# Schiff's base Fufural Phenylhydrazone as a Potential Corrosion Inhibitor for Mild Steel in Hydrochloric Acid Solution

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*Abstract: Heterocyclic Schiff base furfural phenylhydrazone [FPH] was prepared and formulated as a corrosion inhibitor for mild steel in 2M hydrochloric acid solution. Mass change measurement and electrochemical methods adapted to study the effectiveness of the FPH during the corrosion process. FPH inhibitor protected 94.53 % corrosion of mild steel at optimum inhibitor strength of 0.0007 M at 303 ±1 K. Route of corrosion protection was interpreted through adsorption of FPH molecules on specimen surfaces in acid solution. Nature of the adsorption was established via Langmuir adsorption isotherm. Stability of the inhibitor was investigated with higher temperatures. Tafel polarization curves revealed, FPH molecules exhibit mixed nature of inhibitor. SEM and AFM images suggested that corroded specimen surface was severely affected in free acid comparatively in presence of FPH inhibitor. FT-IR analysis proved that, chemical interaction takes place between specimen surface atoms with FPH molecules and established chemical bond between them.*

*Keywords: Schiff base, Corrosion inhibitor, Tafel plots, Corrosion rate.*

## I. INTRODUCTION

In various industrial processes mineral acids like hydrofluoric acid, hydrochloric acid, nitric acid, chloroacetic acid and sulfuric acid are most preferably used in order to improve oil/gas recovery and also to removal of iron oxides and salt deposits [1, 2]. Mild steel is also an extensively utilized important structural & industrial material in various chemical industries due to its natural availability, toughness and possessed better-quality mechanical properties [3]. These materials react with acids to form stable compounds, in which the loss of metals take place and the metal surface becomes corroded, leading to enormous economic and material wastes. Corrosion can be controlled or reduced by means of various ways [4, 5]. For controlling the corrosion of metallic substrate, different approaches such as coating and lining, cathodic protection and use of corrosion inhibitors frequently applied in a variety of industrial applications [6]. Several researchers reported that molecules of organic inhibitors possessed N, O, & S hetero atoms, imine group, multiple bonds and aromatic rings are effectively interact to surface of metals and reduce the corrosion in acidic solutions [7, 8].

Recently many Schiff's bases had been developed as inhibitors for various metals and alloys in acidic media and these inhibitors contain heterocyclic atoms, aromatic systems and electronegative atoms in the molecule. Recent developed few Schiff's bases are 2,20-dithiobis(3-cyano-4,6-dimethylpyridine) [9], 1-methyl-3-pyridin-2-yl-thiourea [10], *N,N-ortho-phenyl*acetylacetone imine and 4-[(3-{[1-(2-hydroxy phenyl)methylene] amino} propyl] ethanemidol]-1,3-benzenediol [11] (NE)-N-(thiophen-3-ylmethylidene)-4-({4-[(E)-(thiophen-2ylmethylidene)amino] phenyl}methyl)aniline [12], 5-(4-Dimethylaminobenzylidene)rhodanine [13] and 2-(3-pyridyl)-3,4-dihydro-4-quinazolinone [14]. These compounds exhibited as good corrosion inhibitors for various metals and alloys.

The present investigation employed for the formulation of furfural phenylhydrazone (*FPH*) as a mild steel corrosion inhibitor in 2M HCl solution using mass change measurement and electrochemical technique. The investigations focused on the effect of concentration of *FPH* and temperature, characterization of surface image and metal-inhibitor interactions.

## II. MATERIALS AND METHODS

### A. Chemicals

Corrosive solution of 2M HCl prepared using AR (36-37%) HCl and used as corrosive medium. Stock solution of the *FPH* inhibitor was prepared by dissolving in a 2M HCl solution. From the stock solution, different concentrations of *FPH* solution were prepared in 2 M HCl then used for the corrosion studies

