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ORIGINAL ARTICLE

Some Imidazoline Derivatives as Corrosion Inhibitors

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Abstract In this study, cationic surfactants having different alkyl chain lengths were prepared by amidation of lauric, myristic, palmitic, stearic, oleic acids with diethylene triamine. The products were quaternized using chloroacetic acid. The chemical structure of the prepared compounds was elucidated using different spectroscopic techniques. The critical micelle concentration (CMC) and the free energy of the micellization and adsorption of these compounds were determined by surface tension and conductivity measurements. The products were evaluated as surface-active agents as well as corrosion inhibitors for steel alloy in 1 M hydrochloric and sulfuric acid, the results indicate that these materials have a high efficiency as corrosion inhibitors and as surface active agents. These results were correlated with the chemical structure of the prepared compounds.

Keywords Cationic surfactants · Imidazoline derivatives · Corrosion inhibitors

Introduction

Corrosion of metals is one of the most important and challenging problems in industry. Therefore, the development of inhibiting films able to protect the underlying metal from corrosion has been the subject of numerous investigations [1, 2]. The corrosion inhibition efficiency of organic compounds is related to their adsorption properties. Adsorption depends on the nature and the state of the metal surface, on the type of corrosive medium and on the chemical structure of the inhibitor [3]. Studies report that the adsorption of the organic inhibitors mainly depends on some physicochemical properties of the molecule, related to its functional groups, to the possible steric effects and electronic density of donor atoms; adsorption is supposed also to depend on the possible interaction of *p*-orbitals of the inhibitor with *d*-orbitals of the surface atoms, which induce greater adsorption of the inhibitor molecules onto the surface of carbon steel, leading to the formation of a corrosion protecting film [4].

Different imidazoline derivatives are employed as steel corrosion inhibitors. Even though they have been specially employed in the oil industry, only recently have many studies been undertaken to understand how they work [5–8]. Ramachandran et al. [9], Wang et al. [10], and Cruz et al. [11] have published important papers concerning the use of imidazoline derivatives as corrosion inhibitors. These gtive rise to some key questions regarding the structure–performance relationships of imidazolines and corresponding amines, they are: (a) the role of the hydrocarbon chain relative to the imidazoline head group and the pendant amine group in film formation; (b) the thickness of the imidazoline film; (c) the stability of the imidazolines film; (d) the solution composition and (e) hydrolysis of imidazoline. Some authors have found that amide and imidazoline have the same efficiency as corrosion inhibitors [12].

The aim of this work was the preparation of some imidazoline derivatives and evaluating them as surface active agents and as corrosion inhibitors for low carbon steel in acid media and correlating these properties with their chemical structure.

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Materials and Experimental

Synthesis of the Imidazoline Derivatives

The imidazoline derivatives were synthesized by the reaction of lauric, myristic, palmitic, stearic and oleic acid, (0.1 mol) with diethylenetriamine (DETA), (0.1 mol) in xylene (100 mL) as the solvent [13]. The synthesis process was carried out in a three-necked flask with thermometer, stirrer and water splitter. The reaction mixture was refluxed at 140 °C for 2.5 h. When the splitting water was close to the theoretically generated amount, the solvent was removed by distillation, then the mixture was warmed up to 200 °C and reacted for 4 h at a constant temperature. When the splitting water was close to the amount demanded, the mixture was cooled down to 70 °C and 30% (wt) chloroacetic acid was slowly dripped into the reactor. Finally, it was refluxed at 80 °C for 2 h.

The diethylenetriamine (DETA), chloroacetic acid were of analytical grade, and the fatty acids and the xylene were of technical grade.

The fatty acids and DETA were reacted to form the corresponding amides which were reacted with chloroacetic acid to generate the corresponding quaternary ammonium salt, as shown in the following Scheme 1:

Confirmation of the Chemical Structure

The chemical structure of the synthesized compounds was confirmed by:

1. Elemental analyses which were carried out in the Micro Analytical Center, Cairo University.
2. FTIR spectra using an ATI Mattson Infinity series^{TM}, Bench top 961 spectrophotometer controlled by Win First^{TM} V2.01 software (Egyptian Petroleum Research Institute).
3. ^{1}H NMR which was measured in DMSO-d_6 by Spect Varian, GEMINI 200 (^{1}H 200 MHz). (Micro Analytical Center, Cairo University).

