



Influence of poly(aminoquinone) on corrosion inhibition of iron in acid media

C. Jeyaprabha, S. Sathiyarayanan, K.L.N. Phani, G. Venkatachari*

Corrosion Science, Engineering Division, Central Electrochemical Research Institute, Ceri Nagar, Karaikudi 630006, India

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Abstract

The inhibitor performance of chemically synthesized water soluble poly(aminoquinone) (PAQ) on iron corrosion in 0.5 M sulphuric acid was studied in relation to inhibitor concentration using potentiodynamic polarization and electrochemical impedance spectroscopy measurements. On comparing the inhibition performance of PAQ with that of the monomer *o*-phenylenediamine (OPD), the OPD gave an efficiency of 80% for 1000 ppm while it was 90% for 100 ppm of PAQ. PAQ was found to be a mixed inhibitor. Besides, PAQ was able to improve the passivation tendency of iron in 0.5 M H₂SO₄ markedly. © 2005 Elsevier B.V. All rights reserved.

Keywords: Iron; Corrosion; Sulphuric acid; Inhibition; Polyaminoquinone

1. Introduction

Inhibition of corrosion of metals is of high technological importance [1] and progress made in this field has been phenomenal in last few decades [2,3]. Acids find immense application in pickling, cleaning, descaling, etc. To avoid base metal attack and to ensure the removal of corrosion products/scales alone, inhibitors are extensively used. The selection of appropriate inhibitors mainly depends on the type of acid, its concentration, temperature, velocity, presence of dissolved solids and the type of metallic materials

involved. An important criterion in characterizing the efficiency of inhibitors is their efficiency/concentration ratio. Schmitt [4] in his review, discussed extensively about the types of inhibitors recommended to protect metallic materials especially ferrous metals and alloys from corrosion in acid solutions during pickling, acid cleaning, scale removing and oil and gas well acidizing.

The important prerequisites for a compound to be an efficient inhibitor are: (i) it should form a defect free, compact barrier film, (ii) it should chemisorb on to the metal surface, (iii) it should be polymeric or polymerise in situ on the metal, and (iv) the barrier thus formed should increase the inner layer thickness. Compounds containing nitrogen, sulphur and oxygen have been established as good inhibitors for iron in

* Corresponding author. Tel.: +91 4565227550;

fax: +91 4565227779.

E-mail address: sathya_ceil@yahoo.co.in (G. Venkatachari).

acidic media [5]. Organic compounds having π bonds are found to inhibit corrosion of steel by getting adsorbed over the electrode surface through electron sharing [6]. Presence of functional groups such as $=\text{NH}$, $-\text{N}=\text{N}-$, $-\text{CHO}$, $\text{R}-\text{OH}$, $\text{R}=\text{R}$, etc., in the inhibitor molecule [7,8] and also the steric factors, aromaticity, electron density at the donor atoms are found to influence the adsorption of the inhibitor molecule over corroding electrode surface. The role of molecular area [9] and molecular weight [10] of the organic molecule on its inhibition efficiency is also reported.

Various organic compounds such as amines, acetylenic alcohols, heterocyclic compounds, etc., have been in use as inhibitors in industries [11–15]. In recent days, polymers [16] and conducting polymers [17,18] have attracted a great deal of attention because of their wide range of industrial applications and economics. Due to the presence of extensive delocalization of π electrons, these polymers could serve as better corrosion inhibitors at very low concentrations.

It has been demonstrated that electrochemically synthesized poly(aminoquinones) [19–21], have got great potential to be an efficient inhibitor because of its special affinity toward metal surfaces. In addition, the abundance of π —electrons and unshared electron pairs on the nitrogen atom can interact with the empty d-orbitals of the iron to provide a protective barrier film. In this paper, the corrosion inhibition performance of chemically synthesized water soluble poly(aminoquinone) on iron in sulphuric acid is presented.

2. Experimental

2.1. Chemical synthesis of water soluble poly(amino quinone)

The synthesis of poly(amino-quinone) was carried out using purified benzoquinone and *o*-phenylenediamine with *p*-toluene sulphonic acid as dopant similar to the preparation of polyaniline [22,23]. Sodium salt of dodecyl benzene sulphonic acid (0.1 M) was used as dopant and was reacted with concentrated HCl. To this solution 0.05 M of benzoquinone and 0.05 M *o*-phenylenediamine dissolved in 0.1 M HCl were added and pre cooled. To this reaction mixture, freshly prepared solution of 0.1 M ammonium persulfate kept

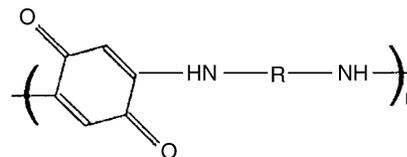


Fig. 1. Structure of poly(aminoquinone).

at a temperature of 5–10 °C was slowly added with constant stirring for two hours. Finally a solution of poly(amino quinone) was obtained. The average molecular weight was determined by GPC method (Shimadzu, Japan) and found to be 1.60×10^5 . The structure of PAQ is shown in Fig. 1.

