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Essential Oil of Lavandula Intermedia Walberton's Silver Edge as Green Corrosion Inhibitor for Carbon Steel in 1 M HCl Solution

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Abstract- The essential oil of *Lavandula intermedia Walberton's Silver Edge (*LAV*)* has been studied as a carbon steel corrosion inhibitor in 1 M hydrochloric acid using electrochemical obstacle spectroscopy (EIS), Tafel's polarization methods. The effect of extract concentration of (LAV) and immersion time on inhibition efficacy were studied. The effect of immersion time on the effectiveness of steel corrosion inhibition in 1M HCl was also studied in the 0.5 – 12 hour time interval. The results revealed that the essential oil of (LAV) tested reduces the kinetic process of the corrosion process of steel in medium 1 M HCl. Its efficiency increases with concentration and reaches a maximum value of 84% to 1% of LAVs. This corrosion inhibition effect attributed to was associated with the formation of a protective film provided to the metal surface by the complex chemical constituents of the oil extracted from (LAV), which is confirmed by surface analysis by scanning electron microscopy spectroscopy (SEM).

Keywords- Lavandula intermedia Walberton's Silver Edge; Carbon steel; Green Corrosion inhibition; Electrochemical Studies

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1. INTRODUCTION

Steel metal materials are most used in various industrial fields because of its good economic and mechanical properties [1]. But when it comes into direct contact with an airy electrolyte, especially in the presence of chloride ions, the steel is subjected to a rapid degradation of corrosion with a change in its physical and chemical properties.

Several recherches [2–9] have studied synthetic organic compounds as steel corrosion inhibitors in acid solutions (HCl, H_2S0_4). The toxicity and economic coercion of synthesis and performance of organic inhibitors has necessitated research activities in recent times to look for other acid corrosion inhibitors from natural sources. Because plant extracts are inexpensive, highly available and renewable material and ecological sources, plant extracts [10–15], essential oils [16–20], have become important and highly effective as green steel corrosion inhibitors in acidic environments in several researches. Phytochemical examination studies on the compounds that are responsible for the inhibitory efficacy of plants and its extracts, have shown that they are rich in alkaloids, tannins, organic acids, amino acids [21].

Lavandula intermedia Walberton's Silver Edge (LAV) is a variety of lavandin hybrid, lavandula genus that belongs to the Lamiaceae family, present in Morocco, Spain, France and Italy [22–24]. It is characterized by a shrub ranging from 60 to 150 cm, with evergreen, broad and rounded, grey-green lined with white. LAV essential oil is a complex hydrophobic mixture of volatile compounds that consists mainly of terpenes (oxygenated hydrocarbons and monoterpens) and sesquiterpenes (oxygenated hydrocarbons and sesquiterpenes) [22]. They also contain phenolic compounds. Thanks to their rich chemical composition, extracts of this lavandin variety have antioxidant, anti-inflammatory, antidepressant, analgesic, antiparasitic, anti-cancer, anti-inflammatory, antimicrobial, antifungal and anticancer properties [25–27].

The purpose of this work is to study the inhibition effect (LAV) extract as a green carbon steel corrosion inhibitor in hydrochloric acid. The mechanism of corrosion inhibition of steel in 1M HCl was studied using electrochemical impedance spectroscopy (EIS) and potentialiodynamic polarization. The morphology of the steel surface was examined by the scanning electron microscope (SEM, EDX)). The effect of the concentration and the immersion time has been also studied. The study shows that lavender extract has a good inhibition effectiveness in hydrochloric acid. Moreover, this extract has never has been studied as a corrosion inhibitor.

2. MATERIALS AND METHODS

2.1. Preparation of inhibitor

(LAV) was harvested in Beni Mellal, Morocco. The leaves of (LAV) were dried in the dark at room temperature and then ground. 100 g of powder obtained of (LAV) was hydrodistilized for 4 hours with the use of a Clevinger type device according to the European method of pharmacopoeia [28] as described by A. Boumezzourh and all [29]. The resulting essential oil was stored in the dark at a 313K temperature.

