



Corrosion inhibition of XC48 steel by *Grewia bicolor* jus leaves extract in 1M HCL acid medium: electrochemical and gravimetric study



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Mohamed Ould Sidine¹, Hana Lahbib², Mohamedou Ba³, Ahmedou Mohamed Vadel Salihi⁴,
Mohammed Benmessaoud⁵, Yaser Ben Amor² and Brahim Ould Elemine^{1*}

¹Unité de Chimie Moléculaire et Environnement, Département de Chimie, FST, UN, Nouakchott 880, Mauritanie.

²Research Laboratory of Environmental Sciences & Technologies, Higher Institute of Environmental Sciences and Technology, Carthage University, Ben Arous 2050, Tunisia.

³Unité de Recherche : Electrochimie, matériaux et environnement (UREME), UR17ES45, Faculté des Sciences de Gabès, Cité Erriadh, 6072 Gabes, Tunisia

⁴Unité de Biodiversité et Valorisation des Ressources Végétales, Faculté des Sciences et Techniques, Université de Nouakchott, Mauritanie

⁵Energy, Materials and Sustainable Development Team CERNE2D, Higher School of Technology Salé, Mohammed V University in Rabat 8007, Morocco

Abstract

The inhibitory effect of the methanol extract of *Grewia bicolor juss* (GB) leaves against XC48 mild steel corrosion, in hydrochloric acid solution (1M) medium, was investigated by potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS) methods for different concentrations at 298 K and weight loss measurements with optimum concentration at different temperatures (298-333 K) and time immersion. The results demonstrated that the components of the extract took action mainly as a mixed-type inhibitor, and the inhibition efficiency values escalated with the progression of extract concentration. The maximum value obtained is about 93% at a concentration of 1500 ppm. This high efficiency can be attributed to the installation of a protecting blanket on the electrode surface. The adsorption of the inhibitor molecules on adsorbate surface obeyed Langmuir isotherm model. The thermodynamic parameter values suggested that physisorption and chemisorption constituted the major contribution to the process of adsorption. Results obtained from weight loss measurements show that the inhibition efficiency decreases with increasing immersion time and temperature. Finally, scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analyses confirmed obtained results, which reveal that adsorbed inhibitor molecules hinder HCl attacks at the steel grain boundaries.

Keywords: Corrosion, *Grewia bicolor juss*. extract, Carbon steel, HCl

1. Introduction

Corrosion designates the alteration of a material by chemical or electrochemical reaction with its environment. Few metals are found in their native state in nature. Metals tend, in the presence of certain environments, to return to the oxidized form which is their thermodynamically stable form [1]. Some manufacturing operations and other industrial procedures largely exploit the strong acids such as nitric, sulfuric, hydrochloric, formic and acetic acids [2-4]. The majority of metals are exposed to oxidation phenomena when in contact with the atmosphere, liquids or other metals [5,6]. The kinetics of the

corrosion can be significant and endanger the metal, so it is necessary in this situation to imagine processes to preserve the metal. The use of corrosion inhibitors is a very effective way to fight against the corrosion of materials. The most frequently used in acidic environments are organic molecules. In general, the capacity of a substance to inhibit the corrosion of metals is essentially based on the existence of heteroatoms (O, N, S...), its structure, its electronic properties, the aromaticity and the nature of the donor electrons π orbital of the donor electrons [7-9]. The most recently used inhibitors are likely to be a source of environmental degradation. For this reason, scientists considered the search for new green

*Corresponding author e-mail: ouldeleminebrahim2009@gmail.com; (Brahim Ould Elemine).

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inhibitors that showed strong inhibitory performance as corrosion inhibitors [10–12]. In this context Several studies on the corrosion-inhibiting performance of plant extracts have been focused on mild steel and low-alloy protection under acid conditions among them: The influence of Roselle (*Hibiscus Sabdariffa*) Leaf extracts on Cu-Zn alloy in 1M HNO₃ solution was studied by Seham Shahan et al. [13]. In another work, the inhibition effect of *Chamaemelum Nobile* extract on corrosion of mild steel electrode in 1 M hydrochloric acid was studied using gravimetric analysis and electrochemical methods, by Ashraf M. Ashmawy et al. [14]. Hydro-Alcoholic extract of Coffee Grounds was investigated as an inhibitor of corrosion phenomena affecting C38 Steel in 1M Hydrochloric by adopting Electrochemical and Thermodynamic Investigation Fatima Bouhlal et al. [15,16]. As a part of our ongoing work to find new green inhibitors for Steel XC48 in acidic medium, we previously reported the inhibitive effect of methanol extract of *Grewia bicolor Juss.* leaves on the corrosion of XC48 steel in H₂SO₄ solution [17]. In continuation of our efforts to improve the quality and efficiency of this extract as a green inhibitor, we describe in the present investigation its effect against XC48 mild steel corrosion, in hydrochloric acid solution (1M) medium. In this study, the corrosion resistance of XC48 steel was evaluated using potentiodynamic measurements and electrochemical impedance spectroscopy.



