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Corrosion Inhibition Performance of Rosmarinus officinalis Methanolic Extract on Carbon Steel XC48 in Acidic Medium (2M HCl)

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ABSTRACT

The inhibition ability of the methanolic extract of *Rosmarinus officinalis* on the corrosion of mild steel (XC48), in HCl solution (2M), was investigated at various temperatures (298–328 K) via weight loss measurements, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS). The results showed that the inhibition efficiency increased with the increase of the extract concentration to reach a maximum of 89,18% at 200 ppm. A further augmentation of the extract concentration led to some deterioration of the inhibition efficiency which dropped to 77,18 % at 400 ppm. This high efficacity may be attributed to the production of a protective layer on the surface of the electrode which is confirmed by the scanning electron microscopy (SEM) images. The investigation of the temperature effect on the inhibition efficiency and the determination of the different thermodynamic parameters, allowed us to deduce a physisorption mechanism of *Rosmarinus officinalis* extract on the surface of the metal. On the other hand, the results of the potentiometric polarization tests revealed that the extract components are acting as a mixed-type inhibitor.

Introduction

Acid solutions are generally used for the removal of undesirable scale and rust in several industrial processes. Hydrochloric acid is widely used in the pickling processes of metal. Mild steel is widely used in the manufacturing of installations for the petroleum, fertilizers, and other industries. However, the main drawback of mild steel is its suffering from corrosion under severe environments [1]. As a result, the need for the corrosion inhibitors becomes increasingly necessary to stop or to delay to the maximum the attack of a metal in an aggressive solution. The use of inhibitors is found to be one of the most practical methods for protection against corrosion, especially in acidic media [2]. The use of inhibitors is one of the most practical methods for protection against corrosion, especially in acidic media [3]. Among numerous inhibitors that have been tested and applied industrially as corrosion inhibitors, those that are non-toxic or low-toxic are now far more strategic than in the recent past [4]. In the 21st century, the research in the field of ''green'' or ''eco-friendly'' corrosion inhibitors has been addressed toward the goal of using cheap, effective compounds at low or ''zero'' environmental impact. In this context, many researchers are focusing on the development of environment-friendly corrosion inhibitors such as natural products [5-9].

This research aims to study the inhibiting action and adsorption properties of *Rosmarinus officinalis* extract, at various temperatures (298–328 K), on the surface of carbon steel in two molars (2M) hydrochloric acid solution. The techniques employed to evaluate the performance of the active compounds are: weight loss, electrochemical impedance spectroscopy, potentiodynamic polarization and scanning electron microscopy.

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Experimental

Specimens Preparation

Tests were performed on XC48 steel specimen cut into a rectangular shape of 2 × 0.9 cm^2 for weight loss test and

1×1 cm^2 for electrochemical tests, as the working electrode (WE). The samples were prepared in inert resin and polished with different grades of emery papers from 400-2500 then cleaned with double distilled water, degreased with acetone and dried.

Inhibitor extraction

Fresh leaves of *Rosmarinus officinalis* were collected from the region of Bordj Bou Arreridj (east of Algeria). They were washed with clean water and dried at 313 K then ground to powder. Thirty grams of dried powder was weighed and extracted with 300 ml of a mixture of methanol/water (8/2 v/v) at ambient temperature with stirring for 24 hours in dark. The extract was then filtered, dried and conserved at 4 °C until use.

Solutions preparation

The aggressive medium used was 2M HCl prepared from 37% analytical grade and distilled water .the concentration range of *Rosmarinus officinalis* extract was 50-400 ppm.

