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Evaluation of Inhibitory Effect of the Methanolic Extract of the Two Parts from Anabasis Aretioides Plant Against the Corrosion of E 24 Steel in a Neutral Solution NaCl 3.5%

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Abstract- In this study, the photochemical compositions of the methanolic extracts of the leaves and root parts from the *Anabasis aretioides* were determined, as well as the evaluation of their effect as a green corrosion inhibitor of mild steel (E24) in a neutral solution (NaCl 3.5%). The photochemical screening performed for the methanolic extracts showed the presence of the polyphenols, tannins, alkaloids, reducing compounds and trace amounts of the proteins, carotenoids, coumarins and cardiac glycosides. The electrochemical study was carried out by using the polarization measurements and electrochemical impedance technique. The analyzing of the surface of the metal was carried out by the scanning electron microscope (SEM) coupled with the EDS. The inhibition efficiency increased with increasing the concentrations and reached the values of 86 and 87% respectively for the 10% concentration of methanolic extracts from the leaves and root parts of the *Anabasis aretioides* plant. The both inhibitors act as a mixed type, with the predominance of the cathodic efficiency for the leaves extract and the anodic efficiency for root extract.

Keywords- *Aretioides Anabasis*, The methanol extract, Mild steel E24, Electrochemical measurements, SEM, Photochemical screening

1. INTRODUCTION

The natural products extracted from plants have a great interest, used as raw material and intended for different industries such as cosmetics, pharmaceuticals, food and industry [1].

The use of plants for therapeutic purposes are reported in the ancient Arabic literature, Chinese, Egyptian, Hindu, Greek, Roman [2]. Their therapeutic power has been known by our ancestors empirically, however, all we knew about the chemical composition of the drugs used every day by many people, for health care, remains narrowed [1].

By combining the scientific research on the active ingredients to the clinical observation and traditional knowledge of plants, it is possible to make an inventory of their therapeutic uses [3].

Moreover, in addition to biological activities of plants, over the past few years, the research attention has focused on the anti-corrosive properties of natural plant products [1].

Indeed, as to the use of increasingly growing metals and alloys in modern life, the corrosion phenomena is a huge problem whose consequences are harmful and invaluable. The use of inhibitors is one of the most practical methods for protecting metals against corrosion in particular in similar solutions to seawater [4,5].

Thus, non-toxic and biodegradable properties of natural products have led to their use as a green corrosion inhibitors called "Friend green inhibitor of the Environment" [6-11].

The natural products such as a plant extracts compounds seem to be an ideal ecological alternative to replace traditional corrosion inhibitors [12].

*Anabasis aretioides*is is a local medicinal plant, belonging to the family of amarantacées, the selection of this species is the result of an ethnobotanical survey by myself [13].

This plant is commonly called sella, lkarma lima ydiha rih or cauliflower Bouamama. Forming compact tufts which hemispherical prevents to exceed 50 cm of diameter, constituted by branches tight against each other and which the interstices are filled with sand (Figure 1), the leaves of *Anabasis aretioides* are opposite, very tight, shabby, hard and each terminated by a spine, however the flowers are inconspicuous, by two or three at the top of branches [14]. Common in the Sahara Oran and in the regions of Beni-Unif and Igli especially rare eastward in areas of Laghouat, Biskra and Touggourt *Anabasis aretioides* lives on hard regs where it often forms extensive stands [15,16].

Fig. 1. *Anabasis aretioides* plant

Based on a literature survey, no previous work has been reported on the corrosion inhibition of *Anabasis aretioides* methanolic extracts for mild steel in a neutral solution (NaCl 3.5%).

Hence, in this work, after performing a phytochemical screening of the methanolic extract of both parts (leaf and root) of *Anabasis aretioides*, we are interested to study their inhibitory effect against the corrosion of mild steel (E 24) in neutral medium (NaCl 3.5%) by the use of the stationary and transient electrochemical methods.

For more performance, we carried out the surface characterization of mild steel samples by using a scanning electron microscopy (SEM) coupled with the energy dispersive spectroscopy (EDS).

2. MATERIAL AND METHODS

2.1. Plant material

The *Anabasis aretioïdes* plant used in this study, was harvested in August during the flowering period, at the region of Errachidia. The both parts of the plant Anabasis aretioides (root and leaf) were rinsed and dried in a dry, airy sheltered from the light then crushed using a cutter mill to obtain a powder.

