

Ni²⁺ cation and imidazole as corrosion inhibitors for carbon steel in sulfuric acid solutions

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Received: 3 January 2010 / Accepted: 26 August 2010 / Published online: 23 October 2010
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Abstract The effect of Ni²⁺ cation, imidazole, and mixtures of them on the corrosion behavior of carbon steel in 0.5 M H₂SO₄ solution was studied by using galvanostatic polarization, potentiodynamic anodic polarization, and weight-loss techniques. Ni²⁺ cation, imidazole, and mixtures of them provide a good protection to carbon steel against pitting corrosion in chloride-containing solutions. The inhibiting solutions were analyzed by using UV–Vis spectrophotometry. The inhibition was explained on the basis of formation of a complex between the two components. The inhibition mechanism was discussed in terms of the results derived from corrosion and UV–Vis spectrophotometric measurements as well as conductometric investigations.

Keywords Ni²⁺ cation · Imidazole · Carbon steel · Inhibition · Corrosion inhibitor

Introduction

Corrosion is a fundamental process playing an important role in economics and safety, particularly for metals and alloys. Steel has found wide application in a broad spectrum of industries and machinery, despite its tendency to corrode. The corrosion of steel is a fundamental academic and industrial concern that has received a considerable amount of attention [1]. Using inhibitors is an important method of protecting materials against deterioration due to corrosion, especially in acidic media [2]. Selection of an

inhibitor for a particular system is a difficult task due to the specificity and selectivity of both the inhibitor and the wide varieties of corrosion systems [2]. The influence of heterocyclic compounds containing nitrogen on the corrosion of steel in acidic solutions has been investigated by some workers [3, 4]. The effect of various metal cations on the corrosion of metals has also been reported [5–9]. Several mechanisms have been proposed to explain both the accelerating and inhibiting effect of metal cations. It was found [10] that the accelerating effect is due to the formation of soluble compounds, while the inhibiting effect was attributed to the formation of insoluble compounds adhering to the metal surface [11, 12]. Recently, it was found that the formation of a donor–acceptor surface complex between a free or π electron of an organic inhibitor and a vacant d orbital of a metal is responsible for the inhibition of the corrosion process [13].

The aim of this work was to study the inhibiting action of Ni²⁺ cation and imidazole as well as a mixture of them on the corrosion behavior of carbon steel (type L-52) used in petrol pipelines (Egypt) in 0.5 M H₂SO₄ solutions using galvanostatic polarization, potentiodynamic polarization, and weight-loss measurements, as well as the ability of these compounds to provide a protection against pitting corrosion.

Results and discussion

Galvanostatic polarization measurements

Figure 1 shows the anodic and cathodic polarization curves of carbon steel in 0.5 M H₂SO₄ in the absence and presence of different concentrations of Ni²⁺ cation as an example. However, similar curves were obtained for

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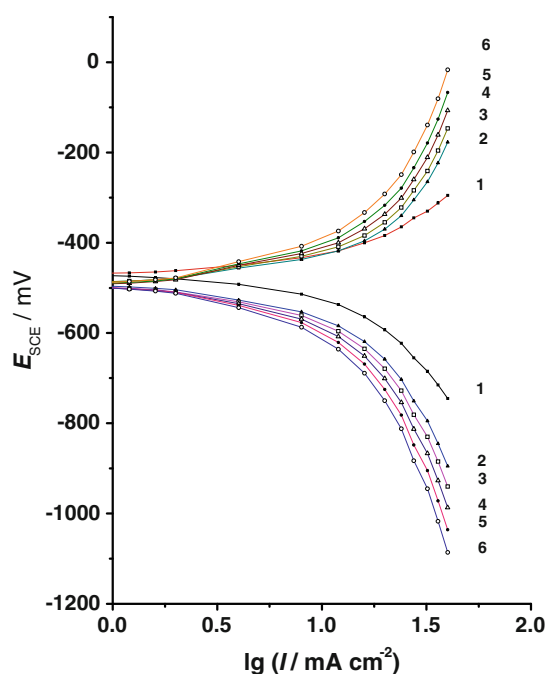


Fig. 1 Cathodic and anodic polarization curves of carbon steel in 0.5 M H_2SO_4 solution with Ni^{2+} cation: 1 blank, 2 5.0×10^{-4} , 3 1.0×10^{-3} , 4 2.0×10^{-3} , 5 3.0×10^{-3} , 6 5.0×10^{-3} M

different concentrations of imidazole and mixtures of different concentrations of Ni^{2+} cation and imidazole (not shown).

The extrapolation method for the polarization curves was applied and the data for corrosion potential (E_{corr}), corrosion

current density (I_{corr}), and Tafel slopes (b_c and b_a) are shown in Table 1.