2.2. Materials and solutions

The corrosive solution 1 M hydrochloric acid was prepared by dilution of 37% HCl analytical hydrochloric acid with distilled water and titled by dosage with 1N NaOH. Essential oil inhibitor solutions (LAV) were prepared in the acid solution 1 M hydrochloric with percentage concentrations of 1% to 0,001%.

Carbon steel samples with a nominal wt. % composition indicated in the composition of the test specimen in Table 1:

Table 1. Percentage nominal composition of carbon steel

Element Symbol	**C**	**Mn**	**Al**	**Cu**	**P**	**Si**	**Cr**	**Fe**
Composition (w%)	0,07	0,19	0,02	0,12	0,02	0,03	0,05	99,5

Before each experiment, the steel samples used of size 5×1×0.2 cm^3 were treated with grades: 180, 400, 600, 800, 1200 successive abrasive paper, then cleaned by distilled water and degreased by acetone [15,30].

2.3. Electrochemical impedance spectroscopy

The electrochemical study was conducted using potentiostat OrigaStat 100, piloted by OrigaMaster software connected to a cell with three electrodes; the saturated calomel electrode and platinum electrode were used as reference and auxiliary electrode and the carbon steel with a surface of 1 cm^2 was used as working electrode. The working electrode was rinsed with distilled water, digressed with acetone and dried before its immersion in hydrochloric acid solution with different concentration of (LAV) essential oil. Before all electrochemical measurement, the working electrode was established at open circuit potential (E_{ocp}) for 30 min at ambient temperature.

Electrochemical impedance spectroscopy (EIS) spectroscopy measurements were performed at OCP (open circuit potential) with sine wave voltage (5 mV) peak to peak in frequency from 1 kHz to 100 mHz. The values of electrochemical parameters as R_t and C_{dl} were obtained from the Nyquist plot representation.

Inhibition efficiency (ER) is calculated by using the following equation (1) [31]:

$$\boldsymbol{E_R}(\%) = \frac{R_t - R_t^0}{R_t} \times \mathbf{100} \qquad (1)$$

Where R_t^0 and R_t represent charge transfer resistances without and with the addition of the (LAV) essential oil as inhibitor respectively.

2.4. Potentiodynamic polarization test

The potentiodynamic polarization tests were conducted at ambient temperature with and without addition of (LAV) in 1 M HCl solution. Potentials were recorded at scan rate of 20 mV/sec in cathodic and anodic direction from from -750 to -100 mV. The Tafel extrapolation method allows us the determination of corrosion current densities I_{corr} in thepresence of different (LAV) concentrations.

The values of inhibition efficiency in function of corrosion current density (E_I) are calculated according to the followed Equation (2):

$$\boldsymbol{E_I}(\%) = \frac{\boldsymbol{I_{corr}} - \boldsymbol{I_{corr}}/_{\boldsymbol{inh}}}{\boldsymbol{I_{corr}}} \times \mathbf{100} \qquad (2)$$

I_{corr} and $I_{corr/\mathrm{inh}}$ represent the corrosion current densities in the presence and absence of (LAV) respectively.

2.5. Scanning electron microscopy (SEM)

The morphology of the corrosion products formed on carbon steel surface in 1 M HCl solution with and without addition of the optimal concentration LAV at ambient temperature, were examined by using Scanning electron microscopy SEM type: JSM-IT10 coupled with EDX analysis.

After 24 h of carbon steel samples immersion in 1 M HCl solution in the presence and absence of 1% LAV at ambient temperature. Carbon steel samples, before their visualization using SEM were polished and rinsed with acetone and dried.

3. RESULTS AND DISCUSSION

3.1. Electrochemical experiments

3.1.1. Potentiodynamic Polarization Curves

The polarization curves for carbon steel in the HCl 1 M solution containing different concentrations of LAV is presented in Fig. 1. Their corresponding electrochemical parameters also inhibition efficiency are shown in Table 2. Based on the curves, it can be observed that the values of the current densities of I_{corr} corrosion decreases with the increase in the concentration of essential oil of LAV, therefore LAV acts as a good inhibitor in a hydrochloric acid solution, with an inhibition efficiency of about 84% for the 1% concentration.

