Inhibitory effects of some imines on the corrosion of mild steel in hydrochloric acid solution

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The inhibition effect of 4-[(E)-(phenylimino)methyl]phenol (PIP), 4-[(E)-(4-fluorophenylimino)methyl]phenol (FIP), 4-[(E)-(4-chlorophenylimino)methyl]phenol (CIP), 4-[(E)-(4-bromophenylimino)methyl]phenol (BIP) and 4-[(E)-(4-nitrophenylimino)methyl]phenol (NIP) on the corrosion of mild steel in hydrochloric acid was investigated. The corrosion inhibiting action was studied through weight loss and electrochemical techniques. The corrosion parameters such as anodic and cathodic Tafel slopes ($b_a$ and $b_c$), corrosion potential ($E_{corr}$), corrosion current densities ($I_{corr}$), surface coverage ($\theta$) and corrosion inhibition efficiencies ($\%IE$) were determined. The polarization measurements indicated that the inhibitors are of mixed type. The adsorption obeyed Langmuir adsorption isotherm. The inhibition efficiencies increased with increase in inhibitor concentration but decreased with increase in temperature and also with increase in acid concentration. The activation and thermodynamic parameters were calculated for different temperatures.

Keywords: Corrosion, Mild steel, Langmuir's isotherm, Imines, Thermodynamic parameters

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Mild steel is one of the industrially important metals and is inevitable to expose its surface to different industrial environments while in use. The steel undergoes corrosion to different extent depending on the aggressiveness of the medium or environment. Generally the service life of steel is enhanced by applying various corrosion control measures. Most of the available methods for controlling corrosion are based on modifying either the surface of the metal or local environment when the metal is exposed. In most of the industries like oil refineries, nuclear power plants, fertilizer and chemical etc. the steel is used as containers, steam generators, liquid carrying pipes and so on. In such cases the corrosion of steel is effectively controlled by the application of suitable inhibitors.

The well known corrosion inhibitors of steel are organic compounds containing elements like S, N, O and P in their functional groups with aromatic and hetero cyclic rings. Many such compounds have been developed as corrosion inhibitors in different media. Most frequently cited compounds are aniline derivatives, aromatic hydrazides, Schiff’s base compounds, thiourea derivatives and azoles. The reported compounds are adsorbed on the metal surface and provide a barrier between metal and environment thereby reducing the rate of dissolution of the corroding metal. The effectiveness of inhibition process is influenced by the nature and surface charge of the metal, the nature of medium, nature and chemical structure of inhibitor molecule. The study of the mechanism of the action of corrosion process is very helpful in developing new corrosion inhibitors.

The present investigation focuses on the study of inhibition action of five compounds namely 4-[(E)-(phenylimino)methyl]phenol (PIP), 4-[(E)-(4-fluorophenylimino)methyl]phenol (FIP), 4-[(E)-(4-chlorophenylimino)methyl]phenol (CIP), 4-[(E)-(4-bromophenylimino)methyl]phenol (BIP) and 4-[(E)-(4-nitrophenylimino)methyl]phenol (NIP) on the corrosion of mild steel in HCl. These were the condensation products of the reaction between $p$-hydroxy benzaldehyde and anilines substituted with halogens and NO$_2$. It is also aimed to predict the thermodynamic feasibility of adsorption of the inhibitor molecules on steel surface and to study their adsorption behaviour. The structures of inhibitors are given in the Table 1.
Experimental Procedure

Mild steel having the chemical composition C = 0.05, Mn = 0.35, P = 0.032, S = 0.033% and the remainder being Fe was used for all experimental investigations. Rectangular specimens of the size 5 x 1 x 0.06 cm were used for weight loss measurements. For potentiodynamic measurements, the working electrode was cut from mild steel and was embedded in chemical resistant epoxy resin, exposing 1 cm² area. The samples were mechanically polished with different grades of emery papers, degreased with trichloro ethylene, washed with double distilled water and finally dried. AR grade chemicals were used for the preparation of solutions.