The results in Fig. 1 and Table 1 illustrate that as the concentration of additives increases there is a shift in the corrosion current density (I_{corr}) to lower values relative to the blank. It is also clear that in the presence of Ni^{2+} cation the corrosion potential (E_{corr}) is shifted to the less negative direction (noble direction), while in the presence of imidazole, E_{corr} was shifted in the more negative direction. On the other hand, in the presence of Ni^{2+} cation + imidazole mixtures, E_{corr} was nearly the same as in the case of blank. These results indicate that the additives inhibit the corrosion of carbon steel electrodes in 0.5 M H_2SO_4 solutions and behave as a mixed inhibitor type. This effect is predominately anodic, cathodic, and ambiodic (i.e., suppressing reactions at both anodic and cathodic sites) for Ni^{2+} cation, imidazole, and a mixture of them, respectively.

The value of I_{corr} decreases with increasing inhibitor concentration and the Tafel slopes remain almost unchanged, indicating that the presence of these compounds in the solution has no effect on the mechanism of the dissolution process of the metal [15, 16] and the adsorbed molecules mechanically screen the coated part of the electrode and therefore protect it from the action of the corrosion medium.

The reaction mechanism of the corrosion process in 0.5 M H_2SO_4 solution free from inhibitor is different from that in the presence of additives. On inspection of data given in Table 1, it is clear that in the presence of inhibitors the value of b_c is more than in free acid solution. This behavior is due to the decrease of the cathodic transfer

Table 1 Corrosion parameters for carbon steel in 0.5 M H_2SO_4 solutions in the absence and presence of different concentrations of inhibitors at 30 °C

Inhibitor system	Conc. (M)	b_a (mV dec ⁻¹)	b_c (mV dec ⁻¹)	E_{corr} (mV _{SCE})	I_{corr} (mA cm ⁻²)	% $I_{\text{E}_{\text{corr}}}$ (%)	% I_{E_w} (%)
–	0.0	62.5	85	–452	0.625	–	–
Ni^{2+} cation	5.0×10^{-4}	133	132	–491	0.430	31	34.5
	1.0×10^{-3}	136	150	–490	0.37	40.8	43.6
	2.0×10^{-3}	153	160	–488	0.325	48	52.72
	3.0×10^{-3}	160	168	–486	0.275	56	58.18
	5.0×10^{-3}	173	180	–483	0.210	66	70
Imidazole	5.0×10^{-4}	111	136	–499	0.45	28	29
	1.0×10^{-3}	123	151	–500	0.362	42	43.63
	2.0×10^{-3}	133	178	–502	0.30	52	52.72
	3.0×10^{-3}	151	191	–505	0.223	64	65.45
	5.0×10^{-3}	186	258	–504	0.113	81.95	82.72
Imidazole + Ni^{2+} cation	$5.0 \times 10^{-4} + 5.0 \times 10^{-4}$	198	269	–515	0.099	84.16	83.63
	$1.0 \times 10^{-3} + 5.0 \times 10^{-4}$	211	281	–517	0.081	87.04	87.27
	$2.0 \times 10^{-3} + 5.0 \times 10^{-4}$	198	271	–513	0.089	85.8	85.45
	$5.0 \times 10^{-4} + 1.0 \times 10^{-3}$	183	261	–513	0.115	81.6	81.81
	$5.0 \times 10^{-4} + 2.0 \times 10^{-3}$	185	258	–514	0.116	81.12	80.90

coefficient, which can be ascribed to the thickening of the electric double layer or the formation of multi-molecular layers on the metal surface [17]. Also, the anodic Tafel slope (b_a) in the inhibited solution is higher than that obtained in the acid inhibitor-free solutions. This increase in anodic Tafel slope suggests a mode of inhibition involving an interposition of the additives into the charge-transfer process for the anodic reaction [18], which may lead to a morphological change of the electrode surface brought about by the anodic dissolution [19].

The percentage inhibition efficiency ($\%IE_{\text{corr}}$) was calculated and listed in Table 1 using the following equation:

$$\%IE_{\text{corr}} = \left(1 - \frac{I_{\text{corr}}}{I_{\text{corr}}^0}\right) \times 100 \quad (1)$$

where I_{corr}^0 and I_{corr} are the uninhibited and inhibited corrosion current densities, respectively. The corrosion current densities are calculated from the intersection parts of the cathodic curves with stationary corrosion potentials (E_{corr}). The obtained data (Table 1) for $\%IE_{\text{corr}}$ values reveal that the inhibition efficiency decreases in the following order: Ni²⁺ cation + imidazole mixture > imidazole > Ni²⁺ cation.

