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Valorization of the essential oil of *Zingiber officinale* by its Use as inhibitor against the corrosion of the carbon steel in acid medium hydrochloric acid 1M

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Abstract: In this work, we are interested in the study of the protection of ordinary steel in acid medium (HCl) 1M by adding *Zingiber officinale* EO rhizomes essential oil at different concentrations: 0.5g / L, 1g / L, 1.5g / L and 2g / L.

The technique used is an electrochemical technique based on the recording of intensity-potential curves in potentio-kinetic mode. The inhibitory action exerted by the essential oil is revealed by tracing the transient curves of the impedance Z to determine in a first step the percentage of inhibition and the stationary curve in order to confirm the first result.

The main results have shown that the essential oil of ginger (*Zingiber officinale*) can slow down or inhibit corrosion in acid medium (hydrochloric acid), steel. The maximum percentage inhibition is 96%. It should be noted that it is obtained with the concentration 1.5g / L of essential oil.

Keywords: *Zingiber officinale* EO; corrosion; inhibition; hydrochloric acid; carbon steel; electrochemical technique.

Introduction

Corrosion affects most industrial sectors and can cause huge losses. In the case of industrial processes, metals are exposed to the action of acids used in chemical processes and during cleaning in several industries. These acids cause the degradation of metals, either by chemical or electrochemical reactions. The best way to protect these materials against corrosion is through the use of inhibitors [1,2]. This use, even in small concentrations has a significant decrease in the percentage of corrosion on the surface of the metal [3,4].

Although these inhibitors exhibit good anticorrosion action, most of them are highly toxic to humans and the environment, hence the importance of converging towards less harmful, natural and biodegradable inhibitors. Recently several studies have been conducted in this direction, using the plants especially their essential oil as inhibitors [5].

One of the widely used metals in industry, particularly for structural applications is carbon steel but being susceptible to rust in humid air and high dissolution in acidic media, its wider use becomes an obstacle [6]. For the cleaning and stripping of steel structures; hydrochloric acid and sulfuric acid, are generally used in industrial processes but are normally accompanied by considerable dissolution of the metal [7].

This work aims to evaluate the inhibitory efficacy of the essential oil *Zingiber officinale* oil on Carbon steel in a 1.0 *M* solution of HCl through the exploitation of potentiodynamic polarization curves, the impedance spectroscopic. The effect of temperature on corrosion is also studied in a range from 298 K to 328 K.

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The thermodynamic parameters, such as *ΔHa* enthalpy and ΔSa entropy, were calculated and analyzed.

Materiel and Methods

Materiel

The tested materiel in this study is carbon steel with the chemical composition listed in Table 1:

Table 1. Chemical composition of the used carbon steel.

Eléments	C	Si	Mn	Cr	Mo	Ni	Al	Co	Cu	V	W	Fe
teneur %	0.11	0.24	0.47	0.12	0.02	0.1	0.03	<0.0012	0.14	<0.003	0.06	98.7

The used carbon steel specimens have a rectangular form with 4cm x 1cm x 0.25 cm. The specimen's surface was prepared by polishing it with emery paper at different grit sizes (from 180 to 1200), rinsing with distilled water, degreasing with ethanol, and drying it at hot air. The aggressive solution of 1 M HCl was prepared by dilution of analytical grade 37 % HCl with distilled water.

Extraction of the essential oil

The essential oil extracted from the rhizomes of *Zingiber officinale* has a complex composition since it consists of a mixture of compounds that belong to different classes of organic chemistry. The composition of the same species can vary according to the geographical location, the climatic conditions, the period of harvest, the part of the plant used and the drying method. The properties can also vary, hence the need to work in the same conditions, to have the reproducibility of the results at the stage of analysis and identification of the molecules contained in the oil. The essential oil was extracted from the rhizomes of *Zingiber officinale* by the "Clevenger" hydrodistillation technique [8]. The apparatus consists of a round-bottomed flask, in which are placed the material containing the volatile oil and a given quantity of water; a separator, in which the oil is automatically separated from the distillate in a graduated tube, thereby permitting a direct reading of the quantity of the oil; and a convenient condenser [9]. The collected oil was kept in an opaque bottle at 4°*C*.

