# New antipyrine derivatives as corrosion inhibitors for C-steel in 1M hydrochloric solutions

A. S. Fouda[1,*], S. A. Abd El-Maksoud[2] and S. R. Abd El-Salam[1]

[1]Chemistry Department, Faculty of Science, El -Mansoura University, El-Mansoura, Egypt.

[2]Chemistry Department, Faculty of Science, Port Said University, Port Said, Egypt.

**ABSTRACT**

The adsorption and inhibition efficiency effect of antipyrine derivatives on the c-steel alloy in 1M HCl were investigated using electrochemical techniques (potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) and electrochemical frequency modulation (EFM)). The inhibition efficiency of the inhibitors increased with increasing the concentration of the derivatives and increased also with increasing the temperature. Polarization curves revealed that these compounds are chemical type inhibitors. The adsorption of these compounds follows Langmuir adsorption isotherm. Some thermodynamic parameters of activation and adsorption processes were also determined and discussed.

## Introduction

Corrosion is a fundamental process playing an important role in economics and safety, particularly for metals. The use of inhibitors is one of the most practical methods for protection against corrosion, especially in acidic media [1].Most well-known acid inhibitor are organic compounds containing nitrogen , sulfur, and oxygen atoms. Among them, organic inhibitors have many advantages such as high inhibition efficiency, low price, low toxicity, and easy production [2-5].Organic heterocyclic compounds have been used for the corrosion inhibition of iron [6-11], copper [12], aluminum [13-15], and other metals [16-17] in different corroding media. The adsorption of the surfactant heterocyclic compounds on the metal surface can markedly change the corrosion- resisting property of the metal [18-19] and so the study of the relations between the adsorption and corrosion inhibition is of great importance. Heterocyclic compounds have shown a high inhibition efficiency for iron in both HCl [20] and H2SO4 [21] solutions.
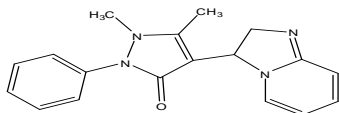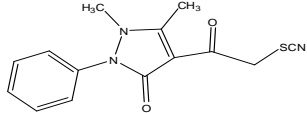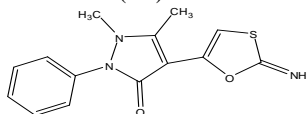
As antipyrine compounds have been studied as corrosion inhibitors before we also used some derivatives of it to inhibit corrosion of c-steel in 1M HCl. For this reason, the objective of the present work is to investigate the inhibiting action of antipyrine derivatives in 1M HCl at 25-45 ºC using different methods.

## Materials and Methods

The working electrode was made from c-steel rod. The rod was mounted into a glass tube and fixed by araldite leaving a circle surface geometry of 1 cm diameter to contact the test solution. Prior to each experiment, the working electrode was polished with a different grades of emery paper up to 1200 grit, rinsed with acetone and finally with doubly distilled water. The auxiliary electrode was platinum wire, while a saturated calomel electrode (SCE) connected to conventional electrolytic cell of capacity 100 ml.

The experiments were conducted in 1M HCl solution and with different concentration (3x10-6 – 18x10-6 M) of organic compounds. The structures and molecular weights of the investigated organic compounds are shown in Figure (1) [22]. All solutions were freshly prepared using analytical grade reagents and doubly distilled water. All experiments were performed at required temperature ±1ºC

| No | Structures and names | Chemical formula | Mol. Wt |
|---|---|---|---|
| 1 | 4-(2,3-dihydroimidazo[1,2-a]pyridin-3-yl)-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one  | $C_{18}H_{18}N_4O$ | 306.36 |
| 2 | 1,5-dimethyl-2-phenyl-4-(2-thiocyanatoacetyl)-1H-pyrazol-3(2H)-one  | $C_{14}H_{13}N_3O_2S$ | 287.34 |
| 3 | 4-(2-imino-1,3-oxathiol-5-yl)-1,5-dimethyl-2-phenyl-1H-pyrazol-3(2H)-one  | $C_{14}H_{13}N_3O_2S$ | 287.34 |

## Results and Discussion

### 1- Polarization measurements

Polarization experiments were carried out in a conventional three-electrode cell with a platinum counter electrode and a saturated calomel electrode (SCE) coupled to a fine Luggin capillary as reference electrode. The working electrode was in the form of a square cut from C-steel embedded in epoxy resin of polytetrafluoroethylene (PTFE) so that the flat surface was the only surface of the electrode. The

working surface area was 1.0×1.0 cm. Tafel polarization curves were obtained by changing the electrode potential automatically from -500 to +500 mV at open circuit potential with a scan rate of 1 mVs-1. Stern-Geary method [23]used for the determination of corrosion current is performed by extrapolation of anodic and cathodic Tafel lines to a point which gives log icorr and the corresponding corrosion potential (Ecorr) for inhibitor free acid and for each concentration of inhibitor. Then icorr was used for calculation of inhibition efficiency and surface coverage (θ) as below:

IE % = θ x 100 = [1- (icorr(inh) / icorr(free) ] × 100　　　(2)

where icorr(free) and icorr(inh) are the corrosion current densities in the absence and presence of inhibitor, respectively.

