

Research Article

Experimental and Theoretical Studies on the Corrosion Inhibition Performance of a Synthesized Schiff Base on the Corrosion of Mild Steel in HCl Solution

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Abstract

Corrosion inhibition performance of a synthesized Schiff base was studied on the corrosion of mild steel in 1.0 M HCl using weight loss and electrochemical methods. The results indicated that the synthesized Schiff base can act as an effective inhibitor in reducing the corrosion of mild steel in 1.0 M HCl solution. The inhibitor efficiency of inhibitor increased with inhibitor concentration and showed maximum inhibition efficiency at 300 ppm concentration. The adsorption of inhibitor molecules obeyed Langmuir Adsorption isotherm. The adsorption mechanism involved both physisorption and chemisorptions. The potentiodynamic polarization studies showed that the inhibitor is a mixed typed inhibitor with more cathodic nature.

Potential of zero charge was also determined and the adsorption mechanism discussed. Quantum mechanical studies showed that the Schiff base molecules have the strong tendency to donate electron pairs to the metallic atoms on the surface.

Keywords: Corrosion, Mild Steel, Inhibitors, PZC, Quantum chemical Studies.

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Introduction

Iron metal and its alloys have most promising features to be used in engineering applications due to their remarkable properties, effective cost and high strength [1]. However, the use of mineral acids in many industries for various applications causes loss of functional properties of metals and alloys due to aggressive reaction. The protection of metals and alloys from corrosion thus, becomes very important to reduce waste of resources and money during industrial applications [2]. Mild steel is widely used in storage tanks, petroleum refineries, oil refining, chemical processing etc., [3-6]. The main problem of using mild steel is its dissolutions in acidic solution [7]. Organic compounds containing oxygen, nitrogen and sulphur atoms in their molecular structure have been found to be of great practical importance in reducing corrosion of metals in acid solution [8-15]. The efficiency of an organic compound to act as corrosion inhibitor generally depends on electron density around the hetero atoms, π electron density, molecular size, mode of adsorption, formation metallic complexes, etc., [16]. Among the organic compounds used as corrosion inhibitors, Schiff bases, possessing azomethine linkage ($-C=N$), find innumerable advantages in corrosion science. The popularity of Schiff bases in the field of corrosion inhibition is due to ease of synthesis from relatively inexpensive starting materials and their eco-friendly or low toxic properties [16-20]. The high inhibitory performance of these compounds resulted from the substitution of different heteroatoms (e.g. N, O, Cl & Br) and π electrons in their structure besides the presence of imine ($-C=N-$) functional group. These molecules normally form a very thin and persistent adsorbed film which leads to decrease in the corrosion rate due to the slowing down of anodic or cathodic reaction or both [16]. Moreover, the adsorption of the organic inhibitors onto the metal surface mainly depends on some physicochemical properties of the molecule which is related to its functional groups, possible steric effects and electronic density of donor atoms [21].

In the present work an attempt has been made to investigate the corrosion inhibition performance of a synthesized Schiff base 2PPT on the corrosion of mild steel in 1.0M HCl solution by weight loss and electrochemical methods. To understand the mechanism of corrosion inhibition, potential of zero charge (PZC) was measured in the presence and

absence of inhibitor film on the mild steel surface. Quantum chemical studies were also made to correlate the observed inhibitor efficiency with the structure of the inhibitor molecule.

Experimental

Materials

Mild steel coupons of dimensions 2.5cm x 1cm x 0.1cm having the composition 0.081%C, 0.035%Mn, 0.028%P, 0.022%S and the remainder being Fe were used for weight loss studies. The mild steel coupons were polished mechanically using the emery papers of grades 220, 400, 600, 800 and 1200, then washed thoroughly with double distilled water. Finally, the specimens were rinsed in acetone and dried. A Teflon coated mild steel rod of exposed area 0.2826cm² and having the composition same as that of the coupons was used for both impedance and polarization studies. Solutions of 1.0 M HCl with and without inhibitor were prepared from the analytical grade reagents using double distilled water. The inhibitor solutions were prepared in 1.0 M HCl solutions containing 5 volume % ethanol for solubility reasons.

Synthesis of Schiff base

2 pyrrole carboxaldehyde and p-toluidine were taken in 1:1 ratio in ethanol and refluxed for six hours in a water bath. The Schiff base formed was separated by vacuum distillation. The structure of Schiff base is given in **Figure 1**. The ethanol solution of the product obtained was distilled to remove the unreacted reactants. The observed disappearance of the characteristic peaks for >C=O and N-H groups at 1675cm⁻¹ and 3347cm⁻¹, respectively, in the IR spectra of the initial compounds along with the appearance of a new peak at 1620cm⁻¹, in the FTIR spectrum (**Figure 2**), of the Schiff base, which is the characteristic of the >C=N- group, confirmed the formation of the Schiff base.