FIGURE 1 : *Grewia bicolor juss* (GB) Leaves

2. Material and methods

2.1. Inhibition extract

The leaves of *Grewia bicolor juss* Figure 1., used in this work, were collected from the Kiffa, town located in the south of Mauritania, about 600 km from the capital Nouakchott in January 2021. The extraction method used was Soxhlet process. 30 g of GB leaves powder extracted with 200 ml of methanol in Soxhlet

apparatus for 8 h. Then, it was filtered by filter paper to get the GB leaves extract [17]. Finally, the extract was concentrated by rotary evaporator at 40°C to obtain a viscous semi solid mass.

2.2. Solutions preparation

The Corrosion solution was prepared from analytical grade of the acid (37% HCl) with distilled water. The inhibitor concentration of GB leaves was 400-1500 ppm.

2.3. Electrochemical measurement

All electrochemical experiments were performed using a VersaSTAT monitored by Versa Studio software, connected to three-electrode cell system, including platinum as an auxiliary electrode, an Ag/AgCl reference electrode, and a working electrode (XC48 steel). Before each test, the working electrode undergoes a surface polishing with abrasive paper of successive grades 400 to 2000. Then, it was rinsed with the acetone and distilled water and dried at room temperature before introducing into the measuring cell. To test the polarization, the scanning rate has been applied at 0.5 mV/s and the potential range was ± 250 mV from E_{corr} . The time limit to reach the steady-state potential before performing an electrochemical measurement was 60 minutes. Electrochemical Impedance Spectroscopy (EIS) measurements were carried out using signal amplitude of 10 mV over a frequency range of 100 kHz to 0.1 Hz with 10 points per decade. The inhibition efficiency was calculated through the data obtained from polarization curves, and impedance diagram using the following equations (1) and (2) respectively [18,19].

$$IE (\%) = \frac{I_{corr}^{\circ} - I_{corr}}{I_{corr}^{\circ}} \times 100 \quad (1)$$

$$IE (\%) = \frac{R_t^{inh} - R_t^{\circ}}{R_t^{inh}} \times 100 \quad (2)$$

Where, I_{corr} and I_{corr}° are the corrosion density currents with and without the inhibitor respectively, R_t^{inh} is the charge transfer resistance in the inhibited solution and R_t° represents the charge transfer in the corrosion medium.

3. Results and discussions

3.1. Electrochemical analysis

3.1.1 Open circuit corrosion potential

The variation in open circuit potential (E_{ocp}) in 1 M hydrochloric solution with or without the presence of

GB leaf extract at various concentrations is given in Fig. 2. The data in this figure exhibits that after the

TABLE 1
The corrosion parameters obtained by fitting of

C(ppm)	E_{corr} (mV/ESC)	I_{corr} ($\mu\text{A}\cdot\text{cm}^{-2}$)	β_c (mV/ESC)	β_a (mV/ESC)	%IE
Blank	-480	547.63	-112.25	114.11	
400	-460	76.12	-123.81	54.91	86.10
800	-455	66.47	-137.05	57.16	87.86
1000	-440	53.89	138.81	65.63	90.16
1500	-431	38.77	135.11	86.76	92.92

addition of the GB leaf extract, the OCP values become more positive due to the adsorption of the inhibitor on the surface of XC48 steel working electrode [20]. These additives may be act as an anodic, cathodic or mixed inhibitor. This could be determined by measuring the gap between the OCP values of the inhibited and uninhibited solutions [21]. The magnitude of this shift until ± 56 mV for the GB leaf extract compared to that of the 1M hydrochloric solution puts forward a mixed type inhibitor [22].