Electrochemical tests

Electrochemical experiments have been performed using a Potentiostat Radiometer Analytical type PGZ 100 to positive direction using Volta Master 4 software which was controlled by a personal computer coupled with an electrochemical cell containing three-electrode cell: a platinum as counter electrode (CE), a carbon steel as a working electrode (with an exposed surface of 1 cm^2), and saturated calomel electrode (SCE) as the reference electrode. All potentials were measured against this later electrode. The potentiodynamic polarization curves were recorded by changing the electrode potential automatically from negative to positive values versus E_{corr} at a scan rate of 1 mV/s after 30 min of immersion time until reaching steady state. The test solution was thermostatically controlled at 298±2 K using 50 ml of test solution.

In order evaluate corrosion kinetic parameters, a fitting by Stern-Geary equation was used. Thus, the overall current density values, i were considered as the sum of two contributions, anodic and cathodic current i_a and i_c, respectively. For the potential domain not too far from the open circuit potential, it may be considered that both processes followed the Tafel law [10]. Thus, it can be derived from equation (1):

$$i = i_a + i_c = i_{corr}\{e^{[b_a(E-E_{corr})]} - e^{[b_c(E-E_{corr})]}\} \quad (1)$$

Where i_{corr} is the corrosion current density ($A\ cm^{-2}$), b_a and b_c are the Tafel constants of anodic and cathodic reactions (V^{-1}), respectively. These constants are linked to the Tafel slopes β(*V/dec*) in usual logarithmic scale given by equation (2):

$$\beta = {}^{ln10}/_{b} = {}^{2.303}/_{b} \quad (2)$$

The corrosion parameters were then evaluated using nonlinear least square method by applying the previous equation using Origin software. However, for this calculation, the potential range applied was limited to ±0.100 *mV/ECS* around E_{corr}, and a significant systematic divergence was sometimes observed for both anodic and cathodic branches.

The corrosion inhibition efficiency ($\boldsymbol{\eta_{pp}}\%$) is evaluated from the corrosion current densities values using the following equation (3) [11]:

$$\boldsymbol{\eta_{pp}}\% = \left[{(\boldsymbol{i^{\circ}_{corr}} - \boldsymbol{i_{corr}})}/{\boldsymbol{i^{\circ}_{corr}}} \right] \times \mathbf{100} \quad (3)$$

Where i^0_{corr} and i_{corr} are the corrosion current densities in absence and presence of the inhibitor, respectively

EIS measurements performed at corrosion potentials, E_{corr}, over a frequency range of 100 *kHz* to 100 *MHz* with an AC signal amplitude perturbation of 10 *mV* peak to peak. The EIS diagrams were done in the Nyquist plot. The results were then analyzed in terms of an equivalent electrical circuit using Brockamp program [12].

The inhibition efficiency ($\eta_{imp\%}$), is calculated using equation (4):

$$\eta_{imp}\% = (R_{CT} - R^{\circ}_{CT}/R_{CT}) \times 100 \quad (4)$$

$$\theta = R_{CT} - R^{\circ}_{CT}/R_{CT}$$

Where R^0_{ct} and R_{ct} are the charge transfer resistance in absence and presence of the inhibitor, respectively, θ is the recovery rate.

Result and discussion

Chemical composition

The analysis of the essential oil of ginger by chromatography in the gaseous stage coupled by a spectre of mass enabled us to identify eighteen compounds (Table 2). The most abundant components are: Geranial (23.30 %), Camphene (12.59 %) Eucalyptol (12.87 %), and alpha-Zingiber ne (7.38 %).

Table 2. Chemical Composition of *Zingiber officinal* essential oil.

Tr	aire	%	Nom
3.866	767203	3.06	alpha.-Pinene
4.256	3154656	12.59	Camphene
4.791	360472	1.44	beta.-Myrcene
5.541	1029433	4.11	D-Limonene
5.690	1995171	7.96	beta.-Phellandrene
5.836	3225554	12.87	Eucalyptol
6.958	614769	2.45	beta.-Linalool
8.618	299462	1.19	Borneol
8.938	1180579	4.71	alpha.-Terpineol
10.032	296378	1.18	Dodecan-2-one
10.291	3299622	23.30	Geranial
12.643	1848341	7.38	alpha.-Zingiber ne
12.780	356637	1.42	beta.-Bisabolene
12.926	274817	1.10	.beta.-Himachalene
13.108	568711	2.27	.beta.-Sesquiphellandrene
13.700	306697	1.22	trans-Nerolidol
13.869	471674	1.88	Elemol
14.570	393856	1.57	alpha.-Bisabolol