## B. *Furfural phenylhydrazone preparation*

Furfural phenylhydrazone *FPH* obtained from refluxing of equimolar alcoholic (ethyl alcohol) solution of furfuraldehyde and phenylhydrazine for two hours at laboratory temperature. Solution further concentrated, cooled and obtained product separated by filtration. The recrystallized product characterized by FT-IR analysis. Fig. 1 represents the molecular structure of *FPH*.
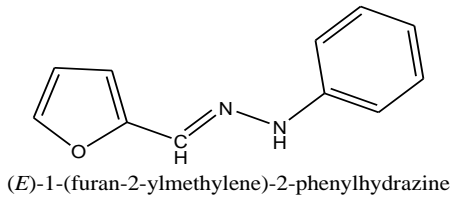


*(E)*-1-(furan-2-ylmethylene)-2-phenylhydrazine

Fig. 1 Molecular structure of Furfural phenylhydrazone

## C. *Electrodes*

Mild steel sheet with compositions of 0.025% C, 0.35% Mn, 0.022% P, 0.036% S and rest being Fe used. Mild steel of rectangular sheet was mechanically pressed cut into its required specimens of dimension 10mm $\times$ 50mm $\times$ 1mm and used for mass change determination. Mild steel specimen exposed area of 1cm$^2$ used for electrochemical determinations. Surface pretreatment process was done mechanically polished using different grades of abrasive papers no. 400, 600, 800, 1500 and 2000 followed by washing with distilled water, degreased with acetone and finally dried.

## D. *Mass change determination*

Mass change determination of test samples in free acid solution (2M HCl) and addition of different amounts of *FPH* in acid solution was performed and calculated the corrosion rate of test samples and percentage inhibition (% IE) of the inhibitor. The mass change in mg ($\Delta$W) of pretreated test samples dipped in 2M HCl with specified immersion time 't' in hours was determined using electronic digital balance. Similar mass change of test samples in presence of different concentrations of *FPH* was determined. Using the values of mass change in free acid and acid containing *FPH*, corrosion rate ($W_{corr}$) of test samples and percentage inhibition efficiency (% IE) of *FPH* were calculated by the Eq.(1) & (2).

$$W_{corr} = \frac{\Delta W}{St} \tag{1}$$

$$\% IE = 1 - \frac{W^1_{corr}}{W_{corr}} x100 \tag{2}$$

Where, 'S' is the specimen exposed surface area (cm$^2$), $W_{corr}$ & $W^1_{corr}$ are rates of specimen corrosion in free acid solution and in presence of *FPH* in acid solution [15, 16]

## E. *Electrochemical measurements*

Polarization and impedance behavior of mild steel in 2M HCl solution done using CHI608 workstation having frequency response analyzer set up at 303±1 K. Three-electrode electrochemical cell consisting of mild steel working electrode to measure the polarised potential of the working electrode against reference electrode (SCE). Platinum electrode used as a counter electrode and hydrochloric acid solution used as electrolyte as well as corrosive medium at room temperature (301 ± 1K). Prior to polarization, the experiment conducted after the specimen immersed for 30 minutes in HCl solutions without and with addition of inhibitor. Tafel curves are constructed by varying electrode potential from -0.2 V to + 0.2 V versus open circuit potential (OCP) at a scan rate of 1.0 mVs$^{-1}$ [17 - 19]. Various parameters like corrosion potential ($E_{corr.}$), cathodic ($\beta_c$) and anodic ($\beta_a$) Tafel slopes and corrosion current density ($I_{corr.}$) obtained by extrapolating the Tafel lines were evaluated. The % IE obtained by the Eq. (3).

$$\% IE = 1 - \frac{I^1_{corr}}{I_{corr}} \quad x\, 100 \tag{3}$$

Where, $I_{corr}$ & $I^1_{corr}$ represents corrosion current density in free HCl solution and HCl solution containing different concentrations of inhibitor respectively.

In electrochemical impedance spectroscopy (EIS), without altering external sources the electrode is allowed to attain steady state potential (OCP) as a function of 180 seconds corresponding to the reference electrode. Electrochemical impedance measurements made from 100 kHz to 10 MHz frequency range with 10 mV amplitude AC signal. Double layer capacitance (Cdl), resistance polarization ($R_p$) & solution resistance ($R_s$) determined from Nyquist plots and percentage inhibition efficiency calculated from ($R_p$) using the Eq. (4).

$$\% IE = 1 - \frac{R^1_p}{R_p} \quad x\, 100 \tag{4}$$

Where $R^1_p$ and $R_p$ are the resistance polarization in presence and absence of the inhibitor respectively [20, 21].