Potentiodynamic polarization measurements

Figure 2 represents the effect of increasing concentrations of imidazole on the potentiodynamic anodic polarization curves of carbon steel in 0.5 M H₂SO₄ solution at a scan rate of 50 mV s⁻¹ as an example. However, similar curves were obtained for different concentrations of Ni²⁺ cation

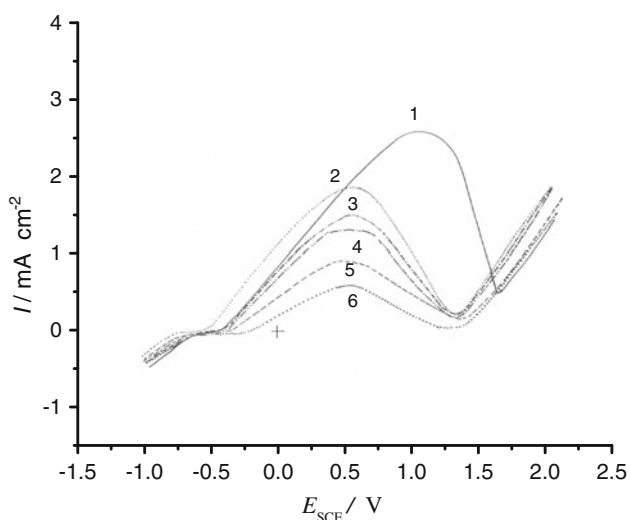
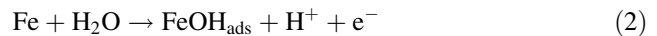


Fig. 2 Potentiodynamic anodic polarization curves of carbon steel in 0.5 M H₂SO₄ solution with imidazole: 1 blank, 2 5.0×10^{-4} , 3 1.0×10^{-3} , 4 2.0×10^{-3} , 5 3.0×10^{-3} , 6 5.0×10^{-3} M

and mixtures of different concentrations of Ni²⁺ cation and imidazole (not shown).

On inspection of the curves of Fig. 2, there is only one anodic peak noted. This peak may correspond to the active dissolution of Fe to Fe²⁺ ion according to the following mechanism:



As the concentration of the additives increases the corrosion current peak I_p decreases and the value of peak potential E_p is shifted to more positive values, which suggests the additives exert an inhibiting effect. The percentage inhibition efficiency ($\%IE_{\text{(add)}}$) was calculated by using the following equation:

$$\%IE_{\text{(add)}} = \left[1 - \frac{I_{\text{p(add)}}}{I_{\text{p(free)}}}\right] \times 100 \quad (5)$$

where $I_{\text{p(add)}}$ and $I_{\text{p(free)}}$ are the peak current densities in the presence and absence of inhibitors. The values of I_p , E_p , and $\%IE_{\text{(add)}}$ are listed in Table 2.

It is obvious from Table 2 that the inhibition efficiency decreases in the following order: Ni²⁺ + imidazole mixture > imidazole > Ni²⁺.

Weight-loss measurements

Figure 3 represents the effect of increasing concentration of mixtures of Ni²⁺ cation and imidazole on the weight loss of carbon steel in 0.5 M H₂SO₄ solutions as an example. However, similar curves were obtained for different concentrations of imidazole and different concentrations of Ni²⁺ cation (not shown).

It is obvious that the weight loss of the inhibitor varied linearly with time and much lower than that obtained in the blank solution. The linearity obtained indicates the absence of an insoluble film during corrosion and that the inhibitors were first adsorbed onto the metal surface and thereafter impede the corrosion process.

The percentage inhibition efficiency ($\%IE_w$) was calculated from the weight-loss measurements and listed in Table 1 using the following relation:

$$\%IE_w = [1 - W/W_0] \times 100 \quad (6)$$

where W_0 and W are the weight loss per unit area in the absence and presence of inhibitor, respectively. Inspection of Table 1 reveals that the percentage efficiency ($\%IE_w$) increases with increasing the additive concentration. Moreover, the percentage inhibition efficiency ($\%IE_w$) is found to decrease in the following order: Ni²⁺ cation + imidazole mixture > imidazole > Ni²⁺ cation.

Table 2 Corrosion parameters for carbon steel in 0.5 M H₂SO₄ solutions without and with different concentrations of inhibitors at 30 °C

