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Original scientific paper

Bidens pilosa extract as a corrosion inhibitor on 1008 carbon steel in neutral medium

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Abstract

The aim of this work is to evaluate the performance of Bidens pilosa *extract as a corrosion inhibitor for 1008 carbon steel in a neutral medium of 0.1 M NaCl. The research has been accomplished by weight loss measurements, linear polarization resistance (R_p) monitoring and electrochemical impedance spectroscopy (EIS). Phytochemical analysis and Fourier transform infrared spectroscopy were performed to determine bioactive components and detect the main functional groups of* Bidens pilosa *extract, respectively. The morphological characterization of the substrate was carried out by optical microscopy (OM). Different isotherms were evaluated to understand more clearly the adsorption mechanism of the inhibitor extract molecules on the surface of the 1008 carbon steel substrate, and the best fit was obtained for the Langmuir isotherm. The results showed that the corrosion rate decreased with an increase of the concentration of the inhibitor up to 1000 ppm, reaching a maximum efficiency value of 82.9 % from gravimetric tests, and 73.1 % from the fitting of EIS data to an equivalent electric circuit. The calculated thermodynamic parameters suggested the formation of a monolayer of inhibitor molecules on the metal surface. The ΔG^o_{ads} value (-22.8 kJ mol^{-1}) determined from the Langmuir isotherm model indicated that adsorption of the inhibitor molecules on the substrate surface follows a physisorption mechanism. This research revealed that* Bidens pilosa *extract can be used as a corrosion inhibitor and emerges as an alternative to replace synthetic corrosion inhibitors that are harmful to health and cause damage to the environment.*

Keywords

Green corrosion inhibitor, corrosion resistance; phytochemical screening; electrochemical impedance spectroscopy; adsorption isotherms

Introduction

Carbon steel is an alloy formed by Fe-C, which is widely used in the industrial field due to its low cost and a variety of useful properties suitable for numerous engineering applications [1]. It is also known that Fe-C material is very susceptible to corrosion, particularly if not alloyed with some additional elements, which implies a need to use coatings or corrosion inhibitors.

Corrosion inhibitors are chemical substances that, added in small quantities, can significantly reduce corrosion rate of a metal [1,2]. One of the main mechanisms of their action is the adsorption of inhibitor molecules on the metal surface, reducing the electrically active areas and hindering the progress of oxidation-reduction reactions [3,4]. This adsorption depends mainly on the presence of functional groups and heteroatoms such as N, O, P and S that contain lone pairs of electrons, which favour the interaction of inhibitor with the metal surface [5]. However, most of these synthetic inhibitors are expensive and highly toxic, causing harm to both humans and the environment [2,6]. Due to this drawback, researchers have been concerned with finding eco-friendly inhibitors with low or no toxicity to the environment, such as inhibitors from natural products. This class of inhibitors, called “green inhibitors” contains a variety of organic compounds, such as pigments, alkaloids, tannins, polyphenols and amino acids, where most of these substances have an effective inhibitory action. In recent years, it has been possible to find in the literature a series of investigations where the efficiency of extracts from medicinal plants in the protection of metal substrates has been verified, in most cases using acid solutions as aggressive media. However, there are still extracts that have not been studied in relation to their anticorrosive properties, which represents an opportunity to continue exploring novel inhibitors with low toxicity and low cost. For example, the extract of *Ceratonia Siliqua* L. seeds was tested as corrosion inhibitor of carbon steel in 1 M HCl medium, reaching an efficiency of around 95 % [7]. On the other hand, *Cucumis Sativus* L., with a concentration of 0.3 g L^{-1}, presented an inhibition efficiency of 92.8 % in the protection of carbon steel when immersed in 0.5 mol L^{-1} H_2SO_4 solution [8]. Another study was carried out on the extract from almond flowers (*Prunus dulcis*), where it was possible to verify the reduction in the corrosion rate of carbon steel when it was immersed in 1 M HCl solution, reaching inhibition efficiency up to 96 % [9]. A variety of recent research on the application of natural extracts as corrosion inhibitors in the protection of carbon steel and other metallic substrates can be found in the literature [10-13].

Bidens pilosa is a plant that belongs to the Asteraceae family and is native to the Americas and some parts of Africa. This plant is used in traditional medicine for the treatment of at least 40 diseases, among them are diabetes, pharyngitis, laryngitis, influence and hypertension [14,15]. The presence of bioactive components, such as tannins and flavonoids, classifies *Bidens pilosa* as a plant with a high potential to present considerable inhibitory properties against corrosion [14,16]. It is also important to emphasize that there is little information in the literature on the use of natural inhibitors in neutral media since most of the studies were performed in acidic media. Therefore, the novelty of this work lies in verifying the effectiveness of the *Bidens pilosa* extract as an inhibitor in the protection against corrosion of carbon steel in a neutral medium of 0.1 M NaCl.

The present study is focused on evaluating the potential performance of the extract from *Bidens pilosa* as a corrosion inhibitor of SAE 1008 carbon steel in the neutral medium of 0.1 M NaCl by gravimetric tests and electrochemical techniques such as linear polarization resistance and electrochemical impedance spectroscopy. Different adsorption isotherms were also analyzed to better understand the adsorption mechanism of extract molecules on the metal substrate.

Experimental

Materials and solutions

In this study, SAE 1008 carbon steel plates were used with the following composition, 0.04 % C, 0.01 % Si, 0.18 % Mn, 0.018 % P, 0.007 % S, 0.029 % Al, 0.0008 % B and 99.72 % Fe). The corrosion inhibitor used in this study was the ethanolic extract of *Bidens Pilosa*. A solution of 0.1 mol L-1 sodium chloride (NaCl) (pH 7) was used as a corrosion medium for gravimetric and electrochemical tests.

Preparation of carbon steel surface

Carbon steel samples with quadrangular geometry and cut to the dimensions of 3×3×0.2 cm and treated with silicon carbide (SiC) emery papers with a grit size of 80, 220, 400 and 600 # for the removal of corrosion products. This procedure was followed by washing it with distilled water, alcohol, and acetone, and finally, it was dried in hot air.