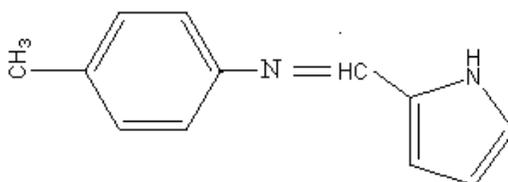


Figure 1 Structure of synthesized Schiff Base (2-PPT)

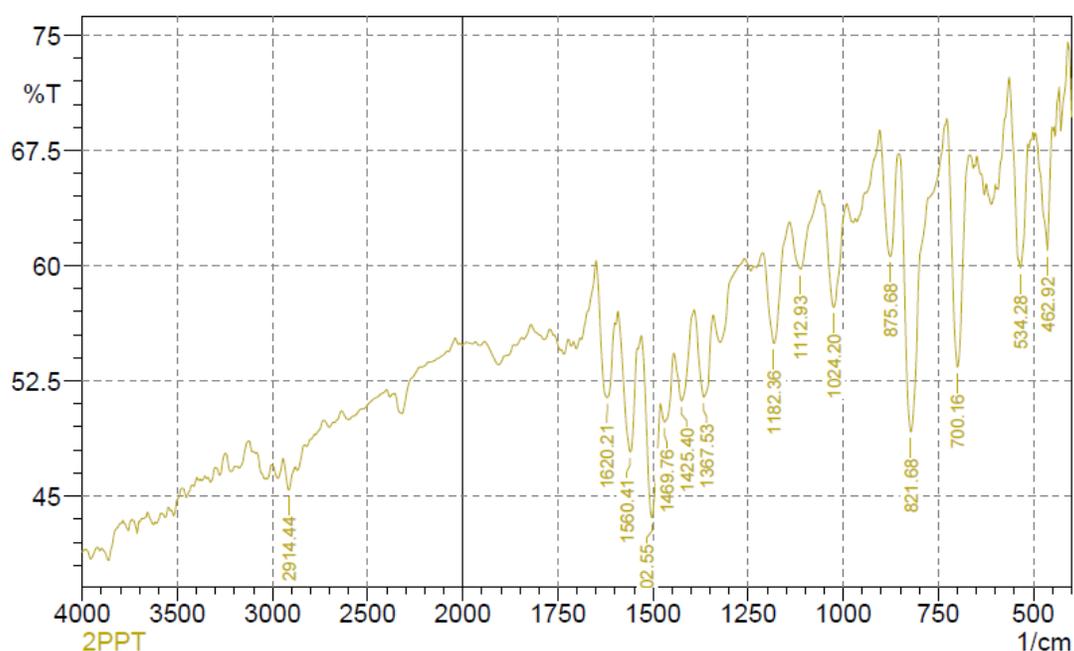


Figure 2 FTIR Spectrum of 2-PPT.

Weight loss measurements

Weight loss measurements were carried out according to the ASTM standard procedure described in [13]. The mild steel specimens in triplicate were immersed for a period of 2 hours in 100ml of the corrosive media with and without inhibitors at room temperature in 1.0 M HCl solutions. The average weight loss of the three specimens was used to calculate the inhibition efficiency employing the formula;

$$IE\% = \left(\frac{w-w'}{w} \right) \times 100 \quad (1)$$

Where w and w' are the weight losses in the uninhibited and inhibited solutions respectively. In the present study, at room temperature the relative difference between replicate experiments have been found to be less than 4% showing good reproducibility.

Impedance measurements

Electrochemical impedance measurements were carried out using a Gamry Reference 3000. The impedance data were analysed using Gamry EChem Analyst software. A three electrode set up was employed with a Pt foil as the auxiliary electrode and a saturated Calomel electrode as the reference electrode. The mild steel cylinder, with surface preparations done as mentioned in the weight loss method, served as the working electrode. The measurements were carried out in the frequency range 10^6 Hz to 10^{-2} Hz at the open circuit potential by superimposing a sine wave ac signal of small amplitude 10mV. The double layer capacitance (C_{dl}) and charge transfer resistance (R_{ct}) were obtained from Nyquist plots as described elsewhere [22]. Since R_{ct} is inversely proportional to corrosion current density, it was used to determine the inhibition efficiency (IE%) from the relationship;

$$IE\% = \left(\frac{R_{ct}' - R_{ct}}{R_{ct}'} \right) \times 100 \quad (2)$$

Where R_{ct} and R_{ct}' are the charge transfer resistance values in the uninhibited and inhibited solutions respectively.

