Inhibition of acid corrosion of mild steel by aqueous nettle extracts

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Abstract

Purpose – The aim of this research was to investigate the use of aqueous extracts of nettle plant (NE) as a green corrosion inhibitor of mild steel in hydrochloric acid solution.

Design/methodology/approach – The inhibition efficiency was investigated by weight loss measurements, potentiodynamic polarizations, electrochemical impedance spectroscopy, SEM observations and EDX analysis.

Findings – The inhibition efficiency increased with an increase in concentration of NE up to a critical concentration of $1.5 \times 10^{-3}\ \mathrm{g \cdot cm^{-3}}$ where the highest inhibition efficiency of 97 percent was obtained. The adsorption of the inhibitor was spontaneous (reflected by the negative value of ΔG^0_{ads}), supported the mechanism of physical adsorption and obeyed to the Langmuir adsorption isotherm. The inhibition action of the extracts was independent on the storage time; it could be conserved without any specific conditions of time and temperature.

Research limitations/implications – The anticorrosion effect can be better understood when the active compound in the extracts is identified and what is the inhibition efficiency of one component in the presence of another in the mixture (synergetic or antagonist effects).

Practical implications – Nettle is a healthy plant, without particular toxicity that can find possible applications as environmentally friendly inhibitor of mild steel used as materials in food industry.

Originality/value – Aqueous nettle extracts were studied for the first time as corrosion inhibitor and its anticorrosion effect was proven by standard methods.

Keywords Corrosion inhibitor, Nettle extracts, Langmuir, Mild steel, EIS, HCl

Paper type Research paper

Introduction

Several corrosion protection methods were used in industrial processes. The application of inhibitors is one of the best techniques that reduces corrosion rate of metallic materials especially in acids and becoming increasingly popular (Shuduan and Xianghong, 2012; Shah *et al.*, 2011; Chaudhari *et al.*, 2007; Eddy, 2010; Sørensen *et al.*, 2009; de Souza and Spinelli, 2009; Uhlig and Revie, 1985; Dupont, 2008; Sastri, 1998; Abdel-Gaber *et al.*, 2009; Bouyanzer *et al.*, 2007; Samide *et al.*, 2005). The efficiency of these inhibiting species is mainly dependent on their ability to be adsorbed on the metal surface.

Nevertheless, the use of chemical inhibitors has been limited because of the environmental threat. Recently, non-toxic, eco-friendly, harmless, readily available and renewable natural products are again used as inhibitors in order to develop new clean chemicals for green environment. Most of the natural products are non-toxic, biodegradable and available.

Zucchi and Omar (1985) have studied the effect of various plants extracts (Papaia, *Poinciana pulcherrima*, *Cassia occidentalis*, etc.) on the corrosion protection of the mild steel in HCl solutions. It was found that the majority of these extracts reduced the corrosion of steel with an efficiency of 88-96 percent in HCl solutions.

Deng and Li (2012) studied the effect of *Ginkgo* leaves extract on the corrosion of cold rolled steel in both HCl and H_2SO_4 media. Oguzie (2005) investigated the anticorrosion effect of *Telfaria occidentalis* extract for mild steel in acid media. The corrosion inhibition of mild steel in HCl media in presence of crude methanolic extract of *Artemisia pallens* was reported by Garai *et al.* (2012). The extracts of *Punica granatum* peel and their main constituents involve ellagic and tannic acids. These chemicals were tested by Behpour *et al.* (2012) in acidic solutions as mild steel corrosion inhibitors. A detailed review has also been reported on the use of natural products as corrosion inhibitors for metals in corrosive media (Bothi and Sethuraman, 2008). The corrosion inhibition activity in many of these plant extracts could be due to the presence of heterocyclic constituents like alkaloids, flavonoids, and others compounds as tannins, cellulose, etc.

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One effective compound in a certain medium with a given metal may be ineffective for the same metal in another media (Okafor *et al.*, 2005).

All these encouraging results obtained by natural plants extracts as corrosion inhibitors of steel in acid solutions permit us to test other plants, namely nettle (*Urtica dioica* L.).

Nettle (*Urtica dioica* L.) is a perennial plant belonging to the family of Urticaceae; it grows in temperate and tropical wasteland areas around the world. Several researches revealed the presence of more than 50 different chemical constituents such as starch, gum, sugar, leanol acid, sterols and steryl glycosides (including 3-beta-sitosterin), scopoletin (a coumarin), secoisolariciresinol, neo-olivil (both lignans), homovanillyl alcohol, minerals, amino acids and active compounds such as tannins, formic acid, salicylic acid, etc. (Guil-Guerrero *et al.*, 2003; Hojnik *et al.*, 2007; Safamehr *et al.*, 2012).

Nettle is a very nutritious food that is easily digested and contains high concentration of minerals like selenium, iron, magnesium, sodium, calcium, potassium, sulfur, vitamins and B-complexes as well as thiamin, riboflavin and niacin. All these compounds are present in nettle in high amounts and act as antioxidants and growth-stimulating properties. Because of its high content of nutritive substances such as amino acids, minerals and vitamins and active compounds such as tannins, formic acid, salicylic acid, carvacrol and thymol, nettle could be used as common dietary additive for humans, in folk remedies (Safamehr *et al.*, 2012).

Therefore, the present work is aimed at investigating the potential of water extracts of nettle (*Urtica dioica* L.) as corrosion inhibitor of mild steel in hydrochloric acid.

The study was carried out by using weight loss measurements, potentiodynamic polarizations and electrochemical impedance spectroscopy (EIS) as techniques to evaluate the corrosion rate of steel and inhibition efficiency of nettle plant (NE) extracts. The steel surface was also examined by scanning electron microscope (SEM) observations and energy dispersive X-rays (EDX) analysis. The effect of the immersion time of steel electrodes in acidic solutions at different temperatures in the absence and presence of the plant extract was also studied. The effect of NE extract aging on the corrosion rate was also evaluated. To our knowledge the extract of this plant has never been used previously in the protection of steel corrosion in acid medium.

Experimental

Solutions preparation

NE extracts were obtained by drying the plant in an oven at 40°C for 24 h. The substrate was then grinded to powder. A sample of 10 g of the dry powder was extracted by reflux in 100 ml of distilled water for 1 h. The refluxed solution was filtered and stored. According to the method described by Abdel-Gaber *et al.* (2006) and Zaabar (2010), 10 ml of the filtrate was evaporated and a solid residue was weighed in order to determine the concentration of the stock solution (expressed in grams per cm^3 of solution ($\mathrm{g \cdot cm^{-3}}$)). Test solutions were prepared by the method of dilution from the stock solution. It consists to add few drops of concentrated HCl to a mixture of an appropriate quantity of the stock solution and distilled water to obtain a total solution volume of 100 cm^3 with a desired concentration of nettle extracts and with a concentration of 0.5 M HCl.