Preparation of Bidens pilosa *extract*

The fresh leaves of the *Bidens pilosa* plant obtained from the Chilca district - Peru (Figure 1), were cut and separated from the stems, flowers and roots to later be dried at room temperature. The previously dried leaves passed through a grinding process to be transformed into powder, which was placed in contact with 96 % ethyl alcohol solvent for 3 days without agitation. The extract was then separated, and the process was repeated 4 times using the same leaves with the purpose of increasing the solid-liquid extraction efficiency. In order to concentrate the extract, an evaporation process was carried out at 45 °C and at reduced pressure (1.38×10^4 Pa) in an equipment called BUCHI R-100 rotary evaporator for a period of 4 h. The resulting extract was finally used to prepare five different concentrations in the range of 350 ppm to 1750 ppm in a neutral 0.1 mol L^{-1} NaCl solution.

Figure 1. *Leaves of the* Bidens pilosa *plant*

Phytochemical screening

A phytochemical is a term that refers to a variety of compounds produced naturally in plants. They are classified into six main types based on their chemical structure and properties. These include carbohydrates, lipids, phenols, terpenoids, alkaloids and other nitrogen-containing compounds. Phytochemicals are mainly classified into primary and secondary metabolites. Phytochemical analysis is a qualitative analytical study that, by certain standardized procedures, can be used to determine the main constituents of a plant extract [17]. Thin layer chromatography (TLC) is a technique generally used to analyse the number and types of components present in a mixture. In TLC, the extracts are loaded in a glass coated with silica gel or other adsorbent, which is then kept in a chromatographic chamber containing a suitable running solvent. This technique mainly consists of a mobile phase and a stationary phase, separating the compounds based on their polarity [18].

Weight loss measurements

For the gravimetric tests, previously sanded and cleaned SAE 1008 carbon steel plates were used, which were cut to the dimensions of 40×10×2 mm and weighed on an analytical balance (Sartorius, Model TE214S) with 0.1 mg precision. Next, the plates were immersed in the 0.1 M NaCl solution in the presence and absence of *Bidens pilosa* extract. Five concentrations of *Bidens pilosa* extract were prepared (350, 700, 1000, 1400 and 1750 ppm) and the immersion time of the samples was 15 days. After this time, they were removed from the immersion, cleaned with a soft brush and washed with distilled water, alcohol, acetone, and then dried and weighed. The tests were carried out in an aerated solution, not stirred, and in duplicate.

Electrochemical tests

In order to evaluate the inhibition efficiency of the *Bidens pilosa* extract, electrochemical techniques such as measurements of open circuit or corrosion potential (E_{oc}), polarization resistance (R_p) and electrochemical impedance spectroscopy (EIS) were used. The inhibitor concentrations evaluated were 350, 700, 1000, 1400 and 1750 ppm in the neutral medium of 0.1 M NaCl. The tests were carried out in a three-electrode electrochemical cell, where $Ag|AgCl|KCl_{sat}$ was used as the reference electrode, a graphite electrode as the counter electrode and a working electrode (SAE 1008 carbon steel) with exposed area of 1.085 cm^2. Electrochemical impedance measurements were performed after 60 min of immersion in the 0.1 M NaCl solution to achieve a steady-state potential. A sinusoidal potential perturbation with an amplitude of 10 mV (root mean square) was used in relation to the open circuit potential and a frequency range of 10 kHz to 10 mHz with 10 measurements for each frequency decade. Linear polarization resistance measurements were obtained at room temperature and after EIS measurements, with a scan rate value of 0.167 mV s^{-1} and scan potential range of -0.01 V < η < +0.01 V, relative to the open circuit potential. All tests were carried out on a Gamry Interface 1010 B potentiostat/galvanostat controlled by Gamry Framework software.

Morphological characterization

The morphological characterization of the surface of the metal substrate in the absence and presence of the inhibitor was carried out by an optical microscope ZEISS Axio Lab. A1, with the help of the ZEN software.

Infrared spectroscopy

The infrared spectroscopy analyzes were carried out on a Bruker Alpha II model, operating in ATR mode and with the help of the OPUS software. The spectrum was obtained at room temperature in the range of 4000 to 400 cm^{-1}.

Results and discussion

Phytochemical analysis

The results of the phytochemical constituents of the ethanolic extract of *Bidens pilosa* are shown in Table 1. Based on the results obtained, it can be observed that the *Bidens pilosa* extract mainly contains compounds such as alkaloids, tannins, reducing sugars, phenols, and in smaller amounts, anthocyanins, lactones, flavonoids and cardenolides. On the other hand, the presence of saponins and amino acids could not be detected. However, the finding of these main bioactive organic compounds reveals the inhibitory characteristics of the *Bidens pilosa* extract due to the presence of potential functional groups and long chains of high molecular weight, which are absorbed on the

surface of the substrate. Thus, the adsorbed film forms a protective barrier against the aggressive environment. Similar results were also found in the literature for the protection of aluminum in an acid medium using *Bidens pilosa* extract as corrosion inhibitor [16].

One of the main drawbacks in the use of plant extracts as corrosion inhibitors is the difficulty of identifying the main active component responsible for the inhibitory action due to the complex matrix of the plant extract. However, the synergism of several of these bioactive components contained in the extract was reached, effectively inhibiting the corrosion of the substrate [19].

Table 1. *Phytochemical analysis of the ethanolic extract of* Bidens pilosa

Phytochemical groups	Result
Anthocyanins	++
Alkaloids	+++
Lactones	++
Flavonoids	++
Cardenolides	++
Tannins	+++
Reducing sugars	+++
Phenols	+++
Saponins	–
Aminoacids	–

+++: very obvious reaction; ++: obvious reaction; –: no reaction

Fourier transform infrared spectroscopy

Fourier transform infrared spectroscopy (FTIR) is a powerful analytical tool that allows determination of the main chemical functional groups contained in a natural or synthetic sample. Figure 2 represents the FTIR spectrum of the liquid extract of *Bidens pilosa* where the most representative absorption peaks were identified.

