Interaction of water soluble polyacrylic acid with mild steel / hydrochloric acid interface

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Abstract: Many of the polymers such as polyanilines, polyanthranilic acids have been reported as one of the efficient corrosion inhibitors for mild steel in acidic media. In view of the major limitation of insolubility of polymers, we have taken water soluble polyacrylic acid for the corrosion inhibition process. The corrosion inhibition property of polyacrylic acid is demonstrated for mild steel in 0.5 M hydrochloric acid using electrochemical impedance spectroscopy, Tafel polarization and weight loss methods. All the results are found to be in well correlation and the inhibition efficiency shows upto 94% in 0.5 M hydrochloric acid for 3hrs duration. Surface studies are also done by scanning electron microscopy.

Keywords: Polymer, Weight loss, Electrochemical calculation, Inhibition

1. Introduction

Deterioration and degradation of metal due to environmental circumstances is known as corrosion. Main cause of corrosion is environmental moisture which leads to the oxidation of metal surface. Metals in ores occur in mixed form of oxides, sulphates; carbonates etc. from those pure metals are extracted. These pure metals are highly unstable and react easily with oxygen and water contents in presence of carbon dioxide.

To reduce the effect of corrosive environment on metals corrosion inhibitors are used. These inhibitors are coated over the metal surface to inhibit the contact of metal surface and moisture. These inhibitors are working on the phenomenon of adsorption and can be explained by mainly Langmuir adsorption isotherm [1]. There are different types of inhibitors used, such as heterocyclic organic compounds, organic dyes, drugs natural products etc. but most efficient corrosion inhibitors are soluble polymers.

Acids are widely used as pickling, descaling, cleaning and for other purposes in different industries [2-6]. Hydrochloric acid is highly corrosive to the metals or mild steel (alloy of pure metals). So, the efficiency of water soluble polymers as corrosion inhibitors is checked in the same solution of 0.5 M hydrochloric acid. Efficiency can be calculated by gravimetric analysis, potentiodynamic polarization and electrochemical impedance spectroscopy. This is also supported by the surface studies done by Atomic force microscopy.

In recent years, studies on corrosion inhibitors focused on different polymers as inhibitor such as polyaniline, polyanthranilic acid, poly-diphenyl amine etc. [7-9]. Most of the polymeric materials are insoluble in nature. This is the major drawback of polymeric material to be used as corrosion inhibitors. For this purpose polymeric materials can be sulphonated, which increases the solubility of polymeric material in acidic medium. Polymeric compounds interact with the metal surface through the pi bond present in moiety [10].

Various factors like steric hindrance, aromaticity and electronic structure of inhibitor molecule affect the adsorption of corrosion inhibitor compounds. In addition of those functional groups;
electron density at donor site, size of the molecule and molecular weight also affect the corrosion inhibition process.

Polymers are excellent corrosion inhibitors for metallic artifacts in acid environment [16-21]. Presence of a small quantity of polymers may be effective in inhibiting the corrosion of metals in aqueous acidic environment [22-23]. The highly insoluble nature of most of the polymers in aqueous medium is the major limitation in the application of corrosion inhibition. Mainly polymers were used as coating material instead of inhibitors. Recently, several attempts have been made to solubilize the polymers for the use of corrosion inhibitor [24-26].

In this article we present the inhibitive behavior of water soluble polyacrylic acid (PAA) on mild steel sample in 0.5M hydrochloric acid solution. The surface coverage of PAA over mild steel surface in corrosive environment was studied using Atomic force microscopic techniques.

2. Experimental:

Polyacrylic acid (PAA) was purchased from Merck India and sulphonated it in laboratory to increase solubility in water.

Weight loss and electrochemical studies were done on mild steel strips of composition reported elsewhere [1]. Hydrochloric acid (AR grade) procured from Merck and double distilled water is used for the dilution of aggressive medium in present study.

2.1. Weight loss Studies

Weight loss measurements were done on the 10cm² surface area of mild steel sample for 3hrs duration and all the experiments were conducted in 0.5M HCl at 35°C for 3 hrs duration. The complete studies were done in the conical flask having glass stopper with different concentration of PAA inhibitor to obtain the optimum inhibitor concentration. Such studies were conducted for optimum concentration of PAA on various temperature ranges, time interval, and concentration of acid. Inhibition efficiency (%) and surface coverage ($\theta$) of the PAA on mild steel were obtained by:

\[
I.E.(\%) = \frac{W_o - W_i}{W_o} \times 100
\]

\[
\theta = \frac{W_o - W_i}{W_o}
\]

$W_o$ and $W_i$ represent the weight loss of metal in the absence PAA inhibitor and in the presence of PAA inhibitor concentration respectively.

