

INHIBITION EFFECT OF SCHIFF BASE COMPOUNDS ON THE CORROSION OF IRON IN NITRIC ACID AND SODIUM HYDROXIDE SOLUTIONS

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Abstract

Studies using chemical and electrochemical techniques were carried out on an iron electrode in 2.0 mo I/liter -nitric acid and 2.0 mol/liter sodium hydroxide solutions to evaluate the corrosion behavior. Schiff bases investigated are found to function well as corrosion inhibitors for iron metal in both nitric acid and sodium hydroxide solutions, but a better performance is noticed in nitric acid solution. This is due to the less negative potential of iron in acidic solution , favoring adsorption of the additives. The Schiff bases act as mixed type inhibitors in acidic solution, whereas they cause only inhibition of the cathode process in alkaline medium and the magnitude of the displacement of the Tafel plot is proportional to its concentration. The additives inhibit the corrosion through their ability of getting adsorbed on the iron surface by interaction with π -electrons of the aromatic rings, and the azo methine group. The adsorption process was found to obey Temkin adsorption isotherm. Also, the adsorbed Schiff base molecules interact with iron ions formed on the metal surface

leading to neutral and cationic iron-Schiff base complexes. Models for the inhibition corrosion behavior were developed based upon various statistical analyses of the experimental data.

Keywords: Corrosion, inhibition, iron, Schiff base, HNO₃and NaOH.

INTRODUCTION

The corrosion behavior of iron and its passivation processes have been studied extensively over the last few decades (1-11). Some Schiff base compounds have recently been reported as effective corrosion inhibitors for mild steels (12,13) in various aqueous solutions. The effects of Schiff base-metal complexes (14) on the corrosion of mild steel were investigated by corrosion weight loss, electrochemical and surface analyses measurements. The molecules of Schiff Base-metal complexes adsorbed on the surfaces of mild steels depress the corrosion. The objective of this work is to study the inhibitive action of some Schiff bases towards the corrosion process of iron in nitric acid and sodium hydroxide solutions and also, to determine the adsorption isotherms and to compare them with kinetic-thermodynamic models of corrosion inhibition. The choice of the inhibitor is based on two considerations. First, it can be synthesized conveniently from relatively cheap raw materials. Secondly the presence of an electron cloud, on the aromatic ring, the electronegative nitrogen, oxygen atoms and the relatively long chain compound in aqueous media can induce greater adsorption of the compound on the iron surface promoting effective inhibition (15)



EXPERIMENTAL

Iron specimens and electrolytes

Iron specimens (0.16% C, 0.05% Si, 0.37% Mn, 0.015% S) were used in the present study. Prior to each experiment, the electrodes were mechanically polished with successive grades of emery paper, degreased in pure acetone, washed 'in running bid stilled water, dried, and weighed before being inserted in the cell to remove any oxide layer or corrosion product from the surface (16,17). 2M nitric acid and 2M sodium hydroxide solutions were prepared by diluting Analar reagents by bidistilled water.

Additives

The structural formulae of the investigated Schiff base derivatives **1-5** are given below. The compounds were synthesized according to the literature ⁽¹⁸⁾. Their purity was checked by melting point determinations and spectroscopy. The Schiff base solutions were prepared by dissolving the appropriate amount of compound in 25 cm³ Analar ethanol. The desired volume of the free inhibitor was added to the electrolyte solution. The solvent effect must be considered by mixing a definite volume of Et OH to the free acid to reach a constant ratio of Et OH in each test in the absence and presence of different concentrations of inhibitor.