The data presented in Table 2 show that the addition of LAV oil shifts the potential for corrosion (E_{corr}) to the cathode values and decreases the current densities. On the other hand, the cathode slope of Tafel (b_c) shows a change with the addition of LAV, suggesting that the inhibitory action occurred by blocking the cathode sites available on the metal surface, which led to a decrease in the exposed area, necessary for the evolution of hydrogen [3]. These results clearly indicate that LAV has a cathodic and anodyne effect. It has been reported in the literature that e when moving in E_{corr} greater than 85 mV compared to White E_{corr} implies that the inhibitor may act as an anodyne or cathode type, while a displacement in E_{corr} less than 85 mV implies that the inhibitor is mixed type [32,33]. In this study, the evolution of E_{corr} values for the oil studied LAV is in the 18-50 mV range, suggesting that LAV oil act as a mixed-type inhibitor, with cathode predominance.

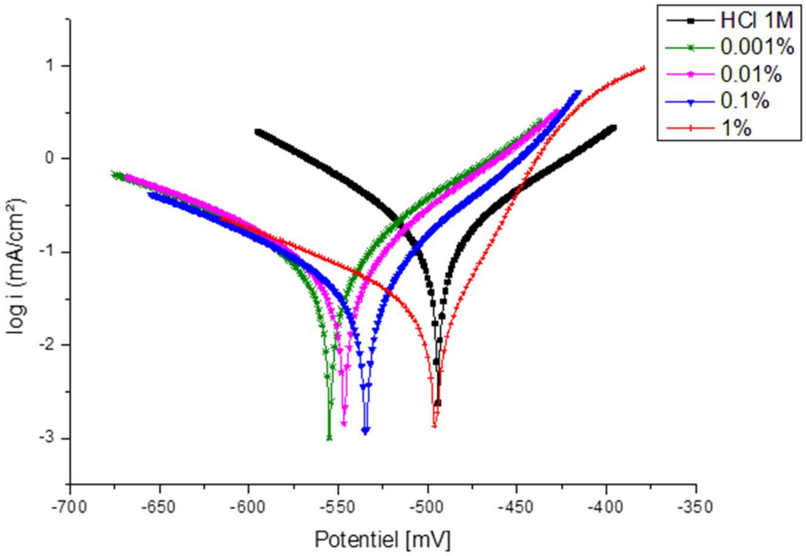

Fig. 1. Potentiodynamic polarization curves for carbon steel in 1M HCl in the absence and the presence different concentrations of LAV

Table 2. Polarization parameters for carbon steel in 1M HCl in the absence and the presence different concentrations of LAV

	C	E(mV)	$I_{corr}(\mu A/cm^2)$	B_c(mV)	B_a(mV)	R_p(ohm.cm^2)	E_I (%)
HCl	1M	- 513.7	0.1930	-104.34	113.85	-	-
LAV	0.00 1%	-554.5	0.0734	-130.61	83.26	265,62	62
	0.01 %	-546.7	0.0564	-122.23	75.12	284,04	71
	0.1 %	-534.5	0.0420	-120.60	63.74	407,83	78
	1 %	-495.5	0.0302	-135.37	37.82	570,11	84

3.1.2. Electrochemical impedance studies

The Nyquist representation of electrochemical carbon steel impedance spectroscopy diagrams in 1M HCl containing different concentrations of LAV after 30 minutes of immersion at E_{OCP} and at temperature of 298K are presented in Fig. 2. For all Nyquist's diagrams, it is observed that they are composed of a single capacitive loop. This means that the corrosion of the steel plate in 1 M HCl acid is controlled by load transfer[34]. On the other hand, it is noted that the loops have a similar shape under different conditions, which means that the addition of LAV is mainly inhibits corrosion by its adsorption on the surface of the steel. With regard to the diameters of capacitive loops, they increase by increasing the concentration of LAV; this may be due to the increase in the obstacle in the inhibited substrate and the formation of a protective film on the surface of the carbon steel plate in the presence of LAV essential oil.