## F. *Adsorption study*

Adsorption studies were very much useful to confirm the mode of corrosion protection by the used inhibitors. Experimental results used to fit with Temkin, Freundlich and Langmuir's adsorption isotherms.

## G. *SEM, AFM & FT-IR Studies*

Surface morphology change of mild steel during corrosion protection with *FPH* was monitored by scanning electron microscopy (SEM: Model-JEOL make, JSM-IT 500LA).The corrosion protection of mild steel in attendance of *FPH* was established via adsorption process. The interaction of the *FPH* functional groups on mild steel surfaces is confirmed through FT-IR analysis using a

Nicolet 6700 FT-IR spectroscopic analyzer with a working range of 400 - 4000 cm$^{-1}$ wave number [22, 23].

## III. RESULTS AND DISCUSSION

### A. Mass change determination

Mass loss of mild steel in 2M HCl solution was found to be more due to the reaction taking place at the metal - solution interface, whereas in case of addition of inhibitor *FPH* in 2M HCl, the mass loss was minimized. Using the values of mass loss in absence and presence of different concentrations of *FPH*, the corrosion rate and percentage inhibition efficiency of the inhibitor were calculated and the results were tabulated (Table I).

It was observed from Table I, the corrosion rate decreased continuously with rising concentration of *FPH* at 303 ± 1 K, meanwhile the % IE value increased continuously and reached maximum % IE of 92.30 at 0.0007 M inhibitor concentration. Additional rise in *FPH* strength causes negligible change in % IE and 0.0007 M treated as optimum *FPH* concentration [24]. In the presence of *FPH* molecules, the corrosion rate of mild steel decreased due to the adsorption of *FPH* on specimen surfaces [25].

### B. Electrochemical investigations

#### Polarization measurement

Potential change from the open circuit potential of a corroding system obtained using Potentiodynamic linear polarization technique. Fig. 2 show the anodic & cathodic polarisation curves in absence and presence of *FPH* inhibitor in 2 M HCl solution at 303 ± 1 K.. Corrosion potential ($E_{corr}$), corrosion current density ($I_{corr}$), Tafel slopes ($\beta_c$ & $\beta_c$) obtained from the anodic and cathodic Tafel lines were determined.

TABLE I  Values of $W_{corr}$ of mild steel and %IE of the inhibitor in 2M HCl at 303 ±1 K.

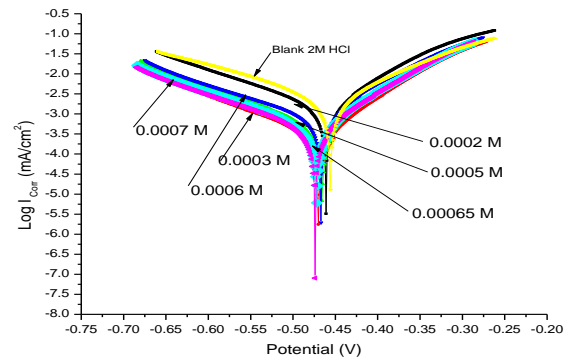| Conc. inhibitor (M) | $W_{corr}$ (mg./cm²/h) | %IE |
|---|---|---|
| Blank | 3.25 | - |
| 0.0002 | 1.5 | 53.84 |
| 0.0003 | 1.35 | 58.46 |
| 0.0005 | 0.95 | 70.70 |
| 0.0006 | 0.6 | 81.53 |
| 0.00065 | 0.45 | 86.15 |
| 0.0007 | 0.25 | 92.30 |



Fig. 2. Polarization curves for mild steel in 2M HCl solution in presence & absence of *FPH*

Using the values of $I_{corr}$ in presence and absence of the inhibitor, % IE was calculated using the equation (4). Table 2 denotes the various polarisation parameters for mild steel in 2M HCl at 303 ± 1K.

