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Inhibition Effect of Natural *Artemisia* Oils Towards Tinplate Corrosion in HCl solution: Chemical Characterization and Electrochemical Study

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The natural *Artemisia* essential oil (*AO)* was tested as a green inhibitor for tinplate in de-aerated hydrochloride solution (0.5M HCl), by means of potentiostatic polarization, in the range of temperature from 298 to 338 K. Polarization data obtained reveal that *AO* performed excellently as a corrosion inhibitor for tinplate corrosion in hydrochloride acid media and acted as a mixed type inhibitor. The inhibition efficiency increases with an increase in *AO* concentration and attains 81% at 0.5 g/l at 298 K. Also, we ensured that the temperature favors the dissolution of tinplate and becomes increasingly important when the temperature increases. The Langmuir adsorption isotherm was tested for their fit to the experimental data. We also present results of an in situ SEM study on the surface morphology of tinplate electrodes in inhibited and uninhibited electrolytes. The parameters (E^*_a, ΔH^*_a, ΔS^*_a, and ΔG^*_a) were estimated and discussed. The fundamental thermodynamic functions were used to glean important information about *Artemisia* oil inhibitory behavior.

Keywords: Corrosion, green inhibitor, *artemisia oil*, tinplate

1. INTRODUCTION

Tinplate is a thin sheet iron or steel coated with tin to prevent rusting, used especially to make cans and pots. The behaviour of tinplate in aqueous environments depends on several parameters such as the surface properties of the material, the temperature, pH and the composition of the aggressive solution [1-5]. Several studies revealed that the degree of dissolution of tin and the appearance of pitting on the surface of tinplate are more important in the presence of Cl^- anions [6-10]. Moreover, protein and amino acid in the canned food usually contain chloride and sulphur resulting in the food can wall would be attacked seriously and that food in the can being polluted as well. Also, the circumstance in the food can is usually aggressive, and it is required that tinplate cannot seriously corrode during a food can's storage life [11].

The use of non-toxic inhibitors called green or eco-friendly environmental inhibitors is one of the solutions possible to prevent the corrosion of the material and to keep the quality of the food conserved. Natural plants have been added in the form of extracts, oils or pure compounds, and may play major roles to keep the environment more healthy, safe, and under pollution control. Among the various natural products, such as the lawsonia extract [12], black peper [13], cedre oil [14], jojoba oil [15], phyllanthus amarus extract [16], fenugreek extracts [17], artemisia oil [18], pennyroyal oil from mentha oulegium [19], hibiscus sabdariffa extract [20], azadiracta indica [21], garcinia kola extract [22], eucalyptus oil [23,24] and thymus oil [25]. All of which have been reported to be good inhibitors for metals and alloys in acidic solutions.

The aim of this paper is to evaluate the effect of the addition of natural Artemisia essential oil (AO) on the corrosion of tinplate alloy in hydrochloride solution (0.5M HCl) taking account the variation of both medium temperature and inhibitor concentration.

2. MATERIALS AND METHODS

2.1. Plant collection and essential oil extraction

Artemisia was collected in the region of Agadir, Morocco. It was taxonomically identified at the National Scientific Institute of Rabat (Laboratory of Botany, Department of Plant Biology). A voucher specimen sample was deposited in the herbarium of the Laboratory, the National School of Applied Sciences, Ibn Zohr University. Agadir, Morocco. The aerial parts of the plant were air-dried in the laboratory at room temperature. A sample of 100 g was subjected to water distillation for 2 hours using a Clevenger-type apparatus recommended by the French Pharmacopoeia [26]. The yield was determined as grams over the 100 g of powder analyzed in percentage, and is shown in table 1. The oil extracted was analyzed using a Hewlett-Packard 5972 MS, fitted with a HP 5890 Series II GC, and controlled by a G1034C Chemstation. A sample of 1µl was injected under the following conditions: DB1 fused silica capillary column (20 m x 0.20 mm, film thickness 0.2 µm); carrier gas helium (0.6 ml/min); injector temperature 523K; column temperature 323-523K at 276K/min; and MS electronic impact 70 eV. The identification of the compounds was achieved by comparing retention times and mass spectra with those of the published standards [27,28].

2.2. Preparation of the electrode

The employed material in this study is the tinplate. It has been selected based on its diverse uses in packing food and beverage cans. For electrochemical tests, the commercial tinplate samples are in the form of a surface disc of 0.5 cm^2 used as a work electrode that is rinsed with acetone then with distilled water before plunging the electrode in the solution.