Weight loss measurement

Cleaned, dried and accurately weighted carbon steel specimens were immersed in 2 M HCl without and with the optimum concentration of 200 ppm of *Rosmarinus officinalis* extract. The effect of the addition of the extract was investigated for different immersion times (5-168 h) at various temperatures (298-328K). To ensure the reproducibility of the weight loss results, each experiment was performed in triplicate and means values are used. The results of weight loss measurements were used to determine the inhibition efficiency and corrosion rates (Wcorr) by applying the equation (1) [10].

$$W_{corr} = \frac{\Delta m}{S \times t} \quad (1)$$

Where Δm is the weight loss (mg), t is the immersion time (h) and S the surface (cm^2).
From the weight loss values obtained the inhibition efficiency and the degree of coverage θ were calculated using the following equations 2 and 3 [11, 12].

$$IE\% = \frac{W_{balnk} - W_{inh}}{W_{blank}} \quad (2)$$

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

Where W_{blank} and W_{inh} are the rate of corrosion without and with the inhibitor respectively.

Electrochemical measurement

All Electrochemical experiments were carried out in a potentiostat-galvanostat Versastat4, controlled by a versastudio software connected to three-electrode.Test coupons with 1 cm^2 exposes areas were used as working electrode XC48, the Platinum electrode as counter electrode and the Ag/AgCl was used as the reference electrode . Before each experiment, the working electrode was immersed in test solution for 60 min at 297 K to attain a stable open circuit potential (OCP) [13] the Potentiodynamic polarization (PDP) measurements were carried by scanning the potential range between -0.25 V and + 0.25 V with respect to the OCP at a scanning rate of 1 mV s^{-1}. The corrosion potential (Ecorr) and corrosion current density (icorr) were acquired by extrapolated the linear Tafel segments of the cathodic curves and anodic Tafel lines to the intersection point, The values of anodic Tafel slope (βa) and cathodic Tafel slope (βc) were determined from the theoretically calculated straight line and experimental line, respectively. The inhibition efficiency IE% of *Rosmarinus officinalis* extract was calculated from the PDP investigation using equation (4) [14].

$$IE\% = \frac{I^{\circ}_{corr} - I_{corr}}{I^{\circ}_{corr}} \quad (4)$$

Where, I_{corr} and I_{corr}^0 are the corrosion density currents with and without the inhibitor

Surface analysis

The surface morphology images of the XC48 steel were performed on specimens exposed to 2 M HCl solutions without and with *Rosmarinus officinalis* extract for 24 h of immersion time at the temperatures 298K and 328K.

Table 1. Weight loss parameters for corrosion of XC48 steel in 2M HCl ,with optimum inhibitor of 200 ppm of *Rosmarinus officinalis* extract at 298 K

Concentration	Blank		200 ppm		
Immersion time (h)	(g.cm^{-2}.h^{-1})	×10^{-2} (g.cm^{-2}.h^{-1})	Δm (g)	×10^{-2} (g.cm^{-2}.h^{-1})	IE%
5	0.18694	3.7388	0.0159	3.18	91.49
24	0.33734	1.405	0.10665	4.443	68.38
72	0.7999	1.111	0.25694	3.568	67.88
168	1.0359	14.7985	0.4555	65.071	56.03

Results and discussions

Weight loss measurement

Immersion time Effect

The adsorption ability of the inhibitor on the surface of the XC 48 steel followed by the weight loss measurements was carried out using the optimum concentration (200 ppm) of the extract, at 298 K and at different immersion times (5–168 h). The various parameters such as IE% ,Wcorr and Δ m are listed in Table 1.
Fig.1 shows that the weight loss of the uninhibited XC 48 steel increases rapidly with time in the corrosive solution, however the presence of the extract causes a reduction in the weight loss. Furthermore, Table 1 illustrates that the extract can retard the corrosion of the metal but the inhibition efficiency decreases with the increase of the exposure time. These results can be explained by the formation of a protective adherent film on the metal surface and suggests that the coverage of the metal surface with this film decreases the double layer thickness [15].