Based on the data in Table 3, it is clear that the values of the effectiveness of inhibition increases with the increase in LAV concentration to reach its maximum value of 84% to 1%, also there is a remarkable decrease in the values of the double layer capacitance (C_{dl}), this result is attributed to the adsorption of LAV compounds on the surface of the carbon steel plate.

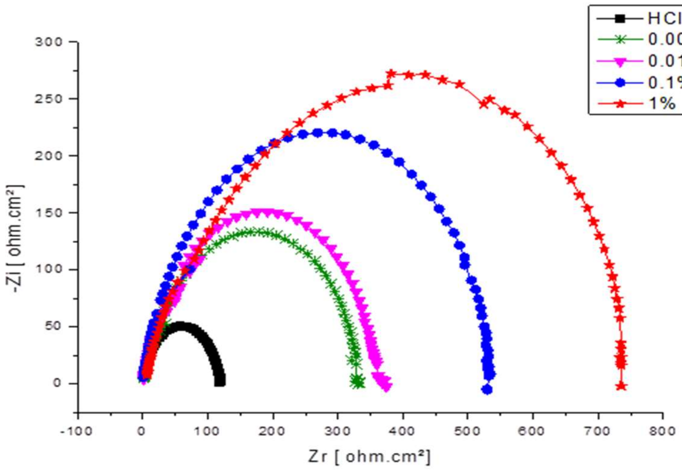

Fig. 2. Nyquist plots of carbon steel in 1 M HCl without and with different concentrations of LAV

Table 3. EIS parameters for the corrosion of ordinary steel in 1 M HCl without and with different concentrations of LAV

	C	**C_{dl}(µF.Cm^{-2})**	**R_s (Ω.Cm^2)**	**R_t(Ω.Cm^2)**	**θ**	**E_R (%)**
HCl	1M	133.61	0.15	119.13	-	-
LAV	0.001 %	107.83	2.30	331.08	0.64	64
	0.01 %	87.60	1.74	374.28	0.68	68
	0.1 %	59.38	1.94	536.05	0.78	78
	1 %	53.27	2.85	749.85	0.84	84

3.2. Immersion Time Effect

The follow-up the evolution of steel load transfer resistance at different immersion times in the aggressive medium without and with the addition of the inhibitor allows for important information about the stability of the protective layer formed on the surface. The electrochemical impedance diagrams obtained for carbon steel in HCl 1M medium in the presence of 1% LAV essential oil after different immersion times (0.5h, 2h, 4h, 6h, 12 h) is shown in Fig. 3 in The Nyquist representation of electrochemical impedance spectroscopy diagrams.

