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Research Article

Marjoram Extract as Corrosion Inhibitor for Dissolution of Zinc in 1.0 M HCl

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In this study, water marjoram (*Origanum marjorana* L.) extract was evaluated as corrosion inhibitor for zinc in 1.0 M HCl solution. The polarization measurements showed that this inhibitor is acting as mixed inhibitors for both anodic and cathodic reactions. The results showed that the inhibition efficiency was increased by increasing the inhibitor doses and reached the maximum at 500 ppm. The adsorption of marjoram extract on zinc surface was found to obey Langmuir type isotherm. The efficiency obtained from the impedance measurements was in good agreement with those obtained from the gravimetrical, thermometric, and polarization techniques which prove the validity of these tolls in the measurements of the tested inhibitor.

1. Introduction

Corrosion inhibitors are widely used in industry to reduce the corrosion rate of metals and alloys in contact with aggressive environment. Most of the corrosion inhibitors are synthetic chemicals, expensive and very hazardous to environment. Therefore, it is desirable to source for environmentally safe inhibitors [1–4]. It has been shown that natural products of plant origin contain different organic compounds (e.g., alkaloids, tannins, pigments, and organic and amino acids, and most are known to have inhibitive action [5, 6]). Other authors have also shown that the inhibitive effect of some plant extract solutions was due to the adsorption of photochemical molecules present in the plant on the metal surface [7], which blocks the metal surface and thus does not permit the corrosion process take place.

The main components of marjoram (*Origanum marjorana* L.) oil extract are thymol (38.4%) and cis-sabinene hydrate (25.3%). Other components detected in lower amounts in oil sample were sabinene and p-cymene (up to 7.4% and 13.9% in), and alpha-terpinene (up to 13.3%) in addition to some active phytochemicals like terpines, flavonoids, and rosmarinic acid is found in big quantities [8, 9]. The objective of the present work is to study the effect of dry plant extracts of marjoram as a corrosion inhibitor for the corrosion of zinc in 1.0 M HCl. Moreover, the effect of temperature on the dissolution of zinc as well as on the inhibition efficiency of the extracted compound was also investigated.

2. Experimental

2.1. Medium. The acid solution of 1.0 M HCl was prepared by dilution of analytical grade HCl (37%) with double distilled water and all experiments were carried out in unstirred solutions.

2.1.1. Extract Preparation. Marjoram plant used in this study was collected and washed to remove impurities such as dust and then dried in an air oven for 3 days at 120°C. The air dried plant materials were ground in a blender with a particular size to ensure the plant powders in identical size. 10 g of plant powder was extracted for 2 days with 200 mL of water at 96°C by a thermowater which is fixed to 180 rpm. The samples were carried to a rotary evaporator to remove water underreduced. The crude extracts were kept in refrigerator in glass bottles until the further experiments.

The extract main components have the following formulas represented in Figure 1.

2.2. Weight Loss Measurements. Zinc strips (99.99% pure), with 3.0 × 2.0 × 0.05 cm sizes for each, were used for weight

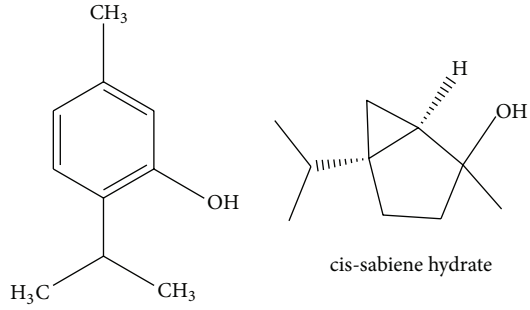

Figure 1: The formulas of marjoram extract main components.

loss measurements. Weight loss experiments were carried out as described elsewhere [10]. The corrosion rate (CR) and the percentage protection efficiency IE (%) were calculated according to the following equations [11, 12]:

$$\mathrm{CR} = \frac{\Delta m}{St},$$
$$\mathrm{IE}\ (\%) = \frac{\mathrm{CR}_{\mathrm{corr}} - \mathrm{CR}_{\mathrm{corr\,(inh)}}}{\mathrm{CR}_{\mathrm{corr}}} \times 100, \quad (1)$$

where Δm (mg) is the mass loss, S (dm^2) is the area, t (h) is the immersion period, and $\mathrm{CR}_{\mathrm{corr}}$ and $\mathrm{CR}_{\mathrm{corr\,(inh)}}$ are the corrosion rates of zinc in the absence and presence of the extract compound.