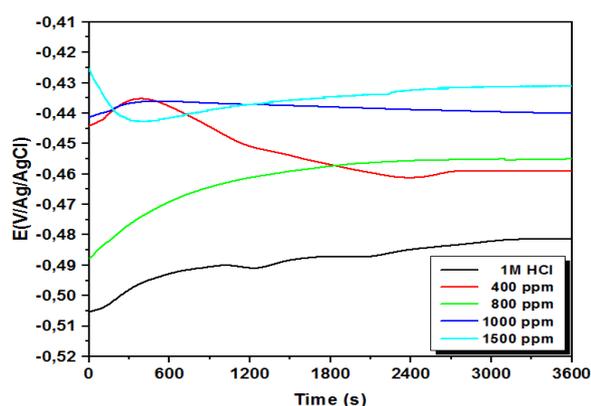


FIGURE 2: Open circuit potential versus immersion time for XC48 steel in 1 M HCl without and with GB leaves extract at different concentrations.

3.1.2. Polarization studies

The curves of polarization for XC48 steel in 1 M hydrochloric medium without and with various concentrations of GB leaf extract are given in Figure 3 and electrochemical parameters such as (E_{corr} , I_{corr} , β_c , β_a , and %IE) retained from the polarization curves by extrapolation, donated in Table 1. It can be seen that the addition of GB leaf extract

polarization plots.

had an effect on corrosion process in 1 M HCl solution and results revealed that the efficiency of inhibitor enhances by adding the GB leaf extract concentration. The uppermost inhibition is around 93% and was achieved after addition of 1500 ppm of GB leaf extract. If the difference in the values of ΔE_{corr} ($\Delta E_{\text{corr}} = \Delta E_{\text{corr}(\text{blank})} - \Delta E_{\text{corr}(\text{inh})}$) is more than 85mV, the inhibitor could be classed as an inhibitor anodic or an inhibitor cathodic. On the other hand, if ΔE_{corr} measures less than 85mV, the inhibitor could be classified as a mixed-type inhibitor [23,24]. Herein the introduction of GB leaf extract into the 1 M HCl solution causes a slight shift in corrosion potential from 20 to 49 mV compared to the blank solution. In addition to this, we can see that the polarization with an increase in inhibitors moves around to the positive (anodic) direction, this indicates that GB leaf extract in the studied solution acts as a mixed inhibitor with a predominant anodic effect [25]. The diminution of density of corrosion current after adding of GB leaf extract occurs due to the adsorption process on the metal surfaces [26].

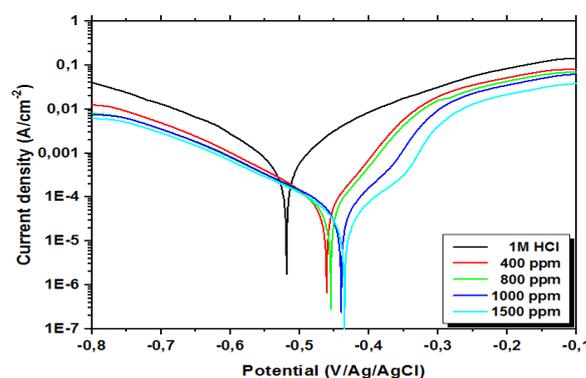


FIGURE 3: The representative Tafel polarization plots in with and without different concentration of GB leaves extract inhibitor.

3.1.3. Electrochemical impedance spectroscopy (EIS)

The EIS study has been adopted as a well-recognized and generally accepted technique to verify the adsorption process at the metal / electrolyte interface. The recorded spectra were reported in the form of Nyquist diagrams, Bode and phase plots in Fig. 4 (a), (b) and (c) respectively.

TABLE 2

EIS parameters and inhibition efficiency for XC48 steel as a function of inhibitor extract concentration at 298K: in 1M HCl.

C(ppm)	R_s (Ω .cm ²)	R_{ct} (Ω .cm ²)	C_{dl} (μ F.cm ⁻²)	E (%)	θ
Blank	2.852	39.05	407.57		
400	2.830	253.15	99.64	84.44	0.8444
800	2.631	306.74	82.23	87.27	0.8727
1000	2.590	369.52	68.26	89.43	0.8943
1500	2.348	406.15	54.68	90.28	0.9028

