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A COMPARATIVE STUDY ON THE CORROSION BEHAVIOR OF MILD STEEL AND ALUMINUM ALLOY IN ACIDIC MEDIUM USING GREEN CORROSION INHIBITOR

ABSTRACT

Many industries worldwide use steel and aluminum as the principal raw material. In one hand, thanks to its lightweight and durability, aluminum is widely used in all sectors of the transportation industry-land, sea, and air. In the other hand, production process of steel equipment’s is generally less expensive than others and its recyclability can be infinite. For this reason, it is one of the most used metals. After the production process, the finished metallic product needs a pickling process using aggressive acids such as hydrochloric acid. Consequently, aluminum and steel products are exposed to the corrosion phenomenon. For this reason, the present work investigates the anticorrosion effect of *Lavandula maroccana* ethanol extract on both aluminum and mild steel corrosion in 1M HCl solution. In fact, plant extracts inhibitors are replacing synthesized compounds that are usually environmentally toxic. Electrochemical measurements are the main data used to analyze the inhibition effect of LM inhibitor using the potentiostat device (PGZ100). Electrochemical impedance spectroscopy technique (EIS) showed an increase in the charge transfer resistance of mild steel specimen. The high inhibition efficiency is 89% for mild steel in 1M HCl using 400ppm of LM extract. For aluminum alloy used in this study, the high inhibition efficiency reached 68%. Scanning electron microscopy (SEM) and UV-visible surface analysis were also applied to investigate the effect of LM extract inhibitor on aluminum alloy and mild steel specimen.

Keywords: Corrosion, Mild Steel, Hydrochloric Acid, Potentiostat (PGZ100), EIS

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1. INTRODUCTION

All industrial metals are exposed to corrosion phenomenon that costs the economy 3-4% of gross national product [1]. Mild steel and aluminum alloys are the most used metals in various industries. In one hand, the extensive use of mild steel in industries including oil production, oil refining, power stations and civil engineering structures has been widespread due to relatively low cost, high strength and availability [2]. On the other hand, the light weight of aluminum alloys, their low density and high thermal and electrical conductivity make them widely used in many industrial applications such as automotive and aircraft industries [3]. However, if the metal suffers from corrosion problem, the previous properties and qualities could be useless for industrials. For this reason, researchers focused their works on corrosion inhibition of metals. The pickling process, that is used to clean every metal surface of the finished product, uses mainly aggressive acids such as sulfuric and hydrochloric acids to remove the surface impurities [4]. Even if aluminum shows a better corrosion resistance than mild steel in different corrosive mediums, thanks to the formation of an adherent passive oxide film [5], this surface film is amphoteric and dissolves substantially when the metal is exposed to high concentrations of acids or bases. Under these circumstances, corrosion inhibitors should be used because the solubility of the oxide film increases above and below pH4-9 range and aluminum exhibits uniform attack [6]. Thus, it is very important to add corrosion inhibitors to prevent metal dissolution and minimize acid consumption. Most of the organic inhibitors contain in their structures mostly N, S, and O atoms. Unfortunately, uses of some organic and inorganic chemical inhibitors are limited because their synthesis compounds are very costly. Moreover, most of them are nonbiodegradable so toxic for the environment. So, it has been focused on the corrosion inhibition properties of plant extracts because plant extracts serve as an incredibly rich sources of natural chemical compounds present (e.g. amino acids, terpenoids, flavonoids, alkaloids, polyphenols, tannins, etc.,) that are environmentally acceptable, of low cost, easily available and renewable sources of materials and extracted by simple procedures [5].