Impedance measurements were carried out in frequency range from 100 kHz to 10 mHz with amplitude of 5 mV peak-to-peak using ac signals at open circuit potential. The experimental impedance were analyzed and interpreted on the basis of the equivalent circuit. The main parameters deduced from the analysis of Nyquist diagram are the resistance of charge transfer Rct (diameter of high frequency loop) and the capacity of double layer Cdl which is defined as:

Cdl =1/ (2 π fmax Rct)　　　(3)

where fmax is the maximum frequency

The inhibition efficiencies and the surface coverage (θ) obtained from the impedance measurements were defined by the following relation:

IE % = θ x 100 = [1- (R°ct/Rct)] ×100　　　(4)

where Roct and Rct are the charge transfer resistance in the absence and presence of inhibitor, respectively.The electrode potential was allowed to stabilize 30 min before starting the measurements. All the experiments were conducted at 25 ± 1°C.

Measurements were performed using Gamry (PCI 300/4) Instrument Potentiostat/Galvanostat/ZRA. This includes a Gamry framework system based on the ESA 400. Gamry applications include DC105 for corrosion measurements and EIS300 for electrochemical impedance spectroscopy along with a computer for collecting data. Echem Analyst 5.58 software was used for plotting, graphing, and fitting data.

## Potentiodynamic polarization measurements

The decrease in the corrosion current density and the increase in the inhibition efficiency may be attributed to the adsorption of the investigated compounds on the alloy surface. The figure below shows the potentiodynamic polarization for highest inhibitor in inhibition the other two inhibitors aren't shown.
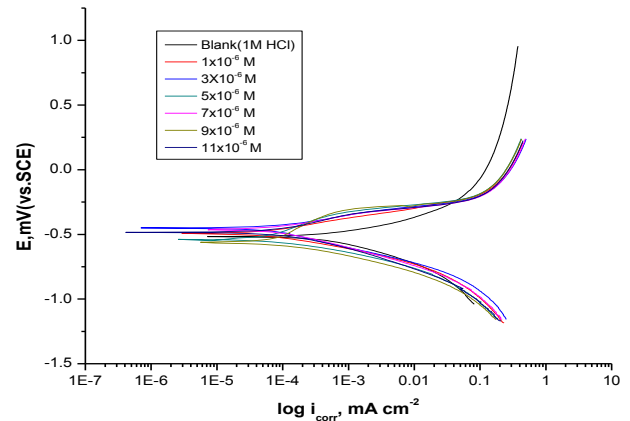


**Figure 1. Potentiodynamic polarization curves for the dissolution of C-steel alloy in 1M HCl in the absence and presence of different concentrations of compound (1) at 25º C**

## Electrochemical frequency modulation measurements

The EFM technique is used to calculate the anodic and cathodic Tafel slopes as well as the corrosion current densities for the investigated compound. Figure (4) show the EFM intermodulation spectra (spectra of current response as a function of frequency) of C-steel in 1M HCl the other two inhibitors aren't shown. The calculated electrochemical parameters (jcorr, βc, βa, CF-2, CF-3 and %IE) are given in Table (2). The values of causality factors obtained in absence and presence of investigated compounds. It shown from the Table that the corrosion current density decreases with increasing the concentration of the investigated compounds with respect to blank and hence the inhibition efficiency increases and indicate that the investigated compounds inhibit the acid corrosion of the alloy through adsorption[24]

**Table 1. The data obtained from potentiodynamic polarization measurements**

| Comp. | [inh] M | -E_corr mV vs SCE | i_corr μA cm$^{-2}$ | β_a mV dec$^{-1}$ | β_c mV dec$^{-1}$ | C.R Mpy | Θ | % IE |
|---|---|---|---|---|---|---|---|---|
| | Blank | 517 | 495.0 | 113 | 158 | 226.0 | ---- | ----- |
| 1 | 1 x 10$^{-6}$ | 491 | 145.0 | 83 | 121 | 66.4 | 0.707 | 70.7 |
| | 3x 10$^{-6}$ | 494 | 106.0 | 65 | 87 | 48.2 | 0.785 | 78.5 |
| | 5 x 10$^{-6}$ | 483 | 83.1 | 130 | 108 | 37.9 | 0.832 | 83.2 |
| | 7 x 10$^{-6}$ | 463 | 79.7 | 101 | 126 | 36.4 | 0.838 | 83.8 |
| | 9 x 10$^{-6}$ | 450 | 54.4 | 77 | 121 | 24.8 | 0.890 | 89.0 |
| | 11 x 10$^{-6}$ | 493 | 51.7 | 96 | 90 | 23.6 | 0.895 | 89.5 |
| 2 | 1 x 10$^{-6}$ | 499 | 139.0 | 72 | 103 | 63.5 | 0.719 | 71.9 |
| | 3x 10$^{-6}$ | 499 | 118.0 | 88 | 115 | 53.8 | 0.760 | 76.0 |
| | 5 x 10$^{-6}$ | 488 | 100.0 | 86 | 109 | 45.7 | 0.797 | 79.7 |
| | 7 x 10$^{-6}$ | 483 | 87.0 | 74 | 114 | 39.7 | 0.824 | 82.4 |
| | 9 x 10$^{-6}$ | 489 | 77.2 | 76 | 109 | 35.2 | 0.844 | 84.4 |
| | 11 x 10$^{-6}$ | 471 | 63.6 | 87 | 137 | 29.0 | 0.870 | 87.0 |
| 3 | 1 x 10$^{-6}$ | 514 | 225.0 | 77 | 98 | 103.0 | 0.545 | 54.5 |
| | 3x 10$^{-6}$ | 515 | 203.0 | 94 | 124 | 92.5 | 0.589 | 58.9 |
| | 5 x 10$^{-6}$ | 501 | 179.0 | 108 | 128 | 81.8 | 0.638 | 63.8 |
| | 7 x 10$^{-6}$ | 498 | 146.0 | 142 | 189 | 66.5 | 0.705 | 70.5 |
| | 9 x 10$^{-6}$ | 506 | 95.1 | 114 | 133 | 60.3 | 0.807 | 80.7 |
| | 11 x 10$^{-6}$ | 494 | 84.4 | 101 | 88 | 38.5 | 0.829 | 82.9 |