The results in Table II indicated that, in absence of the *FPH* inhibitor, the $I_{corr}$ value for mild steel is high and its value continuously decreased with addition of different concentrations of the *FPH* in acid solution. Percentage inhibition efficiency of the inhibitor increases with increase in the inhibitor concentration and reaches a maximum of 86.07 % at optimum 0.0007 M concentration at 303 ± 1 K. On the other hand *FPH* molecules blocked both anodic and cathodic reaction sites without much alter the cathodic & anodic Tafel slopes and worked as a mixed type of inhibitor [18, 26].

The mechanism action of corrosion inhibitor is based on the adsorption on the mild steel surface to form a protective molecular layer which displace water from the metal surface and protect it against deteriorating. At low concentration of the inhibitor, the metal surface partially covered by the inhibitor molecules, as the concentration of the inhibitor is enhanced, larger surface area occupied by the inhibitor molecules, which leads to greater reduction in the corrosion rate. The used inhibitor readily got adsorbed on metal surfaces due to the presence of electron rich centers in the inhibitor.

TABLE II Values of $I_{corr}$, $E_{corr}$, $\beta_c$ & $\beta_c$ and % IE of mild steel at 303 ± 1 K.

| Conc. (M) | $I_{corr}$ (µA cm$^{-2}$) | $E_{corr}$ (mV) | $\beta_c$ (mV dec$^{-1}$) | $\beta_a$ (mV dec$^{-1}$) | % IE |
|---|---|---|---|---|---|
| Blank | 2.6218 | -455.3 | 8.2 | -5.67 | - |
| 0.0002 | 1.2702 | -460.9 | 12.5147 | -6.9741 | 51.55 |
| 0.0003 | 0.8450 | -467.9 | 10.53 | -6.4546 | 67.77 |
| 0.0005 | 0.6514 | -464.9 | 14.5 | -6.673 | 75.15 |
| 0.0006 | 0.4798 | -466.0 | 11.8 | -7.477 | 81.69 |
| 0.00065 | 0.3918 | -470.5 | 12.33 | -7.063 | 85.05 |
| 0.0007 | 0.3650 | -473.2 | 12.30 | -7.0 | 86.07 |

## C. Electrochemical Impedance Spectroscopy (EIS)

EIS is the multifrequency measurement mode because it measures the electrical impedance of a metal solution and is offenely used to calculate the corrosion rate. Electrochemical AC impedance measurements were made for mild steel in 2M HCl solution and added with varying concentrations of *FPH* at $303 \pm 1$ K. Nyquist plots for mild steel are depicted in Fig. 3 [27].
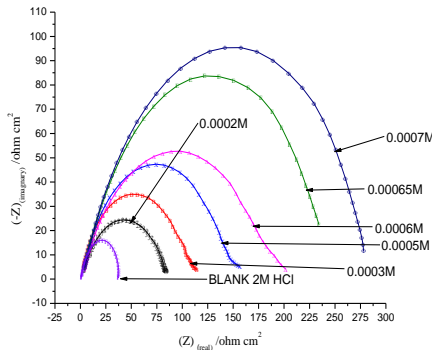


Fig. 3. Nyquist plots for mild steel in 2M HCl in presence and absence of inhibitor at $303 \pm 1$K

To study the impedance plots containing a single capacitance semicircle loop, the standard Randles circuit has been referred (Fig. 4). The circuit contains a solution resistance parameter ($R_s$), a constant phase element (CPE) which is parallel to polarization resistance component ($R_p$) [28]. The use of CPE-type impedance has been largely explained in accordance of equation (5) with corresponding reports [29, 30].

$$Z_{CPE} = [Y_0^{-1}(jw)^{-n}] \qquad (5)$$

## D. Temperature effect study

Temperature influence on corrosion rate and percentage inhibition efficiency of *FPH* was studied by mass change method at different temperatures [33]. Rate of corrosion ($W_{corr}$) and % IE at the optimized inhibitor concentration with immersion period of 2 hours and at temperature range (303 to $343 \pm 1$K) were mentioned in the Table IV.

Data from Table IV revealed that the corrosion rate was increased and percentage inhibition efficiency decreased as the temperature increased from $303 \pm 1$ to $343 \pm 1$ K. It revealed that, as the temperature increased the migration of $H^+$ ions enhanced toward metal face and induced to increase reduction reaction rate.

