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Corrosion inhibition of Aluminum in 1 M phosphoric acid solutions using some Chalcones derivatives and synergistic action with halide ions

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The purpose of this paper is to investigate the inhibition effect of some Chalcone derivatives on the corrosion behavior of aluminum in 1 M phosphoric acid solutions with and without Chalcone derivatives and to investigate the influence of halide additives namely: KCl, KBr and KI on the inhibition efficiency of aluminum. For this purpose, polarization (DC) and weight loss measurements were used. To increase the inhibition efficiency, halide anions were added to the corrosive medium. The effect of temperature also was studied over the range 30 to 50°C. Thermodynamic parameters (ΔH*, ΔS*) were calculated and discussed. The results indicated that the corrosion of aluminum was affected by the addition of Chalcone derivatives in 1 M H_3PO_4. The efficiency of Chalcone derivatives increases with the increase in the derivatives concentration but decreases with a rise in temperature. The effect of addition of halide ions to the corrosive solution increases the inhibition efficiency by more than 30%. These compounds were adsorbed on aluminum surface obeying Freundlich adsorption isotherm and they behave as mixed type inhibitors. The main objectives of this study were to gain some insight into the protection of aluminum in 1 M H_3PO_4 medium using some Chalcone derivatives.

Key words: Corrosion inhibition, aluminum, synergistic effect, H_3PO_4, Chalcone derivatives.

INTRODUCTION

Aluminum has a remarkable economic and industrial importance owing to its low cost, light weight, high thermal and electrical conductivity. The most important feature in aluminum is its corrosion resistance to the atmosphere and too many aqueous media due to the presence of a rapidly formed thin and highly protective barrier oxide film that is bonded strongly to its surface, which separates the bare metal from the corrosive environment. It is commonly stated that the oxide film is stable over a pH range of about 4.5 to 8.5 (Mazhar et al., 1986). Organic compounds containing polar functions with N, S, or O in conjugated system have been reported as effective inhibitors of metal corrosion (Obot et al., 2009; Obot et al., 2008a,b). The nature of inhibitor adsorption depends on the number and types of adsorption sites on the metal surface as well as the type of interaction between the organic molecule and the metallic surface (Tang et al., 2006).

A number of studies have recently appeared in the literature (Ebenso et al., 2001; Fouda et al., 1986a; El-Dahan et al., 2005; Okafor et al., 2004; Khalil et al., 2003; Oguzie et al., 2004; Ambat et al., 2006; Ashassi-Sorkhabi et al., 2005; Rajan et al 2006) on the topic of the corrosion of aluminum in acidic solutions.

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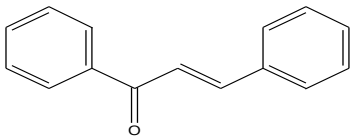
Chalcones were used as inhibitors for metals and alloys (Fouda et al., 2002, 2013; Ayyannan et al., 2013; Bouklah et al., 2006; Selim et al., 1996; Ganesan et al 2013).

The present study aimed to investigate the inhibition efficiency of the investigated Chalcone derivatives as corrosion inhibitors for aluminum in phosphoric acid solution by weight loss and galvanostatic polarization methods, the effect of addition of different anions like I^-, Br^- and Cl^- on the corrosion inhibition was studied. Also, the effect of temperature on the rate of corrosion was studied in order to calculate some thermodynamic parameters related to the corrosion process.

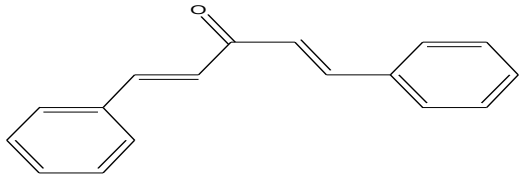
EXPERIMENTAL TECHNIQUES

Materials

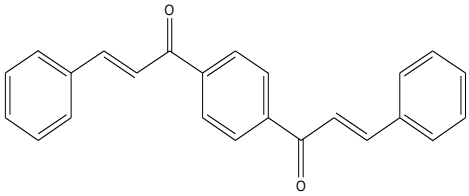
The experiments were performed with pure aluminum sheets having the chemical composition in weight %: Si 0.483, Fe 0.180, Cu 0.001, Mg 0.405, Zn 0.017, Ti 0.015, Cr 0.004, Ni 0.005 and Al balance. The selected inhibitors used in this study were:



(I) Benzalacetophenone



(II) Dibenzalacetone



(III) Dibenzal-1, 4-Diacetylbenzene

Preparation of phosphoric acid and inhibitors solutions

Phosphoric acid solutions were prepared by diluting the appropriate volume of the concentrated chemically pure acid (85%, BDH grade) with doubly distilled water and its concentration was checked by sodium hydroxide. 100 ml stock solutions (10^{-3}M) of compounds (I to III) were prepared by dissolving an accurately quantity of each material in the appropriate volume of ethanol, then the required concentrations (1×10^{-6} – 11×10^{-6}M) were prepared by dilution with doubly distilled water. 100 ml stock solutions (1 M) of each halide salt (BDH grade) were prepared by dissolving an accurately quantity of each material in the appropriate volume of doubly distilled water, from these stock solutions exactly $1x10^{-2}$ M was prepared by dilution with doubly distilled water. Alkaline degreasing mixture (El Hosiery et al., 1972) was prepared by using pure grade sodium carbonate (BDH) 15 g and the same quantity of trisodium phosphate (BDH) and dissolving them in one liter of doubly distilled water. The solution was heated to 80 to 85ºC before being used for degreasing the aluminum pieces for 30 s. Two different techniques have been employed for studying the inhibition of corrosion of aluminum by these compounds, these are: Chemical (Weight loss method) and electrochemical (Galvanostatic polarization) techniques.

Chemical technique (Weight loss method)

The reaction basin used in this method was a graduated glass vessel 6 cm inner diameter having a total volume of 250 ml. 100 ml of the test solutions were employed in each experiment. The test pieces were cut into 2 x 2 cm. They were abraded with emery papers (a coarse paper was used initially and then progressively finer grades were employed), ultrasonically degreased in alkaline degreasing mixture, rinsed in doubly distilled water and finally dried between two filter papers and weighed. The test pieces were suspended by suitable glass hooks at the edge of the basin, and under the surface of the test solution by about 1 cm. After specified periods of time, the test pieces were taken out of the test solution, rinsed in doubly distilled water, dried as before and weighed again. The average weight loss at a certain time for each set of the test pieces was recorded to the nearest 0.0001 g.

Electrochemical technique (Galvanostatic polarization method)

Three different types of electrodes were used during polarization measurements: The working electrode was aluminum electrode, which cut from aluminum sheets. The electrodes were of dimensions 1 × 1× 0.2 cm and were weld from one side to a copper wire used for electric connection. The samples were embedded in glass tube using epoxy resin. Saturated calomel electrode (SCE) and a platinum foil (1 cm^2) were used as reference and auxiliary electrodes, respectively.

A constant quantity of the test solution (100 ml) was taken in the polarization cell. A time interval of about 30 min was given for the system to attain a steady state. Both cathodic and anodic polarization curves were recorded galvanostatically using Amel Galvanostatic (Model-549) and digital Multimeters (Fluke-73) were used for accurate measurements of potential and current density. All the experiments were carried out at 30 ± 1°C by using ultra circulating thermostat.

RESULTS AND DISCUSSION

Weight-loss measurements

Weight loss of aluminum was determined, at various time intervals, in the absence and presence of different concentrations of the investigated compounds. The obtained weight loss-time curves are represented in (Figure 1) for inhibitor (III), the most effective one. Similar curves were obtained for other inhibitors (not shown). The degree of dissolution, of course, dependent on the surface area of the metal exposed and the time of exposure; hence the amount of corrosion is given with

