

Corrosion Inhibition of Carbon Steel in HCl by Cyanoacetate and Oxobutanoate Derivatives

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Abstract- The corrosion behavior of carbon steel in HCl solution containing various concentrations of cyanoacetate and oxobutanoate derivatives was investigated. The corrosion inhibitive action of these compounds on carbon steel was studied using chemical and electrochemical methods. The results showed that the inhibition efficiency increases by increasing the concentration and decreases by increasing the temperature. The synergistic effect of potassium iodide (KI) in presence of various concentrations of these compounds was also investigated. Polarization curves revealed that these compounds are mixed type inhibitors. The adsorption of these compounds follows the kinetic thermodynamic model and Temkin adsorption isotherm. Some thermodynamic parameters of activation and adsorption processes were also determined and discussed.

Keywords- Carbon Steel; Corrosion Inhibition; HCl; Cyanoacetate and Oxobutanoate Derivatives

I. INTRODUCTION

The corrosion of carbon steel is the most common form of corrosion, especially in acid solution. It has practically importance, for example in the acid pickling of iron and steel, chemical cleaning and processing, ore production, oil recovery and petrochemical industry, and other electrochemical systems. Hydrochloric acid is also an important mineral acid with the corrosion inhibitors [1-5]. Corrosion inhibitors play a very important role in protecting many metals and alloys. This leads the researchers to study the use of organic compounds as corrosion inhibitors. Adsorption behavior of organic molecules on the surface of metals depend on their molecular structures of the organic compounds, surface charge density and zero charge potential of the metals [6-19]. Most of the well-known organic compounds containing nitrogen, sulphur, oxygen with aromatic and heterocyclic rings through which they are adsorbed on the metal surface have been reported to be effective inhibitors for the corrosion of steel in acid media by several authors [20-45].

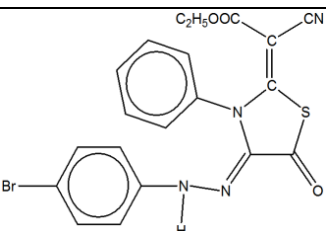
The present investigations aim to study the effect of cyanoacetate and oxobutanoate derivatives as corrosion inhibitors on the corrosion of carbon steel in 2 M HCl using two methods (weight loss and polarization measurements). The synergistic effect of KI was also studied. Effect of temperature was also studied and some thermodynamic parameters were computed and discussed.

II. EXPERIMENTAL

A. Cell and Materials

Measurement was conducted at room temperature. The working electrode was machined to have a fixed exposed surface area of 1 cm² from C-steel sample with composition in weight % of: 0.200 C, 0.350 Mn, 0.024 P, 0.003 Si and the balance Fe. The test electrode was abraded with different grades emery papers, degreased with acetone, washed by bi-distilled water and dried in air. Before polarization experiments, (open circuit potential) OCP of the working electrode was measured vs. time till reach a quasi-stationary value. The molecular structure formulae and molecular weights of the investigated compounds are given in Table I.

TABLE I CHEMICAL STRUCTURE OF THE INVESTIGATED ORGANIC COMPOUNDS

Comp.	Structure	Name	Molecular weight and, Mol. Formula
(A)		(2E)-ethyl 2-((Z)-4-(2-(4-bromophenyl)hydrazono)-5-oxo-3-phenylthiazolidin-2-ylidene)-2-cyanoacetate.	C ₂₀ H ₁₅ BrN ₄ O ₃ S 471.33

(B)		(2E)-ethyl 2-((Z)-4-(2-(4-carboxyphenyl)hydrazono)-5-oxo-3-phenylthiazolidin-2-ylidene)-2-cyanoacetate.	C ₂₁ H ₁₆ N ₄ O ₅ S 436.44
(1)		(2E)-ethyl 2-((Z)-4-(2-(o-tolylhydrazono)-5-oxo-3-phenylthiazolidin-2-ylidene)-3-oxobutanoate	C ₂₂ H ₂₁ N ₃ O ₄ S 423.48
(2)		(2E)-ethyl 2-((Z)-4-(2-(m-tolylhydrazono)-5-oxo-3-phenylthiazolidin-2-ylidene)-3-oxobutanoate	C ₂₂ H ₂₁ N ₃ O ₄ S 423.48

B. Solutions

1) Hydrochloric Acid:

The corrosive medium (2 M HCl) was prepared from a stock 8 M HCl solution by dilution with bi-distilled water from the concentrated acid solution (37 %, Merck). The concentration was checked by standard solution of Na₂CO₃. 2 M HCl solutions were prepared by dilution from 8 M acid with bi-distilled water. This solution was used as a blank.

2) Inhibitors:

An appropriate weighted amount from each compound was dissolved in alcohol to prepare 0.001 M stock solution. Different concentrations of (1x10⁻⁴ - 5x10⁻⁷ M) inhibitors were prepared by dilution from the stock solution using bi-distilled water.