**Table 2.The following table shows the electrochemical frequency modulation results**

| Inhibitor | [inh] | $i_{corr}$ | $\beta_a$ | $\beta_c$ | CF-2 | CF-3 | C.R | Θ | % IE |
|-----------|-------|-----------|-----------|-----------|------|------|-----|----|----|
| Blank | 0 | 812.1 | 105 | 134 | 1.38 | 3.06 | 371.1 | - | - |
| 10 | 1 x10⁻⁶ | 219.4 | 77 | 200 | 1.97 | 2.55 | 100.2 | 0.729 | 72.9 |
|  | 3 x10⁻⁶ | 216.5 | 142 | 300 | 2.01 | 1.07 | 98.9 | 0.733 | 73.3 |
|  | 5 x10⁻⁶ | 166 | 162 | 232 | 1.84 | 1.06 | 75.8 | 0.795 | 79.5 |
|  | 7 x10⁻⁶ | 138.4 | 84 | 120 | 2.08 | 1.53 | 63.2 | 0.829 | 82.9 |
|  | 9 x10⁻⁶ | 119.3 | 151 | 228 | 2.04 | 3.60 | 54.5 | 0.853 | 85.3 |
|  | 11 x10⁻⁶ | 119.2 | 127 | 213 | 2.04 | 1.45 | 54.4 | 0.853 | 85.3 |
| 12 | 1 x10⁻⁶ | 247.8 | 86 | 91 | 1.71 | 2.84 | 113.2 | 0.694 | 69.4 |
|  | 3 x10⁻⁶ | 209.4 | 81 | 102 | 1.94 | 2.70 | 95.7 | 0.742 | 74.2 |
|  | 5 x10⁻⁶ | 193.1 | 83 | 96 | 1.90 | 2.98 | 88.2 | 0.762 | 76.2 |
|  | 7 x10⁻⁶ | 180 | 80 | 99 | 1.91 | 1.92 | 82.2 | 0.778 | 77.8 |
|  | 9 x10⁻⁶ | 179 | 78 | 113 | 1.96 | 2.67 | 81.8 | 0.779 | 77.9 |
|  | 11 x10⁻⁶ | 173.6 | 91 | 210 | 1.94 | 3.90 | 79.3 | 0.786 | 78.6 |
| 13 | 1 x10⁻⁶ | 422.5 | 106 | 127 | 1.93 | 2.70 | 193.0 | 0.479 | 47.9 |
|  | 3 x10⁻⁶ | 370.3 | 203 | 461 | 1.95 | 3.23 | 169.2 | 0.543 | 54.3 |
|  | 5 x10⁻⁶ | 353.7 | 84 | 105 | 1.92 | 2.84 | 161.6 | 0.564 | 56.4 |
|  | 7 x10⁻⁶ | 280 | 83 | 90 | 1.69 | 3.10 | 127.9 | 0.655 | 65.5 |
|  | 9 x10⁻⁶ | 269.6 | 97 | 102 | 1.30 | 3.09 | 123.2 | 0.667 | 66.7 |
|  | 11 x10⁻⁶ | 182 | 126 | 160 | 1.99 | 3.49 | 115.2 | 0.775 | 77.5 |
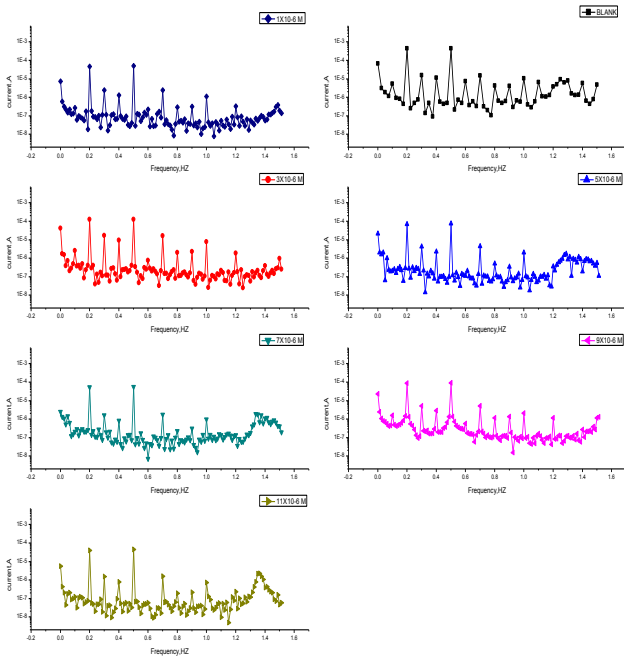


