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Full Paper

Evaluation of a New Formulation Derived from *Aleurites moluccana* Seeds Oil as a Green Corrosion Inhibitor for Iron in Acidic Medium

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Abstract- The aim of this work is to characterize the seed oil of *Aleurites moluccana* and to develop a new formulation based on this oil (noted as "ALM"), which was used as a green corrosion inhibitor for iron in acidic solution. The fatty acid composition was determined using the chromatography gas GC/MS analysis. The Electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization measurements were carried out for different concentrations of the ALM formulation, immersion times and rotation speeds. The surface analyses was performed by scanning electron microscopy (SEM) coupled with the energy dispersive X-Ray analysis (EDX). The results showed that the seeds oil of *Aleurites moluccana* was principally composed of unsaturated fatty acids (Oleic, Linoleic and Linoleinic acids) reached a value of 88%. The inhibition efficiency increases with increasing of concentration to attain a value of 97% at 250 ppm of the ALM formulation and acts as a mixed type inhibitor. This efficiency is reinforced by increasing immersion time and the rotation speeds values of the working electrode.

Keywords- *Aleurites moluccana*, Formulation ALM, Corrosion inhibitor, Iron, Acid rainwater solution

1. INTRODUCTION

The use of plant oils extracts as corrosion inhibitors is one of the best way available to minimize the metallic corrosion in contact with aggressive media such as acid solution.

The adoption of this kind of inhibitors will be establish, the economic and environmental goals [1-4].

The *Aleurites moluccana* frequently called (kukui nut or candlenut) is native to Pacific islands and Southeast Asia regions. The tree can grow up to 20 m in height [5] and it grew throughout a tropical latitude. *Aleurites moluccana* can thrive in moist or dry climates but does not tolerate hard freezes that last more than a few days [6].

Aleurites moluccana has an ornamental, widespread branching system where flowers and subsequently, nuts are produced.

The nuts can have oil contents ranging from 45-65%, and this high oil content is likely behind the common name of the kukui tree: the candlenut tree [6].

This extractable oil used specifically in the cosmetics industry [7] for the production of biodiesel intended to seriously replace petroleum-based diesel [6-10], also used for lighting, for the manufacture of soap and including as the fast-drying waterproof oil for boats and paper. The remaining press cake of the extraction is used as fertilizer [5].

Untill now, there is no already reported studies evaluated the effect of *Aleurites moluccana* seeds oil used as a green corrosion inhibitor to protect the iron against corrosion in acidic medium.

Then the aim of this study is to evaluate the potential of the oil extracted from *Aleurites moluccana* seeds, developed as a new formulation, which can used as green corrosion inhibitor to protect the iron against corrosion in acid rainwater solution (pH=3.6).

The fatty acid composition of this seeds oil was determined using the chromatography gas GC/MS analysis and the inhibition efficiency was evaluated using electrochemical techniques. Indeed, to better understand the inhibiting mechanism of the formulation ALM against the iron corrosion in the acidic solution, we are interesting to study the effect of the concentration of the inhibitor, immersion time and the rotation speeds of the electrode.

The obtained results was confirmed by the surface analysis using Scanning Electron Microscopy (SEM) coupled with elemental chemical analysis (EDX).

2. MATERIAL AND METHODS

2.1. Oil Extraction processes

The fruits of *Aleurites moluccana* used in this study were bring from the Comoros islands. The nuts were crushed to obtain the seeds powder then extracted by a Soxhlet apparatus for 6 hours, using cyclohexane as solvent.

The organic phase was removing using a rotary evaporator. The obtained oil was filtered and stored at 4 °C.

2.2. Fatty acid analysis

Fatty acids were trans-esterified into methyl esters (FAME) [11], were identified by gas chromatography (GC3900), CP-Select CB for FAME fused silica WCOT (50 m×0.25 mm×0.25 µm film thickness), and equipped with flame ionization detector (FID). The flow rate of the carrier gas helium is 1.2 ml/min. The injector and FID temperatures were set at 250 °C. The initial column temperature is programmed to 140°C with 5 °C/min to 180 °C and kept 5 min at 180 °C, then 45 °C/min until 250 °C and kept 10 min at 250 °C. Peaks were identified by comparing retention times with those of standard fatty acid methyl esters.

2.3. Electrochemical measurements

2.3.1. Corrosive medium

The corrosive solution was a synthetic solution of acid rainwater, noted ″AR" correspond to acid rainwater in urban areas near seaside with a high degree of pollution.

This solution was prepared by the addition of 0.2 g/l of Na_2SO_4, 0.2 g/l of $NaHCO_3$ and 0.2 g/l of NaCl in distilled water, adjusted at pH=3.6 by the addition of a few drops of H_2SO_4 [2,12].

2.3.2. Corrosion inhibitor "ALM Formulation"

The corrosion inhibitor used in this study will be noted "ALM", indeed this later contained the seeds oil of the *Aleurites moluccana* prepared according to the process cited in the already patented work [13].