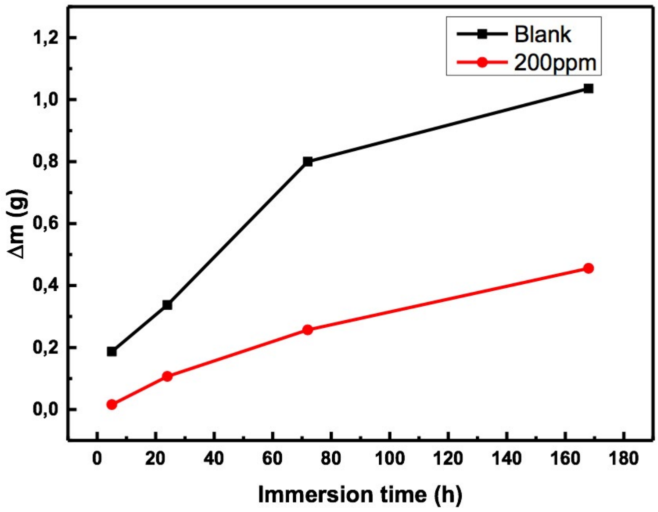


Fig. 1. Weight loss for corrosion of XC48 steel in 2M HCl, with optimum inhibitor concentration of 200 ppm of *Rosmarinus officinalis* extract at different times.

Temperature effect

In order to study the effect of temperature on the corrosion inhibition property of *Rosmarinus officinalis* extract, weight loss measurements were conducted, during 24 h of immersion, in the temperature range of 298–328 K, in the absence and presence of 200ppm of inhibitor. The parameters of the obtained data, listed in Table 2, clearly show an increase in the corrosion rate (W_{corr}) with the rise of temperature.
We also note that the inhibition efficiency (Figure 2) depends on the temperature and decreases with the rise of temperature from 298 to 328 K. This is due to the shift of the adsorption/desorption equilibrium towards the *Rosmarinus officinalis* extract desorption and hence to the decrease of the degree of surface coverage [16].

The relationship between corrosion rate W_{corr} and temperature T can be expressed in the Arrhenius equation (Eq. 5) [17].

$$W_{corr} = A \exp(-\frac{E_a}{RT}) \quad (5)$$

where E_a is the apparent effective activation energy, T is the absolute temperature , R is the general gas constant and A is the Arrhenius pre-exponential factor .
The values of apparent activation energy E_a for corrosion process in the presence and absence of inhibitor solution ,obtained by linear regression of Ln W_{corr} vs 1000/T shows in Fig. 3 and Table 3.

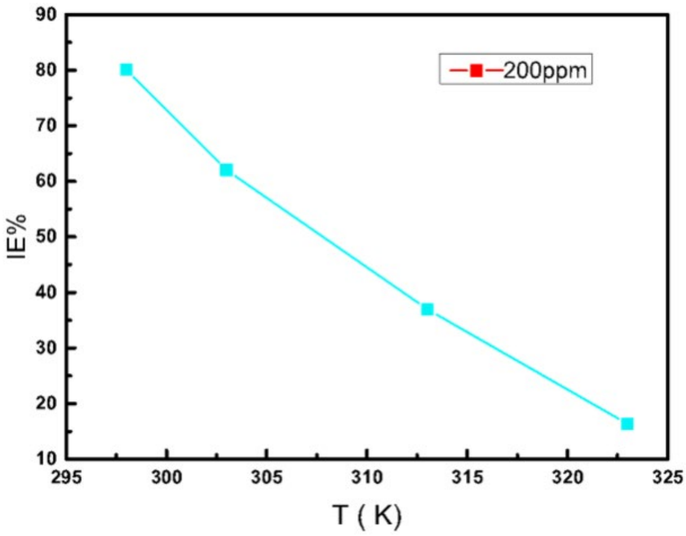


Fig. 2. Inhibition efficiency for corrosion of XC48 steel in 2M HCl, with an optimum inhibitor concentration of 200 ppm of *Rosmarinus officinalis* extract at different temperatures.

The value of E_a in the presence of inhibitor is greater than that in absence of inhibitor which deviously signalize that in presence of *Rosmarinus officinalis* extract additional energy barrier warried out for XC48 steel corrosion due to forming protective film that diminution the corrosion rate [18].

