these natural inhibitors are low-cost, highly available, and contribute to the planet's circular economy [5]. These natural inhibitors have in their composition as a main characteristic a variety of organic compounds, such as flavonoids, tannins, quinones, phenols and nitrogenous bases, which have heteroatoms that are responsible for the inhibitory properties of these substances [3,6].

Due to the great importance of using these environmentally friendly corrosion inhibitors, a variety of investigations have been carried out in recent years. Maestro *et al.* [7] evaluated the effectiveness of garlic peel as a corrosion inhibitor. The study was conducted in different electrolytic solutions (HCl, NaCl and NaOH). For the concentration of 0.44 g L^{-1} of the inhibitor, the authors were able to verify the protective effect of the inhibitor due to a decrease in the corrosion rate in all the media studied. This decrease is due to the barrier effect shown by the inhibitor when adsorbed on the metal surface, which prevents the entry of aggressive species. On the other hand, the extract of *Praecitrullus fistulosus* was tested as a green corrosion inhibitor in the protection of mild steel in 1.0 mol L^{-1} HCl solution. Thermodynamic studies determined that the behaviour of such an inhibitor is of the mixed type, which is related to the inhibition of cathodic and anodic reactions. The maximum efficiency achieved was 87.11 % for an inhibitor concentration of 200 mg L^{-1} [8]. In another study conducted by Li *et al.* [9] the extract of the leaves of *Jasminum nudiflorum* Lindl. was used as a possible inhibitor of cold-rolled steel using HCl of concentration 1.0 mol L^{-1} HCl. The results confirmed the adsorption of the inhibitor on the substrate, forming a single monolayer (Langmuir isotherm) and exhibiting a mixed behaviour where the cathodic reactions of hydrogen formation and metal dissolution are inhibited. In a recent study, ostrich oil was used to evaluate its effectiveness in protecting C38 steel immersed in a 1.0 mol L^{-1} HCl solution. The results showed significant corrosion inhibition by ostrich oil, reaching an efficiency of 94 % due to the chemical adsorption of fatty compounds onto the metal surface [10]. Most of the research reported in the scientific literature supports the effectiveness of using natural extracts as active agents that block oxidation and reduction reactions at the metal-surface interface [5,11-13].

Oreocallis grandiflora is an Ecuadorian species belonging to the Proteaceae family, commonly known as cucharillo (Loja and Zamora provinces), cucharilla (Sierra region), gañal (Bolivar province), and algil (Chimborazo province). This plant is widely used for gastric ulcers and liver (complaints), injuries, and kidneys. According to ethnobotanical information, it may be useful to treat hernias, liver diseases, intestinal tract infections, cholesterol, nephritis, diabetes, gastric ulcer, inflammations, eye problems, and influenza [14,15]. Other important characteristics that could be attributed to this *Oreocallis grandiflora* extract compared to other green inhibitors are its simple extraction and preparation process, rapid biodegradability, low toxicity, renewable and available raw material, antioxidant properties, and its ability to form protective surfaces against corrosive media due to the high concentration of phenolic compounds and tannins. Furthermore, these phenolic compounds and flavonoids not only inhibit corrosion but may also exhibit antimicrobial activity, which is particularly useful in marine environments where biofouling is prevalent [16].

The presence of active agents containing heteroatoms in the composition of *Oreocallis grandiflora* extract makes it a potential candidate to present inhibitory properties against corrosion [17,18]. Our primary interest in evaluating the inhibitory properties of *Oreocallis grandiflora* extract against the corrosion of ASTM A1011 steel using electrochemical techniques stemmed from the fact that no articles in the scientific literature have previously carried out such research.

Therefore, the novelty of this research lies in being the first article to evaluate the performance of *Oreocallis grandiflora* extract as a promising inhibitor in the corrosion mitigation of ASTM A1011 steel in a dynamic system with a recirculation rate of 3.5 wt.% NaCl containing different concentrations of *Oreocallis grandiflora* extract. Different electrochemical techniques, such as open circuit or corrosion potential (OCP) measurements and potentiodynamic polarization curves (PPC), were used.