Polarization measurements

The potentiodynamic polarization curves were recorded using the same cell setup employed for impedance measurements. The potentials were swept at the rate of 1.67mVs^{-1} , primarily from more negative potential than E_{oc} to the more positive potential than E_{oc} through E_{corr} . The inhibition efficiencies were calculated using the relationship [23];

$$IE\% = \left(\frac{i_{corr} - i_{corr}'}{i_{corr}} \right) \times 100 \quad (3)$$

where i_{corr} and i_{corr}' are the corrosion current densities in the absence and in the presence of inhibitor respectively in the corrosive media.

Measurement of potential of zero charge

The electrochemical impedance spectra were recorded at 200 Hz with the AC amplitude of 10mV for different applied DC potentials. The double layer capacitance values obtained were plotted against the applied DC potentials to determine the potential of zero charge

Results and discussion**Weight loss measurement**

The corrosion inhibition efficiency values calculated using weight loss data for mild steel corrosion in 1.0 M HCl medium at different concentrations of the inhibitor are presented in **Table 1**. It is seen that the inhibition efficiency increases with increase in inhibitor concentration. This behavior can be explained on the basis of strong interaction of the inhibitor molecule with the metal surface that results in adsorption [24]. The extent of adsorption increases with

increase in concentration of the inhibitor leading to increased efficiency. The acid solutions the maximum inhibition efficiency was observed at 300 ppm of inhibitor concentration. Generally organic inhibitors suppress the metal dissolution by forming a protective adsorbed film on the metal surface and separate it from the corrosion medium [25-29]. The inhibiting nature of the inhibitor molecule originates from the tendency to form either strong or weak chemical bonding with Fe atoms using the lone pair of electrons present on the O and N atoms and the π electrons of $>C=N$ - moiety and aromatic rings [16].

Table 1 Inhibitor efficiency of 2 PPT in 1.0 M HCl for the mild steel corrosion

S.No.	Concentration of Inhibitor (ppm)	Corrosion rate (mpy)	Inhibitor efficiency (IE%)
1	Blank	178	-
2	25	49.48	72.20
3	50	38.09	78.60
4	100	31.84	82.11
5	200	29.53	83.41
6	300	17.77	90.01

Electrochemical impedance spectroscopy

The impedance spectra recorded at room temperature in the presence and in the absence of inhibitor in 1.0 M HCl solution are shown in **Figure 3**. The Nyquist plots are simple semi circles, free from inductive loop etc. The electrical structure of any heterogeneous interface can be best described using an equivalent circuit, which is very useful to obtain the electrochemical impedance parameters of the system under study [30]. The simple $-R(CR)$ - model is expected to best describe the situation at the metal/solution interface. But the complex plane plots obtained are depressed semi-circles showing a non-ideal capacitive behavior of the electrochemical solid/liquid interface [7, 30]. This capacitance dispersion at the solid surfaces is attributed to the surface roughness, degree of polycrystallinity, chemical inhomogeneities and anion adsorption [31]. A precise modeling of the experimental behaviour is given by replacing capacitor (capacitive element) by a constant phase element (cpe) in the equivalent circuit (**Figure 4**), which has the impedance function represented by the expression [31];

$$Z_{cpe} = \frac{1}{Y_0 (j\omega)^n} \quad (4)$$

Where Y_0 is the admittance of the corrosive system at 1 rad s^{-1} and n is a constant ($-1 \leq n \leq 1$). When $n = 0$, the cpe represents a pure resistor, if $n = +1$ the cpe represents a pure capacitor and if $n = -1$ it represents an inductor [32].