Weight loss measurements were carried out by weighing the specimens before and after immersion in 50 mL acid solutions for 4 h in the absence and presence of various concentrations of PIP, FIP, CIP, BIP and NIP. Runs were also done at different acid concentrations (0.5, 1, 2 and 5 M) and at different temperatures (303, 313, 323 and 333 K). To know the durability of adsorbed layer, the effect of immersion time (1-12 h) and stirring effect was carried out. Duplicate experiments were performed in each case and the mean value of the weight loss was determined.

A conventional three electrode cell consisting of mild steel as working electrode (WE) with exposed area of 1 cm², Platinum foil as counter electrode (CE) and Saturated Calomel Electrode (SCE) as reference electrode was used for polarization studies. The anode and cathode potential values were measured under galvanostatic conditions.

The surface of mild steel specimen immersed for 4 h in 1 M HCl solution with and without the inhibitors has been investigated by using JEOL JSM 6380 LA Analytical Scanning Electron Microscope. The energy of the acceleration beam employed was 20 kv.

Results and Discussion

Weight loss measurements

Table 2 gives the corrosion rate and the inhibition efficiency calculated from weight loss measurement for different concentration of inhibitor for an immersion period of 1-12 h in 1 M HCl. The
inhibition efficiency, $% \text{IE}$ was calculated by applying the following relationship\(^{23}\):

$$\% \text{IE} = \frac{W_o - W}{W_o} \times 100 \quad \ldots (1)$$

where $W_o$ is the average weight loss of mild steel sample in 1 M HCl without inhibitor and $W$ is the average weight loss of the same in 1 M HCl containing the inhibitor. The $% \text{IE}$ increases with increasing inhibitor concentration whereas it decreases in the following order: PIP > FIP > CIP > BIP > NIP. The PIP has shown maximum inhibition of 92% at 100 mg L\(^{-1}\) concentration and NIP exhibited lowest inhibition of 71% at the same concentration. It is observed from Table 2 that all the compounds show increase in the $% \text{IE}$ with increase in immersion time from 1 to 4 h and further increase in immersion period has no effect. This shows the persistency of the adsorbed imines over the test period\(^{24}\). It was also observed that there was no appreciable change in $% \text{IE}$ with the stirring of the solution indicating the firm adsorption of inhibitor molecules on the surface of metal.

It is well known that the inhibitive action of organic compounds containing S, N and O is due to the formation of co-ordinate type bond between the metal and the lone pair of electrons of hetero-atom in the inhibitor. The tendency to form a co-ordinate bond and hence the extent of adsorption can be enhanced by increasing the effective electron density at the functional group of the additive. The effective electron density at the hetero atom of the functional group depends on the nature of substituents in aromatic ring.

The adsorption of imines on the metal surface occur either directly on the basis of donor-acceptor interactions between $\pi$-electrons of the inhibitor molecules and the vacant d-orbitals of iron surface or by the interaction of inhibitor with already adsorbed chloride ions\(^{25}\). The $% \text{IE}$ of PIP is more than NIP because the OH group, being electron donating increases the localization of lone pair of electrons on

**Table 2 — Corrosion parameters obtained from weight loss (mg) measurements in 1 M HCl containing different concentrations of inhibitors at 303 K**