Inhibitor system	Conc. (M)	I_p (mA cm ⁻²)	E_p (V _{SCE})	%IE _(add) (%)
–	0.0	2.40	1.049	–
Ni ²⁺ cation	5.0×10^{-4}	1.71	0.522	28.75
	1.0×10^{-3}	1.50	0.593	37.50
	2.0×10^{-3}	1.35	0.596	43.75
	3.0×10^{-3}	1.14	0.608	52.50
	5.0×10^{-3}	0.90	0.60	62.50
Imidazole	5.0×10^{-4}	1.74	0.47	27.50
	1.0×10^{-3}	1.41	0.467	41.25
	2.0×10^{-3}	1.20	0.469	50.00
	3.0×10^{-3}	0.84	0.471	65.00
	5.0×10^{-3}	0.54	0.472	77.50
Imidazole + Ni ²⁺ cation	$5.0 \times 10^{-4} + 5.0 \times 10^{-4}$	1.80	0.598	77.10
	$1.0 \times 10^{-3} + 5.0 \times 10^{-4}$	1.50	0.60	80.90
	$2.0 \times 10^{-3} + 5.0 \times 10^{-4}$	1.70	0.61	78.80
	$5.0 \times 10^{-4} + 1.0 \times 10^{-3}$	2.10	0.582	73.80
	$5.0 \times 10^{-4} + 2.0 \times 10^{-3}$	2.20	0.584	72.00

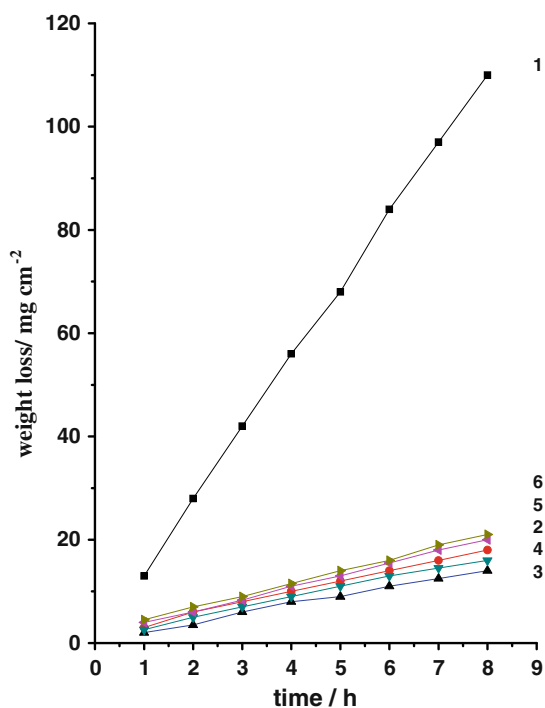


Fig. 3 Weight loss as a function of time of carbon steel in 0.5 M H₂SO₄ solution without and with imidazole + Ni²⁺ cation: 1 blank, 2 $5.0 \times 10^{-4} + 5.0 \times 10^{-4}$, 3 $1.0 \times 10^{-3} + 5.0 \times 10^{-4}$, 4 $2.0 \times 10^{-3} + 5.0 \times 10^{-4}$, 5 $5.0 \times 10^{-4} + 1.0 \times 10^{-3}$, 6 $5.0 \times 10^{-4} + 2.0 \times 10^{-3}$ M

Pitting corrosion

Potentiodynamic anodic polarization curves were traced for carbon steel in solutions of 0.5 M H₂SO₄ + 0.5 M NaCl (as a pitting corrosion agent) devoid of and

containing different concentrations of Ni²⁺ cation, imidazole, and mixtures of them at a scanning rate of 1 mV s⁻¹.

The potential was swept from negative potential towards anodic direction up to the pitting potential, and no anodic oxidation peaks were observed in all anodic scans. The pitting potential (E_{pitt}) was taken as the potential at which the current flowing along the passive film increases suddenly to higher values, denoting the destruction of the passive film and initiation of visible pits. The effect of addition of increasing concentration of Ni²⁺ cation, imidazole, and mixtures of them on the values of the pitting potential is presented in Fig. 4.

This figure represents the relationship between E_{pitt} and logarithmic molar concentrations of additives. It is clear from this figure that as the concentration of these additives increases the pitting potential is shifted to more positive values according to the following equation:

$$E_{pitt} = a + b \lg C_{add} \quad (7)$$

where a and b are constants depending on the type of additives used and the nature of the metal. The positive shift of E_{pitt} indicates the increased resistance to pitting attack.

At one and the same inhibitor concentration, the marked shift of potential in the positive (noble) direction decreased in the following sequence: Ni²⁺ cation + imidazole > imidazole > Ni²⁺ cation.

The different techniques used in this study gave the same order of inhibition efficiency but yielded different absolute values, probably due to the different experimental conditions.