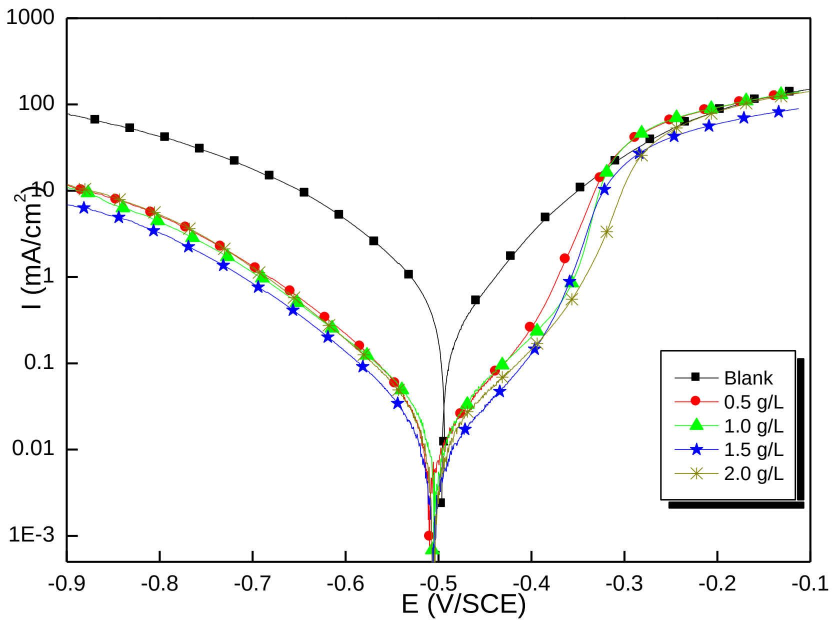

Figure 1. Polarization curves of carbon steel in 1 M HCl with and without *Zingiber officinale* EO at various concentrations at 298 K

Electrochemical studies

Polarization studies

Figure 1 represents the cathodic and anodic polarization curves of carbon steel, in 1M HCl, with and without inhibitor at different concentrations, the electrochemical parameters: anodic Tafel constancy *(βa)*, cathodic Tafel constancy *(βc)*, corrosion potential (E_{corr}) and the corrosion current density (i_{corr}) and inhibition efficiency **(**η_{pp}%) are given in Table 3.

It is clear from Fig. 1 that the addition of the *Zingiber officinale* EO reduces both the cathodic and the anodic currents and therefore hinders the acid attack of the carbon steel electrode in 1M HCl. The significant change of Tafel slopes of the anodic (*βa*) and cathodic (*βc)* in the presence of *Zingiber officinale* EO can be observed as a result of adsorption the molecules of inhibitor. The corrosion process decrease can be explained by both metallic dissolution and hydrogen evolution at the metal surface due to the adsorption of an organic compound (heteroatom such as oxygen) [13,14,15].

Based on these results, the *Zingiber officinale* EO can be considered as mixed type inhibitor in 1M HCl. From Table 3, it is clear that the i_{corr} values decrease considerably with the increase of the concentration of *Zingiber officinale* EO. That means that the inhibitory actions are due to their adsorption on the carbon steel surface. Accordingly, **(**η_{pp}%) values increase with increasing the inhibitor concentration reaching a maximum value of 97.4% with 1.5 g/L of ginger oil.

Table 3. Polarization data of carbon steel in 1.0 M HCl without and with *Zingiber officinale* EO at 298 K.

Compound	Conc. g/L	- E_{corr} mV/SCE	i_{corr} µA cm^{-2}	$-\beta_c$ mV dec^{-1}	β_a mV dec^{-1}	η_{PP} %
HCl 1.0M	--	498	983	140	150	-
EO	0.5	506	42	127	67	95,7
	1.0	498	39	128	92	96,0
	1.5	504	25	125	77	97,4
	2.0	503	30	119	83	96,9

EIS studies

The corrosion behavior of mild steel in 1.0 *M* HCl with and without *Zingiber officinale* EO is also investigated by EIS at corrosion potential. The nyquist plot, for carbon steel in 1.0 M HCl *with Zingiber officinale* EO at 298 K, is shown in Figure 2. The Nyquist spectra exhibit a single capacitive loop single wave attributed to charge transfer of the corrosion process [16]. These Nyquist plots are not perfect semi-circles, and this may be attributed to the frequency dispersion of interfacial impedance [17].

This behavior is a result of the surface roughness, the chemical heterogeneity of surface, and the adsorption-desorption process of inhibitive molecules on carbon steel surface [18]. Furthermore, the diameter of the semicircles in the presence of *Zingiber officinale* EO is larger than observed in blank solution (1.0 *M* HCl) and increases with increasing inhibitor concentration, which may be related to the increase of surface coverage of inhibitive molecules on the carbon steel surface.