TABLE IV $W_{corr}$ of mild steel & % IE of *FPH* in 2 M HCl with 2 h immersion period.

| Temperature [$\pm 1$K] | $W_{corr}$ (2M HCl) (mg./cm²/h) | $W_{corr}$ (2M HCl + 0.0007 M *FPH*) (mg./cm²/h) | (%IE ) | $\Theta$ |
|---|---|---|---|---|
| 303 | 3.32 | 0.25 | 92.46 | 0.925 |
| 313 | 5.63 | 0.63 | 88.80 | 0.888 |
| 323 | 8.33 | 1.3 | 84.39 | 0.844 |
| 333 | 16.0 | 3.5 | 78.12 | 0.781 |
| 343 | 30.95 | 9.55 | 69.14 | 0.619 |

At higher temperature the stability of the *FPH* adsorbed layer weakened due to increase in the local thermal energy of the adsorbed molecules and start molecules detaching from the specimen surface, as a result, corrosion rate increased and % IE was decreased [34, 35].

## E. Adsorption isotherm study

Corrosion inhibition in presence of inhibitor was explained based on adsorption of inhibitor molecules on the surface of the mild steel. The adsorption of inhibitors depends on the nature of the metal, molecular structure and temperature of the medium. The metal atoms having vacant orbitals and electron rich centres possessed by the inhibitor molecules favor the adsorption route. The metal-inhibitor interactions providing important clues to the type of adsorption isotherm takes place such as Langmuir, Temkin, Frumkin and Freundlich [36].

The mild steel – *FPH* interactions fitted to the Langmuir adsorption isotherm given by the Eq.(6). This was confirmed by the graph plotted Log [$\Theta$ /1-$\Theta$] against Log $C_{inh}$ (Fig. 5) give a curve fitted in the graph is a straight line with regression coefficients is around unity ($R^2 = 0.9892$). Langmuir adsorption isotherm studies support that corrosion control tendency by the *FPH* molecules takes place via adsorption process.

$$\text{Log } [\Theta/1-\Theta] = \text{Log } K_{ads} + \text{Log } C \qquad (6)$$

Where C is the molar *FPH* concentration, '$\Theta$' is the degree of surface coverage and determined from the Eq. (7) and $K_{ads}$ is the equilibrium constant of the adsorption process (Eq. 8)

$$\Theta = 1 - \frac{W}{W_0} \qquad (7)$$

Where $W_0$ and W represent the mass loss of mild steel in uninhibited and inhibited solutions respectively.

$$K_{ads} = \Theta / C_{inh} (1 - \Theta) \qquad (8)$$

Here $Y_0$ is a proportionality factor, j is an imaginary number, and w is the angular frequency in rad$^{-1}$ (w = $2\pi f_{max}$), $f_{max}$ is the highest frequency of the imaginary component of the impedance. The degree of non-ideality in capacitance behavior and its value makes it possible to differentiate between the behavior of an ideal capacitor (n = 1) and of a CPE (n < 1) is obtained by the Eq. (5). This observation indicated that, the corrosion of mild steel retarded through a charge transfer controlled in an acid medium [31].

The values of resistance polarization ($R_p$), solution resistance ($R_s$), CPE, % IE and CPE exponent (n) were mentioned in Table 3. Change of n values as addition of *FPH* to the HCl media is an indication of change in the surface analogy by virtue of adsorption process of inhibitor on the utmost active place of mild steel.

The impedance of mild steel progressively increased as increasing the addition of inhibitor in acid solution. Further maximum % IE of 87.82 was attained at inhibitor concentration of 0.0007 M as it gives much larger semicircle diameter then the rest of lower concentrations of inhibitor [24, 32].
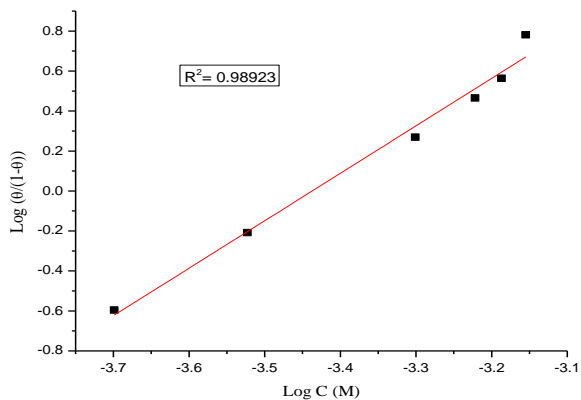
Fig. 5 Langmuir adsorption isotherms for the adsorption of *FPH* molecules on mild steel surface in 2M HCl