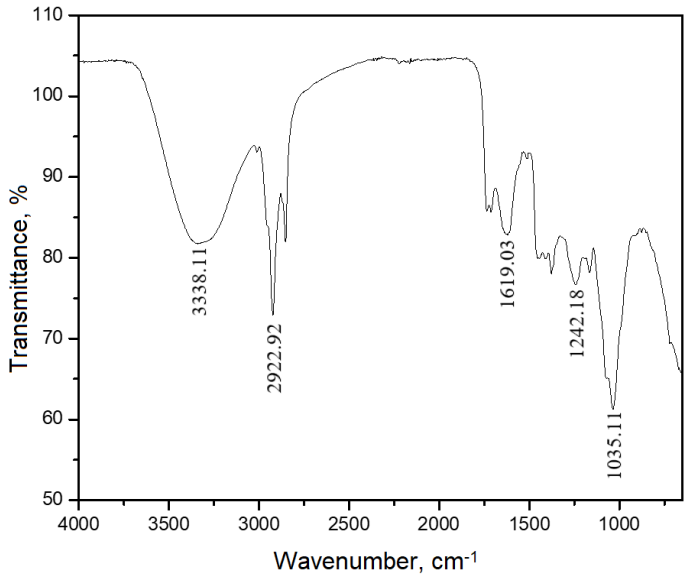

Figure 2. *FTIR spectrum of Bidens pilosa extract*

The first broad and strongly pronounced peak around 3338.11 cm^{-1} can be attributed to a tension vibration between the O-H group bonds and could correspond to the presence of simple phenolic compounds and tannins [1,13,20-22], which is confirmed by phytochemical analysis (Table 1). The peaks located around 2922.92 and 1619.03 cm^{-1}, can be attributed to tension vibrations of the -CH and C=C groups, respectively, due to the presence of aromatic rings as part of the flavonoid structure

or alkaloids [20,23,24]. The peak at 1242.18 cm^{-1} is related to the vibration of the C-N group and finally, the peak located at 1035.11 cm^{-1} can be attributed to the C-O group [25].

According to the results of the analysis by FTIR, it was possible to detect the presence of heteroatoms such as C, N and O, which belong to different functional groups found in the structure of the *Bidens pilosa* extract and which are responsible for the corrosion inhibition process on metal substrates. In the literature, it is possible to find several research works on natural inhibitors where the presence of these heteroatoms is reported [4,26-29].

Weight loss measurements

The results of the gravimetric tests were obtained by immersing 1008 carbon steel samples for a period of 15 days in 0.1 M NaCl solution in the absence and presence of different concentrations of *Bidens pilosa* extract. Important parameters such as corrosion rate (C_R), inhibition efficiency (η) and degree of surface coverage (θ) were obtained from this gravimetric study and are presented in Table 2. The corrosion rate expressed in mm $year^{-1}$ (1 mm $year^{-1}$ = 39.37 mpy (mils per year)) was calculated using Equation (1), where ΔW / g is the change in mass, A /cm^2 is the total area exposed to the solution, D is density of the metal equal to 7.86 g cm^{-3}, t / h is immersion time in the solution and K is a conversion factor equal to 8.76×10^4.

$$C_R = K\left(\frac{\Delta W}{ADt}\right) \quad (1)$$

The inhibition efficiency was calculated using Equation (2) where C_{Ro} is corrosion rate in the absence of an inhibitor and C_{Ri} is corrosion rate in the presence of an inhibitor. The degree of surface coverage θ was calculated based on the inhibition efficiency values using Equation (3).

$$\eta = \frac{\left(C_{Ro} - C_{Ri}\right)}{C_{Ro}} 100 \quad (2)$$

$$\theta = \frac{\eta}{100} \quad (3)$$

Table 2. *Corrosion rate, inhibition efficiency and degree of coverage calculated from gravimetric tests for SAE 1008 carbon steel during immersion time of 15 days in 0.1 M NaCl in the absence and presence of different concentrations of* Bidens pilosa *extract*

Concentration of inhibitor (ppm)	C_R / mpy	C_R / mm $year^{-1}$	η / %	θ
0.0 (blank)	3.1233 ± 0.012	0.0793 ± 0.0003	-	-
350.0	1.321 + 0.011	0.0336 ± 0.0002	57.7	0.577
700.0	0.584 ± 0.008	0.0148 ± 0.0002	81.3	0.8129
1000.0	0.531 ± 0.013	0.0135 ± 0.0003	83.0	0.8299
1400.0	0.604 ± 0.014	0.0153 ± 0.0004	80.7	0.8067
1750.0	0.670 ± 0.001	0.0170 ± 0.00003	78.5	0.7854

The results presented in Table 2 show an increase in the inhibition efficiency up to the maximum value of 83.0 %, obtained for the inhibitor concentration of 1000 ppm. As seen in the FITR analysis, this can be attributed to the presence of *Bidens pilosa* extract molecules on the surface of the substrate, generating a protective barrier layer that inhibits the corrosion process. Similar results were also reported in the literature for the protection of carbon steel using natural corrosion inhibitors [24,27].

Furthermore, the degree of coverage also increases with the *Bidens pilosa* extract concentration, which indicates a possible increase in the adsorption effectiveness of the *Bidens pilosa* extract

molecules on the substrate surface. However, for the last two concentrations (1400 ppm and 1750 ppm) a slight increase in the corrosion rate is observed (Figure 3).