Table 1. Chemical composition of *Artemisia* essential oils.

Plant species	Family	Yield (%)[a]	Major constituents	(%)[b]
Artemisia	Astéracées	1.5	Camphor, ($C_{10}H_{16}O$)	46.0
			α-thuyone, ($C_{10}H_{16}O$)	33.2
			β-thuyone, ($C_{10}H_{16}O$)	09.0
			1,8-cineole, ($C_{10}H_{18}O$)	06.4
			Camphene, ($C_{10}H_{16}$)	08.5

[a] Grams of extracted oil as percentage over the 100 g of powder analysed.

[b] Grams over total extracted oil in percentage.

2.3. Electrochemical tests

The electrochemical study was carried out using a potentiostat PGZ100 piloted by Voltamaster soft-ware. This potentiostat is connected to a cell with three electrode thermostats with double wall (Tacussel Standard CEC/TH). A saturated calomel electrode (SCE) and platinum electrode were used as reference and auxiliary electrodes, respectively. The working electrode is in form of disc from tinplate of the surface 0.5cm^2. Potentiodynamic polarization curves were plotted from -700 to -500 mV/SCE at a polarization scan rate of 0.5 mV/s. Before all experiments, the potential was stabilized at free potential during 30 min.

The electrochemical impedance spectroscopy (EIS) measurements are carried out with the electrochemical system (Tacussel), which included a digital potentiostat model Voltalab PGZ100 computer at E_{cor} after immersion in solution without bubbling. After the determination of steady-state current at a given potential, sine wave voltage (10mV) peak to peak, at frequencies between 100 kHz and 10 mHz are superimposed on the rest potential. Computer programs automatically controlled the measurements performed at rest potentials after 0.5, 2, 4 and 24 hours of exposure. The impedance diagrams are given in the Nyquist representation.

The solution 0.5M HCl was prepared by dilution of concentrated HCl with double distilled water. The solution tests are freshly prepared before each experiment by adding the oil directly to the corrosive solution. The solution test is there after de-aerated by bubbling nitrogen. Gas pebbling is maintained prior and through the experiments. Experiments are repeated three times to ensure the reproducibility.

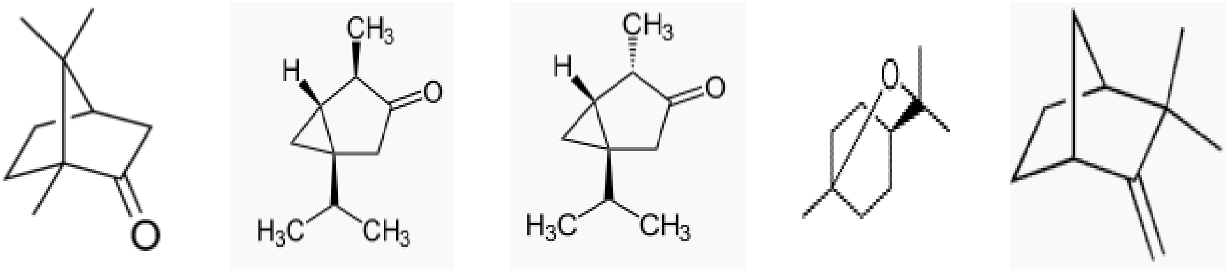
2.4. Surface morphology

The surfaces (0.9 cm X 0.8 cm X 0.1 cm) of tinplate immersed for 1 day in 0.5M HCl solution with and without 0.5 g/l of *AO* were analyzed by scanning electron microscopy (SEM) JOEL JSM-5500 type.

3. RESULTS AND DISCUSSION

3.1. Artemisia oil (AO) analysis

GC-mass spectrum analyzes permit the identification of the composition of *Artemisia* essential oil. The main components are listed in table 1. *AO* mainly contains camphor, α-thuyone, β-thuyone, camphene, 1,8-cineole (Figure 1).



Camphor α-thuyone β-thuyone 1,8-cineole Camphene

Figure 1. Molecular structure of the major components of *Artemisia* essential oil.

3.2. Polarization curves

The composition of the medium and its temperature are essential parameters affecting the phenomenon of the corrosion. Electrochemical steady state measurements are taken at various temperatures (298-338K) in the absence and presence of *Artemisia* essential oil at different concentrations (0.05 to 6g/l). The anodic and cathodic polarization curves obtained are shown in Fig. 2 and the corresponding electrochemical parameters deduced from these curves such as corrosion potential (E_{corr}), corrosion current density (I_{corr}), anodic (βa) and cathodic (βc) Tafel slopes estimated by using Tafel ruler are all given in table 2.