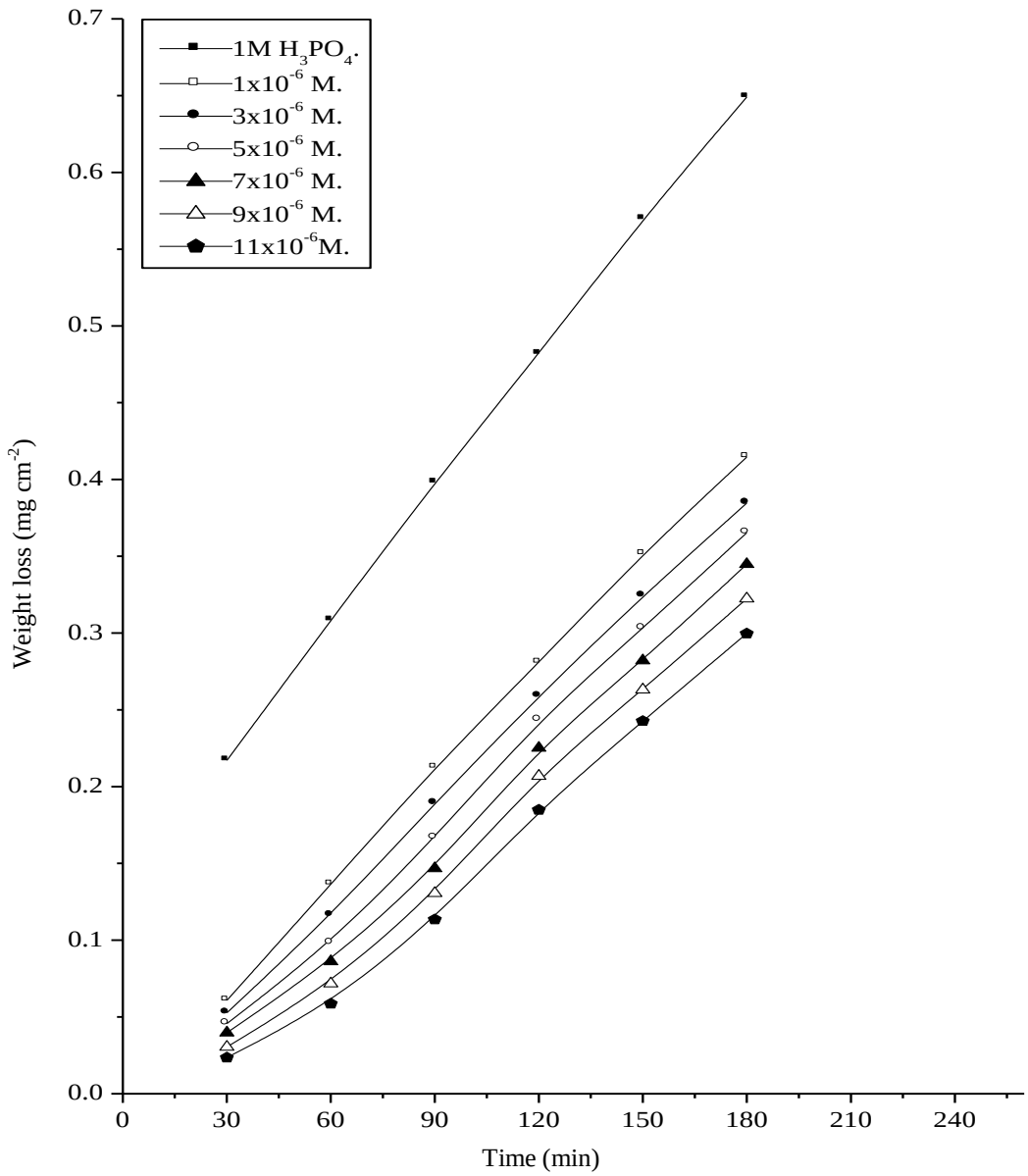


Figure 1. Weight loss – Time curves for the corrosion of Aluminium in 1 M H_3PO_4 in absence and presence of different concentrations of compound (III) at 30°C.

respect to area and time. The resulting quantity, corrosion rate, is thus a fundamental measurement in corrosion science. Corrosion rates can be evaluated by measuring weight of a specimen before and after exposure and applying Equation (1):

$$\Delta W = (W_B - W_A) \quad (1)$$

The percentage of inhibition efficiency (% IE) and the degree of metal surface coverage (θ) of the investigated compounds were computed by Equation (2):

$$\% IE = \theta \times 100 = [1 - (\Delta W_{inh} / \Delta W_{free})] \times 100 \quad (2)$$

where ΔW_{inh} and ΔW_{free} are the weight losses of metal per unit area in the presence and absence of the inhibitors respectively at given time period and temperature. In order to get a comparative view, the variation of the inhibition efficiency (% IE) of the investigated compounds with their molar concentrations was calculated according to Equation (2). The values obtained are summarized in Table 1. Careful inspection of these results showed that, the rate of corrosion was reduced significantly in the presence of these additives indicating that these compounds actually inhibited the corrosion of aluminum. At the same inhibitor concentration, the order of decreasing inhibition efficiency

Table 1. Values of % inhibition efficiencies of Chalcone derivatives for the corrosion of aluminum in 1 M H_3PO_4 from weight-loss measurements at different concentrations and at 30°C.

Concentration (M)	% Inhibition efficiency (% IE)		
	(I)	(II)	(III)
$1x10^{-6}$	20.78	38.07	42.80
$3x10^{-6}$	28.86	41.52	47.28
$5x10^{-6}$	34.09	41.93	47.87
$7x10^{-6}$	36.81	47.76	53.05
$9x10^{-6}$	40.22	52.09	57.87
$11x10^{-6}$	44.53	57.70	62.34

Table 2. % Inhibition efficiency (% IE) at different concentrations of the Chalcone derivatives with addition of $1x10^{-2}$M KI for the corrosion of aluminum after 120 min immersion in 1 M H_3PO_4 at 30°C.

Concentration (M)	% IE		
	(I)	(II)	(III)
$1x10^{-6}$	34.90	47.58	60.25
$3x10^{-6}$	37.48	50.63	63.77
$5x10^{-6}$	41.08	55.16	67.84
$7x10^{-6}$	44.21	58.29	72.38
$9x10^{-6}$	47.89	61.50	75.59
$11x10^{-6}$	52.97	65.65	80.20

of the investigated compounds is as follows: III > II > I.

Compound III is the most efficient inhibitor, due to its higher molecular size and the presence of extra sites such as O atom. Compound II comes after compound III in inhibition efficiency, due to its lower molecular size than compound III and the presence of one active center (O atom). Compound I is the least efficient one, due to the its lower molecular size than compounds II and III.