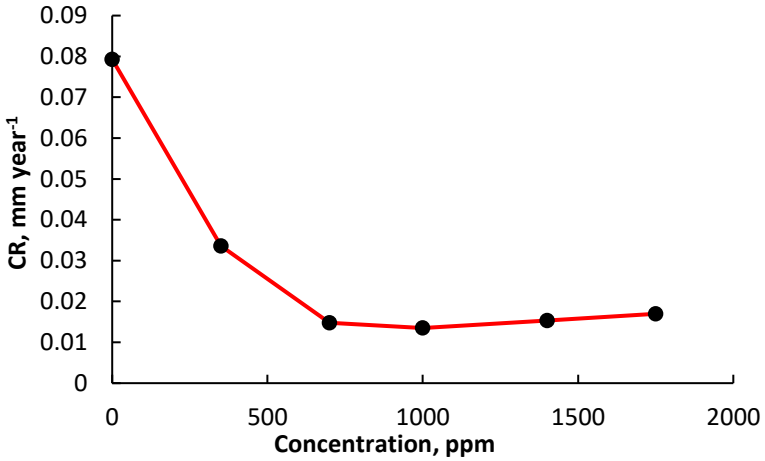

Figure 3. *Corrosion rate of 1008 carbon steel immersed in a neutral solution of 0.1 M NaCl from gravimetric tests at different concentrations of* Bidens pilosa *extract*

A possible explanation for this fact is related to the repulsive forces between the inhibitor molecules which causes a desorption of the inhibitor. Therefore, the unprotected sites are formed, that are prone to corrosion and generate an increase in corrosion rate values [27,30]. Other investigations argue that when high amounts of inhibitor are added, the adsorption on the surface of the substrate is perpendicular due to the repulsion between molecules and this generates unprotected sites since the parallel adsorption of the inhibitor molecules normally covers a greater area of the substrate [27]. For higher concentrations of inhibitor molecules beyond the optimal one, some of the adsorbed and self-assembled inhibiting molecules undergo a kind of disorder provoked by repulsive interactions among them, diminishing the inhibitor efficiency.

Open circuit potential

Open circuit potential (OCP) measurements for carbon steel 1008 were carried out during 3600 s of immersion time in 0.1 M NaCl solution in the absence and presence of different concentrations of *Bidens pilosa* extract. In Figure 4 it is possible to observe the same trend of decreasing potential for all inhibitor concentrations in reaching steady state values at longer immersion time.

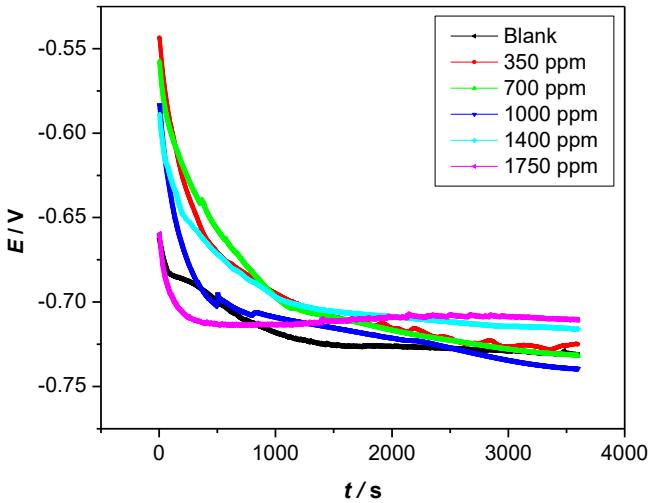

Figure 4. *Time changes of open circuit potential for 1008 carbon steel in 0.1 M NaCl solution in the absence and presence of different concentrations of* Bidens pilosa *extract*

For the concentrations of 350, 700 and 1000 ppm the steady state was reached in approximately 3300 s, while for other concentrations of 1400 and 1750 ppm the stabilization time was shorter, reaching a steady-state value within approximately 1500 s. The addition of the inhibitor to the corrosive medium changes the behaviour of the OCP, leading to more positive steady-state potentials in the case of 1400 and 1750 ppm, and to the most negative potential for the concentration of 1000 ppm. It is known that when the differences in the corrosion potential values in relation to the blank are at least 85 mV, the inhibitor can be classified as a cathodic or anodic type inhibitor for negative or positive differences, respectively. A mixed type of inhibitor is assumed for slight differences, less than 85 mV [28,30,31], as this would be the case in present experiments.

Electrochemical impedance spectroscopy

Figure 5 represents the Nyquist (a) and Bode ((b) and (c)) plots for 1008 carbon steel in 0.1 M NaCl solution in the absence and presence of different concentrations of *Bidens pilosa* inhibitor extract. In all Nyquist diagrams, the presence of a single capacitive arc was observed, indicating that the corrosion kinetics is controlled by the charge transfer resistance and the corrosion mechanism is not affected by the presence of the inhibitor [9,25,32,33]. It is also possible to observe an increase in the diameters of capacitive arcs up to a concentration value of 1000 ppm, which indicates an increase in the charge transfer resistance due to the formation of adsorbed film on the metal surface with protective properties against corrosion [1,8,30,34].