2.3. Thermometric Measurements. The procedure used for determining the corrosion behavior by this method has been described elsewhere by other authors [13–15]. In this technique, the corroding (HCl) concentration was kept at 1.0 M. The volume of test solution used was 50 mL. The initial temperature in all experiments was kept at 30°C. The corrosion reaction progress was monitored by determining the changes in temperature with time using a calibrated thermometer (0–100°C) to the nearest ±0.05°C. This method enabled the computation of the reaction number (RN) which is defined as

$$\mathrm{RN}\left(^{\circ}\mathrm{C\,min}^{-1}\right) = \frac{T_m - T_i}{t}, \quad (2)$$

where T_m and T_i are the maximum and initial temperatures, respectively, and t is the time (min) taken to reach the maximum temperature. The inhibition efficiency IE (%) was evaluated from percentage reduction in the reaction number using the following equation:

$$\mathrm{IE}\,(\%) = \frac{\mathrm{RN}_{\mathrm{free}} - \mathrm{RN}_{\mathrm{add.}}}{\mathrm{RN}_{\mathrm{free}}} \times 100, \quad (3)$$

where $\mathrm{RN}_{\mathrm{free}}$ is the reaction number in the absence of Marjoram extract (blank solution) and $\mathrm{RN}_{\mathrm{add.}}$ is the reaction number of 1.0 M HCl containing the extracted compound.

2.4. Potentiodynamic Polarization. Potentiodynamic measurements were carried out using three-compartment glass cell and PS remot potentiostat with PS6 software for calculation of electrochemical parameters. Platinum electrode was used as a counterelectrode (separated from the cell solution by a sintered glass frit) and a saturated calomel electrode (SCE) inside a luggin's probe as a reference electrode. A cylindrical rod embedded in araldite with an exposed surface area of 0.5 cm^2 was used. The electrode surface was polished with different grades of emery paper, degreased with acetone, and rinsed with distilled water.

The inhibition efficiency IE (%) was calculated from polarization measurements according to the relation given below:

$$\mathrm{IE}\,(\%) = \frac{I_{\mathrm{free}} - I_{\mathrm{add.}}}{I_{\mathrm{free}}} \times 100, \quad (4)$$

where I_{free} and $I_{\mathrm{add.}}$ are uninhibited and inhibited corrosion current densities, respectively. They are determined by extrapolation of Tafel lines to the respective corrosion potentials.

2.5. Electrochemical Impedance Spectroscopy. The impedance measurements were carried out at open circuit potential (E_{ocp}) in the frequency range from 10 kHz to 100 mHz with signal amplitude perturbation of 5 mV by using a computer-controlled potentiostate (Auto Lab 30, Metrohm). All experiments were performed using three-electrode system.

The percentage inhibition efficiency due to the extracted compound was calculated from the charge transfer resistance values using the following equation:

$$\mathrm{IE}_{(R)}\,(\%) = \left[\frac{\left(1/R_{\mathrm{ct}}\right)_0 - \left(1/R_{\mathrm{ct}}\right)}{\left(1/R_{\mathrm{ct}}\right)_0}\right] \times 100, \quad (5)$$

where $(R_{\mathrm{ct}})_0$ and (R_{ct}) are the uninhibited and inhibited charge transfer resistance, respectively [16].