2.4. Electrochemical methods

The Iron samples used in this study was produced in the framework of the European PROMET project [14], thier chemical composition (Si: 0.201wt %; Mn: 0.519 wt %; C: 0.157 wt %; P: 0.007 wt %; S: 0.009 wt % and Fe>99 wt %) was determined by EDS analysis.
For the electrochemical tests, we used a traditional cell with three electrodes:

Reference electrode is a saturated calomel electrode (SCE), a large platinum surface as a counter electrode, and a working electrode of iron, coated in an impermeable resin, fixed on a revolving support. The rotating disk electrode (CTV 101) was used for the rotation speeds tests.

The potentiodynamic and the electrochemical impedance spectroscopy (EIS) techniques were carried out using a potentiostat model SP-200 apparatus, with a sweeping speed of 1 mV/s. In this work, the EIS measurement used the field of frequencies extends from the 100 KHz to 10 mHz.

2.5. Surface analysis

The morphology of the samples was recorded using FEI microscopy (model quanta FEG 450) and the surface characterization was evaluated by flash X-Ray (model 6130 Bruker brand) with an acceleration voltage of 20 KV.

This investigation techniques was realized at the Moroccan Foundation for Advanced Science Innovation and Research (MAScIR) Rabat - Morocco.

3. RESULTS AND DISCUSSION

3.1. Fatty acid composition of extracted oil

In this present study, we have found that the oil yield of the *Aleurites moluccana* seeds obtained after 6 h of extraction by the Soxhlet apparatus was 60.65%. This value is in agreement with that found in literature [5].

The fatty acid profiles for *Aleurites moluccana* seeds oil are presented in (Table 1) and compared with the results reported in the reference.

Table 1. Fatty acid composition of *Aleurites moluccana* seeds oil

Fatty acids	**Present study** **Amounts of fatty acid (%)**	**Ref.** [15]
$C_{12:0}$ Lauric	0.31	–
$C_{14:0}$ Myristic	0.22	–
$C_{16:0}$ Palmitic	6.13	5.51
$C_{18:0}$ Stearic	2.65	2.60
$C_{18:1}$ Oleic	22.33	15.5
$C_{18:2}$ Linoleic	37.80	42.9
$C_{18:3}$ Linoleinic	27.81	32.9
$C_{20:1}$ Gadoleic	0.48	–

The fatty acid composition of the seeds oil of *Aleurites moluccana* shows in (Table 1) contains a high quantity of polyunsaturated acids (Linoleic and Linoleinic acids) 65.61%, a moderate quantity of the mono-unsaturated acids (Oleic and Gadoleic acid) 22.81% and a little quantity of saturated acids principally a Palmitic acid with the presence of Stearic acid and a hint of Lauric and Myristic acids [15].

The total of the unsaturated fatty acids was around 88.42% (Oleic, Linoleic and Linoleinic acids). This high proportion indicates that the seeds oil of *Aleurites moluccana* as an unsaturated type.

3.2. Electrochemical tests

3.2.1. Open circuit potential (OCP)

The evaluation of the variation of the potential as a function of the immersion time is an important parameter, indicating the stability of the working electrode in the electrolyte and predicting the domain of the reaction of action of the inhibitor (anodic or cathodic) and determine threshold concentrations. The results of the open-circuit potential (OCP) variation of the iron substrate in acidic solution in the absence and presence of different concentrations of ALM formulation at 20°C are reported in (Fig. 1).

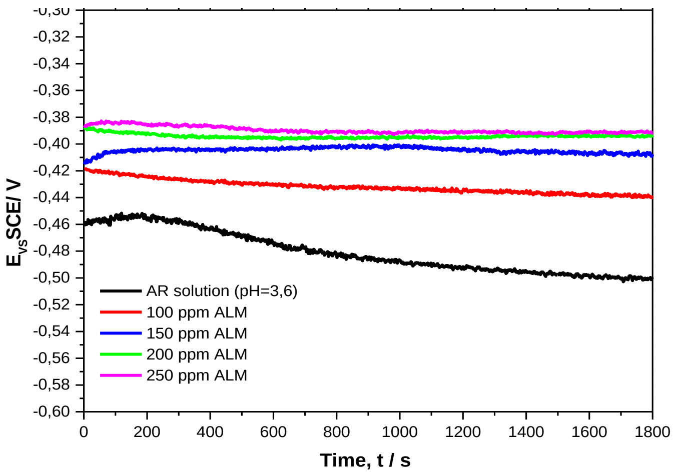

Fig. 1. Variation of the open-circuit potential of iron in acidic rainwater solution at different concentrations of ALM formulation

As shown in (Fig. 1), in the absence of ALM formulation, the potential tends to stabilize at -0.5 V, after a delay of 26 min. On the other hand, the addition of the ALM formulation results in a displacement of the corrosion potential to positive values, which increases with the increase of the inhibitor concentration. This significant change in corrosion potential may indicate the important anodic effect of the ALM formulation, as well as efficiency for the different inhibitor concentrations used in this study.

3.2.2 Concentration effect of the ALM formulation

3.2.2.1. Potentiodynamic polarization

The aim of these investigations is to determine the influence of the ALM formulation on the electrochemical behaviour of iron in AR solution at pH=3.6.