The inhibition effect of green products extracted from the plant leaves have been reported by several authors. James et al.studied the corrosion inhibition of aluminum in 2.0 M hydrochloric acid solution by the acetone extract of red onion skin [6]. They found that WMRE can be physically adsorbed on the mild steel surface in acidic media following a Langmuir adsorption isotherm. Dahmani et al. studied Corrosion Inhibition of C38 Steel in 1 M HCl as a comparative study of black pepper extract and its isolated piperine. The inhibition efficiency of black pepper extract increases with the increase of its concentration to attain 95.8% with 2g/l as inhibitor concentration [7]. Nasibi et al. investigated the inhibition effect of chamomile extract on mild steel corrosion in hydrochloric. CE acts as a mixed-type corrosion inhibitor with predominantly anodic behavior. CE was adsorbed physically on the metal surface and obeyed the Langmuir adsorption isotherm. Maximum inhibition efficiency (IE) was 93.28%, which was obtained at 22°C in 7.2g/L of inhibitor in 1 M HCl solution [8]. Soror, et al. studied Saffron extracts as environmentally safe corrosion inhibitor for aluminum in 2M HCl solution [9]. Results manifested that the highest corrosion inhibition was achieved using 600ppm of the extract (84%). Mild steel corrosion inhibition by Parsley (Petroselium Sativum) extract in acidic media was investigated by Benarioua et al.Weight loss method showed a maximal inhibition efficiency at 5g/l of 87.65% [10]. Thus, this study aimed to investigate the anticorrosion effect of Lavandula maroccana ethanol extract on both the corrosion of aluminum and mild steel samples in 1M HCl solution. Tow electrochemical applications were applied for the investigation: potentiodynamic polarization (PDP), electrochemical impedance spectroscopy (EIS). SEM- EDX mapping and UV-visible were applied to give more understandings for inhibition behavior.

2. RESEARCH SIGNIFICANCE

In the present work, corrosion inhibitors are used as a prevention way to decrease the corrosion rate of aluminum alloy and mild steel in 1M hydrochloric acid. Most of chemical inhibitors are expensive and not safe for environment [11]. Thus it's necessary to develop eco-friendly inhibitors for both aluminum and steel in acidic mediums [12]. Hence, we have chosen *Lavandula marocanna* ethanol extract as green corrosion inhibitor to compare its inhibition efficiency in hydrochloric acid. Electrochemical impedance spectroscopy and potentiodynamic polarization have been used to evaluate the anticorrosive behavior. The metals' surface morphology was studied by SEM investigation and solid ultraviolet absorption spectrum.

3. EXPERIMENTAL METHOD-PROCESS

3.1. Test Solutions

Hydrochloric acid is the corrosive acidic medium used in this experimental study; 1M concentration was prepared using 37% commercial solution and distilled water. Four concentrations were also prepared using 1M HCl solution and the powdered ethanol extract of *Lavandula marocanna* plant as follow: 0.05, 0.1, 0.2 and 0.4g/l.

3.2. Metal Specimens

According to EDS analysis, aluminum alloy used in this study belongs to 4XXX group [13]; Aluminum alloyed with silicon. Silicon is good in metallic alloys used for casting. This is because it increases the fluidity of the melt, reduces the melting temperature, decreases the contraction associated with solidification and is very cheap as a raw material [14]. For mild steel specimens, it contains 99.2% of Fe, 0.21% of C and other elements that help to maintain good chemical and mechanical properties. Both aluminum and steel specimens were rectangular; $0.8cm^2$ and $0.5cm^2$ are respectively coupons areas used in electrochemical studies. They were mechanically cut and abraded with (600, 800, 1000 and 1200 grade) emery papers. Then, the coupons were cleaned and dried with dryer.

3.3. Preparation and Constituents of LM Extract

L. maroccana is an endemic species of Morocco, located in the high Atlas Mountains (1700m of altitude). Taxonomic determination was performed by Pr. Ahmed OUHAMMOU, Faculty of Sciences Semlalia University Cadi Ayyad. A voucher specimen was deposited in the regional herbarium of the Cadi Ayyad University, Marrakesh, Morocco, under N° MARK 8443. The aerial parts of Lavandula marocanna were used to prepare various extract (Ethanol and aqueous extracts). A quantity of 40g of the aerial's parts powder was macerated with 200mL of solvents for 24h at room temperature using a magnetic stirrer. The extract was then shacked, filtered and evaporated in a rotating evaporator under reduced pressure until dryness [15]. The extracts were stored in sealed glass vials at 4 to 5°C prior to analysis. Each extraction was performed in triplicate.