Synergistic effect

The corrosion of aluminum in 1 M phosphoric acid in the absence and presence of different concentrations of the Chalcone derivatives with addition of a specific concentration (10^{-2} M) of KI, KBr and KCl, respectively was studied. The values of inhibition efficiency (% IE) for specific concentration of KI, KBr and KCl salts in the presence of various concentrations of inhibitors are given in Tables 2, 3 and 4 respectively. From these values, it is observed that % IE of the inhibitors increases on addition of halide solutions and each anion plays a certain effect on the decreasing of corrosion rate process according to the following order (Oguzie, 2004; Jayateetha et al 2012). The order of synergism of halide ions with the tested inhibitors has been found to be I^- > Br^- > Cl^- which on the basis of ionic radii (Rudresh et al., 1979), The reason for better synergism with iodide ion is due to the large size and ease of polarizability of I^- ion, which facilitates chemisorptions on the aluminum surface. The strong chemisorptions of iodide, bromide and chloride ions on the metal surface is responsible for the synergistic effect of these anions in combination with cation of the inhibitor. The cation is then adsorbed by columbic attraction on the metal surface where these ions are already adsorbed by chemisorptions. Stabilization of adsorbed anions with cations leads to greater surface coverage and therefore greater inhibition (Maitra et al., 1983; Gomma, 1998).

The order of decreasing inhibition efficiency of the investigated compounds on addition of a specific concentration of the investigated halides is as follows: III > II > I. It can been seen from Table 2 that the addition of a specific concentration (10^{-2} M) of KI, KBr and KCl inhibits the corrosion of aluminum in 1 M phosphoric acid in the absence and presence of different concentrations of investigated compounds to a large extent. This can be interpreted according to Schmitt and Bedbur (1985), which proposed two types of joint adsorption namely competitive and cooperative. The synergistic inhibition effect was evaluated using a parameter, S_θ, obtained from the surface coverage values (θ) of the anion, cation and both. Aramaki and Hackerman (1969) calculated the

Table 3. % Inhibition efficiency (% IE) at different concentrations of the Chalcone derivatives with addition of $1x10^{-2}$ M KBr for the corrosion of aluminum after 120 min immersion in 1 M H_3PO_4 at 30^oC.

Concentration (M)	% IE		
	(I)	(II)	(III)
$1x10^{-6}$	30.76	41.94	52.74
$3x10^{-6}$	33.72	46.87	57.67
$5x10^{-6}$	38.26	51.41	62.68
$7x10^{-6}$	41.39	55.95	67.68
$9x10^{-6}$	45.07	60.09	71.36
$11x10^{-6}$	49.19	63.77	75.51

Table 4. % Inhibition efficiency (% IE) at different concentrations of the Chalcone derivatives with addition of $1x10^{-2}$ M KCl for the corrosion of aluminum after 120 min immersion in 1 M H_3PO_4 at 30^oC.

Concentration (M)	% IE		
	(I)	(II)	(III)
$1x10^{-6}$	25.51	40.06	48.54
$3x10^{-6}$	30.44	44.99	52.06
$5x10^{-6}$	35.91	50.47	57.04
$7x10^{-6}$	41.39	55.48	61.11
$9x10^{-6}$	46.95	60.09	65.73
$11x10^{-6}$	52.04	65.18	70.35

synergism parameter, S_{θ} using Equation (3)

$$S_{\theta},= [1- (\theta_{1+2}/1- \theta'_{1+2})] \quad (3)$$

where $\theta_{1+2} = (\theta_1+\theta_2) – (\theta_1\theta_2)$, θ_1 is the surface coverage by anion, θ_2 is the surface coverage by cation and θ'_{1+2} is the measured surface coverage by both the anion and the cation. The values of S_{θ} are nearly equal to unity, which suggests that the enhanced inhibition efficiency caused by the addition of iodide, bromide and chloride ions individually to the investigated compounds is due mainly to the synergistic effect.

Adsorption isotherm

Adsorption isotherm equations are generally of the form (Khamis et al., 2000):

$$f (\theta, x) \exp (-a, \theta) = K C \quad (4)$$

where f (θ, x) is the configurationally factor that depends essentially on the physical model and assumptions underlying the derivation of the isotherm, 'a' is the molecular interaction parameter depending upon molecular interactions in the adsorption layer and the degree of heterogeneity of the surface. Attempts were made to fit θ values to various isotherms including Frumkin, Langmuir, Flory-Huggins, Temkin, Freundlich and El-Awady kinetic/ thermodynamic model, best fit for the experimental data were the Freundlich isotherm and correlation coefficient (R^2) values were used to determine the best fit isotherm. The Freundlich isotherm is given by:

$$\log \theta = \log Kads + n \log C \ (0 < n < 1) \quad (5)$$

Plots of log θ vs. log C (Freundlich adsorption plots) for adsorption of the used compounds on the surface of aluminum in 1 M phosphoric acid at 30°C are shown in (Figure 2). The data gave straight lines of intercept log Kads and slope n indicating that Freundlich adsorption isotherm is valid for these compounds. From the Figure the order of decreasing inhibition efficiency of the Chalcone derivatives is as follows: (III > II > I). All adsorption expressions include the equilibrium constant of the adsorption process, Kads, which is related to the standard free energy of adsorption (ΔG°ads.) by the following equation (Kliskic et al., 1997; Abdallah, 2000):