Weight loss measurements

The working electrode tested in this work is made from mild steel. The chemical composition (wt.%) of the material is presented in Table I. The weight loss measurements and surface analysis were carried out on sheets with dimensions of 1 cm × 2 cm × 0.09 cm. Cylindrical rods having a geometric area of 0.785 cm^2 were used for the electrochemical investigations. Before use, the different electrodes were polished with emery papers to 1200 grit (15 μm) and rinsed with distilled water.

The weight loss was determined at different immersion times at 25 ± 1°C, for a total period of 12 days, by weighing the cleaned samples before and after hanging the coupon into 100 cm^3 of the corrosive solution, namely 0.5 M HCl in the absence and presence of various concentrations of NE. Inhibition efficiency (η) was calculated from the weight loss measurements using the following equation (Rahim *et al.*, 2007; Bouyanzer *et al.*, 2007; Oguzie *et al.*, 2010; Eddy and Odiongenyi, 2010):

$$\eta(\%) = \left[\frac{w - w_i}{w}\right] \times 100 \tag{1}$$

where w and w_i are the weight losses of the coupon steel in free and inhibited solutions, respectively.

Electrochemical tests

For electrochemical measurements, the experiments were carried out in a conventional three-electrode double walled cylindrical glass cell. A saturated calomel electrode (SCE) and a platinum electrode were used as reference and auxiliary electrode, respectively. The polarization curves, at a scan rate of 1 mV s^{-1}, were performed using a potentiostat/galvanostat (PGP 201) coupled with an HP personal computer under a voltamaster software. The fraction of the surface covered by the adsorbed molecules (θ) was determined from this equation:

$$\theta = \left[1 - \frac{i_{corr}}{i^0_{corr}}\right] \tag{2}$$

where i^0_{corr} and i_{corr} are the uninhibited and inhibited corrosion current densities, respectively, obtained from the Tafel parameters.

The inhibition efficiency (η) is calculated from equation (3) (Negm *et al.*, 2012; Grosser and Gonçalves, 2008; Fallavena *et al.*, 2006):

$$\eta(\%) = \theta \times 100 \tag{3}$$

EIS was carried out at E_{corr} potential in aerated solution using an AUTOLAB potentiostat/galvanostat (PGSTAT30) under FRA software. We measured the response of our electrochemical system to AC excitation with a frequency ranging from 100 kHz to 10 mHz and peak to peak AC amplitude of 10 mV. The impedance diagrams are given in the Nyquist representation. The data emulation program of the type Setup_ZSimDemo_320 was used to treat the impedance

Table I Composition in wt.% of the mild steel used in this study

Element	Fe	Mn	Al	Si	C
Composition (wt.%)	98	0.94	< 1	0.31	0.2

data and to determine the equivalent circuit which describes the reaction between the solution and the sample surface. Analysis of the impedance spectra was done by fitting the experimental data using the nonlinear least square fitting procedure. The quality of fitting to the equivalent electrical circuit was judged by the error distribution versus frequency, comparing experimental with simulated data. Good agreement between the fitted and experimental data was obtained. (The calculation error given by the model is less than 4 percent.)

In this part, our intention is not to give a thorough modeling of the interface metal/electrolyte, but to schematize the electrochemical system coarsely in order to extract the values from the parameters allowing evaluating the effectiveness of the used inhibitor (Amar *et al.*, 2006).

Surface analysis

The surface of the mild steel electrodes, before and after their exposure in the studied solutions (with and without inhibitor) were examined with a Philips FEI Quanta 200 SEM with a tungsten filament with a link to a Genesis X-ray analysis system EDX using an electron acceleration of 20 keV to determine the elemental composition of the films formed onto the electrode surfaces. During analysis, the sample was glued with conductive cement on a stub. The EDX collector time was 100 s.

Results and discussion

To test the reliability and reproducibility of the measurements, duplicate experiments were performed in each case and in the same conditions.

Weight loss study

Weight loss measurements provide reliable results concerning the efficiency of a given inhibitor, so that the corresponding corrosion data approach service conditions more accurately than the data obtained with any other test. Figure 1(a) and (b) present the results of weight loss measurements for the corrosion of steel, in 0.5 M HCl solutions, and inhibition efficiency (η%) with different concentrations of NE at 25°C. The data obtained reveal that the weight losses decreased markedly and the η% increased in the presence of NE.

These results indicated clearly the inhibitive effect of the added extract on steel corrosion in the acidic solution showing that the inhibition efficiency was improved with the increase of the concentration of the extract in the solution. The inhibiting action was more pronounced with $1.5 \times 10^{-3}\,\mathrm{g \cdot cm^{-3}}$ of NE where the highest inhibition efficiency of 97 percent was obtained. Beyond $1.5 \times 10^{-3}\,\mathrm{g \cdot cm^{-3}}$ of NE, the efficiency was maintained constant, so this value of $1.5 \times 10^{-3}\,\mathrm{g \cdot cm^{-3}}$ was chosen as the optimum value which gave the best results in relation with steel corrosion protection.

Polarization measurements

In order to gain a better knowledge concerning classification of a compound as an anodic, cathodic or mixed type inhibitor and its effect on the kinetics of the anodic and cathodic reactions, etc. polarization measurements were carried out.

The effect of natural substance (NE) concentration on the typical anodic and cathodic polarization curves of steel in 0.5 M HCl solutions was studied and presented in Figure 2.

Figure 1 (a) Variation of the weight loss data with the immersion time recorded for a mild steel electrode in 0.5 M HCl solution in the absence and presence of various concentrations of NE, at 25°C and (b) inhibition efficiency (η%) vs the concentration of the inhibitor, deduced from (a)

