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Ciprofloxacin as Eco-Friendly Corrosion Inhibitor for Carbon Steel in Hydrochloric Acid Solution

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Ciprofloxacin antibiotic was studied as a new corrosion inhibition for carbon steel (CS) in 1M hydrochloric acid solution using (mass loss (ML), hydrogen evolution (HE) and thermometric) as chemical methods at different temperatures, AC impedance (EIS), potentiodynamic polarization (PP) and electrochemical frequency modulation (EFM) as electrochemical techniques at 25°C. Polarization technique showed that the ciprofloxacin performs as a mixed-type inhibitor and give protection percentage reached to 91% in the presence of 300 ppm of the drug. Adsorption of the ciprofloxacin on surface of CS has obeyed Langmuir isotherm. Thermodynamic adsorption and activation parameters were calculated and discussed. Surface analyses have evaluated using different techniques.

Keywords: CS, Corrosion inhibition, HCl, ML, HE, PP, EFM, EIS, SEM, EDX.

1. INTRODUCTION

The corrosion of CS is a fundamental academic and industrial concern that has received a considerable amount of attention [1]. The utilized of inhibitors is the excellent practical tests for protection of metals against corrosion, especially in acidic media [2]. It has well recognized that N-heterocyclic compounds are measured to be the best corrosion inhibitors of steel in solution contain acid [3]. Most inhibitors utilized in industrial are organic assembles including of donor atoms like S, O and N. Inhibitors including triple or double bond performance significant role in simplifying the absorption of these compounds. A bond formed between the cloud π -electron and/or the electron pair of the donor atoms and the metal surface, thus bond lowering corrosive attack in acidic medium [4-13]. Many authors study the influence of some examines drugs (such as ampiclox, ampicillin, tetracycline, Cloxacillin, methocarbamol, orphenadrine, azithromycin, penicillin G, etc.) have been found to be

good inhibitors for the corrosion of metals. Numerous researches generally agree that drugs are inhibitors that can compete favorably with green corrosion inhibitors and that most drugs can be synthesized from natural products. The choice of some drugs used as corrosion inhibitors is based on the following: (a) drug molecules include O, S and N as active center, (b) drugs are reportedly environmentally friendly and significant in biological reactions and (c) simply created and purified [14-18].

In recent years, many authors use drugs as corrosion inhibitors for different metals, namely: Norfloxacin, Cefatrexyl, Ofloxacin drugs, Tacrine [19-21]. Ciprofloxacin drug is second-generation synthetic fluoroquinolone antibacterial agent, having greater activity against Gram-(+) bacteria and anaerobes [22].

In the current study, the corrosion inhibition of CS in 1 M HCl at different temperatures in the existence of ciprofloxacin antibiotic using chemical and electrochemical methods was tested. Morphology of the CS coins was examined using scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) without and with the drug.

2. EXPERIMENTAL TESTS

2.1. Materials

The chemical composition of CS present in this research (weight %) is: 0.2 % C, 0.003 % Si, 0.024 % P, 0.35 % Mn and the remainder Fe. The specimens were mechanically cut into about 2 cm \times 2 cm \times 0.2 cm sizes for ML, and 1cm \times 1cm for electrochemical tests. These specimens were polished with sand paper 1000, 1500 and 2000 to a metallic shine, degreased with absolute ethanol, washed with bidistilled water and finally dried.

2.2. Inhibitor

The pharmaceutical compound which used in this study is called ciprofloxacin, is easily soluble in water, has larger molecular weight, contains many donating atoms (P, N and O), and easily available as pharmaceutical drugs, non-toxic and its structure is listed below:



C₁₇H₁₈FN₃O₃ 331.346

Figure 1. Molecular structure of Ciprofloxacin

2.3. Solutions

The corrosive solutions, hydrochloric acid was diluted to 1M HCl as a corrosive medium and the investigated inhibitor was prepared by dissolving the appropriate weight of the solid pharmaceutical compound in bidistilled water, the other concentrations of the drug (0.05 - 0.3 M) were prepared by dilution with bidistilled water. All the materials utilized were of AR grade and utilized as received.