$$K_{ads} = 1/ 55.5 \exp (-\Delta G°_{ads}/ RT) \quad (6)$$

where R is the universal gas constant, T is the absolute

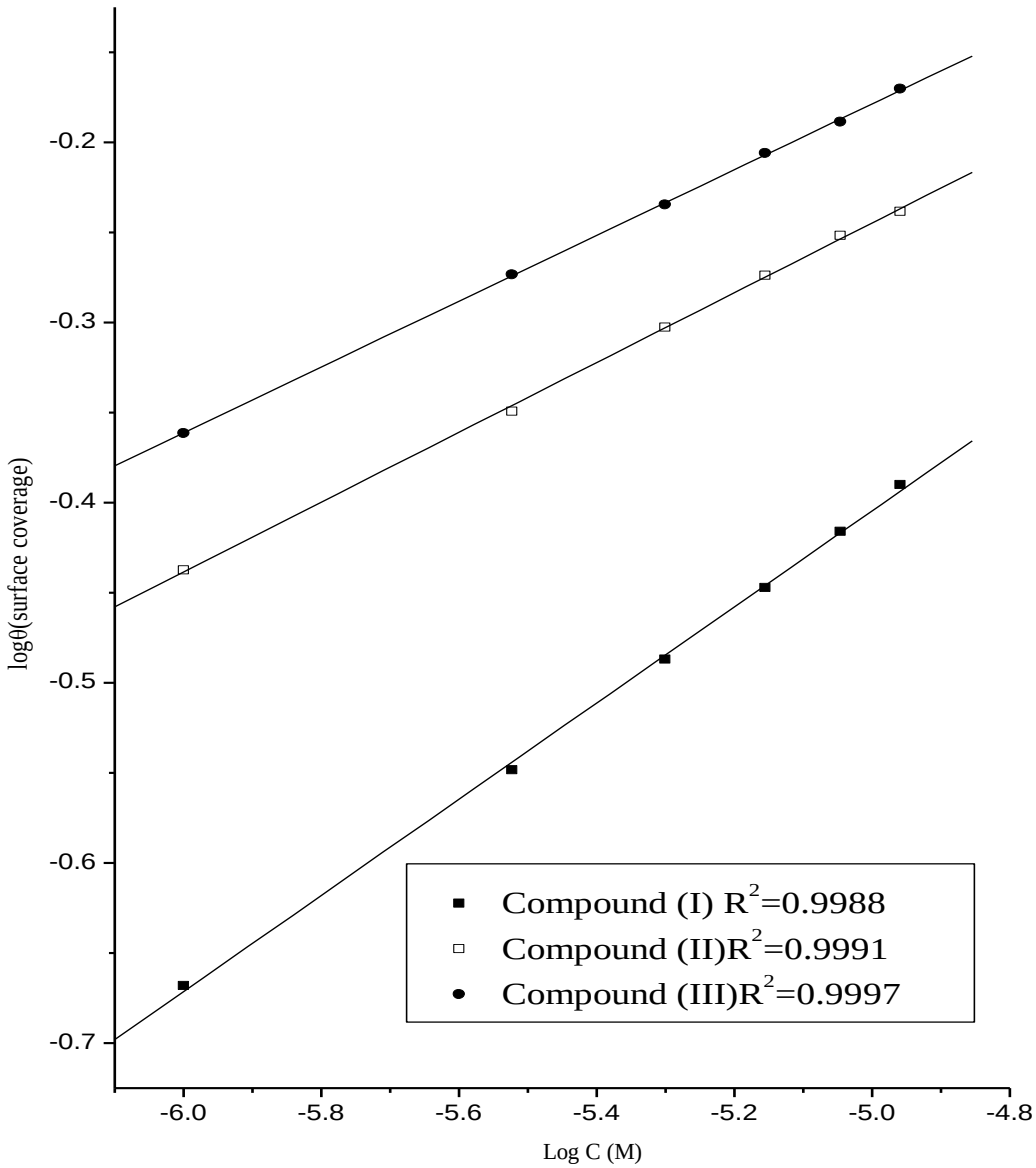


Figure 2. Curve fitting of corrosion data for Aluminium in 1 M H_3PO_4 in presence of different concentrations of the used compounds to Freundlish adsorption isotherm at 30°C.

Table 5. Equilibrium constant and adsorption free energy of the Chalcone derivatives adsorbed on aluminum surface.