Weight loss measurements

The reaction basin used in this method was a graduated glass vessel of 6 cm inner diameter and a total volume of 250 cm^3 . 100 cm^3 of the test solution at $303.0 \pm 1.0 \text{ K}$ were employed in each experiment. The iron pieces (2x 2x 0.lcm) were prepared as described before, weighed, and suspended under the surface of the test solution by about 1cm by suitable glass hooks. After specified periods of time, three pieces of iron were taken out of the test solution, rinsed with doubly distilled water, dried, and re-weighed. The average weight loss at a certain time for each of the three test samples was taken. The percentage of inhibition (% In) of different concentrations of the inhibitors was calculated according to:

% In =
$$\underline{\text{Wt. loss (pure)- Wt. loss(inh)}}$$
 x 100
Wt. loss (pure)

Thermometric measurements

The reaction vessel used was basically the same as that described by Mylius $^{(19)}$. An iron piece (1 x 10 x 0.1 cm) was immersed in 30 cm³ of either 2M HNO₃ or 2M NaOH in the absence and presence of additives, and the temperature of the system was followed as a function of time. The procedure for the determination of the metal dissolution rate by the thermometric method has been described previously $^{(19,20)}$. The reaction number (RN) is defined $^{(21)}$ as:

$$RN = (T \max -Ti)$$

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T max and Ti, are the maximum and initial temperatures, respectively, and t is the time (in minutes) required to reach the maximum temperature. The percent reduction in $RN^{(22)}$ is then given as:

% reduction in
$$RN = (RN free - RN inh)$$
 x 100
RN free

Polarization measurements

A conventional three - electrode cell was used with a 1.0 cm² Pt sheet as the counter electrode which was separated from the main cell compartment by a glass sinter. The potentials of the working electrode were referred to a saturated calomel electrode (SCE). In order to avoid contamination, the reference electrode was connected to the working- electrode through a salt bridge filled with the test solution. The tip of the bridge was pressed against the working electrode in order to compensate the ohmic drop. Prior to each experimental measurement, the solution under investigation (25 cm³) was freed of oxygen by passing prewashed pure nitrogen through it for a sufficient time. Measurements were performed on a planar disk electrode (A= 1cm²). The iron electrodes were carefully degreased, and the edges were masked by appropriate resins (Duracryle, Spofa-Dental, and Praha). The surface of the iron electrodes were prepared by mechanical grinding and polishing as given elsewhere (16,17). The electrodes were rinsed in an. ultrasonic bath containing bid stilled water and finally washed with bid stilled water immediately before being immersed in the



cell. The pretreatment procedure was repeated before each experiment.

Anodic and cathodic potentiostatic polarization of iron electrodes were measured with a (Wenking Potentioscan model POS 73). Potentials and currents were determined by digital multi meters. Corrosion current densities (*Icorr*) were determined by extrapolation of the anodic and cathodic Tafel lines to the free corrosion potential value (Ecorr). Each experiment was conducted with a freshly prepared solution and with newly polished electrodes. The cell temperature was kept constant at 303.0 ± 1.0 K in an ultra-thermostat.

RESULTS AND DISCUSSION

Weight loss measurements

The behavior of corrosion inhibition for iron by the tested Schiff bases is investigated by the corrosion weight loss measurements in either 2M HNO₃ acid or 2M NaOH solutions as shown in **Fig.1**. The percentage inhibition efficiency (% In) values of Schiff bases on the corrosion of iron in either acidic and/or alkaline media indicate that the inhibition efficiency depends on both the nature and the concentration of the additive used. The curves obtained in the presence of Schiff bases in 2M HNO₃ are characterized by a rapid rise as shown in **Fig. 1(A)** for compound **1** as an example since, the oxide film formed on the iron surface offers no protection against acid attack. The corresponding curves in 2M NaOH solution (Fig. 1(B) are straight lines starting from the origin because the oxide film originally formed is easily removed by alkaline solution and weight loss gradually increases by increasing the time. The curves obtained



in the presence of Schiff base additives fall below that of the free acid and alkaline as given in **Figure 1 (A) and (B)**.