<table>
<thead>
<tr>
<th>Inhibitor</th>
<th>Conc. (mg L(^{-1}))</th>
<th>1 h</th>
<th>2 h</th>
<th>4 h</th>
<th>8 h</th>
<th>12 h</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Wt % Wt % Wt % Wt % Wt %</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Nil</td>
<td>0 41.5 - 54.2 - 63.2 - 67.4 - 72.1 -</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>PIP</td>
<td>10 12.9 69 12.5 77 11.2 82 11.5 83 13 82</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>20 12 71 10.8 80 9.6 85 10.1 85 11.5 84</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>30 10.8 74 8.7 84 8.4 87 9.4 86 9.4 87</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>50 9.1 78 6 89 5.6 91 6.7 90 5.8 92</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>100 9.5 77 6 89 5.2 92 6.1 91 6.5 91</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>FIP</td>
<td>10 14.5 65 13.5 75 12 81 12.8 81 13 82</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>20 13.7 67 12.5 77 10.8 83 10.8 84 12.3 83</td>
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<td></td>
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<td></td>
</tr>
<tr>
<td></td>
<td>30 12.9 69 12.5 77 10 84 11.5 83 12.3 83</td>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>50 12.5 70 11.9 78 8.8 86 9.4 86 10.1 86</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>100 11.6 72 11.4 79 9.6 85 9.4 86 10.8 85</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CIP</td>
<td>10 18.3 56 19.5 64 18.4 71 18.9 72 20.2 72</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>20 16.6 60 16.8 69 16 75 17.5 74 18 75</td>
<td></td>
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<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>30 14.5 65 14.6 73 13.2 79 14.1 79 14.4 80</td>
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<td></td>
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<td></td>
</tr>
<tr>
<td></td>
<td>50 13.3 68 12.5 77 10 84 10.1 85 11.5 84</td>
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</tr>
<tr>
<td></td>
<td>100 12.9 69 11.9 78 11.2 82 11.5 83 11.5 84</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>BIP</td>
<td>10 18.7 55 20 63 18.4 71 18.9 72 21.6 70</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>20 15.8 62 16.3 70 15.2 76 16.2 76 18 75</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>30 13.7 67 14.1 74 13.2 79 13.5 80 15.1 79</td>
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<tr>
<td></td>
<td>50 12.5 70 11.9 78 10.8 83 11.5 83 12.3 83</td>
<td></td>
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</tr>
<tr>
<td></td>
<td>100 12.9 69 11.9 78 11.2 82 11.5 83 13 82</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NIP</td>
<td>10 20.3 51 21.7 60 23.6 63 23.6 65 26 64</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>20 19.5 63 20.6 62 20.4 68 22.2 67 23.1 68</td>
<td></td>
<td></td>
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</tr>
<tr>
<td></td>
<td>30 18.3 56 19 65 18.8 70 19.5 71 20.9 71</td>
<td></td>
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<td></td>
</tr>
<tr>
<td></td>
<td>50 17 59 17.3 68 17.2 73 18.2 73 21.2 72</td>
<td></td>
<td></td>
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</tr>
<tr>
<td></td>
<td>100 16.6 60 17.9 67 18.4 71 18.2 73 19.5 73</td>
<td></td>
<td></td>
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</tr>
</tbody>
</table>
nitrogen atom of \(-\text{C}=\text{N}−\) group which is in between phenyl and benzene rings but halides and \(-\text{NO}_2\) being electron withdrawing when present in benzene ring decrease the localization of lone pair of electrons on nitrogen atom. The electron density on nitrogen atom decreases in the order \(\text{PIP} > \text{FIP} > \text{CIP} > \text{BIP} > \text{NIP}\). Also the interaction of compound with metal and protection efficiency was in the same order.

The effect of HCl concentration on the \(\%\text{IE}\) of inhibitors is given in Table 3. The \(\%\text{IE}\) for the given concentration of the inhibitor increases in acid concentration up to 1 M and later decreases with increasing the concentration of the acid. At lower acid concentration up to 1 M, the adsorption of inhibitor molecules increase continuously, blocks the anodic sites which results in increase in \(\%\text{IE}\). But further increase in acid concentration up to 5 M causes decrease in \(\%\text{IE}\) for all the compounds. This is due to the increased aggressiveness of acid medium and dissolution of metal at anode sites which are not blocked by inhibitor molecules\(^{26}\).