3. Result and Discussion

3.1. Weight Loss Measurements. The corrosion rates of the zinc coupons in 1.0 M HCl without and with different concentrations of marjoram extract were determined. The results obtained are summarized in Table 1. As inspection of Table 1, the corrosion rates decrease with increasing concentration of the extracted compound. This indicates that extract in the solution inhibits the corrosion of zinc in HCl and the extent of corrosion inhibition depends on the amount of the extract present. Also shown in Table 1 are the calculated values of inhibition efficiency for the extract. It is observed that the inhibition efficiency IE (%) increases with increasing concentration of the extract. The maximum inhibition efficiency was observed to be 91.80% at 500 ppm. The corrosion inhibition can be attributed to the adsorption of the main components and phytochemicals molecules present within the marjoram extract on the metal surface. Owing to the complex chemical composition of

Table 1: Corrosion parameters for zinc in aqueous solution of 1.0 M HCl in absence and presence of different concentrations of marjoram extract from weight loss measurements at 30°C for 1/2 h.

Inhibitor concentration (ppm)	Weight loss ($mg\,dm^{-2}$)	CR ($mg\,dm^{-2}\,h^{-1}$)	IE (%)
Blank	843	252.90	—
100	341	98.00	61.30
200	243	73.60	71.00
300	183	52.40	79.30
400	98	28.00	88.90
500	69	21.20	91.80

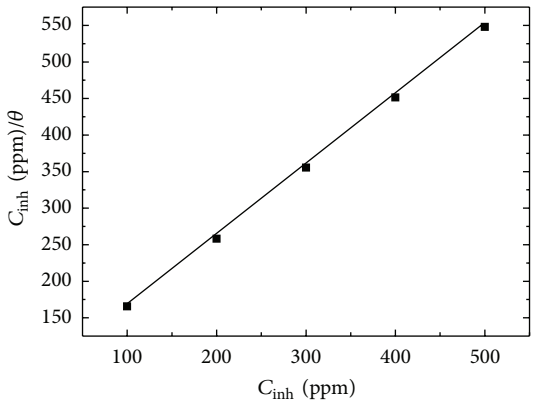

Figure 2: Langmuir adsorption isotherm plot of Marjoram extract adsorption on the surface of zinc in 1.0 M HCl.

the extract, it is quite difficult to assign the inhibitive effect to a particular constituent. However, mutual effects of these compounds and other components present in the extract cannot be ruled out. The adsorption of these compounds on the zinc surface reduces the surface area available for corrosion. The values of surface coverage (θ = IE/100) for different concentrations of marjoram extract obtained from the weight loss measurements at temperature of 303 K were tested graphically by fitting to various isotherms. A straight line (Figure 2) was obtained on plotting C_{inh} versus C_{inh}/θ, from which it is observed that the adsorption of the studied extract on zinc surface obeys Langmuir adsorption isotherm:

$$\theta = \frac{W_o - W}{W_o}. \quad (6)$$

3.2. Thermometric Studies. Thermometric technique has proved to be of considerable value and help in studying corrosion behavior of a number of metals and alloys in various corroding environments [17, 18]. The technique is also useful in evaluating the inhibitor efficiency of a number of organic compounds [19]. Figure 3 shows the variation of temperature with time for the corrosion reaction of zinc in 1.0 M HCl solution in the absence and presence of different concentrations of the extracted compound.

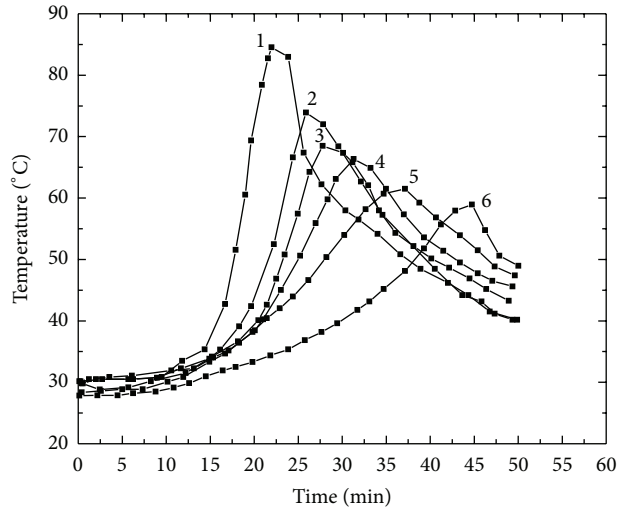

Figure 3: Temperature-time curves for zinc corrosion in 1.0 M HCl solutions containing different concentrations of Marjoram extract. (1) 0.00 ppm, (2) 100 ppm, (3) 200 ppm, (4) 300 ppm, (5) 400 ppm, and (6) 500 ppm.