Schiff base compounds may exist in acid solution as neutral molecules or in the form of cations and it is the neutral molecules that cover the metal surface via the chemisorptions mechanism (23) involving the sharing of electrons between the nitrogen atom and iron metal atoms. Chemisorptions involves the share or transfer of charge from the molecules to the surface metal ions forming a coordinate type bond. Electron transfer is typical for transition metals having vacant low-energy electron orbitals. As for inhibitors, the electron transfer can be expected with compounds having relatively loosely bound electrons (24). In nitric acid and sodium hydroxide solutions, the corrosion rate of iron is decreased with increasing concentration of the Schiff base inhibitors as well as with increasing the number of methylene groups. The Schiff base compounds act as effective corrosion inhibitors in 2 M HNO₃ solution. The higher inhibition efficiency of the compounds in acidic than in alkaline media may be due to the less negative potential of iron in nitric acid solution, favoring adsorption of the additive. The inhibition efficiency of the Schiff base inhibitors under investigation increases in the order 1 > 2 > 3 > 4 > 5 respectively. The inhibition effect may be explained by considering the adsorption of the Schiff base molecules (with high negative charge density at the hetero atom) on the metal surface^(25,26) consisting of iron atoms with incomplete d shell⁽²⁷⁾ and interaction of π -electrons with the metal.

Also, formation of a metal-Schiff base complex on the corroding iron surface (surface chelation) may play a role (13,28) either in acidic or alkaline solutions.

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In general, the amine groups such as - NH-and - NH₂ respectively, ionize mainly as -NH₂⁺ - and - NH₃⁺ in acidic media. These ionized groups adsorb on iron surface to form a film on iron, which inhibits the corrosion of iron. Many Schiff base – metal complexes seem to be adsorbed on the surface of iron by those having a 5- or 6-membered chelate ring (n = 2 or 3) with a metal ion. This interpretation is supported experimentally by spectroscopic analysis (mainly ultraviolet spectra) as shown in Fig.2. Holm et al. (29) have already reported that the size of the metal ion accommodated in stable complexes is not critical if the ionic radius is in the range 0.53 for Fe³⁺, hence, the chelating of tri-and tetravalent ions of the lower transition series is possible (29). The Temkin adsorption isotherm is found to be ideally obeyed in acidic and alkaline solutions as represented in Fig.3, indicating that the main inhibition process takes place through an adsorption (30,31) process. Assuming, that the inhibitor forms a monolayer on the surface of iron at the maximum inhibition, the surface coverage (Θ) value is calculated from the results of corrosion weight loss measurements. Here, (Θ) can be given as: $(\Theta) = (1 - u / u_0)$ Where u_0 and u are the corrosion weight losses of the uninhibited and inhibited iron, respectively. The plots of logarithmic concentration of inhibitors ($\log C$) vs. the surface coverage (Θ) of inhibitors (**Fig.3**) gave straight lines. Consequently, the adsorption of Schiff bases and Schiff base-metal complexes on the surface of iron was found to be governed by the Temkin adsorption isotherm (32) namely:

$$\Theta = a + \ln b C$$



Where C is the concentration of the additive in the bulk of the solution, (Θ) is the degree of coverage of the iron surface by the adsorbed molecules, and a and b are constants.

Thermometric measurements

The temperature change of the system involving iron in 2M HNO₃ acid solution was followed in the absence and presence of different concentrations of the inhibitor **Fig.4**. Upon increasing the concentration of the additive, the time required to reach T max. increases. This indicates that the inhibitor retards the dissolution of iron in the corrosive media, presumably by adsorption onto the surface of the metal. The extent of retardation depends on the degree of coverage of the metal surface with the adsorb ate. The temperature vs. time curves provides a means of differentiating between weak and strong adsorption (20). Strong adsorption is noted, since a simultaneous increase in t and a diminution in T max. take place, and both factors cause a large decrease in RN (reduction in reaction number) of the system. The results reported in **Table 1** reveal that the inhibition efficiency of the additive, as determined from the percentage reduction in RN, increases with increasing concentration of the additives.