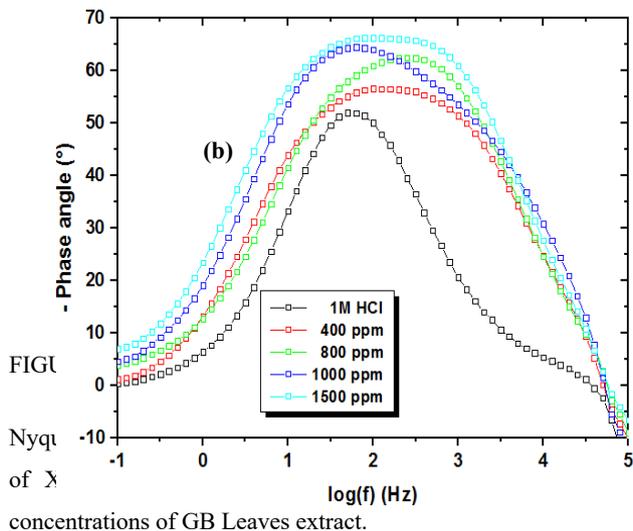
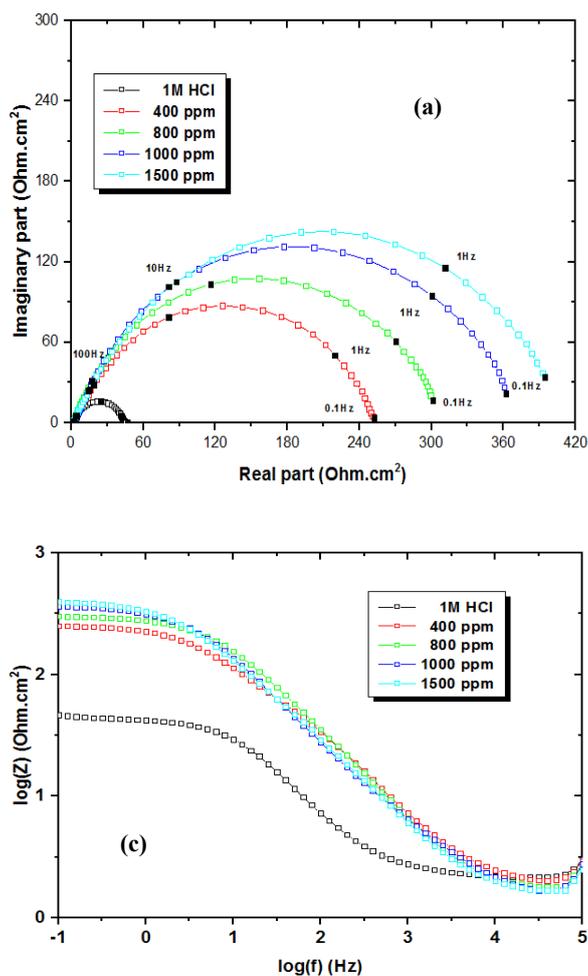


FIGURE 4

Nyquist
of Z

concentrations of GB Leaves extract.

All impedance spectrums in the Nyquist plots **Figure 4 (a)** for 1M hydrochloric solution without and with GB leaf extract demonstrate a single semicircle with a diameter that gets bigger with gradation of inhibitor concentration this means that the process of corrosion was mainly mastered by a charge transfer reaction [27,28], and the immensity of the loops expands, with a significant resemblance between forms of impedance curves at all concentrations. It indicates that the addition of GB leaf extract efficaciously slows up the corrosion reactions without modification of the corrosion mechanism [29]. It can be seen that the magnitude of values of $\log |Z|$ in the Bode modulus raises conjointly with the phase angle (φ) in the presence of the inhibitor and the effect is monotonic with the GB extract concentration. Furthermore, all phase-frequency angle plots indicate a single wave, confirming the unique constant obtained by the Nyquist diagrams. According to Akhil Saxena [30].

3.1.4. Adsorption isotherm models

Many models of adsorption isotherms were tested so as to better understand the interaction between GB leaf extract components and the surface of XC48 steel in the corrosive 1 M HCl solution, applying the values of surface coverage (θ) determined by the electrochemical data. The best-fitting isotherm has been identified from the linear correlation coefficient (R^2) value, with the Langmuir adsorption isotherm being the most appropriate. The surface coverage is linked to the concentration of inhibitor by following Equation [31]:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (3)$$

Where K_{ads} represents the equilibrium constant of the adsorption process and C_{inh} the concentration of inhibitor. The constant K_{ads} can be found from **Figure 4**. It is combined with the standard free adsorption enthalpy ΔG_{ads} by the relation below [32].

$$K_{ads} = \frac{1}{55.55} \exp\left(-\frac{\Delta G_{ads}}{RT}\right) \quad (4)$$

Where, T is absolute temperature (298 °K), R is the ideal gas constant (8.314 J.mol⁻¹. K⁻¹) and 55.55 represents water molar concentration in the bulk solution.