Table 2: Calculated values of reaction number and percentage reduction in RN for Zn dissolution in 1.0 M HCl containing marjoram extract from thermometric method.

Extract concentration (ppm)	Reaction number (RN) ($°C\,min^{-1}$)	Reduction in RN (% IE)
Blank	2.47	—
100	1.72	30.40
200	1.43	42.30
300	1.22	50.60
400	0.89	64.00
500	0.68	72.50

As shown in Figure 3, the temperature of the system rises gradually due to the exothermic corrosion reaction to reach a maximum value, T_m, which was attained at a very short time (t) by the acid-free additive solution. This corresponds to a reaction number (RN) of $2.47°C\,min^{-1}$ (Table 2). It is also shown from Figure 3 that on the addition of the extracted compound the maximum temperature attained decreased and the time required to reach it increases. This gives an indication that the various additives inhibit the corrosion of zinc in the acid solution, probably by adsorption on the metal surface [20]. The extent of inhibition depends on the degree of coverage of the metal by the adsorbed extract molecules. Strong adsorption is achieved at higher concentration of the extract as depicted by the decrease in (T_m) attained and a corresponding increase in time (t) taken to reach it, resulting in a large decrease in the RN of the system (Table 2). The decrease of temperature for all systems after reaching their maximum values could be attributed to decreasing the corrosion rate and, hence, a decrease in quantity of produced heat during the reaction.

From inspection of the results given in Table 2, it is very clear that reaction number decreased in the presence of

the extracted compound compared to the blank solution. Also, the percentage reduction in reaction number increases with increase in concentration of the extract. This is the same trend obtained from weight loss measurements.

3.3. Polarization Studies. The effect of addition of various concentrations of the natural extracted compound on the cathodic and anodic polarization curves of zinc in 1.0 M HCl solution at 30°C (Figure 4) was studied. Electrochemical parameters such as corrosion current density (i_{corr}), corrosion potential (E_{corr}), Tafel constants (b_a and b_c), and percentage inhibition efficiency IE (%) were calculated from Tafel plots shown in Table 3. It is evident from the table that both anodic and cathodic Tafel slopes increase upon the addition of the extract compound indicating a mixed anodic and cathodic effect on the mechanism of corrosion inhibition [21]. Also, it is observed that the corrosion potential shifted to more positive values and i_{corr} decreased when the concentration of the inhibitor was increased indicating the inhibiting effect of this extract compound resulting in an increase in the percentage inhibition efficiency IE (%).

3.4. Effect of Temperature. The effect of temperature on the corrosion rate of zinc in acid-free additives and in the presence of 500 ppm of extract was studied in the temperature range of 303 to 353 K using potentiodynamic polarization measurements.

The data listed in Table 4 show that E_{corr} shifted to more negative values whereas the values of i_{corr} increased with the increase in temperature, but by different amounts, indicating that the natural extracted compound affected the zinc electrochemical dissolution. On the other hand, the increase in temperature led to a decrease in the inhibition efficiency and the best inhibition efficiency was obtained at 303 K.

In examining the effect of temperature on the corrosion process in the presence of the extract, the Arrhenius equation below was used [22]:

$$K = \frac{Ae^{-E_a}}{RT}, \tag{7}$$

where K is the rate constant of the metal dissolution reaction that is directly related to corrosion current density i_{corr} [23], A is the frequency factor, T is the absolute temperature, and E_a is the activation energy. By plotting log K versus $1/T$ the values of E_a can be calculated from the slope of the obtained straight lines (Figure 5). The values of E_a determined from the Arrhenius plots correspond to 9.83 kJ mol^{-1} in absence and 13.18 kJ mol^{-1} in presence of inhibitor. From the obtained values of E_a, it is observed that the presence of the extract compound increased the activation energy values and consequently decreased the metal corrosion rate. These findings indicate that extracted compound acted as inhibitor and affect on the corrosion process through increasing the activation energy of metal dissolution by making a barrier to charge and mass transfer by their adsorption on metal surface.