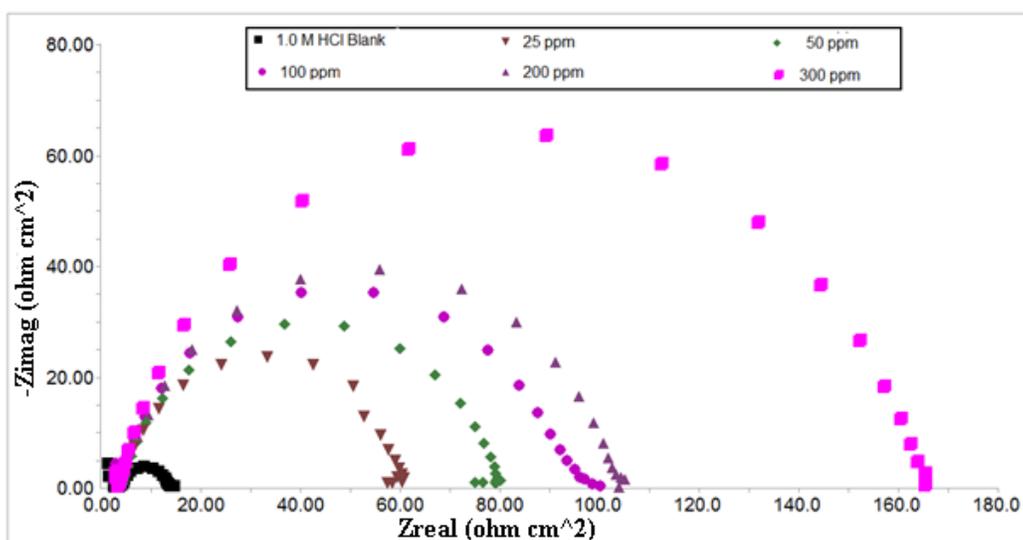


Figure 3 Nyquist plots for mild steel in 1.0 M HCl in the absence and presence of 2 PPT

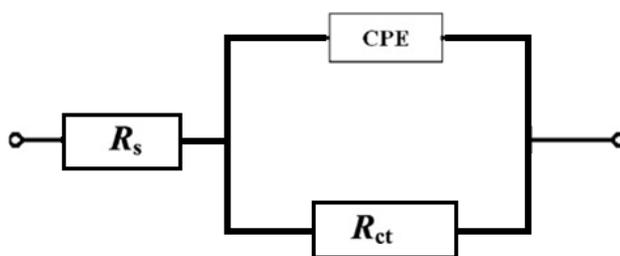


Figure 4 Equivalent circuit for mild steel corrosion in 1.0 M HCl solution.

As it can be seen from **Table 2**, the values of 'n' for the corroding electrode is 0.902 in 1.0M HCl and it increases with increase in inhibitor concentration. The value of 'n' is 0.952 at 300 ppm concentration. This indicates that the CPE behaves nearly as a capacitor. From the cpe parameter the idealized capacitance value (C_{id}) is calculated using the expression [33, 34];

$$C_{id} = \frac{Y_0 \omega^{n-1}}{\sin\left(\frac{n\pi}{2}\right)} \quad (5)$$

Where ω is the angular frequency at the maximum value of imaginary part of impedance spectra ($-z''$). It is further seen that this value of angular frequency is calculated using the expression;

$$\omega = \left(\frac{1}{R_{ct} Y_0}\right)^{\frac{1}{n}} \quad (6)$$

Hsu and Mansfeld [32] used the following equation to calculate the double layer capacitance;

$$C_{dl} = Y_0 \omega^{n-1} \quad (7)$$

Table 2 Electrochemical Impedance parameters of mild steel corrosion in 1.0 M HCl in the absence and presence of inhibitor.

S.No.	Concentration of Inhibitor (ppm)	$R_{ct} \Omega \text{ cm}^2$	$Y_0 \mu\Omega \text{ cm}^{-2}$	n	$C_{dl} \mu\text{F cm}^{-2}$	Inhibitor efficiency (IE%)
1	Blank	17.30	323.1	0.902	128	-
2	25	61.20	134.8	0.903	49.3	71.70
3	50	78.32	91.25	0.912	43.7	78.32
4	100	82.05	74.22	0.922	38.3	82.05
5	200	83.31	47.52	0.919	29.4	83.31
6	300	89.41	39.81	0.935	25.1	89.41

As it is seen from Table 2, the R_{ct} values increased and the values of C_{dl} decreased with increase in inhibitor concentration both in 1.0 M HCl solution. The increase in R_{ct} values with increase in inhibitor concentration (Figure 3) is the result of an increase in the surface coverage by the inhibitor molecules that led to an increase in inhibitor efficiency [26, 35].

The decrease in C_{dl} values with increase in inhibitor concentrations is due to the adsorption of Schiff base with water replacement at the metal solution interface that led to decrease in local dielectric constant and/or an increase in the thickness of the electrical double layer [35]. Hence, the change in C_{dl} values caused by the gradual displacement of water molecules by the adsorption of the Schiff base at the metal solution interface decreases the extent of the corrosion [36].