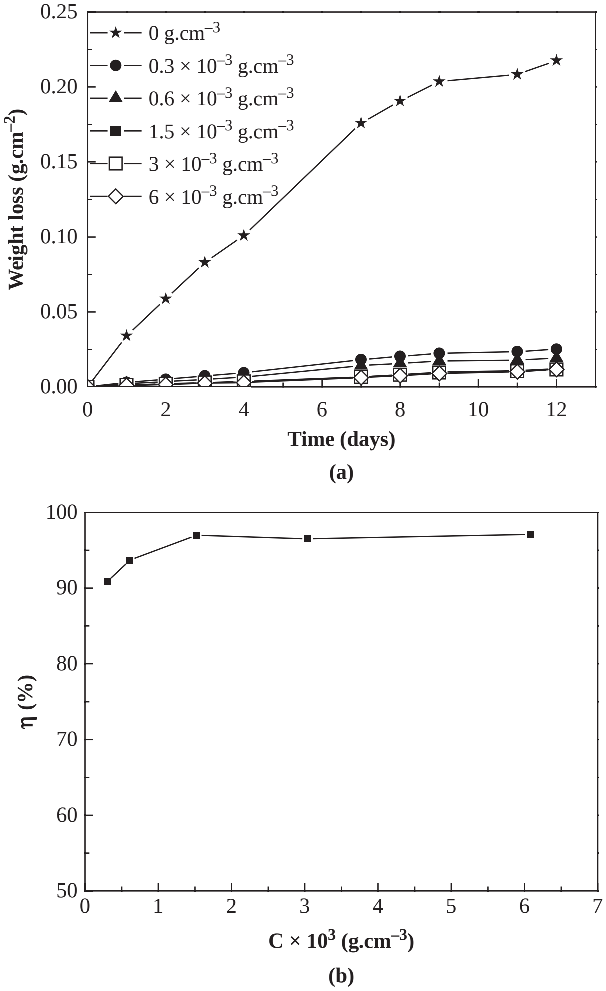
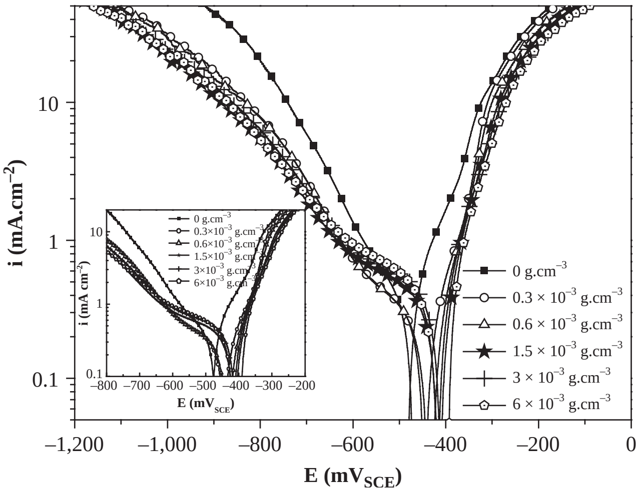


Figure 2 Potentiodynamic anodic and cathodic polarization curves of a mild steel electrode in 0.5 M HCl solution without and with different concentrations of NE at a scan rate of 1 mVs^{-1}, at 25°C



As it would be expected, both anodic and cathodic reactions of steel electrode corrosion were inhibited by increasing the concentration of NE up to a critical concentration value of $1.5 \times 10^{-3}\,g \cdot cm^{-3}$. As NE concentration increased up to $3 \times 10^{-3}\,g \cdot cm^{-3}$, there was no significant change in polarization curve. At this critical value of concentration of NE, the cathodic and anodic current densities values deduced from individual cathodic polarization (at potential -900 mV/SCE) and anodic polarization (at potential $+100$ mV/SCE) curves were, respectively, 3.4 and 1.4 times lower than that recorded without inhibitor. This NE substance acted as a typical mixed inhibitor, implying that the addition of NE reduced mainly the anodic dissolution and also slightly retarded the hydrogen evolution reaction. The action of NE might be attributed to the adsorption and formation of a barrier film on the steel surface. Note that the addition of inhibitor had a beneficial effect even at low concentration. It was also observed that an increase in NE concentration shifts the corrosion potential (E_{corr}) in the positive direction (noble direction) indicating the formation of a protective layer of the inhibitor on the electrode surface. The formation of such barrier film was confirmed by EDX analysis of the electrode surface (as will be showed later). These findings show that NE extracts are good candidates as corrosion inhibitors and the protection against corrosion must be attributed to a barrier effect (Martinez and Metikos-Hucovic, 2003).

In addition, the Tafel lines remained almost unchanged (same shapes) upon addition of inhibitor. This result suggests that the adsorbed inhibitor acted by simple blocking of active sites for both anodic and cathodic processes. In other words, the inhibitor decreased the surface area for corrosion without affecting the corrosion mechanism and only causes inactivation of a part of the surface with respect to the corrosive medium (Martinez and Metikos-Hucovic, 2003; Hazzazi, 2007; de Souza and Spinelli, 2009). The increasing steel surface coverage as a function of NE concentration is also shown in Table II. As can be seen, both θ and η% increased with increasing concentration of NE up to optimum concentration.

The variation of η% determined by the two methods, namely gravimetric and polarization curves, depending on the concentration of NE in 0.5 M HCl is shown in Table III. The values of inhibition efficiency determined by the polarization curves were slightly lower than those determined by the other method.

A similar observation on the difference between the two used methods has been reported before (Al-Turkustani *et al.*, 2011; Rengamani *et al.*, 1994; Popova *et al.*, 2004). The observed differences might be attributed to the fact that the gravimetric method gives average corrosion rates, whereas electrochemical methods give instantaneous corrosion rates. Indeed, there is formation of a protective film more important in the case of gravimetric study in which the immersion time is 12 days, which explains the discrepancy between the obtained values.

Table II The electrochemical parameters (i_{corr}, E_{corr}, η% and θ) associated with polarization measurements of mild steel electrode in 0.5 M HCl solution in the absence and presence of different concentrations of NE

Concentration of NE × 10^3($g \cdot cm^{-3}$)	i_{corr} ($mA \cdot cm^{-2}$)	E_{corr} (mV_{SCE})	η%	θ
0	1.17	−477.00	–	–
0.3	0.40	−446.14	65.8	0.658
0.6	0.35	−434.11	70.1	0.701
1.5	0.19	−419.06	83.8	0.838
3	0.25	−416.05	78.6	0.786
6	0.23	−406.27	80.3	0.803

Table III The variation of η% determined by the two methods, namely gravimetric and polarization curves

Concentration of NE × 10^3 ($g \cdot cm^{-3}$)	Tafel	Weight loss
0	–	–
0.3	65.8	90.83
0.6	70.1	93.66
1.5	83.8	97.0
3	78.6	96.5
6	80.3	97.0

Impedance measurements

EIS is a well-established and powerful tool in the study of corrosion. Surface properties, electrode kinetics and mechanistic information can be obtained from the impedance diagrams (Lorenz and Mansfeld, 1981; Li *et al.*, 2012a, b). The corrosion behaviour of steel, in acidic solution in the absence and presence of NE (until $1.5 \times 10^{-3}\,g \cdot cm^{-3}$ of NE which is the optimum concentration) was investigated by EIS method at E_{corr} (Figure 3(a)). These curves show a single semicircle indicating the occurrence of a single charge transfer reaction. These curves are depressed in nature with their center below the *x*-axis. This observation is due to the origin of micro roughness and other inhomogeneties of the solid/solution interface formed during corrosion (Madhankumar and Rajendran, 2012).

By using Setup_ZSimDemo_320 program, the impedance diagrams can be fitted by a simple equivalent circuit (Figure 3(b)) where R_s represents the solution resistance, R_t is the charge transfer resistance and CPE is the constant phase element of double layer capacitance (C_{dl}).