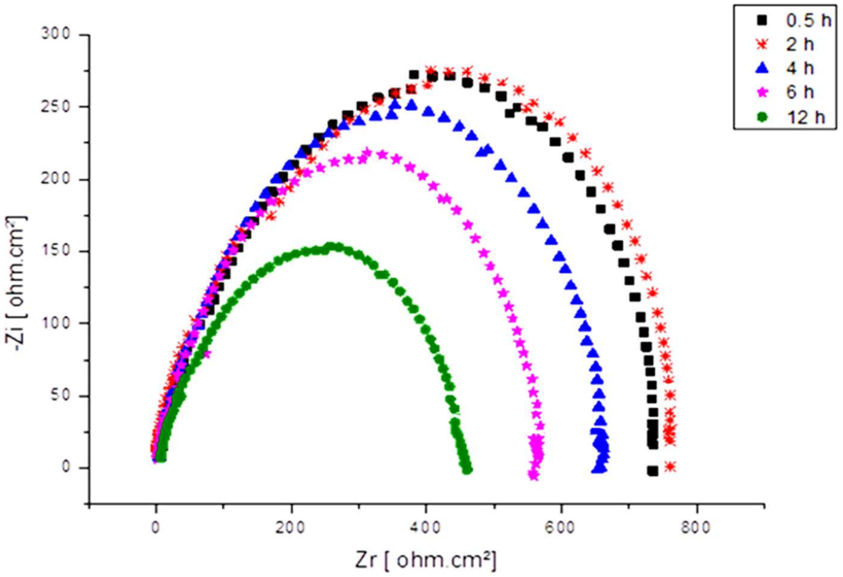

Fig. 3. Nyquist plots spectra for carbon steel in 1 M HCl in the presence of 1% of LAV at different immersion time

Table 4. Electrochemical impedance parameters for carbon steel immerged in 1 M HCl with and without 1% of LAV at different immersion times

Immersion Time (h)	**C_{dl}(µF.Cm^{-2})**	**R_t(Ω.Cm^2)**	**E_R (%)**
0.5	53.27	749.85	84
2	53.14	774.98	85
4	53.78	663.00	82
6	58.90	540.40	78
12	69.56	458.10	74

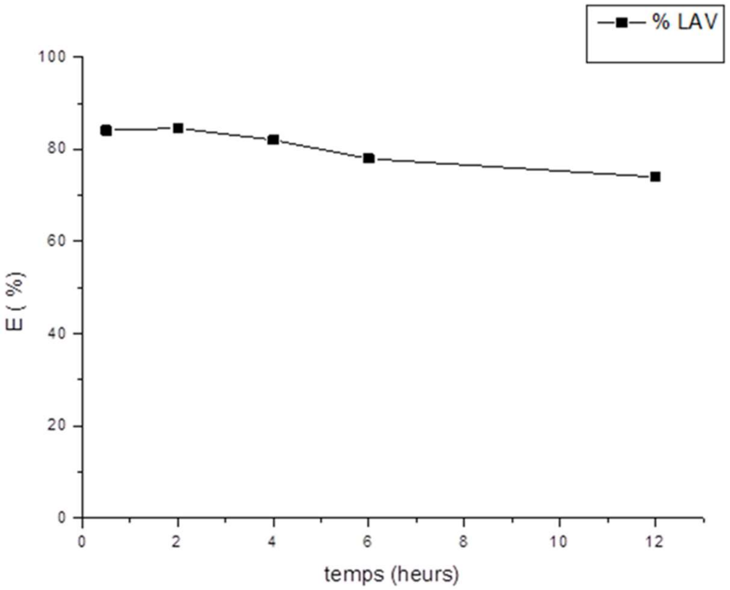

Fig. 4. Influence of immersion time on inhibition efficiency (E %) of carbon steel in 1M HCl in the presence of 1% of LAV

Fig. 4 more clearly illustrates the temporal evolution of the corrosion inhibition efficiency of steel to carbon (E %) in HCl 1M containing 1% of LAV essential oil.

Table 4 shows the values of load transfer resistance, double layer of steel to carbon, inhibition rates at different immersion times for LAV extract as inhibitor.

After analyzing these results, we note that all Nyquist diagrams were composed of a single capacitive loop with their diameter increased with immersion time up to 2 hours and decreased after that time. From the electrochemical parameters shown in Table 4, R_t values increase with immersion time to reach a maximum of 84% at 2 hours at the same time there is a decrease in C_{dl} values. This evolution of the R_t and C_{dl} is mainly due to the movement of water molecules by the ions of the acid and the adsorption of the molecules constituting oil inhibitory on the metal [35]. After the 2-hour immersion time, LAV still has an inhibitory effect with a slight decrease in R_t values and an increase in C_{dl} values and a decrease in R_t values. This result can be explained by the desorption of LAV molecules from the carbon steel surface.

3.3. Adsorption Isotherm

The adsorption of organic molecules provides information on the interaction between the adsorbed molecules themselves and their interaction with the surface of the electrode.