Polarization measurements

The polarization curves for mild steel in 1.0 M HCl solutions in the absence and in the presence of inhibitor are shown in **Figure 5**. From this figure it is clear that the addition of inhibitor to corrosive media changes the anodic and cathodic Tafel slopes showing the influence of the inhibitor both in cathodic as well as anodic reactions. However, the influence was more pronounced in cathodic polarization plots compared to anodic polarization plots. The electrochemical parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}) and cathodic and anodic slopes (β_c and β_a), surface coverage and the inhibitor efficiency are given in **Table 3**. Even though β_c and β_a values change with increase in inhibitor concentrations, the higher β_c value is higher than β_a [37] and the E_{corr} is also shifted to more negative side. This indicated that the Schiff base reduces cathodic reaction to a higher extent than the anodic reaction. This is attributed to the decrease in the rate of hydrogen evolution reaction on mild steel surface caused by the adsorption of the Schiff base on the metal surface [38].

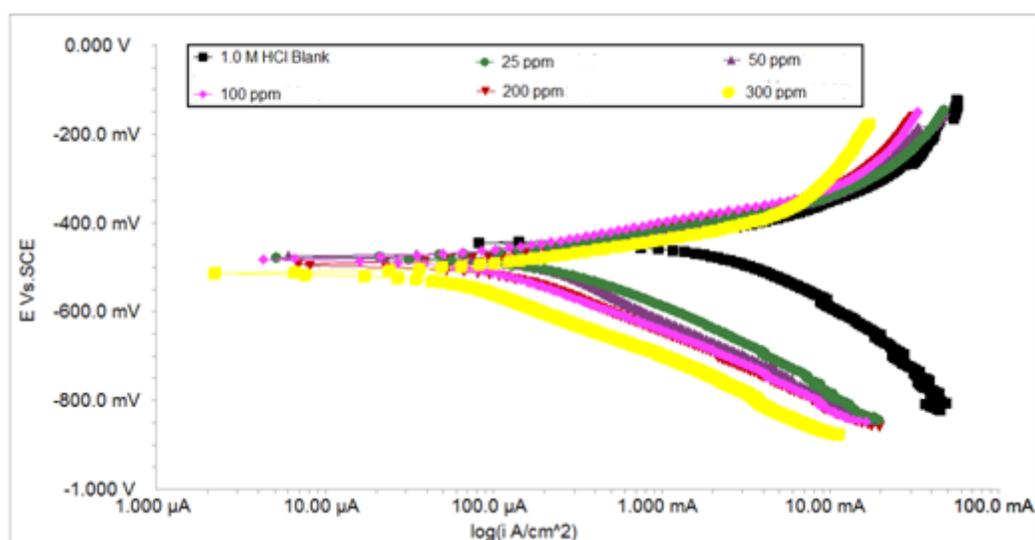


Figure 5 Tafel plots for mild steel in 1.0 M HCl in the absence and presence of 2 PPT

Table 3 Electrical parameters measured from potentiodynamic polarization studies on corrosion of mild steel in 1.0 M HCl in the presence and absence of inhibitor

S.No.	Concentration of Inhibitor (ppm)	E_{corr} (mV/SCE)	I_{corr} (mA cm ⁻²)	β_c	β_a	Inhibitor efficiency (IE%)
1	Blank	-450	1.96	227	135	-
2	25	-463	0.601	219	109	69.30
3	50	-468	0.443	198	98	77.42
4	100	-471	0.356	218	96	81.84
5	200	-481	0.345	219	90	82.39
6	300	-504	0.215	216	91	89.03

Potential of zero charge and inhibition mechanism

Organic inhibitors generally get adsorbed onto the metal surface. Their adsorption mainly depends on surface charge of the metal, the charge or dipole moment of the inhibitor molecule and the other ions that are specifically adsorbed onto the metal surface [39]. The surface charge of the metal is defined by the position of open circuit potential with respect to the potential of zero charge [40]. The dependency of double layer capacitance on the applied DC potential is graphically represented in **Figure 6a** and **6b**. The values of PZC and E_{ocp} for mild steel in the inhibited and uninhibited solutions of 1.0M HCl are given in **Table 4**. The surface charge of the mild steel at the open circuit potential was found out using the equation $E_r = E_{\text{ocp}} - E_{\text{PZC}}$, where E_r is Antropov's "rational" corrosion potential [37]. The surface charge of mild steel at the open circuit potential was found to be positive both in the inhibited and uninhibited 1.0 M HCl solutions. Based on the surface charge, the following mechanisms have been proposed for the