C. Weight Loss Measurements

Carbon steel specimens sized 2 x 2 x 0.05 cm were abraded with different grades of emery papers, washed by bidistilled water and degreased with acetone before immersion in the corrosive solution. The samples were allowed to stand for 3 hours in blank solution in absence and presence of different concentrations of inhibitors and KI. The weight loss of carbon steel specimens in 2 M HCl with and without addition of inhibitors was used to calculate the inhibition efficiency In (%) by using Eq. (1), and all the tests were repeated three times. All tests were performed at different temperature from 30 to 50 °C.

$$\% \text{ In} = [W^{\circ} / (W - W^{\circ})] \times 100 \quad (1)$$

where W° and W are the weight losses of carbon steel specimens in HCl without and with addition of inhibitors and KI, respectively.

D. Potentiodynamic Polarization Measurements

Potentiodynamic measurements were conducted in a conventional three-electrode glass cell of capacity 100 ml. Three different types of electrodes were used; saturated calomel electrode (SCE) and a platinum foil were used as reference and auxiliary electrodes, respectively. The working electrode was C- steel electrode, which cut from C- steel sheets with thickness 0.2 cm. The electrode was of dimensions 1 cm x 1 cm and was weld from one side to a copper wire used for electric connection. The sample was embedded in a glass tube using epoxy resin. The electrode was prepared before immersion in the test solution as in case of weight loss method. A constant quantity of the test solution (100 ml) was taken in the polarization cell. A time interval of about 30 minutes was given for the system to attain a steady state. All the experiments were carried out at 30 ± 1 °C by using an ultra circulating thermostat. The potentiodynamic current potential curves were recorded by changing the electrode potential automatically from - 500 to + 800 mV versus open circuit potential (E_{ocp}) with a scan rate of 0.5 mV s⁻¹ using an electrochemical measurement system Potentiostat / Galvanostat / ZRA (Gamry PCI 300/4). This includes Gamry Framework system based on the ESA400, and a personal computer with DC 105 software for dc corrosion measurements.

Echem Analyst software 5.1 was used for plotting, graphing and fitting data.

The inhibitors were of mixed-type. Corrosion inhibition efficiency (% In) was calculated using equation (2):

$$\% \text{ In} = [1 - (i_{\text{corr}} / i_{\text{corr}}^{\circ})] \times 100 \quad (2)$$

where (i_{corr} and i_{corr}°) are the corrosion current densities in the absence and presence of inhibitors respectively.

III. RESULTS AND DISCUSSION

A. Weight Loss Measurements

Fig. 1&2 show the effect of increasing the concentration of investigated compounds 1&2, A&B respectively, on the weight loss of carbon steel in 2 M HCl solutions. It is obvious that the weight loss of the inhibitor containing solutions varied with immersion period, which is lower than that obtained in the blank solution. The relatively large decrease in the weight loss in presence of different concentrations of additives indicates that the additives under study have inhibition effect on the corrosion of carbon steel in 2 M HCl solution. The percentage inhibition efficiency was calculated from the weight loss measurements and was listed in Table II. The results showed that inhibition efficiency increased as the concentration of inhibitors rose. Moreover, the percentage inhibition efficiency is found to decrease in the following order: $A > B$ & $2 > 1$.

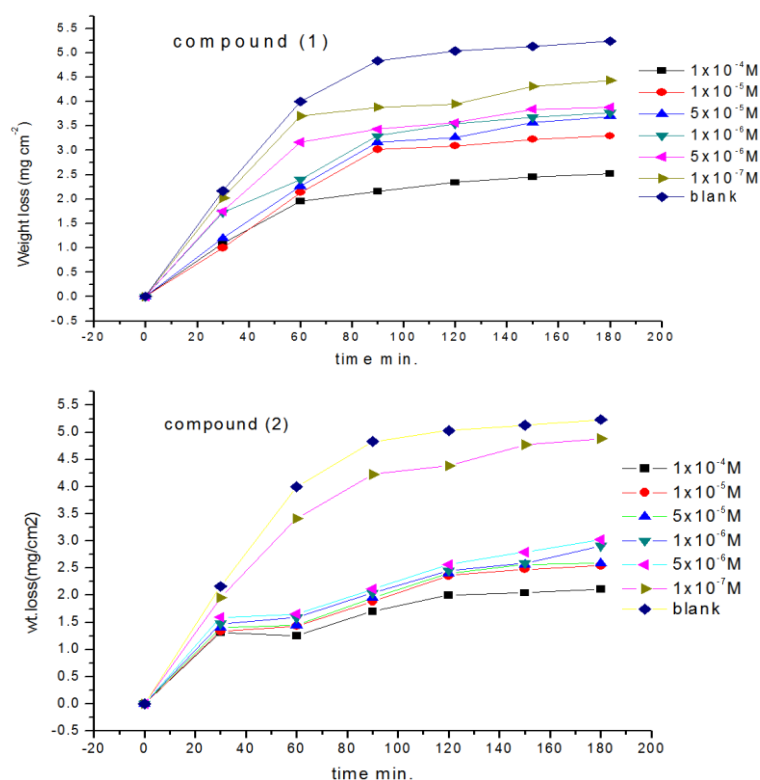
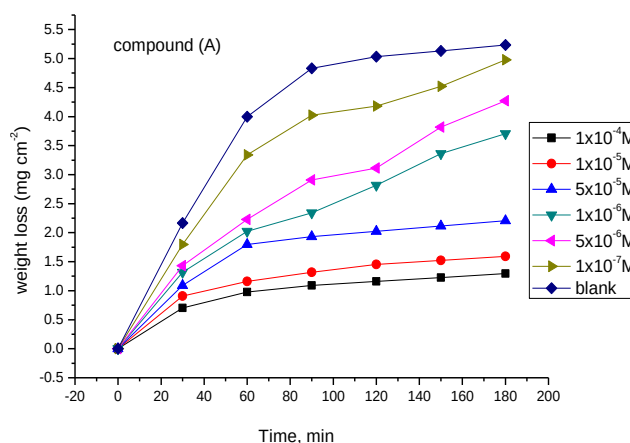