**Figure 2. EFM intermodulation spectra of C-steel alloy in 1M HCl for inhibitor (1)at 25ºC**

**Electrochemical impedance spectroscopy**

The effect of inhibitor concentration on the impedance behavior of C-steel alloy in 1M HCl solution at 25 ℃ Is presented in Fig(7) The curves show a similar type of Nyquist plots for C-steel in the presence of various concentrations of inhibitor (1) the other two inhibitors aren't shown . The existence of single semi-circle showed the single charge transfer process during dissolution which is unaffected by the presence of inhibitor molecules.

Deviations from perfect circular shape are often referred to the frequency dispersion of interfacial impedance which arises due to surface roughness, impurities, dislocations, grain boundaries, adsorption of inhibitors, and formation of porous layers and in homogenates of the electrode surface[25].
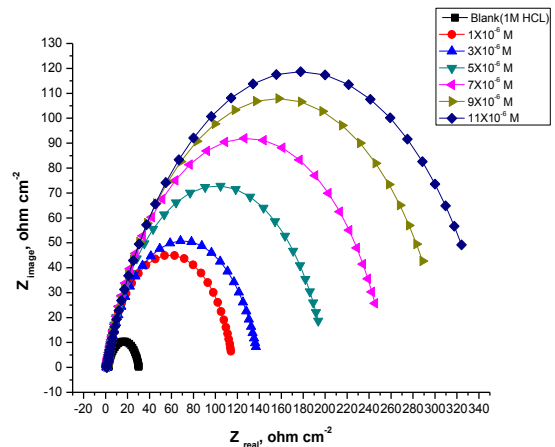


**Figure (3a). The Nyquist plots for corrosion of C-steel in 1 M HCl in the absence and presence of different concentrations of inhibitor (1) at 25ºC**
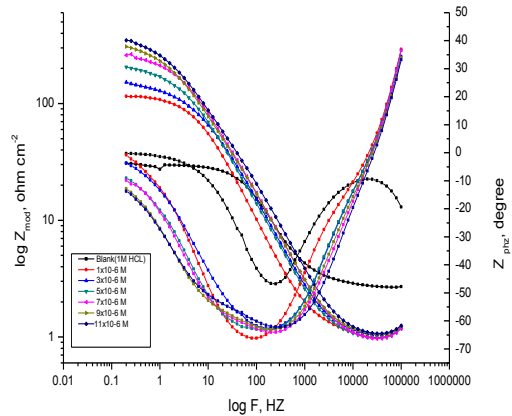


**Figure (3b). The Bode plots for corrosion of C-steel in 1 M HCl in the absence and presence of different concentrations of inhibitor (1) at 25ºC.**

**Table 3. Electrochemical kinetics parameters obtained by EIS technique for three inhibitors in 1M HCl at 25ºC**

| Inhibitor | [inh] M | $R_{ct}$, $\Omega$ cm$^2$ | $R_s$, $\Omega$ cm$^2$ | $C_{dl}$, $\mu$Fcm$^{-2}$ | $\Theta$ | % IE |
|---|---|---|---|---|---|---|
| Blank | 0 | 27.5 | 2.67 | 9.92 | ----- | ------ |
| 1 | 1 x10$^{-6}$ | 115.2 | 1.01 | 2.38 | 0.760 | 76.0 |
| | 3 x10$^{-6}$ | 139.1 | 1.00 | 1.97 | 0.801 | 80.1 |
| | 5x10$^{-6}$ | 200.9 | 0.939 | 1.36 | 0.862 | 86.2 |
| | 7 x10$^{-6}$ | 256.2 | 0.914 | 1.07 | 0.892 | 89.2 |
| | 9 x10$^{-6}$ | 311.4 | 0.977 | 0.881 | 0.911 | 91.1 |
| | 11 x10$^{-6}$ | 352.4 | 0.959 | 0.779 | 0.921 | 92.1 |
| 2 | 1 x10$^{-6}$ | 90.6 | 2.67 | 3.02 | 0.694 | 69.4 |
| | 3 x10$^{-6}$ | 90.8 | 3.00 | 3.01 | 0.696 | 69.6 |
| | 5 x10$^{-6}$ | 96.6 | 3.20 | 2.83 | 0.714 | 71.4 |
| | 7 x10$^{-6}$ | 112.9 | 3.04 | 2.43 | 0.755 | 75.5 |
| | 9 x10$^{-6}$ | 126.2 | 3.97 | 2.17 | 0.781 | 78.1 |
| | 11 x10$^{-6}$ | 146.9 | 2.53 | 1.86 | 0.812 | 81.2 |
| 3 | 1 x10$^{-6}$ | 54.7 | 3.70 | 5.01 | 0.496 | 49.6 |
| | 3 x10$^{-6}$ | 65.4 | 7.04 | 4.19 | 0.579 | 57.9 |
| | 5x10$^{-6}$ | 86.6 | 2.49 | 3.16 | 0.681 | 68.1 |
| | 7 x10$^{-6}$ | 88.6 | 2.64 | 3.09 | 0.689 | 68.9 |
| | 9 x10$^{-6}$ | 109.5 | 2.50 | 2.50 | 0.748 | 74.8 |
| | 11 x10$^{-6}$ | 110 | 4.86 | 2.49 | 0.749 | 74.9 |