The polarization curves of the iron have been obtained after 30 min of immersion time in acidic solution without and with addition of various concentrations of the ALM inhibitor at corrosion potential E_{corr} and after performing the automatic Ohmic drop compensation (ZIR). These polarization curves are represented in (Fig. 2).

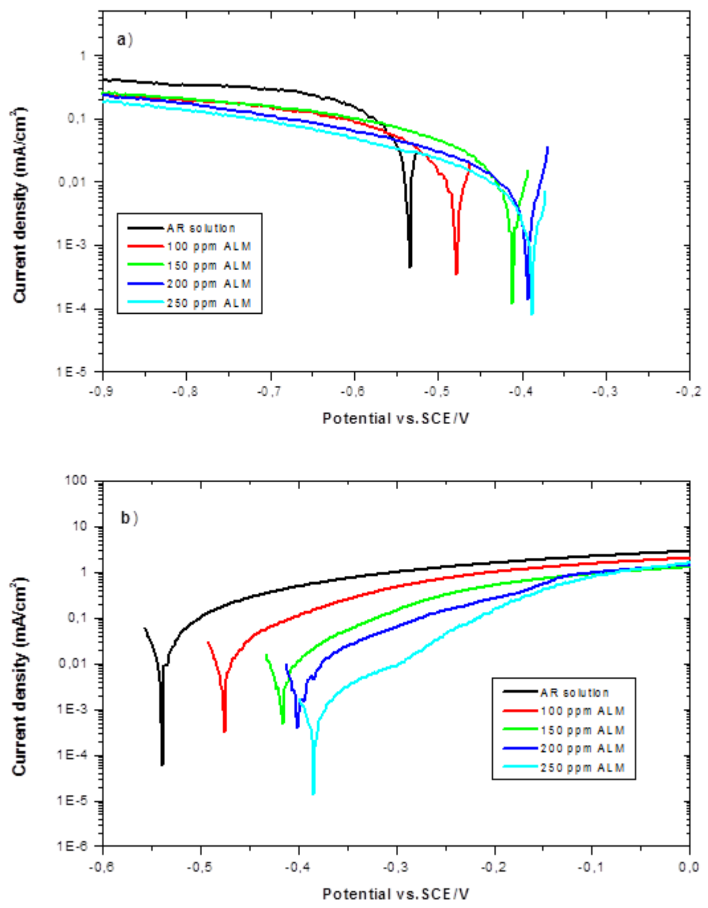

Fig. 2. Potentiodynamic polarization curves of iron in the AR solution at different concentrations of the ALM formulation in the cathodic **(a)** and the anodic **(b)** domains, respectively

As can been seen from (Fig. 2a), in the absence of the ALM inhibitor, the shape of the cathodic curve shows a current plateau, which can be attributed to the oxygen diffusion process [16], indicating that the mass transport is an important factor which must be considerate in the cathodic process [3,17], according to the following reaction [18]:

$$2H_2O + O_2 + 4e^- \rightarrow 4OH^-$$

Taken into account the presence of the hydrogen reduction effect:

$$2H^+ + 2e^- \rightarrow H_2$$

In the presence of the ALM formulation the cathodic current density values decreased, the diffusion plateau obtained in the cathodic domain, in the case of the blank essay disappeared gradually. We can report that to the ALM inhibitor addition in the AR solution leads to changing the cathodic behavior mechanism [3].

In (Fig. 2b), we can observed that the anodic current density value in the absence of the inhibitor increased significantly at the vicinity of the corrosion potential (E_{corr}) and it becomes slightly in the high anodic overvoltage, in the domain studied.

On the other hand, the addition of the ALM formulation in the corrosive medium leads to a decrease of the anodic current density values for all concentrations of the tested inhibitor. At 250 ppm of the ALM inhibitor. Furthermore, beyond -320 $mV/_{SCE}$, we noted an increase in the current density value, may be due to the initiation of pitting corrosion favoraited by the germination process [17].

However, the current density value remains relatively low compared to that observed in the absence of the ALM inhibitor. Therefore the presence of the protective film can prevents the propagation of pitting [2,17].

From the (Fig. 2a) and (Fig. 2b), we notice that both anodic and cathodic reactions of corrosion process were inhibited by the addition of the ALM inhibitor to the corrosive solution and the corrosion potential (E_{corr}) value displaced toward to the positive potential direction, may be indicates that the ALM inhibitor has an anodic effect more marked in the vincity of corrosion potential, even though, the cathodic current densities values notably also decreased [2]. The ALM formulation could be classified as a mixed type corrosion inhibitor with more pronounced to the anodic effect.

Table 2. Kinetic parameters determined from polarization curves of the iron substrate in the AR solution without and with a different ALM concentrations

ALM concentration (ppm)	E_{corr} (mV/SCE)	I_{corr} (μA/cm^2)	-βc (mVdec^{-1})	βa (mVdec^{-1})	IE (%)
Blank	-538	73.24	---	153	---
100	-477	26.09	243	125	64.4
150	-417	17.55	208	129	76.0
200	-401	7.14	140	104	90.3
250	-382	2.02	93	124	97.2

We noted that the corrosion current density was determined by the EC- lab software, after correction of the diffusion, by using the following relation [19,20]:

$1/I=1/I^{*}+1/I_L$

where: I: current density at mixed process

I*: corrected current density

I_L: limited current density

The kinetic parameters of the corrosion of the iron electrode are summarized in (Table 2).