2.2. Characterisation of poly(aminoquinone)

The synthesized poly(aminoquinone) was characterized by UV–vis (HITACHI – U 3400), FT-IR (PERKIN ELMER – PARAGON 500) spectroscopy.

2.3. Electrochemical measurements

The electrochemical experiments were made using a conventional three electrode cell assembly at 28 ± 1 °C. All the solutions were prepared using AR grade chemicals using triple distilled water and was deaerated by purging purified nitrogen for 0.5 h before the start of the experiments. The working electrode was a pure iron sample (Johnsons Matthey Ltd., UK) of 1 cm² area with the rest being covered with araldite epoxy and a large rectangular platinum foil was used as counter electrode and saturated calomel electrode as reference electrode. The working electrode was polished with different grades of emery papers, washed with water and degreased with trichloroethylene. The polarization and impedance studies were made after 30 min of immersion using Solatron Electrochemical Analyser (Model 1280 B). The polarization measurements were carried out using a Corware software from a cathodic potential of -0.2 V to an anodic potential of $+1.5$ V with respect to the corrosion potential at a sweep rate of 0.5 mV/s in order to find out the effect of inhibitors on corrosion and passivation. The data in the Tafel region (-0.2 to $+0.2$ V versus corrosion potential) have been processed for evaluation corrosion kinetic parameters evaluation by plotting E versus $\log I$ curves. The linear

Tafel segments of the anodic and cathodic curves were extrapolated to corrosion potential for obtaining the corrosion current values.

The inhibition efficiency was evaluated from the measured i_{corr} values using the relationship:

$$IE\% = \left\{ \frac{i_{\text{corr}} - i'_{\text{corr}}}{i_{\text{corr}}} \right\} \times 100$$

where i_{corr} and i'_{corr} are the corrosion current values without and with the addition of various concentrations of inhibitors.

For LPR measurements, the potential of the electrode was scanned from -0.02 to $+0.02$ V versus corrosion potential at a scan rate of 0.5 mV/s and the polarization resistance (R_p) was measured from the slope of η versus i curve in the vicinity of corrosion potential. From the measured polarization resistance values, the inhibition efficiency has been calculated using the relationship:

$$IE\% = \left\{ \frac{R'_p - R_p}{R'_p} \right\} \times 100$$

where R_p and R'_p are the polarization resistance values without and with the addition of inhibitors.

The impedance measurements were carried out using ac signals of 10 mV amplitude for the frequency spectrum from 100 KHz to 0.01 Hz. The Nyquist representations of the impedance data were analysed with Zview software. The charge transfer resistance (R_{ct}), is obtained from the diameter of the semicircle in Nyquist representation. The inhibition efficiency of the inhibitor has been found out from the relationship:

$$IE\% = \left\{ \frac{R'_{\text{ct}} - R_{\text{ct}}}{R'_{\text{ct}}} \right\} \times 100$$

where R_{ct} and R'_{ct} are the charge transfer resistance values in the absence and presence of inhibitors.

The interfacial double layer capacitance has been estimated from the impedance value of the frequency having maximum imaginary component in the Nyquist plot by using the following equation:

$$C_{\text{dl}} = \frac{1}{2\pi f_{\text{max}} R_{\text{ct}}}$$

and the surface coverage θ by the inhibitor molecule is given by [24,25]:

$$\theta = \frac{C_{\text{dl}} - C'_{\text{dl}}}{C_{\text{dl}}}$$

where C_{dl} and C'_{dl} are the double layer capacitance values in the absence and presence of inhibitors.

2.4. Surface examination

The morphologies of iron surface after corrosion in the presence and absence of inhibitors in 0.5 M H_2SO_4 under the magnification of 1 K were examined by scanning electron microscope (HITACHI S 3000H).