**Polarization measurements**

Figures 1 and 2 show the effect of addition of the inhibitors BIP and NIP at different concentrations, as typical examples, on the anodic and cathodic polarization of mild steel in 1 M HCl. Similar curves were obtained for other compounds. Various corrosion parameters such as corrosion current density \(i_\text{corr}\), corrosion potential \(E_\text{corr}\), Tafel constants \(b_a\) and \(b_c\), inhibition efficiencies \(\%\text{IE}\) and surface coverage degrees (\(\theta\)) obtained from polarization measurements are given in Table 4.

The percentage inhibition efficiency \(\%\text{IE}\) and the degree of surface coverage, \(\theta\) values were calculated from the following equations\(^{27}\),

\[
\%\text{IE} = \frac{i_o - i_i}{i_o} \times 100 \quad \ldots(2a)
\]

<table>
<thead>
<tr>
<th>Solution</th>
<th>Weight loss (mg)</th>
<th>Corrosion rate (mg cm(^{-2}) h(^{-1}))</th>
<th>Inhibition efficiency (% IE)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5 M HCl +</td>
<td>55.2</td>
<td>1.38</td>
<td>-</td>
</tr>
<tr>
<td>PIP</td>
<td>6</td>
<td>0.15</td>
<td>89</td>
</tr>
<tr>
<td>FIP</td>
<td>11.2</td>
<td>0.28</td>
<td>80</td>
</tr>
<tr>
<td>CIP</td>
<td>9.2</td>
<td>0.23</td>
<td>84</td>
</tr>
<tr>
<td>BIP</td>
<td>12</td>
<td>0.3</td>
<td>78</td>
</tr>
<tr>
<td>NIP</td>
<td>15.2</td>
<td>0.38</td>
<td>73</td>
</tr>
<tr>
<td>1 M HCl +</td>
<td>63.2</td>
<td>1.58</td>
<td>-</td>
</tr>
<tr>
<td>PIP</td>
<td>5.6</td>
<td>0.14</td>
<td>91</td>
</tr>
<tr>
<td>FIP</td>
<td>8.8</td>
<td>0.22</td>
<td>86</td>
</tr>
<tr>
<td>CIP</td>
<td>10</td>
<td>0.25</td>
<td>84</td>
</tr>
<tr>
<td>BIP</td>
<td>10.8</td>
<td>0.27</td>
<td>83</td>
</tr>
<tr>
<td>NIP</td>
<td>17.2</td>
<td>0.43</td>
<td>73</td>
</tr>
<tr>
<td>2 M HCl +</td>
<td>92</td>
<td>2.3</td>
<td>-</td>
</tr>
<tr>
<td>PIP</td>
<td>15.2</td>
<td>0.38</td>
<td>84</td>
</tr>
<tr>
<td>FIP</td>
<td>20</td>
<td>0.5</td>
<td>78</td>
</tr>
<tr>
<td>CIP</td>
<td>18</td>
<td>0.45</td>
<td>80</td>
</tr>
<tr>
<td>BIP</td>
<td>26</td>
<td>0.65</td>
<td>72</td>
</tr>
<tr>
<td>NIP</td>
<td>29.2</td>
<td>0.73</td>
<td>69</td>
</tr>
<tr>
<td>5 M HCl +</td>
<td>145.2</td>
<td>3.63</td>
<td>-</td>
</tr>
<tr>
<td>PIP</td>
<td>36</td>
<td>0.9</td>
<td>75</td>
</tr>
<tr>
<td>FIP</td>
<td>45.2</td>
<td>1.13</td>
<td>69</td>
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<tr>
<td>CIP</td>
<td>42</td>
<td>1.05</td>
<td>71</td>
</tr>
<tr>
<td>BIP</td>
<td>49.2</td>
<td>1.23</td>
<td>66</td>
</tr>
<tr>
<td>NIP</td>
<td>54</td>
<td>1.35</td>
<td>63</td>
</tr>
</tbody>
</table>

*Fig. 1 — Polarization curves of mild steel in 1 M HCl in the presence of different concentrations of BIP at 303 K*