Experimental

Materials and solutions

The ASTM A1011 steel samples had a chemical composition of 0.02-0.15 % C, 0.60 % Mn, 0.0035 % S max., 0.20 % Cu max., 0.20 % Ni max., 0.15 % Cr max., 0.06 % Mo max., 0.0008 % V mx., 0.0008 % Cb max. and 0.025% Ti max. The corrosive medium was prepared using a 3.5 wt.% sodium chloride solution (NaCl), in which the inhibitor was dissolved at different concentrations to obtain the values for the electrochemical measurements.

Surface treatment of ASTM A1011 steel

SAE 1011 steel samples were cut in cylindrical form and were polished internally using emery papers from 80 to 600 grit. The samples were then cleaned with deionized water and absolute ethanol to remove dust and grease. Finally, all samples were dried with compressed air. The steel sample area was 175.58 cm².

Preparation of *Oreocallis grandiflora* extract

The leaves of the *Oreocallis grandiflora* plant (Figure 1) were collected and dried in shade for 20 days. The leaves were then crushed and sieved on 1 mm grids to obtain a homogeneous particle size. For the extraction process, the maceration method was employed, where the powder obtained was mixed with absolute ethanol in a 3:8 ratio, respectively, and then placed in a water bath at 38 °C for eight hours. Afterwards, the solution was sonicated for 60 min at room temperature. Finally, the excess solvent was evaporated at 40 °C and under a reduced pressure of 1.69×10^4 Pa in a BUCHI R-100 rotary evaporator for a period of 4 h. The resulting *Oreocallis grandiflora* extract was thoroughly washed and then dried in an oven at 38 °C for 24 hours.



Figure 1. Leaves of the *Oreocallis grandiflora* plant

Phytochemical characterization of Oreocallis grandiflora extract

Phytochemical screening is one of the most versatile tools for identifying secondary metabolites found in plants. The importance of this technique lies in its ability to characterize the chemical structure of bioactive compounds, thereby explaining the mechanism of interaction with the metallic substrate. Among the main bioactive compounds found in plants are polyphenolic compounds, tannins, alkaloids, vitamin A, steroids, and nitrogen-based compounds [19]. It is well known that the primary mechanisms explaining the interaction between these bioactive compounds and the metallic substrate are the phenomena of physical and chemical adsorption. In both cases, this phenomenon is due to the presence of heteroatoms in the structure of the molecule, such as O, S, and N, which, according to the scientific literature, confer inhibitory properties to these compounds [20,21].

Preparation of dynamic system with recirculation

For the preparation of the dynamic system with recirculation, a 3.5 wt.% NaCl solution containing increasing concentrations of OGE in a total volume of 8 L of distilled water was used as an electrolytic medium. First, a certain amount of powder inhibitor is weighed and dissolved in 5 mL of absolute ethanol using a thermostatic bath at 40°C. Then, the inhibitor is mixed with the electrolyte solution. For the test with the blank, only the 3.5 wt.% sodium chloride electrolyte solution is used. When the system is in operation, the velocity of the solution is 0.5 L s⁻¹. The cables are then connected to the electrodes and the potentiostat to initiate the electrochemical measurements, as shown in Figure 2.



Figure 2. Dynamic system with recirculation

Electrochemical tests

The use of electrochemical tests, such as open circuit potential (OCP) measurements and Tafel polarization curves, was necessary to determine the effectiveness of the OGE in protecting the substrate against corrosion. The inhibitor concentrations evaluated were 125, 250, 375 and 500 ppm in a neutral medium of 3.5 wt.% sodium chloride (NaCl). A working electrode (ASTM A1011 steel pipe) with an exposed area of 175.58 cm², an AISI 310 stainless steel rod as the counter electrode, and saturated calomel Hg/Hg₂Cl₂, KCl_{sat} as the reference electrode. The open circuit potential (OCP) was reached after one hour of substrate immersion. Once OCP was established, polarization tests were performed using a range of -250 mV to +250 mV and considering 0.5 mV s⁻¹. The equipment used for the electrochemical tests was a potentiostat/galvanostat Gamry, model 1010B. The tests were controlled by the Gamry Framework software.