Table 2. Weight loss parameters for corrosion of XC48 steel in 2M HCl ,with optimum inhibitor of 200 ppm of *Rosmarinus officinalis* extract at different temperature.

Concentration	Blank	200 ppm	
Temperature (K)	$\times10^{-3}$ (g.cm^{-2}.h^{-1})	$\times10^{-3}$ (g.cm^{-2}.h^{-1})	IE%
298	0.749	0.221	70.50
308	2.52	1.10	56.35
318	5.37	3.60	32.96
328	8.79	7.36	16.27

Table 3. Thermodynamic parameters for mild steel in presence of *Rosmarinus officinalis* extract in 2 M HCl.

C	E_a (KJ.mol^{-1})	- ΔH (J.mol^{-1})	- ΔS (J.mol^{-1}.K)
Blank	81.075	323.58	196.65
200 ppm	107.74	410.29	196.39

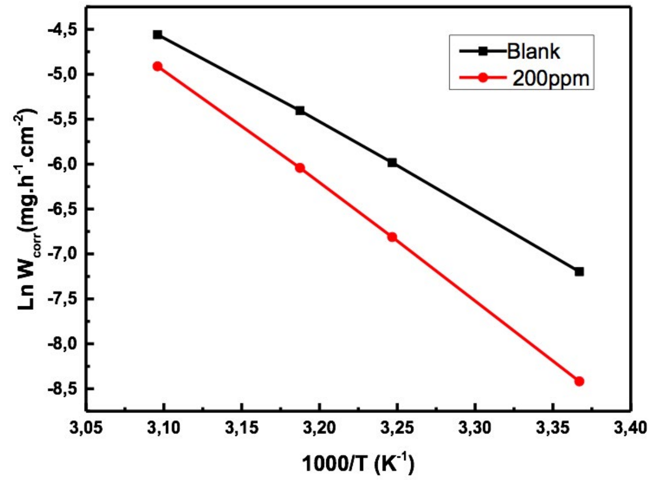


Fig. 3. Arrhenius plots of Ln W_{corr} versus 1000/T for XC48 in 2 M HCl in the absence and presence of *Rosmarinus officinalis* extract at at different temperatures .

An alternative formulation of Arrhenius equation is [19]:

$$W_{corr} = \frac{RT}{Nh} \exp\left(\frac{\Delta S_a}{R}\right) exp - \left(\frac{\Delta H_a}{RT}\right) \quad (6)$$

Where *h* is the plank's constant, N is Avogadro's number, ΔS_a the entropy of activation and ΔH_a is the enthalpy of activation. Fig. 4 illustrates a plot of Ln W_{corr}/T against (1/T) plot from which values ΔH_a and ΔS_a are computed and reported in Table 3. The negative values of ΔH_a indicates that the adsorption phenomenon is exothermic. Besides, the change of values of entropies (ΔS_a) imply that the activated complex represented the rate-determining step for the association rather than the dissociation step [20].

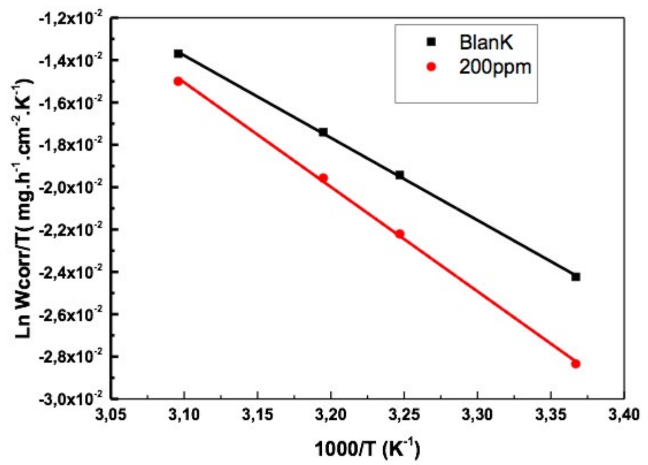


Fig. 4. Transition-state plots corrosion rate (W_{corr}) in acidic media in absence and presence of *Rosmarinus officinalis* extract at different temperatures.