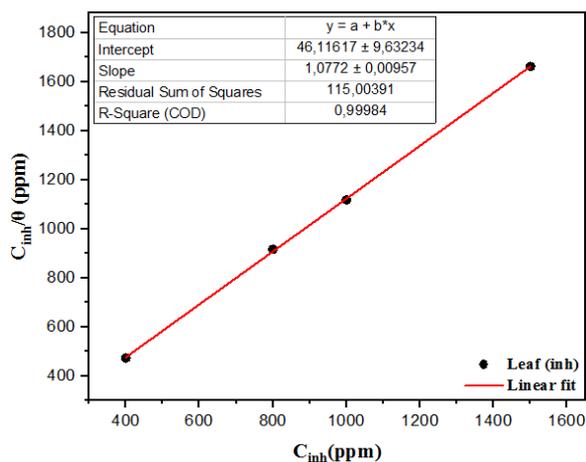


FIGURE 5: Langmuir adsorption isotherm for XC48 steel in 1 M HCl solution in the presence of GB Leaves.

TABLE 3

The thermodynamic parameters of the corrosion of the XC48 in 1 M HCl medium containing the inhibitor, revealed of Polarization.

Inhibitor	K	R ²	ΔG KJ mol ⁻¹
Leaves	2.1 10 ⁴	0.9998	-34.61

The calculated value of ΔG_{ads} is -34.61 KJ mol⁻¹, who suggested the occurrence of both physisorption and chemisorption processes, with a major contribution from the latter [33]. Moreover, the negative value shows that the adsorption process is spontaneous and the high values of K_{ads} (**Table 3**) give thought to the strong adsorption of inhibitor on metal surface [34].

3.2. Weight loss measurements

Mass loss measurements of the samples were performed in accordance with the standard ASTM G31-72 method for determining the corrosion behavior of steel. XC48 steel samples were placed in 1 M hydrochloric acid mediums at different temperatures (298 to 333) and various immersion time. A reference solution without inhibitor was also used. To determine the effectiveness of inhibition of the corrosion process, XC48 samples were cleaned with distilled water, dehydrated with acetone and reweighed. Triplicate experiments were carried out for each operating condition and the average weight loss value registered. The corrosion rate CR (g cm⁻² h⁻¹) and inhibition efficiency IE (%) were determined according to the following equations [35-36]:

$$CR = \frac{\Delta m}{S \times t} \quad (5)$$

$$IE (\%) = \frac{CR_0 - CR}{CR_0} \times 100 \quad (6)$$

Where Δm is the specimen weight loss in g, S is the specimen total surface area in cm^2 and t is the immersion time in hour, CR_0 and CR are the weight loss rates of carbon steel in the acid medium in the absence and presence of the extract, respectively.

3.2.1. Immersion time Effect

The adsorption ability of the inhibitor on the surface of the XC48 steel followed by the weight loss measurements was carried out using the optimum concentration (1500 ppm) of the extract, at room temperature 298 K and at different immersion times (6–168 h). The different values are given in **Table 4**. It can be observed that the corrosion rates obtained in the absence and presence of inhibitor act as a function of immersion time. It should be noted that the maximum IE was 93,02% at 24 hours and a slight decrease in the effectiveness of inhibition at 48h, but after 168 h of immersion a considerable decrease in inhibition efficiency may be due to desorption of inhibitor molecules [35].

TABLE 4
Efficiency of corrosion inhibition of XC48 steel during different time at 298 K in HCl 1 M with the optimum concentration

Time (h)	Blank		1500 ppm	
	$CR \times 10^{-3}$ (g cm^{-2} h^{-1})	$CR \times 10^{-3}$ (g cm^{-2} h^{-1})	$CR \times 10^{-3}$ (g cm^{-2} h^{-1})	IE (%)
6	7,82	0,75		90,41
24	6,46	0,45		93,03
48	4,18	0,57		86,36
72	2,97	0,71		76,09
168	3,61	1,12		68,97

3.2.2. Temperature effect

The influence of temperature on the corrosion behavior of XC48 steel was investigated at temperatures of 298, 308, 318, and 333K during 24 h of immersion in the absence and presence of 1500 ppm of inhibitor. Corrosion rate values for XC48 steel and inhibition efficiency measured by weight loss are shown in **Table 5**. The data revealed that increasing temperature leads to an increase in steel dissolution and corrosion rates, and a marked decrease in inhibition efficiency at high temperatures. This is due to the destabilization of the interaction between the inhibitor and the metal surface, moreover temperature is a kinetic parameter; high values can perturb the solution/surface system, affecting the inhibitor's adsorption capacity [37].