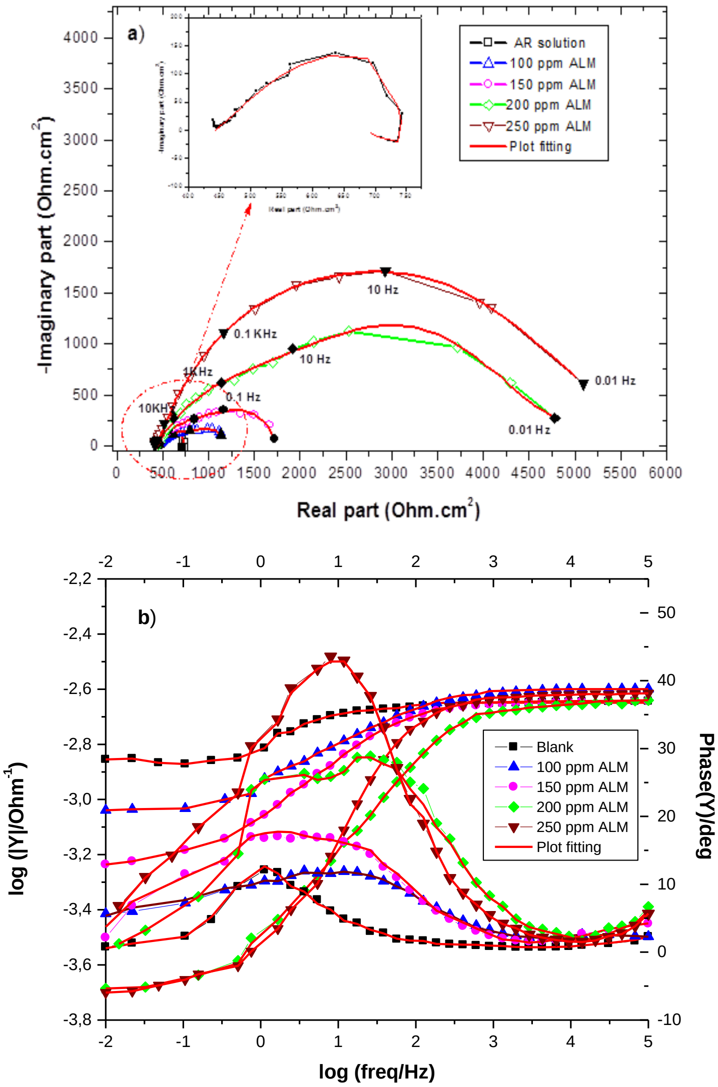

Fig. 3. Nyquist **(a)** and Bode **(b)** impedance plot of the iron substrate in the AR solution without and with addition of different concentrations of the ALM formulation at 20 °C

As shown in (Table 2), the current density I_{corr} values decreased with increasing of the concentrations of the inhibitor, while the inhibition efficiency increased to reach a maximum value of 97% at a concentration of 250 ppm.

3.2.2.2. Electrochemical Impedance Spectroscopy (EIS)

The inhibition property of the ALM formulation against corrosion of iron substrate in the AR solution at pH=3.6 was also examined by the electrochemical impedance spectroscopy technique (EIS) (Fig. 3).

As can been seen in the Fig. 3a, in the absence of inhibitor, the Nyquist diagrams indicated the presence of two capacitives loops. At the high frequencies range, the loop (R_{HF}, C_{HF}) can be attributed to the charge transfer. In the low frequencies range, the loop (R_{LF} C_{LF}) may be attributed to the diffusion phenomenon.

We noted also, the presence a small inductive loop in the low frequencies range indicated the beginning of the inductance effect [3,17], this loop may be attributed to the relaxation process obtained by adsorption of the elements present in the corrosive solution on the electrode surface. It may also be attributed to the re-dissolution of the passivated surface [21-23].

On the other hand, in the presence of the ALM formulation, the shape and size of the loops are more flattened, this later increases with the increase of the inhibitor concentration.

We notice the presence of two capacitives loops, the first loop observed in the high frequencies range was attributed to the formation of the film (R_f, C_f) and the second loop marked in the low frequencies range was attributed to the charge transfer (R_{dl}, C_{dl}), with disappearance of the inductive element (L) in the impedance spectrum. [2,3,17].

This means that there is a change of mechanism, as well as the corrosive ions responsible of this induction loop cannot be adsorbed on the metal surface and its removed by adding of ALM formulation to the corrosive medium.

At a higher concentrations of the ALM formulation, the two capacitives loops were overlapped, and become hardly distinguished.

These results are confirmed by Fig. 3b, represented the bode spectrum, show large amplitudes band of impedance spectrums and indicate the presence of more than one time constant. Also, the value of the angular phase increasing with increasing of the ALM inhibitor concentration.