It's observed that the anodic and cathodic branches of polarization curves show the same characteristics with and without the addition of the inhibitor at all temperatures studied. Extrapolation of this linear corrosion potential leads to the current density of corrosion. In anodic domain, an anodic active passive transition is observed in uninhibited and inhibited acid.

Analysis of the polarization curves indicates that the presence of *Artemisia* oil tested leads to decreases in anodic current densities where the cathodic current densities are nearly unchanged. The inhibited systems were slightly shifted toward anodic potentials, emphasizing that the studied compounds act as an anodic type inhibitor. We remark also that cathodic current potential curves give rise to parallel Tafel lines indicating that the hydrogen evolution reaction is activation controlled and the addition of the inhibitor studied does not modify the mechanism of this process.

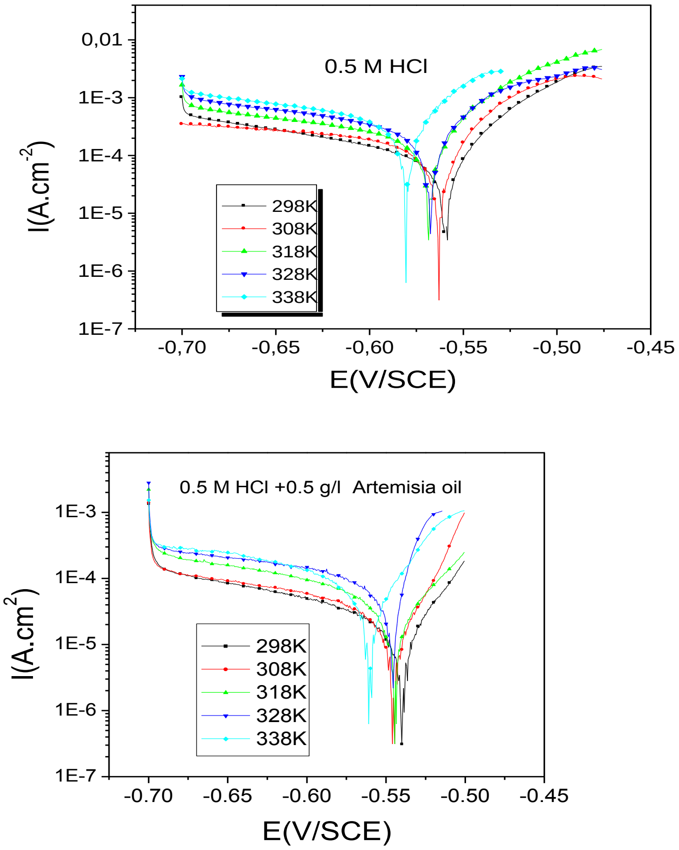


Figure 2. Polarization curves of tinplate in 0.5M HCl in the absence and presence of 0.5g/l of *Artemisia* essential oil at various temperatures of the corrosive medium.

In a reducing acid where hydrogen is evolved from a tin surface, the exchange current is relatively small. It may be increased and the cathodic Tafel slope decreased by providing local noble-metal cathodes. In other words, the dissolution of tin is possible but the rate of corrosion may be very slow. The hydrogen overpotential of tin is high [29].

As can be seen from table 2 that the increasing of temperature from 298 to 338 K acts on all electrochemical parameter values; we observe that the corrosion current densities (I_{corr}) increase and the corrosion potential values (E_{corr}) are moved toward negatives values.

The inhibition efficiency E% can be calculated by the following equation:

$$E\% = (1 - \frac{I'_{corr}}{I_{corr}})x100 \qquad (1)$$

Where I_{corr} and I'_{corr} are, respectively, the uninhibited and inhibited corrosion current densities determined by extrapolation of the cathodic Tafel lines to corrosion potential (E_{corr}).

Table 2. Corrosion parameters obtained from potentiostatic polarization at various temperatures studied for tinplate in 0.5M HCl containing various concentrations of *Artemisia* oil.