3.4. Phytochemical Screening

The phytochemical screening of *L. maroccana* aerial parts was characterized by high proportions of reducers compounds, flavonoids and catechin tannin compounds.

Table 1. Preliminary phytochemical screening of aerials parts of L. marocanna Murb

		Aerials Parts
Catechin Tannins		+
Gallic Tannins		+
Reducers Compounds		+
Carotenoids		-
Anthocyanins		+
Leucoanthocyanins		+
Alkaloids	Mayer's Test	-
	Wagner's Test	-
Flavonoids		+++
Sterols		+++
Terpenoids		+++
Coumarines		+
Terpenoids		+++
Free Quinones		+++

(+): Presence, (-): Absence

3.5. Electrochemical Experiments

Electrochemical measurements were obtained using an electrochemical cell with three electrodes: Working electrode (WE) of metal (Aluminum/Mild steel), platinum Counter electrode (CE) and saturated calomel electrode as reference electrode (RE). The cell was connected to Potentiostat/Galvanostat PGZ100 analyzer assisted with a computer. All the corrosion data are obtained from polarization curve using Volta-Master (Version 4) software. All tests were performed at 35°C. The samples were first immersed into the solution for 30 min to establish a steady state open circuit potential. After measuring the open circuit potential between sample electrode and reference electrode, EIS test was carried out over a frequency range of 100 KHz to 10 mHz using a 10-mV sine wave AC voltage. All EIS information was fitted to proper proportional circuit utilizing the EC-Lab V10.40 software. The results of EIS were tested and deduced by using the equivalent circuit [16]. The (% IE) of the investigated LM extract inhibitor from the impedance tests can be computed from Eq.1:

$$\%IE=[1-(R°_{ct}/R_{ct})]x100 \quad (1)$$

Where $R°_{ct}$ and R_{ct} are the charge transfer resistances without and with inhibitor, individually. For Aluminum working electrode, potentiodynamic polarization measurements were obtained by sweeping the electrode potential from -1000 mV to -500 mV at a scan rate of 1 mV /sec. At the same scan rate, Tafel curves for mild steel electrode were obtained over a potential of -800mV to -200mV. The inhibition efficiency IE% was calculated using the following equation:

$$\%IE=(\frac{I_0-I_{inh}}{I_0}) \quad (2)$$

where I_0 and I_{inh} are the corrosion current densities of mild steel/aluminum in the absence and presence of inhibitor, respectively.

3.6. Surface Analysis

Layers aspects of aluminum surface was studied by scanning electron microscopy (SEM) using a JEOL JSM-IT100 microscope. The composition of the film formed on the alloy surface was analyzed by energy dispersive spectroscopy (EDS). Three specimens were scanned: Before immersion in the test solution and after 24 hours of immersion in solution with absence and presence of inhibitor in 1M HCl solution [1]. The measurements of UV-visible reflection spectra of surface species on the aluminum and mild steel specimens were performed by using UV/Vis spectrophotometer V-670 series, attached with ILN-725 Integrating Sphere. This spectrophotometer is controlled with the Spectra Manager software. The coupons were mechanically polished and immersed in the test solution during 3 hours for aluminum and 6 hours for mild steel specimens based on weight loss time measurements.