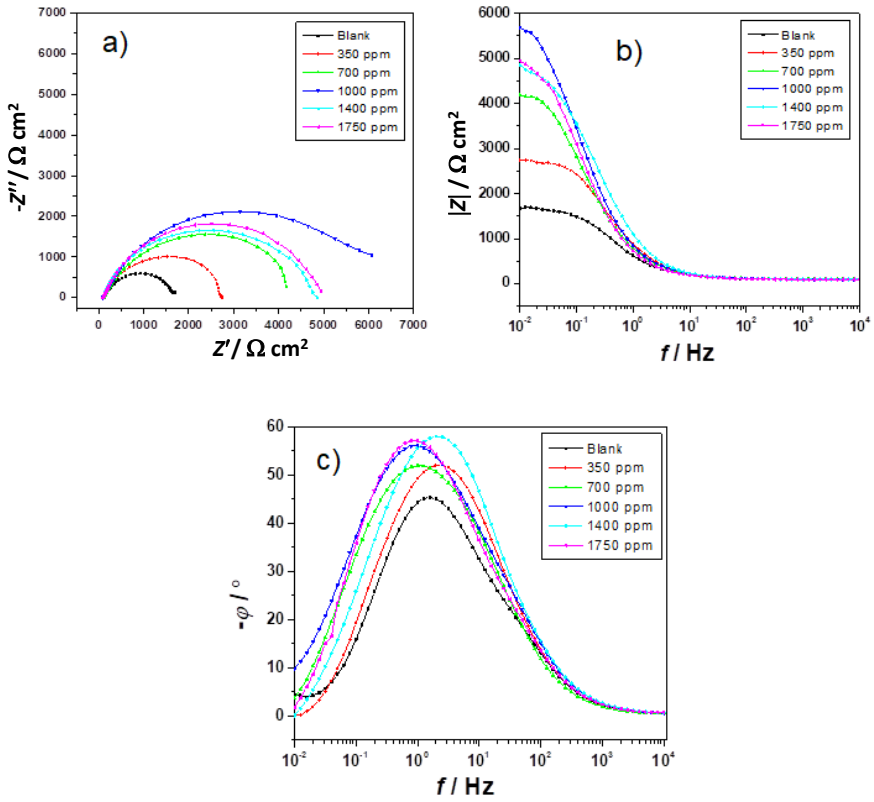

Figure 5. *Nyquist (a) and Bode (b) and (c) diagrams for 1008 carbon steel in 0.1 M NaCl solution in the absence and presence of different concentrations of* Bidens pilosa *extract*

In relation to the Bode modulus diagrams (Figure 5b), it is possible to observe that at low frequencies, there is an increase in the value of the impedance modulus as the concentration of the inhibitor increases up to 1000 ppm and then there is a decay of impedance modulus values as the concentration of the inhibitor is further increased. It is important to mention that the Bode phase

diagrams in Figure 5c, show the presence of a single time constant for all the conditions studied, which is related to the formation of an electrical double layer at the metal-solution interface [13,25].

The values obtained in the electrochemical impedance measurements were fitted to an equivalent electric circuit with the objective of obtaining the electrochemical parameters and, thus, quantitatively comparing the effects of the different concentrations of the *Bidens pilosa* extract in the protection of 1008 carbon steel. Figure 6 represents the classic Randles equivalent circuit, which was used to simulate the metal/electrolyte system, where R_s corresponds to the resistance of the electrolyte, R_{ct} is the resistance to charge transfer in parallel with the capacitance of the electrical double layer represented by a constant phase element (CPE_{dl}). The consideration of a CPE is due to the inhomogeneities found in the steel surface. This equivalent circuit has already been found in the literature to fit properly EIS data for studies of different corrosion inhibitors [7,13,35,36].

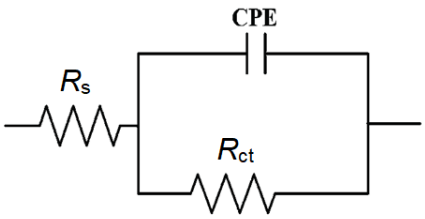

Figure 6. *Equivalent electrical circuit used to fit EIS data of carbon steel in 0.1 M NaCl solution in the absence and presence of different concentrations of* Bidens pilosa *extract*

The CPE_{dl} element was introduced to replace the pure capacitor representing the electrical double layer. The impedance of CPE_{dl} is expressed mathematically according to Equation (4) [1,30,32,33,37].

$$Z_{CPE} = Y_0 (j\omega)^{-\alpha} \quad (4)$$

In Equation (4) Y_0 is the constant parameter of CPE_{dl}, ω the angular frequency, j^2 is equal to -1 and α is the dispersion factor, which values are between 0 and 1. Previous studies have reported that the value of α can inform on different physical defects on the metal surface, such as surface inhomogeneity, impurities, inhibitor adsorption, formation of layers with porosity, *etc.* [26,30,37,38].

The impedance parameter values obtained from the adjustment of the EIS data to the proposed equivalent circuit are presented in Table 3. The χ^2 values were also calculated to provide support for the adjustment made. The χ^2 values were mostly lower than 10^{-3}, indicating a good fit for the proposed equivalent circuit [25,32].

Table 3 shows that the values of α range between 0.749 and 0.808, which leads to using a CPE_{dl} and not a pure capacitor. In relation to the R_{ct} values, it is possible to observe an increase as the inhibitor is added to the solution, where the maximum R_{ct} value was obtained for a concentration of 1000 ppm of inhibitor. This behaviour is related to an increase in the degree of coverage of the substrate due to the inhibitor molecules adsorption, indicating a decrease in the area exposed to the corrosive medium due to the displacement of water molecules and/or chloride ions by the inhibitor molecules [21,28,39]. The inhibition efficiency (IE) values shown in Table 3 were calculated using Equation (5), where $R_{ct,0}$ is the charge transfer resistance in the absence of an inhibitor and R_{ct} represents the charge transfer resistance in the presence of the inhibitor.

$$IE = \frac{R_{ct} - R_{ct,o}}{R_{ct}} 100 \quad (5)$$

Table 3. *Corrosion parameters obtained from fits of EIS data (Fig. 5) to Randles equivalent circuit model (Fig. 6) for 1008 carbon steel immersed in 0.1 M NaCl solution in the absence and presence of different concentrations of* Bidens pilosa *extract*

C (ppm)	R_s / Ω cm^2	R_{ct} / Ω cm^2	CPE_{dl}, μF cm^{-2} s$^{(\alpha-1)}$	α	χ^2	IE, %	θ
Blank	103.00	1587	381.6×10^{-6}	0.766	7.399×10^{-4}	-	-
350	93.84	2636	251.0×10^{-6}	0.794	4.248×10^{-4}	39.8	0.398
700	99.60	4303	330.8×10^{-6}	0.749	7.035×10^{-4}	63.1	0.631
1000	112.10	5894	301.0×10^{-6}	0.774	1.365×10^{-4}	73.1	0.731
1400	92.30	4443	207.2×10^{-6}	0.806	4.148×10^{-4}	64.2	0.642
1750	105.80	4632	323.8×10^{-6}	0.808	1.908×10^{-3}	65.7	0.657

Based on the inhibition efficiency values determined by Equation (5), it is possible to observe that the efficiencies were in the range of 39.8 to 73.1 %, where the best result (73.1 %) was achieved for the concentration of 1000 ppm of inhibitor. It is interesting to mention that for the conditions studied (neutral NaCl medium), an efficiency value greater than 70 % was determined, which, according to what is reported in the literature, is the minimum value that all types of corrosion inhibitors must show to be considered as effective [1,25,32,40]. It is important to stress that EIS results are in close agreement with weight loss measurements.