The C_{dl} was estimated from the impedance value of the frequency (f_m) having maximum imaginary component with Nyquist plot by using following relationship (Bhandari *et al.*, 2010; Ashassi-Sorkhabi *et al.*, 2005, 2008; Dong *et al.*, 2011; Abd El Rehim *et al.*, 2004; Makhloufi et al., 1998; Gabrielli *et al.*, 2007):

$$C_{dl} = \frac{1}{2\pi f_m R_t} \tag{4}$$

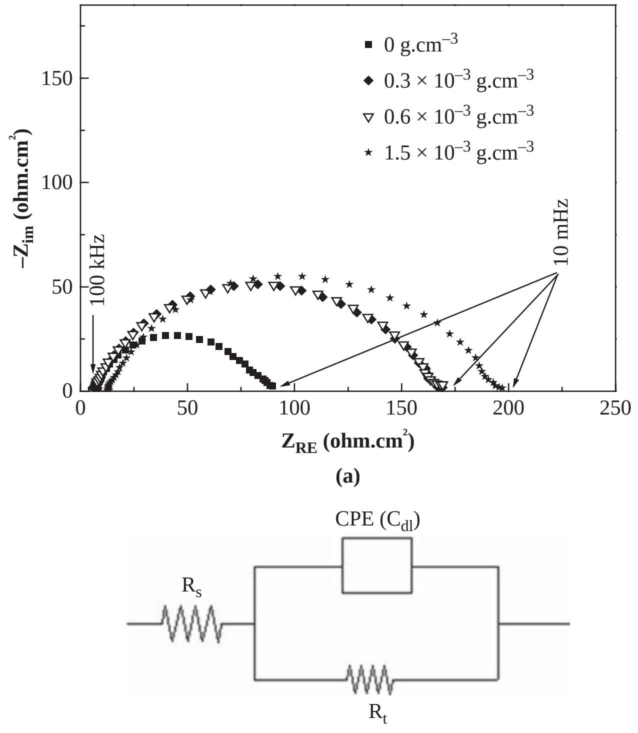
The charge transfer resistance (R_t) values used in equation (4) were given by the Setup_ZSimDemo_320 program by fitting the Nyquist plots.

The impedance parameters mentioned in Table IV indicate that the value of R_t is found to increase by increasing the inhibitor concentration up to a critical concentration, while the value of C_{dl} were found to decrease.

This behaviour could be associated with the adsorption mechanism of the inhibitor at steel/acid interface leading to the formation of an insoluble barrier film (Hazzazi, 2007; Khaled, 2006) which leads to the protection of the metal against corrosion. In these conditions, the variation of R_t and C_{dl} with NE concentrations is shown in Figure 4.

The expression of the double layer capacitance presented in the Helmholtz model is given by equation (5) (Grosser and Gonçalves, 2008; Muralidharan *et al.*, 1995; Li *et al.*, 2012):

Figure 3 (a) Nyquist diagrams of a mild steel electrode in 0.5 M HCl solution with and without different concentrations of NE at E_{corr}, at 25°C and (b) equivalent electrical circuit for modeling the Nyquist diagrams of (a)



(a)

(b)

Notes: R_s – solution resistance, R_t – charge transfer resistance and CPE is the constant phase element of double layer capacitance C_{dl}

Table IV Impedance parameters for mild steel corrosion in 0.5 M HCl without and with NE at different concentrations

Concentration of NE × 10^3 (g·cm^{-3})	R_t (Ω·cm^2)	C_{dl} (mF·cm^{-2})
0	80.3	0.20
0.3	157.2	0.13
0.6	145.5	0.14
1.5	184.5	0.14

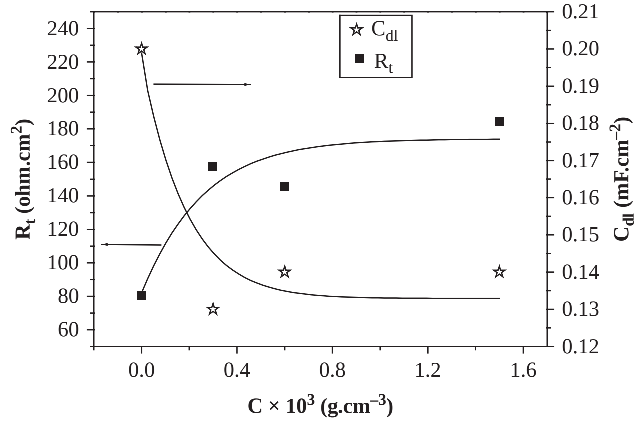
$$C_{dl} = \frac{\varepsilon\varepsilon_0}{d} S \tag{5}$$

where d is the distance between the plates of the capacitor (dual layer), S is the electrode surface, ε_0 is the permittivity of the medium and ε is the dielectric constant.

The decrease in the C_{dl} was caused by the reduction in the local dielectric constant and/or by the increase in the thickness of the electrical double layer. This fact suggests that the inhibitor molecules acted by adsorption at the metal/solution interface (Hazzazi, 2007; Ashassi-Sorkhabi *et al.*, 2005).

The double layer formed at the electrode/solution interface is considered as an electric capacitor, whose capacitance decreases due to the displacement of water molecules and other ions originally adsorbed on the electrode by organic constituents of NE, forming a protective film. The morphology, the texture and the thickness of the formed film seem to change with increasing concentrations of the inhibitor, since more NE adsorbs on the surface, resulting in lower C_{dl} values (de Souza and Spinelli, 2009; Barchiche *et al.*, 2004). Such behaviour and this data interpretation are in good agreement with previous reports on the inhibition of low carbon steel corrosion by succinic acid (Amin *et al.*, 2007).

Figure 4 Variations of the double layer capacitance and resistance transfer as a function of inhibitor concentrations



Note: Values derived from Figure 3(a) by using the Setup_ZSimDemo_320 program by fitting the Nyquist plots

Adsorption isotherms

To determine which adsorption isotherm fits best the surface coverage and to calculate the free-energy of adsorption, the respective plots were obtained. We try to correlate the experimental results with the course of adsorption isotherms deduced from weight loss curves. Thus, several adsorption isotherms (Langmuir, Temkin and Frumkin) were tested to find the suitable adsorption isotherm, according to the following equations (Grosser and Gonçalves, 2008; Bentiss, 2006; Benchikh *et al.*, 2009):

$$Langmuir : \frac{C}{\theta} = \frac{1}{K} + C \tag{6}$$

$$Temkin : \exp(-2a\theta) = KC \tag{7}$$

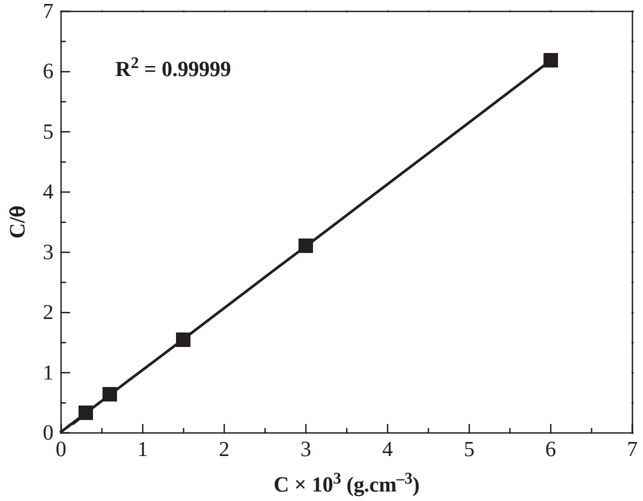
$$Frumkin : \left(\frac{\theta}{1-\theta}\right) \exp(-2a\theta) = KC \tag{8}$$

where θ is the fraction of the surface covered deduced from weight loss curves, a is the adsorbate interaction parameter, K the adsorption-desorption equilibrium constant and C the inhibitor concentration.