Figure 5 shows the relation between % reduction in RN and the molar concentration of different Schiff base additives. The curves obtained are invariably sigmoid in nature, substantiating that the present inhibitors retard the corrosion rate by adsorption according to the Temkin isotherm⁽³³⁾, i.e. the

adsorption is taking place during the inhibitor action⁽³³⁾. Schmitt et al⁽³⁴⁾ reported, that the corrosion efficiency strongly depends on the structure and chemical properties of the layer formed on the metal surface under particular conditions. In case of the heterocyclic nitrogen compounds under investigation in acidic solutions, the adsorption is ascribed to effects connected with the aromatic rings. It has been reported that the adsorption of the heterocyclic compounds occurs with aromatic rings parallel to the surface. The adsorption of the investigated Schiff bases depends mainly on the electronic structure of the molecules and the inhibition efficiency increases with increasing the length of the hydrocarbon chain and also the number of aromatic rings ^(35, 36). The antipyrine category has a large size that makes better surface coverage and enhances the inhibition efficiency values. So, the inhibitive action of the Schiff Base additives can be attributed to its adsorption on the iron surface and inhibiting the dissolution of the metal by blocking the active sites on its surface

Polarization measurements

Anodic and cathodic polarization of iron metal was carried out under potentiostatic conditions in 2M HNO₃ acid and 2M NaOH solutions in the absence and presence of different concentrations of the Schiff bases .The data indicate that the additives act as mixed type inhibitors in acidic medium, whereas the inhibitors act only as cathodic inhibitors in alkaline solution, the magnitude of displacement of the Tafel plots is proportional to its concentration . **Figure 6(A)** shows *the* polarization curves of iron in 2M nitric acid solution at different concentrations of compound **1**(as an example). The results obtained for the other compounds were quite similar. The inhibition efficiency depends on



many factors including the number of adsorption sites or functional groups, basicity, and molecular size. As the concentration of the Schiff base additive was increased both I $_{corr}$. and R $_{corr}$. are decreased and the percentage inhibition efficiency (%In.) increased. The plot of %In. vs. the molar concentration has the sigmoid nature (**Fig. 7**), indicating that *an* adsorption process is taking place during the inhibitor action $^{(33)}$. The investigated Schiff base compounds contain C = N bond thus exhibit basic properties in addition to the presence of lone pair of electrons on the nitrogen atoms. Also, the Schiff base additives have the ability to form complexes with the dissolved iron ions either in acidic or alkaline corrosive solutions.

Cathodic polarization curves obtained for iron in 2M NaOH solution in **Fig.6** (**B**) show that, the Schiff bases are only cathodic inhibitors in alkaline medium. The comparison between the inhibition efficiency values of the Schiff bases tested as inhibitors and the compatibility of the results obtained using weight loss, thermometric and potentiostatic polarization methods shows that, the three methods gave consistent results.

From **Table 2** it can be seen that the three different techniques gave the same results for the inhibition of corrosion of iron in both acidic and alkaline solutions.

Skeletal representation of Schiff base compounds

Skeletal representation of the proposed mode of adsorption of the tested Schiff base additives is represented in **Fig. 8**. Figure 8 show that the Schiff bases have

two active adsorption centers. Compounds 1 and 2 have the highest percentage inhibition efficiency. This is due to the large size of antipyrine category which makes better surface coverage and hence the highest inhibition efficiency is obtained. Compound 1 displays the highest inhibition efficiency, this is due to the presence of additional benzene ring in its structure which enhances the electron density on the inhibitor in addition to its higher molecular area. The inhibition efficiency of compound 3 is higher than that of compound 4, due to the presence of an additional ring in the arylidene part of the additive, which increases the area of the adsorbed molecule and consequently makes better surface coverage on the metal surface than in the case of the other additives 4 and 5. The inhibition efficiency of compound 4 is higher than that of compound 5, which can be explained on the basis that, the basic strength of the C = Ngroup is insufficient to permit the formation of stable complex through a simple coordination bond with the metal ion through the lone pair of the nitrogen atom. Therefore, a functional group with a replaceable hydrogen ion (OH group) in the ortho position of compound 4 enhances the formation of a stable chelate complex with the corroding metal ions. Consequently, both the Schiff base inhibitor and Schiff base-metallocomplexes can be adsorbed or deposited on the metal surface, hence causing an additional inhibition of iron corrosion (13, 14). Finally, compound 5 comes at the end of all the tested Schiff base additives in its percentage inhibition efficiency value, owing to the presence of OH group in the para position. The adsorption process of Schiff base molecules on the metal surface interfere with the adsorption of the anions (39) present in the corrosive media.