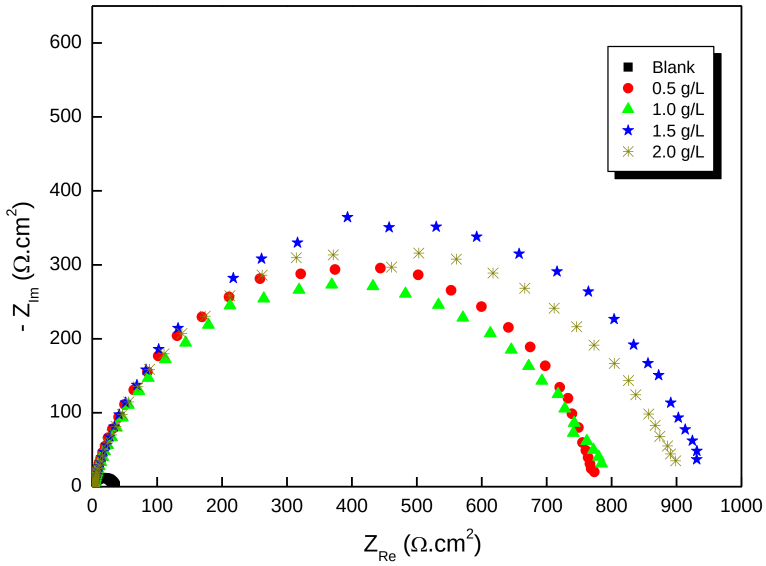

Figure 2. Impedance plots for steel interface in 1.0M HCl medium, presented in Nyquist plan, absence and presence of different concentrations of *Zingiber officinale* EO at 298 K

Accordingly, the simple electrical equivalent circuit (Figure 3) has been proposed to model the experimental data. The employed circuit allowed the identification of the solution resistance (Rs) and charge transfer resistance (R_{ct}). It is noteworthy that the double layer capacitance (C_{dl}) value was affected by imperfections of the surface, and this effect was simulated via a constant phase element (CPE) [19]. CPE was used as the substitute for the capacitor to fit more accurately impedance behavior of the electric double layer. The impedance of the CPE is expressed as [20]:

$$Z_{CPE} = Y^{-1}(j\omega)^{n} \quad (5)$$

Where Y is the magnitude of the CPE; ω the angular frequency; n is an empirical exponent which measures the deviation from the ideal capacitive behavior [21]. Depending on the values of n, CPE can represent resistance (n = 0), capacitance (n = 1), inductance (n=-1) and Warburg impedance (n = 0.5) [22,23]. Also, the double layer capacitances, Cdl, for a circuit including a CPE were calculated by using the equation (6) [24]:

$$C_{dl} = Q\,(\omega_{max})^{n-1} \quad (6)$$

Where $\omega_{max} = 2\pi f_{max}$ is the angular frequency at the maximum value of the imaginary part of the impedance spectrum, Q is the constant phase element (CPE).

The impedance spectrums were fitted, and their parameters were calculated according to different models and listed in Table 4.

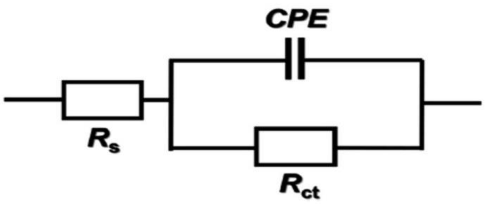

Figure 3. The electrochemical equivalent circuit used to fit the impedance spectra

Inspection of the results in Table 4 indicated that R_{ct} value increased and C_{dl} decreases with the increased concentration of the inhibitor. This is attributed to the increase in surface coverage of carbon steel by inhibitor molecule leading to enhanced inhibition efficiency [25]. The increase in R_{ct} value is attributed to the formation of a protective film on the metal/solution interface. The impedance study also gave the same efficiency trend as found in Tafel polarization method.

Table 4. Electrochemical impedance parameters of carbon steel in 1*M* HCl medium in the absence and the presence of the inhibitor at different concentrations at 298 K.