4. FINDINGS AND DISCUSSIONS

4.1. Electrochemical Study of Inhibition Corrosion of LM Extract on Mild Steel in 1M HCl

4.1.1. Electrochemical Impedance Spectroscopy (EIS)

The open circuit potential of electrode surfaces mild steel specimens in investigating solution was immersed for 30 min to allow the inhibition and kinetics of the corrosion processes to stabilize before running EIS and polarization experiments. Nyquist plots of mild steel in 1M HCl (Figure 1) show one capacitive loop which means that the corrosion process is mainly charge transfer controlled. The shape is maintained throughout the whole concentrations, indicating that almost no change in the corrosion mechanism occurred due to the inhibitor addition. In contrast to corrosion inhibition effect of LM extract on aluminum alloy in the test solution, the polarization resistance, as well as the inhibition efficiency, increases with the increase of inhibitor concentration from 15 to 80 ohm.cm² for 0.05 to 0.4g/l as it is presented in the Table 2. C_{dl} values are given in the same table. The C_{dl} values for the inhibitor are generally lower than the acid blank values.Thus,the inhibitor adsorbed on the steel surface thereby forming a protective layer on the steel surface to reduce the rate of charge transfer process [17]. The maximum inhibition efficiency was 81% which means that LM ethanol extract is a good corrosion inhibitor for mild steel in 1M HCl medium. In fact, Heterocyclic constituents like alkaloids, flavonoids could be the major responsible of the inhibition activity in many plant extracts. Even the presence of tannins enhances the film formation over the metal surface, thus aiding corrosion [18]. In the same manner, a decrease in C_{dl} is attributed to decrease in dielectric constant and increase in the thickness of the electrical double layer, suggesting that the AERST molecules are adsorbed at the metal/solution interface. This behavior is in accordance with Helmholtz model [19].

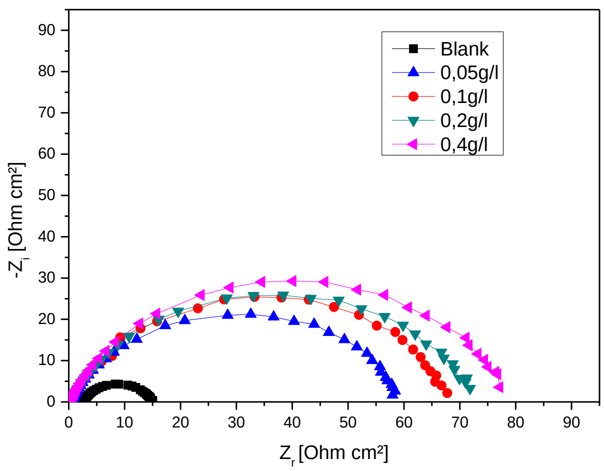


Figure 1. Nyquist plots of mild steel in 1 M HCl with and without different concentrations of LM extract at 35°C

Table 2. Impedance parameters of mild steel in 1 M HCl at 35°C containing different concentrations of LM extract.

	C(g/l)	Rct ($\Omega.cm^2$)	Cdl ($\mu F/cm^2$)	E(%)
Blank	0	15	116	-
Inhibitor LM Extract	0.05	57.32	70.61	73.88
	0.1	67.6	95.16	77.81
	0.2	71.84	89.83	79.12
	0.4	79.36	100.8	81.09

4.1.2. Polarization Measurements

Polarization plots of mild steel in 1 M HCl solution in the absence and presence of different concentrations of LM extract are shown in Figure 2. Corresponding electrochemical parameters are summarized in Table 3. Mild steel polarization plots illustrated that the addition of LM extract decreases the current density of both anodic and cathodic reactions. Also, it can be clearly seen that the inhibition efficiency of LM extract increases with inhibitor concentration to reach a maximum value of 81.52. This behavior shows that LM extract acts as a good inhibitor for the corrosion of mild steel in HCl media. Moreover, in the presence of LM extract the values of corrosion potential E_{cor} are nearly constant; Therefore, LM extract could be classified as a mixed-type inhibitor [20]. An inhibitor can be classified as cathodic or anodic type if the displacement in corrosion potential is more than 85 mV with respect to corrosion potential of the blank and when the when the E_{cor} shifts slightly, the inhibitor act as mixed type inhibitor [21]. The parallel Tafel lines indicates that the addition of LM extract did not change the dissolution mechanism during the retardation of the anodic metal dissolution and the cathodic reduction reactions [22].