**Weight loss measurements**

The weight loss-time curves of C-steel with the addition of inhibitor (1) in 1M HCl at various concentrations is shown in Fig (10) the other inhibitors aren't shown. The curves of Fig(10a) shows that the weight loss values of C-steel in 1M HCl solution containing investigated inhibitors decrease as the concentration of the inhibitors increases; i.e., the corrosion inhibition strengthens with the inhibitor concentration, this is appear in the Table(4).This result comes from the fact that the adsorption of inhibitor on the C-steel increases with the inhibitor concentration thus the C-steel surface is efficiently separated from the medium by the formation of a film on its surface[26-27].Other figures and tables for inhibitors at different temperatures are not shown.

**Table 4.Variation of inhibition efficiency (%IE) of different compounds with their molar concentrations at 25C$^0$ from weight loss measurements at 120 min immersion in 1M HCl.**

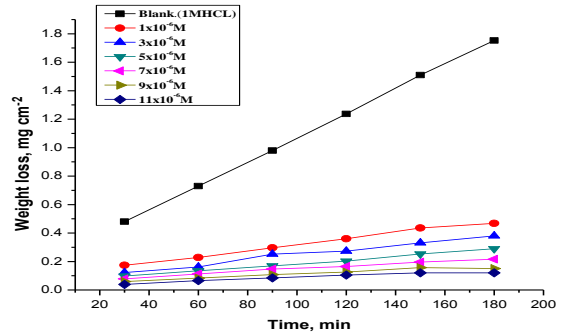| [Inh] M, x 10$^6$ | 1 | | 2 | | 3 | |
|---|---|---|---|---|---|---|
| | %IE | C.R. | %IE | C.R | %IE | C.R. |
| 1 | 70.9 | 0.0030 | 64.6 | 0.0036 | 51.0 | 0.005 |
| 3 | 77.9 | 0.0022 | 71.2 | 0.0029 | 58.9 | 0.0042 |
| 5 | 83.6 | 0.0016 | 75.8 | 0.0024 | 64.9 | 0.0036 |
| 7 | 86.7 | 0.0013 | 80.0 | 0.0020 | 69.0 | 0.0031 |
| 9 | 89.7 | 0.0010 | 83.3 | 0.0017 | 72.2 | 0.0028 |
| 11 | 91.5 | 0.0008 | 86.3 | 0.0014 | 75.9 | 0.0024 |



**Figure 4a. Weight- loss time curves for the dissolution of C-steel in absence and presence of different concentration of inhibitor (1) at 25ºC**
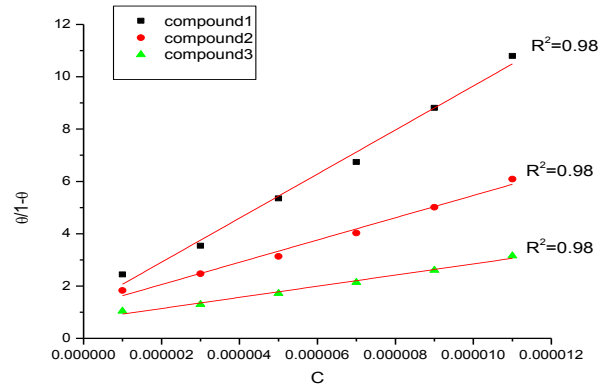


**Figure (4b): Langmuir adsorption isotherm of inhibitors on C-steel surface in 1 M HCl at 25º C**
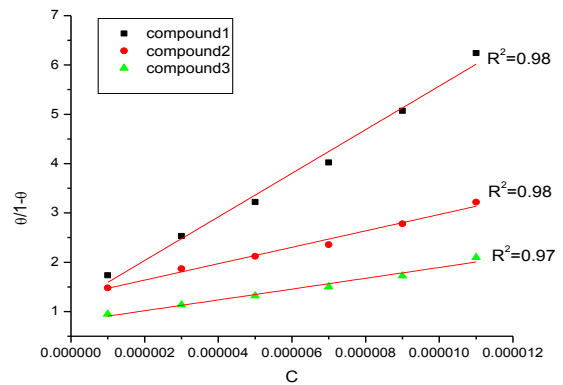