Morphological characterization

Scanning electron microscopy (SEM) was necessary to observe the surface morphology of the ASTM A101 before and after immersion in the 3.5 wt.% sodium chloride solution. The SEM used was the ThermoFisher Scientific, model Axia ChemiSEM with a beam voltage of 20 kV and coupled with an energy-dispersive X-ray spectrometer (EDS).

Infrared spectroscopy

The molecular structure of OGE was determined using infrared spectroscopy. The spectrum was recorded in the 4000 to 500 cm⁻¹ range using a Bruker Alpha Model II spectrophotometer with a spectral resolution of 2 cm⁻¹ and controlled by the OPUS software.

Results and discussion

Phytochemical analysis

Table 1 summarizes the phytochemical constituents of OGE identified through analytical and quantitative techniques. Based on these results, it is possible to verify the notable presence of tannins and phenolic compounds in the OGE, which promotes strong electrostatic attractions, facilitating effective adhesion of the inhibitor molecules to the substrate surface. Other less representative compounds include flavonoids, alkaloids, leucoanthocyanins, quinones, steroids, and vitamin A. It is essential to note that the organic compounds present in OGE have both a polar hydrophilic component (such as -OH) and a nonpolar hydrophobic component, namely the carbon chain. These components are adsorbed on the substrate surface, forming an obstacle that prevents the migration of corrosive species toward the metal. Other past research has also demonstrated the effectiveness of these plant extracts on different types of substrates [11,13]. One of the main challenges for a better understanding of the corrosion inhibition mechanism of these natural extracts is the process of selectively separating each component through techniques such as column chromatography. Separating each component can provide a better understanding of the contribution of each active component to the corrosion protection of different metallic substrates [21].

Table 1. Phytochemical analysis of *Oreocallis grandiflora* extract

Phytochemical groups	Presence
Leucoanthocyanin	+
Alkaloids	+
Cardenolides, lactone rings	-
Flavonoids	+
Tannins	+++
Quinone	+
Phenolic compounds	+++
Steroids, triterpenes	+
Vitamin A	+

+++ strong presence; + present; - absent

Fourier transform infrared spectroscopy

From the FTIR analysis, it is possible to confirm the presence of characteristic functional groups containing heteroatoms, which are responsible for the interaction between the OGE molecules and the 310 stainless steel. Figure 3 shows the FTIR spectrum of the liquid extract of *Oreocallis grandiflora* where a first broad peak appears at 3355.97 cm⁻¹, which is related to the -OH functional group belonging to the structure of phenolic compounds and tannins [22,23]. Other peaks observed were

around 2917.01 and 2850.4 cm^{-1} related to -CH bonds, and another peak at 1602.75 cm^{-1} attributed to C=C stretching vibrations. The flavonoids and alkaloids found in the phytochemical analysis of the extract may support the presence of double bonds as part of the aromatic ring composition [24]. Another peak of lower intensity was located at 1443.7 cm^{-1} , which may be associated with tensions between C-N bonds. Finally, the C-O group was assigned in the band at 1034.53 cm^{-1} [25].

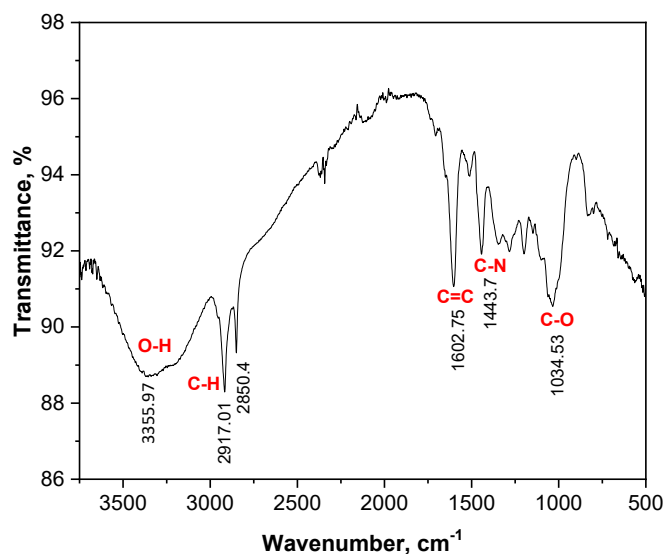


Figure 3. FTIR spectrum of *Oreocallis grandiflora* extract

It is important to mention that the inhibitory properties of these extracts are based mainly on the presence of heteroatoms (N and O), which facilitate the adsorption of the inhibitor molecules on the metal surface, forming a protective barrier against corrosive species [26].