The obtained results show that the best fit was obtained for the curve given by the plot of (C/θ) as a function of concentration (Figure 5), the R^2-value obtained was 0.9999; this shows that the inhibitor adsorption on the steel surface in hydrochloric acid obeys the Langmuir adsorption isotherm (Eddy and Ebenso, 2010; Ibrahim *et al.*, 2012). The above other equations (7) and (8) were tested but did not show a good fit (the R^2-values obtained were 0.7331 and 0.8999 for Frumkin and Temkin isotherms, respectively).

The free-energy of adsorption (ΔG^0_{ads}) was calculated from the slope of the Langmuir isotherm, according to the equation (El-Etre, 2006; de Souza and Spinelli, 2009):

Figure 5 Langmuir isotherm for the adsorption of NE on the surface of a mild steel in 0.5 M HCl, at 25°C



$$K = \exp\left(\frac{-\Delta G^0_{ads}}{RT}\right) \quad (9)$$

where K is the equilibrium constant ($L \cdot kg^{-1}$), R the gas constant and T the temperature (°K).

The adsorption-desorption equilibrium constant K was determined as $13{,}612\,L \cdot kg^{-1}$, leading to $\Delta G^0_{ads} = -23.57\,kJ \cdot mol^{-1}$. The negative value of ΔG^0_{ads} reflects a spontaneous process of adsorption of the organic constituents of NE on the surface of the metal. It is well known that the values of ΔG^0_{ads} of the order of $-20\,kJ \cdot mol^{-1}$ or lower indicates a physisorption, while those of $-40\,kJ \cdot mol^{-1}$ or higher involve charge sharing or a transfer from the inhibitor molecules to the metal surface to form a co-ordinate type of bond which indicates chemisorption (Soltani *et al.*, 2012; Negm *et al.*, 2012; Moretti and Guidi, 2002).

Surface analysis

As shown by the above methods of study, a significant efficiency of the steel protections by NE were observed, so, the study was completed by observing the electrode surface by SEM and EDX analysis.

The formation of a protective surface film of inhibitor on the electrode surface was further confirmed by SEM micrographs and EDX analysis. This later was used to determine which elements were present on the electrode surface before and after exposure to the inhibitor solution. Figure 6 shows SEM images recorded for steel samples exposed for seven days in 0.5 M HCl solutions without and with $1.5 \times 10^{-3}\,g \cdot cm^{-3}$ of NE. For comparison, a SEM image of polished steel surface (control sample) is shown in Figure 6(a).

The morphology of the surface exposed to the inhibitor-free solution (Figure 6(b)) shows that the surface is highly damaged in the absence of the inhibitor (the electrode corner is highly attacked). While, in presence of the inhibitor (Figure 6(c)), the electrode surface is almost free from corrosion due probably to the formation of an adsorbed film of the inhibitor on the electrode surface.

The EDX analysis performed on the surface of the control sample (polished surface) (Figure 7(a)) shows the presence of peaks attributed to Fe and C, specific elements of the steel. The spectra of steel sample exposed in the corrosive solution without inhibitor (Figure 7(b)) presents the same peaks as the polished sample with the appearance of another peak of oxygen. The presence of the latter confirms the presence of iron oxide (or iron hydroxide) on the steel surface (corrosion products). In presence of NE, the EDX spectra shows an additional lines characteristic of the existence of C, O, Na, Mg, Si, P, Cl, K and Ca, undoubtedly due to the inhibitor (as shown in Figure 7(c)). In this case, the suppression of the Fe lines occurred because of the overlying inhibitor film (Hazzazi, 2007; Amin *et al.*, 2007, 2009; Abdel Rehim *et al.*, 2008). This result confirms the adsorption observed by electrochemical methods presented previously. The oxygen peak which appears in this case is not assigned to an iron oxide or iron hydroxide, but it is rather assigned to the inhibitor composition as shown in plant extract spectra (Figure 7(d)).

Effect of immersion time

EIS was also used in this part to study the effect of immersion time on the behaviour of the mild steel electrode in the corrosive solution in the presence of the NE at its optimal concentration of $1.5 \times 10^{-3}\,g \cdot cm^{-3}$. The obtained results show that the immersion time had a significant influence on the size and shape of the impedance spectra (Figure 8(a)). When the immersion time was longer than 2 h, the impedance diagrams could be interpreted by using two time constants. The high-frequency (HF) loop, the smaller one, could be attributed to the film formation at the steel surface (adsorption of NE) while the low-frequency (LF) loop, the larger one, could be attributed to the charge transfer reaction (for example and for clarity of the figure, is presented in the insert of Figure 8(a), only the zoom of the HF loop for an immersion time of 8 h) (Lebrini *et al.*, 2011; Amar *et al.*, 2006; Gonçalves *et al.*, 2002). By using Setup_ZSimDemo_320 program, an equivalent electrical circuit was used for modeling the EIS data obtained with the immersion time (Figure 8(b)).

Here, R_s is the solution resistance, R_f and C_f (CPE) are, respectively, the resistance and the capacitance of the adsorbed inhibitor film, R_t is the charge transfer resistance, C_{dl} (CPE) is the double layer capacitance that characterizes the charge separation between metal and electrolyte interface.