Concentration (g/l)	T (K)	E_{corr} (mV/SCE)	I_{corr} (mV/dec.)	βc (mV/dec.)	βa (mV/dec.)	E (%)
Blank	298	-560	102	-127	26	-
	308	-563	124	-96	15	-
	318	-567	127	-91	16	-
	328	-566	172	-90	19	-
	338	-580	221	-70	15	-
0.05	298	-599	42	-82	22	59
	308	-610	66	-65	15	47
	318	-609	70	-42	14	45
	328	-612	98	-30	13	43
	338	-610	132	-23	10	40
0.1	298	-551	25	-113	24	75
	308	-543	44	-106	17	65
	318	-552	49	-99	14	61
	328	-563	86	-46	16	50
	338	-590	105	-103	15	52
0.5	298	-539	19	-119	26	81
	308	-546	32	-94	20	74
	318	-545	41	-56	18	68
	328	-547	57	-35	15	67
	338	-559	90	-34	14	59
1	298	-570	10	-99	19	90
	308	-565	24	-48	16	81
	318	-559	37	-32	14	71
	328	-557	51	-26	13	70
	338	-571	82	-18	10	63
4	298	-493	8	-106	23	92
	308	-490	20	-91	22	84
	318	-492	31	-101	19	76
	328	-510	46	-78	22	73
	338	-531	77	-73	20	65
6	298	-565	5	-95	19	95
	308	-543	15	-67	14	88
	318	-536	27	-60	21	79
	328	-545	41	-30	16	76
	338	-542	62	-75	19	72

It's known that the inhibition efficiency depends on the molecular structure of the inhibitor and the adsorption of the molecule on the metal surface occurs through the hetero-atoms such as oxygen (O). If we go back to Fig.1, we find that all molecules of *AO* components contain one hetero-atom of oxygen with the exception of Camphene component. Moreover, the presence of oxygen atom may increase the polarity and the absorbability of the oil on the surface of tinplate. For this reason, the molecule of camphene doesn't participate in the phenomenon of adsorption. Thus, the inhibiting effect of *AO* may be attributed essentially to camphor and α-thuyone which are the major components of the oil.

The variation of the logarithm of I_{corr} of tinplate in 0.5M HCl at various concentrations of *AO* as function of temperature reciprocate was illustrated in Fig. 3.

Figure 3 shows that the corrosion reaction can be regarded as an Arrhenius-type process (Equation 2). The activation parameters for the studied system (E_a, ΔH* and ΔS*) were estimated from the Arrhenius equation and transition state equation (Equation 2 and 3):

$$I_{cor} = A\exp(-\frac{E_a}{R.T}) \qquad (2)$$

$$I = \frac{RT}{Nh}.\exp(\frac{\Delta S^*}{R}).\exp(-\frac{\Delta H^*}{RT}) \qquad (3)$$

where A is Arrhenius factor, E_a is the apparent activation corrosion energy, N is the Avogadro's number, h is the Plank's constant, R is the perfect gas constant, ΔH* and ΔS* are the enthalpy and the entropy changes of activation corrosion energies for the transition state complex.

Transition state plots for the corrosion rates (I_{corr}) of the tinplate in the absence and presence of different concentrations of the inhibitor are given in Fig. 4 and the corresponding activation parameters E_a, ΔH* and ΔS* for the corrosion process were estimated and listed in Table 3. The change in the activation free energy (Δ*G**) of the corrosion process can be calculated at each experimental temperature by applying the much known equation:

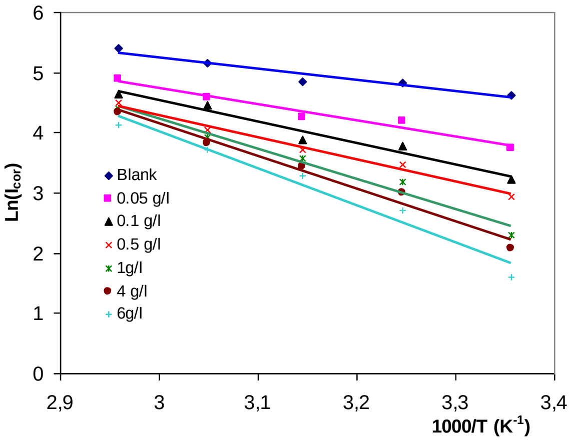


Figure 3. Arrhenius plots of ln(I_{corr}) versus 1/T at various concentrations of *AO*.