In general, the adsorption of organic molecules at the metal electrode surface depends on the molecular size, charge distribution and deformability of the active center as well as the charge on the metal surface undergoing corrosion. Thus, the increased formation of Schiff base-metal complexes leads to the formation of an insoluble film of the complex on the metal surface, which furnishes an additional inhibitive property to that of the investigated Schiff base inhibitors.

Kinetic - thermodynamic model of corrosion inhibition

To evaluate the kinetic parameters and correlate them to the corrosion inhibition mechanism, it is of value to analyze the kinetic data obtained in the presence of Schiff base inhibitors from the standpoint of the generalized mechanistic scheme proposed by El-Awady et al ^(38, 39). The equations valid for this treatment are

$$\theta / (1 - \theta)$$
 = K [I inh.]^y.....(1)

Where (y) is the number of inhibitors molecules [I inh.] occupying one active site., and K is a constant. The binding constant (Kb) corresponding to that obtained from the known adsorption isotherms curve fitting is given by $^{(38)}$.

$$Kb = K^{(1/y)}$$
(2)

Where (1/y) represents the number of active sites of the metal surface occupied by one inhibitor molecule. A plot of $\log (\theta/1-\theta)$ versus $\log C$ inh. for the tested Schiff base compounds give satisfactory linear relationship of slope (y) and intercept at $(\log K)$. The slope values do not equal unity (gradient slopes < 1),



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hence the adsorption of the Schiff base compounds on Fe surface does not obey a Langmuir adsorption isotherm. Values of (1/y) greater than unity imply the formation of multilayer of the inhibitor molecules on the metal surface, however values of (1/y) less than unity, mean that a given inhibitor molecule will occupy more than one active site (33).

Table 3 comprises the values of 1/y which give the number of active sites occupied by a single organic molecule; K is the binding constant (40). The values of B (equilibrium constant) and f (lateral interaction parameter) are also reported in **Table 3**. The large negative values of AG° indicate that the reaction proceeds spontaneously and is accompanied by an efficient adsorption. Larger values of K and B point to better inhibition efficiency of the tested Schiff base compounds, i.e. stronger electrical interaction between the double layer at the phase boundary and the adsorbed molecules.

The lack of the compatibility between the calculated (K) and (B) values may be attributed to the fact that, Temkin adsorption isotherm equation is only applicable to the cases in which one active site per inhibitor molecule is occupied.

In general, the equilibrium constant of the adsorption process was found to rise with increasing the inhibition efficiency.

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Table 1. Effect of 4-(2-hydroxynaphthylidene amino) antipyrine concentration on the thermometric parameters for iron in $2M\ HNO_3$ acid.

Conc.	Ti	T max.	t	Δt	Log t	Ө	R N C ^o	% red.
mol/L	C^{o}	Co	min.	min.	min.		min ⁻¹	in RN
0.0	19.5	50.8	68				0.460	
$5x10^{-7}$	17.8	46.8	130	62	1.792	0.515	0.223	51.6
1x10 ⁻⁶	17.5	46.5	141	73	1.863	0.554	0.205	55.4
$5x10^{-6}$	17.0	45.7	155	87	1.939	0.597	0.185	59.8
$1x10^{-5}$	18.0	44.1	177	109	2.037	0.680	0.147	68.0
$5x10^{-5}$	18.0	44.0	190	122	2.086	0.702	0.136	70.3
$1x10^{-4}$	18.0	41.0	200	132	2.120	0.750	0.115	75.0

Table 2. Comparison between the efficiency of corrosion inhibition as determined by weight - loss, thermometric and polarization at $1x10^{-4}$ M concentration at 303 K.