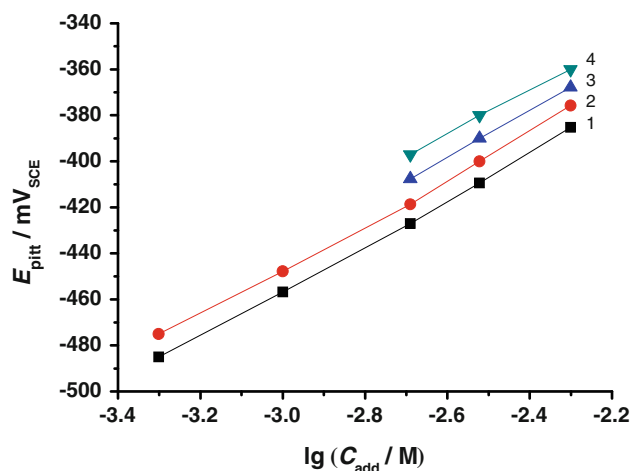


Fig. 4 Relationship between pitting potential of carbon steel and logarithm of the concentration of 1 Ni²⁺, 2 imidazole, 3 imidazole + Ni²⁺, and 4 Ni²⁺ + imidazole mixtures

Qualitative analysis of inhibited media

In order to clarify the inhibition mechanism of carbon steel corrosion in 0.5 M H₂SO₄ solutions containing additives, UV–Vis spectroscopic investigations on the testing solutions before and after some corrosion experiments were performed. Also, conductometric titrations of Ni²⁺ cation using imidazole as a titrant were performed.

UV–Vis spectroscopic investigation

Figure 5a represents the spectrum of Ni²⁺ cation (curve 1), imidazole (curve 2), and a mixture of them (curve 3) before anodic and cathodic polarization measurements.

It is obvious that the spectrum of imidazole shows a shoulder band at 215 nm, corresponding to the π – π^* of the aromatic system. This band is shifted to a higher value (230 nm) in the case of the Ni²⁺ cation + imidazole mixture. The intensity of the band obtained in the case of the Ni²⁺ cation + imidazole mixture is lower than that obtained for imidazole in 0.5 M H₂SO₄ solutions.

For otherwise identical conditions, the spectra of both imidazole and Ni²⁺ cation + imidazole mixture after polarization measurements (Fig. 5b, curves 2 and 3) show the same absorption band values as obtained from those before polarization measurements but at lower intensities. This means that imidazole and the Ni²⁺ cation + imidazole complex formed in 0.5 M H₂SO₄ solution are adsorbed on the carbon steel surface.

Conductometric titration of Ni²⁺ cation using imidazole

The conductometric titration curve obtained for Ni²⁺ cation with imidazole as a titrant is shown in Fig. 6. The

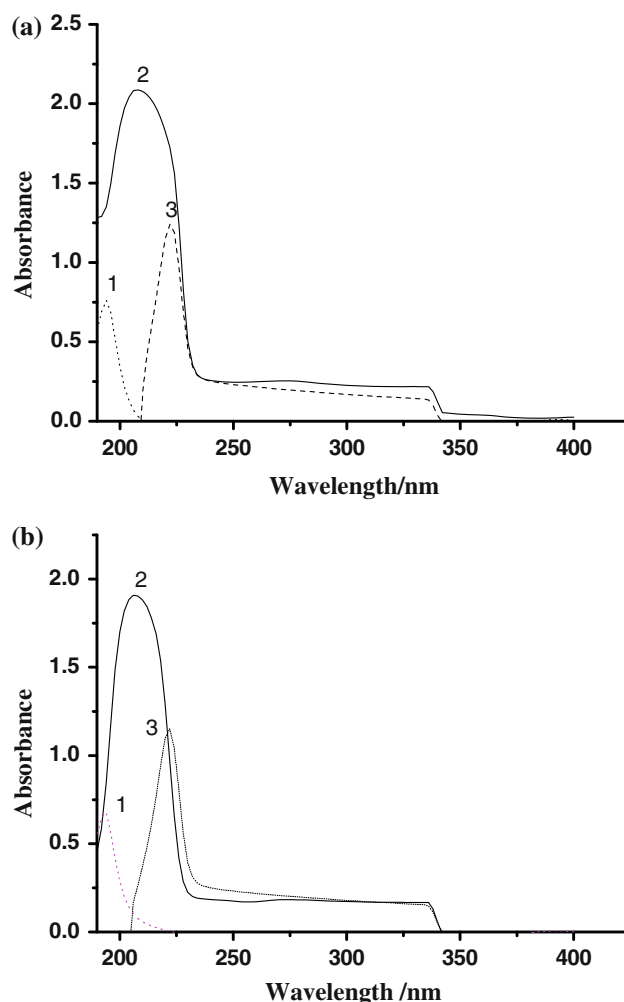


Fig. 5 UV spectra of the additives used as inhibitors for carbon steel corrosion in 0.5 M H₂SO₄ solutions **a** before and **b** after measurements: 1 5.0 × 10^{−4} M Ni²⁺ cation, 2 5.0 × 10^{−4} M imidazole, 3 5.0 × 10^{−4} M Ni²⁺ cation + 5.0 × 10^{−4} M imidazole

conductometric titration of 5.0 × 10^{−4} M Ni²⁺ cation with 5.0 × 10^{−3} M imidazole shows an obvious maximum in the conductometric curve, which corresponds to a molar ratio of 1.0 (metal cation):2.0 (additive). This observation gives a strong indication for the formation of a complex compound between the two additives in a molar ratio of 1.0 (Ni²⁺ cation):2.0 (imidazole).