corrosion of mild steel in the inhibited and uninhibited HCl solutions [36-38]. In the uninhibited HCl solution, the mechanism of anodic dissolution involves the reversible adsorption of the anions (Cl^-) onto the mild steel surface, release of electrons from the anions adsorbed onto the metal surface and desorption of adsorbed species with Fe^{+2} ions after picking up electrons from the mild steel surface [36,38]. The schiff base molecules existing in the protonated form through nitrogen atom in 1.0 M HCl solution are in equilibrium with the corresponding molecular (unprotonated) form. Thus, it is very difficult for the positively charged inhibitor to approach the positively charged metal surface because of electrostatic repulsion. The protonated molecules can get adsorbed onto the mild steel surface via Cl^- ions that form interconnecting bridges between the positively charged metal surface and the protonated organic inhibitors [41,42]. In addition to this type of physical adsorption, the adsorption of unprotonated form of schiff base can also take place through donor acceptor interactions between π electrons of $>\text{C}=\text{N}-$ group, benzene ring and the lone pair of electrons on N and S atoms present in the unprotonated form of schiff base and the vacant 'd' orbitals of surface iron atoms.

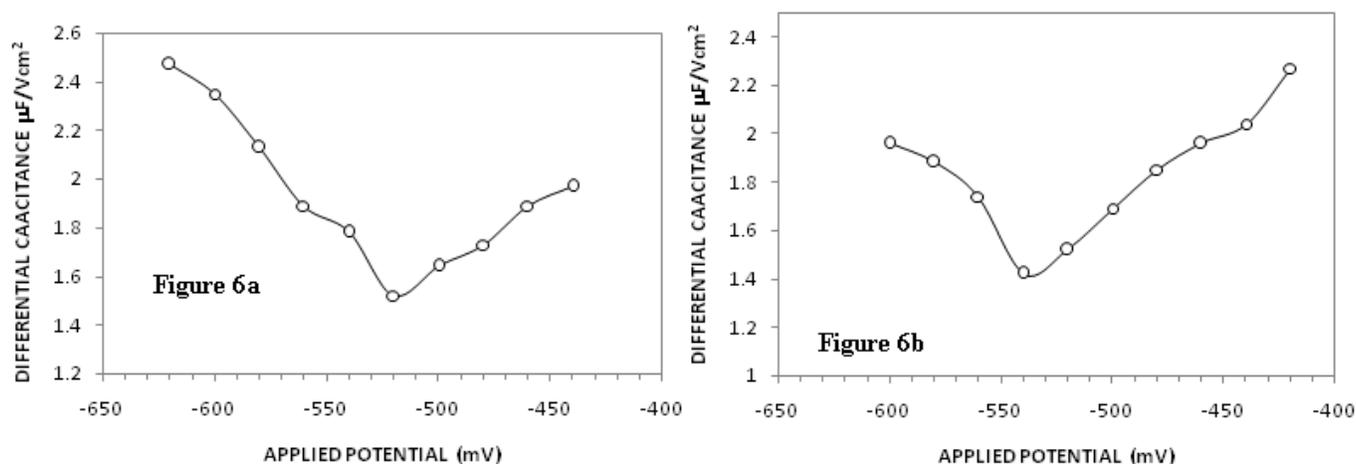
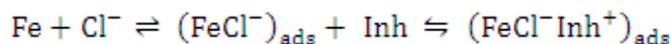


Figure 6 Plot of Applied potential vs differential capacitance for mild steel in 1.0 M HCl (a) and 1.0 M HCl containing 300 ppm of 2 PPT (b)

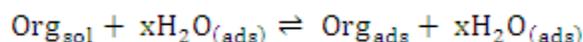
Table 4 Excess Surface charge on mild steel surface in 1.0 M HCl in the absence and presence (300 ppm) of inhibitor

S.No	Medium	E_{OCP} (mV/SCE)	PZC (mV/SCE)	Excess charge
1	1.0 M HCl	-450	+70	+
2	1.0 M HCl + 300 ppm of 2 PPT	-504	+36	+



Adsorption isotherm

The inhibitive action of the Schiff base molecules in the acid media for mild steel is due to its adsorption at the metal solution interface, which can be regarded as a substitution adsorption process between organic compound (org sol) in the aggressive media and the water molecules on the metallic surface $\text{H}_2\text{O}_{(\text{ads})}$ [43].



Where 'x' is the size number representing the number of water molecules replaced by an adsorbate molecule of the Schiff base. For organic compounds, which impede the metal dissolution reaction in acid media, the surface coverage can be evaluated as a function of inhibitor efficiency (IE%). The link between surface coverage and inhibitor efficiency at constant temperature gives an insight into the adsorption process.

