Reviewed articles

# Poly(styrene sulphonic acid)-doped polyaniline as an inhibitor for the corrosion of mild steel in hydrochloric acid

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Abstract

Poly(styrenesulphonic acid)-doped polyaniline has been synthesised and the influence of this polymeric compound on the inhibition of corrosion of mild steel in 1M HCl has been investigated using weight loss measurements, galvanostatic polarisation studies, electropermeation studies and a.c. impedance measurements. The polymer acts predominantly as an anodic inhibitor. Hydrogen permeation studies and a.c. impedance measurements clearly indicate a very effective performance of the compound as a corrosion inhibitor. The adsorption of the compound on the mild steel surface obeys Temkin's adsorption isotherm.

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Volume 49 · Number 1 · 2002 · pp. 19–26

DOI 10.1108/00035590210413566

## Introduction

A thorough and systematic study on the mechanism of action of corrosion inhibitors has considerable significance owing to its usefulness in developing new inhibitors for their effective use in several industries. Usually mineral acids such as hydrochloric and sulphuric acids are used to remove millscale and the rust formed on ferrous alloys and steel before further processing. Inhibitors are used in industrial processes to minimise both the metal loss and acid consumption. Many organic compounds have been examined for their utility as corrosion inhibitors for different corrosive media for the past few years (Hackerman, 1962; Machu, 1971; Schmitt, 1984; Trabanelli, 1991). For the inhibition of corrosion by most of the organic compounds, their adsorption on the metal surface is considered as the initial step. Usually, organic compounds which serve as very effective corrosion inhibitors have at least one functional group which acts as the reaction centre for the adsorption process. The presence of hetero atoms such as nitrogen, sulphur and phosphorus in the organic compound and also the presence of an aromatic ring, in the molecular structure are found to have a significant influence on the extent of adsorption of the organic compound on the metal surface and thereby the extent of inhibition of corrosion. In general, nitrogen containing heterocyclic compounds are found to function as very effective corrosion inhibitors for many metals and alloys in different aggressive corrosive media (Chin and Nobe, 1971; Box and Bradley, 1977; Leroy, 1976; Vasudevan *et al.*, 1995; Muralidharan and Venkatakrishna Iyer, 1997; Quraishi *et al.*, 1996, 1997) It has been observed that compounds containing both nitrogen and sulphur perform as very effective corrosion inhibitors (Muralidharan *et al.*, 1993, 1995; Quraishi *et al.*, 1996, 1997, 1998).

The utility of polymers as inhibitors for corrosion has been recognised very recently. Joshi and Srinivasan (1989) have examined the effectiveness of water soluble melamine formaldehyde as an inhibitor for the corrosion of mild steel in HCl. The inhibitive action of proparyl alcohol which can polymerise *in situ* on the substrate metal has been studied by Bartos and Hackermann (1992) for the corrosion of iron in HCl and also by Aramaki

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**Table I** Corrosion kinetic parameters for the corrosion of mild steel in 1M HCl containing different concentrations of the polymer

| Inhibitor concentration | $E_{corr}$ (mV) | Tafel slope (mV. dec$^{-1}$) $b_a$ | $b$ | $i_{corr}$ mA.cm$^{-2}$ | Inhibition efficiency (%) |
|---|---|---|---|---|---|
| Blank (1M HCl) | −525 | 65 | 120 | 3.40 | – |
| 10 | − 505 | 67 | 120 | 2.50 | 26.4 |
| 20 | − 492 | 70 | 123 | 2.00 | 41.2 |
| 30 | − 485 | 75 | 125 | 1.74 | 48.5 |
| 40 | − 475 | 75 | 125 | 1.74 | 48.5 |
| 50 | − 460 | 83 | 127 | 1.48 | 56.4 |
| 60 | − 455 | 90 | 130 | 1.00 | 70.5 |
| 70 | − 450 | 93 | 135 | 0.48 | 85.9 |

and Fujikoa (1997). Silverman *et al.* (1995) studied the influence of pH on the inhibition of corrosion of steel in HCl by polyaspartic acid by weight loss and a.c. impedance measurements. Muralidharan *et al.* (1995) have investigated the utility of polyaminobenzoquinone as an inhibitor for the corrosion of mild steel in acidic solution. Karpagam *et al.* (1996) have examined the usefulness of acrylamide-thiomalic acid polymer as an inhibitor for the corrosion of mild steel in acidic solutions. Elsayed (1996) studied the inhibiting action of a few polymers such as pectin, polyvinyl alcohol, polyacrylic acid and polyethylene glycol for the corrosion of iron in HCl, $H_2SO_4$ and $HClO_4$.