Fig. 1 Weight loss a-time curves of carbon steel in 2 M HCl solution without and with different concentrations of compounds (1) & (2).



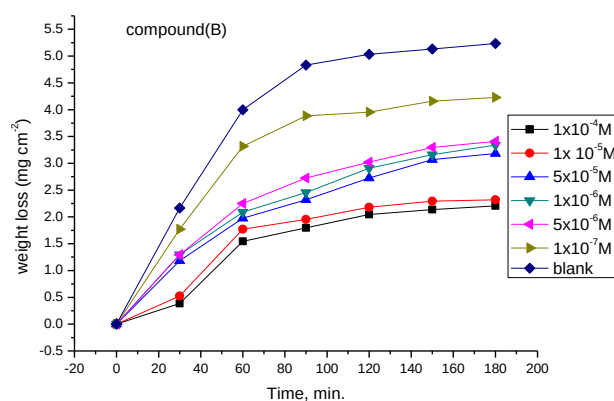


Fig. 2 Weight loss a-time curves of carbon steel in 2 N HCl solution without and with different concentrations of compounds (A) & (B).

TABLE II DATA FROM WEIGHT LOSS OF CARBON STEEL IN 2 M HCL FOR VARIOUS CONCENTRATIONS OF ALL COMPOUNDS AFTER 90 MIN IMMERSION

concentration M	% Inhibition			
	(A)	(B)	(1)	(2)
5×10^{-7}	16.8	2.8	19.6	12.5
1×10^{-6}	39.8	31.8	29.0	56.3
5×10^{-6}	51.6	38.6	31.8	57.7
1×10^{-5}	60.0	42.1	34.5	59.6
5×10^{-5}	72.7	51.1	37.5	61.0
1×10^{-4}	77.4	55.1	55.3	64.7

1) Synergistic Effect:

The results indicate that there is a synergistic effect between KI and the inhibitor for inhibition efficiency and this is illustrated in Table III and Fig. 3.

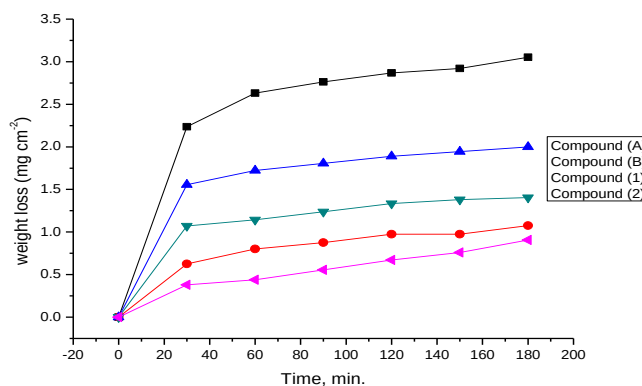


Fig. 3 Weight loss- time curves of carbon steel in 2 M HCl solution without and with 1×10^{-5} M different compounds + 1×10^{-2} M KI.