2.2. Electrochemical Studies

Three electrode cell assembly was used to conduct all the electrochemical studies including tafel polarization, linear polarization and electrochemical impedance spectroscopy [29, 30] at 35°C. Mild steel sample will be used as working electrode. 1 cm² area of the mild steel was exposed and the rest of the strip covered as reported elsewhere [1]. Standard calomel electrode is used as reference electrode whereas Counter electrode is made up platinum. All the experiments were conducted on Potenentiostat / galvanostat of Gamry instruments Inc. USA, (model G-300) and EIS software. All the electrochemical measurements were carried out after stabilization of the open circuit potential in the aerated environment.

Tafel polarization, linear polarization resistance and electrochemical impedance spectroscopy experiments were carried out on the detail parameters reported elsewhere [1]
3. Result and discussion:

3.1. Weight-loss method

3.1.1. Effect of inhibitor concentration

Weight loss techniques were done for the various inhibitor concentration of PAA. The weight loss in mg, % inhibition efficiency and corrosion rate in mmpy were listed in table 1. Trend of the inhibition efficiency with respect to increase in PAA concentration graph is plotted in figure1. Tendency of increment of % inhibitor efficiency with the increase in PAA concentration in 0.5 M hydrochloric acid solution is already revealed from figure1. From table 1, it is clearly visible that the % inhibition efficiency increases and corrosion rate decreases with the increment of concentration of PAA. Maximum inhibition efficiency shown at 100 ppm concentration of PAA used i.e. upto 94.6%. The graphical representations of error bar have also been shown in figure1 represent the variability of data used to indicate the error or uncertainty in a reported measurement. It gives a general idea of mean, standard deviation and precision of measurement during triplicating observation.

Table 1. Corrosion inhibition data from weight loss studies of mild steel of surface area 10cm$^2$ for 3hrs at 35°C in aqueous acidic solution of 0.5N hydrochloric acid in absence of inhibitor and in presence of various concentrations of Polyacrylic acid.

| S.No | Inhibitor concentration (ppm) | Weight loss (mg) | IE (%) | CR (mmpy) |
|------|------------------------------|------------------|--------|-----------|
| 1.   | Nil                          | 81.05            | -      | 30.18     |
| 2.   | 15                           | 41.82            | 48.4   | 15.57     |
| 3.   | 25                           | 27.95            | 65.5   | 10.41     |
| 4.   | 50                           | 16.85            | 79.2   | 6.27      |
| 5.   | 75                           | 8.59             | 89.4   | 3.20      |
| 6    | 100                          | 4.37             | 94.6   | 1.63      |

Figure 1. Change in % inhibition efficiency with the varying concentration of Polyacrylic acid in 0.5M HCl on the surface of mild steel at the temperature 35°C in 3hrs immersion time

3.1.2. Effect of Immersion time
Figure 2 shows the % inhibition efficiency of PAA against the variation in immersion time. From the figure, it is clearly revealed that the inhibition efficiency slowly decreases with increase in time interval. This result is suggesting that the PAA inhibitor is adsorbed firmly on the surface of mild steel and the very slow rate of desorption of inhibitor molecule [27].

![Inhibition Efficiency vs Time](image1.png)

**Figure 2.** Change in % inhibition efficiency in 0.5M hydrochloric acid on the surface of mild steel of 10cm² of exposed area with different time intervals at 100 ppm concentration of PAA at 35°C temperature

3.1.3. Effect of solution temperature

![Inhibition Efficiency vs Temperature](image2.png)

**Figure 3.** Trend in % inhibition efficiency in 0.5M HCl on the surface of mild steel of 10cm² exposed areas at varying temperature range for 3hrs immersion time at optimum concentration of PAA inhibitor
Effect of temperature on % inhibition efficiency graph is shown in figure 3. This was studied at the 100 ppm of the concentration of PAA at wide temperature range of 298-328K. It is evident from graph that the % inhibition efficiency is lowering with increase in the temperature of the aqueous acid solution of hydrochloric acid. Decrease in the % inhibition efficiency with the rise of solution temperature may due to the desorption of the molecule of PAA inhibitor from the surface of mild steel [1]

3.1.4. Effect of acid concentration

Figure 4 clearly shows the trend of % inhibition efficiency with the variation of the acid concentration. It is clearly evident from the figure 4 that the inhibition efficiency is decreasing with the increase in the hydrochloric acid concentration. This clearly indicates that the PAA is a very good corrosion inhibitor for the less acidic medium. It gives best result in 0.5M hydrochloric acid solution.