Open circuit potential

The open-circuit potential (OCP) measurements for ASTM A1011 steel were established after one hour of immersion in a 3.5 wt.% NaCl solution for different concentrations of OGE. Figure 4 shows that OCP values are stabilized in all cases studied after 60 min of exposure. In particular, in the blank solution, the OCP values begin to decrease due to chloride attack ions on the surface of the substrate as a consequence of the corrosive medium. Additionally, the OCP values are lower than those of the blank, suggesting that OGE exerts a noteworthy anticorrosive influence on the cathodic reaction and therefore could be classified as a cathodic inhibitor [24,27].

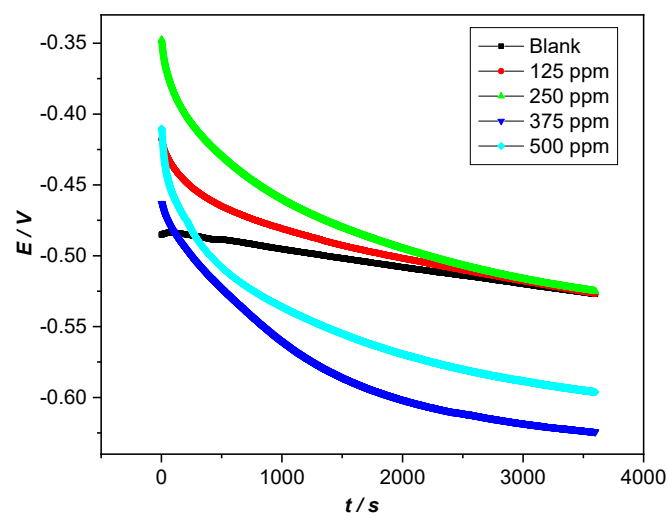


Figure 4. Open circuit (corrosion) potential measurements for ASTM A1011 steel in 3.5 wt.% NaCl solution in the absence and presence of different concentrations of *Oreocallis grandiflora* extract

Potentiodynamic polarization curves

Figure 5 depicts the potentiodynamic polarization curves for ASTM A1011 steel in 3.5 wt.% NaCl solution in the absence and presence of different concentrations of OGE after the OCP experiments and 60 min of immersion. These curves allow us to understand the inhibitor's behaviour and thus classify it as a cathodic, anodic, or mixed inhibitor. The results showed that for all concentrations studied, there was a decrease in corrosion current densities in the presence of OGE. This effect may be related to changes in the reaction process due to the working electrode surface being coated with a protective film that blocks the active sites. This film effectively inhibits the cathodic reaction at the corrosion potential and the same in its vicinity [28]. Similar studies were also found for other green inhibitors in the protection of metallic substrates [29,30].

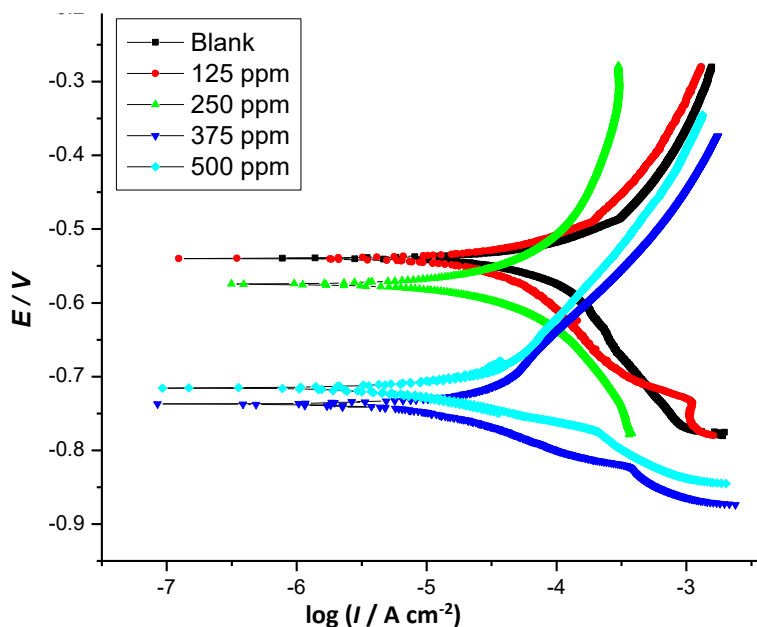


Figure 5. Polarization curves for ASTM A1011 steel in 3.5 wt.% NaCl solution in the absence and presence of different concentrations of *Oreocallis grandiflora* extract