TABLE III DATA FROM WEIGHT LOSS OF CARBON STEEL IN 2 N HCL IN THE PRESENCE OF 10^{-2} M KI + 1×10^{-5} M OF INVESTIGATED COMPOUNDS AFTER 90 MIN IMMERSION

compound	Weight loss (mg cm^{-2})	% In
(A)	0.875	68.33
(B)	1.556	43.7
(1)	1.238	55.19
(2)	0.439	84.13

2) Effect of Temperature:

Fig. 4&5 represents the relationship between $\log k$ (rate constant of corrosion) vs. $1/T$ (T = absolute temperature) for 1×10^{-5} M inhibitors (1, 2) and (A, B) respectively, after 90 min in 2 M HCl solution using weight loss measurements. These relations gave straight lines. The results show that the rate of corrosion increases as the temperature increases, i.e. the inhibition efficiency of the additives decreases with rise in temperature. The values of the rate constants obtained at different temperatures permit the calculation of the Arrhenius, enthalpy, ΔH^* , entropy, ΔS^* , and free energy of activation, ΔG^* , of

carbon steel in 2 M HCl (Table IV). These thermodynamic functions were calculated from the following equations [26]:

1- ΔE^* from:

$$\log k = -\Delta E^*/2.303RT = \text{constant} \quad (3)$$

2- ΔH^* from:

$$\Delta H^* = \Delta E^* - RT \quad (4)$$

3- ΔG^* and ΔS^* using the transition state theory:

$$\Delta G^* = RT (\ln k' T/h - \ln k) \quad (5)$$

$$\Delta S^* = \Delta H^* - \Delta G^*/T \quad (6)$$

where R = gas constant, k = the rate constant , k' = Boltzmann constant, and h = Planck's constant

These results reveal that the energy and free energy of activation increase with increase in the concentration of the additive which retards the corrosion reaction. Also the results show that the efficiency of different inhibitors at all temperature studied (30-50 °C) was found decreasing in the order: 2 > 1 & A > B

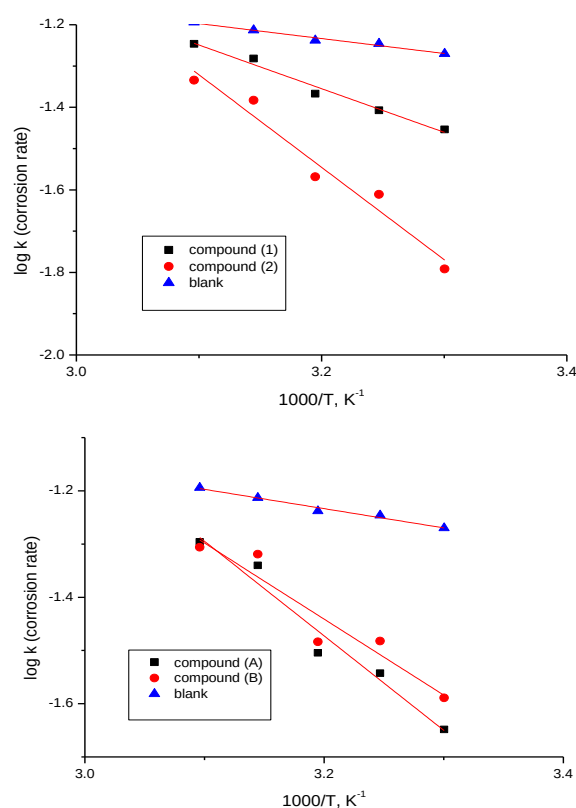


Fig. 4, 5 Log k vs $1/T$ curves for carbon steel in 2 M HCl solution in the presence and absence of different inhibitors using weight loss measurements

TABLE IV ENERGY (E^*), ENTHALPY (ΔH^*), ENTROPY (ΔS^*), AND FREE ENERGY (ΔG^*) OF ACTIVATION FOR CARBON STEEL IN 2 M HCL IN PRESENCE OF 1×10^{-5} M OF DIFFERENT INHIBITORS AT DIFFERENT TEMPERATURES

Temperature, °C		30	35	40	45	50
2 M HCl	E^* in kcal mol ⁻¹	6.9				
	ΔH^* in kcal mol ⁻¹	4.39	4.34	4.30	4.26	4.22
	$-\Delta G^*$ in kcal mol ⁻¹	45.92	46.78	47.54	48.41	49.25
	$-\Delta S^*$ in kcal mol ⁻¹	137.08	137.78	138.15	138.85	139.42
Inhibitor (1)	E^* in kcal mol ⁻¹	20.19				
	ΔH^* in kcal mol ⁻¹	17.67	17.63	17.59	17.55	17.50
	$-\Delta G^*$ in kcal mol ⁻¹	44.86	45.83	46.77	48.93	48.93
	$-\Delta S^*$ in kcal mol ⁻¹	206.37	206.03	205.62	138.9	205.68
Inhibitor (2)	E^* in kcal mol ⁻¹	41.45				
	ΔH^* in kcal mol ⁻¹	38.93	38.89	38.85	38.81	38.77
	$-\Delta G^*$ in kcal mol ⁻¹	42.90	44.63	45.57	47.38	48.38
	$-\Delta S^*$ in kcal mol ⁻¹	270.08	271.18	269.71	271.04	269.82
Inhibitor (A)	E^* in kcal mol ⁻¹	33.93				
	ΔH^* in kcal mol ⁻¹	31.41	31.37	31.33	31.29	31.25