% Inhibition								
Inhibitor	Weight-loss		Thermo	ometric	Polarization			
	HNO ₃	NaOH	HNO ₃	NaOH	HNO ₃	NaOH		
1	78.4	58.3	75.9		73.8	63.2		
2	76.1	55.3	75.0		72.0	58.1		
3	75.2	54.1	74.1		71.1	62.3		
4	74.1	52.7	73.7		69.8	56.8		
5	73.1	48.2	71.5		68.6	55.3		



Table 3. Curve fitting data to the kinetic-thermodynamic model (r = 0.94) and the Temkin adsorption isotherm for Schiff base inhibitors in acidic and alkaline solutions at 303 K.

Inhibitor	Medium	K	inetic mod	lel	Temkin adsorption isotherm			
		1/y	K	-ΔG ^o	-f	В	-ΔG ^o	
				kJ/mol			kJ/mol	
1	HNO ₃	10.44	$1.8 \text{x} 10^8$	37.75	23.46	36307	16.33	
	NaOH	4.80	$3.3x10^4$	16.01	31.55	13803	13.89	
2	HNO ₃	10.00	$1.2x10^8$	36.81	24.16	31622	15.98	
	NaOH	4.54	$2.2x10^4$	15.11	33.30	12302	13.60	
3	HNO_3	9.32	$7.3x10^7$	35.50	24.44	30408	15.88	
	NaOH	4.16	$1.8x10^4$	14.53	33.97	11800	13.50	
4	HNO_3	7.98	$5.3x10^7$	34.67	24.82	28575	15.75	
	NaOH	3.85	$9.7x10^3$	13.01	34.47	9332	12.91	
5	HNO ₃	6.73	$4.2x10^7$	34.18	25.15	27227	15.60	
	NaOH	3.72	8.6×10^3	12.71	37.86	8917	12.30	



4-(2-Hydroxynaphthylideneamino)antipyrine (1)

$$\begin{array}{c} OH \\ \\ CH=N \\ \\ H_3C \\ \\ CH_3 \\ \end{array}$$

4-(2-Hydroxybenzylideneamino)antipyrine (2)

$$N = CH$$

$$N = CH$$

$$N = CH$$

3-(2-Hydroxynaphthylideneamino)-1,2,4-triazole (3)

$$N = CH$$

$$N = N = CH$$

$$N = CH$$

3-(2-Hydroxybenzylideneamino)-1,2,4-triazole (4)

$$N \longrightarrow N = CH \longrightarrow OH$$

3-(4-Hydroxybenzylideneamino)-1,2,4-triazole (5)

Structural formulae of investigated Schiff base compounds

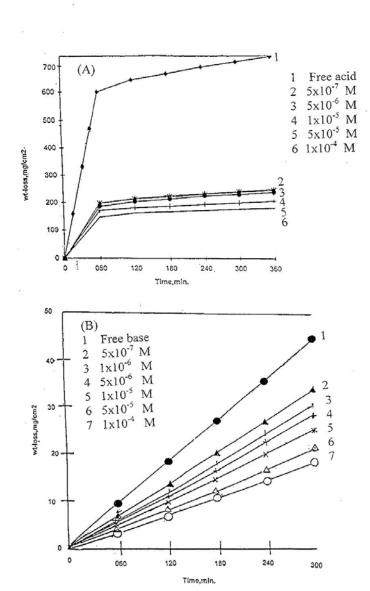


Fig. 1. Effect of 4-(2-hydroxynaphthylidene amino) antipyrine concentration on weight - loss - time curves for Fe in:

(A) 2M HNO₃ acid (B) 2M NaOH at 30 °C.