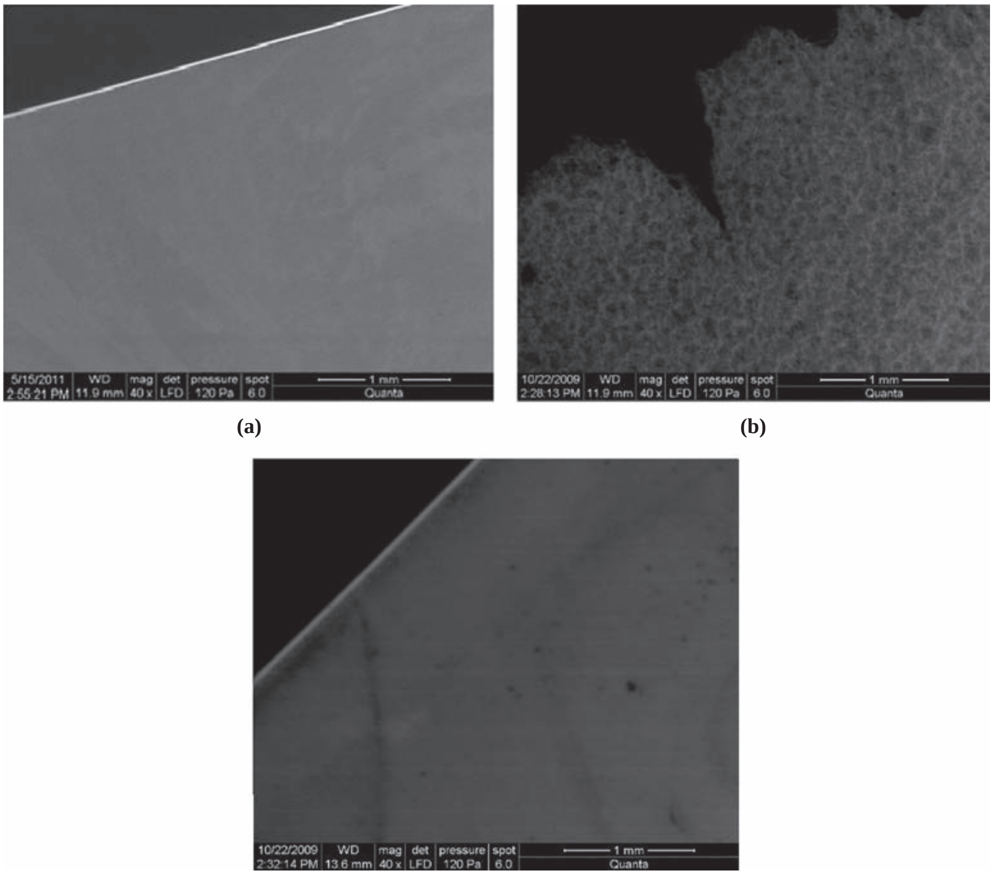
In this case, as given in the literature (Solmaz *et al.* (2008) and the references therein), we consider the change of the polarization resistance R_p (which includes the charge transfer resistance R_t and the film resistance R_f caused by the adsorption of the inhibiting species) as a function of the immersion time instead of the only change of the charge transfer resistance R_t. R_p is given by the following equation:

$$R_p = R_t + R_f \quad (10)$$

Thus, as can be seen in Figure 9, the polarization resistance R_p increased during the initial 180 min of the immersion time and remained fairly constant afterward.

This means that an inhibitory layer was formed by adsorption and the surface was almost covered after a period of 180 min (Amin *et al.*, 2007). These results could be explained by considering that the adsorption process, which takes place on the electrode surface, was strongly dependent on the immersion time. By keeping the inhibitor concentration constant

Figure 6 SEM images of mild steel at 25°C



(a) (b)

(c)

Notes: (a) Before immersion; (b) after seven days of immersion in 0.5M HCl without NE (border attacked by corrosion); (c) after seven days of immersion in 0.5M HCl with 1.5×10^{-3} g· cm^{-3} of NE

and increasing the immersion time, the polarization resistance was enhanced, improving the inhibitive properties.

Effect of temperature

The effect of temperature on the corrosion of steel in free and inhibited 0.5 M HCl solutions was studied using polarization measurements in the range of 20-80°C. The i_{corr} values were obtained by extrapolation of the Tafel lines of experiments carried out at 20, 30, 60 and 80°C, in the absence and the presence of $1.5 \times 10^{-3}\,\mathrm{g \cdot cm^{-3}}$ of NE in the solution (Figure 10). Corresponding data was given in Table V.

The results show that the i_{corr} values increased with the increase of the temperature but are less pronounced in the presence of NE in the solution, indicating that the NE could protect the steel against corrosion at high temperature.

An Arrhenius-type (according to equation (11)) dependence was observed between the corrosion rate and temperature. From this law, the apparent activation energy (E_a) of the corrosion process could be calculated. Some conclusions on the mechanism of the inhibitor action could be obtained by comparing E_a, both in the presence and absence of the corrosion inhibitor:

$$i_{corr} = K\exp\left(\frac{-E_a}{RT}\right) \qquad (11)$$

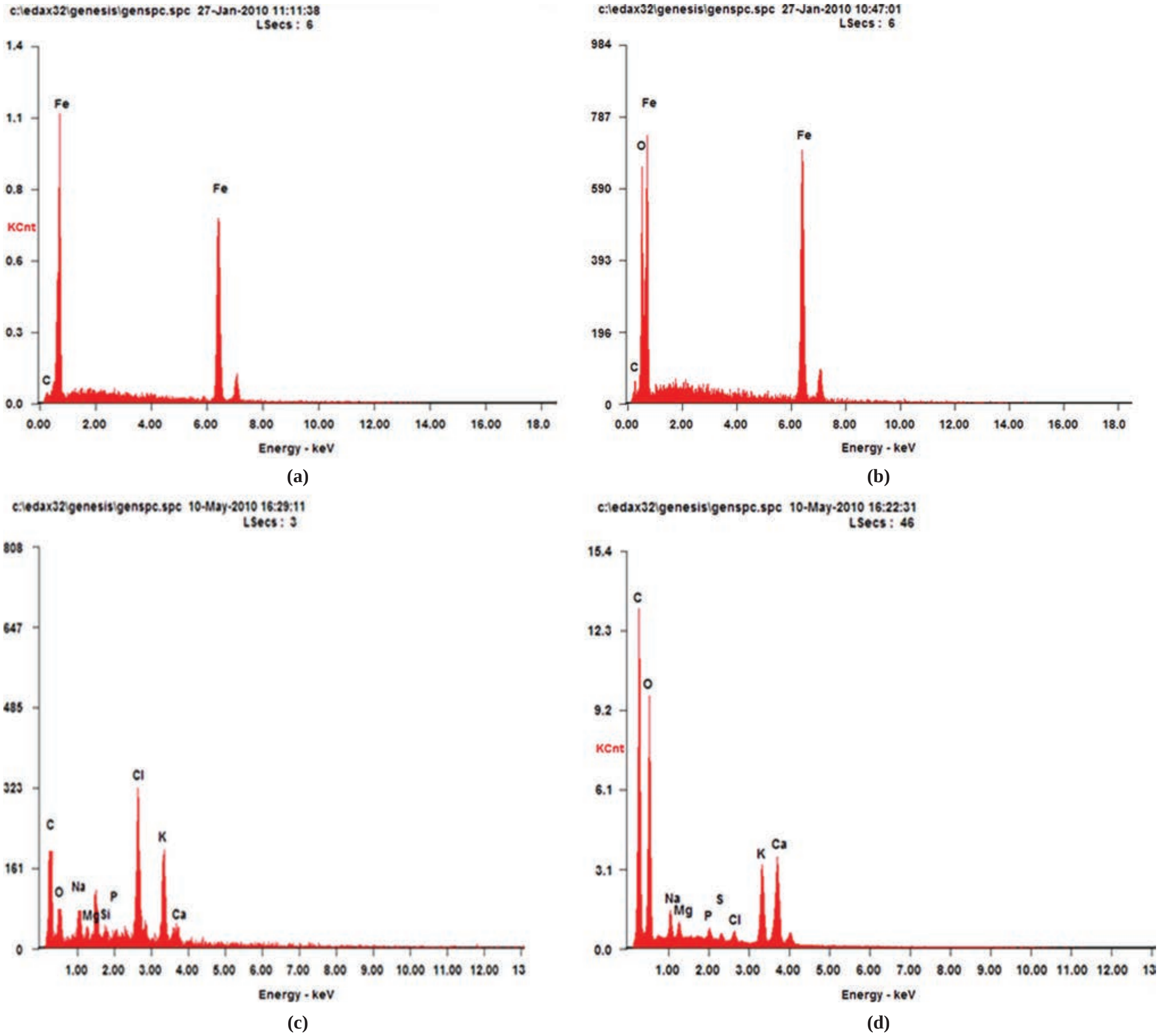
The corresponding Arrhenius plots (plotting the corrosion rate (i_{corr}) vs 1/T) are shown in Figure 11.