Surface Properties Measurements

The CMC for various imidazoline derivatives were determined from surface tension measurements of the prepared compounds solutions at 30 °C using a Du-Nouy tensiometer (Krüss K6).

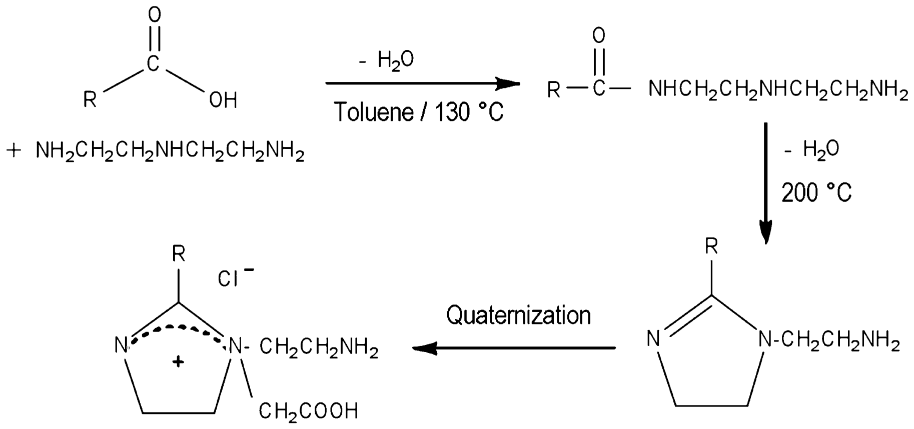
Corrosion Inhibition Measurements

Two techniques were used to determine the inhibition efficiency of the prepared compounds—the electro-polarization and weight loss.

The Electro-Polarization Technique

The electro-polarization technique was employed using a Radiometer analytical Potentiostat PGZ 3O1 (Volta Master 4). The concentration range of imidazoline derivatives employed was 10 to 200 ppm in 1 M HCl. The corrosion inhibition tests were carried out on electrodes cut from sheets of low carbon steel. Steel strips containing 0.09% P, 0.38% Si, 0.01% Al, 0.05% Mn, 0.21% C, 0.05% S and the remainding iron were used for the measurement of weight loss and electrochemical studies. The surface preparation of the specimens was carried out using emery paper, grades 600 and 1,200; they were degreased with ethanol and dried at room temperature before use. The solutions (1 M HCl) were prepared by dilution of analytical reagent grade 37% HCl with doubly distilled water.

Scheme 1 Reaction sequence



R = C11, C13, C15, C17and unsaturated C17

Weight Loss Technique

A weight-loss technique [ASTM G31-72 (Reapproved 2004)] was used to measure the inhibition efficiency of the prepared imidazoline amphiphiles for low carbon steel in 1 M HCl or 1 M H_2SO_4 solutions at 25 °C for 72 h. The dissolved oxygen range was 6–8 ppm. The low carbon steel specimen was machined into regular shapes of 55.8 cm^2 cross-sectional area. The specimens were sequentially abraded with different emery papers, degreased with acetone, washed with distilled water and dried. The corrosive solutions (1 M HCl or 1 M H2SO4) in the absence and presence of the inhibitors at concentrations ranging from 50 to 200 ppm were prepared from doubly distilled water the average weight loss of three coupons was recorded.

Results and Discussion

Structure Confirmation

The chemical structures of the prepared imidazoline derivatives were confirmed by different techniques.

Elemental Analysis

The chemical structure of the synthesized surfactants based on imidazoline as shown in Fig. 1 were confirmed using micro elemental analyses, Table 1.

FTIR Spectroscopy

The prepared imidazoline derivatives showed different FTIR bands as shown in Table 2, which indicates that they were prepared by the right method.