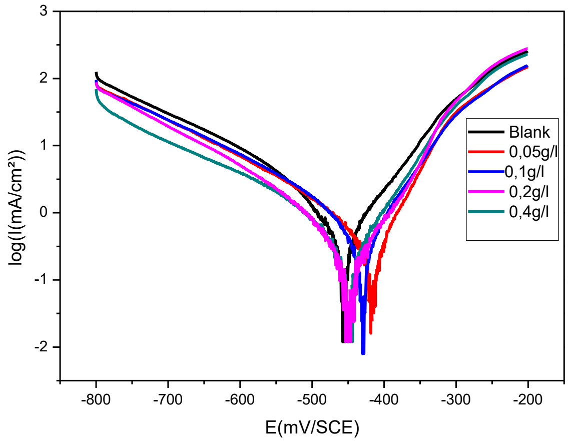


Figure 2. Polarization curves of mild steel in 1 M HCl at 35°C containing different concentrations of LM extract

Table 3. Kinetic parameters of mild steel in 1 M HCl at 35°C containing different concentrations of LM extract

	C(g/l)	icorr (mA/cm²)	Ba mV	βc mV	E(i=0) mV	E(%)	θ
Blank	-	1.7103	108.2	-192.8	-457.7	-	-
Inhibitor concentration	0.05	0.794	89.1	154.9	-417.6	53.57	0.5357
	0.1	0.562	64.8	164.1	-430.3	67.13	0.6713
	0.2	0.398	91.3	244.8	-450.3	76.72	0.7672
	0.4	0.316	80	165.4	-450	81.52	0.8152

4.1.3. Adsorption Isotherm

According to the experimental results obtained in electrochemical tests (Table 3), plots of the data for isotherm showed LM extract adsorption agreed with the Langmuir isotherm as seen in Figure 3. R^2 value is approximately close to 1. Consequently, the isotherm adsorption obeys to Langmuir model equation Equation 3 as follow:

$$C_{inh}/\theta = 1/K_{ads} + C_{inh} \qquad (3)$$

Where C_{inh} the inhibitor concentration and K_{ads} is the equilibrium constant of the adsorption process [23]. Thus, the inverse of the intercept with C_{inh}/θ axis gives the experimental value of K_{ads} which is equal to 28.26 l/g. Moreover, the standard Gibbs energy of adsorption, $\Delta G°ads$ can be calculated using the following Equation 4:

$$K_{ads} = \frac{1}{C_{H2O}} \exp\left(\frac{-\Delta G°ads}{RT}\right) \qquad (4)$$

Where R is the universal gas constant, T the thermodynamic temperature and the concentration of water in the solution is 1000 g/l. According to that, the value of $\Delta G°ads$ is -26.23 Kj/mol. Thus, the negative values of $\Delta G°ads$ indicates that the adsorption process is spontaneous and is consistent with physisorption.

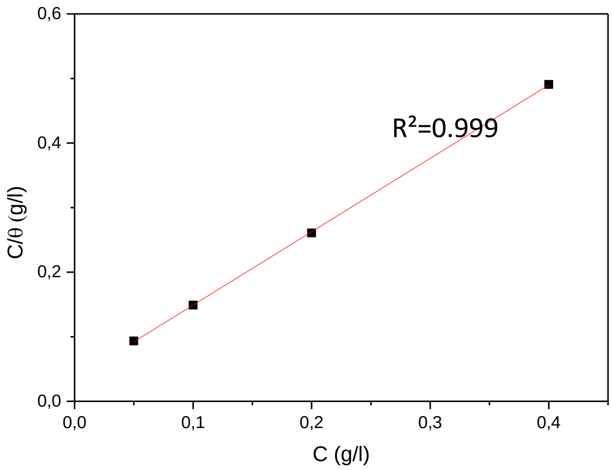


Figure 3. Experimental results at 35°C according to the Langmuir isotherm for mild steel