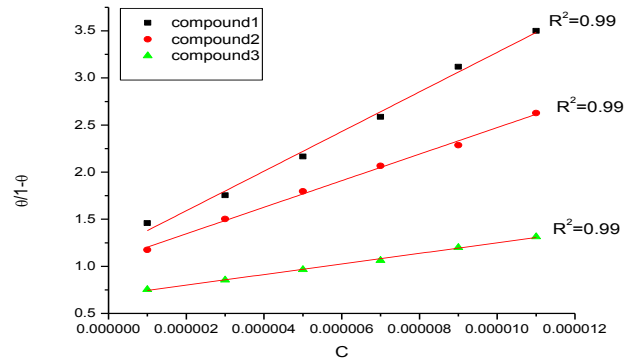**Figure 5b. Langmuir adsorption isotherm of inhibitors on C-steel surface in 1 M HCl at 30º C**



**Figure (5b). Langmuir adsorption isotherm of inhibitors on C-steel surface in 1 M HCl at 35º C**
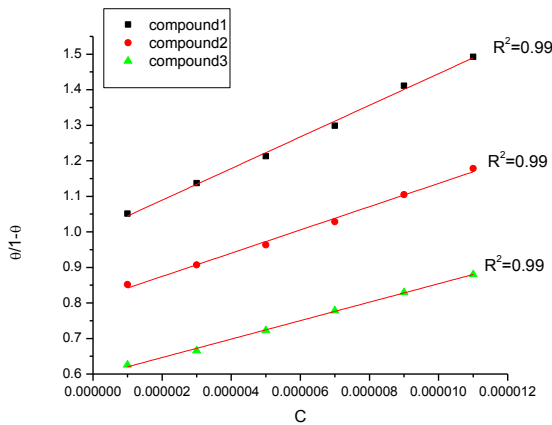
**Figure 6. Langmuir adsorption isotherm of inhibitors on C-steel surface in 1 M HCl at 40º C**
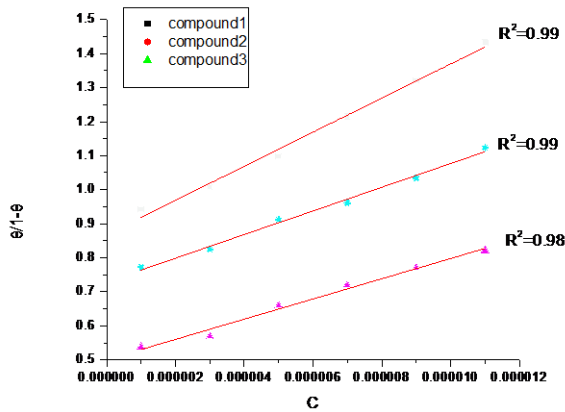
**Table 5.Thermodynamic parameters for adsorption of inhibitors on C-steel surface in 1M HCl at different temperature**

| Compound | Temperature ºC | $K_{ads} \times 10^{-3}$ M$^{-1}$ | $-\Delta G°_{ad}$ kJ mol$^{-1}$ | $\Delta H°_{ads}$ kJ mol$^{-1}$ | $\Delta S°_{ads}$ J mol$^{-1}$ k$^{-1}$ |
|---|---|---|---|---|---|
| 1 | 25 | 0.866 | 9.597 | 11.586 | 71.086 |
| | 30 | 0.869 | 9.766 | | 70.473 |
| | 35 | 0.926 | 10.092 | | 70.386 |
| | 40 | 1.0007 | 10.455 | | 70.422 |
| | 45 | 1.163 | 11.019 | | 71.089 |
| 2 | 25 | 0.873 | 9.615 | 18.581 | 94.619 |
| | 30 | 0.932 | 9.943 | | 94.139 |
| | 35 | 1.069 | 10.459 | | 94.287 |
| | 40 | 1.236 | 11.005 | | 94.524 |
| | 45 | 1.365 | 11.444 | | 94.42 |
| 3 | 25 | 1.228 | 10.462 | 20.067 | 102.447 |
| | 30 | 1.254 | 10.691 | | 101.512 |
| | 35 | 1.453 | 11.242 | | 101.661 |
| | 40 | 1.682 | 11.808 | | 101.836 |
| | 45 | 1.999 | 12.452 | | 102.262 |



**Figure 7. Langmuir adsorption isotherm of inhibitors on C-steel surface in 1 M HCl at 45ºC**



**Figure 7c. Transition plots for C-steel after immersion 120 minutes of immersion in 1M HCl in absence and presence of various concentrations of inhibitor (1)**

**Table 6. Activation parameters for adsorption of inhibitors on C-steel in 1M HCl in absence and presence of various concentrations of inhibitors**