Linear polarization resistance measurements

Linear polarization resistance (R_p) measurements were obtained after 80 min of immersion of the 1008 carbon steel substrate in 0.1 M NaCl solution in the absence and presence of different concentrations of the *Bidens pilosa* extract. The corrosion rate expressed in mm year^{-1} was calculated using Equation (6), where i_{corr} / A is the corrosion current, Eq_{metal} /g is the equivalent weight of the metal, A / cm^2 is the total area exposed to the solution, D is the density of the metal equal to 7.86 g cm^{-3} and K' is conversion factor equal to 3.27×10^3.

$$C_R = K'\left(\frac{i_{corr}Eq_{metal}}{AD}\right) \tag{6}$$

The value of the corrosion current (i_{corr}) is obtained by the Stern-Geary Equation (7):

$$i_{corr} = \frac{b_a|b_c|}{2.3\left(b_a+|b_c|\right)}\frac{1}{R_p} \tag{7}$$

where the values of b_a and b_c are Tafel slopes, whose values are 0.12 V decade^{-1}.

Figure 7 shows the experimental measurements of R_p, adjusted to straight lines with the purpose of obtaining slopes that represent the values of R_p. It is seen that for the solution containing 1000 ppm of *Bidens pilosa* extract, a higher polarization resistance (R_p = 7070.96 Ω) compared to the other concentrations is obtained, which is due to the barrier effect caused by the adsorption of inhibitor molecules on the surface of carbon steel.

In relation to Table 4, which summarizes measured polarization resistance values, it is possible to observe an increase in the value of the polarization resistance and, therefore, a decrease in the corrosion rate as the concentration of *Bidens pilosa* extract in the 0.1 M NaCl solution increases up to 1000 ppm concentration. For higher concentration values (1400 and 1750 ppm), the R_p value began to decrease due to the inhibitor's low corrosion protection efficiency.

This fact can be attributed to the change in orientation that some adsorbed *Bidens pilosa* extract molecules may have from the flat or parallel to a vertical orientation (>1000 ppm). This reorient-tation causes some parts of the substrate to be exposed to the aggressive solution and causes an increase in the corrosion rate and, consequently, a decrease in the polarization resistance [24].

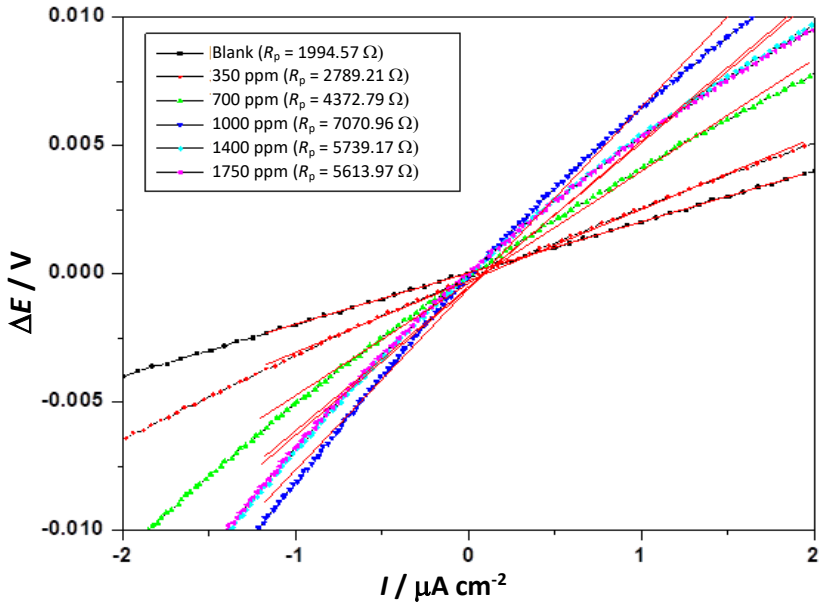

Figure 7. *Linear polarization resistance measurements (*R_p*) for carbon steel in 0.1 M NaCl solution in the absence and presence of different concentrations of* Bidens pilosa *extract*

It is important to highlight that the results obtained from R_p follow a similar trend to those obtained in the mass loss and electrochemical impedance spectroscopy tests, confirming the inhibitory properties of *Bidens pilosa* extract on the 1008 carbon steel substrate.

Table 4. *Polarization resistance and corrosion rate values of 1008 carbon steel obtained from* R_p *measurements after 80 min immersion in 0.1 M NaCl solution in the absence and presence of different concentrations of* Bidens pilosa *extract*

Concentration of inhibitor, ppm	R_p / Ω	C_R / mpy	C_R / mm year^{-1}	IE, %
0.0 (blank)	1994.57 ± 117.33	5.51 ± 0.38	0.14 ± 0.01	-
350.0	2789.21 ± 610.14	3.94 ± 0.89	0.10 ± 0.02	28.5
700.0	4372.79 ± 406.77	2.51 ± 0.26	0.06 ± 0.01	54.4
1000.0	7070.96 ± 1584.87	1.55 ± 0.35	0.04 ± 0.01	71.8
1400.0	5739.17 ± 1291.31	1.91 ± 0.42	0.05 ± 0.01	65.2
1750.0	5613.97 ± 249.51	1.96 ± 0.07	0.05 ± 0.002	64.5

Adsorption isotherms

The use of adsorption isotherms is a very useful tool to understand the adsorption mechanism of the inhibitor on the surface of carbon steel in the aggressive medium. Generally, values around -20 kJ mol^{-1} or less negative are related to electrostatic interactions between the inhibitor molecules and the charged surface of the metal (physisorption) and those values around -40 kJ mol^{-1} or more negative involve a sharing or transfer of charge from the inhibitor molecules towards the metal surface to form coordinated covalent type bonds [25,41,42].