2.3. Methods

2.3.1. Chemical calculations

A. ML Measurements

The pretreated CS coins were inundation in 100 ml solution of 1.0M HCl in the existence and nonexistence of different concentration of ciprofloxacin at 25°C, 30°C, 35°C, 40°C and 45°C for 3h. The range of ciprofloxacin concentration in test solutions, has selected to be 50- 300 ppm. After indicating the time the CS specimens had removed from corrosive solution, rinsed with water distilled, dried and massed [23]. The ML has recorded to nearest 0.0001g. Inhibition efficiency was measured from the following relations [24]

 $\% IE = \theta \times 100 = [1 - (W/W^{\circ}) \times 100$ (1)

Where W° and W are the ML in the uninhabited and inhibited of inhibitor, correspondingly.

B. Thermometric test

This method was used in case of exothermic reactions. Rise of temperature with time (in minute) due to the reaction of the metal with the environment was recorded and plotted. The reaction number (RN) and corrosion efficiency (% I) were measured using equations 2 and 3 correspondingly:

RN (°C min) = (T_m - T_i) / t (2) %I = [(RN_b - RN_w)/RN_b] x 100 (3)

Where T_m is the maximum temperature and T_i is the initial temperature attained by the system at time t, RN_b and RN_w are the reaction numbers for the unprotected and protected systems,

C. Gasometric test

correspondingly.

Measurements of liberated hydrogen were carried out at 25° C. From the volume of liberated hydrogen per minutes, corrosion efficiency (% I) and degree of surface coverage (θ) were measured utilizing equations 4 and 5, correspondingly:

$$\% I = 100 x (1 - V'Ht / V'Ht)$$
(4)

$$\theta = \% \mathrm{I} / 100 \tag{5}$$

Where V'Ht is the liberated hydrogen volume at time t for inhibited solution and V^0 Ht is the volume of liberated hydrogen for uninhibited solution.

2.3.2. Electrochemical measurements

The behavior of ciprofloxacin as an inhibitor for corrosion of CS in 1.0 M HCl was further examined by PP, EIS and EFM. All electrochemical experiments were achieved at 25°C using threeelectrode cell setup consists of (SCE) a saturated calomel electrode as electrode reference, (Pt) platinum wire as counter electrode and CS as (WE) working electrode. The WE was prepared as follows at first one side of a CS sheet. Subsequently, the copper wire which attached to CS sheet was inserted into a glass tube and then fixed by epoxy resin to make the size of CS visible to the test solutions is 1 cm². The sample was pretreated as mentioned before and the potential of electrode was allowed to stabilize for half hour in order to reach a steady state. For PP test the potential was initiated from -500 to 500 mV vs OCP with rate of scan 1 mVs⁻¹. EIS tests were done in a range of frequency from 10 mHz to 1000 kHz with an amplitude of 10 mV peak to peak under open circuit condition. EFM was used as nondestructive method for dissolution of CS data without knowledge of Tafel slopes. EFM technique with amplitudes 10 mV to 2 and 5 Hz. The electrochemical methods were performed using Gamry Instruments Series (G750TM Potentiostat / Galvanostat / ZRA with Gamry applications contain DC105 software for PP, EFM140 software for EFM, and EIS300 software for EIS analysis. Echem Analysts 5.5 Software loaded at computer that used for finding fitting data.

2.3.3. SEM-EDX Analysis

SEM and EDX tests were tested for surface morphology of CS exposed to 1 M HCl contain 300 ppm of the ciprofloxacin for 24 hours at 25 °C, and determining the weight % of the atoms on the surface by (EDX) using (SEM: JOEL 840,Japan) with a magnifying power of (x2000) speed.

3. RESULTS AND DISCUSSION

3.1. Chemical Measurements

A. ML Measurements

Table (1) displays the mass loss data; (CR) corrosion rate, (%IE) inhibition efficiency, and (θ) surface of the CS coverage in 1.0 M HCl with and without different concentrations of ciprofloxacin which can be measured by equation (1). The addition of ciprofloxacin attended by decreasing in ML and CR while, raising in %IE and θ . This ensures that efficiency of ciprofloxacin as (%IE) at an optimum concentration 300 ppm reach 91%. Figure (1) shows ML-time curves of CS with and without different concentrations of ciprofloxacin at 25°C. From table (1) by raising temperature, ML, increases indicating that the CR of CS increases while the protection efficiency of the ciprofloxacin decreases