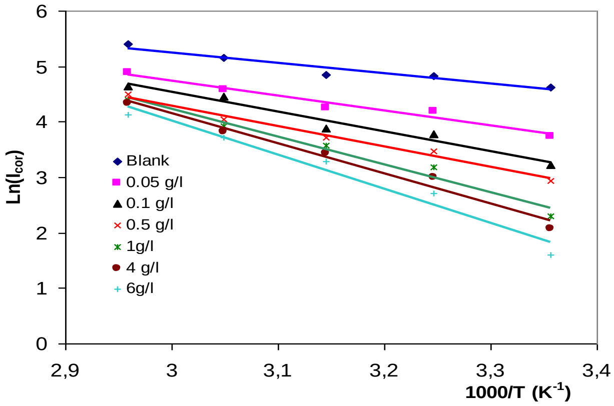


Figure 4. Arrhenius plots of ln(I_{corr}/T) versus 1/T at various concentrations of *AO*.

$$\Delta G^* = \Delta H^* - T\Delta S^* \qquad (4)$$

The obtained Δ*G**values was also listed in Table 3.

Table 3. The value of activation parameters E_a, ΔH*, ΔS* and ΔG*, and for tinplate in 0.5M HCl in the absence and presence of different concentrations of *AO*.

Concentration (g/l)	E_a ($kJ.mol^{-1}$)	ΔH* ($kJ.mol^{-1}$)	ΔS* ($J.mol^{-1}.K^{-1}$)	ΔG* ($kJ.mol^{-1}.K^{-1}$) at 298 K
Blank	15.57	12.93	-163.23	61,57
0.05	22.47	19.84	-146.78	63,58
0.1	29.68	27.05	-126.78	64,83
0.5	30.86	28.23	-125.29	65,56
1	41.73	39.10	-93.14	66,85
4	45.07	42.44	-83.86	67,43
6	50.93	48.30	-67.39	68,38

From table 3, it can be concluded that:

- As observed, the trend of E_a for the studied inhibitor is not the same with that obtained from inhibition efficiency. The higher activation energy for oil as compared to that of free acid may be explained according to Riggs and Hurd [30], as they stated that at higher levels of surface coverage the corrosion process may proceed on the adsorbed layer of inhibitor and not on the metal surface leading to a decrease in the apparent activation energy and in some cases become less than that obtained in the absence of inhibitor.
- The positive signs for both E_a and ΔH* reflecting the endothermic nature of the corrosion process. It is obviously that the activation energy strongly increases in the presence of the inhibitor. Some authors [31-33] have attributed this result to the inhibitor species being physically adsorbed on the metal surface. In this respect, the comparison of the inhibiting action of the investigated compounds in HCl will be of definite interest.
- The negative values of ΔS* pointed to a greater order produced during the process of activation. This can be achieved by the formation of an activated complex representing an association or fixation with consequent loss in the degrees of freedom of the system during the process [34].
- The ΔG* values for the inhibited systems were more positive than that for the uninhibited systems revealing that in presence of inhibitor addition the activated corrosion complex becomes less stable as compared to its absence [30].
-

The fraction of the surface covered by adsorbed molecules of the oil (θ) was determined by the ratio E%/100. Data were tested graphically by fitting to various isotherms. The plot of (C/θ) against the inhibitor concentration (C) yields a straight line with correlation coefficient equal unity (Fig. 5).

The strong correlation shows that *Artemisia* essential oil tested was adsorbed on the tinplate surface electrode according to the Langmuir isotherm [33]:

$$\frac{C}{\theta} = \frac{1}{K} + C \tag{5}$$

$$K = \frac{1}{55.5}\exp\left(-\frac{\Delta G_{ads}}{RT}\right) \tag{6}$$

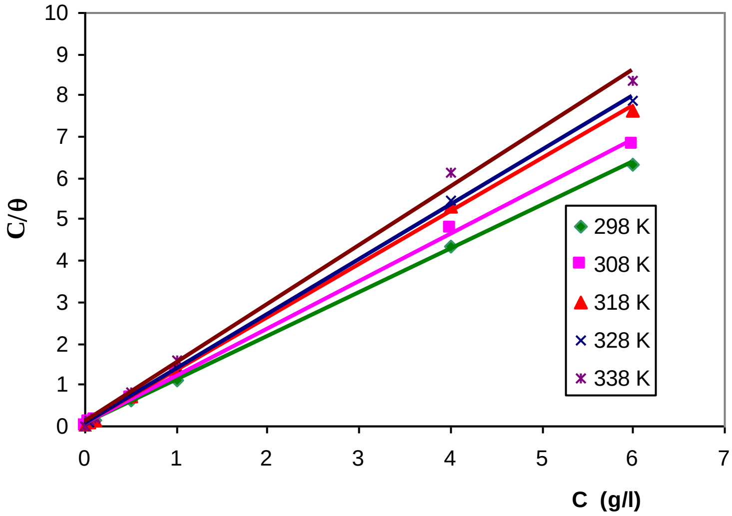


Figure 5. Curves fitting of the corrosion data for tinplate in 0.5M HCl in the presence of *Artemisia* oil according to Langmuir thermodynamic kinetic model at various temperatures.