2.2. Preparation of Extract

The methanolic extracts of the two parts leaf and root of the plant *Anabasis aretioides* were prepared by maceration of 5 g of the powder in 40 ml of methanol for 24 h with stirring for 15 minutes at the beginning, at a temperature of 25 °C. Then the mixture was filtered by filter paper and the extracts obtained are stored at 4 °C in sealed vials.

2.3. Screening phytochemical

The phytochemical screening is a qualitative analyzis, used in this study to indicate the presence of the different families of secondary metabolites existing and present in the both metanolic extracts from the leaves and root parts of the *Anabasis aretioides* plant.

This characterization is based principaly on the precipitation phenomena or on the reaction stainings using reagents specific to identified each family of compounds [17,18].

2.3.1. Test for Polyphenols

The reaction with ferric chloride ($FeCl_3$) permitted to characterize the presence of polyphenols, by the addition a drop of 2% ferric chloride alcoholic solution to 2 ml of the methanolic extract.

The appearance of a more or less dark blue-blackish or green coloration, indicate the presence of polyphenols compounds [19].

2.3.2. Test for Tannins

In a tube, we introduced 5 ml of methanolic extract and we added 1 ml of aqueous solution of $FeCl_3$ at 1%. the development of a blackish or greenish blue color indicate the presence of tannins.

However, the Stiasny reaction indicates the presence of catechin tannins and the addition of sodium acetate may confirm the presence of gallic tannins [20].

2.3.3. Test for Coumarins

5 ml of methanolic extract was evaporate in the open air, then we added to the residue obtained, 2 ml of hot water.

The solution was shared between two test tubes. Then we added to one of them a volume of 0.5 ml of ammonia diluted to 25% and we observed the fluorescence under UV 366 nm. The appearance of fluorescence in the tube, where ammonia was added indicates the presence of coumarins [18].

2.3.4. Test for Flavonoids

a) Cyaniding

1 ml of methanolic extract was added to 1 ml of hydrochloric alcohol and to 1 ml of isoamyl alcohol solution, then we introduced into a tube some magnesium chips.

The Cyanding reaction occurred for a few minutes and the appearance of the isoamyl alcohol supernatant layer of orange rose coloration indicates the presence of flavones, purple rose coloration characterizes the flavanones and a red coloration showed the presence flavanones and flavanonols.

b) Anthocyanins

This test is performed by adding 5 ml of methanolic extract to 5 ml of sulfuric acid and to 5 ml of NH_4OH introduting in a test tube. the color deepens by acidification in the presence of anthocyanins and then turns purplish blue in a basic medium.

c) Leucoanthocyanins

As in the case of the cyaniding reaction, we heated the mixture in a water bath for 15 minutes but without the addition of magnesium chips.

On the one hand, in the presence of leucoanthocyane a cherry red color with purplish was developed, on the other hand the red-brown color indicates the presence of catechol [21].

2.3.5. Test for Alkaloids

This test is carried out by precipitation reaction using the Dragendorff reagent. 1 ml of extract introduced into test tubes and we added 5 drops of Dragendorff reagent. the appearance of an orange precipitate, is found to reveal the presence of alkaloids [22].

2.3.6. Test for Reducing compounds

1 ml of the methanolic extract was introduced into a test tube with addiding a 2 ml of Fehling solution, then the tubes was heated in the water bath at 90 °C. Obtaining a brick red precipitate indicates the presence of reducing compounds [22].

2.3.7. Test for Protein

The Biuret reaction allowed us to identify the protein, therfore we performed a maceration of 1 g of powder samples in 10 ml of methanol for 24 h in the darkness, then we filtered and we added to the aliquot of the residue dissolved in 2 ml of NaOH aqueous solution at 20%, 2 to 3 drops of $CuSO_4$ aqueous solution at 2%.

The appearance of a purple color, sometimes with a reddish hue, indicating the presence of protein [23].

2.3.8. Test for Carotenoids

After the evaporation of 5 ml of methanolic extract until dry, we added 2 to 3 drops of antimony trichloride ($Sbcl_3$) saturated in a chloroform solution. The presence of carotenoids is indicated by a blue color toned red [18].

2.3.9. Test for Glycosides cardiac

2 ml of chloroform has addition to 1 ml of the methanolic extract, the appearance of a brown-reddish coloring after the addition of H_2SO_4 indicates the presence of the cardiac glycosides [24].

2.4. Electrochemical measurements

The electrochemical measurements were carried out using three-electrode setup comprised of a counter electrode (large platinum surface), the reference electrode (saturated calomel one (SCE)) and a working electrode (mild steel which had a cylindrical form and the surface area of 1 cm^2, coated in an impermeable resin), The samples of mild steel used in this study have a composition indicated in Table 1.