3. Results and discussion

3.1. Corrosion inhibition by *o*-phenylenediamine

The effectiveness of inhibitor *o*-phenylenediamine has been found out by tafel polarization, linear polarization and impedance method. The polarisation behaviour of iron in 0.5 M H_2SO_4 with the addition of various concentrations of *o*-phenylenediamine in the tafel region is shown in Fig. 2. The corrosion kinetic parameters derived from these curves are given in Table 1. From the table, it is found that the addition of

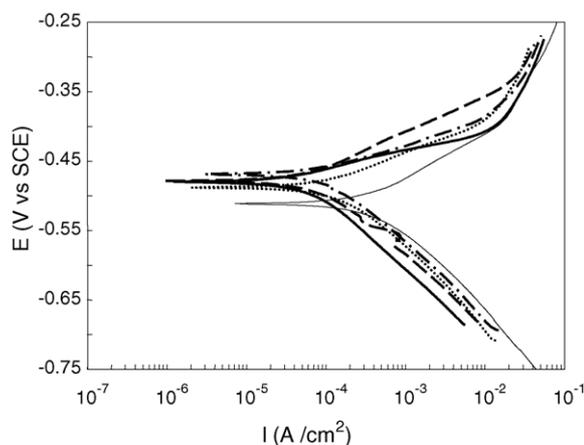


Fig. 2. Potentiodynamic polarization curves for iron in 0.5 M H_2SO_4 with the addition of *o*-phenylenediamine (—) blank; (···) 100 ppm; (—•) 250 ppm; (—■) 750 ppm; (—) 1000 ppm.

Table 1
Corrosion kinetic parameters of pure iron in 0.5 M H₂SO₄ with *o*-phenylenediamine at 28 °C

Concentration of <i>o</i> -phenylenediamine (ppm)	E_{corr} (mV) vs. SCE	ba (mV/dec)	bc (mV/dec)	i_{corr} ($\mu\text{A}/\text{cm}^2$)	Inhibition efficiency (%)
Blank	-508	76	109	410	–
100	-514	60	105	223	45
250	-492	50	103	160	61
500	-486	68	92	125	70
750	-490	65	100	98	76
1000	-483	50	103	74	81

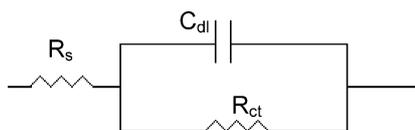
o-phenylenediamine in the concentration range 100–1000 ppm decreases the dissolution rate of iron in 0.5 M H₂SO₄. The corrosion current value (i_{corr}) is decreased from 410 $\mu\text{A cm}^{-2}$ for the inhibitor free solution to 74 $\mu\text{A cm}^{-2}$ at the highest concentration of *o*-phenylenediamine studied.

The steady state corrosion potentials are not altered significantly by the addition of *o*-phenylenediamine. Further the anodic and cathodic tafel slopes are in the range of 65 ± 8 and 105 ± 8 mV, respectively. Since there is no marked change in the corrosion potential and tafel slopes in the presence of inhibitor, it is inferred that the inhibitor is first adsorbed onto iron surface and blocks the reaction sites without altering the anodic and cathodic reaction mechanisms [26].

The polarization resistance (R_p) values in the presence and absence of inhibitor are given in Table 2. The R_p values showed an increase from 34 to 205 Ωcm^2 with the addition of *o*-phenylenediamine inhibitor. An inhibitor efficiency of 83% has been observed for the highest studied concentration.

The Nyquist representation of the impedance behaviour of iron in 0.5 M H₂SO₄ with and without

the addition of various concentrations of *o*-phenylenediamine is shown in Fig. 3. The existence of a single semi circle shows the presence of single charge transfer process during dissolution which is unaffected by the presence of inhibitor molecules. The slightly depressed nature of the semi circle which has the center below the x -axis is the characteristic for solid electrodes and such frequency dispersion has been attributed to roughness and other inhomogenities of the solid electrode [27–29]. The charge transfer resistance (R_{ct}) and the interfacial double layer capacitance (C_{dl}) values were derived by using the following equivalent circuit:



The impedance values are given in Table 2. R_{ct} value is increased from 33 to 235 Ωcm^2 and C_{dl} value is decreased from 2244 to 123 $\mu\text{F cm}^{-2}$ in the presence of highest concentration of *o*-phenylenediamine inhibitor.

