Eco-friendly *Chamaerops humilis L.* fruit extract corrosion inhibitor for mild steel in 1 M HCl

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Abstract

Given the great interest in the application of medicinal and aromatic plants in several fields, including the control of corrosion of metals in different environments and in order to minimize the use of synthetic inhibitors, this is the objective of our research. In this work, ethanol and hexane extracts of *Chamaerops humilis L*. fruits were investigated as green corrosion inhibitors for mild steel in 1 M HCl solution. Corrosion rates were evaluated at 308 K using weight loss, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) techniques. Electrochemical techniques reveal that ethanol and hexane extracts exhibit excellent inhibition efficiency indicated that the corrosion mechanism is controlled by charge transfer process and function as mixed type inhibitors at temperature studied. Adsorption studies showed that the process follows the Langmuir adsorption isotherm. The weight loss measurements show that ethanol and hexane extracts of *Chamaerops humilis L*. fruits are excellent inhibitors in 1 M HCl medium and its inhibition efficiency (%*IE*) increases with an increase in the concentration of the inhibitors and reaches 88% and 80% at concentrations of 1 g/l of ethanol and hexane extracts, respectively. The mechanism for inhibiting corrosion of mild steel by ethanol and hexane extracts of *Chamaerops humilis L*. fruits has been proposed.

Keywords: *mild steel, Chamaerops humilis L., extracts, EIS, corrosion, weight loss, electrochemical.*

Received: January 15, 2020. Published: April 6, 2020 doi: 10.17675/2305-6894-2020-9-2-4

1. Introduction

Corrosion of metals is a major industrial problem that has attracted much investigations and researches. During certain operations like cleaning, pickling, descaling or even transportation it may come in the contact with hydrochloric acid and get severely corroded [1]. The use of inhibitors has been found to be one of the best options available for the protection of metals against corrosion [2–5]. Though many synthetic compounds showed

good anti-corrosive activity, most of them are highly toxic to both human beings and environment [6], expensive and are effective only at high concentrations. Natural products as corrosion inhibitors for various metals have been widely studied by several authors [7–11]. The metal corrosion inhibition activity of plant extracts can be attributed to the presence of heterocyclic compounds like alkaloids, flavonoids, and other compounds such as tannins, cellulose and polycyclic compounds. These compounds form a protective layer on the metal surface, thus preventing corrosion.

Chamaerops humilis L. is a medicinal plant which belongs to the Arecaceae family. It is frequently found in the North Africa especially occidental Mediterranean area [12–15]. Several studies have been shown the beneficial effects of *Chamaerops humilis* against chronically diseases such as cancer, ulcer, kidney stones [16–22]. Nevertheless, there is insufficient information regarding the corrosion inhibition study of *Chamaerops humilis L*. Benali *et al.* [23] studied the corrosion and inhibition behaviors of mild steel in sulfuric acid + 5% of ethanol in the presence of tannin extract of *Chamaerops humilis* plant (LF-Ch) and potassium iodide (KI).

In the present study, the adsorption and the corrosion inhibition effect of ethanol and hexane extracts of *Chamaerops humilis L*. fruit extract was investigated using weight loss, potentiodynamic polarization and electrochemical impedance spectroscopy methods.

2. Materials and methods

2.1. Material

Hydrochloric acid, ethanol and hexane were purchased from (E. Merck). All other chemicals and solvents were of the highest analytical grade and used as supplied.

The fruits of the *Chamaerops humilis* plant were collected from Chaouia-Ouardigha located at western Morocco in July 2014, Figure 1. The plant sample was identified by the authors. The seeds are separated from the pulps, dried, peeled, finely ground and then packaged to protect from light and moisture.

(a) (b)

Figure 1. *Chamaerops humilis L*. from Chaouia-Ouardigha (a: leaves, b: fruits).

2.2. Preparation of extracts

Extracts with ethanol or hexane were prepared by the procedure described in our previous work [24].

2.3. Preparation of mild steel coupons

Mild steel sheet was obtained commercially. The used mild steel coupons have percent composition (%wt.) of 0.09% P, 0.38% Si, 0.01% Al, 0.05% Mn, 0.21% C, 0.05% S and 99.21% Fe. The coupons were polished using emery paper of grade ranging from N°400 to N°1200; and then rinsed with distilled water, degreased with acetone and dried with warm air before use. Different inhibitor concentrations (0.125, 0.25, 0.5 and 1 g/L) were prepared from the methanol or hexane extract in 100 mL of 1 M HCl with stirring at room temperature. 100 mL of 1 M HCl without inhibitor was used as blank test solution. All tests were performed in aerated medium. The tests were done at 308 K atmospheric pressure.