Additionally, the polarization curves also show that the variation of the corrosion potential (ΔE_{corr}) in relation to the blank for concentrations of 125 and 250 ppm is 0 and 35 mV, respectively, so the inhibitor at these concentrations is indicated as a mixed inhibitor with cathodic character, while for concentrations of 375 and 500 ppm the variations were much greater, reaching values of 197 and 176 mV respectively, which indicates a strictly cathodic behaviour of the inhibitor at higher concentrations, that is, the inhibitor molecules are preferentially adsorbed in the cathodic zones and tend to alter the mechanism of the cathodic processes, responsible for the oxygen reduction (ORR) and water hydrogen reduction (HER) [31,32].

Tafel extrapolation plots were used to obtain the basic parameters, such as corrosion potential (E_{corr}) and corrosion current density (I_{corr}), while β_a and β_c represent the anodic and cathodic Tafel slopes, respectively. The inhibition efficiency (IE) was calculated through equation (1). The results are reported in Table 2.

$$IE = [1 - (I_{\text{corr}} / I_{\text{corr}}^0)] 100 \quad (1)$$

where I_{corr} and I_{corr}^0 are the corrosion current densities in the solution with inhibitor and blank solution, respectively. As can be seen in Table 2, the I_{corr} value decreased from 0.149 mA cm⁻² to 0.0165 mA cm⁻² compared to the blank, reaching a maximum inhibitory efficiency of 88.93 % at 500 ppm of OGE.

Table 2. Electrochemical parameters obtained from polarization curves for ASTM 1011 steel in 3.5 wt.% NaCl solution in the absence and presence of different concentrations of *Oreocallis grandiflora* extract

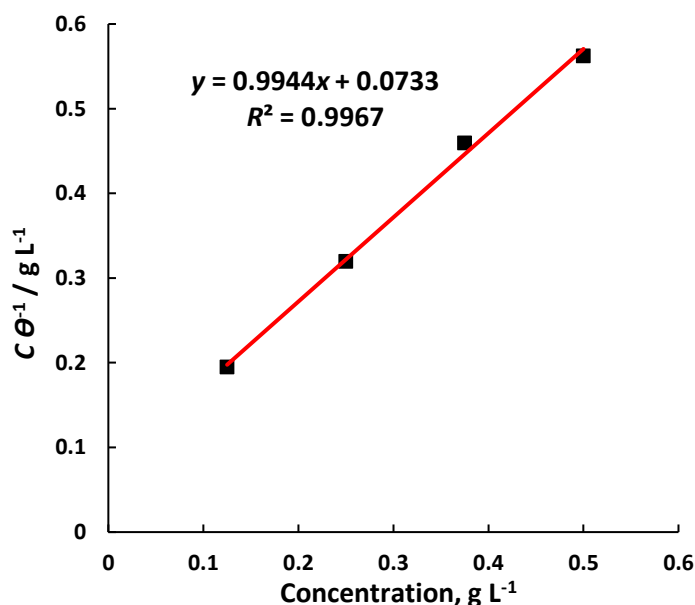
C / ppm	$E_{\text{corr}} / \text{V}$	$I_{\text{corr}} / \text{mA cm}^{-2}$	$\beta_a / \text{mV decade}^{-1}$	$\beta_c / \text{mV decade}^{-1}$	IE, %	θ
Blank	-0.540	0.1490	138.50	364.60	-	-
125	-0.540	0.0534	89.10	218.40	64.16	0.642
250	-0.575	0.0325	108.70	111.90	78.19	0.782
375	-0.737	0.0274	176.90	92.80	81.61	0.816
500	-0.716	0.0165	105.60	72.10	88.93	0.889