4.2. Comparative Study of LM Inhibition Effect on Mild Steel and Aluminum Alloy in 1M HCl

4.2.1. EIS Analysis Method

After analyzing the shape of the mild steel Nyquist plots previously presented, Figure 4 shows that the corrosion mechanism of aluminum and mild steel in 1M HCl solution is totally different. In general, both the nature of metal and chemical structure of inhibitor influence the phenomenon of adsorption.[24].To compare aluminum an mild steel corrosion behavior we have chosen 0.1g/l as an optimum concentration for both metals. Aluminum alloy Nyquist plots show a capacitive which means that the corrosion process is charge transfer controlled at high frequencies. Moreover, the semi-circle diameter increases with increasing of LM extract concentration. Charge transfer resistance increases with the increase of LM extract concentration until 0.1g/l. The maximal value was 99 ohm.cm² for 0.1g/l as corrosion inhibitor concentration giving a maximum inhibition efficiency of 61%. Thus, it is important to mention that LM extract acts as a threshold inhibitor for aluminum alloy in 1M HCl solution. In fact, above this concentration, the behavior of the corrosion inhibitor changes to accelerate the corrosion effect on aluminum alloy [9]. Otherwise, Nyquist plots of aluminum electrode show an inductive loop at low frequencies. At LF, time constant is higher thus, ions (other molecules in LM extract) have sufficient time to interact with surface and we get a pseudo-capacitive loop (inductive behavior in impedance spectra). Moreover, an inductance behavior in electric circuits is generally due to an opposing current created by an inductor and that is resisting to the electric current in the circuit. Hence, there could be, on the electrode surface, some different potential regions which produce an electric field opposing the external electric field causing the Nyquist plot to show inductive behavior. EDX technique has shown the addition of new chemical components on the aluminum surface after immersion in the test solution containing the inhibitor. According to Figure 4 and Figure 5, the inhibition efficiency of 0.1g/l of LM extract for mild steel is higher than aluminum alloy (81% and 61% respectively).

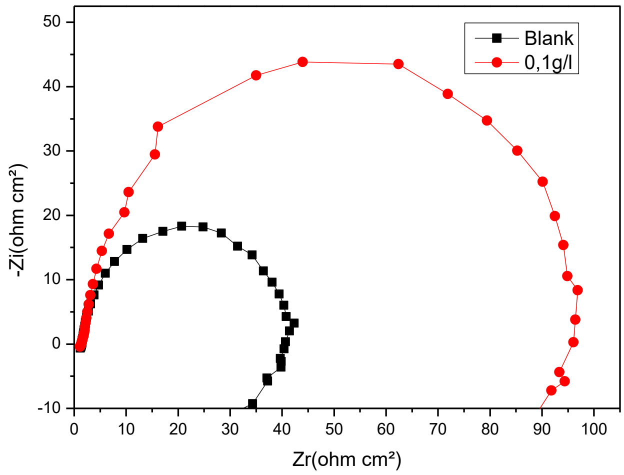


Figure 4. Nyquist plots of aluminum alloy in 1 M HCl in absence and presence of 0.1g/l of LM extract