2.4. Weight loss, polarization and EIS measurements

Weight loss measurements were carried out in a double walled glass cell equipped with a thermostat-cooling condenser. The solution volume was 100 ml. The used steel specimens had square shape with the following dimensions (1.5 cm × 1.5 cm × 0.05 cm). They were immersed in the tested extract for 6 hours at 35°C. After that, the specimens were carefully washed with double-distilled water, dried and then weighed. From the weight loss measurements, the corrosion rate expressed in $mg \cdot cm^{-2} \cdot h^{-1}$ was calculated. Electrochemical measurements were carried out in a conventional three-electrode electrolysis cylindrical Pyrex glass cell. The working electrode (WE) in the form of disc cut from mild steel has an area of 1 cm^2 and was embedded in polytetrafluoro ethylene. A saturated calomel electrode (SCE) and a disc platinum electrode were used, respectively, as reference and auxiliary electrodes. The temperature was thermostatically controlled at 35±1°C. The electrochemical study was carried out using a potentiostat PGZ100 piloted by Voltamaster software. This potentiostat was connected to a cell with three electrode thermostats with double wall. A saturated calomel electrode (SCE) and platinum electrode were used as reference and auxiliary electrodes, respectively. Anodic and cathodic potentiodynamic polarization curves were plotted at a polarization scan rate of 0.5 mV/s. The potential was stabilized at free potential for 30 minutes before any run. The polarization curves are obtained from −800 mV to −200 mV at 308 K. The solution test was de-aerated by bubbling nitrogen. The electrochemical impedance spectroscopy (EIS) measurements were carried out with the electrochemical system, which included a digital potentiostat model Voltalab PGZ100 computer at E_{corr}. After the determination of steady-state current at a corrosion potential, sine wave voltage (10 mV) peak to peak, at frequencies between 100 kHz and 10 mHz are superimposed on the rest potential. Computer programs automatically controlled the measurements performed at rest

potentials after 30 minutes of exposure at 308 K. The impedance diagrams are shown in the Nyquist representations. Experiments were repeated three times to ensure the reproducibility of results.

3. Results and discussion

3.1. Weight loss technique

The corrosion behavior of mild steel in 1 M HCl in the absence and presence of hexane and ethanol extracts was studied at temperature 308 K using weight loss technique and data obtained at time (6 h) are shown in Table 1.

The corrosion rate (v) is calculated using the following equation:

$$v = \frac{W}{St} \tag{1}$$

Where: W is the average weight loss, S the total area, and t is immersion time. With the corrosion rate calculated, the inhibition efficiency (E_w) is determined as follows:

$$E_W\% = \frac{v_0 - v}{v_0} \times 100 \tag{2}$$

Where: v_0 and v are, respectively, the values of corrosion rate with and without inhibitor.

Table 1. Impedance parameters with corresponding inhibition efficiency for the corrosion of mild steel in 1.0 M HCl at different concentrations of hexane and ethanol extracts.

Inhibitor	***C* (g/L)**	***C*R (mg/cm²·h)**	***E* (%)**
HCl 1 M	–	0.903	–
Hexane extract	1.000	0.174	80
	0.500	0.249	72
	0.250	0.283	68
	0.125	0.313	65
Ethanol extract	1.000	0.101	88
	0.500	0.114	87
	0.250	0.120	86
	0.125	0.172	80

From Table 1, it is clear that the corrosion rate of the mild steel is reduced in the presence of inhibitors as compared to the free acid solution. It is also clear that as the concentrations interval increases the inhibition efficiency also increases. Higher inhibition efficiency was shown at 1 g/L for ethanol extract (88%). The increased inhibition efficiency with concentrations interval indicates that more inhibitors molecules are

adsorbed on the mild steel surface as the immersion time 6 h, leading to greater surface coverage and hence the formation of a protective film.

In order to obtain a better understanding of the corrosion protection mechanism of hexane and ethanol extracts corrosion of mild steel in 1 M HCl, a detailed study was carried out using electrochemical techniques.