Potentiodynamic polarization measurements

The polarization curves of carbon steel in 2 M HCl solution in the absence and presence of Inhibitors at 297K are shown in Fig. 5 .

The electrochemical parameters such as E_{corr} , I_{corr}, β_a , $\beta_a$and IE% obtained by extrapolations of the Tafel lines were given in Table 4.

From Fig. 5,It can be observed,that the addition of inhibitor causes a decrease of the current density as well as a slight modification of the cathodic Tafel slope constant (β_c) and the anodic Tafel slope (β_a). in the Table 4 it is evident that the introduction of the *Rosmarinus officinalis* extact into 2M HCl solution leads to small displacement in the corrosion potential between 2and 47 mV in comparison to the blank solution. These values are less than the value of Ecorr shift of ±85 mV generally taken as a benchmark for an inhibitor to be classified as anodic or cathodic, [21] hence *Rosmarinus officinalis* extact could be considered to function as a mixed-type inhibitor.

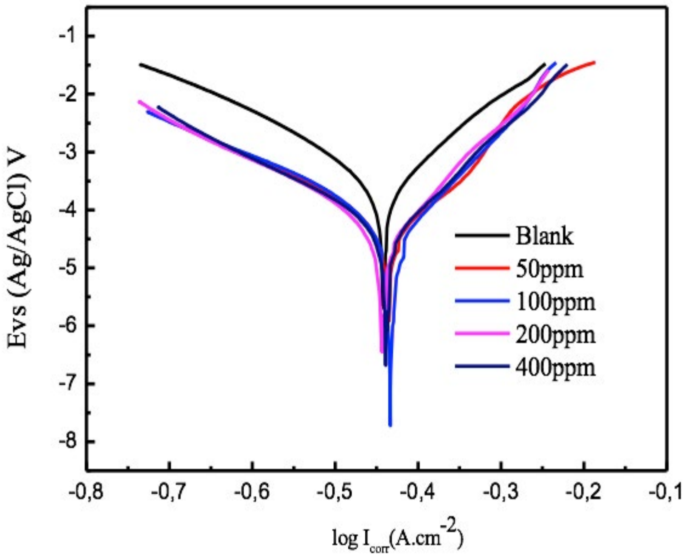


Fig. 5. Polarization curves for XC48 steel in 2M HCl in the absence or presence of different concentrations of *Rosmarinus officinalis* extract at 298K.

The current density I_{corr} value decreases with the increase in *Rosmarinus officinalis* extact concentration until 200 ppm, then I_{corr} reincreases again ,which can be due the surface becomes slightly bare and vulnerable to corrosion attack as will be discussed,This phenomenon causes that the inhibitor efficiency increase untile 75% at 200 ppm, confirming that the oxidation-reduction reactions are been affected due to the block of the active sites [22].

Electrochemical impedance spectroscopy (EIS)

The corrosion inhibition efficiency of *Rosmarinus officinalis* extract on carbon steel was also investigated by electrochemical impedance spectroscopy (EIS). The spectra obtained for carbon steel in 2 M HCl in the absence and presence of different concentrations of the plant extract at the open circuit potential are displayed in Fig. 6 as Nyquist plots. All diagrams exhibited single semicircles with diameters proportional to the concentration of the inhibitor. This may explain the fact that there are some active molecules adsorbed on the carbon steel surface forming a protective film. Considering the electrochemical impedance plots, it can be concluded that there is only one occurring phenomenon which may be explained by the charge transfer process between the XC48 steel/ solution. Furthermore, the single semi-circle presented in the Nyquist diagrams of all solutions proves that the addition of plant extract does not change the dissolution mechanism of the XC48 steel but the resistance of the carbon steel increased compared with the blank solution[23].