TABLE 5

Corrosion rate of XC48 steel as a function of temperature in HCl 1 with and without the inhibitor, and inhibition efficiency of Grewia bicolor juss leaves extract on corrosion of XC48.

Temperature (K)	Blank	1500 ppm	
	$CR \times 10^{-3}$ (g cm^{-2} h^{-1})	$CR \times 10^{-3}$ (g cm^{-2} h^{-1})	IE (%)
298	6,46	0,45	93,03
308	8,02	1,43	82,16
318	9,98	2,54	74,55
333	12,39	5,30	57,22

3.2.3. Thermodynamic and Activation Parameters

The effect of temperature on the corrosion inhibition process cannot be elucidated without evaluation of the activation factors. Accordingly, the gravimetric measurements are carried out in 1 M HCl solution at 303–333 K for the optimum concentration of Grewia bicolor juss extract. The logarithmic form of the Arrhenius relation allows to determine graphically the activation energy E_a and ΔH_a , respectively, by the following Equations [34,38]:

$$\ln (CR) = -\frac{E_a}{2.303 RT} + \ln A \quad (7)$$

$$\ln \left(\frac{CR}{T} \right) = \left(\ln \left(\frac{R}{Nh} \right) + \frac{\Delta S}{2.303R} \right) - \frac{\Delta H}{2.303RT} \quad (8)$$

E_a is the apparent activation energy, R represents the universal gas constant, T is the absolute temperature and A is the frequency factor. In the other Equation h is Planck's constant, N is for the number of Avogadro constant, T is absolute temperature (i.e. $273 + \theta$)° K, ΔS represents the change in entropy of activation (J/mol.K), and ΔH_a is the apparent enthalpy of activation (kJ/mol) [39]. The Arrhenius plots ($\ln CR$ vs. $1/T$) and the transition state plots ($\ln (CR/T)$ vs. $1/T$) for XC48 mild steel corrosion in HCl 1 M without and with optimum concentration (1500 ppm) of Gb extract are given in **Figures 6** (a) and (b) respectively and the values of E_a , and ΔH_a are determined from the slopes of the straight lines obtained are reported in **Table 6**.

Solutions	E_a (kJ mol^{-1})	ΔH_a (kJ mol^{-1})	ΔS_a (J $\text{mol}^{-1}\text{K}^{-1}$)
Blank	33,86	27,72	-434,50
1500 ppm	102,69	96,55	-244,12

TABLE 6

Thermodynamic parameters for the adsorption of Grewia bicolor juss leaves extract on XC48 mild steel in HCl 1 M at different temperatures.

The positive value of enthalpy of activation in the absence and presence of inhibitor reflects the endothermic nature of the XC48 steel dissolution process, indicating that steel dissolution is difficult [40]. The value increased in the presence of the inhibitor compared to the uninhibited solution, indicating a higher level of protection it can be explained as follows in the free acid solution: the rate-determining recombination step's transition state represents a more orderly arrangement than the initial state, resulting in a high negative value for activation entropy ΔS_a . The rate-determining step in the presence of an inhibitor, however, is the discharge of hydrogen ions to form adsorbed hydrogen atoms. Because the metal surface is covered with inhibitor molecules, the discharge of hydrogen ions at the metal surface is slowed, causing the system to move away from a random arrangement, increasing the entropy of activation. As a result, the entropy of activation increases (in the presence of inhibitors, the disordering from reactant to activated complex increases) [34,41].