Determining the type of isotherm requires the calculation of the recovery rate of the metal surface, the latter was evaluated by electrochemical impedance spectroscopy using the following equation [36].

$$\theta = \frac{R_t - R_t^0}{R_t} \qquad (3)$$

Based on the results obtained in Table 4, the value of (θ) increases with the addition of the LAV extract. The data were tested graphically by adjusting Langmuir, Temkin and Frumkin's isotherms corresponding to Fig. 5 Are given by the following equations [37]:

$$\frac{C}{\theta} = C + \frac{1}{K} \qquad \text{Langmuir} \quad (4)$$

$$C.K_{ads} = exp(-2a\theta) \qquad \text{Temkin} \quad (5)$$

$$C.K_{ads} = \frac{\theta}{1-\theta} exp(-2a\theta) \qquad \text{Frumkin} \quad (6)$$

where θ is the fractional recovery rate; C the concentration of inhibitor; K_{ads} is the constant balance of the adsorption-desorption process.

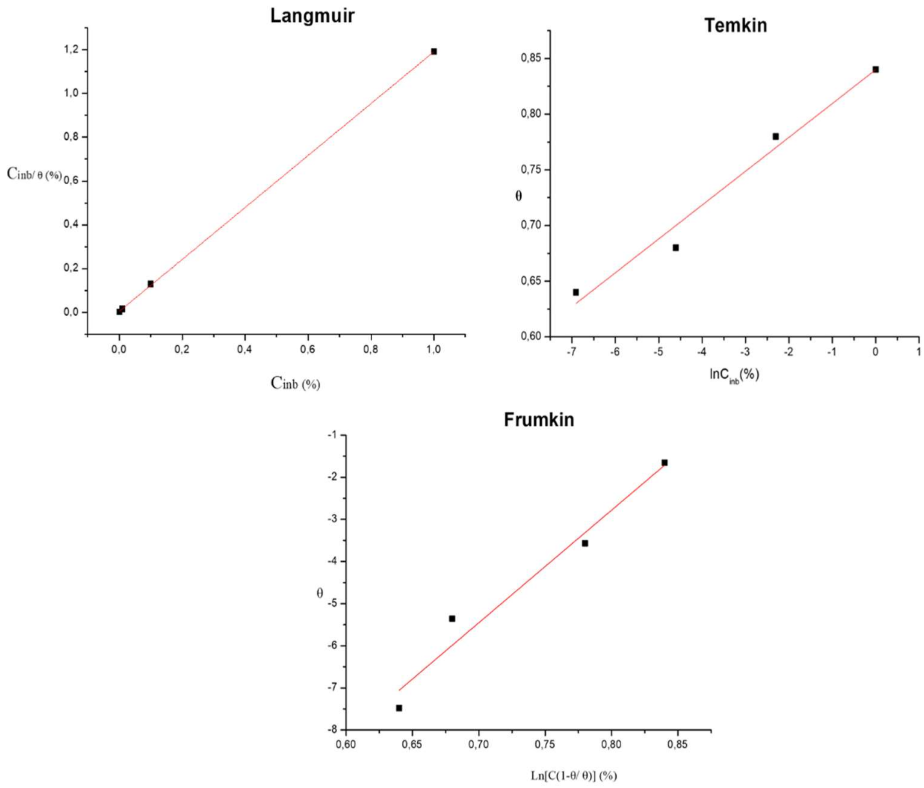

Fig. 5. Langmuir, Temkin and Frumkin Adsorption Isotherms obtained for carbon steel in HCl 1 M in the presence of LAV extract (obtained from the EIS) at 298 K

Table 5. Correlation coefficient values for different adsorption isotherms

Isotherme	**Langmuir**	**Frumkin**	**Temkin**
R^2	0.9999	0.964	0.948

Based on the results of Table 5, we found a correlation coefficient (R^2) up to 0.9999 for LAV essential oil, the slope is very close to the unit. These results suggest that the experimental data are well described and better Langmuir's isotherm. This isotherm implies the hypothesis, that all adsorption sites are equivalent and that particle bonds occur independently of the sites close to whether or not they are occupied [38,39], therefore, the inhibitory molecules are adsorbed to the metal surface to form a film that insulates the surface of the steel.