Inhibitors	$-\Delta G°_{ads}$ (kJ mol^{-1})	K_{ads},x 10^{-2} (M^{-1})
(I)	4.7	11.77
(II)	5.5	16.51
(III)	5.8	18.44

temperature and the value 55.5 is the concentration of water in mol/l. The value of $\Delta G°_{ads}$ were calculated and are listed in Table 5. It is clear that the value of $\Delta G°_{ads}$ increases with the increasing luminium energy of adsorbed species which in turn increases with increasing the size of the molecule (Blomgren et al., 1961). The

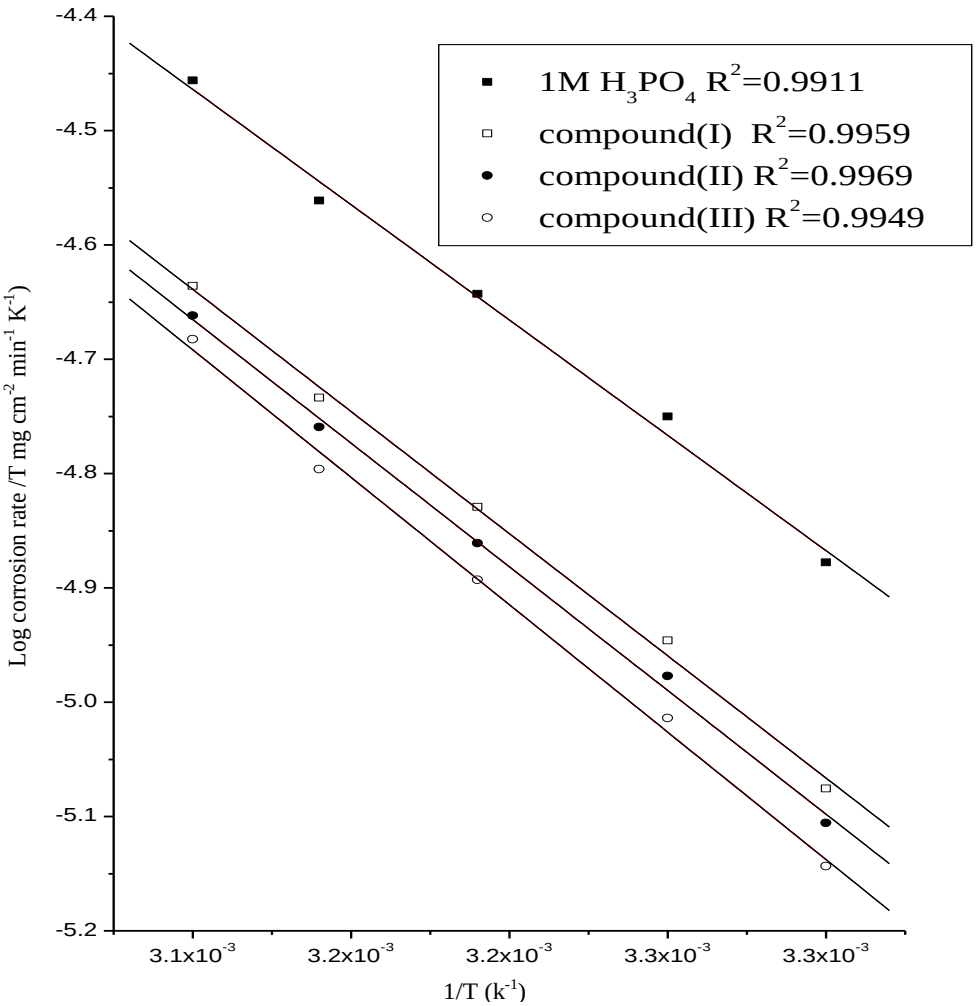


Figure 4. Log (corrosion rate/T) – (1/T) curves for the corrosion of Aluminium in 1 M H_3PO_4 at $5x10^{-6}$ M after 120 min for the used compounds.

negative values of $\Delta G°_{ads}$ obtained here indicate that the adsorption process of these compounds on the metal surface is spontaneous one.

Effect of temperature and activation parameters of inhibition process

The effect of temperature on the corrosion rate of aluminum in 1 M H_3PO_4 over the temperature range (30 to 50°C) in absence and presence of different concentrations of the investigated compounds has been studied. The % inhibition efficiency is found to decrease with increasing the temperature; this may be attributed to increase in the solubility of the protective films and of any reaction products precipitated on the surface of the metal that may otherwise inhibit the reaction rate. As reported before (Ferreira et al., 2004), a decrease in the inhibition efficiency with increasing temperature indicates inhibitor physisorption on the corroding metal surface while the reverse behavior implies chemisorptions. Similar observations have been reported elsewhere (Popova et al., 2003; Oguzie, 2007). Plots of logarithm of corrosion rate (log k), with reciprocal of absolute temperature (1/T) for aluminum in 1 M H_3PO_4 at $5x10^{-6}$ M after 120 min for the Chalcone derivatives are shown in (Fig. 3). As shown from this Figure, straight lines with slope of $-E_a^*/ 2.303$ R and intercept of A were obtained according to Arrhenius-type equation:

$$k = A \exp(- E_a^*/ RT) \quad (7)$$

where: k is the corrosion rate, A is a constant depends on a metal type and electrolyte, and E_a^* is the apparent activation energy.