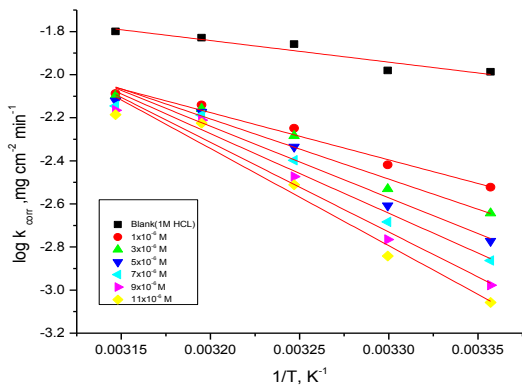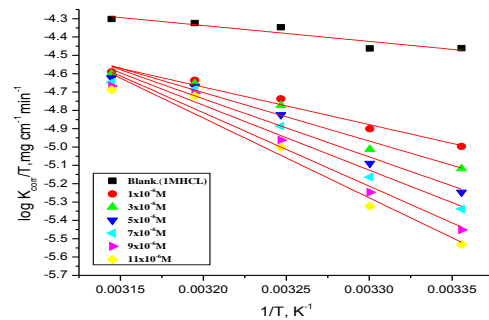| Compound | [inh] M | $E_a^*$ kJ mol$^{-1}$ | $\Delta H^*$ kJ mol$^{-1}$ | $-\Delta S^*$ J mol$^{-1}$k$^{-1}$ |
|---|---|---|---|---|
| Blank | 0 | 19.20 | 7.2 | 227.56 |
| 1 | 1 x10$^{-6}$ | 41.98 | 17.03 | 161.57 |
| | 3 x10$^{-6}$ | 53.46 | 21.99 | 125.56 |
| | 5 x10$^{-6}$ | 63.58 | 26.34 | 94.13 |
| | 7 x10$^{-6}$ | 70.70 | 29.42 | 72.13 |
| | 9 x10$^{-6}$ | 79.22 | 33.31 | 44.22 |
| | 11 x10$^{-6}$ | 86.36 | 36.18 | 23.62 |
| 2 | 1 x10$^{-6}$ | 38.73 | 16.04 | 168.28 |
| | 3 x10$^{-6}$ | 48.87 | 20.11 | 138.65 |
| | 5 x10$^{-6}$ | 51.07 | 21.07 | 131.99 |
| | 7 x10$^{-6}$ | 56.88 | 23.59 | 113.74 |
| | 9 x10$^{-6}$ | 62.65 | 26.10 | 95.74 |
| | 11 x10$^{-6}$ | 68.69 | 28.73 | 76.92 |
| 3 | 1 x10$^{-6}$ | 30.19 | 11.57 | 199.40 |
| | 3 x10$^{-6}$ | 36.76 | 14.87 | 175.32 |
| | 5 x10$^{-6}$ | 42.98 | 17.01 | 159.94 |
| | 7 x10$^{-6}$ | 44.33 | 17.86 | 154.20 |
| | 9 x10$^{-6}$ | 47.91 | 19.38 | 143.41 |
| | 11 x10$^{-6}$ | 52.46 | 21.44 | 128.55 |



**Figure 7b. Arrhenius plots for C-steel corrosion rates after 120 minutes of immersion in 1M HCl in the absence and presence of different concentrations of inhibitor (1)**

**SEM/EDX examination**



(a)



(b)



(c)