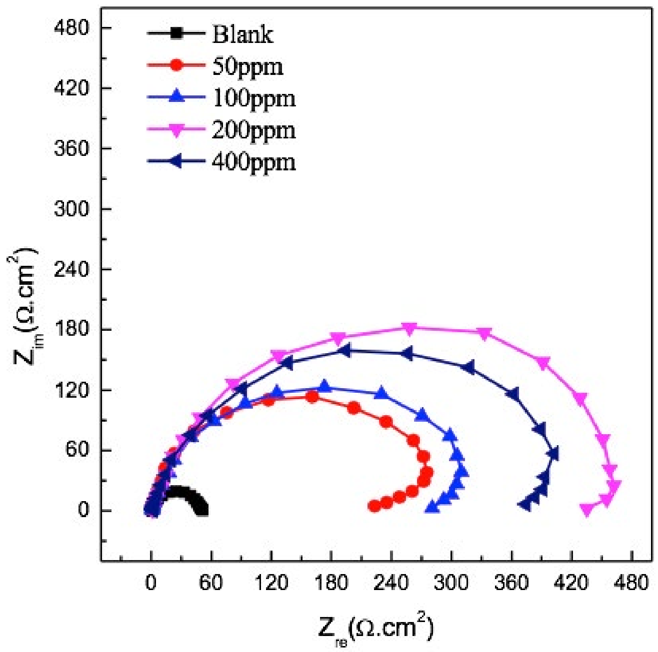


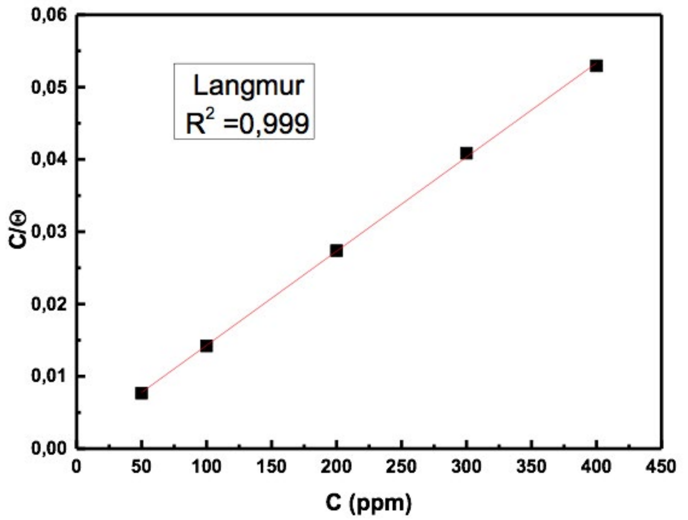
Fig. 6. Nyquist plot of carbon steel in 2 M HCl in the absence and presence of different concentrations of *Rosmarinus officinalis* extract at 298K .

Adsorption isotherm models

The type of interactions between *Rosmarinus officinalis* extact molecules and metal surface can be evaluated by different isotherms: Langmuir, Temkin, Frumkin and Flory–Huggins. The best fit isotherm was determined from the values of the linear correlation coefficient (R^2) where Langmuir adsorption isotherm was found to be the most suitable. According to this isotherm, θ is related to the inhibitor concentration by the relation 7 [24].

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (7)$$

Where, K ads is the equilibrium constant of the adsorption process, C_{inh} is the inhibitor concentration and θ the degree of surface coverage In our case the Langmuir isotherm was fitted with a linear relation of $R^2 > 0.99$ (Fig. 7). These observations disclose that the adsorption isotherm obeyed Langmuir isotherm [25].



Fir. 7. Langmuir isotherm adsorption model of *Rosmarinus officinalis* extract on the mild steel surface in 2 M HCl at 298 K.