3.2. Adsorption isotherm

An adsorption isotherm is a graphical representation of variation of extent of adsorption with pressure or concentration of adsorbate at a given constant temperature. The phenomenon of adsorption plays a vital role during the action of corrosion inhibitors. Hence a thorough knowledge about the adsorption isotherm is pre-requisite in understanding the nature of interaction prevailing between the inhibitor molecules and the metal surface [25].

As seen from Figure 2, the plot of C_{inh}/θ *versus* C_{inh} yields a straight line with a correlation coefficient more than 0.999, showing that the adsorption of hexane and ethanol extracts inhibitors in acidic solutions is fitted to Langmuir adsorption isotherm.

These results show that the inhibition of mild steel in HCl solutions by hexane and ethanol extracts is an adsorptive process, (equation 3).

$$\frac{C}{\theta} = \frac{1}{K} + C \tag{3}$$

This isotherm assumes that the adsorbed molecules occupy only one site and there are no interactions between the adsorbed species. The K_{ads} values can be calculated from the intercept lines on the C/θ-axis. This value is also related to the standard free energy of adsorption (ΔG^0_{ads}), by the following equation (4), where C_{inh} is the inhibitor concentration, K_{ads} is the adsorption equilibrium constant, ΔG^0_{ads} is the standard free energy of adsorption, 55.5 is the concentration of water in the solution in mol·dm^{-3}, R is the universal gas constant and T is the absolute temperature in Kelvin:

$$\Delta G^0_{ads} = -RT \ln(55.5 K_{ads}) \tag{4}$$

The values of K_{ads} and ΔG^0_{ads} for hexane and ethanol extracts are given in Table 2.

The standard free energy of adsorption ΔG^0_{ads}, which can characterize the interaction of adsorption molecules and metal surface, was calculated. The large negative values of ΔG^0_{ads} ensure the spontaneity of the adsorption process and the stability of the adsorbed layer on the mild steel surface as well as a strong interaction between different organic molecules forming the hexane and ethanol extracts molecules and the mild steel surface.

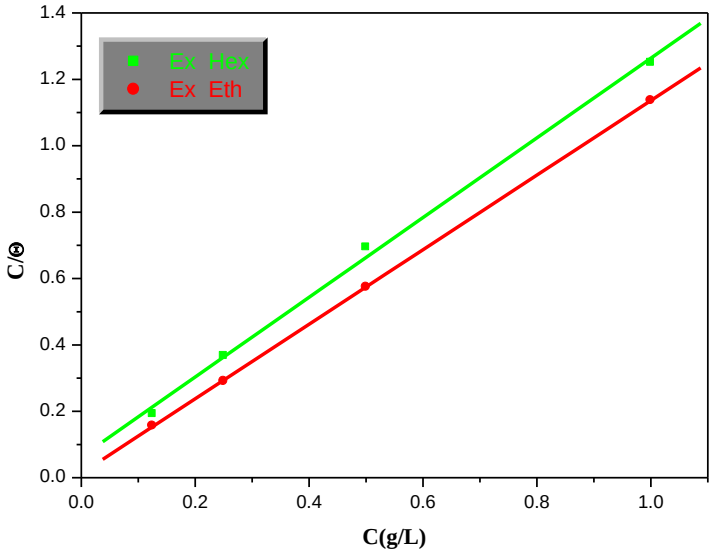

Figure 2. Langmuir isotherm of mild steel in the 1 M HCl in presence hexane and ethanol extracts calculated by weight loss technique at 308 K.

Table 2. Equilibrium constant and free energy of adsorption values in presence of the studied inhibitors in 1 M HCl on mild steel at 308 K.

Inhibitors	**Linear coefficient regression (r)**	**Slope**	K_{ads} **(L·mol^{-1})**	ΔG^0_{ads} **(kJ·mol^{-1})**
Hexane extract	0.99876	1.20038	15.7678966	–17.34
Ethanol extract	0.99998	1.12273	75.5857899	–21.35

Generally, values of ΔG^0_{ads} up to –20·kJ·mol^{-1}, the types of adsorption was regarded as physisorption, the inhibition acted due to the electrostatic interactions between the charged molecules and the charged metal, while the values around –40·kJ·mol^{-1} or smaller were associated with chemisorption as a result of sharing or transfer of electrons from organic molecules to the metal surface to form a coordinate type of bond (chemisorption) [26].

Here, the calculated ΔG^0_{ads} values are ranging between –17.34 and –21.35 kJ·mol^{-1}, indicating that the adsorption mechanism of hexane and ethanol extracts molecules on mild steel in 1 M HCl solution at the studied temperature is physisorption.