Conc.,	Conc., 25°C		30	30°C		35°C		40°C		45°C	
ppm	CR	% IE	CR	% IE	CR	% IE	CR	% IE	CR	% IE	
50	0.096	71.4	0.138	68.9	0.220	66.6	0.345	59.6	0.465	52.8	
100	0.087	74.3	0.125	71.7	0.203	69.1	0.331	61.3	0.428	56.5	
150	0.079	76.7	0.088	74.3	0.181	72.5	0.301	64.8	0.392	60.2	
200	0.057	83.2	0.100	77.5	0.167	74.6	0.260	69.5	0.351	64.3	
250	0.038	88.8	0.085	80.8	0.146	77.8	0.234	72.6	0.310	68.5	
300	0.032	90.5	0.074	83.2	0.127	80.8	0.214	74.9	0.268	72.8	

Table 1. Effect of ciprofloxacin concentration on CR and inhibition efficiency (% IE) in 1.0M HCl at different temperatures at 120 min immersion





A.1. Temperature Effect

The temperature effect on the CR of CS in 1.0 M HCl at different concentrations from ciprofloxacin was test by ML method over a temperature range from 25 to 45°C. The influence of temperature on the CR and IE% obtained was obtained by using ML method. The obtained data showing that, the CR improves as the temperature rise and lowered as the concentration of ciprofloxacin rise Figure (3).

The CR dependence on temperature can be represented by Arrhenius relation:

 $CR = A \exp(-Ea^*/RT)$

Where A = factor pre-exponential and E_a^* is the energy of activation for process of corrosion. Figure (4) display the Arrhenius curves with and without ciprofloxacin. E_a^* data measured from the slopes of these linear diagrams are written in Table (5) which indicated that the E_a^* data for corrosive solution is maximum than that for unprotected solution, suggesting that dissolution of CS is smaller in the existence of the drug and can be assumed that the adsorption of drug molecules on CS surface is due to physical adsorption [25-26].



Figure 3. Effect of temperature on CR and protection efficiency (%IE) of CS in 1M HCl without and with different concentrations of ciprofloxacin



Figure 4. Arrhenius plots of CS corrosion in 1.0 M HCl with and without various concentrations ciprofloxacin



Figure 5. Log (CR/T) vs 1/T for CS corrosion in 1.0 M HCl with and without various concentrations of ciprofloxacin

Table 2. Thermodynamic activation parameters for CS corrosion with and without different concentrations of ciprofloxacin in 1.0 M HCl

Concentration	Activation parameters						
Concentration,	$E_a^* kJ mol^{-1}$	ΔH^*	$-\Delta S^*$ J mol ⁻¹ K ⁻¹				
ppm	kJ mol ⁻¹	kJ mol ⁻¹	J mol ⁻¹ K ⁻¹				
Blank	42.2	41.6	114.3				
50	61.2	61.4	60.1				
100	62.8	63.0	55.0				
150	63.1	63.3	53.8				
200	64.4	64.7	50.1				
250	66.5	66.9	45.2				
300	66.9	68.1	44.2				

Enthalpy and entropy of activation (ΔH^* , ΔS^*) for the process of corrosion was measured from the transition state relation:

 $CR = RT/Nh \exp (\Delta S^*/R) \exp (-\Delta H^*/RT)$

(7)

Where, h is the Boltzmann constant and N is Avogadro's number. ΔH^* and ΔS^* can be obtained through the intercept and slope of curves of log (CR/T) vs (1/T) as shown in Figure (5). The data of E_a^* , ΔH^* and ΔS^* are recorded in Table (2), negative value of ΔS^* meaning that there is a decrease in disorder occurring during movement of transition from reactants to the activated complex [27].

A.2. Adsorption Isotherms

Adsorption isotherms have utilized to recognize the mechanism of protection on the metal surface [28]. The best fit was obtained using Langmuir isotherm, which is indicated in Figure (6) for ciprofloxacin expressed by the next relation (8):

$$C/\theta = 1/K_{ads} + C$$
(8)

Where C is the concentration of drug in the bulk electrolyte (Mol L⁻¹) and K_{ads} is the adsorption equilibrium constant. Gibbs free energy (ΔG^o_{ads}) can be calculated by the relation (9):

$$K_{ads} = (1/55.5) \exp \left(\Delta G^{o}_{ads} / RT\right)$$
(9)

Where 55.5 is the molar concentration of water in the bulk solution in M^{-1} , R is gas constant, T is the temperature in Kelvin.