An alternative formulation of the Arrehnius equation is the transition state equation [24, 25]:

$$K = \frac{RT}{Nh}\exp\left(\frac{\Delta S^*}{R}\right)\exp\left(\frac{-\Delta H^*}{RT}\right), \tag{8}$$

where h is Planck's constant, N is Avogadro's number, ΔS^* is the entropy of activation, and ΔH^* is the enthalpy of activation.

Figure 6 shows a plot of $\log(K/T)$ versus $(1/T)$ giving a straight lines with a slope of $(-\Delta H^*/2.303R)$ and an intercept of $(\log R/Nh + S^*/2.303R)$ from which the values of ΔH^* and ΔS^* are calculated and listed in Table 5.

As shown from Table 5, the positive values of ΔH^* reflect a strong adsorption of the inhibitor on the surface. The values of entropy of activation ΔS^* in the absence and presence of the studied extract compound are negative, and this indicates an increase in the system order in which the activated complex in the rate determining step represents an association rather than a dissociation step [25].

3.5. Electrochemical Impedance Spectroscopy Studies. Electrochemical impedance spectroscopy (EIS) measurements were carried over the frequency range from 10 kHz to 100 mHz at open circuit potential. The sample equivalent Randle circuit is shown in Figure 7 obtained for zinc in 1.0 M HCl with and without inhibitor, where R_s (Ωcm^2) represents the solution and corrosion products film; the parallel combination of resistor, R_{ct}, (charge transfer resistance) and capacitor, C_{dl}, (double-layer capacitance) which represents the corroding interface.

The Nyquist plots for zinc in 1.0 M HCl solution without and with different concentrations of inhibitor at 30°C are shown in Figure 8. The charge transfer resistance values (R_{ct}) were calculated from the difference in impedance at higher and lower frequencies, as suggested by Haruyama et al. [26].

From inspection of the impedance quantitative results listed in Table 6, it is seen that the R_{ct} values of the investigated extract compound increase with increasing its concentration. At the same time the C_{dl} has opposite trend in the same concentration range. These observations clearly bring out the fact that the corrosion of zinc in 1.0 M HCl is controlled by a charge transfer process. The decrease of C_{dl} values by the increase of the extract concentration is due to its adsorption on the electrode surface leading to a barrier film formation on the Zn surface and hence decreasing the extent of dissolution reaction [27]. Inspection of Tables 1, 2, 3, and 6 reveals that the inhibition percentages are increased as the concentration of marjoram extract is increased for all used methods, but the inhibition percentages calculated from polarization measurements for all concentrations of the extracts are ways higher than those obtained from chemical measurements. Similar observation on the difference between chemical and electrochemical measurements has been reported before [28]. The differences observed can be attributed to the fact that the chemical methods give average corrosion rates, whereas electrochemical methods give instantaneous corrosion rates [29].

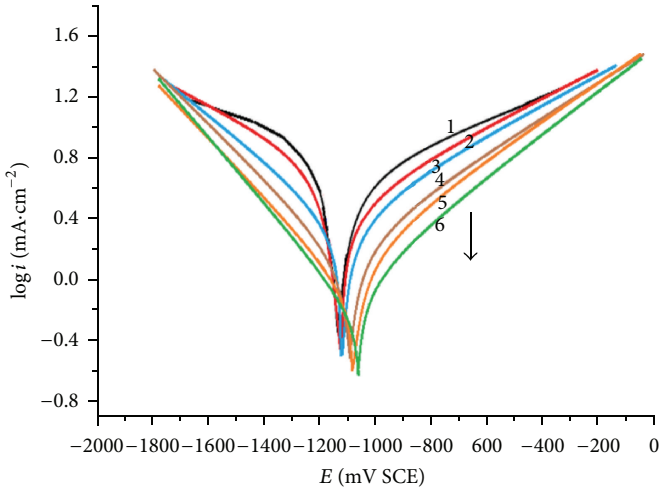

Figure 4: Potentiodynamic polarization curves of zinc electrode in 1.0 M HCl solutions containing different concentrations of Marjoram extract at 30°C. (1) 0.00 ppm, (2) 100 ppm, (3) 200 ppm, (4) 300 ppm, (5) 400 ppm, (6) 500 ppm.