The free energy of standard adsorption $-\Delta G_{ads}$ (kJ / mol) is evaluated from the following equation:

$$K_{ads} = \left(\frac{1}{55.55}\right) \exp\left(\frac{-\Delta G_{ads}}{RT}\right) \qquad (7)$$

where, R is the perfect gas constant (8.314 J. mol^{-1}. K^{-1}), T is the absolute temperature, and the value 55.55 is the concentration of water in solution expressed in M.

Table 6. Langmuir Isotherption Adsorption parameters for Carbon Steel in HCl 1M containing different concentrations of LAV

R^2	Slop	Intersection	ΔGads (kJ/mol)
0,9999	1.19	0.004	-14.06

Based on the results obtained in Table 6, the value of ΔG_{ads} is -14.06 (kJ/ mol) for LAV oil. This negative value of ΔG_{ads} for the system studied implies that the inhibitor's adsorption on the surface of carbon steel was spontaneous, and the film adsorbed on the metal surface is stable. In this case, we noticed that the value of adsorption is around -20 kJ/ mol, this corresponds to electrostatic interactions between the molecules charged thus the loads of the metal (physisorption). According to D. SEIFZADEH the adsorption mechanism is physical [40]. It is assumed that the anion Cl^- adsorb first to the positively charged metal surface by physical attraction and then the inhibitory molecules are adsorbed by electrostatic interactions between positively charged molecules and negatively charged metal surface. Molecules LAV also adsorbed on cathode sites in competition with hydrogen ions, which will be reduced by the following mechanism [40].

$$4H^+ + O_2 + 2e^- \rightarrow 2H_2O \qquad (8)$$

As a result, the activation polarization of the cathode reaction increases, leading to the reduction of the associated current.

3.4. Surface analysis

Fig. 6(a) shows carbon steel-scanning electron micrographs after 24-hour immersion in 1 M HCl solution. The surface of the specimen in Fig. 6(a) was strongly corroded in the acid medium in the absence of the LAV inhibitor, The EDX spectrum give the peaks that characterizes certain components constituting the carbon steel sample after a 24-hour immersion in 1 M HCl in the absence of inhibitor, which shows the presence of iron and oxygen, thus suggesting the formation of iron oxide produces corrosion.

In the presence of 1% LAV inhibitor in the corrosive solution, there is the formation of a thick film protective against adsorbed corrosion on the carbon steel surface as a watch Fig. 6 (b). Same results obtained by S. Merah and all [41]. Based on the analysis of the EDX spectrum in the presence of the LAV inhibitor, it is noted that the peak that characterizes Fe increases relative to the non-inhibitory steel surface sample. This result is explained by the protection of steel by the inhibitory film formed by the LAV constituents.

Comparison of the spectra and mass percentages shown in Fig. 7 and Table 7 in the presence of 1% of LAV with those of corroded steel in HCl 1M clearly shows that the peak of chlore and oxygen decreases sharply on the EDX spectra of the extract. These observations confirm the formation of a pre-detector layer that limits the electrolyte's access to the steel surface.

These results confirm those obtained by polarization measurements, which suggest that a thick protective film is formed on the metal surface and thus delayed anode and cathode reactions.

Table 7. Mass percentage of the various elements from the EDX analysis of the surface of steel to carbon in HCl 1M medium and in the presence of 1% of LAV extract

Elements	**% mass in corroded carbon steel**	**% mass in steel in the presence of LAV**
Fe	49.68	54.39
C	33.96	33.98
O	15.73	11.39
Cl	0.63	-