Figure 6. Plots Langmuir isotherms for fitting corrosion value for CS in 1.0 M HCl in the presence and absence of different concentrations of ciprofloxacin



Figure 7. Log K_{ads} vs (1000 / T) for corrosion of CS in 1.0 M HCl in the presence of ciprofloxacin.

Thermodynamic adsorption parameters of the ciprofloxacin on CS surface in 1.0 M HCl at different temperatures were record in the Table (3). It was establish that ΔG°_{ads} has –ve data among 20.6 to 22 kJ mol⁻¹ describing that the ciprofloxacin adsorption is a spontaneous process and belong to physisorption mechanism [29].

The standard enthalpy ΔH°_{ads} and ΔS°_{ads} can be measured using relation (10):

$$\Delta G^{\circ}_{ads} = \Delta H^{\circ} ads - T\Delta S^{\circ}_{ads}$$
(10)

The ΔH°_{ads} data was evaluated from the plot of Log K_{ads} vs1/T see Figure (7). The –ve sign data of ΔH°_{ads} ensures that the process of adsorption is an exothermic one. This can described on the basis that rises of temperature causes desorption of some adsorbed ciprofloxacin molecules from the CS surface and accordingly lead to decrease the protection efficiency.

 $-\Delta G^{o}_{ads}$ $-\Delta H^{o}_{ads}$ $\overline{\Delta S^{o}}_{ads}$ Kadş, Temp M^{-1} kJ mol $J mol^{-1} K^{-1}$ °C $kJ mol^{-1}$ 25 19.7 51.2 66.0 30 43.4 19.6 64.6 35 36.7 19.5 33.5 63.2 40 29.8 19.2 61.5 45 21.3 18.7 58.7

Table 3. Parameters for the corrosion of CS in 1.0 M HCl at different temperatures

B. Hydrogen Evolution (HE)



Figure 8. Volume of hydrogen evolved against time for CS in 1.0 M HCl solutions with different concentrations of ciprofloxacin at 25 $^{\circ}$ C

Slopes of lines of Figure (8) are measured and obtained as the CR as confirmed before. The linearity in the relations indicates the presence of insoluble film on the CS surface throughout corrosion.

Conc., ppm	$(CR),$ $ml \ cm^{-2} \ min^{-1}$	θ	% IE
Blank	0.4191		
50	0.16441	0.608	60.8
100	0.13572	0.676	67.6
150	0.12548	0.701	70.1
200	0.0718	0.828	82.8
250	0.0497	0.881	88.1
300	300 0.0387		90.7

The measured CR obtained from HE vs. concentration are shown in Table 1. As observed, CR lowered with increase ciprofloxacin concentration. This is due to the adsorption of drug molecules on CS surface [30].

C. Thermometric tests

In this test, the temperature variation has observed with and without different concentrations of the ciprofloxacin. Figure (9) characterized the performance detected in the existence of different concentrations of drug. All diagrams of the tested material are categorized by an initial slow rise [31] followed by a sharp rise and finally by a breakdown in temperature after achieving a maximum. The diagrams for adding drug fall below that of the free acid. This indicates that the adding drug behaves as inhibitor over the added concentration range. The percentage reduction in RN of the drug has tabulated in Table (5). The obtained data are reported in Table (5) showing the protection inhibition as calculated from the RN of different concentrations of the drug.

Table 5. The protection efficiency of ciprofloxacin from thermometric tests

Con, M/L	R.N C/min	θ	% IE
Blank	0.200		
0.05	0.094	0.528	52.8
0.1	0.077	0.614	61.4
0.15	0.056	0.722	72.2
0.2	0.041	0.796	79.6
0.25	0.034	0.828	82.8
0.3	0.023	0.890	89.0



Figure 9. Temperature--time curves for CS corrosion with and without various concentrations of The drug

- 3.2. Electrochemical measurements
- A. Potentiodynamic Polarization Measurements.