Medium	C (g/L)	R_s (Ω cm^2)	R_{ct} (Ω cm^2)	C_{dl} (μF cm^{-2})	n_{dl}	Q (μF.S^{n-1})	θ	η_{imp} %
HCl	--	1.12	34.7	121.0	0.773	419	-	-
EO	0.5	2.87	774	41.96	0.856	68.55	0.955	95,5
	1.0	1.43	779.8	55.67	0.804	102.7	0.956	95,6
	1.5	1.31	951.8	68.62	0.806	116.5	0.964	96,4
	2.0	0.84	902	46.34	0.795	88.7	0.962	96,2

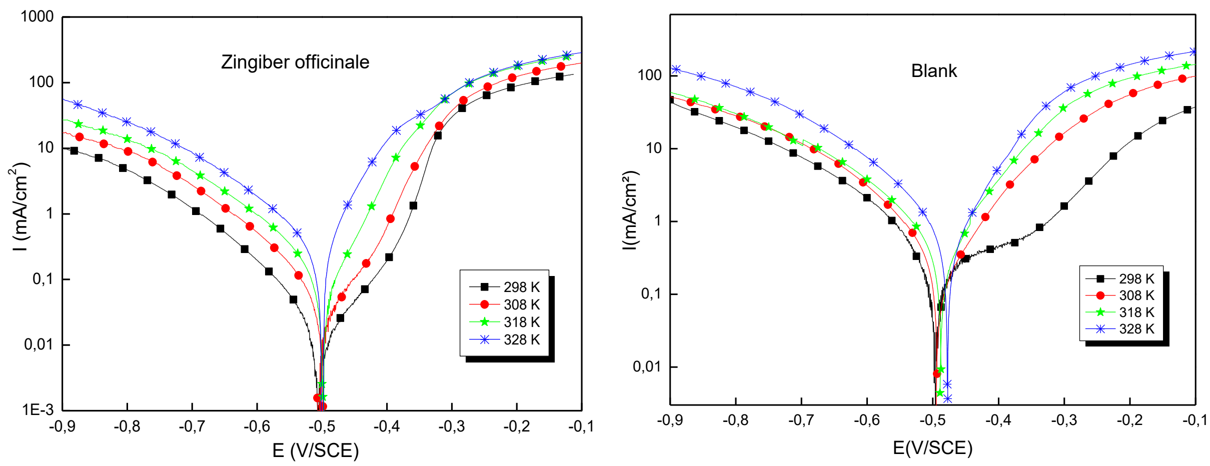

Figure 4. Temperature effect in 1.0 M HCl, with 1.5 g/L of Zingiber *officinale* EO and uninhibited solution

Effect of temperature

In order to get more information about the adsorption type of *Zingiber officinale* EO and its effectiveness at higher temperatures, potentiodynamic polarization measurements were used in the temperatures range from 298 to 328 K for carbon steel electrode in 1.0 M HCl without and with 1.5 g/L of *Zingiber officinale* EO after 30 mn of immersion time (Figure 4). It is remarked that these curves exhibited the Tafel regions. It is also noted that the anodic and cathodic branches increased with the increase of temperature.

The values of corrosion current density (i_{corr}), Corrosion potential of steel (E_{corr}), and the inhibitory efficiency of both inhibitors as a function of temperature are given in Table 5.

Table 5. Influence of temperature on electrochemical parameters of steel in 1.0M HCl without and with addition of 1.5 g/L of *Zingiber officinale* EO.

Compound	Temperature K	$-E_{corr}$ mV/SCE	i_{corr} µA cm^{-2}	$-\beta_c$ mV dec^{-1}	β_a mV dec^{-1}	η_{PP} %
Blank	298	498	983	140	150	-
	308	491	1200	184	112	-
	318	475	1450	171	124	-
	328	465	2200	161	118	-
inh	298	504	25	125	77	97,4
	308	497	71	135	53	94,1
	318	496	131	145	75	90,9
	328	499	269	147	83	87,7

It can be seen that the i_{corr} increased with increased temperature, both in uninhibited and inhibited solutions, and the values of the inhibition efficiency of *Zingiber officinale* EO decreased with higher temperatures. Thus, the inhibition efficiencies of *Zingiber officinale* EO are temperature-dependent.

Activation parameters of corrosion process

The activation thermodynamic parameters of the corrosion process can be determined using Arrhenius Eq. and Eq. transition state [26]:

$$\ln i_{corr} = \ln A - \frac{Ea}{RT} \quad (7)$$

$$\ln\frac{i_{corr}}{T} = \left(\ln\left(\frac{R}{Nh}\right) + \frac{\Delta Sa}{R}\right) - \frac{\Delta Ha}{RT} \quad (8)$$

Where Ea is the apparent activation energy, R is the gas constant; A is the Arrhenius pre-exponential factor, N is Avogadro's number, h is Plank's constant, ΔSa is the entropy of activation and ΔHa is the enthalpy of activation.