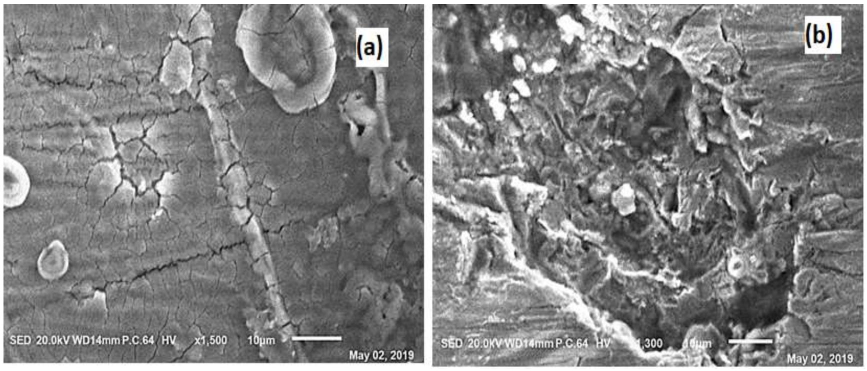

Fig. 6. SEM of carbon steel immersed in (a) 1 M HCl for 24 h (b) 1 M HCl in the presence of 1% LAV for 24 h

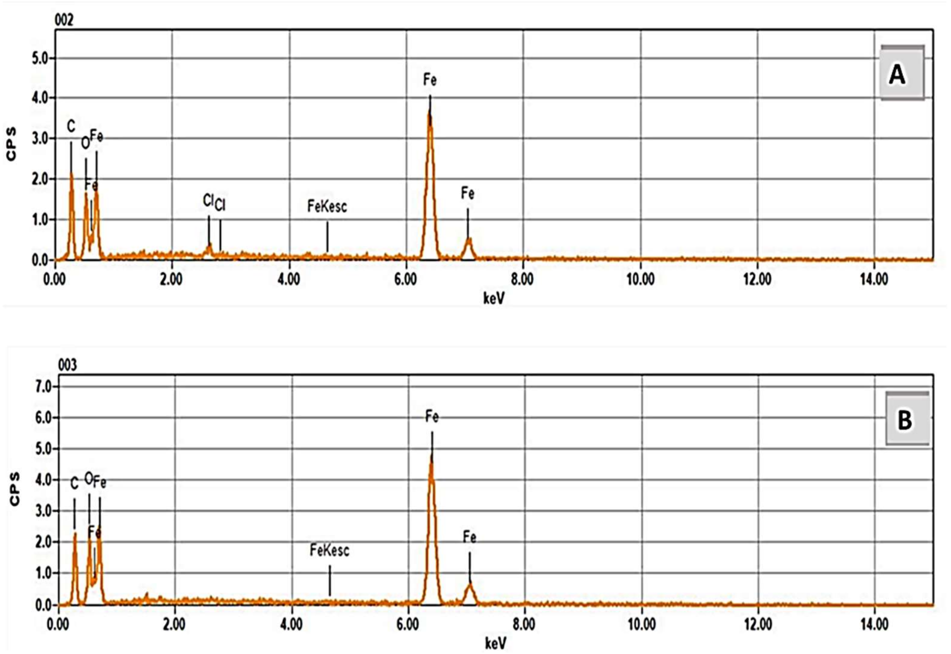

Fig. 7. EDX spectra of the surface of carbon steel (A) after immersion in HCl 1M at 298K (B) in the presence of 1% in LAV extract

4. CONCLUSION

The influence of LAV essential oil on corrosion behavior as a green corrosion inhibitor of carbon steel in 1 M HCl using different electrochemical methods and surface analysis by SEM, and it has been found that:

- LAV reacts as a good carbon steel corrosion inhibitor in 1M HCl and decreases both anodic and cathode reactions.
- The effectiveness of inhibition increases with LAV concentrations to a maximum of 84% to 1% in LAV.
- Intensity-potential polarization curves have shown that LAV acts as a mixed-type inhibitor, with cathode predominance.
- The effectiveness of LAV inhibition decreases slightly with immersion time after 2 h.
- The SEM analysis indicated that LAV essential oil acts by forming a protective film on the surface of carbon steel.
- The adsorption of LAV essential oil can be described by the Langmuir adsorption isotherm.

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