Table 2
Electrochemical impedance and linear polarization parameters for pure iron in 0.5 M H₂SO₄ with *o*-phenylenediamine at 28 °C

Concentration of <i>o</i> -phenylenediamine (ppm)	Impedance method				LPR method	
	R_{ct} (Ωcm^2)	C_{dl} ($\mu\text{F cm}^{-2}$)	Inhibition efficiency (%)	Surface coverage (θ)	R_p (Ωcm^2)	Inhibition efficiency (%)
Blank	33	2244	–	–	34	–
100	51	760	35	0.6613	62	45
250	80	447	59	0.8008	72	52
500	130	300	75	0.8663	139	76
750	138	246	76	0.8904	180	81
1000	235	123	86	0.9452	205	83

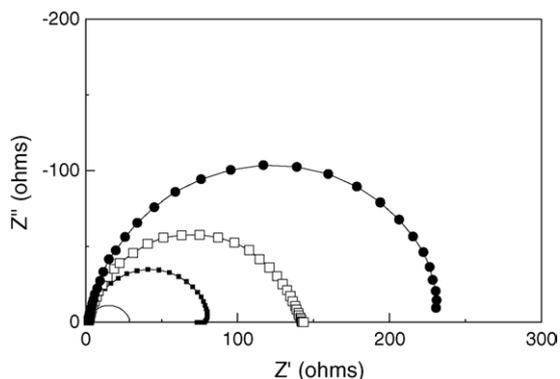


Fig. 3. Impedance plot for iron in 0.5 M H_2SO_4 with the addition of *o*-phenylenediamine (—) blank; (●) 250 ppm; (□) 750 ppm; (●) 1000 ppm.

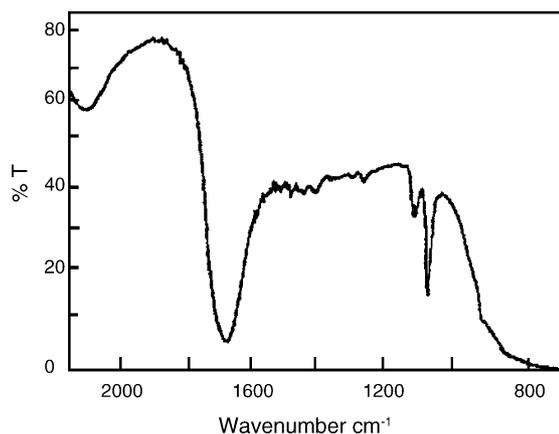


Fig. 5. FT-IR spectra of poly(aminoquinone).

3.2. Corrosion inhibition by poly(aminoquinone)

The UV–vis spectrum of poly(amino quinone) is shown in Fig. 4. The adsorption peak at 245–270 nm shows the incorporation of benzoquinone in to the poly(phenylenediamine) polymer and the peak at 321 nm shows the π – π^* transition in the benzenoid ring. The peak at 348 nm indicates the linkage of quinone unit to the poly(phenylenediamine) polymer at 1,4 position. The IR absorption band at 1636 cm^{-1} (Fig. 5) indicates the linkage of quinone unit to the poly(phenylenediamine) polymer at 1,4 position and the characteristic peaks for amino polymers are observed at 1543, 1459, 1044 and 920 cm^{-1} .

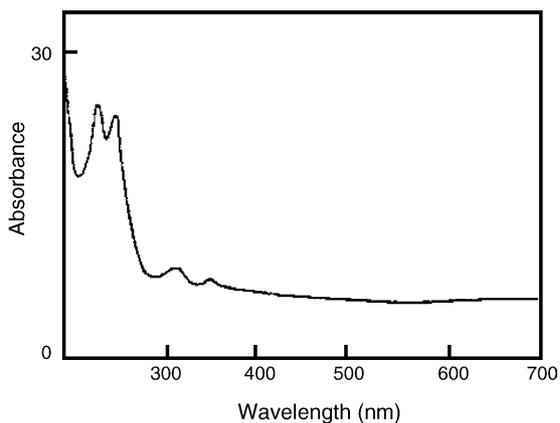


Fig. 4. UV–vis spectra of poly(aminoquinone).

The tafel polarization curves for iron in 0.5 M H_2SO_4 with the addition of various concentrations of poly(aminoquinone) are shown in Fig. 6. The corrosion kinetic parameters derived from these curves are given in Table 3. It is evident from the table that the corrosion current value (i_{corr}) is decreased from $410\text{ }\mu\text{A cm}^{-2}$ of that of blank solution to $143\text{ }\mu\text{A cm}^{-2}$ with the addition of 10 ppm of poly(aminoquinone) and it gets further reduced gradually with increasing concentration of the inhibitor and reaches a value of $38\text{ }\mu\text{A cm}^{-2}$ with 100 ppm of the added inhibitor. The corrosion potential remains unaffected with the addition of PAQ. This shows the mixed type inhibition offered by PAQ.