The shape of the conductometric curve depends on the concentrations of all species present during the titration process as well as other factors such as viscosity, dielectric constant, solvation, complexation, and proton transfer [20]. On the other hand and as given below, imidazole is an organic compound with two nitrogen atoms in the heterocyclic ring [21]. One of them is of pyrrole type, and the other a pyridine-like nitrogen atom. Consequently, imidazole is basic, and can become protonated in acid solution in the manner shown in Fig. 7.

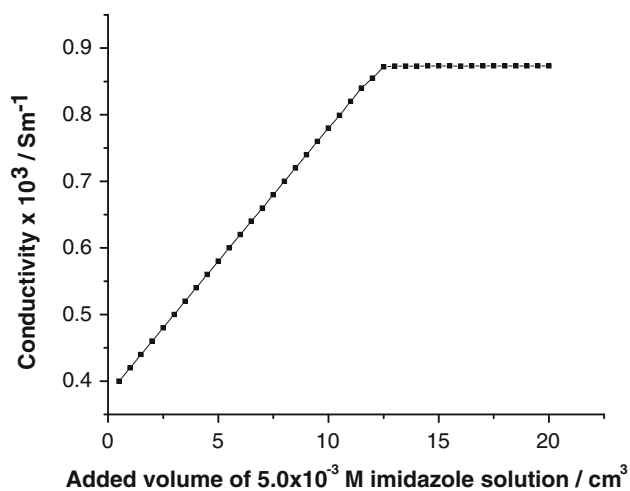


Fig. 6 Conductometric titration curve for 50 cm³ 5.0 × 10⁻⁴ M Ni²⁺ cation titrated with different volumes of 5.0 × 10⁻³ M imidazole solution

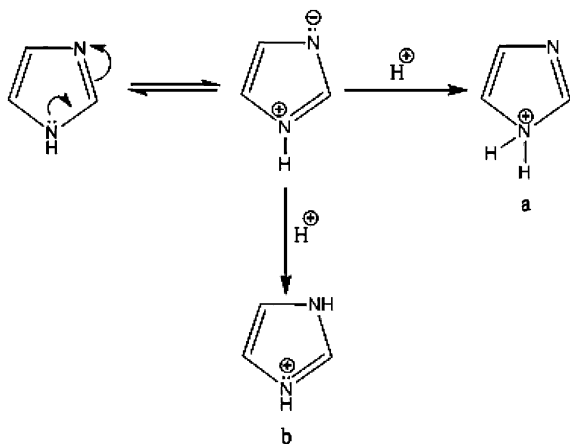


Fig. 7 Protonation of imidazole in acidic medium

In the present work, the conductance of the titrated solution is mainly due to Ni²⁺ cation and partially to the hydrogen ions resulting from the dissociation of the protonated imidazole in the acidic medium. After the complete formation of 1.0 (Ni²⁺ cation):2.0 (imidazole) complex, the conductance values remain constant (Fig. 6). This indicates that the increase in the conductance with increasing the amount of titrant is due to the increase of the mobility of the formed complex and also to the increase of ions in the medium [22]. After the complex is completely formed with the ratio 1:2 (Ni²⁺ to imidazole), the mobility of that complex becomes less than that of the ions present in the solution so the conductance has no change with increasing the amount of titrant at 25 °C. Consequently it can be concluded that the Ni²⁺ cation–imidazole complex is of the following type as shown in Fig. 8.

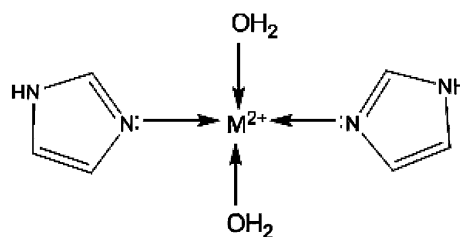


Fig. 8 1:2 complex formation between metal cation and imidazole

It is shown that the protonated N atom of the imidazole molecules is assumed to participate in the complex formation with Ni²⁺ cations. The participation of the N atom (i.e., the nitrogen of pyridine type) of some organic compounds like imidazole and phenylazo-hydroxyquinoline in the complex formation with Ni²⁺ cation in H₂SO₄ solutions is reflected in many publications [23, 24].