The use of conducting polymers for the inhibition of corrosion is an area which is very recently gaining increasing attention. Among the conducting polymers, those derived from aniline and its derivatives have been predicted to be futuristic materials because of their greater environmental stability, cheapness of raw materials used, easy synthesis and their unique conduction mechanism. But these polymers are not found to be processible due to the rigid backbone structure, because of the extensive delocalisation of $\pi$-electrons. This large availability of $\pi$-electrons enable them to function as very effective corrosion inhibitors. But, these polymeric compounds suffer from the drawback that they are not soluble in most of the common solvents and aqueous acidic solutions. Recently, soluble forms of polyaniline have been prepared by modifying the polymeric chain by introducing ethoxy and a methoxy groups at ortho position to N–H group of polyaniline and they have been examined as inhibitors of corrosion for mild steel in acidic solutions (Sathyanarayanan *et al.*, 1992, 1994). Recently Sathiyanarayanan *et al.* (1999) have studied the influence of a completely water soluble and a commercially available polyaniline, namely acid black II on the inhibition of corrosion of mild steel in HCl. In the present study the efficacy of a completely water soluble poly(styrene sulphonic acid) doped polyaniline on the corrosion of mild steel in 1M HCl has been examined using weight loss measurements, galvanostatic polarisation studies, hydrogen permeation measurements and impedance measurements and UV reflectance measurements.

## Experimental

### Materials

*Mild steel strips of the composition*

Carbon (C) = 0.07 per cent
Sulfur (S) = Nil
Phosphorus (P) = 0.008 per cent
Silicon (Si) = Nil
Manganese (Mn) = 0.34 per cent, and
Iron (Fe) = remainder

and of size 5×2 × 0.025cm were used for weight loss and hydrogen permeation studies. These strips were given fine mechanical polishing and then degreased with trichloroethylene before use. For polarisation measurements, a cylindrical mild steel rod of the same composition as above and embedded in Araldite with an exposed area of 0.283cm$^2$ was used. The electrode was polished using a sequence of emery papers of different grades and then degreased with trichloroethylene.

AR grade HCl was used for preparing solutions. Sodium salt of poly(styrene sulphonic acid) (PSSA-Na) and ammonium persulphate were of Aldrich make. Aniline

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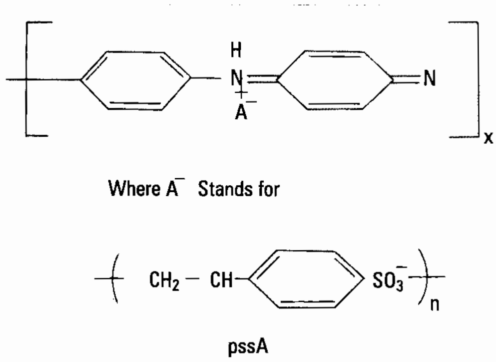
(Merck) was procured from E-Merck (India) Ltd., Mumbai.

### 1.2 Synthesis of poly(styrene sulphonic acid)—doped polyaniline

Poly(styrene sulphonic acid)-doped polyaniline was prepared as described earlier (Li *et al.*, 1987; Shannon and Fernandez, 1994). The method consists in adding an aqueous solution of ammonium persulphate slowly to a solution of 1:1mol. ratio of aniline and a proton acid namely poly(styrene sulphonic acid) at room temperature. After allowing the reaction to continue for a duration of 2 hours, the dark solid product formed was separated by filtration, washed with a small amount of sulphuric acid, and then with water and dried in vacuum for 48 hours. The compound is freely soluble in water.

The polymer was characterised by recording its infra red spectrum using Nicolet Fourier Transform Infra red Spectroscopy (FTIR) using a KBr pellet. FTIR spectrum of the compound shows sulphate C–H stretching over 2915 and 2850cm$^{-1}$ (PSSAH backbone) which shows the presence of PSSA in the compound. The molecular formula of the doped polymer is shown in Figure 1.