*Fig. 2 — Polarization curves of mild steel in 1 M HCl in the presence of different concentrations of NIP at 303 K*
These results show that the inhibitors bring down $I_{\text{corr}}$ value at all concentrations and maximum decrease of $I_{\text{corr}}$ value was obtained at 50 mg L$^{-1}$ of the inhibitor concentration in 1 M HCl. It is obvious from the polarization curves that both anodic as well as cathodic curves shift towards lower current density values in the presence of inhibitors indicating that the inhibitors act in such a manner that both anodic as well as cathodic reactions are influenced by it simultaneously almost to the same extent showing a mixed nature of inhibitor\(^{28}\).

**Adsorption isotherms**

Basic information on the interaction between the inhibitor and the metal surface is provided by adsorption isotherms. This can be obtained from degree of surface coverage ($\theta$) and fraction of unoccupied surface ($1 - \theta$) which is a function of exposed area. If the adsorption of the inhibitor follows the Langmuir adsorption isotherm, the adsorption rate should be proportional to the inhibitor concentration ($C_{\text{inh}}$) and the fraction of unoccupied sites ($1 - \theta$).

$$V_{\text{ads}} = k_{\text{ads}} (1 - \theta) C_{\text{inh}}$$

Inversely, the desorption rate is proportional to the fraction of occupied sites ($\theta$).

$$V_{\text{des}} = k_{\text{des}} \theta$$
At equilibrium, both rates are equal:

\[ k_{\text{ads}} (1-\theta) C_{\text{inh}} = k_{\text{des}} \theta \]

Consequently, the degree of surface coverage (\( \theta \)) is given by

\[ \theta = \frac{bC_{\text{inh}}}{1+bC_{\text{inh}}} \] ... (3)

where ‘\( b \)’ designates the adsorption co-efficient. The Eq. (3) can be formulated as,

\[ C_{\text{inh}}/\theta = \frac{1}{b} + C_{\text{inh}} \] ... (4)

The plot of \( C_{\text{inh}}/\theta \) versus \( C_{\text{inh}} \) gave a straight line (Fig. 3) and thus followed the Langmuir adsorption isotherm\(^{29-31} \). This suggests that the inhibitor molecules are adsorbed over the metal surface forming a barrier which prevents further contact of the metal with electrolyte\(^{32} \).

**Effect of temperature**

The effect of temperature (303-333 K) on the corrosion inhibition of mild steel in 1 M HCl with and without inhibitors is shown in Table 5. It has been found that the %IE decreased with increase in temperature. Using Arrhenius equation

\[ \text{C.R.} = A \exp(-E_a/RT) \] ... (5)

Activation energy \( E_a \), has been obtained by plotting Log C.R. versus \( 1/T \) (Fig. 4). The activation energy as shown in Table 5 is higher in the presence of the inhibitor than in the absence. This reveals that higher activation energy reduces the rate of dissolution reaction of mild steel and it indicated the adsorption of inhibitor on metal surface. However, with an increase in temperature there is an appreciable decrease in the rate of adsorption of the inhibitor on the metal surface and a corresponding rise in the rate of dissolution reaction occurs\(^{33} \).

**Thermodynamic parameters**

The equilibrium constant \( ( K ) \) for adsorption and desorption process was calculated from the equation

\[ K = \frac{\theta}{C(1-\theta)} \] ... (6)