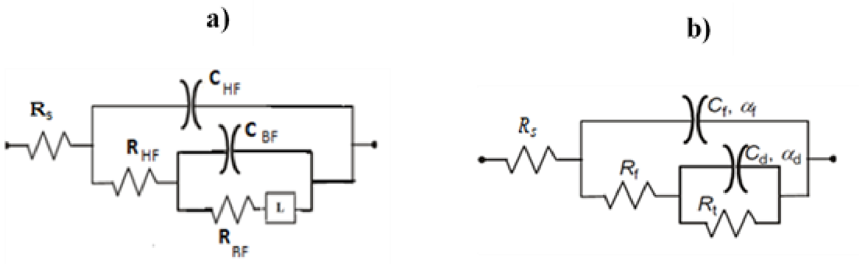

Fig. 4. Scheme of the equivalent electrical circuit used to fit EIS data for iron substrate **a)** In the blank solution (AR) and **b)** In the presence of the ALM formulation

According to these observations, we are proposed two equivalent electrical circuit (EEC), one in case in the absence and the other in the presence of ALM inhibitor, are presented in Fig. 4.

Electrochemical parameters resulting to the EIS diagrams associated to the EEC are shown in Table 3.

Table 3. Electrochemical data derived from EIS spectra for iron substrate in the AR solution at various concentrations of the ALM formulation

Blank	**R_s (Ohm.cm^2)**	**C_{HF} (µF/cm^2)**	**R_{HF} (Ohm.cm^2)**	**C_{BF} (µF/cm^2)**	**R_{BF} (Ohm.cm^2)**	**R_p (Ohm.cm^2)**	
AR (solution)	433	82.15	164	702.10	203	367	
ALM Concentrations	**R_s (Ohm.cm^2)**	**C_f (µF/cm^2)**	**R_f (Ohm.cm^2)**	**C_{dl} (µF/cm^2)**	**R_t (Ohm.cm^2)**	**R_p (Ohm.cm^2)**	**IE (%)**
100 ppm	448	36.69	367	493.43	428	794	53.8
150 ppm	445	26.04	517	254.24	830	1346	72.7
200 ppm	447	7.73	1739	75.58	2792	4531	91.9
250 ppm	424	6.92	1944	67.53	3124	5068	92.8

As indicated in the Table 3, the capacitance of the film C_f values decreases with increasing of the ALM inhibitor concentration, also the double layer capacitance (C_{dl}) has decreased, this decrease was probably due to the adsorption of the ALM formulation on the metal surface and the formation of a protective layer [2,3,17].

Moreover, we noted that the polarization resistance R_p values increase with an increase of the ALM concentrations, then the inhibition efficiency reached a maximum value of 93% at 250 ppm concentration of inhibitor.

3.2.3. Rotating disc electrode effect of the ALM formulation

In this investigation, we used the rotating disc electrode to evaluate the inhibiting effect of ALM inhibitor at concentration of 250 ppm, with different rotation speeds of the iron electrode. The rotation speeds range was selected between 0 and 1500 rounds per minute (rpm), at 20 °C. The cathodic and anodic polarization curves are reported in the (Fig. 5).

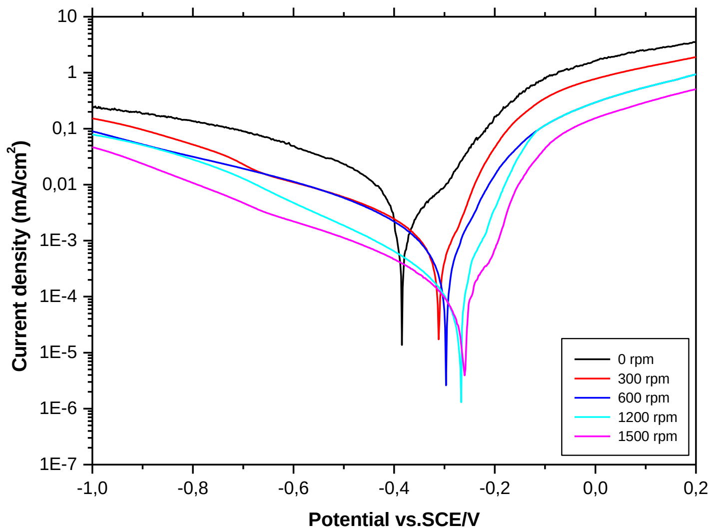

Fig. 5. Potentiodynamic polarization curves of iron substrate in the AR solution containing 250 ppm concentration of the ALM inhibitor, at various rotation speeds, after the automatic Ohmic drop compensation (ZIR)

As shown in the (Fig. 5), we observe that the increase of the rotation speed, leads a displacement of the corrosion potential E_{corr}, in the positive direction. The polarization curves in the cathodic and anodic parts are affected by the rotation speeds effect.

The electrochemical parameters derived from the potentiodynamic polarization curves were determined by the Ec- Lab software. The obtained results are summarized in (Table 4).