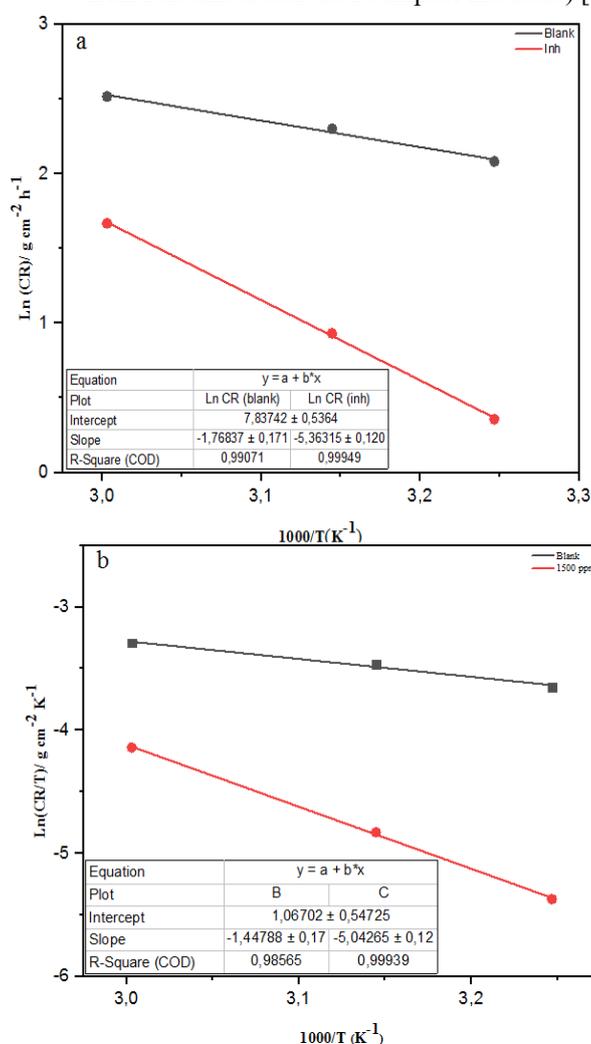


FIGURE 6: Arrhenius plots of Ln (CR) (a) and Transition state plots of Ln (CR/T) (b) vs 1/T for XC48 mild steel in

HCl 1 M in the absence and presence of optimum concentration (1500 ppm) of GBJ extract.

The values of E_a obtained for XC48 steel in hydrochloric acid solution containing inhibitor are greater with respect to those retrieved for the blank system. This is probably due to the formation of a protective film adsorbed on the metal surface of the working electrode, as previously mentioned by Deyab and Abd El-Rehim [42], the adsorbed inhibitive molecules generate a physical barrier hindering both mass and electrical charges transfer. The adsorption phenomenon can be classified either as physical or chemical. E_a values smaller than 80 kJ mol^{-1} generally reflect a physisorption mechanism, whereas greater values are related to a chemical adsorption mechanism [43]. From the achieved results, it can be concluded that the *Grewia bicolor* juss leaves extract adsorbs on the XC48 steel in hydrochloric acid solution via chemisorption.

3.3. SEM and EDX examination

The surface of the metal immersed for 24 hours in 1 M HCl without and with 1500 ppm of *Grewia bicolor* juss (GB) leaf extract was analyzed using SEM and the results are shown in Figure 7: (a) represents the SEM image for the surface of XC48 steel before immersion, then (b) and (c) after immersion in the corrosive solution in the absence and presence of the inhibitor, respectively. It is observed that the surface of steel after immersion in the corrosive solution (1M HCl) became damaged with large and deep holes, but after addition of 1500 ppm of the extract of GB leaves there is a formation of a protective layer on the surface of metal.

TABLE 7

EDS characterization of St37 steel surface after 24 h of immersion in 1M HCl without and with the presence of 1500 ppm of GB leaves extract.

Element (%)	Fe	C	O	F	Si	Cl	Mn	Zn
Bare XC48 steel	87.25	5.84	-	1.49	0.96	-	3.30	1.16
Blank	55.54	4.64	35.60	0.34	-	1.04	1.06	-
1500 ppm	68.89	4.11	23.37	0.44	0.38	0.72	2.1	-