Table 3: Electrochemical parameters for zinc in absence and presence of different concentrations of Marjoram extract in 1.0 M HCl solution at 30°C obtained from Tafel polarization curves.

Inhibitor concentration (ppm)	$-E_{corr}$ (mV SCE)	b_a (V dec^{-1})	b_c (V dec^{-1})	I_{corr} (mA cm^{-2})	Θ	IE (%)
HCl (1 M)	996	0.43	0.49	2.41	—	—
100	989	0.56	0.56	0.93	0.61	61.40
200	985	0.63	0.60	0.65	0.73	73.00
300	978	0.71	0.68	0.47	0.80	80.50
400	976	0.80	0.73	0.24	0.90	90.00
500	974	0.86	0.79	0.18	0.92	92.50

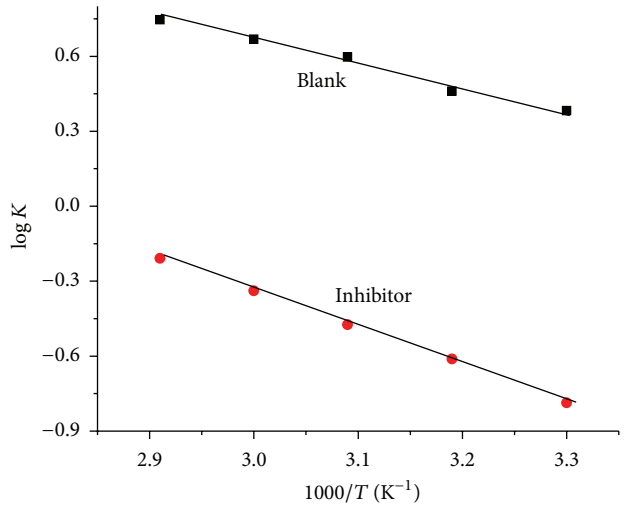

Figure 5: Relation between log K and the reciprocal of the absolute temprature of zinc electrode in 1.0 M HCl devoid of and containing 500 ppm of Marjoram extract.

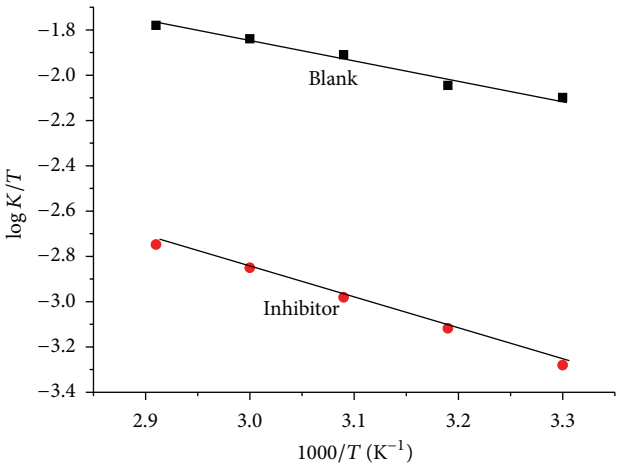

Figure 6: Arrhenius plot of log K/T versus $1/T$ for the dissolution of zinc in 1.0 M HCl in the absence and presence of Marjoram extract.

4. Inhibition Mechanism

The inhibitive action of naturally occurring Marjoram extract toward the corrosion of zinc metal could be attributed to the adsorption of its components on the metal surface. The adsorbed layer acts as a barrier between the metal surface and aggressive solution leading to a decrease in the corrosion rate. From the inspection of chemical composition of Marjoram extract, it appears that this extract is available source of many natural organic components as mentioned above. These components are chemical organic compounds that contain hetero atoms with free electron pairs and, hence, can

Table 4: Effect of temperature on the corrosion parameters of zinc in 1.0 M HCl and 1.0 M HCl + 500 ppm of marjoram extract.