The activation energy values (E_a) for the corrosion process in absence and presence of inhibitor determined from the slope of the Arrhenius plots corresponds to 21.8 and 26.9 $\mathrm{kJ \cdot mol^{-1}}$ in the absence and in the presence of NE, respectively. In the literature, the lower activation energy value of corrosion process in the presence of inhibitor (rather than the absence of inhibitor) is attributed to its chemisorption, while it is found to be the opposite in the case of physical adsorption (Larabi *et al.*, 2005). It was found that the energy barrier of the corrosion process increased with the presence of NE. E_a increased in the presence of the inhibitor in 0.5 M HCl suggesting that adsorbed inhibitor molecules created a physical barrier to charge and mass transfer, leading to reduction in corrosion rate (Oguzie, 2006; Ibrahim *et al.*, 2012).

Effect of solution aging on the inhibition efficiency of the extract

The main disadvantage of using plants materials as corrosion inhibitors is their instability with storage time (they are readily biodegradable). The effect of storage time, at ambient temperature, of the stock solution of aqueous nettle extracts on the steel corrosion was investigated by polarization measurements. The Tafel plots obtained for steel in 0.5 M

Figure 7 EDX spectra of mild steel specimens at 25°C



(a) (b) (c) (d)

Notes: (a) Before immersion; (b) after seven days of immersion in 0.5M HCl without NE; (c) after seven days of immersion in 0.5M HCl with 1.5×10^{-3} g· cm^{-3} of NE; (d) of plant extract

HCl solution with two aqueous extracts of nettle (one of them was freshly prepared and the other one stored up to two months) are presented in Figure 12.

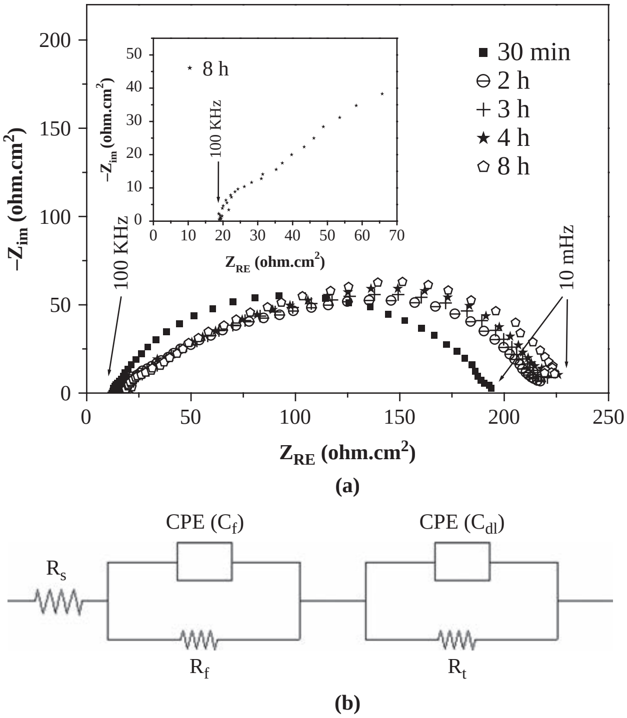
As observed, the plots were unchanged; this suggests that the inhibition mechanism of the extracts was independent on the storage time, so it could be conserved without specific condition of time and temperature. This result was confirmed in the literature for some natural inhibitors (Abdel-Gaber *et al.*, 2008).

Conclusion

From this study, we conclude that aqueous extracts of nettle (NE) are an adsorption inhibitor for the corrosion of mild steel in 0.5 M HCl solutions. The inhibition efficiency increased with increasing inhibitor concentrations till an optimum value of $1.5 \times 10^{-3}\,\mathrm{g \cdot cm^{-3}}$ of NE where a maximum inhibition efficiency value of 97 percent was attained. Polarization measurements showed that NE compounds acted as a mixed inhibitor. The EIS experiments showed that charge transfer resistance (R_t) increased and double layer capacitance (C_{dl}) decreased in the presence of the inhibitor; this indicated the adsorption of the inhibitor at the surface of steel. The adsorption of the inhibitor was spontaneous (reflected by the negative value of ΔG^0_{ads}), supported the mechanism of physical adsorption and obeyed to the Langmuir adsorption isotherm. The inhibition action is independent on the storage time.

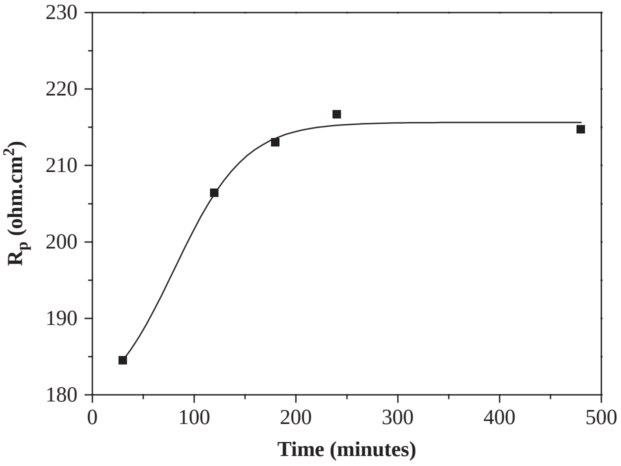
Nettle is a healthy plant, without particular toxicity, as demonstrated by its long history as a food plant, so, its extracts could be used as an inhibitor of mild steel used as materials in food industry.