NMR Spectroscopy

As shown in Fig. 2, the ^{1}H-NMR spectra of OII compound show the following signals:

CH3 (0.85, 0.87 and 0.94 ppm, $(CH2)_n$ (1.26 ppm). CH_2 attached to the Q nitrogen of the imidazoline ring and the carbon of carboxylic acid (1.59), CH_2 attached to the nitrogen of NH_2 group (1.99). CH_2 attached to the carbon of the imidazoline ring (2.24 ppm), CH_2 attached to the nitrogen of the imidazoline ring (2.66, 2.75, 2.8 ppm), CH_2 attached to the carbon of the imidazoline ring 2.36, 2.37 and 2.39 ppm), CH_2 in the ring attached to the t nitrogen and CH_2 in the ring attached to Q nitrogen (3.61, 3.616 ppm), H of carboxylic group (5.33 ppm), H of NH_2 group (7.27 ppm).

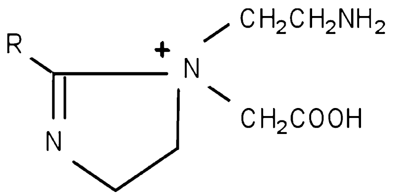


Fig. 1 The chemical structure of imidazoline derivatives (R = C11, C13, C15, C17 and unsaturated C17)

Table 1 The elemental analysis of the prepared imidazoline compounds

Elem. comp.	C (%)		N (%)		Cl (%)	
	Calc.	Found	Calc.	Found	Calc.	Found
LII	59.75	60.56	11.61	8.41	9.82	7.72
MII	60.22	58.69	1,078	7.28	9.11	7.44
PII	63.23	60.78	10.06	6.61	8.5	7.88
SII	64.64	62.21	9.42	6.88	7.97	7.52
OII	64.93	59.87	9.47	7.18	8.00	7.66

Table 2 The FTIR bands (cm^{-1}) of the function groups of prepared compounds

	–C–H CH_2, CH_3	C=N	Imidaz. ring	N–H bending	$(CH_2)^n$ skeletal
LII	2,922	1,631	1,601	1,549	716
MII	2,919	1,635	1,602	1,549	717
PII	2,915	1,636	1,602	1,548	711
SII	2,922	1,637	1,600	1,546	716
OII	2,916	1,640	1,602	1,540	716

Surface Parameters

Surface Tension (γ) and Critical Micelle Concentration

Figure 3 represents the variations of surface tension versus the solution concentration of the synthesized surfactants (LII, MII, PII, SII and OII) at 30 °C. It's clear that the surface tension decreased as the solution concentration increased. Surfactant molecules first adsorbed at the liquid/air interface until the surface of the solution was completely occupied. Then, the excess molecules tended to self-aggregate in the bulk to form micelles. From the intersection points of γ versus solution concentration curves, the critical micelle concentration (CMC) was determined, Table 3. The highest CMC value was observed for OII (2.5×10^{-4}), while, the lowest value was observed for MII 0.7×10^{-4} at 30 °C. This may be due to the difference in the alkyl chain lengths. It is clear that as the alkyl chain length increased the hydrophobicity of the molecule increased, this led to a decrease in the CMC value