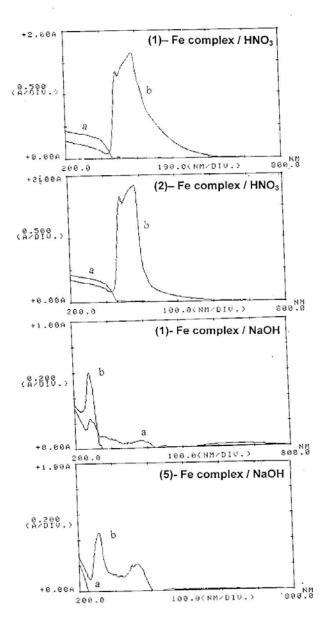


Fig. 2. Absorption spectra of Fe in 2M HNO₃ acid and 2M NaOH containing 10^{-4} M 1, 2 and 5 inhibitors: (a) no corrosion (b) corrosion at 30 °C

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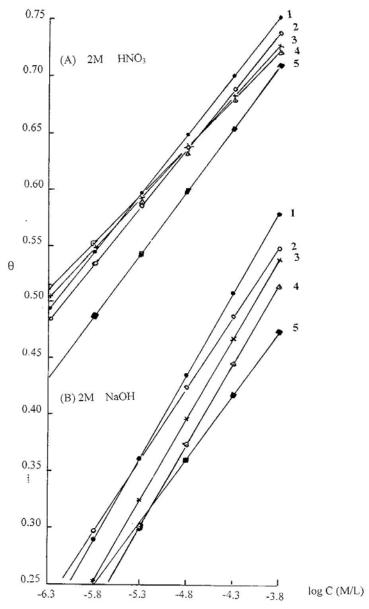


Fig. 3. Variation of iron surface coverage (θ) with the logarithmic concentration of different Schiff base additives



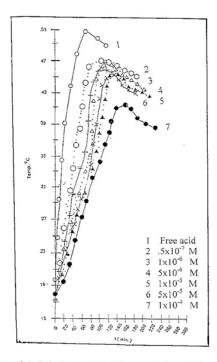


Fig. 4. Effect of 4-(2-hydroxynaphthylidene amino) antipyrine concentration on the thermometric curves for Fe in 2M HNO3 acid.

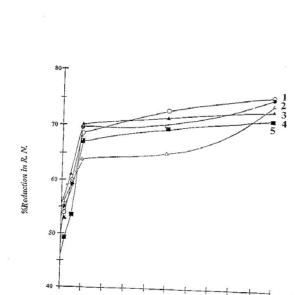


Fig. 5. Effect of concentration of Schiff base additives on % reduction in RN for Fe in 2M HNO $_3$ acid .

conc. (M/L)

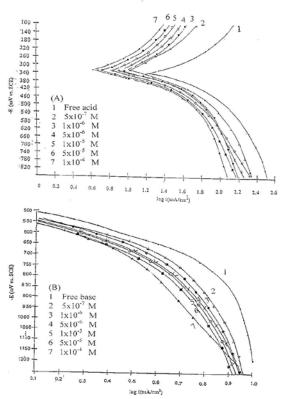


Fig. 6. Potentiostatic polarization curves of iron in the absence and in the presence of different concentrations of compound 1 in : (A) 2M HNO3 acid (B) 2M NaOH at 30 °C

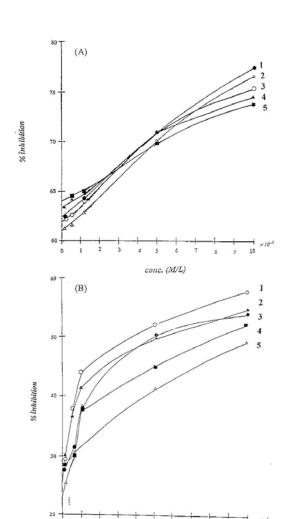
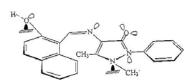


Fig. 7. Effect of concentration of Schiff base additives on the % inhibition for Fe in : (A) 2M HNO₃ (B) 2M NaOH



4-(2-Hydroxynaphthylideneamino)antipyrine (1)

4-(2-Hydroxybenzylideneamino)antipyrine (2)

3-(2-Hydroxynaphthylideneamino)-1,2,4-triazole (3)

3-(2-Hydroxybenzylideneamino)-1,2,4-triazole (4)

3-(4-Hydroxybenzylideneamino)-1,2,4-triazole (5)

Fig. 8. Skeletal representation of the proposed mode of adsorption of the investigated Schiff base additives