**Figure 1** Molecular formula of poly(styrene sulphonic acid)-doped polyaniline




## 2 Methods

### 2.1 Weight loss measurements

Weight loss measurements were carried out as described in an earlier paper (Muralidharan *et al.*, 1995). Already weighed triplicate mild steel specimens were suspended from glass hooks fused to a glass rod in 200ml of inhibited and uninhibited solutions kept in a 250ml beaker for a duration of 2 hours. At the end of the period, specimens were taken out, washed well with running water, rinsed with distilled water, dried thoroughly and finally weighed. Inhibition efficiency was calculated from the weight losses of specimens in the absence and presence of the inhibitor using the equation:

$$\text{Inhibition efficiency (\%)} = \frac{\text{Weight loss in the presence of the inhibitor}}{\text{Weight loss in the absence of the inhibitor}} \times 100$$

### 2.2 Galvanostatic polarisation studies

Galvanostatic polarisation experiments were carried out in the range of 0.1–100mA cm$^{-2}$ at a temperature of 30 ± 1°C using an indigenously fabricated galvanostat. A platinum foil of dimensions 3cm × 3cm was used as the auxiliary electrode and a Hg/$Hg_2Cl_2$/1M HCl electrode was used as the reference electrode. The experimental solution was stirred thoroughly using a magnetic stirrer.

### 2.3 Hydrogen permeation measurements

Hydrogen permeation studies were carried out at a temperature of 30± 1°C using an adaptation of Devanathan and Stachursky's modified two compartment cell (Devanathan and Stachursky, 1962; Srinivasan *et al.*, 1993). Hydrogen permeation currents were recorded using an X–Y–T Rikadenki recorder in the absence and presence of the polymeric compound in 1M HCl.

### 2.4 Electrochemical impedance spectroscopy

Impedance measurements were carried out at the open circuit potential after immersing the mild steel specimen in the experimental solution using Electrochemical system (EG & G.PAR) which includes a digital potentiostat Model 273 and a lock-in-amplifier Model 5208 and IBM PC in the frequency range 60KHz to mHz. The applied voltage of the sinusoidal wave was 5mV.

### 2.5 UV reflectance spectroscopic studies

UV diffuse reflectance studies were carried out on the surfaces of polished, corroded and corrosion inhibited mild steel specimens in the range of 200–700 nm using normal incident angle (90°) using UV visible NIR

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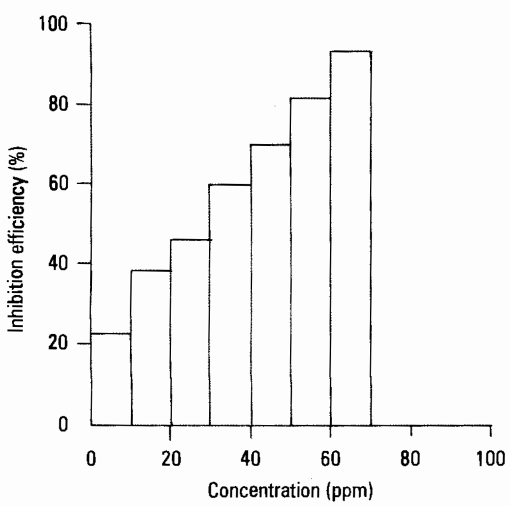
spectrophotometer (Model U-3400, Hitachi, Japan).

## 3 Results and discussion

### 3.1 Weight loss measurement

Figure 2 shows the variation of the inhibition efficiency with the concentration of doped polyaniline in the form of a chart. It is found that the inhibition efficiency increases with increase in the concentration of the polymeric compound. The good performance of the compound can be due to its strong adsorption on the metal surface. It is found that a concentration as low as 70ppm is found to give an inhibition efficiency of 90.0 per cent. This can be attributed to greater availability of $\pi$-electrons in the aromatic ring which facilitates a flat orientation of the polymer on the metal surface. The formation of positively charged quarternary ammonium ions at the nitrogen atom in acidic solution also helps in the strong adsorption of the polymeric compound on the metal surface (Frignani *et al.*, 1991). The formation of quarternary nitrogen has been confirmed from IR spectral lines where the -NH absorption due to quarternary nitrogen is 1116cm$^{-1}$. Since absorption is also observed at 1454–1580cm$^{-1}$, it holds out the fact that all the nitrogen in the polymer are not quarternary nitrogen (Sathyanarayanan *et al.*, 1994). As chloride ions are strongly adsorbed on the metal surface rendering it more negatively charged, the strong adsorption of quarternary ammonium ions is favoured. Large molecular area of the polymer which enables more coverage of the metal surface may also be responsible for the high values of inhibition efficiency.