3.3. Electrochemical impedance spectroscopy

Figures (3 and 4) show electrochemical impedance spectra (Nyquest plots) at open circuit potential for mild steel corrosion in different concentrations of 1 M HCl containing hexane and ethanol extracts at 308 K. A significant increase in the impedance system causing

enhance in the resistance to charge transfer process is the result as formation of an insulated layer on the mild surface. It is obvious that the measurements of the impedance spectroscopy determine the performance of inhibitors. The inhibition efficiency of the inhibitor was calculated from the charge transfer resistance values using the following equation:

$$E\% = \frac{R_{ct} - R_{ct}^{0}}{R_{ct}} \times 100 \quad (5)$$

Where, R_{ct}^{0} and R_{ct} are the charge transfer resistance in absence and in presence of inhibitor, respectively.

These plots having the shape of a semicircle for all the concentrations of hexane and ethanol extracts examined indicate that the corrosion is mainly inhibited by charge transfer process. The electrochemical impedance parameters namely the solution resistance (R_s), charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) are derived from these curves are given in Table 3. It could be observed that the R_{ct} increased with increase in the concentration of hexane and ethanol extracts signifies that the insulated film obtained acts as a barrier layer to the corrosion process that clearly proves the existence and formation of the film [27–33].

Table 3. Electrochemical parameters for mild steel in 1 M HCl without and with different concentrations of hexane and ethanol extracts at 308 K.

	Concentration (g/L)	R_{ct} **(ohm·cm^2)**	C_{dl} **(μF/cm^2)**	E **(%)**
HCl 1 M	–	20.77	200	–
Ethanol extract	1.000	99.25	142	79
	0.500	89.43	150	77
	0.250	66.80	160	69
	0.125	56.61	180	63
Hexane extract	1.000	92.74	146	78
	0.500	79.45	152	74
	0.250	77.96	157	73
	0.125	58.67	171	65

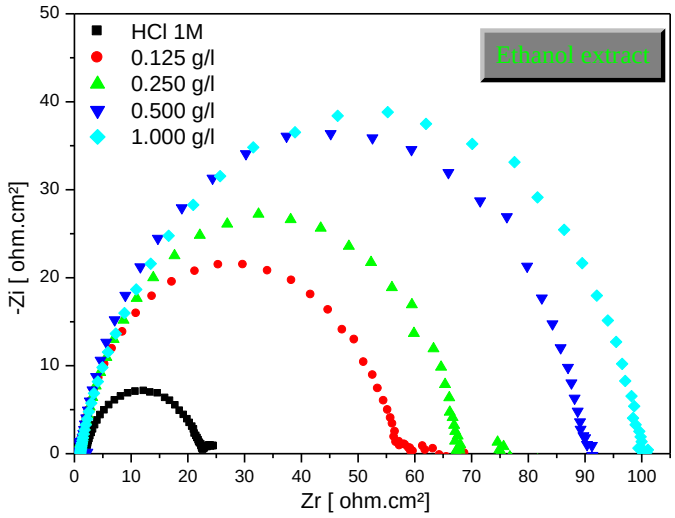

Figure 4. Nyquist plot for mild steel in 1 M HCl in the absence and presence of ethanol extract at temperature 308 K.

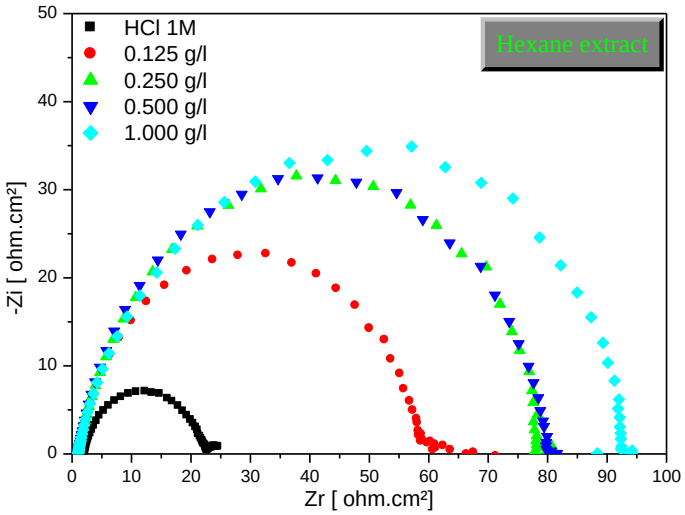

Figure 5. Nyquist plot for mild steel in 1 M HCl in the absence and presence of hexane extract at temperature 308 K.