	$-\Delta G^{\circ}$ in kcal mol ⁻¹	43.73	45.03	45.95	47.64	48.62
	$-\Delta S^{\circ}$ in kcal mol ⁻¹	40.65	44.35	46.71	51.42	53.79
Inhibitor (B)	E° in kcal mol ⁻¹	27.28				
	ΔH° in kcal mol ⁻¹	24.76	24.72	24.68	24.64	24.60
	$-\Delta G^{\circ}$ in kcal mol ⁻¹	44.07	45.39	46.07	47.77	48.56
	$-\Delta S^{\circ}$ in kcal mol ⁻¹	63.74	67.1	68.36	72.74	74.19

3) Adsorption Isotherm:

To understand the mechanism of corrosion inhibition, the adsorption behavior of the inhibitors adsorbents on the metal surface must be known. There are a number of mathematical expressions having thus developed to take into consideration of non-ideal effects. The most used isotherms are, Frumkin, De Boer, Parsons, Temkin, Flory-Huggins and Bockris-Swinkless [27-31]. The values of surface coverage, θ , corresponding to different concentrations of inhibitors at 30 °C have been used to explain the best isotherm to determine adsorption isotherm process. Fig. 6 showed the plotted relation between θ against $\log C$ (Temkin isotherm) which has the following Equation:

$$-2a\theta = \ln K_{ads} + \ln C \quad (7)$$

where C is the inhibitor bulk concentration in mol L⁻¹, K_{ads} (M⁻¹) is the equilibrium constant of adsorption, a is lateral interaction parameter which describe the molecular interactions in the adsorbed layer.

The equilibrium constant (K_{ads}) in M⁻¹ is related to the standard free energy of adsorption (ΔG°_{ads}) by [32]:

$$K_{ads} = 1/55.5 \exp(\Delta G^{\circ}_{ads}/RT) \quad (8)$$

where R is the universal gas constant, 55.5 is the concentration of water in the bulk of solution in mol L⁻¹ and T is the absolute temperature.

K_{ads} can be calculated from the intercept of lines in Fig.6 and ΔG°_{ads} . Can be calculated using equation (7). The results are shown in Table (V). A Plot of $\log \theta/1-\theta$ against $\log C$ (Fig.7) at different concentrations of all compounds, straight lines were obtained indicating that adsorption follows kinetic thermodynamic model according Equation (9) [33]:

$$\log \theta/1-\theta = \log (K') + y \log C \quad (9)$$

The equilibrium constant of adsorption is $K_{ads} = K'^{(1/y)}$, where $1/y$ is the number of surface active sites occupied by one inhibitor molecules and C is the bulk concentration of the inhibitor. Also we can calculate The results are shown in Table (V).

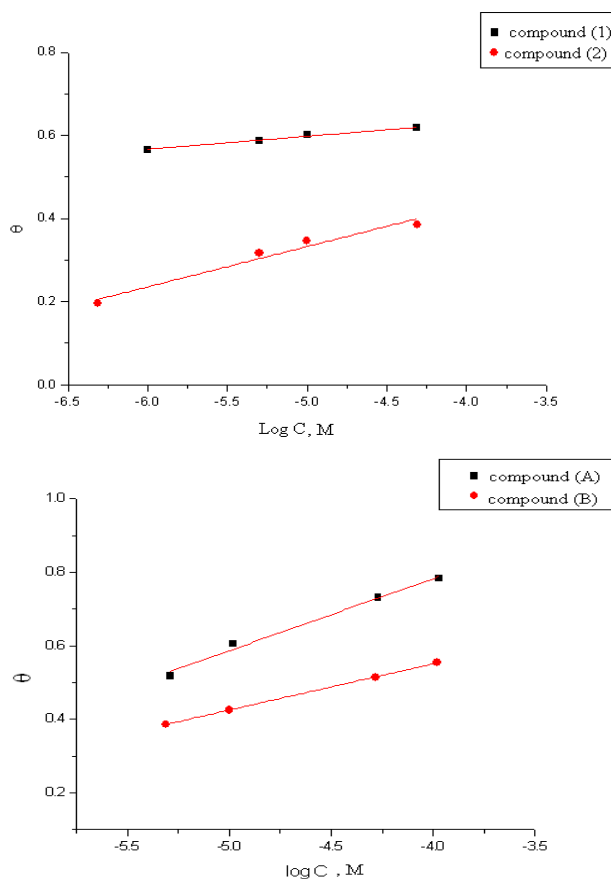


Fig. 6 The Temkin adsorption isotherms for the inhibitors, determined in a 2 M HCl solution at 30