Adsorption isotherms

Adsorption isotherms are mostly used to gain a better understanding of the adsorption mechanism of inhibitor molecules on metallic substrates. Of the different models that exist to describe the adsorption process of the inhibitor on the substrate, the Langmuir isotherm has become one of the most popular for this type of process, where a monolayer is formed by the adsorption of inhibitor molecules at a specific site on the substrate, without any interaction or stereo impediment between them [29,33]. To obtain the Langmuir isotherm, the values of (C/θ) versus the inhibitor concentration (C) were adjusted to a straight line with a correlation coefficient (R^2) very close to unity and an intercept equal to $1/k_{\text{ads}}$. The value of k_{ads} represents the adsorption-desorption constant and θ is the fractional surface coverage of the metal.

The Langmuir plot for ASTM A1011 steel in 3.5 wt.% NaCl solution with different concentrations of *Oreocallis grandiflora* extract is presented in Figure 6. It is necessary to highlight that θ values were obtained from the polarization results (Table 2).

From Figure 6, a strong linear correlation ($R^2 = 0.9967$) is observed between the two fitted variables (C/θ and C), indicating the formation of an adsorbed monolayer on the metal surface. This monolayer, which acts as a barrier, is composed primarily of inhibitor molecules, which independently occupy active sites on the substrate surface [22,34]. Based on previous research, the Langmuir isotherm intercept is $1/k_{\text{ads}}$, and the k_{ads} value is thus calculated ($k_{\text{ads}} = 13.64 \text{ L g}^{-1}$). Higher values of k_{ads} confirm the high affinity between the adsorbent and the adsorbate, which provides excellent corrosion protection properties for the inhibitor under study [9,35].

**Figure 6.** Langmuir isotherm for ASTM A1011 steel in 3.5 wt.% NaCl solution containing different concentrations of *Oreocallis grandiflora* extract

Through the adsorption and desorption constant k_{ads} it is possible to calculate another important parameter, such as the adsorption energy ($\Delta G^{\circ}_{\text{ads}}$). The adsorption energy can be calculated using equation (2) [25,36]:

$$\Delta G^{\circ}_{\text{ads}} = -RT \ln(C_{\text{H}_2\text{O}} k_{\text{ads}}) \quad (2)$$

where $C_{\text{H}_2\text{O}} = 55 \text{ mol L}^{-1} = 1000 \text{ g L}^{-1}$, T is the temperature expressed in Kelvin (298.15 K) and R is the gas constant. Substituting these values (R , T , $C_{\text{H}_2\text{O}}$ and k_{ads}) in equation (2), the adsorption energy was about $-23.59 \text{ kJ mol}^{-1}$. This negative value of the adsorption energy indicates that the adsorption process is spontaneous and that the monolayer formed on the metal surface is stable over time [3].

Another detail is that this calculated value is very close to -20 kJ mol^{-1} , which, according to the literature, is characteristic of a physical adsorption process associated with weak electrostatic interactions between the charged molecules of the inhibitor and the charged metal surface [10]. However, when adsorption energy values are close to -40 kJ mol^{-1} , these processes can be considered a type of chemical adsorption, which involves interactions between heteroatoms and vacant d orbitals of iron [37]. In conclusion, for this study, it was possible to verify that the molecules of the OGE were physically adsorbed on the steel substrate due to the polar functional groups and the non-polar part of the carbon chains, which form a physical barrier that prevents the diffusion of corrosive ions towards the substrate [38].

Morphological characterization

Figure 7 presents the SEM images of the ASTM A1011 steel before immersion, after immersion in 3.5 wt.% NaCl solution without inhibitor and with the concentration that provided the best efficiency in the previous tests (500 ppm). In Figure 7a, a surface free of corrosion products is visible, albeit with only minor scratches resulting from the grinding procedure, which is part of the sample preparation process.

In relation to Figure 7b, a much more attacked surface is observed with the presence of large craters and cracks as a result of the dissolution of iron atoms in the corrosive solution due to the anodic reaction. However, in the presence of an OGE inhibitor (Figure 7c), with an exposure time of 60 min, the surface became more homogeneous, with a lower degree of attack and a minor presence of pores. This demonstrates that the inhibitory molecules are bound to the substrate surface, forming a physical barrier that prevents the diffusion of chloride ions into the substrate.