Figure 10. Potentiodynamic Polarization curves for the dissolution of CS in 1.0 M HCl with and without various concentrations of ciprofloxacin at 25°C

Figure (10) demonstrates that the polarization performance of CS corrosion in 1.0 M HCl with and without various ciprofloxacin concentration. The measured data (i_{corr}), (E_{corr}), (β_c), (β_a), (θ) and (% IE) were estimated from the curves of Figure (10) and determined from eq. 12:

% IE =
$$\Theta \ge 100 \left[1 - (i_{\text{corr(inh)}} / i_{\text{corr(free)}}) \right]$$
 (12)

Table 6 reported the data of PP. The data of %IE in Table 6 are in large validly with data obtained from ML and H_2 evolution. In addition, the addition of ciprofloxacin lowered both oxidation of metal and H_2 release, furthermore lower the current in both cathodic and anodic. The drug behaves as a mix-kind inhibitor by way of the measured data confirm the retardation in i_{corr} , which are in good valid calculation with the greater IE% in comparing various dissolution tests [32].

Table 6. PP parameters	for CS with and v	vithout different	concentratio	ns of ciprof	loxacin At	$25^{\circ}C$

Conc, ppm	i _{corr.} mA cm ⁻²	-E _{corr.} mV vs SCE	β_a mV dec ⁻¹	β_c mV dec ⁻¹	CR mpy	θ	% IE
Blank	10.985	443	398	481	210	-	-
50	3.295	450	311	390	5.6	0.700	70.0
100	2.965	460	303	372	6.8	0.731	73.1
150	2.526	450	301	365	7.1	0.773	77.3
200	1.933	455	272	325	8.1	0.824	82.4
250	1.757	461	250	291	8.6	0.840	84.0
300	1.203	463	245	278	5.9	0.893	89.3

B. (EIS) Measurements



Figure 11. Nyquist diagrams for CS in existence of various concentrations of ciprofloxacin at 25°C

Figure (11) demonstrations the Nyquist diagrams for CS with and without different concentrations of ciprofloxacin at 25°C. These curves have a semicircle form; it designates that the corrosion of ciprofloxacin is mostly controlled by charge transfer résistance. The Bode curves for the CS is presented in Figure (12) where the great frequency boundary resembles to electrolyte resistance R_{Ω} , however the little frequency limit signifies the summation of $(R_{\Omega} + R_p)$. Various parameter obtained from EIS such as (R_{ct}) , (C_{dl}) and (% IE) were calculated and are recorded in Table (7). The obtained data showed that the values of (R_{ct}) improve and the values of layer (C_{dl}) decreased with

raising the concentration of ciprofloxacin, because the adsorption of ciprofloxacin and forming film on the CS surface. The obtained Nyquist curves have not gave perfect semicircle. Due to the heterogeneity of the CS surface [33-34]. In 1.0M HCl and existence of different concentrations of ciprofloxacin, the impedance demonstrates the similar trend. Nevertheless, the capacitive loop diameters improves with raising concentration of ciprofloxacin.



Figure 12. Bode curves for CS with and without various concentrations of ciprofloxacin at 25°C and Equivalent circuit utilized to fit the impedance data

Conc, ppm	$R_p, \Omega cm^2$	$C_{dl}, \mu F cm^{-2}$	θ	% IE
0.0	12.3	220		
50	53.7	177	0.771	77.1
100	72.5	129	0.830	83.0
150	76.5	100	0.839	83.9
200	79.5	83	0.845	84.5
250	96.9	111	0.873	87.3
300	125.3	85	0.901	90.1

Table 7. EIS data of ciprofloxacin with and without different concentrations of at 25°C

C. EFM test

EFM is not-destructive corrosion test that give an exact and rapid determine of the corrosion current of CS without prior knowledge of Tafel slopes. These indispensability of EFM data support and allow us to governor for corrosion procedure [35]. The causality factor have very important for thought EFM which have internally forms of validly EFM for determine data. Figure (13) indicates EFM spectrum, involve the quantity of current as frequencies' a purposes. The high peaks were applied

to figure out the (CF-2 & CF-3), ($\beta_c \& \beta_a$) and the dissolution current density (i_{corr}), %IE were estimated from equation (13).

$$\% IE_{EFM} = [1 - (i_{corr}/i_{corr}^{0})] \times 100$$
(13)

Electrochemical data at several concentrations for created inhibitors in destructive solution 1.0 M HCl at 298K display in Table (8) which explain that by increasing the concentration of the drug, %IE _{EFM} increased. Theoretically, the data of CF-2 and CF-3 are nearly 2 and 3 present that the obtained values calculated are in large validity [36].