Table 1. Mild steel composition of the samples used in this study [25]

Elements	**Si**	**Mn**	**C**	**Cr**	**Ni**	**Mo**	**Cu**	**Al**	**Fe**
Weight (%)	0.24	0.47	0.11	0.12	0.1	0.02	0.14	0.03	98.7

The sample surface was polished using abrasive paper grades until 1200. Then, it was cleaned by the acetone and distilled water and dried at room temperature before dipping to corrosion solution.

The corrosive solution was a simulated solution of seawater; it is prepared by the addition of 35 g of one liter NaCl in distilled water.

The corrosion inhibitor used in this study was the methanolic extracts of leaves and root from *Anabasis Aretioïdes* plant. The methanolic extracts concentration was varied from 1, 5, 7 and 10% v/v in 100 ml of 3.5% wt NaCl solution.

The polarization and electrochemical impedance spectroscopy (EIS) measurements were carried out by using a Bio-Logic (SP-200) apparatus connected to a computer, with a sweeping speed of 1 mV/s and 10 mV of amplitude. The time limit for reaching equilibrium potential before doing electrochemical measurement was 30 minutes, In the electrochemical impedance tests, the frequency spectrum range extends from 100 KHz to 10 mHz at the open circuit potential (OCP).

2.5. Surface characterization by SEM-EDS

The morphology and a chemical composition of the surface of E24 steel substrates immersed for 24 h in a solution of NaCl 3.5%, without and with 10% v/v of leaves and roots methanolic extracts from *Anabasis Aretioïdes* plant, were investigated by scanning electron microscopy (SEM) coupled with the energy dispersive spectroscopy (EDS).

The images were recorded using a HITACHI S-3200N model scanning microscope and the surface morphology was evaluated by Thermoscientific -Ultradry Energy Dispersion Spectroscopy with an acceleration voltage of 20 kV.

3. RESULTS AND DISCUSSION

3.1. Identification of the active compounds in the methanolic extract of the two parts of the plant

The results of the phytochemical tests of both the methanolic extract of the *Anabasis aretioides* plant parts, are summarized in a Table 2.

Table 2. Identification of the polyphenols, tannins, coumarins, flavonoids, alkaloids, proteins, carotenoids, cardiac glycosides and reducing compounds in the methanolic extract of the *Anabasis aretioides* plant.

Research	*Anabasis aretioides*	
	Leaf	**Root**
polyphenols	++	++
tannins	++	++
catechin tannins	++	++
gallic tannins	+++	+++
coumarins	+	-
anthocyanins	-	-
genins flavonoid	-	-
leucoanthocyanins	-	-
catechols	-	-
alkaloids	++	++
reducing compounds	+	++
protein	+	+
carotenoids	+	+
cardiac glycosides	+	+

+++: Strongly positive; ++: Moderately positive; +: Weak Positive; -: Negative. According to Table 1, we note the presence of polyphenols and alkaloids in both parts of the plant Anabasis aretioides, as well as their average

The gallic tannins are present in larger quantities than those of catechin tannins, which have smaller.The coumarins, reducing compounds, proteins, carotenoids and cardiac glycosides are weakly present in both parts of the plant. We also notice a complete lack anthocyanins, buffy anthocyanins, flavonic genins and catechols in leaf and root parts of the *Anabasis aretioides* plant. These families of detecting compounds are the same as those revealed and found in the work of Bentabet-Lasgaa et al. [26]

3.2. Electrochemical Measurements

3.2.1. Open circuit potential

The variation of potential with time was determined of the working electrode in a solution in absence and presence of different concentrations of leaves and root methanolic extracts from *Anabasis Aretioïdes* plant. Results are reported in Fig. 2.