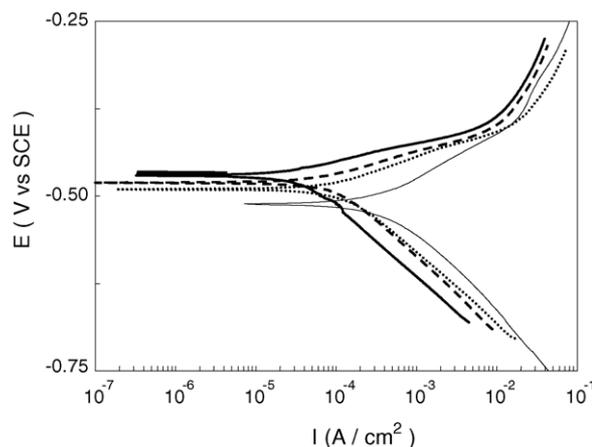


Fig. 6. Potentiodynamic polarization curves for iron in 0.5 M H_2SO_4 with the addition of poly(aminoquinone) (—) blank; (···) 10 ppm; (---) 50 ppm; (—) 100 ppm.

Table 3

Corrosion kinetic parameters of pure iron in 0.5 M H₂SO₄ with poly(aminoquinone) at 28 °C

Concentration of poly(aminoquinone) (ppm)	E_{corr} (mV) vs. SCE	ba (mV/dec)	bc (mV/dec)	i_{corr} ($\mu\text{A}/\text{cm}^2$)	Inhibition efficiency (%)
Blank	−508	76	109	410	–
10	−508	53	100	143	65
25	−496	51	106	130	68
50	−496	53	102	92	78
75	−507	50	100	41	90
100	−489	51	100	38	91

From the linear polarization studies (Table 4), it has been observed that the polarization resistance R_p increases from 34 $\Omega \text{ cm}^2$ of that of blank to 920 $\Omega \text{ cm}^2$ due to the adsorption of PAQ molecules on iron electrode surface and the highest inhibition efficiency of 96% is observed at 100 ppm PAQ.

As in the case of the *o*-phenylenediamine monomer, the Nyquist representation of the complex impedance of iron in 0.5 M H₂SO₄ (Fig. 7) shows the presence of depressed semicircles indicating the activation controlled nature of the corrosion process with micro roughness of the electrode. The charge transfer resistance (R_{ct}), interfacial double layer capacitance (C_{dl}) values and the surface coverage (θ) values derived from these figures are given in Table 4. The R_{ct} values are increased from 33 to 282 $\Omega \text{ cm}^2$ with a corresponding decrease in C_{dl} values from 2244 to 34 $\mu\text{F cm}^{-2}$ for the added poly(aminoquinone). In contrast to the θ values of *o*-phenylenediamine monomer, the θ value is increased to 0.98 by the addition of 100 ppm of poly(aminoquinone) whereas it is 0.94 by the addition of 1000 ppm of the monomer.

Fig. 8 shows the FT-IR spectrum of iron surface exposed to 0.5 M H₂SO₄ containing 50 ppm of poly(aminoquinone). The shift of characteristic band of imino-quinone moiety of poly(aminoquinone) from

1635 to 1660 cm^{-1} confirming the strongly adsorbed state of polymer on iron surface.

The UV reflectance spectra of iron in 0.5 M H₂SO₄ with the addition of *o*-phenylenediamine and poly(aminoquinone) is shown in Fig. 9. It can be seen that, the percentage reflectance is high for polished iron specimen and is very much reduced for iron treated with 0.5 M H₂SO₄. The reflectance in the case of iron treated with 0.5 M H₂SO₄ in presence of *o*-phenylenediamine is intermediate and that of poly(aminoquinone) is high confirming the better inhibition efficiency of the poly(aminoquinone).

Amines in aqueous solutions may exist as either neutral molecules or in the form of cations [30] depending upon the concentration of H⁺ ions in the solutions. In acidic solutions, they predominantly exist as cations and adsorb through electrostatic interaction between the positively charged anilinium cation and negatively charged metal surface [31]. In the case of aromatic amines, the interaction between π -electron of benzene ring and the positively charged metal surface also play an important role [32]. The lone pair of electron density of nitrogen atom is diminished by resonance effect of phenyl group and the possibility of electron donation to the metal via nitrogen is very less. Further, the pzc of iron in sulphuric acid is positive

Table 4

Electrochemical impedance and linear polarization parameters for pure iron in 0.5 M H₂SO₄ with poly(aminoquinone) at 28 °C