![Figure 4. Trend in % inhibition efficiency on surface of mild steel of exposed area 10cm² at 35°C at 100 ppm concentration of PAA immersed for 3 hrs with different acid concentration](image)

3.1.5. Adsorption isotherm

“Adsorption isotherm study is the tool to explain the behavior of adsorption of PAA molecule on the surface of the metal to know the mechanism of adsorption. Langmuir, Temkin, and Frumkin are the most widely used isotherms to explain the mechanism of adsorption” [9]. The surface coverage values (θ) required for the adsorption isotherm calculation was deduced from the weight loss parameters given in table 1.

In all the three isotherms were tested and Langmuir adsorption isotherm were found most appropriate adsorption isotherm to explain. Langmuir adsorption isotherm is expressed by:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh}$$

where, concentration of PAA corrosion inhibitor represented by $C_{inh}$ and equilibrium constant of adsorption is represented by $K_{ads}$ in acid medium. Straight trend line with the rigration coefficient (R) is almost unity which is obtained by $C_{inh} vs C_{inh}/θ$ graph (Figure 5). Unity value of R represent the best description of adsorption behavior through Langmuir adsorption isotherm [31,32].
3.2. Tafel polarization study:

Figure 6 represents the effect of PAA corrosion inhibitor on both anodic and cathodic polarization curves on the surface of mild steel in 0.5M hydrochloric acid solution. The potentiodynamic polarization study was carried out to understand kinetics of both anodic and cathodic reactions. After examination of figure 6, both the cathodic and anodic reactions were minimising with the addition of the PAA inhibitor in this study. This result gives the inference that inhibitor reduced the reaction of anodic dissolution and on the same time it suppresses the hydrogen evolution reactions on cathode.

Corrosion potential (E$_{corr}$) and corrosion current density (I$_{corr}$) like electrochemical kinetic parameters can be obtained from the extrapolation of both anodic and cathodic polarization curves and the values are reported in Table 2. Decrease in the corrosion current density (I$_{corr}$) by addition of inhibitor molecule. Variation in more than 85mV corrosion potential with respect to blank solution, results the cathodic or anodic type of inhibitor [33, 34]. In this study, change in corrosion potential between tafel plot uninhibited and inhibited sample is only 02mV which clearly indicates that the PAA is a mixed type of inhibitor. % inhibition efficiency calculated from tafel polarization is in good agreement with the weight loss results.

Table 2. Tafel polarization and linear polarization data for mild steel corrosion without and with PAA inhibitor in 0.5M aqueous hydrochloric acid solution

| Inhibitor Conc. (PPM) | Tafel data | Linear Polarization data |
|-----------------------|-----------|-------------------------|
|                       | $E_{corr}$ (mV vs SCE) | $b_a$ (mV dec$^{-1}$) | $b_c$ (mV dec$^{-1}$) | $I_{corr}$ (µA cm$^{-2}$) | IE (%) | $R_p$ (Ω cm$^2$) | IE (%) |
| Nil                   | 469       | 75                      | 150                  | 2140                  | -      | 20.1              | -      |
| 100                   | 467       | 69                      | 158                  | 148                   | 93.1   | 395.5             | 94.9   |
3.3. **Linear polarization Study:**

Polarization resistance parameters and % inhibition efficiency were calculated and reported in table 2. Calculation of Polarization resistance from the slope of the potential current lines by:

\[ R_p = A \frac{dE}{di} \]  \hspace{1cm} (7)

In above reaction A is area of the working electrode surface, dE and di is change in potential, and change in current respectively. It is clearly revealed from the data listed in table2 that the inhibition efficiency increases with the addition of corrosion inhibitor.