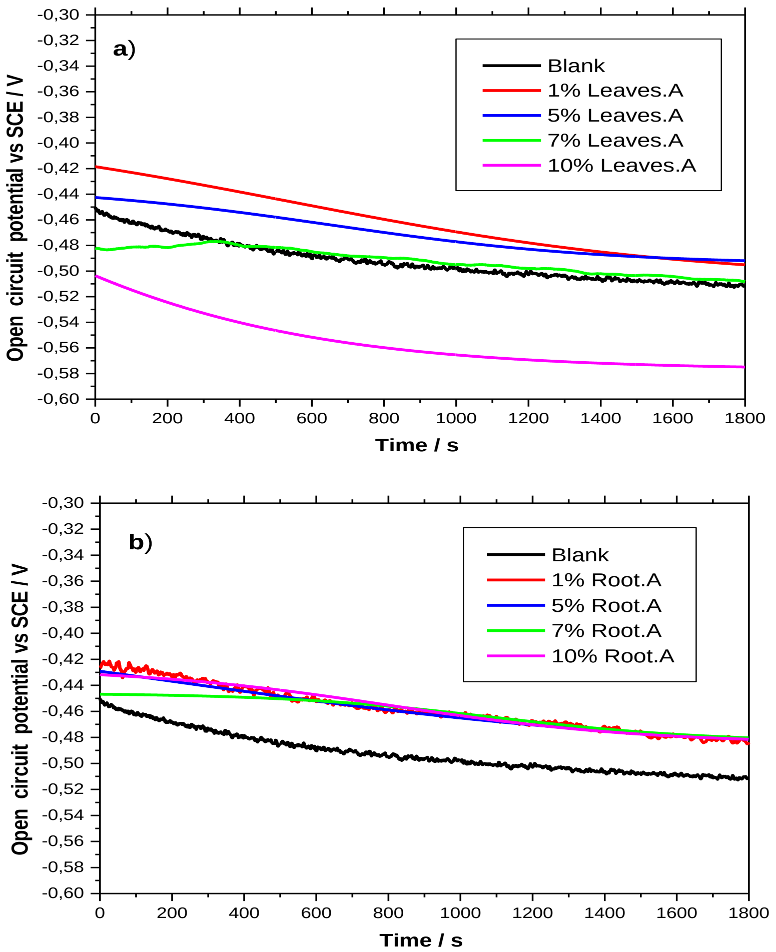

Fig. 2. Variation of the open-circuit potential (OCP) of the mild steel substrate in a neutral solution at various concentration of a) leaves methanolic extract and b) root methanolic extract from *Anabasis Aretioïdes* plant

As shown in Fig. 2, in absence of the inhibitors the potential tends to stabilize at -0.507 $V/_{SCE}$, after 25 min. The addition of the ro ot methanolic extract (see Fig. 2b) leads to a shift corrosion potential to positive direction and the potential tends to stabilize in the same potential value at -0.478 $V/_{SCE}$ for all concentration of extrait. Furthermore, for the leaves methanolic extract(Fig. 2a), at the beginning the corrosion potentiel is located in the positive values, but with the increase of the extract concentration, the potentiel decreased by heading to the most negative values, then stabilized at -0.572 $V/_{SCE}$, after 25 min for concentration of 10% of leaves methanolic extract from *Anabasis Aretioïdes* plant.

This important shift of corrosion potential may indicate an important anodic inhibiting effect of root methanolic extract and cathodic inhibiting effect of leaves methanolic extract from *Anabasis Aretioïdes* plant.

3.2.2. Potentiodynamic measurements

The cathodic and anodic polarization curves of mild steel substrate in the NaCl 3.5% solution in the absence and in the presence of different concentrations of the methanol extracts from leaves (Fig. 3a) and roots (Fig. 3b) of *Anabasis Aretioïdes* plant are respectively presented in Fig. 3.

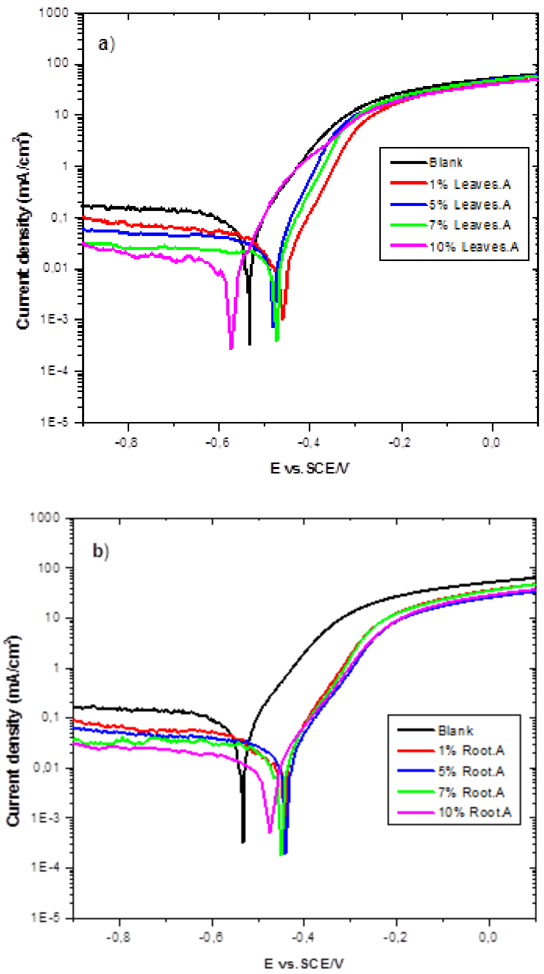

Fig. 3. Potentiodynamic polarization curves for mild steel in 3.5% NaCl solution without and with different concentrations of a) leaves and b) root methanolic extracts from *Anabasis Aretioïdes* plant