Plots of log (corrosion rate/ T) vs. 1/ T for aluminum in 1 M H_3PO_4 at $5x10^{-6}$ M after 120 min for the Chalcone derivatives is shown in (Figure 4). As shown from this

Table 6. Activation parameters of the corrosion of aluminum in 1 M H_3PO_4 at $5x10^{-6}$ M after 20 min immersion for the Chalcone derivatives.

Inhibitors	Activation parameters		
	E_a* (kJ mol^{-1})	ΔH* (kJ mol^{-1})	-ΔS* (J $mol^{-1}K^{-1}$)
Free acid	43.667	38.612	163.22
(I)	45.953	40.896	159.48
(II)	46.451	41.397	158.44
(III)	47.696	55.48	155.05
$9x10^{-6}$	46.95	60.09	-ΔS* (J $mol^{-1}K^{-1}$)
$11x10^{-6}$	52.04	65.18	163.22

figure, straight lines with slope of (-ΔH*/ 2.303R) and intercept of (log R/ Nh + ΔS*/ 2.303R) were obtained according to transition state equation:

$$\text{Rate} = RT/ Nh \exp (\Delta S^*/ R) \exp (-\Delta H^*/ RT) \quad (8)$$

where h is Planck's constant, N is Avogadro's number, ΔH* is the activation enthalpy and ΔS* is the activation entropy. The calculated values of the apparent activation energy, E_a*, activation enthalpies, ΔH* and activation entropies, ΔS* are given in Table 6. The entropy of activation (ΔS*) in the blank and inhibited solutions is large and negative indicating that the activated complex represents association rather than dissociation step (Gomma et al., 1995; Marsh, 1988; Soliman, 1995). The value of the activation energy for the corrosion of aluminum in 1 M H_3PO_4 solution in absence of the additives is equal to 43.7 kJ mol^{-1}, which is in the same order of the magnitude as those observed by Yadav et al. (1999), 54.8 kJ mol^{-1} for the corrosion of aluminum 3003 in 20% HNO_3. The order of decreasing inhibition efficiency of the investigated compounds as gathered from the increase in E_a* and ΔH^*_{ads} values and decrease in ΔS^*_{ads} values, is as follows: III > II > I.

Galvanostatic polarization measurements

Figure 5 shows the galvanostatic polarization curves for aluminum dissolution in 1 M H_3PO_4 in the absence and presence of different concentrations of inhibitor (III) at 30°C. Similar curves were obtained for other inhibitors (not shown). Corrosion kinetic parameters such as corrosion current density (I_{corr}), corrosion potential (E_{corr}), Tafel slopes (β_a and β_c), degree of surface coverage (θ) and inhibition efficiency (% IE) with the concentrations of the investigated compounds are given in Table 7. The lower current density (Icorr) values in the presence of inhibitors without causing significant changes in corrosion potential (Ecorr) suggest that the compounds are mixed type inhibitors and are adsorbed on the metal surface there by blocking the corrosion reaction. The Tafel constant ba and bc are both affected and there is no definite trend in the shift of Ecorr values. This suggests that the compounds are mixed type inhibitors. The higher the values of (β_c) over those of (β_a) suggests that the cathodic reaction is the predominate factor and that the additives act mainly as cathodic inhibitors of the blocking type (Fouda et al., 1986b). The order of decreasing inhibition efficiency of the investigated compounds is as follow: III > II > I. This is also in agreement with the observed order of corrosion inhibition by the weight loss method.

Chemical structure of the inhibitors and its effect on the corrosion inhibition

Inhibition efficiency of the additive compounds depends on many factors which include the number of adsorption active centers in the molecule and their charge density, molecular size, and mode of interaction with metal surface. It is generally believed that the adsorption of the inhibitor at the metal/solution interface is the first step in the mechanism of inhibitor action in aggressive acid media. The order of decreasing inhibition efficiency of these tested compounds is III > II > I, due to three reasons:

1) Conjugation of these compounds is clearly appeared especially in compound (III) which is more efficient and conjugated, so electron density increases and reactivity of this inhibitor becomes larger than compound (II) and compound (I) and probability of its attachment to the metal increases.
2) Molecular size of the molecules (III > II > I).
3) Polarization in compound (III) [–C=C–C= O] (α, β-unsaturated ketone) make more partial negative on oxygen atom and more partial positive on carbon atom in position β than other compounds (II and I). So, more