<table>
<thead>
<tr>
<th>Temp (K)</th>
<th>C.R. (mg.cm(^{-2}).h(^{-1}))</th>
<th>%IE</th>
<th>C.R. (mg.cm(^{-2}).h(^{-1}))</th>
<th>%IE</th>
<th>C.R. (mg.cm(^{-2}).h(^{-1}))</th>
<th>%IE</th>
<th>C.R. (mg.cm(^{-2}).h(^{-1}))</th>
<th>%IE</th>
<th>C.R. (mg.cm(^{-2}).h(^{-1}))</th>
<th>%IE</th>
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<tbody>
<tr>
<td>303</td>
<td>1.58</td>
<td>91</td>
<td>0.22</td>
<td>86</td>
<td>0.25</td>
<td>84</td>
<td>0.27</td>
<td>83</td>
<td>0.43</td>
<td>73</td>
</tr>
<tr>
<td>313</td>
<td>2.13</td>
<td>87</td>
<td>0.34</td>
<td>84</td>
<td>0.43</td>
<td>80</td>
<td>0.43</td>
<td>80</td>
<td>0.64</td>
<td>70</td>
</tr>
<tr>
<td>323</td>
<td>2.55</td>
<td>82</td>
<td>0.56</td>
<td>78</td>
<td>0.62</td>
<td>75</td>
<td>0.62</td>
<td>76</td>
<td>0.87</td>
<td>66</td>
</tr>
<tr>
<td>333</td>
<td>3.23</td>
<td>75</td>
<td>0.9</td>
<td>72</td>
<td>1.00</td>
<td>69</td>
<td>0.97</td>
<td>70</td>
<td>1.16</td>
<td>64</td>
</tr>
<tr>
<td>( E_a (kJ) )</td>
<td>18.4</td>
<td>46.0</td>
<td>40.4</td>
<td>39.8</td>
<td>35.8</td>
<td>29.1</td>
<td></td>
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</tbody>
</table>
The enthalpy of adsorption ($\Delta H$) was calculated from the plot of $\log K$ versus $1/T$. The thermodynamic parameters, free energy ($\Delta G$) and entropy ($\Delta S$) of adsorption were calculated from the following thermodynamic relations:

$$K = \frac{1}{55.5 \exp(-\Delta G/RT)}$$  \hspace{1cm}   \hspace{1cm} \ldots(7)

and

$$\Delta G = \Delta H - T\Delta S$$  \hspace{1cm}   \hspace{1cm} \ldots(8)

The calculated values of $K$, $\Delta H$, $\Delta G$ and $\Delta S$ over the temperature range of 303-333 K are recorded in the Table 6. The value of $K$ decreases with increasing temperature indicating that the interactions between adsorbed molecules and the metal surface are weakened and consequently, the adsorbed molecules become easily removable. Such data explains the decrease in % $IE$ with increase in temperature. The negative values of $\Delta H$ and $\Delta G$ and positive values of $\Delta S$ indicate the spontaneous adsorption of the additives on the surface of mild steel. Decrease in enthalpy and free energy is highest for PIP and lowest for NIP indicating PIP is readily adsorbed. This agrees with the observed %$IE$ (Table 2).

**Scanning electron micrograph**

Representative scanning electron micrographs...
(SEM) of mild steel surface that are formed in 1 M HCl solution without and with PIP as inhibitor are shown in Fig. 5. In the absence of the inhibitor the surface was covered with a high density of pits (Fig. 5a). In the presence of the inhibitor the micrograph shows no evidences of pitting but shows the formation of thick films on the steel surface (Fig. 5b). This result is due to the adsorption of inhibitor molecules around the pits. This passive film blocks the active sites present on the iron surface.

**Conclusions**

From the above results and discussions, the following conclusions are drawn:

(i) All the imines are proved to be efficient inhibitors for corrosion of mild steel in 1 M HCl. The order of inhibition efficiency is PIP > FIP > CIP > BIP > NIP. All are mixed type inhibitors.

(ii) The inhibition efficiency was found to increase with increase in the inhibitor concentration but decreases by increasing the temperature and acid concentration.

(iii) The %IE obtained from polarization measurements show good agreement with those obtained from weight loss measurements.

(iv) Adsorption of the compounds on mild steel obeyed Langmuir adsorption isotherm.

(v) The thermodynamic parameters indicate spontaneous adsorption of inhibitor molecules. However, increase in temperature will weaken the forces of attraction between inhibitor molecules and metal surface that results in decrease of %IE.

**References**