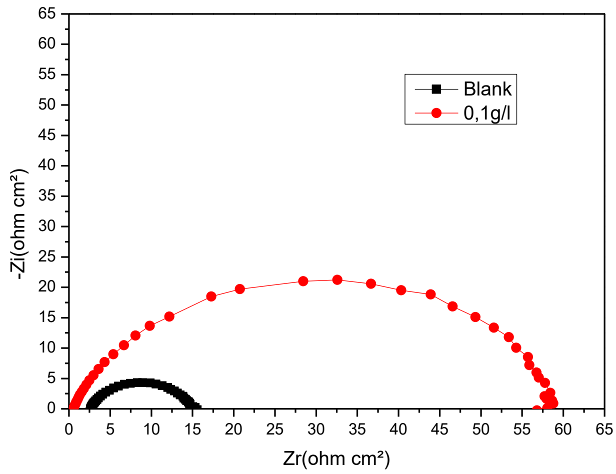
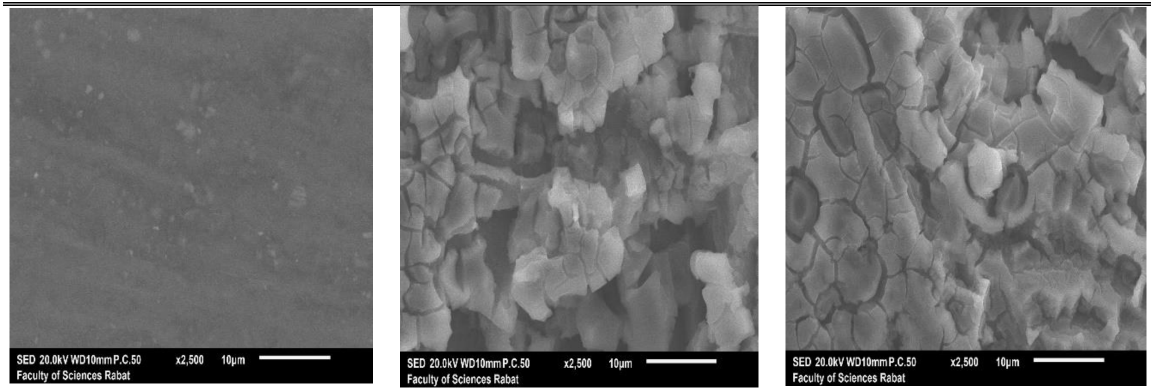


Figure 5. Nyquist plots of mild steel in 1 M HCl in absence and presence of 0.1g/l of LM extract

4.2.2. Scanning Electron Microscopy

SEM micrographs and the corresponding EDS spectra of the surface of the aluminum specimens, after 24 hours of immersion in 1 M HCl solution in the absence and presence of LM extract, are shown in Figure 6. The images reveal that in the absence of LM extract, the aluminum surface is damaged which is illustrated clearly with black areas on Figure 6b. After 24 hours of immersing the aluminum alloy in 1M HCl containing 0.1g/l as inhibitor concentration, black areas decreased lightly on the surface of aluminum specimen as it's illustrated in Figure 6c. which confirms the average inhibition efficiency of LM extract on aluminum alloy with maximum value 61%. The film produced on the external surface may reduce the oxygen reduction reaction on the metal surface. For this reason, the uniform corrosion is reduced too [9].



(a) (b) (c)

Figure 6. SEM micrographs of the surface of aluminum specimens after electrochemical tests (polarization measurements) in 1 M HCl: (a)before immersion in acid (free) (b)without henna extract; (c)containing 0.1 g/l LM extract

4.2.3. UV-Visible Reflection Measurements

The present surface analysis gives the reflectance of aluminum and mild steel specimens before and after immersion in 1M HCl in absence and presence of the inhibitor. The results shown in Figure 7 and Figure 8 indicate that the reflectance of both aluminum and mild steel specimen has decreased after immersion in 1M HCl solution in absence of LM extract. Whereas, the addition of the corrosion inhibitor (0.1g/l as an optimum concentration) to the test solution increases the reflectance value until it is close to the specimen reflectance before immersion in the acidic solution.