Several isotherm models exist, such as Temkin, Freundlich and Langmuir, but the latter is the most frequently used [9]. In this model, it is assumed that the inhibitor molecules occupy a single active site on the metal surface and that there is no interaction between neighbouring molecules. Equation (8) represents the Langmuir isotherm model

$$\frac{C}{\theta} = \frac{1}{k_{ads}} + C \qquad (8)$$

where C, k_{ads} and θ are the concentration of the inhibitor, the equilibrium constant of the adsorption process and the covered surface of the metal, respectively.

Figure 8 was plotted with the values of concentration (expressed in g L^{-1}) and θ obtained from Table 2 corresponding to the gravimetric tests, which fits perfectly to a straight line with a value of R^2 = 0.9868, proving that the adsorption of the inhibitor follows the Langmuir model (C/θ *vs*. C) due to the adsorption of a monolayer of inhibitory molecules on the surface of carbon steel [19]. The value of the equilibrium constant (k_{ads}) was determined from the intercept of the Langmuir isotherm at the *y* axis is 9.94 L g^{-1}, where this k_{ads} value obtained suggests strong adsorption of the *Bidens pilosa* extract molecules on the substrate surface. k_{ads} shows the interaction strength between the adsorbate and adsorbent in the electrolyte. Higher values of k_{ads} indicate effective adsorption of inhibitor molecules on the metal surface and high inhibition efficiency [28,34,43].

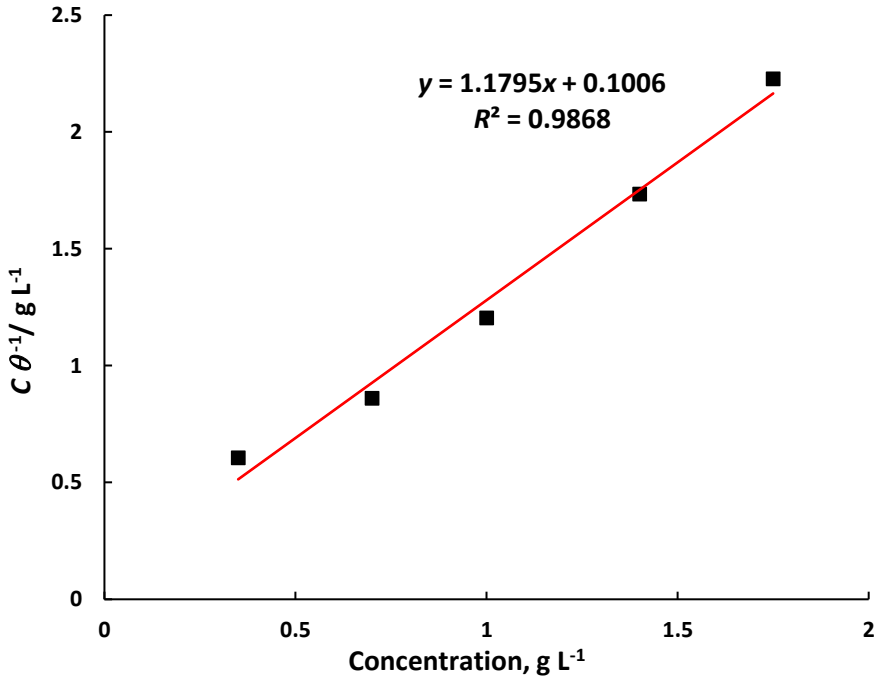

Figure 8. *Langmuir isotherm for carbon steel 1008 in neutral medium of 0.1 M NaCl containing different concentrations of* Bidens pilosa *extract*

With the value of the adsorption constant k_{ads} the Gibbs free energy of adsorption (ΔG^o_{ads}) was determined using Equation (9) [25]:

$$\Delta G^0_{ads} = -RT \ln(C_{H_2O}\, k_{ads}) \quad (9)$$

where T is the absolute temperature of the system under study, R is the universal gas constant (8.3147 J mol^{-1} K^{-1}), C_{H2O} is the concentration of water, and its value is 1000 g L^{-1} (55.5 mol L^{-1}) [25]. Substituting all needed values into Equation (9), a value of ΔG^o_{ads} = -22.8 kJ mol^{-1} is obtained. The negative value of the Gibbs adsorption free energy indicates that the inhibitor molecules spontaneously adsorb on the surface of the carbon steel. On the other hand, it can be observed that the value of ΔG^o_{ads} is around -20 kJ mol^{-1}, indicating that the inhibitory molecules coming from functional groups contained in the *Bidens pilosa* extract and being responsible for the decrease in the corrosion rate are physically adsorbed on the surface of the substrate [44].

In the case of the Freundlich and Temkin isotherms, the fitting was also carried out with θ values taken from Table 2, and the results are presented in Table 5, where the correlation coefficient values (R^2) were far from 1 (0.6092 and 0.5925, respectively), and therefore they will not be considered for this particular study.

Table 5. *Parameters obtained for different adsorption isotherms*

Isotherms	Equations	Linear fit and correlation coefficient (R^2)
Langmuir (C/θ *vs.* C)	$C/\theta = 1/k + C$	$C/\theta = 0.1006 + 1.1795C$ $R^2 = 0.9868$
Freundlich ($\log \theta$ *vs.* $\log C$)	$\log \theta = 1/k_f + (1/n) \log C$	$\log\theta = -0.1133 + 0.1876 \log C$ $R^2 = 0.6092$
Temkin (θ *vs.* $\log C$)	$\theta = (-2.303/a) \log k + (-2.303/a) \log C$	$\theta = 0.7754 + 0.2935 \log C$ $R^2 = 0.5925$

The results in Table 5 clearly show that the Langmuir isotherm presented the highest correlation coefficient value (0.9868) compared to the Freundlich (0.6092) and Temkin (0.5925) isotherms. In the literature, it is possible to find other studies using natural corrosion inhibitors for the protection of carbon steel where the adsorption mechanism was adequately adjusted to the Langmuir isotherm model [9,19,45,46].