Figure 8 (a) Nyquist diagrams with the immersion time for mild steel electrode in 0.5 M HCl solution containing $1.5 \times 10^{-3}\,g \cdot cm^{-3}$ of NE, at E_{corr} and at 25°C and (b) equivalent electrical circuit for modeling the Nyquist diagrams of (a)



Notes: For example, and for clarity of the figure, is presented in the insert of (a), only the zoom of the HF loop for an immersion time of 8 h; R_s – solution resistance, R_t – charge transfer resistance and CPE is the constant phase element of double layer capacitance C_{dl}; C_f and R_f are, respectively, the film capacitance and the film resistance caused by the adsorption of the inhibiting species

Figure 9 Variation of R_p with the immersion time for the mild steel electrode in 0.5 M HCl solution containing $1.5 \times 10^{-3}\,g \cdot cm^{-3}$ of NE at 25°C



Note: Values derived from (a) by using the Setup_ZSimDemo _320 program by fitting the Nyquist plots

Figure 10 Effect of temperature on the cathodic and anodic responses for mild steel in 0.5 M HCl solution (a) without and (b) with $1.5 \times 10^{-3}\,g \cdot cm^{-3}$ of NE

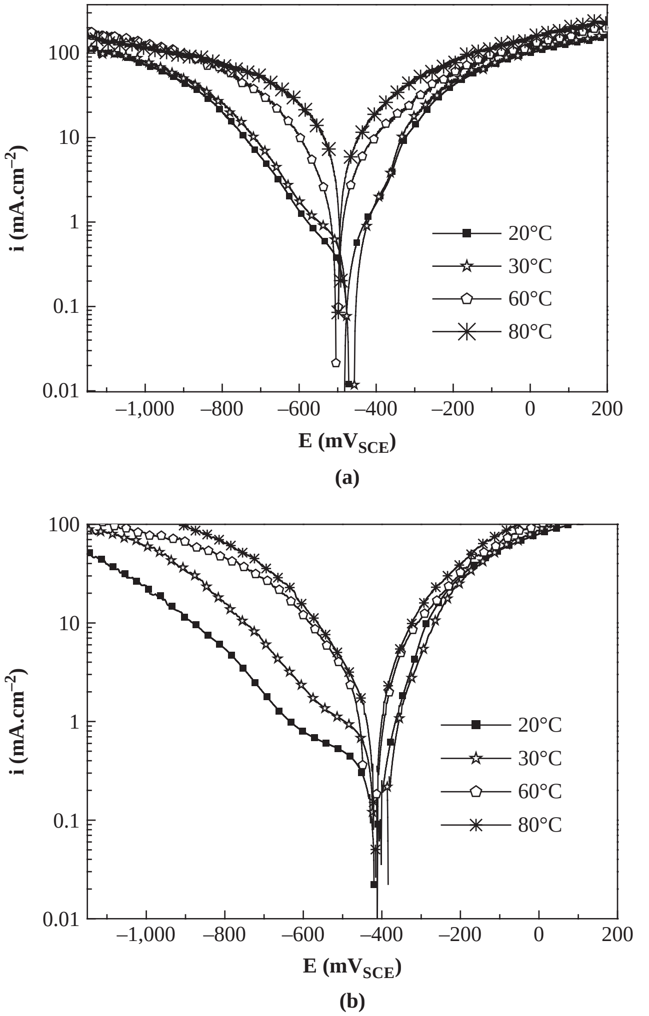


Table V Corrosion rates of mild steel in solution of 0.5 M HCl free and inhibited by $1.5 \times 10^{-3}\,g \cdot cm^{-3}$ of NE at different temperatures, determined by Tafel method extrapolation

Temperatures (°C)	Concentration of NE × 10^3 ($g \cdot cm^{-3}$)	E_{corr} (mV$_{SCE}$)	i_{corr} ($mA \cdot cm^{-2}$)	η%
20	Free	− 477.00	1.17	–
	1.5	− 419.06	0.19	83.8
30	Free	− 467.90	1.58	–
	1.5	− 408.84	0.69	56.3
60	Free	− 501.00	4.56	–
	1.5	− 414.14	1.75	61.6
80	Free	− 493.86	6.42	–
	1.5	− 414.14	1.91	70.2

Note: η% is the corrosion protection efficiency

Figure 11 Relation between corrosion rate and reciprocal of temperature for blank and $1.5 \times 10^{-3}\,g \cdot cm^{-3}$ of NE solution

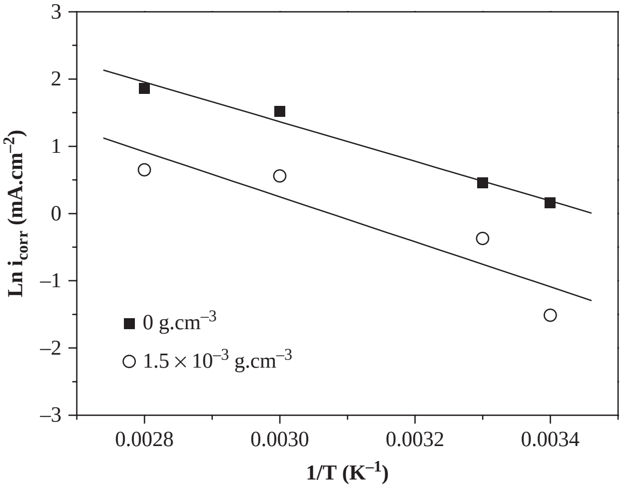
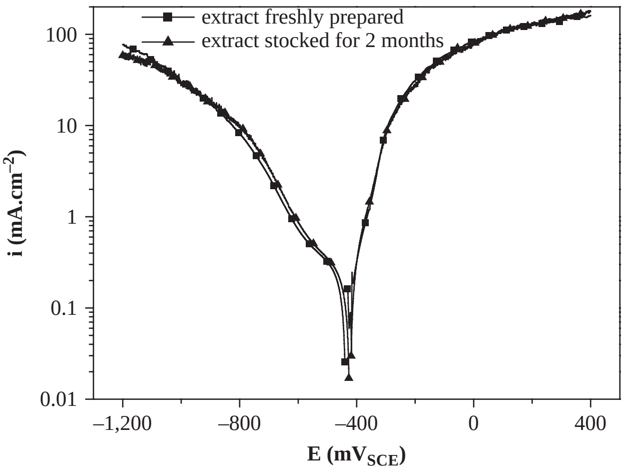


Figure 12 Polarization curves of mild steel in 0.5 M HCl in the presence of $1.5 \times 10^{-3}\,g \cdot cm^{-3}$ nettle extract at different storage times



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