Table 4. Kinetic parameters obtained from polarization curves of iron substrate in the AR solution in the presence of 250 ppm of the ALM formulation at different rotation speeds

250 ppm of ALM Rotation speeds (rpm)	E_{corr} (mV/SCE)	I_{corr} (μA/cm^2)	-βc (mVdec^{-1})	βa (mVdec^{-1})	IE (%)
0	-384	2.03	89	128	97.2
300	-311	0.48	120	58	99.3
600	-297	0.34	122	58	99.5
1200	-268	0.12	176	47	99.8
1500	-260	0.07	178	55	99.9

We observed also, in the Table 4, the current densities values decrease significantly with the increase of the rotation speeds, consequently the inhibition efficiency values increased and reached a maximum value of 99.9% at 1500 rpm.

The (Fig. 6) represent the impedance spectra obtained at E_{corr} (corrosion potential), at 20 °C, in presence of 250 ppm of ALM inhibitor, a various rotation speeds.

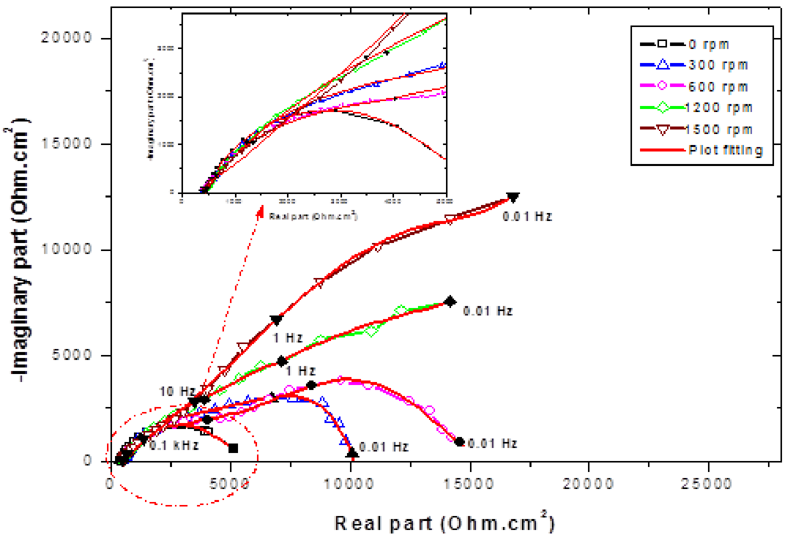

Fig. 6. Impedance diagrams of iron substrate in the AR solution at 250 ppm of the ALM formulation at different rotation speeds

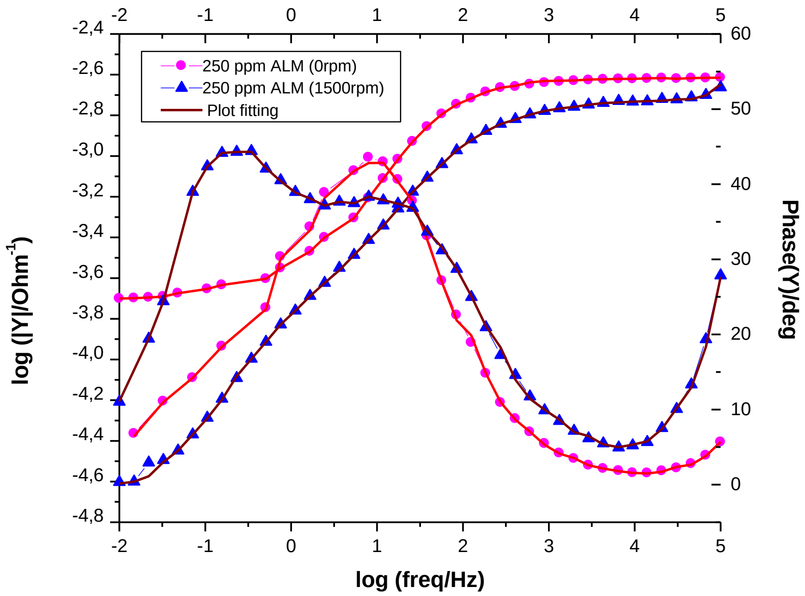

Fig. 7. Bode diagrams for iron in AR medium, in the presence of 250 ppm of ALM, under the hydrodynamic condition of rotating disk electrode

The inspection of the impedance diagrams (see Fig. 6) reveals that the two capacitive loops became more distinguish by the rotating disc electrode effect and the size of these capacitive loops increase significantly by increasing of the rotation speeds. These results are supported by those shown in the Fig. 7.

The electrochemical parameters associated to the EIS spectra extracted by using the experimental fitting Ec-Lab program, are given in (Table 5).