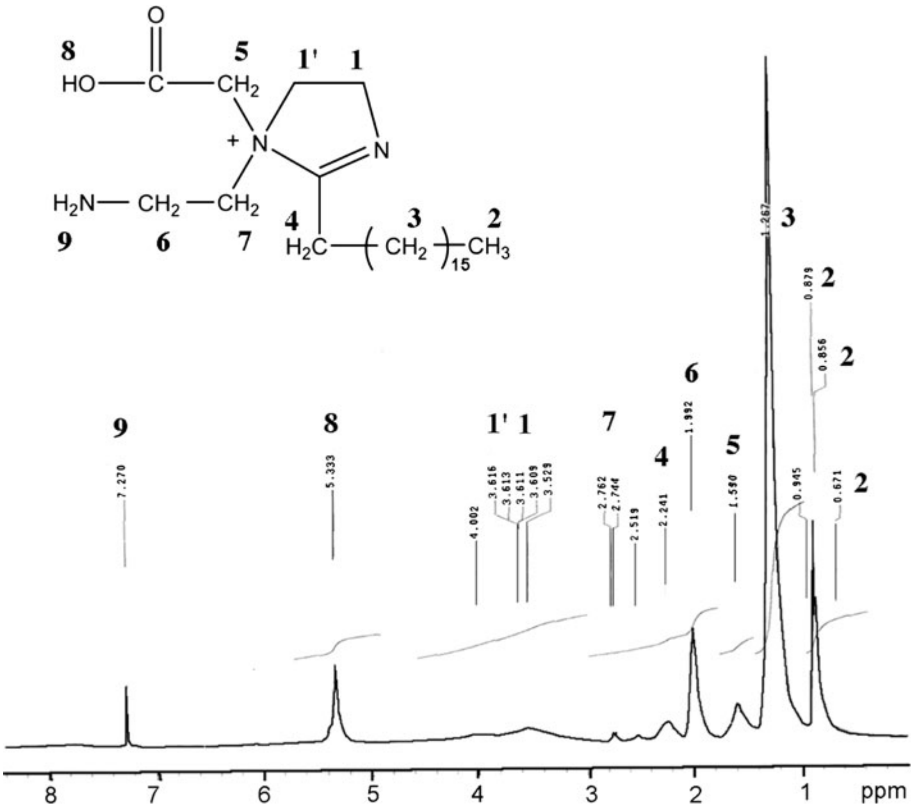


Fig. 2 The ^{1}H-NMR spectra of C18 unsaturated imidazoline derivatives

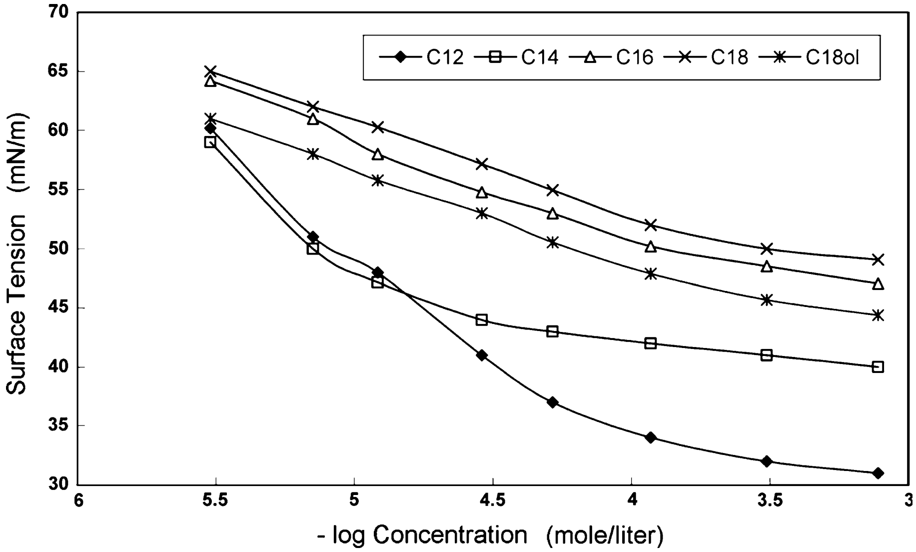


Fig. 3 Surface tension of aqueous solutions of different compounds versus concentration

of the molecule, which can be referred to the differences in their structures where, the OII compound behave nearly the same as PII.

Effectiveness (π_{cmc}), Maximum Surface Excess (Γ_{max}) and Minimum Surface Area (A_{min})

The difference between the surface tension value of the surfactant solution at its CMC and that of corresponding distilled water is defined as its effectiveness (π_{cmc}). The most efficient is the one that gives the greatest lowering in surface tension at the critical micelle concentration (CMC). According to the values of the effectiveness shown in Table 3, SII is found to be more efficient, it achieve the maximum reduction of the surface tension at its CMC.