Fig. 3 shows that in the absence of inhibitor, the current density increases rapidly in the vicinity of the corrosion potential. This increase was fast and attained the current limit diffusion level, giving a current plateau, which is attributed to the diffusion limit of the current dissolved of oxygen [5,27] in the cathodic domain.

In the presence of inhibitor, it can be clearly seen that the addition of the extracts decreases the current density in the both cathodic and anodic domains. However, the corrosion potential

E_{corr} value for electrode without inhibitors was -532 mV/SCE and it shifted to -573 mV/SCE for leaves extract and -473 mV/SCE for root extract at concentration of 10%.

Even if, the anodic and cathodic corrosion reactions of mild steel electrode were inhibited by increasing the concentration of the inhibitors, the addition of the leaves extract reduced the cathodic current density with a slight affection of the active anodic dissolution in the polarization curves. Contrary to the addition of the root extract which caused a more marked change in the anode branches. Therefore, the leaves extract could be classified as a mixed type inhibitor with the predominant of the cathodic efficiency and the root extract could be also classified as a mixed type inhibitor, but with predominant of the anodic efficacy [28].

The values of the current density corrosion (I_{corr}), the corrosion potential (E_{corr}), the Tafel slopes of cathodic (βc) and anodic (βa) and the inhibiting efficiency (IE %) for different concentrations of the two extracts are given in Table 3.

The inhibition efficiency IE% was calculated from polarization measurements according to equation [29]:

$$IE\ (\%) = \frac{I^0_{corr} - I^{inh}_{corr}}{I^0_{corr}} \times 100$$

Where I^0_{corr} and I^{inh}_{corr} are without and with inhibited corrosion current densities, respectively.

Table 3. Electrochemical parameters from polarization measurements on mild steel in 3,5% NaCl without ant with different inhibitors concentrations

Blank	**E_{corr} (mV/SCE)**	**I_{corr} (μA/cm^2)**	**βc (mV/dec)**	**βa (mV/dec)**	**IE (%)**
NaCl 3.5%	-532.24	42.05	-225.4	77.5	-----
Leaves of *Anabasis. A*					
1%	-459.76	12.04	-165.2	60.8	71.37
5%	-479.90	10.47	-109.1	44.0	75.10
7%	-474.44	9.69	-165.7	48.6	76.96
10%	-572.97	5.52	-146.6	59.8	**86.87**
Root of *Anabasis. A*					
1%	-442.43	11.07	-209.8	55.4	73.67
5%	-439.105	10.21	-154.5	66.6	75.72
7%	-450.02	8.34	-155.5	59.6	80.17
10%	-473.35	6.76	-257.5	74.8	**83.92**

As shown in the table 3, the values of I_{corr} decreased by the addition of the extracts to the corrosive medium, from 42.05 in the absence of inhibitor to 5.52 and 6.76 µA/cm^2 in the presence of 10% for leaves and root extracts, respectively. This decrease of I_{corr} values can be due to the adsorption of molecules the extract on the mild steel surface.

The inhibition efficiency increased with an increase of the inhibitors concentrations, although it differs slightly from one extract to another, we can noted that the inhibition efficiency value reaching of 87 and 84% at 10% concentration of leaves and root extracts from *Anabasis Aretioïdes* plant, respectively. So, the inhibition effect of the methanol extracts is more pronounced at 10% of leaves extract.