3.4. Potentiodynamic polarization study

Tafel found that a linear relationship exists between electrode potential (E) and logarithm of current ($\log i$), if an electrode is polarized to sufficiently large potentials, both in anodic and cathodic directions. The regions in which such relationship exists are known as Tafel regions. Biasing the potential on the anodic and cathodic sides of the Tafel region linearly and extrapolating them to corrosion potential (E_{corr}) give the corresponding corrosion

current density (i_{corr}) values. The plot of *E versus* log *i* in the anodic and cathodic Tafel regions gives the corresponding Tafel slopes (β_a and β_c).

The percentage inhibition efficiency (E_p%) were calculated using the following equation (6):

$$E_p\% = (i_{corr(0)} - i_{corr(inh)} / i_{corr(0)}) \times 100 \quad (6)$$

Where, $i_{corr(0)}$ and $i_{corr(inh)}$ represent corrosion current density values without and with inhibitor, respectively;

Polarization curves for mild steel in 1 mol·L^{-1} HCl solution at temperature 308 K in the absence and presence of the hexane and ethanol extracts are shown in Figures 6 and 7. All the calculated electrochemical corrosion parameters such as corrosion current density (i_{corr}), corrosion potential (E_{corr}), anodic and cathodic Tafel slopes (β_a and β_c, respectively) are presented in Table 4.

It is clear from the table, that the i_{corr} decreases considerably in the presence of individual inhibitors and it reaches a minimum value for the inhibitors. These results clearly reveal that the added inhibitors act as an effective corrosion inhibitor of mild steel in 1 mol·L^{-1} HCl. As it can be seen from the table, no significant shifts are observed in the E_{corr} values for both the inhibitors which indicate that the studied systems are functioning *via* a mixed type mechanism.

Also, only slight shifts were observed in the anodic and cathodic Tafel slopes, indicating that the studied inhibitor systems under investigation act as efficient corrosion inhibitors, suppressing both anodic dissolution and cathodic hydrogen evolution reaction by getting adsorbed onto the mild steel surface blocking the active sites, without altering the mechanism of corrosion reaction.

Table 4. Polarization parameters and inhibition efficiency for the corrosion of mild steel in 1 M HCl in the absence and presence of inhibitors (hexane and ethanol extracts) at 308 K.

	Concentration (g/L)	**E_{corr} (mV/SCE)**	**β_a (mV/dec)**	**β_c (mV/dec)**	**I_{corr} (mA/cm^2)**	**E_p (%)**
Blank	0.000	–454		–182	1.9477	–
Ethanol extract	1.000	–457	67	–151	0.2469	87
	0.500	–459	71	–147	0.2713	86
	0.250	–463	72	–142	0.2972	84
	0.125	–466	84	–164	0.4912	74
Hexane extract	1.000	–461	78	–160	0.3947	79
	0.500	–473	87	–163	0.4224	78
	0.250	–461	81	–159	0.4955	74
	0.125	–459	87	–160	0.6850	64

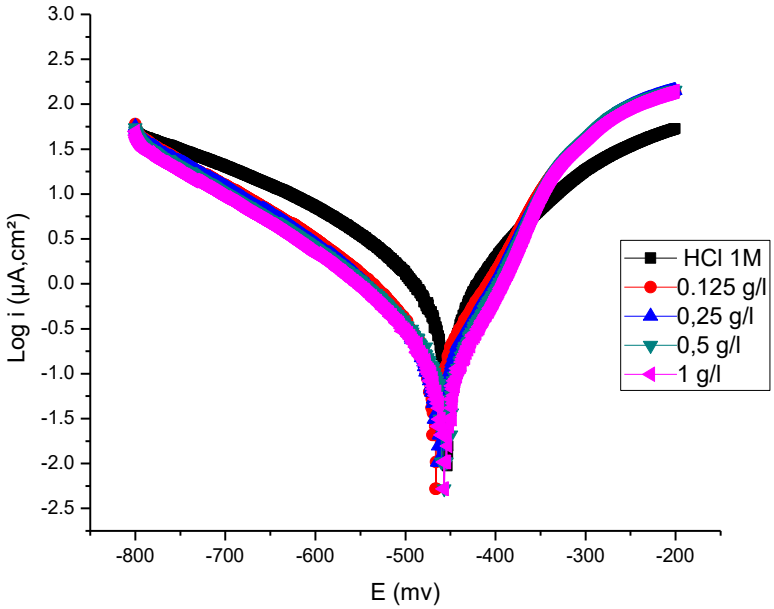

Figure 6. Potentiodynamic polarization curve for mild steel in 1 M HCl in the absence and presence of ethanol extract at the temperatures 303 K.