In the present study various adsorption isotherms were tested and it was found that the adsorption of Schiff base on the mild steel surface in acid media follows the Langmuir adsorption isotherm [44], given by the expression

$$\frac{C_{inh}}{\theta} = C_{inh} + \frac{1}{K_{ads}} \quad (8)$$

Where C_{inh} is inhibitor concentration, θ is fraction of total surface covered by inhibitor molecules and K_{ads} is equilibrium constant. A plot of C_{inh} vs C_{inh}/θ is shown in Figure.7.

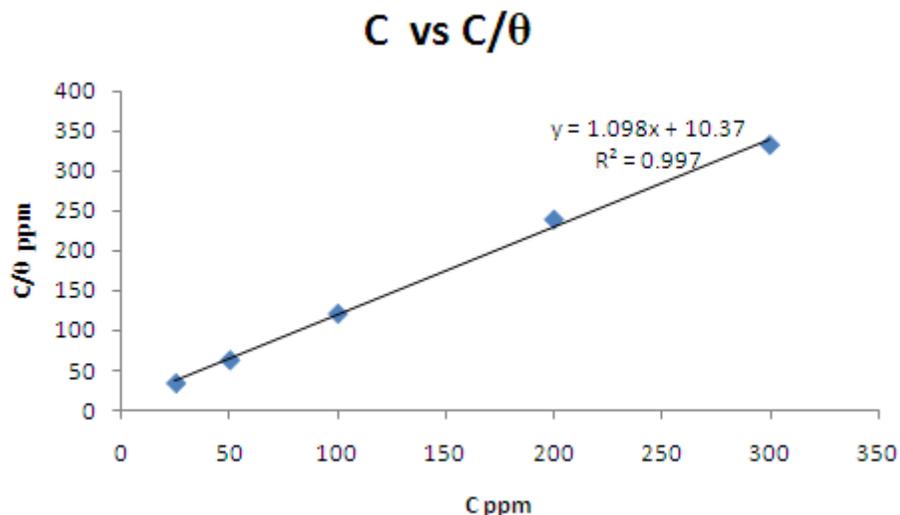


Figure 7 Langmuir's Isotherm for adsorption of inhibitor molecules onto mild steel surface in 1.0 M HCl solution.

The value of K_{ads} , determined from the plot of C_{inh}/θ vs C_{inh} at constant temperature is used to calculate the value of standard free energy of adsorption (ΔG_{ads}) using the expression [45]

$$K_{ads} = \frac{1}{55.5} \exp\left(\frac{-\Delta G_{ads}}{RT}\right) \quad (9)$$

The plots obtained are linear in both the media with a correlation coefficients higher than 0.99. The calculated value of ΔG_{ads} at room temperature in 1.0 M HCl is $-38.17 \text{ kJmol}^{-1}$. The negative values of ΔG_{ads} show the strong interaction and spontaneous adsorption of the inhibitor molecules on to the mild steel surface [39, 41]. In general the values of $-\Delta G_{ads}$ upto 20 kJmol^{-1} or less than 20 kJmol^{-1} imply the coulombic electrostatic interaction between the charged molecules and the charged metal surface. The values greater than 40 kJmol^{-1} imply formation of chemical bond between inhibitor molecule and metal surface through charge sharing or charge transfer [46]. The calculated ΔG_{ads} values close to -40 kJmol^{-1} and far from -20 kJmol^{-1} . This indicates that the adsorption of Schiff bases on to the mild steel surface involves both physical as well as chemical interactions [47-50].

Molecular structure and Quantum Chemical investigations

Quantum chemical method has strong influence to design and develop corrosion inhibitors. Molecular geometries and electron distributions can be calculated precisely using density functional theory (DFT) [7]. Figure 8a and 8b show the electron populations in the HOMO and LUMO of the inhibitor molecule. From these figures it is seen that the electron density is high in E_{HOMO} and it can donate electrons to the surface metal atoms to form dative bond.

Conclusion

The adsorption and inhibition effect of the synthesized Schiff base on the corrosion behavior of mild steel in 1.0 M HCl solution was studied by weight loss and electrochemical methods from which the following conclusions have been arrived.

- In acid medium, the inhibitor efficiency of the newly synthesized Schiff base increases with increase in inhibitor concentration and maximum inhibitor efficiency was observed at an optimum concentration of 300 ppm.
- The results obtained show that the synthesized Schiff base can act as a good inhibitor for the corrosion of mild steel in 1N HCl.
- The inhibition efficiencies calculated from weight loss, electrochemical impedance and potentiodynamic polarization studies show the same trend.
- The potentiodynamic polarization curves indicated that the Schiff base acts as a mixed type of inhibitor with more polarized cathodic curves than anodic curves.
- The adsorption of the Schiff base onto the metal surface follows Langmuir adsorption isotherm. The negative values of the ΔG_{ads} indicate that the adsorption process is spontaneous and exothermic.
- The value of free energy of adsorption is very close to -40kJmol^{-1} and far away from -20kJmol^{-1} , indicating that the adsorption of the inhibitor onto the metal surface follows both electrostatic coulombic attraction and chemisorption.
- Determination of PZC indicated the formation of chloride inter connecting bridges between the protonated Schiff base molecules and the metal surface.
- Quantum chemical studies indicated that the molecule has a strong electron donating tendency to the metal.