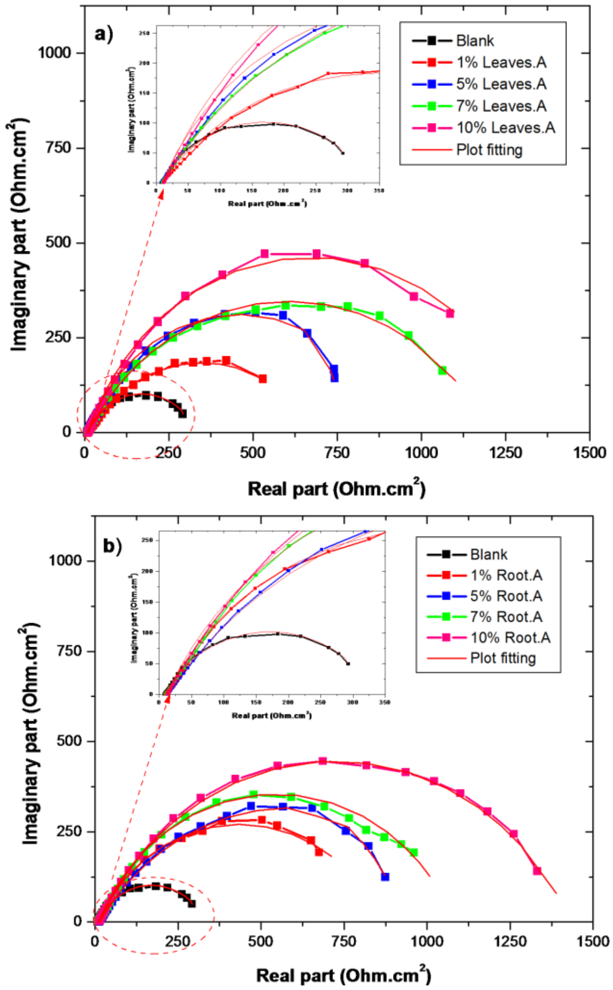

Fig. 4. Nyquist plots recorded for mild steel substrate in 3.5% NaCl medium in the absence and the presence of various concentrations of a) leaves methanolic extract and b) root methanolic extract, parts from *Anabasis Aretioïdes* plant

3.2.3. Electrochemical impedance spectroscopy measurements

The electrochemical impedance spectroscopy (EIS) measurements, at the corrosion potential, have been used to evaluate the inhibition effect of the inhibitors, on the corrosion of mild steel in 3.5% NaCl solution.

The Nyquist plots obtained, for the electrode in the seawater solution without and with the leaves extract (Fig. 4a) and root extract (Fig. 4b) from *Anabasis Aretioïdes* plant, after 30 min of the immersion time, at different concentrations, are presented in Fig. 4.

As shown in the Fig. 4, we noted that the diameter of the capacitive loop was affected by the addition of inhibitors and increasing with increases of the extracts concentrations, and in all concentrations used for both extracts, we observed the presence of a single capacitive loop (one time constant), corresponding to the charge transfer resistance [30].

These results are confirmed by Fig. 5, represented the bode spectrum for mild steel in 3.5%NaCl solution, in the absence and in the presence of leaves extract and root extract from Anabasis Aretioïdes plant, at concentration of 10%.

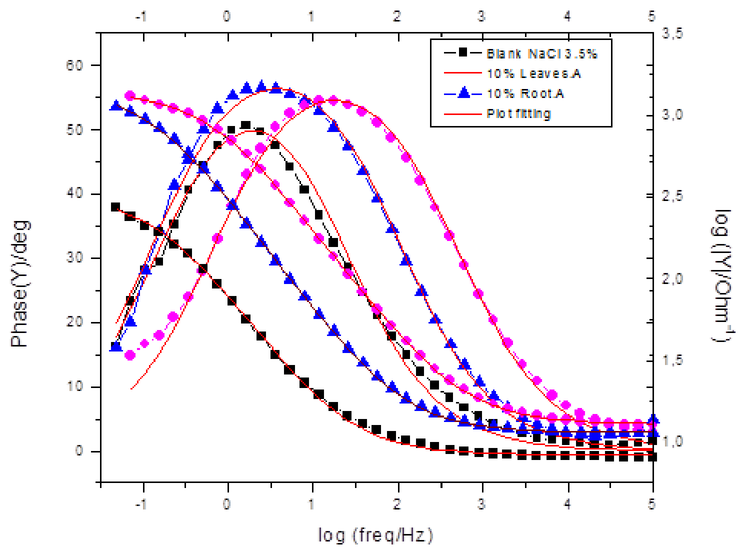

Fig. 5. Bode plots for mild steel in 3.5% NaCl medium without and with 10% of leaves extract and root extract from *Anabasis Aretioïdes* plant

According to Fig. 5, the Bode plots for the mild steel in the presence of inhibitors show higher impedance magnitudes at the low frequency than in the absence of inhibitors [31]. Moreover, in the phase angle, when adding the inhibitors, the range of circles is increased. This means improvement of the corrosion resistance of the metal in solution of 3.5% NaCl [28].