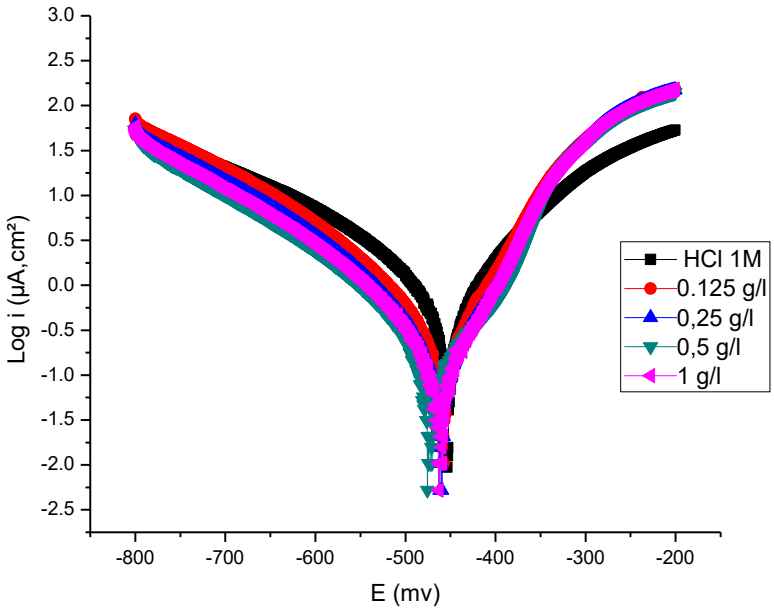

Figure 7. Potentiodynamic polarization curve for mild steel in 1 M HCl in the absence and presence of hexane extract at the temperatures 303 K.

3.5. Corrosion inhibition mechanism

It is a fact that extraction solvents differ in terms of extraction preference and resistance. Differences in preference and extraction strength may have an effect on the inhibition performance of a plant extract dissolution. The inhibition efficiency values of ethanol and hexane extracts of *Chamaerops humilis L*. fruits obtained from the various techniques applied are given in the Tables (1, 3, 4). The data in the table show that the extraction solvent has an effect on the corrosion inhibition performance of the extract. On average, the performance order of corrosion inhibition by the extract is ethanol > hexane.

Knowing that hexane is a non-polar solvent, therefore it can extract non polar and very slightly polar molecules such as fatty acids, tocopherols (vitamin E), sterols, while ethanol is a polar solvent that has the capability of extracting more compounds than hexane. It can also extract polyphenols, flavonoids and tannins. The total polyphenol content (phenolic acids, flavonoids, tannins) in ethanol extracts explains why ethanol extracts have a more remarkable antioxidant and therefore anti-corrosion activity compared with hexane extract [24].

The overall corrosion inhibition may possibly be a synergistic interaction between the different adsorbed constituents on the metal surface.

4. Conclusions

The principle finding of the present work could be summarized as follows:

Results from the techniques employed show that ethanol and hexane extracts of *Chamaerops humilis L.* fruits are effective inhibitors for the corrosion of mild steel in 1 M HCl.

- Electrochemical studies show that the inhibitors are more effective at the studied temperature (308 K). Higher inhibition efficiency is shown at concentration 1 g/L.
- Potentiodynamic studies reveal that the inhibitors act as mixed type.
- The adsorption process of the inhibitors obeys Langmuir adsorption isotherm at 308 K. The values of standard free energy of adsorption (ΔG_{ads}) indicate that adsorption of inhibitors is neither typical physisorption.
- Charge transfer resistance increases and double layer capacitance decreases due to adsorption of the inhibitors' molecules on the mild steel surface.
- The inhibition efficiency values obtained from weight loss, polarization curves, and EIS are in good agreement.
- However, neither the molecular mechanism of the anticorrosive nor the specific compound or mixture of compounds responsible for this bioactivity is well known.

Acknowledgments

The authors gratefully acknowledge financial support of this work by the CNRST of Morocco.

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