Table 5. Electrochemical data derived from the EIS spectra of iron substrate in the AR solution containing 250 ppm of the ALM inhibitor at different rotation speeds

Rotation speeds (rpm)	**R_s (Ohm.cm^2)**	**C_f (µF/cm^2)**	**R_f (Ohm.cm^2)**	**C_{dl} (µF/cm^2)**	**R_t (Ohm.cm^2)**	**R_p (Ohm.cm^2)**	**IE (%)**
0	424	6.92	1944	67.53	3124	5068	92.8
300	421	5.45	3727	52.00	6006	9733	96.2
600	412	3.15	6318	39.64	7878	14196	97.4
1200	442	2.89	6896	25.50	18156	25052	98.5
1500	444	2.73	7284	17.14	27002	34286	98.9

The results reported in (Table 5), showed that the values of the film and the charge transfer resistances (R_f and R_t) increased respectively with an increase of the rotation speeds. While the film and the double layer capacitances (C_f and C_{dl}) values decreased simultaneously to with the increase of the inhibiting effect, due to the impact of the rotating disc electrode [17].

As previously discussed for the polaraization curves and the impedance diagrams, we found that the rotation speeds effect can improve the inhibition efficiency of the ALM formulation, probably due to the movement of the molecules from the electrolyte towards the surface of the electrode, which help the adsorption process of these constituents and the formation of a protective layer against corrosion of iron substrate in acidic medium [23].

3.2.4. Immersion time effect of the ALM formulation.

The Fig. 8 presents the effect of the immersion time on the impedance spectra at the corrosion potential E_{corr}, at the static conditions, in the presence of 250 ppm of the ALM formulation, at 20 °C.

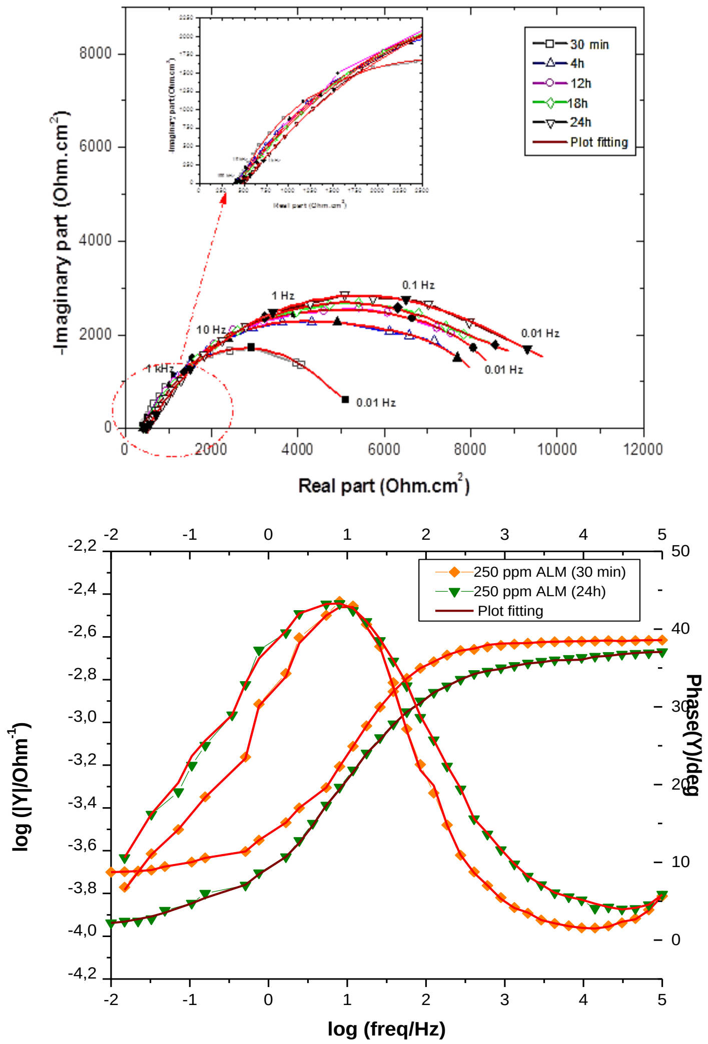

Fig. 8. a) Nyquist impedance diagrams of iron electrode in the AR solution containing 250 ppm of the ALM formulation at different immersion time; **b)** Bode diagrams for iron in AR solution, containing 250 ppm of ALM, under the time immersion

Table 6. Electrochemical impedance parameters determined from Nyquist plot of iron substrate in the AR solution containing 250 ppm of the ALM inhibitor at different immersion time

Immersion time	R_s (Ohm.cm^2)	C_f (μF/cm^2)	R_f (Ohm.cm^2)	C_{dl} (μF/cm^2)	R_t (Ohm.cm^2)	R_p (Ohm.cm^2)
30 min	424	6.92	1944	67.53	3124	5068
4h	454	6.45	2087	34.53	6110	8197
12h	452	5.91	2275	30.30	6964	9239
18h	452	5.47	2460	28.68	7356	9816
24h	476	5.12	2627	26.58	7936	10564

The electrochemical impedance parameters derived from this investigation are given in Table 6.

From the Fig. 8. a) and Fig. 8. b), we found that the shape of these curves is very similar to that obtained by the effect of the inhibitor concentration (see Fig. 3.a) and Fig. 3.b)), as well as the size of the curves increases with increasing of the immersion time.