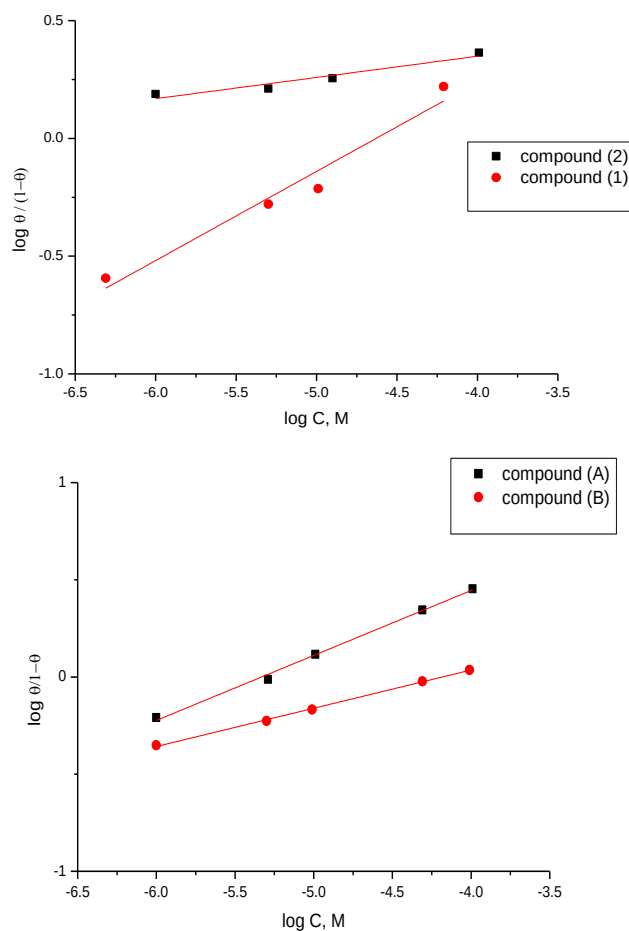


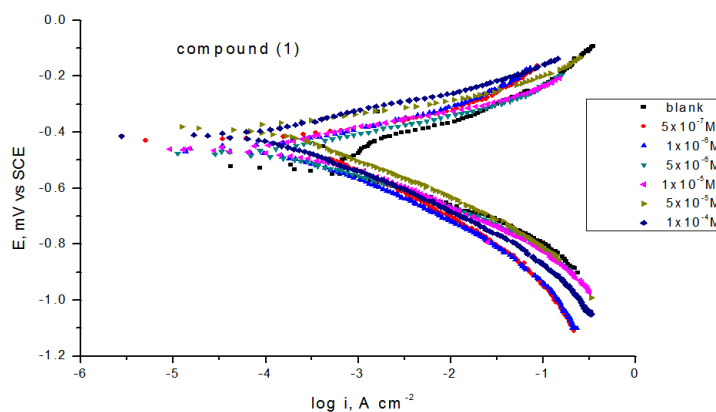
Fig. 7 The kinetic thermodynamic isotherms for the inhibitors, determined on carbon steel in a 2M HCl solution at 30.

TABLE V INHIBITOR EQUILIBRIUM CONSTANT (K_{ads}), FREE ENERGY (ΔG°_{ads}), LATERAL INTERACTION PARAMETER (A) AND NUMBER OF ACTIVE SITES AT $1 \times 10^{-5}M$ FOR ALL INHIBITORS FOR CORROSION OF CARBON STEEL IN A 2 M HCL SOLUTION AT 30 °C

Inhibitor	Temkin isotherm			Kinetic model		
	K_{ads}	a	ΔG°_{ads}	K_{ads}	1/y	ΔG°_{ads}
(A)	3.0×10^3	12.83	20.195	237.06	6.93	23.85
(B)	5.9×10^3	21.49	21.887	81.30	11.92	21.16
(1)	5.7×10^3	27.63	21.788	53.98	9.56	20.13
(2)	4.9×10^3	16.22	31.56	3487.23	49.31	30.61

B. Potentiodynamic Polarization Measurements

Fig. 8 represents the potentiodynamic polarization curves for carbon steel in 2 M HCl solution in the absence and presence all studies compounds in 1 M HCl solution. The electrochemical parameters and inhibition efficiencies (In %) of all studied compounds are given in the Table (VI).