Inhibition mechanism

The results discussed above indicate that the influence of the additives as inhibitors for carbon steel corrosion in 0.5 M H₂SO₄ solutions depends on the nature and size of the additives. The inhibition action of Ni²⁺ cation is ascribed to the plating of the corresponding metal on the carbon steel surface [25], which forms a protective layer with consequently more complete anodic passivation of the anodic sections of the substrate.

The inhibition of carbon steel corrosion by imidazole is attributed to the adsorption of the inhibitor molecules on the metal surface. As shown above, the imidazole molecule shows three different anchoring sites suitable for surface adsorption: the nitrogen atom with its *sp*² lone pair, the active hydrogen atom attached to the nitrogen atom, and the π bond of the aromatic ring. As previously stated [25], most of the organic compounds containing nitrogen atoms have an electron-donating ability and could be adsorbed via electron donation from the N atom and by bridging with the active hydrogen atom attached to the N atom. On the other hand, the π bond of the aromatic ring is considered as the center for the adsorption process [26, 27], in which the main adsorption mode of some organic compounds is attributed to the interaction of the π electrons with surface atoms of the adsorbent.

The adsorption of the imidazole molecule through its N⁺–H protonated center onto the carbon steel surface is excluded because the carbon steel surface is assumed to be positively charged in sulfuric acid solution [26], in which positively charged centers of the organic molecules are not attached to the same positively charged metal surface. Also, the fact that participation of the active hydrogen atom attached to the N atom in the inhibition processes is excluded could be due to its ability to form an

intermolecular hydrogen bonding with another imidazole molecule and/or with H₂O from the medium [21]. Consequently, it can be concluded that the adsorption of the imidazole molecules onto the carbon steel surface arises via one anchoring site, that is the π bond of the aromatic ring. Thus, it is reasonable to assume that the imidazole molecule is adsorbed onto the carbon steel surface in a flat orientation through a monodentate form as shown in the model in Fig. 9a.

The flat model for adsorption is stable and preferred [28] since the covered fraction surface is high, even at low inhibitor concentration.

In the case of addition of Ni²⁺ cation + imidazole mixture, the results demonstrated the complex formation between the two components, which was much more effective than the inhibiting action of each additive separately (Table 1). The conductometric studies revealed the participation of two imidazole molecules in the complex formation with Ni²⁺ cation. This finding suggests that the molecular size of the complex formed and consequently the number of adsorption centers play an important role in the enhancement of the protection of carbon steel against corrosion. Thus, it is reasonable to suggest that the adsorption of the complex molecule onto the carbon steel surface occurs in a flat orientation through the π bond of the aromatic rings in a bidentate form (Fig. 9b), which covers a large surface area as compared to the case of imidazole additive.

It is of interest to remark that when the Ni²⁺ cation + imidazole mixture is added to the corrosive medium in higher stoichiometric amounts of Ni²⁺ cation than required to form a complex with imidazole in a ratio of 1.0 (Ni²⁺ cation):2.0 (imidazole), the value of percentage inhibition efficiency is decreased (Table 1). The same

results are obtained from the weight-loss experiments (Fig. 3c; Table 1). This behavior could be explained on the basis that although the presence of a high stoichiometric amount of Ni²⁺ cation permits a formation of the complex compound in a 1 (Ni²⁺ cation):2 (imidazole) ratio, some of the Ni²⁺ cations are still free in the acid solution. So it is expected that there is a competition between Ni²⁺ cations and the complex ions to form a protective layer on the carbon steel surface. This competition may slow down the rate of film formation and consequently reduce the percentage inhibition efficiency (%P). However, the experimental results (Table 1) revealed that, although the percentage inhibition efficiency is decreased when the concentration of Ni²⁺ cation is higher than that of imidazole, its value is still high as compared with those obtained in the case of using different concentrations of Ni²⁺ cation separately. This finding suggests that the carbon steel surface is protected by layers most probably composed of adsorbed Ni²⁺ cation–imidazole complex ions. This assumption is further confirmed from the near constancy of the E_{corr} value obtained in the case of adding Ni²⁺ cation + imidazole mixtures with various ratios of the two components.

Conclusion

The corrosion inhibition of carbon steel in 0.5 M H₂SO₄ solution containing Ni²⁺ cation and imidazole separately and together was studied by using galvanostatic polarization, potentiodynamic anodic polarization, and weight-loss techniques. All additives provide adequate inhibition for carbon steel corrosion in sulfuric acid solution. The results revealed a complex formation between Ni²⁺ cation and imidazole, which has a much larger inhibition effect than each additive separately. The inhibition action of Ni²⁺ cation is ascribed to the plating of the corresponding metal on the carbon steel surface. In contrast, the corrosion inhibition by imidazole as well as its complex compound with Ni²⁺ cation is attributed to the adsorption of the additives onto the carbon steel surface. The adsorption is assumed to arise from the π bond of the imidazole rings and the carbon steel surface. Ni²⁺ cation, imidazole, and mixtures of them provide protection against pitting corrosion of carbon steel in the presence of chloride ions.