As shown in (Table 6), the capacity of the C_f film remains partially constant and the C_f values are less than 10 µF/cm^2 [3,24-26]. The resistance of the film R_f increases slightly with increasing of immersion time, as well as the polarization resistance R_p (determined at the low frequency limit of the impedance spectrum) doubles and vary from 5.07 kΩ.cm^2 after 30 min to 10.56 kΩ.cm^2 after 24 h of immersion time.

These results reveal the protective effect of the ALM formulation and indicate that the thickness of the film seems to be enhanced by the immersion time [2,3,4].

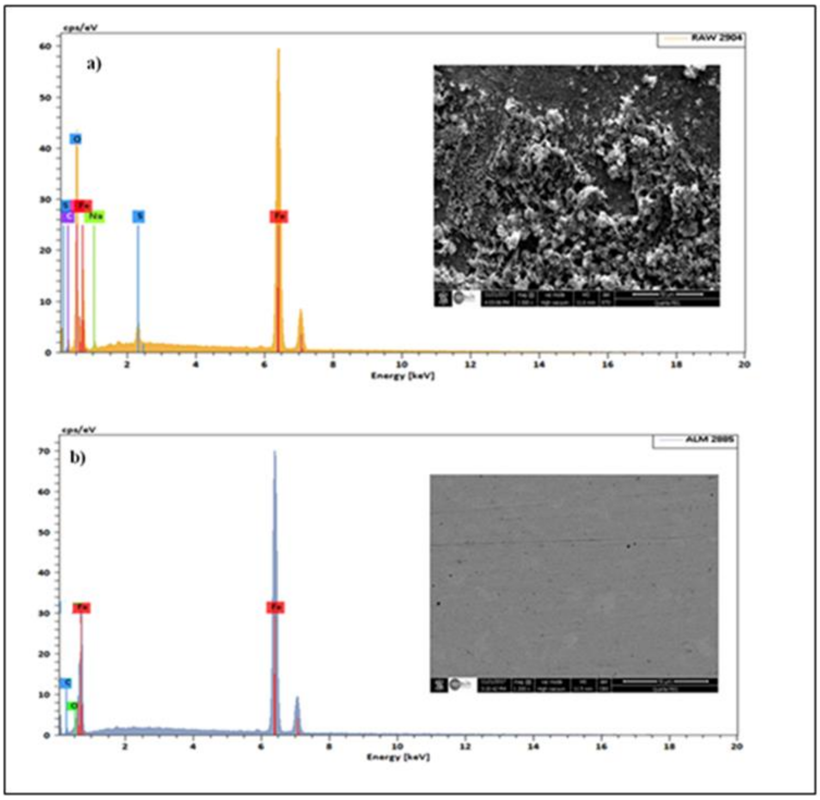

Fig. 9. SEM micrographs and analysis Spectrum EDX of the iron surface in the AR solution: **a)** without and **b)** with 250 ppm of the ALM inhibitor, after 24 h of immersion time

3.3. Surface analysis (SEM/EDX)

In order to provide insight into the state of the iron surface with the best concentration of the inhibitor and to analyses the chemical nature of the inhibitor/iron interface, the scanning electron microscopy (SEM) coupled with the energy dispersive X-Ray analysis (EDX) were carried out on the iron substrate surface after 24 hours of immersion time in acidic solution in the absence (Fig. 9a) and in the presence of 250 ppm of ALM formulation (Fig. 9b).

As can be seen in (Fig. 9a), in the absence of the ALM inhibitor, the metal surface of the iron is very corroded, prove a severe attack by the AR solution [2,3,17]. The SEM micrograph revealed the presence of small crystalline globules (sandy crystals) and the appearance of the fine plates ('flowery' structures) typical of lepidocrocite (γ-FeOOH), a component of rust developed in the atmospheric corrosion conditions [27].

A qualitative analysis carried out by the EDX analysis marked the presence of elements characterizing of the AR solution such as (O, Na and S) identified on the iron substrate surface.

In the presence of the ALM inhibitor (Fig. 9b), the SEM micrograph revealed a uniform and smooth metal surface due to the formation of the inhibitor film.

This result was confirmed by the EDX spectrum showing the decreases of oxygen amount at the metal surface, as well as the disappearance of the chemical elements from the corrosive medium.

4. CONCLUSION

In this study the determination of the fatty acid composition of the seeds oil of *Aleurites moluccana* showed that it’s a long chain unsaturated oil, attains a value of 88% of unsaturated fatty acids. The potentiodynamic and the electrochemical impedance measurements results indicated that the ALM formulation acts as a mixed type inhibitor with the predominance of the anodic efficiency. The inhibition efficiency increases with increasing of the ALM concentration and reached a maximum value of 97% at 250 ppm. This inhibiting efficiency was improved with immersion time and rotation speeds effect. The good protective effect of the ALM formulation is attributed to the formation of a thick film that act as a barrier layer on the iron surface to minimize the contact area with corrosive solution and hinder metal oxidation. This result was confirmed by SEM analysis coupled with the EDX. Therefore, the ALM formulation is a good green corrosion inhibitor of iron in acid rainwater solution, which is an ecological environmentally safely product.

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