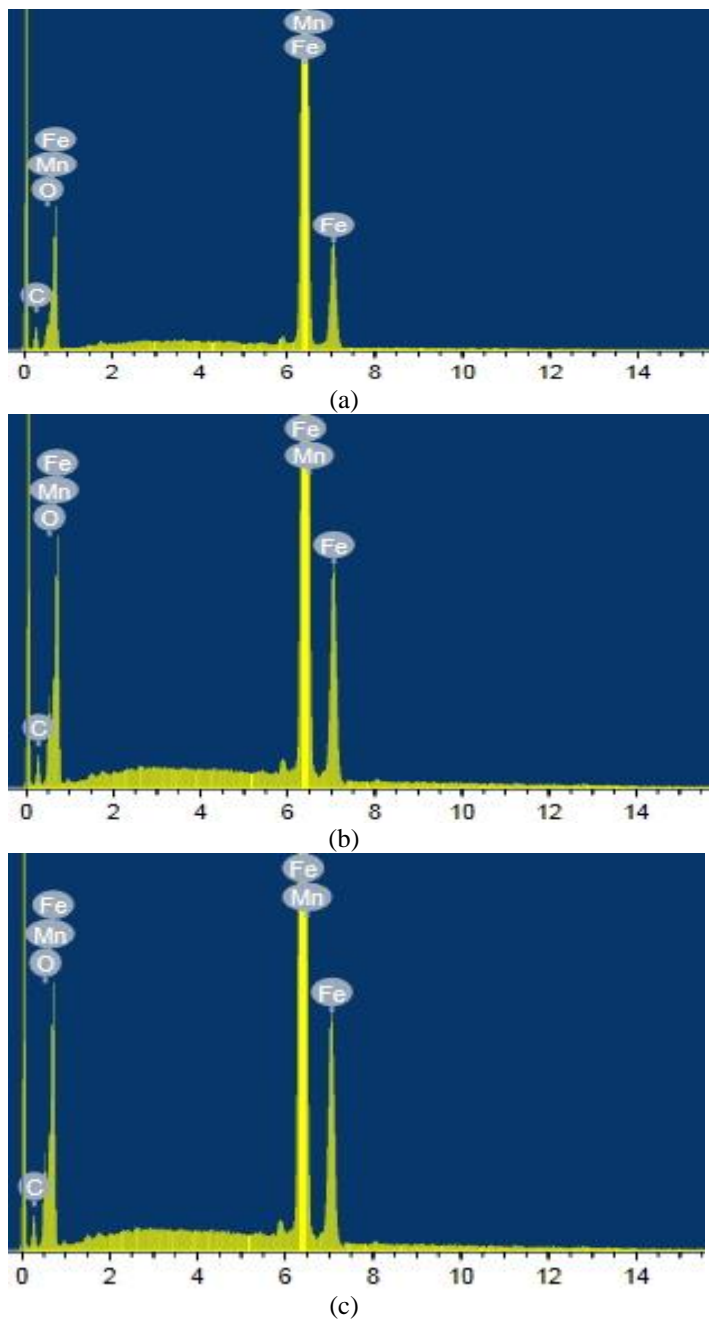**Fig8. EDS spectra of C-steel in 1M HCl in presence of (18x10$^{-6}$M ) of inhibitor(1)(a), (18x10$^{-6}$M) OF inhibitor(2)(b),(18x10$^{-6}$M) inhibitor(3)(c).**
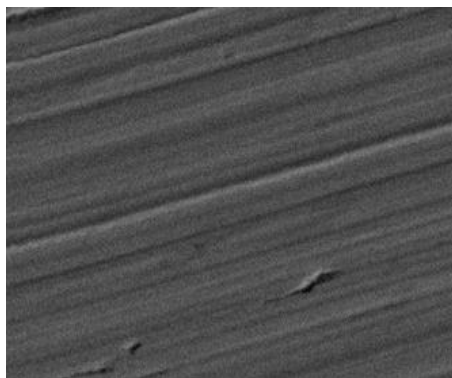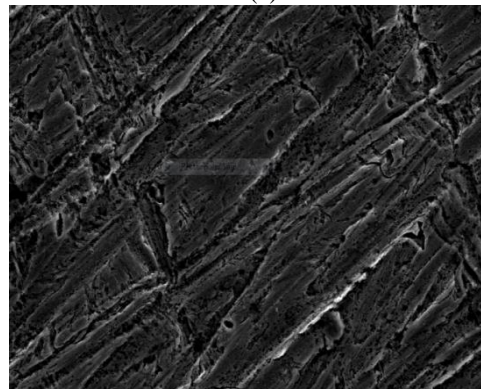


Blank(a)
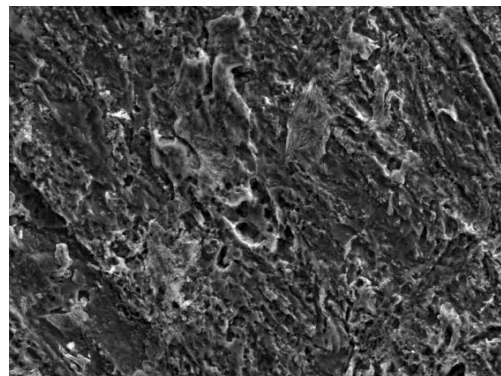


(b)
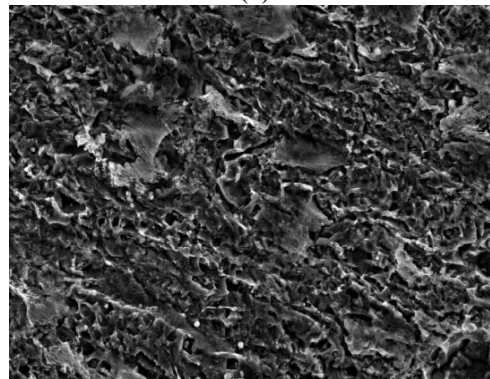


(c)



(d)

**Fig9. SEM micrographs of C-steel surface (a) before immersion in 1M HCl ,(b) after 12h of immersion for inhibitor(1), (c) after 12h of immersion for inhibitor (2) ,(d) after 12h of immersion for inhibitor(3).**

**Conclusions**

From the overall experimental results the following conclusions can be deduced:

1-The investigated compounds are good inhibitors and act as physical type inhibitors for C-steel corrosion in 1M HCl solutions.

2-Reasonably good agreement was obtained between the values observed by the weight loss and electrochemical measurements were in good agreement .The order of %IE of these investigated compounds is in the following order: 1>2>3

3-The results obtained from all electrochemical measurements showed that the inhibiting action increases with inhibitor concentration increases and also decrease with increasing temperature.

4-Thermodynamic parameters showed that the inhibition of corrosion by studied compounds is due to the formation of physical adsorbed film on the metal surface.

5-The adsorption of inhibitor on C-steel surface in 1M HCl solution follows Langmuir isotherm for these compounds.

6-The negative values of ΔG0 ads indicate spontaneous adsorption of the inhibitors on the surface of C-steel.

7-The values of inhibition efficiencies observed from the different independent quantitative techniques used reveal the validity of the results.

**References**

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