The maximum surface excess is expressed as the concentration of surfactant molecules at the interface per unit area (Γ_{max}). Meanwhile the minimum surface area is

Table 3 Surface properties of prepared imidazoline surfactants

Comp.	S.T. (mN/m)	CMC × 10^{-4}	Π_{CMC} (mN/m)	Γ_{max} (mol $cm^{-2} \times 10^{10}$)	A_{min} (nm)	$-\Delta G^{\circ}_{mic}$	$-\Delta G^{\circ}_{ads}$
LII	43	1	38	1.42	1.12	23.2	25.8
MII	45	0.7	31	1.2	1.38	24.1	26.6
PII	54	1.7	23.5	0.755	2.2	21.8	24.9
SII	56	2.1	22	0.702	2.37	21.8	24.4
OII	53	2.5	27	0.77	2.15	20.9	24.4

defined as the area occupied by each molecule in nm^2 at the interface. Using the adsorption law of molecules at the interfaces, (Γ_{max}) values were calculated according to the following equation.

$$\Gamma_{max} = \left(\frac{d\gamma}{d \log C}\right) \Big/ (8.3 \times 10^7 \times RT) \qquad (1)$$

where dγ/d log C is the surface pressure. R, the universal gas constant and T, the absolute temperature. Regarding the results listed in Table 3 we can see that Γ_{max} was decreased by increasing the alkyl chain length so that the lowest value is for SII which may be attributed to the dissolution of molecules from the interface and their dissolution in the aqueous phase [14], the OII compound behaves nearly the same as PII.

The minimum surface area occupied by each surfactant molecules at the air\water interface (A_{min}) is calculated according to the following equation.

$$A_{min} = 1/N\,\Gamma_{max} \qquad (2)$$

where (A_{min}) values increase with the increase in the alkyl chain length, the OII compound behaves nearly the same as PII.

Standard Free Energies of Micellization and Adsorption $(-\Delta G^{\circ}_{mic}, -\Delta G^{\circ}_{ads})$

Understanding the process of micellization and adsorption are important for explaining the effects of structural and environmental factors on the values of the CMC and to predict the effect on the CMC of new structural and environmental variations, $-\Delta G^{\circ}_{mic}$ and $-\Delta G^{\circ}_{ads}$ have played an important role in such understanding. The $-\Delta G^{\circ}_{mic}$ and $-\Delta G^{\circ}_{ads}$ are given by:

$$-\Delta G^{\circ}_{mic} = RT \ln \text{CMC} \qquad (3)$$

$$\Delta G^{\circ}_{ads} = \Delta G^{\circ}_{mic} - 6.023 \times 10^{-1} \pi_{cmc} A_{min}. \qquad (4)$$

As shown in Table 3 the $-\Delta G^{\circ}_{mic}$ and $-\Delta G^{\circ}_{ads}$ values were always negative, indicating the spontaneity of these two processes. However, there was a greater increase in the negativity of $-\Delta G^{\circ}_{ads}$ than that of $-\Delta G^{\circ}_{mic}$, indicating the tendency of the molecules to be adsorbed at the interface water/air or water/metal [15].

Corrosion Inhibition

The inhibition efficiency (η) of the synthesized imidazoline can be calculated from the weight loss in the corrosive medium according to the following equation:

$$\eta\,(\%) = [(W_0 - W)/W_0] \times 100 \qquad (5)$$

The W and W_0 are the weight loss in the steel's coupon in the presence and absence of inhibitor, respectively. Tables 4, 5, and 6 represent the variation of inhibition efficiencies of the synthesized inhibitors in different acidic media for low carbon steel with a wide range of doses (from 50 to 200 ppm). It is clear that the inhibition efficiency towards corrosion process increases by increasing the inhibitor dose. The maximum corrosion inhibition was found at 200 ppm for all the synthesized inhibitors in 1 M HCl or H_2SO_4 solutions. Increasing the inhibition efficiencies with increasing the concentration of the targeted inhibitors is mainly due to the adsorption of those inhibitors on the metal surface. The adsorption

Table 4 The corrosion inhibition efficiency of the prepared imidazoline surfactant at different doses (weight loss method) in 1 M HCl

Dose (ppm)	Eff. (%)				
	LII	MII	PII	SII	OII
50	35	34	46	43	33
100	60	56	62	68	57
150	85	75	79	85	72
200	89	86	84	94	79

Table 5 The corrosion inhibition efficiency of the imidazoline surfactants (weight loss method) in 1 M H_2SO_4 solution

Dose (ppm)	Eff. (%)				
	LII	MII	PII	SII	OII
50	33	61	22	9	–
100	77	68	31	21	40
150	85	83	32	36	52
200	91	85	36	48	59

Table 6 The corrosion inhibition efficiency of compounds PII, SII and OII in 10% ethanol 1 M H_2SO_4 solution (weight loss method)