Concentration of poly(aminoquinone) (ppm)	Impedance method				LPR method	
	R_{ct} ($\Omega \text{ cm}^2$)	C_{dl} ($\mu\text{F cm}^{-2}$)	Inhibition efficiency (%)	Surface coverage (θ)	R_p ($\Omega \text{ cm}^2$)	Inhibition efficiency (%)
Blank	33	2244	–	–	34	–
10	79	95	58	0.9577	168	80
25	88	81	63	0.9639	252	87
50	115	89	71	0.9603	330	90
75	230	46	85	0.9795	400	92
100	282	34	88	0.9849	920	96

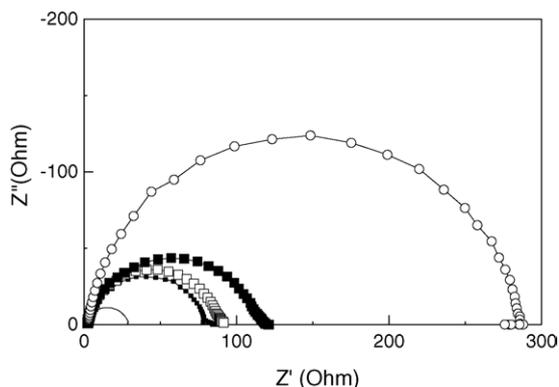


Fig. 7. Impedance plot for iron in 0.5 M H_2SO_4 with the addition of poly(aminoquinone) (—) blank; (●) 25 ppm; (□) 50 ppm; (●) 75 ppm; (○) 100 ppm.

with respect to corrosion potential [33]. Hence iron surface is positively charged and interaction between the π -electron of benzene ring with iron surface is more probable. At low concentration, *o*-phenylenediamine offers lower inhibition efficiency. This may be due to the lower coverage of the surface by formation of a “quasi complex” [34]. The inhibitive effect of the same compound at higher concentration may be due to higher coverage with blockage of the more active sites of the metal surface. The inhibitive property of poly(aminoquinone) is mainly due to the adsorption of polymer molecules on the metal surface in the form of the adsorption through π -electron interaction between quinonoid structure of the molecule and the metal surface.

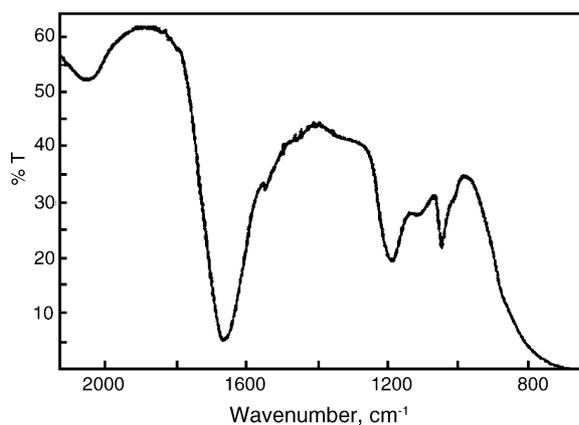


Fig. 8. FT-IR spectra of poly(aminoquinone) adsorbed on the iron surface.

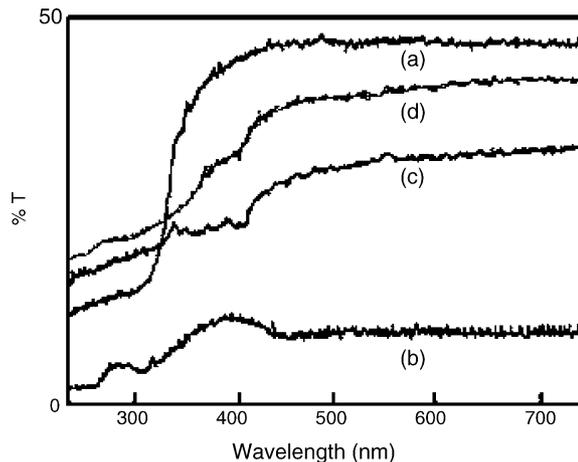


Fig. 9. UV-reflectance curves for iron under different conditions (a) polished, (b) iron treated with 0.5 M H_2SO_4 , (c) iron treated with 0.5 M H_2SO_4 in the presence of 1000 ppm *o*-phenylenediamine, and (d) iron treated with 0.5 M H_2SO_4 in the presence of 100 ppm poly(aminoquinone).