**Figure 2** Variation of inhibition efficiency with the polyaniline concentration of poly(styrene sulphonic acid)-doped polyaniline in 1.0M HCl




### 3.2 Galvanostatic polarisation studies

Figure 3 shows galvanostatic polarisation curve for mild steel in 1.0M HCl which illustrates the evaluation of corrosion kinetic parameters such as $b_a$, $b_c$, $E_{corr}$ and $i_{corr}$. Table II gives the values of these parameters for different concentrations of poly(styrene-sulphonic acid) doped polyaniline in 1.0M HCl obtained from galvanostatic polarisation curves. It can be seen from this table that the values of $b_a$, $b_c$ and $i_{corr}$ given in the table are almost the same as reported earlier (Muralidharan *et al.*, 1995). It is found that an increase in the concentration of the polymer enhances the values of both $b_a$ and $b_c$; but the values of $b_a$ are enhanced to a greater extent than $b_c$ values. So, the polymeric inhibits the corrosion of mild steel in HCl by mixed type of control; but, predominantly, under anodic control. The formation of a film of a surface complex may be responsible for the decrease in anodic dissolution of mild steel in HCl (Donahue and Nobe, 1967). The adsorbed film on the metal surface was retained for a period of time when specimens dipped the inhibited acid were transferred to fresh acid without the polymer. This observation supports the formation of an insoluble film on the metal surface when brings down corrosion. $E_{corr}$ is shifted to less negative values in the presence of different concentrations of the polymer. Values of inhibition efficiency obtained from $i_{corr}$ values in the presence of different concentrations of

**Figure 3** A typical galvanostatic polarisation curve for mild steel in 1.0M HCl which illustrates the evaluation of corrosion kinetic parameters such as $b_a$, $b_c$, $E_{corr}$ and $i_{corr}$

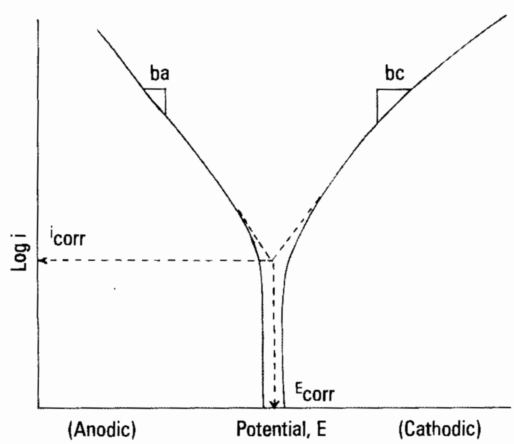


**Table II** Impedance parameters for the corrosion of mild steel in 1M HCl in the presence of different concentrations of the polymer

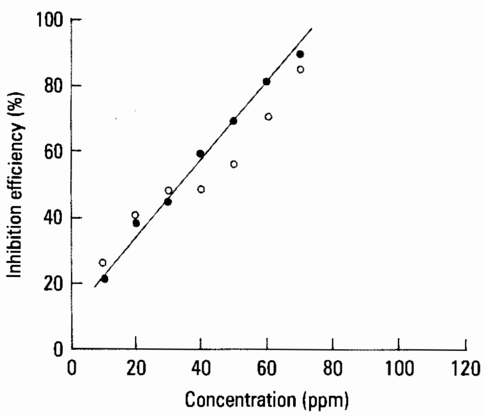
| Inhibitor concentration ppm | $R_t$ $\Omega.cm^2$ | $C_{dl}$ $\mu F.cm^{-2}$ | $i_{corr}$ $mA.cm^{-2}$ | Inhibition efficiency (%) |
|---|---|---|---|---|
| **Blank (1M HCl)** | 5.2 | 252 | 3.34 | – |
| **20** | 15.3 | 162.1 | 1.50 | 55.1 |
| **40** | 23.4 | 140.0 | 1.26 | 62.3 |
| **60** | 32.2 | 132.2 | 0.732 | 78.0 |

the polymer shows fairly good agreement with those obtained from weight loss measurements in most of the cases, as seen from Figure 4.