In Figure 6 shows the evolution of Ln (K) versus the inverse of temperature.

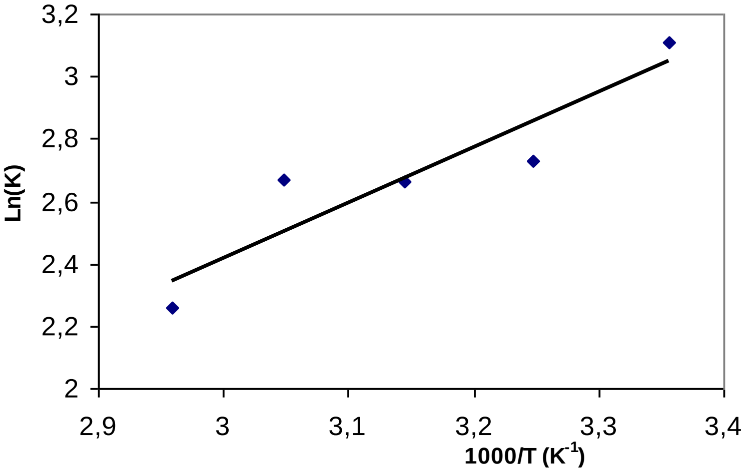


Figure 6. Variation of ln(K) versus 1/T.

ΔH_{ads} found by Van't Hoff equation can also be calculated by the equation of Gibbs-Helmotz:

$$\frac{\Delta G_{ads}}{T} = \frac{\Delta H_{ads}}{T} + cte$$

The Figure 7 represents the variation of ΔGads/T versus the inverse of temperature.

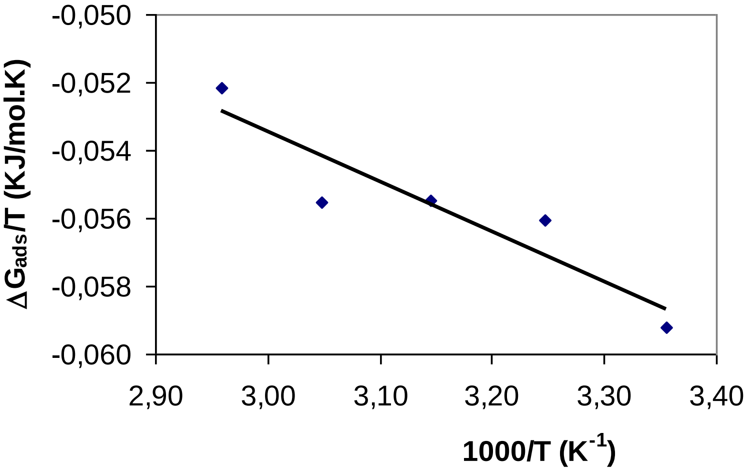


Figure 7. Relation between $\Delta G_{ads}/T$ et 1/T

The enthalpy of AO oil adsorption on the tinplate surface can be deduced from the following equation:

$$\Delta G_{ads} = \Delta H_{ads} - T\Delta S_{ads} \qquad (7)$$

All the results are summarized in Table 4.

Table 4. The value of Activation parameters ΔH_{ads} ΔS_{ads} and ΔG_{ads} for tinplate in 0.5M HCl in the absence and presence of different temperature.

T (K)	K	ΔG_{ads} (kJ.mol^{-1})	ΔH_{ads} (kJ.mol^{-1})	ΔS_{ads} (J.mol^{-1})
298	22.37	-17.64	-14.8	9.70
308	15.31	-17.26		8.16
318	14.31	-17.64		9.10
328	14.39	-18.22		10.56
338	9.56	-17.62		8.50

The negative values of ΔG_{ads} and the higher values of K reveal the spontaneity of adsorption process and are characteristic of strong interaction and stability of the adsorbed layer with the tinplate surface. The obtained values of the adsorption free energy were >- 36 to - 44 kJ mol^{-1} are indicative of chemisorption 35 [35]. The adsorption of an organic adsorbate at the metal/solution interface is considered a ‘‘substitutional adsorption’’ phenomenon [36]. Therefore, the positive values of ΔS_{ads} and ΔH_{ads} related to ‘‘substitutional adsorption’’ can be attributed to the increase in the solvent entropy and to a more positive water desorption enthalpy.