3.4. **Electrochemical impedance spectroscopy:**

Electrochemical impedance spectroscopy measurements were carried out to investigate the corrosion behavior of mild steel surface in 0.5 M aqueous hydrochloric acid with and without PAA inhibitor. A Nyquist plot of the impedance spectra of the corrosion of mild steel is shown in figure 7. In the figure 7, at high frequency a single semicircle has clearly been shown which attributed to charge transfer of the process of corrosion and the diameter of semicircle was increased with addition of inhibitor. In the figure 8 impedance spectra shows depressed semicircle under real axis and look like a depressed capacitive loop. This is due to the heterogeneity on the mild steel surface. This heterogeneity may arise by the surface roughness, distribution of the active sites dislocations or adsorption of the inhibitor molecules [35-37]. Electrochemical impedance data must be correlated with various theoretical circuit proposed by F. Mansfeld. The equivalent circuit having constant phase angle was introduced to explain the impedance data which is applicable to explain the iron / acid interface model [38]. From the fitting of equivalent circuit of electrochemical impedance parameters (\( R_p, R, Y_o \), and \( n \)) were obtained and reported in Table3. Constant phase angle calculation was done as reported by J.R. McDonold [39]. In the iron/acid interface, roughness or uneven current distributions on the electrode surface ideal capacitor behavior is not observed which results the dispersion in frequency [36,40,41]. \( C_{dl} \) values were derived and listed in table3 from the Constant phase element parameters calculated by using equation [42]:

\[ C_{dl} = (Y_o \cdot R^{1-n})^{1/n} \]  \hspace{1cm} (9)
It is clearly evident from the data represented in table 3 that the $R_t$ values increase with the addition of optimum concentration of PAA corrosion inhibitor. Increase in $R_t$ value is attributed that inhibitor molecule from a protective film on the interface of metal and solution. The higher value of $n$ in the inhibited sample in comparison to the uninhibited sample is due to the decrease in surface heterogeneity. Surface heterogeneity may decrease due to adsorption of PAA inhibitor molecules on the active adsorption sites [43]. Double layer capacitance ($C_{dl}$) values were decreased with addition of PAA inhibitor. Following equation shows the relation between Protective layer thicknesses ($d$) and double layer capacitance $C_{dl}$ [44]:

$$C_{dl} = \frac{\varepsilon \varepsilon_0}{d}$$

(10)

In above equation $\varepsilon$ is dielectric constant of protective layer and $\varepsilon_0$ is permittivity of free space.

**Table 3:** Electrochemical impedance parameters calculated without inhibitor and with the optimum concentration of PAA inhibitor molecule for the corrosion of mild steel in 0.5M hydrochloric acid solution

| Inhibitor concentration (PPM) | $R_s$ ($\Omega \text{ cm}^2$) | $R_p$ ($\Omega \text{ cm}^2$) | $Y_e$ ($\mu F \text{ cm}^2$) | $N$ | $C_{dl}$ ($\mu F \text{ cm}^2$) | IE (%) |
|------------------------------|------------------------------|------------------------------|-----------------------------|-----|-----------------------------|-------|
| Nil                          | 1.23                         | 8.3                          | 1181.5                      | 0.858 | 55                          | 55    |
| 100                          | 1.19                         | 260.2                       | 69.3                        | 0.812 | 27                          | 96.8  |

Inhibition efficiency data reported in Table 3 is in quite good agreement with the other techniques used in the studies.

**Figure 7:** Nyquist plot of corrosion of mild steel in absence and presence of optimum concentration of PAA inhibitor in 0.5M hydrochloric acid solution

**3.5. Surface Morphology of Specimen**

Surface morphology of the mild steel sample was studied before and after corrosion in presence and absence of the inhibitors. The surface morphology supported the formation of adsorbed polymer film over the steel samples. In the figure 8 (b) it is clearly shown in that the steel samples are getting cracks due to the acid corrosion. However, the presence of polyacrylic acid as inhibitor retard the corrosion and surface showed more smoothness in figure 8 (c) which is less smooth than figure 8 (a) which is more smooth as the polished one.
Figure 8. Surface morphological analysis through atomic force microscopy of mild steel sample

4. Conclusion:
- PAA exhibit good inhibition towards corrosion on the surface of mild steel in the medium of aqueous hydrochloric acid.
- From the potentiodynamic polarization techniques it is clear that the used PAA inhibitor exhibit mixed-type character and alter both the anodic as well as cathodic type of reactions.
- Increase in the PAA inhibitor concentration up to the optimum concentration of 100 ppm results the increase in inhibition efficiency.
- These studies suggest that the polyacrilic acid exhibit good corrosion inhibition property towards the mild steel surface in the lower concentration of aqueous hydrochloric acid solution.
- Good inhibition property of PAA against the mild steel corrosion was also supported by the atomic force microscopic characterization.

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