A plot of ln (i_{corr}) vs 1000/T obtained gave a straight line with regression coefficient close to unity, as shown in Figure 5. The Arrhenius plot of ln (i_{corr}/T) vs 1000/T (Figure 5) which gave straight lines with slope ΔHa/R and intercept (ln (R/N.h) + ΔSa/R) from which ΔHa and ΔSa values were calculated. The values of ΔHa and ΔSa were calculated and listed in Table 6.

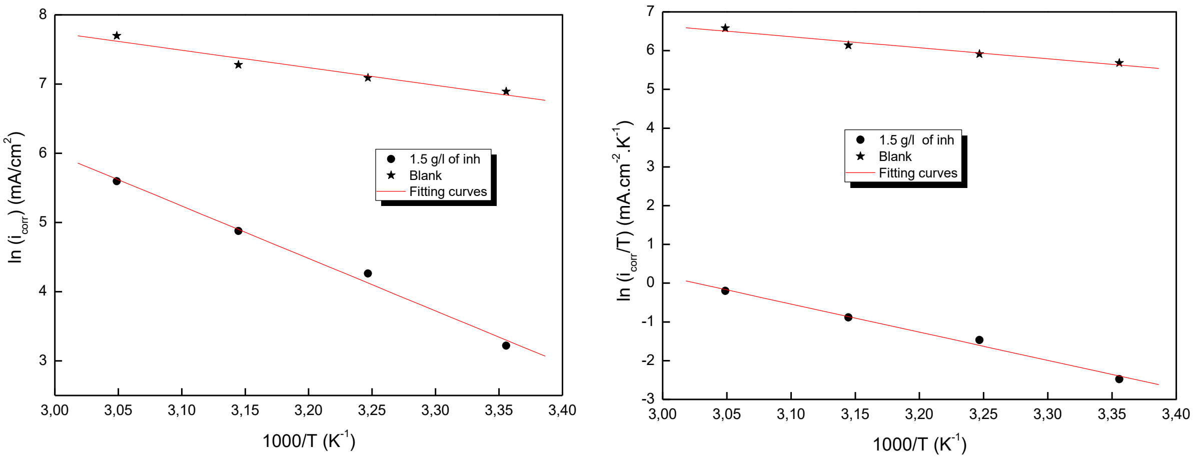

Figure 5. Arrhenius curves of carbon steel in 1 M HCl with and without 1.5g / L of essential oil of *Zingiber officinale*

Table 6. The value of the activation parameters Ea, ΔHa and ΔSa of carbon steel in 1M HCl in the absence and in the presence of 1.5g / L of essential oil of *Zingiber officinale*

	Ea (KJ/mol)	ΔHa (KJ/mol)	ΔSa (KJ/mol.K)
Blank	21	18.5	-126
1.5 g/L of inh	63	60.4	-14.7

It was observed that the value of apparent activation energy Ea are higher in the presence of inhibitor than in its absence. This result indicated that the energy barrier for the corrosion reaction increases in the presence of inhibitor is associated with physical adsorption or weak chemical bonding between the inhibitor specie and the steel surface [27,28] The positive values of ΔHa mean that the dissolution reaction is an endothermic process indicating difficulty of carbon steel dissolution [29,30]. The entropy ΔSa increases negatively in the presence of *Zingiber officinale* EO which reflects that the activated complex represents an association rather than a dissociation step which means the formation of an ordered, stable layer of this inhibitors on the carbon steel surface [31,32].

Conclusion

The following conclusions can be derived from the study of Corrosion inhibition characteristics of *Zingiber officinale* EO on carbon steel corrosion in 1 M HCl medium:

- ✓ The *Zingiber officinale* EO are efficient corrosion inhibitor for carbon steel in the studied medium.
- ✓ Polarization measurement showed the *Zingiber officinale* EO are mixed type inhibitor.
- ✓ Electrochemical impedance results show that the obtained Nyquist diagrams exhibit one single capacitive loop, indicating that carbon steel corrosion in essentially under charge transfer control, leading to the formation of a protective layer on the metal surface, which limits the steel corrosion.
- ✓ The study of the influence of temperature on the inhibitory efficiency shows that this is decreasing with increasing temperature. That confirms that the *Zingiber officinale* EO adsorption on the steel surface is done by physisorption type.
- ✓ Reasonably good agreement was observed between the obtained measures from potentiodynamic polarization curves and electrochemical impedance spectroscopy techniques.

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