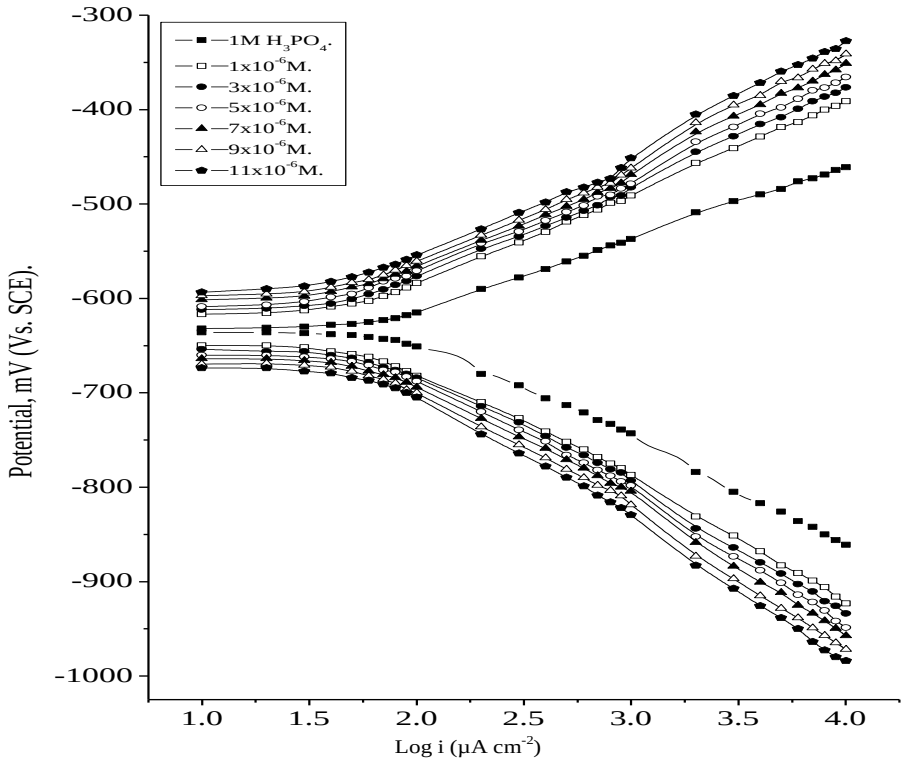


Figure 5. Galvanostatic polarization curves for the corrosion of Aluminium in 1 M H_3PO_4 at $5x10^{-6}$ M in absence and presence of different concentrations of compound (III) at 30°C.

Table 7. Electrochemical parameters for aluminum in 1 M H_3PO_4 in the absence and presence of different concentrations of Chalcone derivatives at 30°C.

Inhibitors	Concentration (M)	$-E_{corr}$ (mV vs SCE)	I_{corr} (µA cm^{-2})	$-\beta_c$ (mV dec^{-1})	β_a (mV dec^{-1})	θ	% IE
	0	630	66.4	89	78	-------	-------
	$1x10^{-6}$	627	51.4	101	80	0.225	22.5
	$3x10^{-6}$	626	46.9	102	81	0.294	29.4
(I)	$5x10^{-6}$	625	43.2	103	82	0.350	35.0
	$7x10^{-6}$	625	41.6	106	83	0.373	37.3
	$9x10^{-6}$	624	39.0	108	83	0.412	41.2
	$11x10^{-6}$	623	36.1	109	84	0.456	45.6
	$1x10^{-6}$	624	40.9	104	81	0.384	38.4
	$3x10^{-6}$	623	37.0	107	83	0.443	44.3
	$5x10^{-6}$	622	34.7	108	86	0.477	47.7
(II)	$7x10^{-6}$	622	32.4	109	87	0.512	51.2
	$9x10^{-6}$	621	30.0	111	88	0.548	54.8
	$11x10^{-6}$	620	28.5	114	90	0.571	57.1
	$1x10^{-6}$	623	37.2	106	83	0.440	44.0
	$3x10^{-6}$	622	34.3	107	88	0.483	48.3
	$5x10^{-6}$	622	33.1	108	91	0.502	50.2
(III)	$7x10^{-6}$	621	30.1	112	92	0.546	54.6
	$9x10^{-6}$	620	27.6	115	93	0.584	58.4
	$11x10^{-6}$	618	23.4	117	94	0.648	64.8

polarizability gives high inhibition efficiency. The order of inhibition efficiency of the additives revealed by the weight loss method is further supported by galvanostatic polarization measurement. The observed agreement among these independent techniques proves the validity of the results obtained and supports the explanation given for the effect of chemical structure on the inhibition action of the investigated compounds.

Conclusions

1. The investigated compounds are efficient inhibitors for aluminum dissolution in 1M H_3PO_4
2. The adsorption of these compounds on the aluminum surface was found to obey Freundlich adsorption isotherm
3. From the effect of temperature, the activation parameters for the corrosion process (E^*_a, ΔH^* and ΔS^*) were calculated
4. Percentage inhibition (% IE) increased in the presence of 1x 10^{-2} M KI, KBr and KCl due to the synergistic effect
5. Galvanostatic polarization data indicated that these compounds influence both cathodic and anodic processes
6. The order of the inhibition efficiency of the inhibitors as given by polarization measurements is in good agreement with that obtained from weight loss measurements. This order was explained on the basis of the chemical structure and adsorption active centers of the compounds.

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