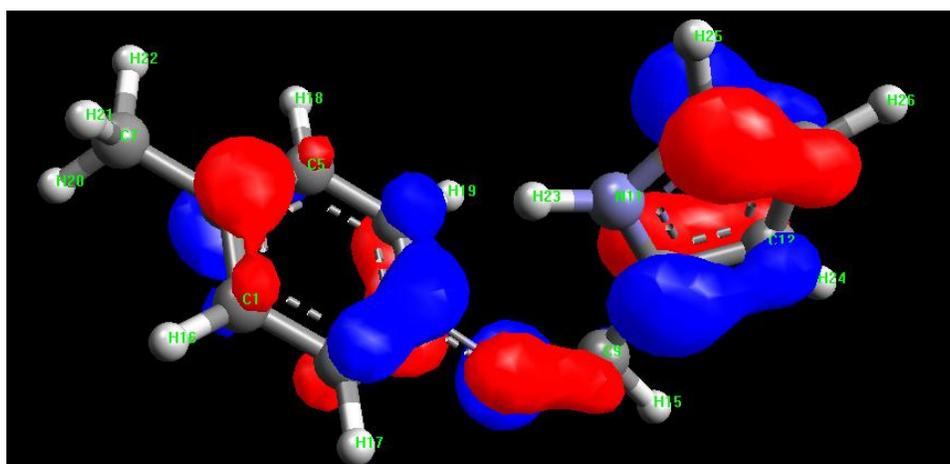


Figure 8a (HOMO)

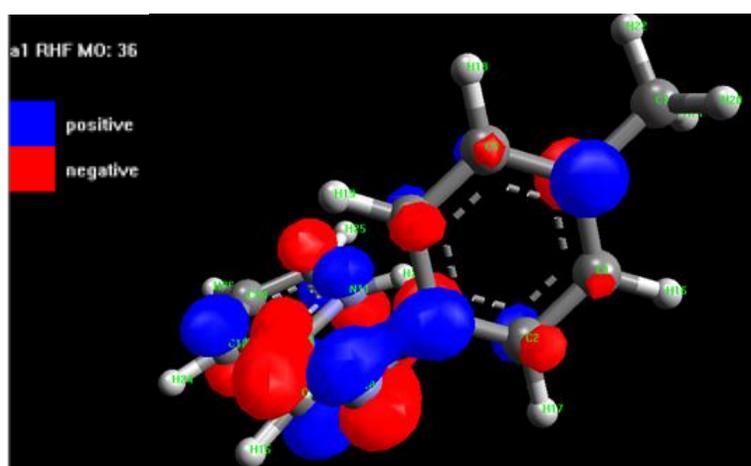


Figure 8b (LUMO)

Figure 8 Stereo view of inhibitor molecule.

Some molecular properties calculated using DFT are given in **Table 5**. E_{HOMO} is often related with electron donating ability of inhibitor molecule, high value of E_{HOMO} indicates the greater tendency of inhibitor molecule to donate electron pairs to acceptor with lower empty orbitals. E_{LUMO} on the other hand indicates the ability of the molecule to accept the electrons [7, 49]. The gap between HOMO and LUMO determines the reactivity of the molecule. From Table 5 it is clear that the inhibitor molecules higher electron donating ability. The parameter ω , electrophilicity, measures the electron accepting power of the molecule, which is low in the present work.

Table 5 Molecular properties calculated using quantum chemical studies.

Total energy (eV)	-1761.20385
Electronic energy (eV)	-9651.26418
core-core repulsion (eV)	7890.06032
ionization potential (eV)	8.904975
HOMO (eV)	-8.905
LUMO (eV)	-0.319
Energy gap (eV)	8.586
Electron affinity (A = - LUMO) (eV)	0.319
Electronegativity $\chi = ((I+A)/2)$ (eV)	4.6119875
Global hardness $\eta = ((I-A)/2)$ (eV)	4.2929875
Softness (1/ η)	0.232938018
Dipole moment $\mu = -\chi$	-4.6119875
Electrophilicity (w)	2.477345753

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