Also, from the K_{ads} calculated from the intercept of the straight, the adsorption$\Delta G°_{ads}$ may be obtained according to equation [26]:

Table 4. Polarization parameters and inhibition efficiency for the corrosion of CX48 steel in 2M HCl with or without different concentrations of *Rosmarinus officinalis* extract at 297K.

C (ppm)	$E_{corr/Ag/AgCl}$ mV	I_{corr} (μA/cm²)	(mV)	(mV)	R_p (ohm.cm²)	E% (I)	E% R
Blanc	-441.93	113.052	68.493	57.226	80.242		
50	-436.248	39.265	95.442	91.971	383.463	65.2682	79.07438
100	-443.657	17.315	50.532	48.923	352.138	75.13711	84.2492
200	-443.5	12.227	52.982	47.779	557.382	89.18462	85.60377
400	-488.779	25.791	94.755	68.491	438.739	77.1866	81.71077

$$\Delta G^{\circ}_{ads} = -RT\ln(55{,}5K_{ads}) \quad (8)$$

Where, R is the universal gaz constant, T is absolute temperature and 55.5 is molar concentration of water in the bulk solution . the values of ΔG°_{ads} at 298K is -12.91 KJ.mol^{-1}. Generally ΔG°_{ads} values of around -20 KJ.mol^{-1} or less negative the adsorption is regarded as the physisorption , those around -40 KJ.mol^{-1} or higher ,the adsorption is regarded as the Chimisorption [27]. In the present study the values of ΔG°_{ads} suggesting that the adsorption of *Rosmarinus officinalis* extract at XC48 steel surface is physisorption.

Surface morphology analysis

The surface of the metal immersed during 24 hours in 2 M HCl without and with 200 ppm of *Rosmarinus officinalis* extract was characterized by SEM and the profiles obtained are given in Fig. 8. The surface of XC48 steel in the polished state (before immersion) is represented in Fig. 8(a), while Fig. 8(b) and (c) show SEM images for the exposed steel in the acidic medium without and with 200 ppm extract, respectively. The corresponding EDS analysis, displayed in table 5, of samples immersed in the blank solution, revealed that the presence of significant amounts of Fe, O and Cl, is probably due to the formation of Fe_3O_4 and $FeCl_3$ [28]. The presence