System	T (K)	E_{corr} (mV SCE)	I_{corr} (mA cm^{-2})	IE (%)
1.0 M HCl	303	−996	2.41	—
	313	−1002	2.81	—
	323	−1001	3.96	—
	333	−999	4.73	—
	343	−1001	5.58	—
1.0 M HCl + 500 ppm inhibitor	303	−974	0.18	92.50
	313	−979	0.25	91.10
	323	−985	0.38	90.40
	333	−990	0.49	89.60
	343	−990	0.66	88.20

Table 5: Activation parameters of the dissolution reaction of zinc in 1.0 M HCl in the absence and presence of 500 ppm of marjoram extract.

System	E_a (kJ mol^{-1})	ΔH^* (kJ mol^{-1})	ΔS^* (J mol^{-1})
1.0 M HCl	9.83	16.12	−209.35
1.0 M HCl + 500 ppm inhibitor	13.18	23.20	−213.40

Table 6: Impedance data and surface coverage for zinc electrode in 1.0 M HCl in absence and presence of different concentrations of Marjoram extract.

Inhibitor conc. (ppm)	R_s Ω cm^2	R_{ct} Ω cm^2	C_{dl} μF cm^{-2}	IE$_{(R)}$ (%)
0.00	0.95	51	22.30	—
100	1.15	198	19.43	74.10
200	1.30	318	17.11	83.50
300	1.95	353	15.13	85.60
400	2.13	385	10.85	86.30
500	2.65	440	9.53	88.80

adsorb on zinc surface through the electron pair on hetero atoms. The adsorption of these components led to decrease the reaction between zinc metal and acid media (HCl) and then decrease corrosion rate.

5. Conclusions

(1) There is a good agreement among the results obtained by different techniques of measurements.

(2) *Marjoram* extract has high inhibitory effect on the corrosion of zinc in 1.0 M HCl and the inhibition efficiency increases with increasing the extract concentration.

(3) The inhibitory effect of *Marjoram* extract results from its adsorption on the metallic surface through its electron-rich functional groups.

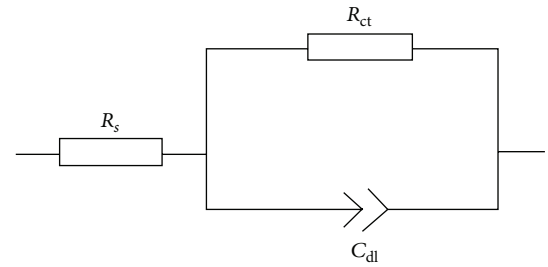

Figure 7: Electrical equivalent circuit ($R_s\Omega$ = uncompensated solution resistance, R_{ct} = charge transfer resistance and C_{dl} = double layer capacitance).

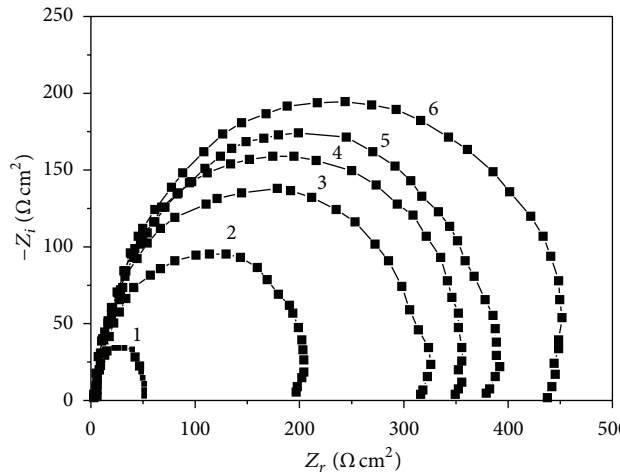

Figure 8: Nyquist plots for zinc electrode in 1.0 M HCl in absence and presence of different concentrations of Marjoram extract: (1) 0.00 ppm, (2) 100 ppm, (3) 200 ppm, (4) 300 ppm, (5) 400 ppm, and (6) 500 ppm.

(4) The adsorption of *Marjoram* extract on a zinc surface follows Langmuir adsorption isotherm.

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