### 3.3 Hydrogen permeation studies

A good corrosion inhibitor, in addition to bringing down metal loss in acidic solutions, should be able to bring down the entry of hydrogen into the metal considerably. In this content the influence of the poly(styrene sulphonic acid) doped polyaniline on the extent of hydrogen permeation through mild steel was studied. Figure 5 shows the permeation current vs time curve for 1 HCl in the presence of different concentrations of the polymer. It can be seen from this figure that the polymer brings down the extent of hydrogen permeated through steel, besides being a very good corrosion inhibitor. This can be attributed to the formation of stable chelates on the metal surface, which brings down the hydrogen entry into the metal.

**Figure 4** A comparison of values of inhibition efficiency for different concentrations of the polymeric compound obtained from weight loss and galvanostatic polarisation measurements.

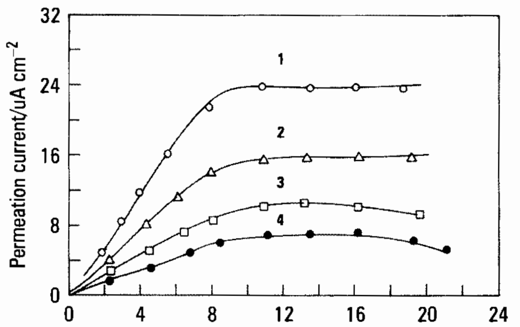



**Key**
○ Weight loss measurements
● Galvanostatic polarisation

### 3.4 ac impedance measurements

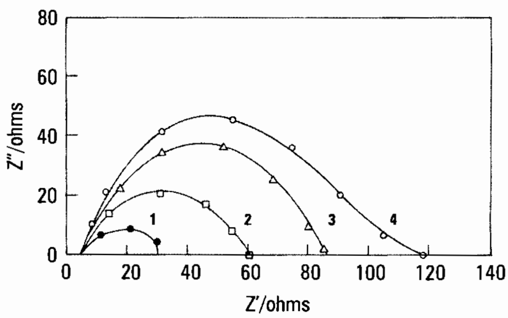
Figure 6 shows Nyquist plots obtained from a.c. impedance measurements for mild steel in 1M HCl in the presence of different concentrations of the polymer. Various parameters such as charge-transfer resistance ($R_t$), double layer capacitance ($C_{dl}$) and $i_{corr}$ were obtained from impedance measurements and are shown in Table II. $R_t$ values were calculated from the difference in impedance at lower and higher frequencies as suggested by Haruyama and Tsuru (1981). $i_{corr}$ values were calculated using the Stern-Geary equation. Values of inhibition obtained by weight loss

**Figure 5** Permeation current vs time curves for mild steel in 1M HCl.




**Key**
1 Blank (1M HCl) 2 20 ppm polymer
3 40 ppm polymer 4 60 mm polymer

**Figure 6** Nyquist plots for mild steel in 1M HCl in the presence of different concentrations of the polymer.




**Key**
1 Blank (1M HCl) 2 20 ppm polymer
3 40 ppm polymer 4 60 ppm polymer

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measurements, galvanostatic polarisation and impedance measurements show fairly good agreement. $C_{dl}$ values were calculated from the frequency at which the imaginary component of impedance was maximum ($Z_{im}$ max) using the reaction:

$$C_{dl} = \frac{1}{2\pi f_{max}} \times \frac{1}{R_t} \quad (1)$$

where $f_{max}$ is the frequency at which the imaginary component of impedance is maximum. The addition of polymer to HCl is found to enhance $R_t$ values and bring down $C_{dl}$ values. These observations clearly bring out the fact that the corrosion of mild steel in HCl is controlled by a charge transfer process and the inhibition of corrosion occurs through the adsorption of the polymer on the metal surface.

### 3.5 UV reflectance studies

The film formation on the metal surface is also supported by reflectance studies for different specimens under similar conditions. Figure 7 shows the reflectance curve for polished mild steel specimen, and polished specimens dipped in HCl in the absence and presence of the polymer. It can be seen that the reflectance is maximum for the polished specimen and it is brought down considerably in the case of specimens dipped in HCl. This clearly reveals that there is a change in surface characteristics due to the corrosion of mild steel in HCl alone. However, in the case of the specimen dipped in HCl containing the polymer, reflectance is decreased only to a very little extent. This observation clearly shows that surface characteristics are not changed very much in the presence of the polymer due to the formation of the film on the metal surface.