3.3. Electrochemical impedance spectroscopy measurements

Fig. 8-9 shows EIS measurements obtained in 0.5 M HCl in the absence and presence of 0.5g/l of Artemisia essential oil at various immersion times.

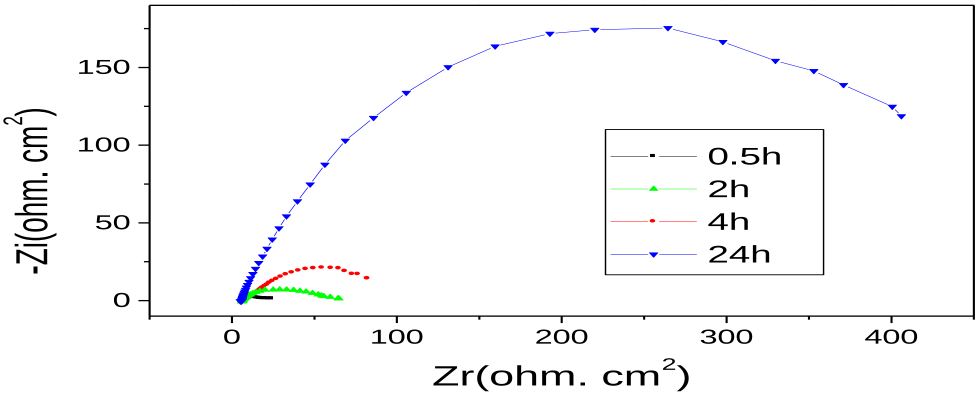


Figure 8. Nyquist plot for tinplate in 0.5 M HCl of different time of immersion at 298K

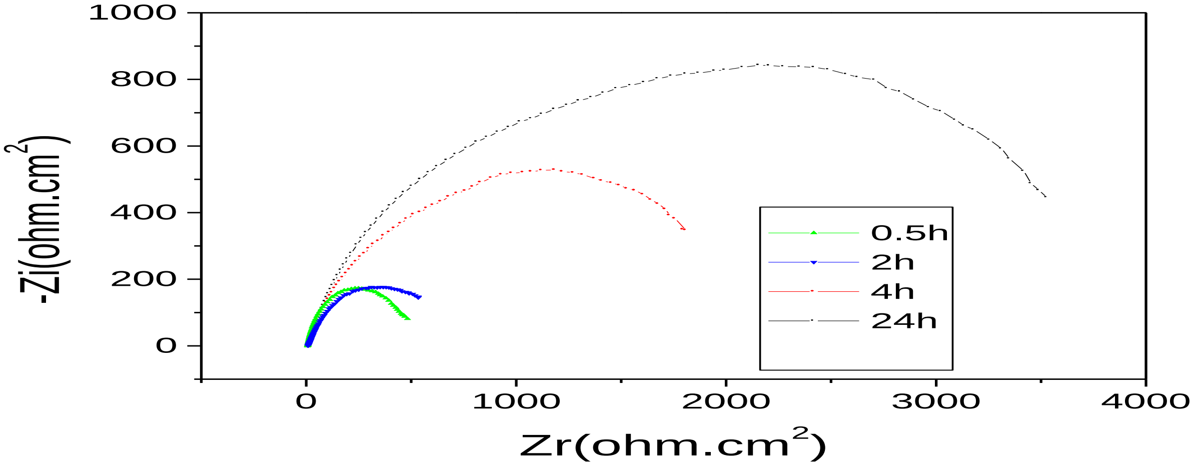


Figure 9. Nyquist plot for tinplate in 0.5 M HCl in the presence of different time of immersion of 0.5 g/l AO at 298 K.

Inspection of Fig. 8 and 9 clearly shows that AO as a corrosion inhibitor for tinplate in 0.5 M HCl. Similar figures have been described in the literature for the acidic corrosion of iron and steel in the presence of inhibitor molecules [37-38].

It can be observed that the size of the capacitive arc in the presence of AO is larger than that in the absence of this inhibitor. This fact is attributed to the inhibition effects of the AO on tinplate corrosion.

Table 5 shows the charge transfer resistance, double layer capacitance and calculated efficiencies obtained from Nyquistic diagram for 0.5 g/l of AO at 298 K.

The inhibition efficiency can be calculated by the following formula:

$$E_{Rt}\% = \frac{(R_t - R^{\circ}{}_t)}{R_t} x100$$

Here R_t and R^0_t are the charge transfer resistances in uninhibited and inhibited solutions respectively.