3.3. Adsorption isotherm of inhibitors

The values of surface coverage (θ) have been obtained from the C_{dl} values for different concentrations of the inhibitors since adsorption of inhibitor is

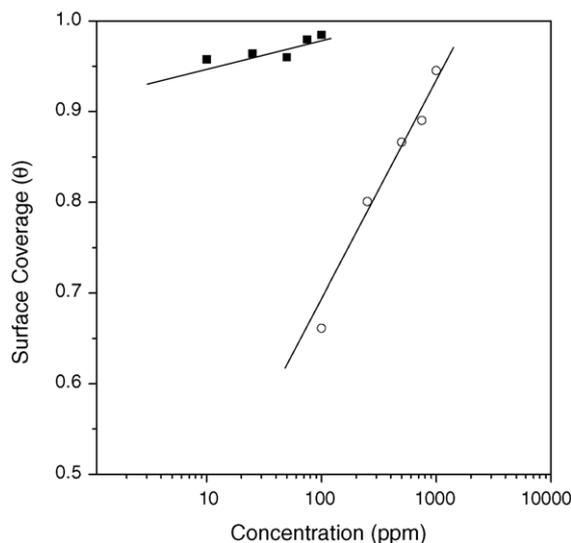


Fig. 10. Temkin adsorption isotherm for iron in 0.5 M H_2SO_4 containing inhibitors (○) *o*-phenylenediamine; (■) poly(aminoquinone).

directly related to the double layer capacitance [35]. Tables 2 and 4 give the surface coverage values for various concentrations of OPD and PAQ. The θ values are found to fit Temkin adsorption isotherm and the corresponding plot is shown in Fig. 10. The free energy of adsorption (ΔG_{ads}) values of OPD and PAQ have been obtained by using the Temkin adsorption equation:

$$\left(\frac{1}{55.5}\right)e^{(-\Delta G_{\text{ads}}/RT)} \times C = e^{f\theta}$$

for the highest surface coverage values of inhibitor. The free energy of adsorption of OPD and PAQ are found to be -13.5 and -21.9 kJ/mol. The energy of

inhomogeneity factor “ f ” values are found to be 3.40 kJ for OPD and 0.04 kJ for PAQ. From the ΔG_{ads} values one can note that the spontaneity of the adsorption process for PAQ is greater than OPD [36]. Generally values of ΔG_{ads} up to -20 kJ/mol are consistent with the electrostatic interaction between the charged molecules and charged metal (physisorption) [37,38].

3.4. SEM studies

The morphologies of iron surface after corrosion in the presence and absence of inhibitors in 0.5 M H_2SO_4 are shown in Fig. 11a–c. It can be seen that the uniform attack is observed in the case of iron exposed to 0.5 M H_2SO_4 (Fig. 11a). The attack is reduced significantly