Morphological characterization

In Figure 9, the surface of the 1008 carbon steel before immersion in the solution of 0.1 M NaCl is presented, where it is possible to see the scratches produced by the grinding process.

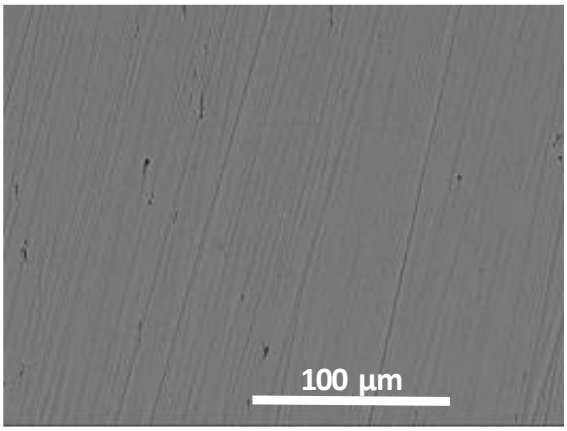

Figure 9. *Micrograph for carbon steel 1008 before the immersion in the electrolyte*

Figure 10a shows the appearance of the carbon steel surface after 60 min of immersion in the electrolytic solution in the absence of the inhibitor. In this condition, it is possible to observe a rough surface due to the drastic attack of the corrosive medium. In contrast, Figure 10b shows a surface with little corrosion products on the sample surface and no attack morphology even after 60 min of immersion. The few spots accentuated in Figure 10b can be associated with previous defects on the steel surface, and the inhibitor was adsorbed all over the steel surface, which forms an efficient protective film on the substrate.

Table 6 compares the efficiencies already obtained with the *Bidens pilosa* extract in relation to the efficiencies obtained using commercial phosphate-based inhibitors for carbon steel in NaCl neutral medium. As can be seen from Table 6, the majority of these commercial inhibitors present a higher efficiency compared to the *Bidens pilosa* extract, but it is important to highlight that our inhibitor is of the natural type and not synthetic, biodegradable and less harmful to health and environment. Another important detail to mention about Table 6 is that the commercial inhibitors chosen to compare the inhibition efficiencies were phosphate-based because the media in which they were tested were also neutral. Most commercial inhibitors present a better performance in an acidic medium than in a neutral medium and in the literature, there is not much information on inhibitors that can present high inhibitor efficiency when the medium is neutral.

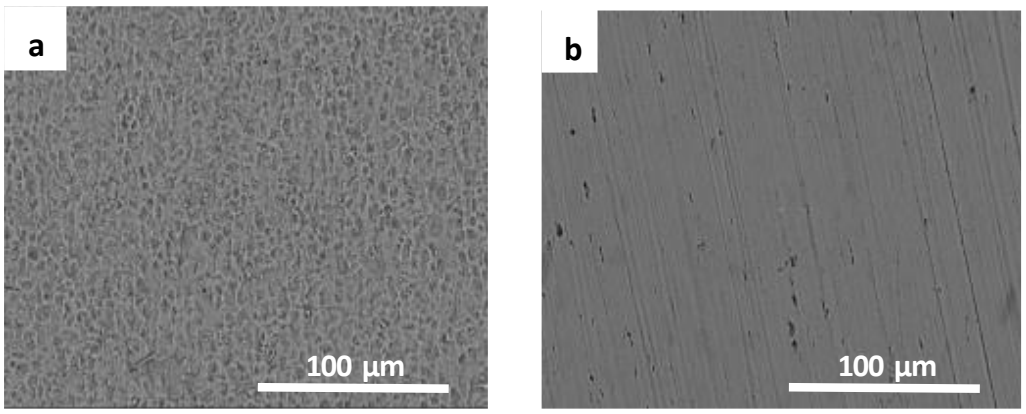

Figure 10. *Micrographs for carbon steel 1008 after immersion in the electrolyte, in the absence (a) and in the presence of* Bidens pilosa *extract inhibitor (b)*

Table 6. *Efficiencies obtained from EIS measurements for commercial phosphate-based inhibitors on carbon steel in NaCl solutions*

Inhibitor	Concentration	Immersion solution	Type of inhibition	IE, %	Ref.
Bidens pilosa extract	1000 ppm	0.1 M NaCl	Mixed	73.1	-
$Na_3PO_4.12H_2O$	0.4 mol per kg	3.5 wt.% NaCl	Cathodic	95	[47]
$Na_3PO_4.12H_2O$	7 %	0.5 M NaCl	Mixed	95.9	[48]
Na_3PO_4	0.6 M	0.6 M NaCl	Cathodic	88.6	[49]
Bis(2-ehylhexyl) phosphate (BEP)	500 ppm	1 wt.% NaCl	-	93.07	[50]

Conclusions

- According to the results obtained, it is possible to conclude that the *Bidens pilosa* extract is a potential and eco-friendly corrosion inhibitor for carbon steel 1008 in 0.1 M NaCl.
- The phytochemical analysis showed the presence of bioactive organic compounds, which confer that *Bidens pilosa* extract has inhibitory properties against corrosion.
- Gravimetric test results showed that the inhibition efficiency increases to a maximum value of 83.0 % for an optimal inhibitor extract concentration of 1000 ppm. The results of electrochemical impedance spectroscopy and polarization resistance (R_p) measurements also confirmed this trend, reaching maximum efficiency values of 73.1 and 75.6 % respectively.
- The thermodynamic study showed that the molecules of the *Bidens pilosa* extract are physically adsorbed (physisorption) on the surface of the carbon steel substrate due to the interaction of the heteroatoms such as carbon, nitrogen and oxygen found in the analyses by FITR.
- The adsorption process obeys the Langmuir isotherm, which assumes that each site is occupied by only one molecule of the inhibitor.
- The images obtained by optical microscopy indicate the reduction of the corrosive process in the presence of the *Bidens pilosa* extract.

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