Therefore, the fitting of all electrochemical impedance spectroscopy (EIS) data was performed using a simple equivalent electric circuit model (shown in Fig. 6), which is typically used for bare and coated mild steel [32]. This model is characterized by a solution resistance

(R_s), a double layer capacitance (C_{dl}) and a charge transfer resistance (R_t) of the faradaic process.

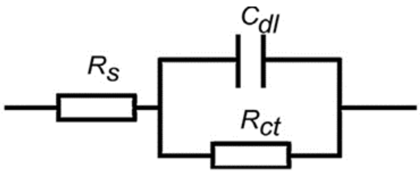

Fig. 6. the equivalent circuit model used to fit EIS experiment data

The data obtained from fitted spectra are listed in Table 4, in which, the inhibition efficiency (IE%) from the impedance curves is calculated using the following equation [33]:

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

Where R^{inh}_t and R_t are the charge transfer resistance values in the absence and in the presence of the inhibitor, respectively.

Table 4. Impedance parameters of mild steel in the 3.5% NaCl medium in absence and in presence of different concentrations of the leaves and root methanolic extracts from *Anabasis Aretioïdes* plant

Blank	**R_s (Ohm.cm^2)**	**f_{max} (Hrz)**	**C_{dl} (mF/cm^2)**	**R_t (Ohm.cm^2)**	**IE (%)**
NaCl 3.5%	8.53	0.1058	4.73	318	----
Leaves of *Anabasis*					
1%	8.89	0.1058	2.252	668.15	52.40
5%	9.33	0.3439	0.543	851.75	62.66
7%	9.34	0.3439	0.388	1193.15	73.35
10%	9.33	0.3439	0.349	1327.86	**76.05**
Root of *Anabasis*					
1%	10.33	0.1568	1.250	812.52	60.86
5%	13.78	0.1568	1.052	965.1	67.05
7%	9.33	0.7547	0.197	1070.91	70.30
10%	9.34	0.7547	0.151	1399.86	**77.28**

As indicate in the table.4, we noticed that the charge transfer resistance value increases with increase of percentages concentration for the both extracts. R_t rises from 318 Ohm.cm^2

value [34] in the absence of inhibitors to 1328 and 1400 Ohm.cm^2 values with addition of 10% of the leaves and root extracts, respectively.