Experimental

Materials

The carbon steel specimen (L-52) used for this study has the following composition: $w_{\text{C}} = 0.26\%$, $w_{\text{Mn}} = 1.35\%$,

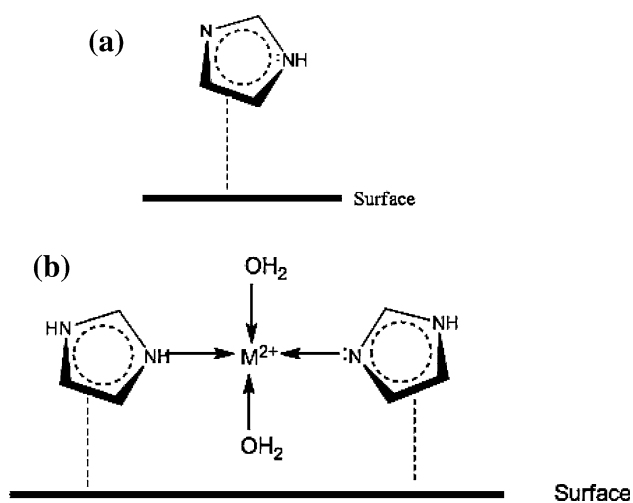


Fig. 9 a Schematic representation of the mode of adsorption of imidazole. b Schematic representation of the mode of adsorption of Ni²⁺ cation–imidazole

$w_P = 0.04\%$, $w_S = 0.05\%$, $w_{Nb} = 0.005\%$, $w_V = 0.02\%$, $w_{Ti} = 0.03\%$, and Fe to balance. The galvanostatic polarization measurements were performed by using specimens in the form of rods of 1-cm² exposed surface area as a working electrode. For weight-loss tests, small rectangular coupons of the dimensions 1.0 × 2.0 × 0.3 cm³ were used.

The testing media were aqueous aerated 0.5 M H₂SO₄ solutions without and with different concentrations of NiSO₄ (as a source for Ni²⁺ cations), imidazole, and NiSO₄ + imidazole mixtures, respectively. All chemicals used in this study were analytical grade (Aldrich) and the desired temperature of each experiment was adjusted to ±1 °C using an air thermostat.

Apparatus

The weight-loss measurements were carried out in large test tubes (20 cm × 2.5-cm diameter) suspended in a thermostated water bath. Each tube was open to air.

The galvanostatic cathodic and anodic polarization measurements were carried out using a three-compartment glass cell and EG&G model 363 potentiostat/galvanostat corrosion measurement system. A platinum electrode was used as a counter electrode (separated from the cell solution by a sintered glass frit) and a saturated calomel electrode (inside a Luggin's probe) as a reference electrode.

The potentiodynamic anodic polarization measurements were performed by using a Wenking potentiostat, type POS 73 and a X-Y recorder, type advanced, HR 2000.

The UV-Vis spectrophotometric experiments were carried out using a JASCO UV-Vis 530 spectrophotometer and a 10-mm matched silica cell.

The conductance measurements were carried out using YSI model 32 conductance meter of cell constant equal to 1.6.

Procedures

Galvanostatic polarization

The working electrode was polished and pre-polarized prior to recording the cathodic and anodic polarization curves at each concentration of the tested solution. All the polarized curves were obtained by using the direct technique at regular intervals. The duration of potential stabilization at each current density value was between 3 and 5 min.

Potentiodynamic anodic polarization

The working electrode is the same as that used before in galvanostatic polarization techniques; the electrode surface was polished and left for 10 min to achieve a pre-steady

state prior to scanning at a rate of 100 mV s⁻¹ at each concentration of the tested solution.

Weight-loss measurements

The coupons were successively abraded with SiC paper to a final finish using 1,200 grade paper. Before immersion in the test solution (50 cm³), the dimensions of each coupon were ascertained. They were then degreased in AR grade acetone, etched in HCl for 30 s, washed with doubly distilled water, followed by acetone, dried, and weighed. The cleaned carbon steel coupons were weighed before and after immersion in 50 cm³ of the test solution for a period of time up to 8 h. The average weight loss for two identical experiments was taken and expressed in milligrams per square centimeter.

UV-Vis spectra

UV-Vis spectra of the electrolyte solution of the inhibited system, before and after polarization measurements, were obtained by using the system described above.

Conductometric titration

Conductometric investigation [14] involving titration of 5 × 10⁻⁴ M Ni²⁺ cation dissolved in 0.5 M H₂SO₄ solution using 0.5 M H₂SO₄ solution containing 5 × 10⁻³ M imidazole as a titrant was performed.

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