Dose (ppm)	Eff. (%)					
	PII	10Eth	SII	10Eth	OII	10Eth
50	22	30	9	40	–	30
100	31	50	21	53	40	72
150	32	73	36	75	52	83
200	36	75	48	82	59	81

mechanism of the inhibitor molecules at metal/solution interfaces is dependent on the chemical structure of the inhibitors and their response towards the environment and governed by one or more of the following factors:

1. Electrostatic attraction between the charged inhibitor molecules and the metal surface.
2. Interaction between the *p*-electrons in the inhibitor molecules and the metal.
3. Interaction between uncharged moieties in the inhibitor molecules and the metal surface.

The chemical structure of the synthesized inhibitors comprises unsaturation sites (conjugation within imidazoline rings) and heteroatom in the amine groups.

The hydrophobic part of the alkyl fatty acid acts as the uncharged moiety which forms the thin film preventing chloride ions from the metal surfaces.

The proposed mechanism of the steel dissolution in the acidic medium was described in the following equations [16]:

$$\mathrm{Fe} + \mathrm{Cl}^- \rightarrow (\mathrm{FeCl}^-)_{\mathrm{ads}}$$
$$(\mathrm{FeCl}^-)_{\mathrm{ads}} \rightarrow (\mathrm{FeCl})_{\mathrm{ads}} + e^-$$
$$(\mathrm{FeCl})_{\mathrm{ads}} \rightarrow \mathrm{FeCl}^+ + e^-$$
$$\mathrm{FeCl}^+ \rightarrow \mathrm{Fe}^{+2} + \mathrm{Cl}^-$$
$$\mathrm{Fe} + \mathrm{H}^+ \rightarrow (\mathrm{FeH}^+)_{\mathrm{ads}}$$
$$(\mathrm{FeH}^+)_{\mathrm{ads}} + e^- \rightarrow (\mathrm{FeH})_{\mathrm{ads}}$$
$$(\mathrm{FeH})_{\mathrm{ads}} + \mathrm{H}^+ + e^- \rightarrow \mathrm{Fe} + \mathrm{H}_2$$

Meanwhile, amine groups are protonated in the acidic medium forming the protonated amine (–N^+H_2C–) which adsorbed physically to the negative species formed during steel dissolution ($FeCl^-$). The same thing occurred with H_2SO_4.

The experimental results of the corrosion processes of the low carbon steel in the HCl media showed high inhibition efficiencies of the synthesized inhibitors, which were increased by increasing the addition dosage of the inhibitor. The efficiency of the LII was 35, 60, 85 and 89% at concentrations of 50, 100, 150 and 200 ppm. Whereas, the maximum inhibitions at concentration of 200 ppm in HCl media were 89, 86. 84, 94 and 79%, for LII, MII, PII, OII and SII, respectively.

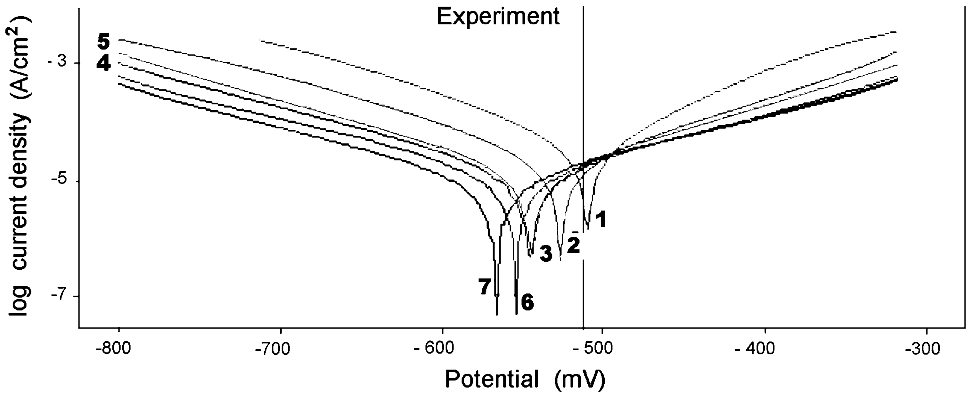
Analyzing the data of corrosion inhibition reveals that the inhibitors which have unsaturated alkyl chain imidazoline exhibit higher efficiencies than those obtained from a saturated one.