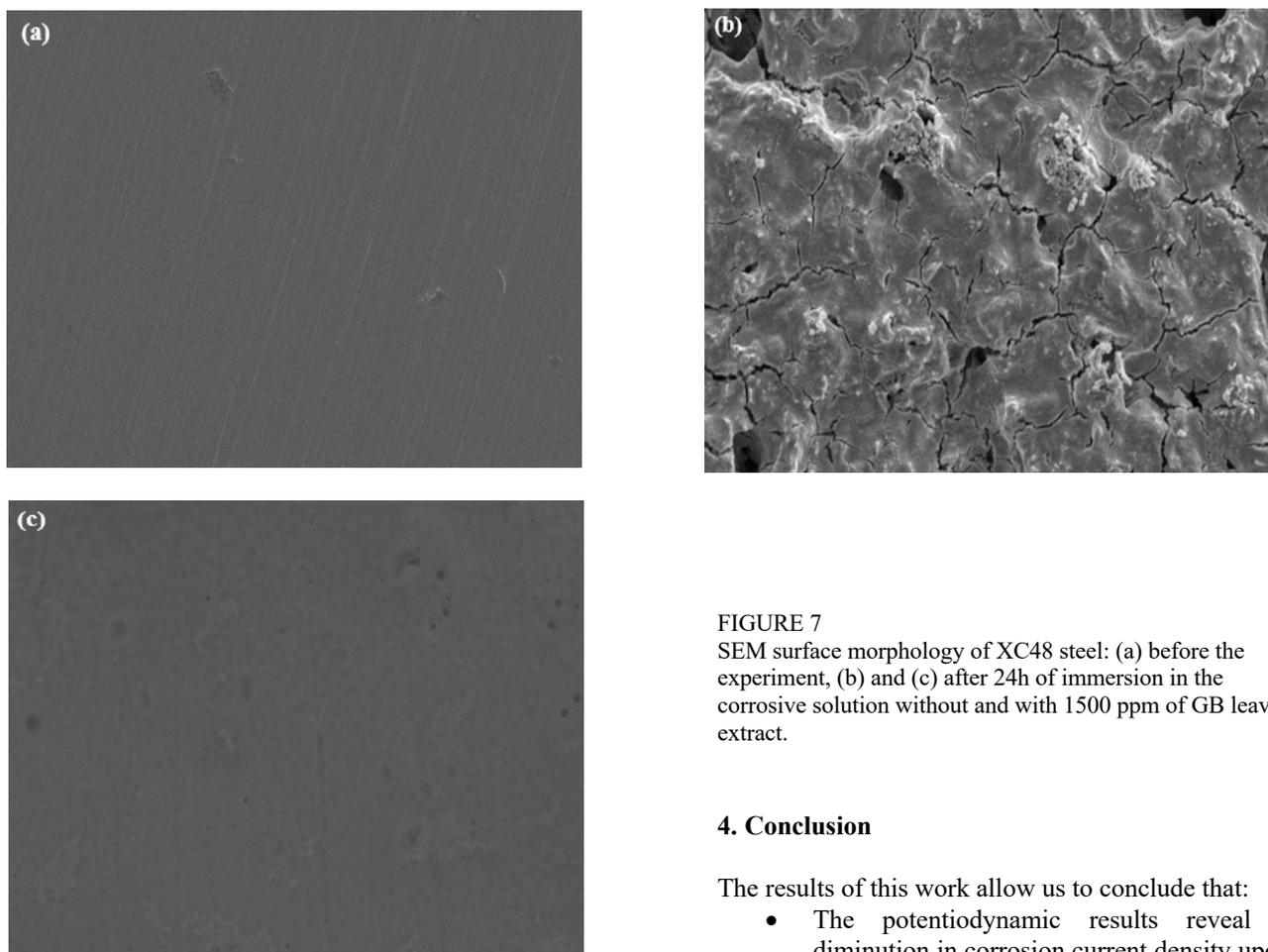


FIGURE 7

SEM surface morphology of XC48 steel: (a) before the experiment, (b) and (c) after 24h of immersion in the corrosive solution without and with 1500 ppm of GB leaves extract.

The corresponding EDS analysis, reported in **Table 7**, of the samples immersed in the blank solution, showed that the presence of significant amounts of Fe, O and Cl, is possibly due to the formation of Fe_3O_4 and FeCl_3 [27,44]. The presence of other elements such as C, Zn, Si and Mn is associated with the composition of the carbon steel sample. In the blank solution, the percentage of Fe, C, Si and Mn reduced compared to that of the XC48 carbon steel rods, showing that the carbon steel sample lost some percentage of its constituent elements due to corrosion in the aggressive environment.

4. Conclusion

The results of this work allow us to conclude that:

- The potentiodynamic results reveal a diminution in corrosion current density upon addition of the *Grewia bicolor juss.* leaf extract, and this inhibitor extract behaved as a mixed inhibitor. Tafel polarization indicated that the extract inhibited 93 % of the corrosion.
- The EIS data indicate the growth in corrosion resistance by dint of the increasing of the extract concentration in the corrosive medium. A highest protection of 90.28% for 1500 ppm of GB leaves extract was achieved in 1M HCl solution.
- Active constituents of the inhibitor adsorbing on the surface of the XC48 steel according to the Langmuir isotherm model.
- The calculated thermodynamic suggested the occurrence of both physisorption and chemisorption processes, with a major contribution from the latter.
- The surface analysis technique supported the electrochemical results and confirmed the adsorption of the active components of the GB leaf extract on the surface of the XC48 Steel.

Conflict of interest disclosure

The authors declare that there are no conflicts to declare.

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