Figure 13. EFM curves for CS in existence of various concentrations of ciprofloxacin at 25°C

Table 8. EFM parameters for CS with and without different concentrations of ciprofloxacin at 25°C

Conc, ppm	$i_{corr.}$ $\mu A \text{ cm}^{-2}$	β_a mV dec ⁻¹	$\begin{array}{c} \beta_c \\ mVdec^{-1} \end{array}$	CF(2)	CF(3)	CR mpy	θ	% IE
0.0	625	87	153	1.98	3.10	285	-	-
50	205	63	143	2.02	3.00	95	0.672	67.2
100	165	59	133	2.01	3.01	75	0.736	73.6
150	115	57	125	2.04	3.04	52	0.816	81.6
200	109	55	120	2.01	3.01	49	0.826	82.6
250	100	54	115	1.98	3.00	43	0.840	84.0
300	67	55	102	1.94	2.97	30	0.893	89.3

3.3 SEM-EDX Measurement

The micrographs of polished CS surface dipped in 1.0M HCl solution with and without 300 ppm ciprofloxacin for 24 h at 25°C are shown in Figure (14a, c). Figure (14a). It is clear that CS surface is very smooth (low corroded), Figure (14b) while the unprotected surface which affected by corrosive medium and suffer from cracking, Figure (14c) in the presence of the ciprofloxacin we found that CS surface resist the corrosion effect due to film formation.



Figure 14(a,b and c): SEM graph of CS surface (a) previously of immersion in acid, (b) after 24 h of immersion in acid and (c) after 24 h of immersion in acid + ciprofloxacin 300 ppm

Figure (15) The EDX spectra display adding lines, representative the presence of C (due to the ciprofloxacin C atoms). The spectra show that the C and O atoms present on the coins surface. This data is due to the inhibitor. It is gotten that, in appending to O, S, and C were existence in the EDX. Similar elements supply is shown in Table (9).



Figure 15. EDX examination on CS with and without ciprofloxacin for 24 h

Table 9. Surface element (weight %) of CS coins with and without various concentrations of
ciprofloxacin in 1.0 M HCl solution at 298K.25°C

(Mass %)	Fe	Mn	Р	0	Ν	С	Cl
Free	98.27	0.81	0.04	-	-	0.79	-
HCl	71.95	0.65	0.03	25.7	-	1.14	0.6
Inhibitor	62.6	0.55	0.01	13.5	11.1	14.5	0.14

3.4. Mechanism of corrosion inhibition

The adsorption of drug molecules can be attributed to the existence of polar unit having atoms of nitrogen and oxygen and aromatic/heterocyclic rings. Therefore, the possible reaction centers are unshared electron pair of hetero-atoms and π -electrons of aromatic ring [37]. The possible explanation of the inhibition is due to adsorption process which is considered as the key of the mechanism of inhibition action. It might be proposed that the drug molecules adhere to the steel surface. This leads to a decrease of the surface area at which cathodic and anodic reactions take place. Inhibition efficiency of the drug molecules depends on many factors [38], which include the number of adsorption active centers in the molecule and their charge density, molecular size, and mode of interaction with metal surface[39- 40]. This molecule will present in the protonated form, so it can adsorb directly on the negative surface of CS in acidic medium by electrostatic attraction as shown below



Table (10) gives a comparison of %IE with different used techniques. The chemical methods considerably significant corrosion %IE compared to other electrochemical methods.

Table 10. Comparison of corrosion efficiencies (verified using all tests at 25°C) obtained for corrosionof CS in 1.0M HCl solutions including 300 ppm of the studied inhibitor

Methods		Chemical		Electrochemical		
	ML	Thermometric	ΗE	EFM	EIS	Polarization
% IE	90.3	89	90.7	89.3	90.1	89.3

4. CONCLUSIONS

The investigated Ciprofloxacin inhibits the corrosion of CS in 1 M HCl. The inhibition is due to adsorption of the drug molecules on the C-steel surface by blocking its active sites. Adsorption of drug fits Langmuir isotherm. Also the results show by increasing inhibitor concentration the inhibition efficiency increased. Polarization data showed that the used inhibitor act as mixed-type inhibitor in 1 M HCl. Results obtained from ML, H₂ evolution, thermometric, PP, EIS and EFM techniques are reasonably in good agreement

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