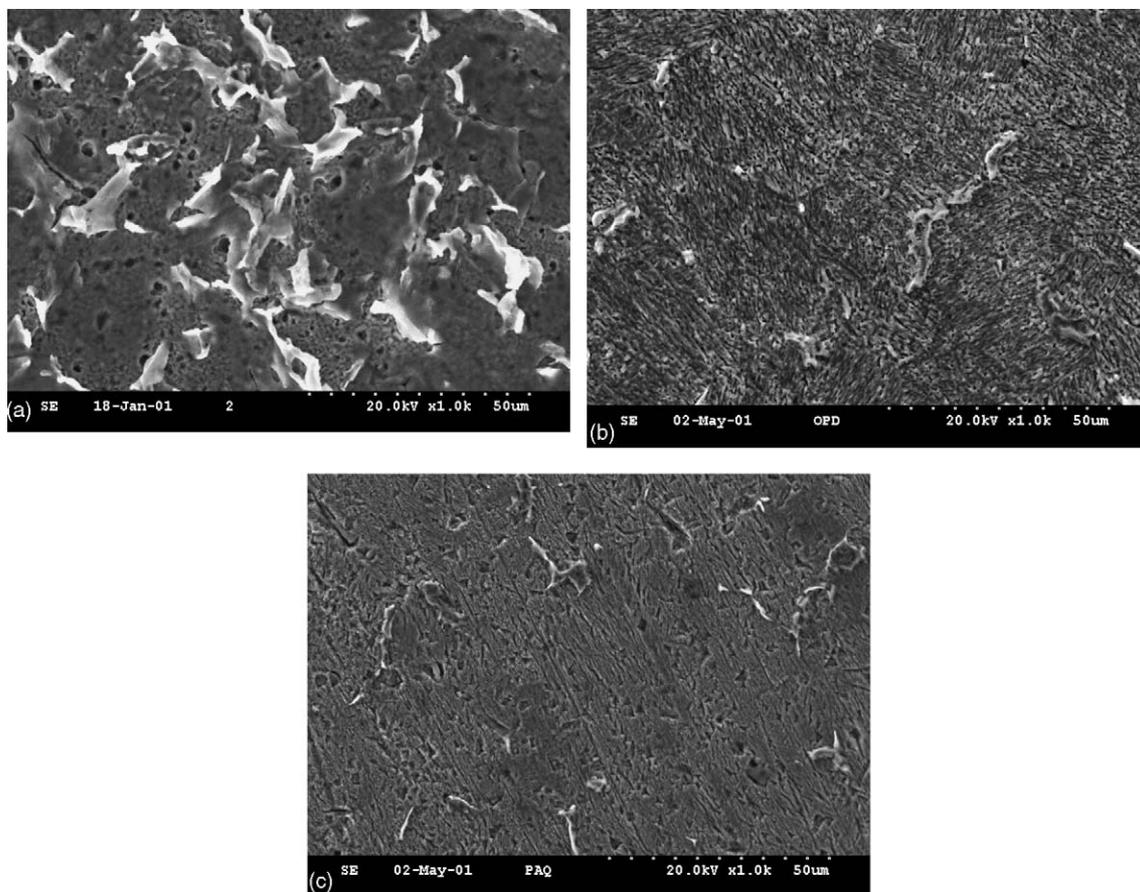


Fig. 11. (a) Scanning electron micrograph of iron after exposure to 0.5 M H_2SO_4 , (b) scanning electron micrograph of iron after exposure to 0.5 M H_2SO_4 containing 1000 ppm *o*-phenylenediamine, (c) scanning electron micrograph of iron after exposure to 0.5 M H_2SO_4 containing 100 ppm poly(amino quinone).

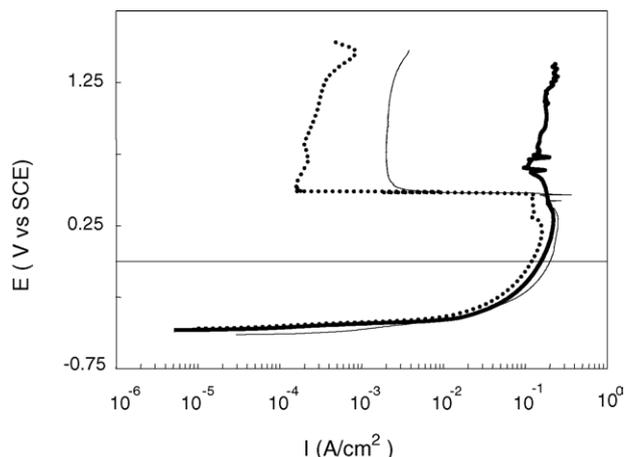


Fig. 12. Anodic polarisation curve of iron in 0.5 M H_2SO_4 with the addition of *o*-phenylenediamine and poly(aminoquinone) (—) blank; (—) 1000 ppm *o*-phenylenediamine; (.....) 100 ppm poly(aminoquinone).

in the 0.5 M H_2SO_4 solution containing 1000 ppm of *o*-phenylenediamine and for the iron surface exposed to 0.5 M H_2SO_4 containing 100 ppm of poly(aminoquinone) (Fig. 11b), where as the formation of thin layer of adsorbed poly(aminoquinone) is observed (Fig. 11c) which is responsible for the greater reduction of corrosion rate.

3.5. Passivation of iron by inhibitors

The passivation behaviour of iron in 0.5 M H_2SO_4 containing *o*-phenylenediamine and poly(aminoquinone) has been found out by potentiodynamic polarization method. Fig. 12 gives the passivation behaviour for the selected inhibitor concentration. It can be seen that the PAQ has been found to improve the passivation characteristics while OPD retards the passivation. In the passive region, where the metal surface is covered by an adherent oxide layer, the direct interaction of nitrogen to an exposed metal atom is a remoter event. Amines are adsorbed onto oxide surface through hydrogen bond formation [39–41]. The presence of more amine group in PAQ has the capability of forming highest N–H linkages that can co-ordinate with the oxide coated surface through hydrogen bonding. Hence, PAQ is able to enhance the passivity. Similar enhancement of passivation for steel in 0.5 M H_2SO_4 by cyclohexylamine has been reported [42]. In the case of OPD, no passivation is

observed. This may be due to the presence of adsorbed OPD layer, which interferes with the formation of passive film.

4. Conclusions

Both *o*-phenylenediamine and poly(aminoquinone) inhibit the corrosion of iron in 0.5 M H_2SO_4 medium. The inhibition performance of poly(aminoquinone) is found to be better than *o*-phenylenediamine due to the presence of extensive delocalised π electrons. The passivation tendency of iron in 0.5 M H_2SO_4 is increased in the presence of poly(aminoquinone).

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