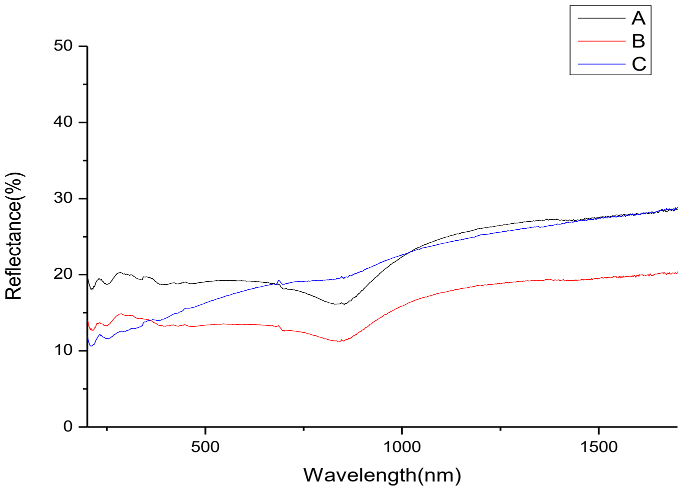


Figure 7. UV spectrum for aluminum specimens in 1M HCl solution in the absence and presence of 0.1g/l of LM extract as corrosion inhibitor: (A) before immersion, (B) immersion in 1M HCl, (C)with LM inhibitor

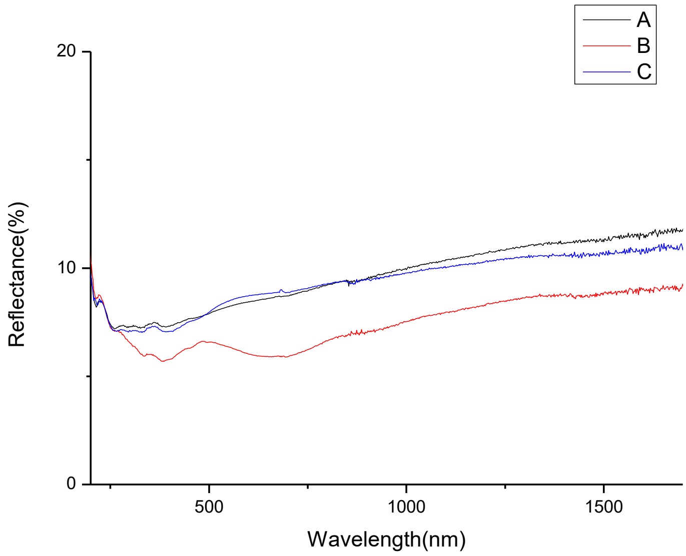


Figure 8. UV spectrum for mild steel specimens in 1M HCl solution in the absence and presence of 0.1g/l of LM extract as corrosion inhibitor: (A) before immersion, (B) immersion in 1M HCl, (C) with LM inhibitor

5. CONCLUSION AND RECOMMENDATIONS

Mild steel and aluminum alloy have different corrosion inhibition behavior in 1M hydrochloric acid solution containing different concentrations of LM ethanol extract. *Lavandula marocanna* ethanol extract is a good corrosion inhibitor for mild steel in 1M HCl solution at 35°C thanks to the synergic effect between the major chemical compounds (reducers compounds, flavonoids and catechin tannin compounds). Its inhibition efficiency increases with increasing the inhibitor concentration to reach a maximum value of 81% corresponding to 0.4g/l (EIS measurements); the capacitive behavior dominates the Nyquist plots obtained. Polarization measurements confirm the EIS results and show that LM extract is a mixed-kind inhibitor. The adsorption of LM extract molecules on mild steel abbeys Temkin isotherm model according to R^2 indicator which was close to 1. Otherwise, the comparative study of mild steel and aluminum alloy using the same inhibitor showed that aluminum alloy behavior was clearly different; In addition to the capacitive loop, aluminum Nyquist plots show an inductive behavior at low frequencies and the inhibition efficiency was lower.SEM and UV-Visible analysis of aluminum have confirmed the average inhibition effect of LM ethanol extract on aluminum alloy specimen in hydrochloric acid.

SYMBOLS

SEM: Scanning Electron Microscopy
EIS: Electrochemical Impedance Microscopy
EDX: Energy Dispersive X-Ray Spectroscopy
LM: Lavandula Maroccana

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