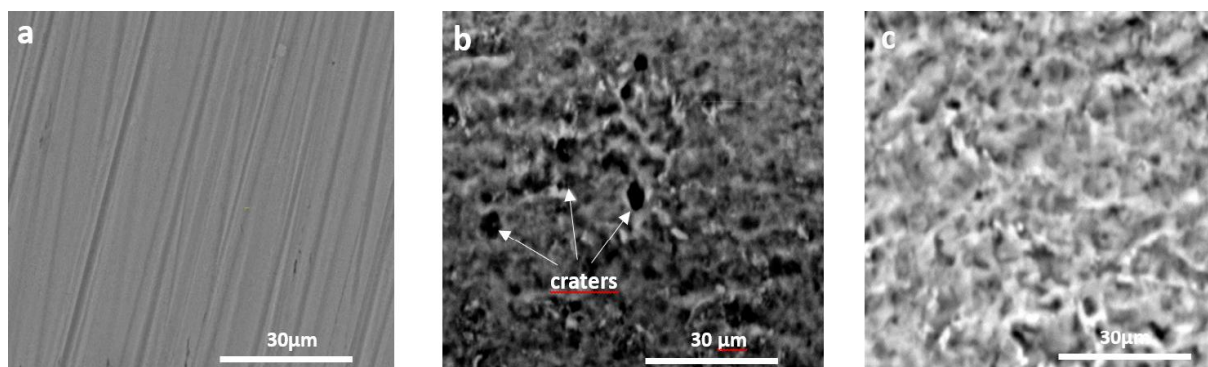


Figure 7. SEM images for ASTM A1011 steel surface: (a) before immersion, and after immersion for 60 min in 3.5 wt.% NaCl (b) without OGE inhibitor, (c) and in the presence of OGE inhibitor

To contextualize the present study, a comparative analysis of recent research using green corrosion inhibitors in saline media is described in Table 3. Table 3 summarizes the key characteristics of some relevant studies, considering the type of metal studied, the concentration and type of inhibitor, the efficiency achieved, and the main findings found in each study.

Table 3. List of green corrosion inhibitors studies in 0.5 M NaCl solution

Green corrosion inhibitor	Concentration	Type of metal	Maximum IE, %	Findings	Ref.
<i>Capsicum pedicles</i>	100 μL	Mild steel	-	Greater corrosion resistance	[39]
Gourd seed extract	40 mg mL ⁻¹	Mild steel	94	Formation of a barrier effect that prevents corrosive molecules to reach the substrate	[40]
Waste pineapple crown	-	Mild steel	80	The inhibitor covers most of the sites on the substrate surface	[41]
Waste lady finger caps	80 mg mL ⁻¹	Copper	89	Adsorption of inhibitor extract onto copper surface blocking its active sites	[42]
Sponge gourd peels	200 mg L ⁻¹	Mild steel	75	Adsorption of inhibitor molecules on the substrate surface	[43]
Watermelon seed	100 μL	Mild steel	68		[44]

Conclusions

- In view of the results obtained under dynamic conditions, it can be concluded that the *Oreocallis grandiflora* extract is an eco-friendly and potential corrosion inhibitor for ASTM A1011 steel in 3.5 wt.% NaCl solution.
- The results obtained through phytochemical screening confirmed a greater presence of tannins and phenolic compounds responsible for the inhibitory effect of the *Oreocallis grandiflora* extract.
- Through polarization curve tests, it was possible to confirm that the inhibition efficiency increased (up to a maximum value of 88.93 %) when a greater amount of *Oreocallis grandiflora* extract was added to the 3.5 wt.% NaCl solution.
- The adsorption of the *Oreocallis grandiflora* extract molecules on the metal surface was found to obey the Langmuir adsorption isotherm.
- SEM images confirmed less attack on the metal surface by the aggressive solution due to the adsorption of the *Oreocallis grandiflora* extract components, which created a protective barrier at the electrolyte/ASTM A1011 steel interface.

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