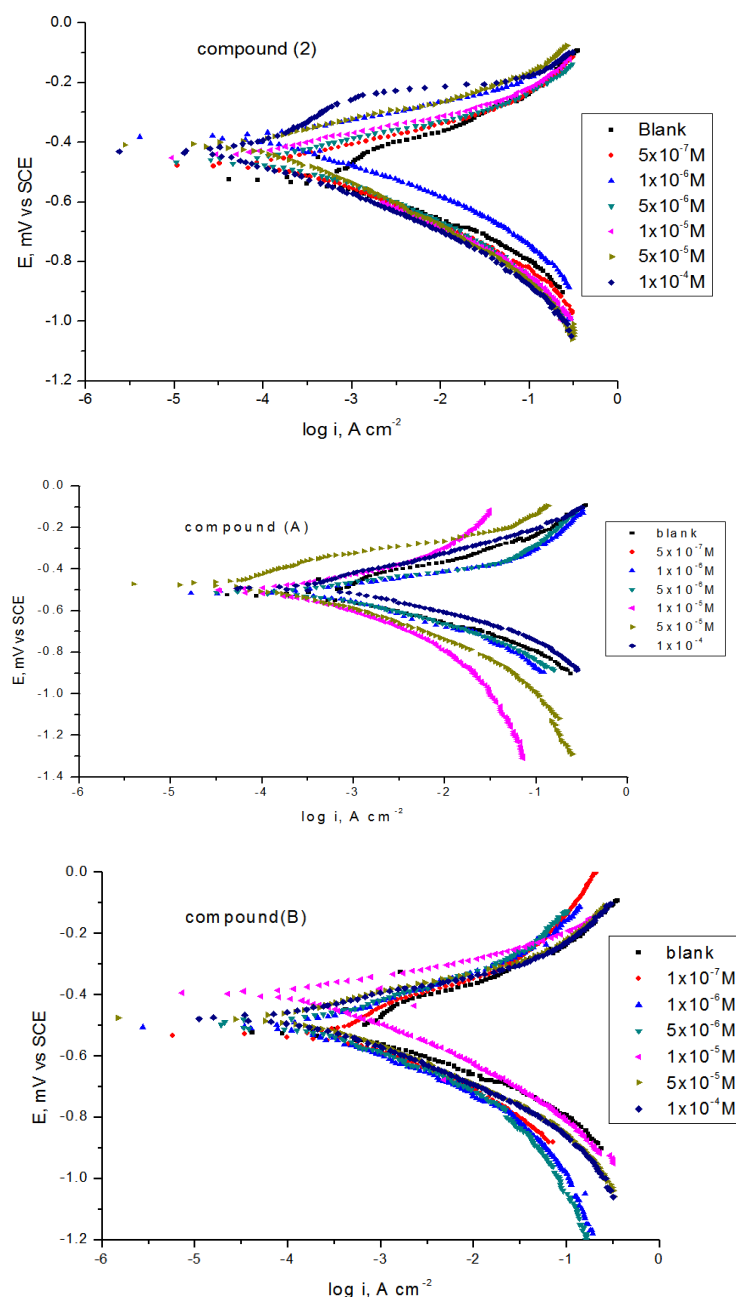


Fig. 8 Polarization curves for carbon steel in 2M HCl as a blank solution and in the presence of inhibitors.

TABLE VI ELECTROCHEMICAL PARAMETERS OF CARBON STEEL IN 2 M HCL SOLUTION CONTAINING DIFFERENT CONCENTRATION OF INHIBITORS AT 30 °C

Conc., M	β_a mVdec ⁻¹	β_c mVdec ⁻¹	i_{corr} mA cm ⁻²	E_{corr} mV vs.SCE	R_p Ω Cm ²	% In
Blank	252.8	375.7	0.808	544.4	7.07x10	-----
Compound(1)						
5x10 ⁻⁷	134.4	276.6	0.560	430.4	7.070x10	31.286
1x10 ⁻⁶	130.7	249.1	0.320	460.2	1.160x10 ²	60.505
5x10 ⁻⁶	109.6	163.3	0.310	467.4	9.280x10	61.990
1x10 ⁻⁵	97.4	162.3	0.220	460.4	1.178x10 ²	72.212
5x10 ⁻⁵	85.4	186.1	0.215	383.5	1.180x10 ²	73.381
1x10 ⁻⁴	99.1	186.9	0.156	409.1	1.804x10 ²	80.698
compound(2)						
5x10 ⁻⁷	109.6	163.4	0.307	430.9	9.280x10	61.990
1x10 ⁻⁶	89	164.7	0.250	409.1	9.940x10	69.048
5x10 ⁻⁶	98.6	162.5	0.228	451.9	1.170x10 ²	71.834
1x10 ⁻⁵	93.3	160.9	0.144	463.1	1.770x10 ²	82.135
5x10 ⁻⁵	99.1	186.9	0.116	382.5	1.80x10 ²	85.651
1x10 ⁻⁴	199.6	166.1	0.118	476.4	3.340x10 ²	85.415

Compound(B)						
5×10^{-7}	180.1	155.1	0.807	525.1	4.480×10	0.0867
1×10^{-6}	150.7	170.4	0.764	509	4.540×10	5.448
5×10^{-6}	156.3	158.2	0.761	510	4.490×10	5.844
1×10^{-5}	211.3	351.5	0.607	500	9.433×10	24.848
5×10^{-5}	281.7	283.9	0.605	467.8	2.030×10^2	24.861
1×10^{-4}	147.1	136.3	0.601	487.7	5.120×10^2	25.653
compound(A)						
5×10^{-7}	250.0	167.3	0.689	534	6.319×10	14.696
1×10^{-6}	147.8	270.3	0.456	505.8	8.927×10	42.441
5×10^{-6}	140.8	264.7	0.375	492.3	1.063×10^2	53.522
1×10^{-5}	91.6	178.3	0.238	394.5	1.102×10^2	70.534
5×10^{-5}	111.4	178.2	0.224	477.1	1.330×10^2	72.316
1×10^{-4}	109.3	177.1	0.220	477.2	1.336×10^2	72.812