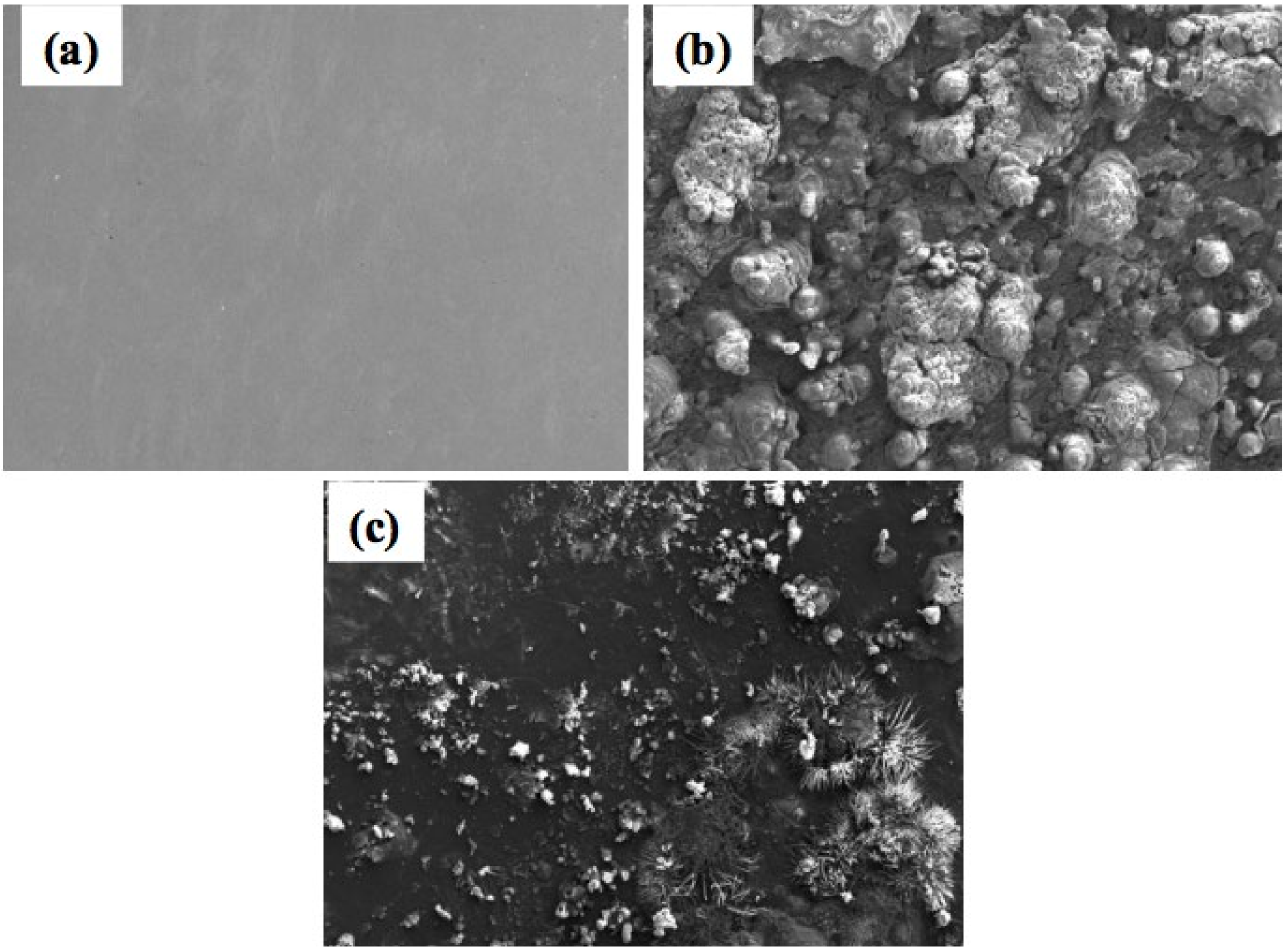


Fig. 8. SEM images: before the experiment (Fig.8a) and after 24h of immersion in the corrosive solution without (Fig.8b) and with (Fig.8c) the Rosmarinus officinalis extract

Table 5. Quantitative analysis for steel surface after 24 hours immersion in 2 M HCl in presence and absence of the *Rosmarinus officinalis* extract from EDX measurements

Element	Fe	C	O	Si	Cl	Ca	Mn
Bare CX48 Steel	94.26	4.8	-	0.030	-	0.59	0.25
Blank HCl 2M	50,15	2,13	35.84	0.01	11.16	0.59	0.12
Blank+200 ppm Rosmarinus officinalis extract	58.75	2.04	27,71	-	5.21	0.58	5.71

of other elements such as C, Ca, Si and Mn was related to the composition of the carbon steel sample. In the blank solution, the percentage of Fe, C, Si and Mn decreased compared to that of XC48 carbon steel rods, indicating that the carbon steel specimen lost some percentage of its constituent elements to corrosion in the corrosive environment.

Conclusion

Weight loss measurements and electrochemical investigations showed that the methanolic extract of *Rosmarinus officinalis* was an effective inhibitor against XC48 steel corrosion in HCl medium. The active molecules in the extract acted as a mixed inhibitor, adsorbing on the steel surface according to the Langmuir isotherm model. The calculated thermodynamic parameters suggested that the adsorption process is spontaneous and exothermic and the inhibitor molecules are adsorbed on the metal surface through physical adsorption. The surface analysis technique supported electrochemical results and confirmed the adsorption of the active components of the plant extract on the surface of the XC48 Steel.

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Conflicts of interest

Authors declare no conflict of interests.

Notes

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