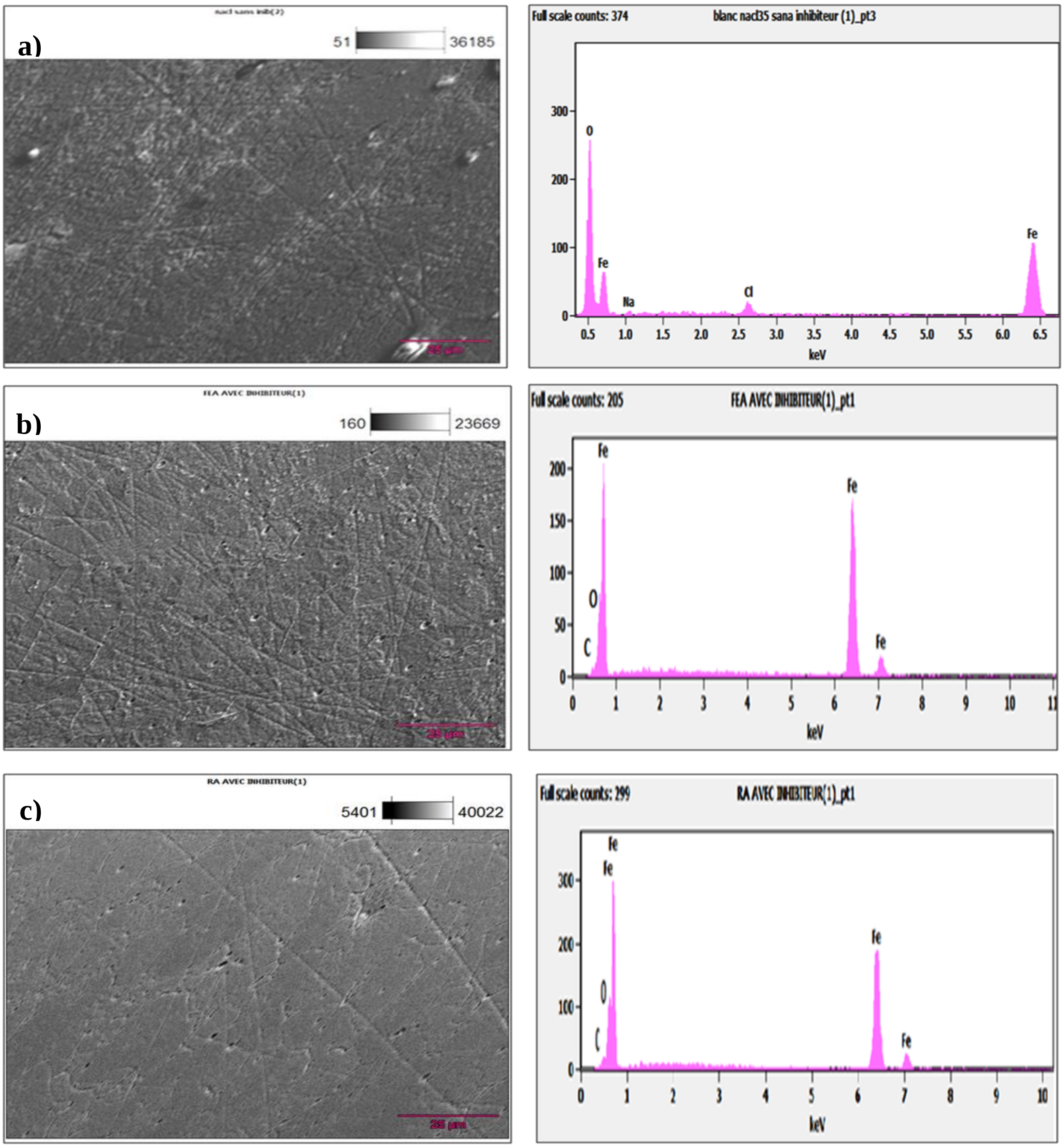

Fig. 7. SEM photograph and Spectrum of EDS analysis of E24 steel immersed for 24 hours, in NaCl 3.5% without (a) and with 10% of methanolic extracts of leaves (b) and root (c) parts from *Anabasis Aretioïdes* plant

As well as, the double layer capacitance value decreases with increasing of the concentration for the both inhibitors, this decrease is may be due to a adsorption of molecules from extracts on the metal/solution interface, probably forming a protective barrier against the corrosion products [34,35].

The inhibition efficiencies values attained a maximum of 76 and 77% at 10% concentration of leaves and root methanolic extracts from *Anabasis Aretioïdes* plant, respectively. These results are not entirely in agreements with that obtained previously, for polarization curves. This may be attributed to the anodic effect of the root extract.

However, both extracts have a protective effect against corrosion to the mild steel substrate in the seawater solution.

3.2.4. Surface characterization by SEM/EDS

The morphologies of E24 steel surface immersed for 24 h in the corrosion solution (NaCl 3.5%) in the absence and in the presence of 10% from methanolic extracts of leaves and root parts from *Anabasis Aretioïdes* plant are presented in Fig. 7a, Fig. 7b and Fig. 7c, respectively.

The surface is highly rusted in the case of the E24 steel immersed in the corrosive solution without inhibitor (see Fig. 7a). The EDS analysis indicate the presence of the elements characteristic of the corrosive medium (O, Na and Cl) on the metal surface [35].

In the presence of inhibitors the SEM photograph showed that the metal surface immersed in NaCl solution with 10% of methanolic extract from leaves (Fig. 7b) is more damaged than that with the presence of 10% of methanolic extract from root (Fig. 7c) and we can observed the presence of some cracks on the surface .Nerveless the EDS spectrum of the both extracts point out the absence of the corrosives ions and the reduce of the oxygen amount .This heterogeneity remarked on the metal surface is probably due to the trace of the polishing.

Furthermore the EDS spectrum detected the presence of carbon element due probably to the organic composition of different extracts.

4. CONCLUSION

The results obtained from this study revealed that the two parts of leaf and root from the *Anabasis aretioides* plant contain significant amounts of polyphenol compounds, as well as condensed tannins, coumarins, reducing compounds, and proteins that could be exploited in several fields (pharmaceutical, food, cosmetics ...).

The both leaves and root extracts from Anabasis Aretioïdes plant can be used as a green corrosion inhibitor, for they have a good inhibition effect against corrosion of mild steel in the seawater solution, this effect is also more pronounced at a concentration of 10% of extracts.

The inhibition efficiency increased with an increase of the inhibitors concentrations, although it differs slightly from one extract to another.

The methanolic extracts from different parts of *Anabasis Aretioïdes* plant could be classified as a mixed type inhibitors with the predominant of the cathodic efficiency for the leaves extract and with predominant of the anodic efficacy for root extract.

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