1) Synergistic Effect of KI:

The polarization curves obtained for 1×10^{-5} M of inhibitors in the presence of 1×10^{-2} M KI added to 2 M HCl are presented in Fig. 9. The synergistic effect of KI in hindering the corrosion is apparent in obtained results (Table VII). The presence of iodide ions shifts E_{corr} more in the anodic direction and further decreases the anodic and cathodic reaction kinetics.

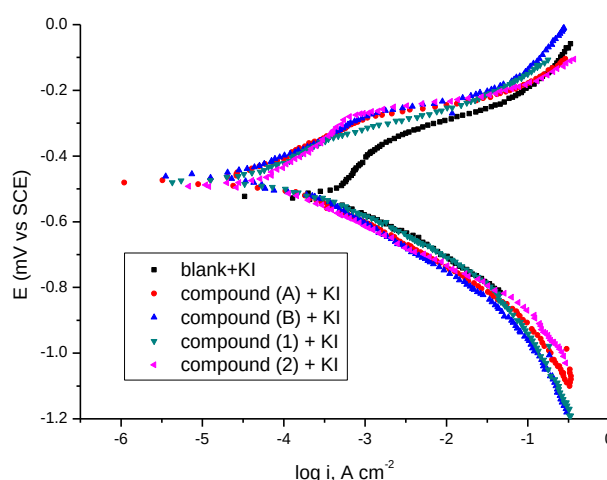


Fig. 9 Potentiodynamic polarization curves for carbon steel in 2 M HCl + 1×10^{-2} M KI in the absence and in presence of 1×10^{-5} M of inhibitor

TABLE VII DATA FROM POTENTIODYNAMIC POLARIZATION OF CARBON STEEL IN 2 M HCL + 1×10^{-5} M INHIBITORS + 1×10^{-2} M

	Conc., M	β_a mv dec ⁻¹	β_c mv dec ⁻¹	$i_{\text{corr.}}$ $\mu\text{A cm}^{-2}$	$E_{\text{corr.}}$ mV vs.SCE	R_p ohm cm ²	% In
blank + 10^{-2} M KI		146	129	234.8	518	1.26×10^2	70.93
Blank + 10^{-2} M KI + 1×10^{-5} M inhibitor	(1)	114	174	41.7	477	7.16×10^2	82.24
	(2)	75	137	9.1	451	2.31×10^3	96.12
	(B)	117	188	60.6	457	5.15×10^2	74.20
	(A)	91	142	16.8	481	1.43×10^3	92.87

2) Effect of Chemical Structure on the Inhibition Efficiency:

The effect of the molecular structure of the investigated compounds on their protective properties will be considered in the following aspects: (i) effect of electronic structure, which determines the electronic density of the molecule, reaction center; (ii) effect of the chemical structure, which includes structural like volume, surface area; (iii) mode of adsorption and formation of metallic complexes [34].

The observed corrosion data in the presence of the inhibitors showed the following: (i) the decrease of the corrosion rate with the increase in the concentration of the inhibitor; (ii) the decrease in the inhibition efficiency with increasing temperature, indicating that the corrosion inhibition occurs by adsorption of the inhibitor molecules on the electrode surface [35]. The nature of the inhibitor interaction on the metal surface during corrosion inhibition has been known from its adsorption characteristics [36].

The obtained results of the first series (Compounds A and B) indicate that Compound (B) has lower inhibition efficiency than Compound (A) in spite of its higher molecular size. This is because of Br atom more basic than $-\text{COOH}$ group and it had a dual effect (behave as donor or acceptor atom) so Br atom is more strongly adsorbed on the metal surface than $-\text{COOH}$ group.

In the second group the two compounds have the same molecular size but Compound (1) gives lower inhibition efficiency

than Compound(2) because the presence of methyl group in Compound(1) in the ortho position may cause steric hindrance for the molecule to adsorb on the metal surface.

IV. CONCLUSIONS

1. All investigated inhibitors used in this study show inhibiting properties for the corrosion of carbon steel in 2 M HCl.
2. The structure of the inhibitor used in this study influences their inhibition efficiency.
3. The inhibitive effect of inhibitors was demonstrated by weight loss method and potentio dynamic polarization method.
4. The adsorption of the inhibitors on the carbon steel surface obeys Temkin's adsorption isotherm.
5. The inhibition efficiency increases with increasing of the compounds concentration.
6. KI synergistically increased the efficiency of inhibitor and this was illustrated in two techniques which we used.

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