The above results were confirmed by the thermodynamic parameters of the micellization and adsorption process, in which it is found that the more negative $-\Delta G^{\circ}_{\mathrm{mic}}$ and $-\Delta G^{\circ}_{\mathrm{ads}}$ corresponding the more corrosion efficiency.

In the case of 1 M H_2SO_4 as shown in Tables 5 and 6 the efficiency of these compounds is dependent on their solubility, the shorter the alkyl chain is the more water soluble and the more efficient it is. So, for the long chain derivatives we added 10% (V/V) ethanol to the corrosive media in order to enhance the solubility. As shown in Table 6 addition of the ethanol enhanced the inhibition efficiency from 36 to 75 for PII, from 48 to 82 for SII and from 59 to 81 for OII.

Tafel Polarization

Figure 4 show a typical record of Tafel polarization measurements for low carbon steel in 1 M HCl in the absence



1Blank, 2 = 10 ppm, 3 = 20 ppm, 4 =30 ppm, 5 = 100 ppm, 6 = 50 ppm and 7 = 200ppm

Fig. 4 The polarization curves of compound PII at 1 M HCl

Table 7 The Tafel polarization parameter values for the corrosion of mild steel in 1 M hydrochloric acid containing different concentrations of the PII

Dose (ppm)	$-E_{corr}$ (mv)	i_{corr} (mA/cm$^{2)}$)	Rp (ohm cm$^{2)}$)	Ba (mv)	Bc (mv)	Corr rat (μm/y)	Eff. (%)
0.0	512.1	0.052	585.8	97.5	−119.9	607.7	0.0
10	529	0.0338	1,240	140.3	−142.0	394.8	35
20	542.8	0.014	1,790	131.6	−139.1	169.9	72
30	547.8	0.013	2,250	154.5	−142.2	157.4	74
50	556.6	0.011	2,610	153	−152.9	135	78
100	546.9	0.012	1,930	124.8	−127	148.8	76
200	568.9	0.008	3,160	148	−142	97.6	84

and presence of the compound PII. As shown in Table 7 the corrosion current density (i_{corr}) of bare low carbon steel electrode was 0.0520 mA cm^{-2} and has a corrosion rate of 607.7 μm/Y. It is clear that addition of PII to the corrosive media decreases the corrosion current density and corrosion rate. This decreasing is depending on the dosage of the PII.

It is clear that the addition of the PII to acid media affected both the cathodic and anodic parts of the curves. Therefore, these compounds behaved as mixed inhibitors. The corrosion potential shifted to negative direction more markedly. This shows that the effect of inhibitors on the cathodic reaction is more observable than on the anodic reaction. Increasing the concentration of the PII caused the corrosion potential to be more inert.

Table 7 lists the polarization parameters for corrosion of low carbon steel in the presence at different concentrations of the investigated compounds. The corrosion inhibition efficiency ($\eta_{P\%}$) is defined as:

$$\eta_{P\%} = \left(1 - \frac{i'_{corr}}{i_{corr}}\right) \times 100 \qquad (6)$$

The i'_{corr} and i_{corr} are the corrosion current densities of corrosive medium with and without inhibitor, respectively.

It is clear that corrosion current density decreases with increasing the concentration of the inhibitors. The depolarization of both anodic and cathodic branches after addition of the inhibitors indicates that not only was dissolution of the metal, but the evolution of hydrogen and oxygen (we did not try to purge the dissolved oxygen out of the cell) was also suppressed. Therefore, the imidazoline derivatives cause a decrease in the corrosion rate of steel in acid media by influencing both the anodic and cathodic reactions. The maximum efficiency was 84% for a concentration of 200 ppm of PII.

Conclusions

The results indicate that all the prepared compounds have good surfactant properties and exhibit good inhibition properties against the corrosion of low carbon steel in 1 M HCl and H_2SO_4 solutions at room temperature, and the inhibition efficiency is increased by increasing the concentration of the inhibitors. However, the corrosion inhibition of the prepared compounds depends mainly on their solubility.

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