The values of the polarization resistance were calculated by subtracting the high frequency intersection from the low frequency intersection [39]. Double layer capacitance values were obtained at maximum frequency (f_m), at which the imaginary component of the Nyquist plot is maximum, and calculated using the following equation.

$$C_{dl} = \frac{1}{2.\pi.f_m.R_t} \qquad (7)$$

With C_{dl} : Double layer capacitance (µF.cm^{-2}) ; f_m : maximum frequency (Hz) and R_t : Charge transfer resistance (Ω.Cm^2)

Table 5. Impedance parameters for the corrosion of tinplate in 0.5 HCl solution in the absence and presence of AO at 0.5 g/l.

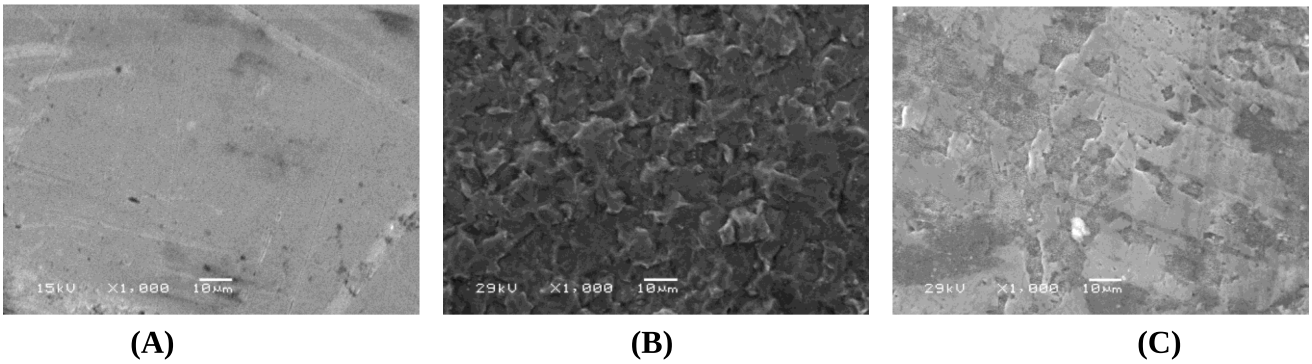
	Immersion time (hours)	R_t (Ω.Cm^2)	C_{dl} (µF.cm^{-2})	E_{Rt} %
Blank	0.5	223.70	89.65	94
0.5g/l		4258.00	0.37	
blanc	2	90.93	875.59	95
0.5g/l		2213.00	0.91	
blanc	4	47.45	1766.24	93
0.5g/l		840.70	4.24	
blanc	24	32.49	3101.94	94
0.5g/l		670.40	59.38	

The examination of data of this table may these conclusions:

1. The nhibitory efficiency is practically independent on immersion time.
2. Values of C_{dl} in inhibited solution are lower than those in uninhibited acid.

3.4. SEM measurements

Fig. 10 shows a scanning electron microscope (SEM) photomicrograph of tinplate surface before and after immersion in 0.5M HCl with and without the addition of the inhibitor at 298 K. The specimen surface in Fig. 10b appears to be roughened extensively by the corrosive environment and the porous layer of corrosion product is present. In presence of AO inhibitor, the data (Fig. 10c) gave the formation of thick films on tinplate surface. This may be intercepted by the adsorption of this inhibitor on the electrode surface. It is thought that the molecule of Aremisia oil depresses the corrosion by the formation of a protective film on the electrode surface.



(A) **(B)** **(C)**

Figure 10. SEM (x1000) of tinplate (a) before immersion (b) after 24 hours of immersion in 0.5M HCl (C) after 24 hours of immersion in 0.5M HCl + 0.5 g/l of *AO*.

4. CONCLUSION

In this work, we have studied the behaviour of corrosion of tinplate in hydrochloride media taking account the effect of the addition of natural *Artemisia* essential oil at different concentrations and the variation of medium temperature. From the obtained results, we can have the following conclusions:

- Increasing of temperature stimulates the active dissolution of tinplate layer in HCl medium.
- Addition of *AO* reduces the corrosion rate of tinplate.
- The inhibitive action of *AO* increases with concentration.
- The best resistance to the corrosion gotten is 81% at temperature 298 K at 0.5 g/l *AO* concentration.
- Adsorption of inhibitor tested fellows Langmuir adsorption isotherm.

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