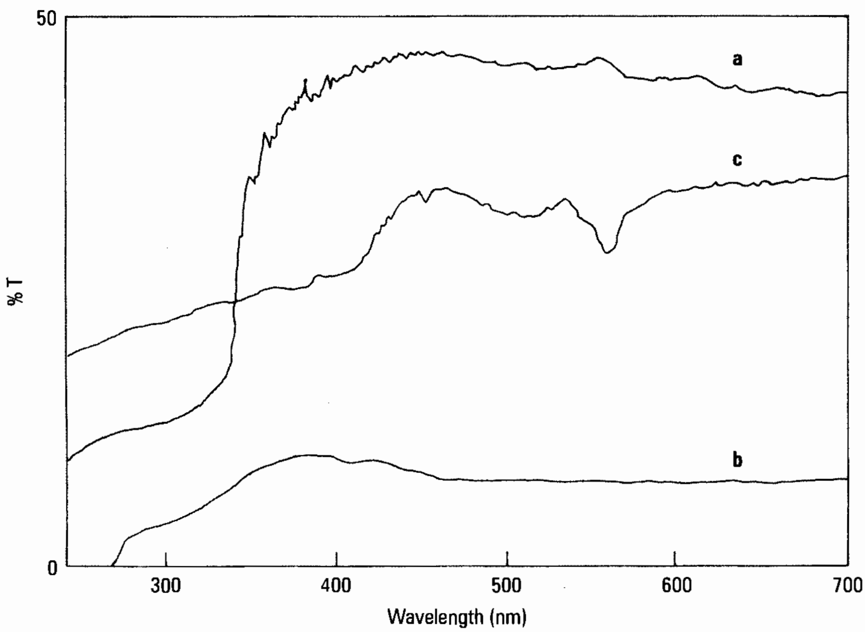
### 3.6 Adsorption isotherm

The nature of the interaction of the polymeric compound with the metal surface which enables the adsorption of the compound on the metal surface leading to corrosion inhibition can be deduced from the adsorption characteristics of the inhibiting compound. The coverage of the metal surface ($\theta$) by the compound is very useful in discussing the adsorption characteristics. For any concentration it can be calculated using the equation:

$$\theta = \frac{W_0 - W}{W_0} \quad (2)$$

Where $W$ and $W_0$ are metal losses in the presence and absence of the organic compound. Surface coverage data were tested graphically for a filling a suitable adsorption isotherm (Figure 8). A straight line

**Figure 7** UV-reflectance curves for mild steel specimens under different conditions.




**Key**
a Polished    b Dipped in 1M HCl
c Dipped in 1M HCl containing the polymer (60 ppm)

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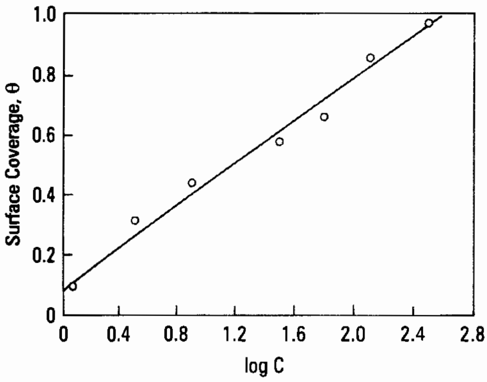
**Figure 8** Temkin adsorption isotherm




relationship was obtained when surface coverage ($\theta$) was plotted against log C, thereby clearly proving that the adsorption of this polymeric compound on the metal surface from 1.0M HCl obeys Temkin's adsorption isotherm.

## 4 Conclusion

The main conclusions drawn from the above study are:

(1) Poly(styrene sulphonic acid)-doped polyaniline inhibits the corrosion of mild steel in HCl.

(2) The inhibitor is found to affect both anodic and cathodic processes, but anodic process to a greater extent.

(3) The inhibition of corrosion of mild steel in HCl by the polymer can be attributed to the formation of a protective film on the metal surface.

(4) The polymer is able to bring down the extent of hydrogen entry into the metal.

(5) The adsorption of the polymer on the mild steel surface from 1M HCl obeys Temkin's adsorption isotherm.

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