## F. Surface interactions studies

### FTIR Analysis

FT-IR spectral technique used to investigate the type of possible bonding interactions and to evaluate the protective layer formed between the organic inhibitor adsorbed on a mild steel surface in acid solution [21]. Fig. 6a is the FT-IR spectrum of recrystallized *FPH* sample and Fig. 6b is the FT-IR spectrum of scratched samples collected from the corroded metal surface containing optimized concentration of *FPH* inhibitor [23].

In Fig. 6a, the characteristic stretching bands at 3317.63 cm$^{-1}$, 1670.48 cm$^{-1}$ and 1598cm$^{-1}$ are indicated as N-H stretching (secondary amine), -C=N stretching and C-C stretching (aromatic) respectively.
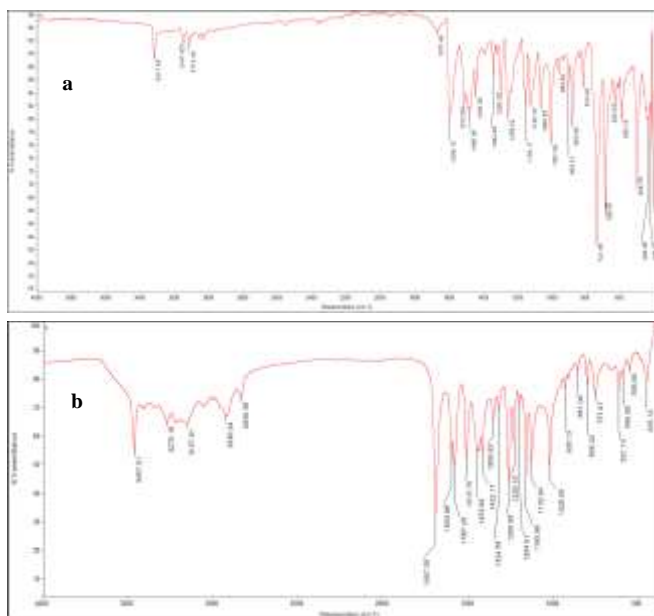


**Fig. 6 :** FT-IR spectra of - **a)** pure *FPH* compound and **b)** scratched sample from the corroded mild steel surface in 2 M HCl with 0.0007 M *FPH*

As in the Fig. 6b these characteristic stretching bands are slightly altered and appeared at 3467.61cm$^{-1}$, 1697 cm$^{-1}$ and 1603.88 cm$^{-1}$ corresponding to –N-H, -C=N, and C-C groups respectively. Such alteration of the corresponding

peaks from pure compound to the scratched sample denotes that nitrogen containing lone pairs of electrons can behave as active centers during the adsorption process on a metal surface. Such modified peaks revealed that *FPH* compound is chemically interplays on metal surfaces and controls deterioration process in HCl medium.

### SEM Analysis

Scanning electron microscopic technique opted to test the surface morphological observations of the corroded test samples. Fig. 7 provides the SEM analysis results for mild steel surface after 2 h immersion in 2M HCl and inhibited by *FPH* inhibitor. Fig. 7a shows the image of a polished mild steel surface and appeared to be smooth. Fig. 7b viewed the corroded sample surface in a 2 M HCl solution. In this case, the test sample surface was strongly damaged and contained several cavities, channels due the surface was severely affected by the acid solution. Fig. 7c specimen surface image with addition of 0.0002M *FPH* in 2M HCl, the surface becomes smoother and most of the surface covered by *FPH* molecules [3]. Figure 7d correspondence to SEM image of the corroded sample surface in presence of optimized inhibitor concentration. The maximum surface area was covered by the *FPH* molecules and the surface was least affected by the acid solution [6]. This illustration discloses that the inhibitor forms passive film, which strongly covers the mild steel surface and effectively controls the corrosion process [5].
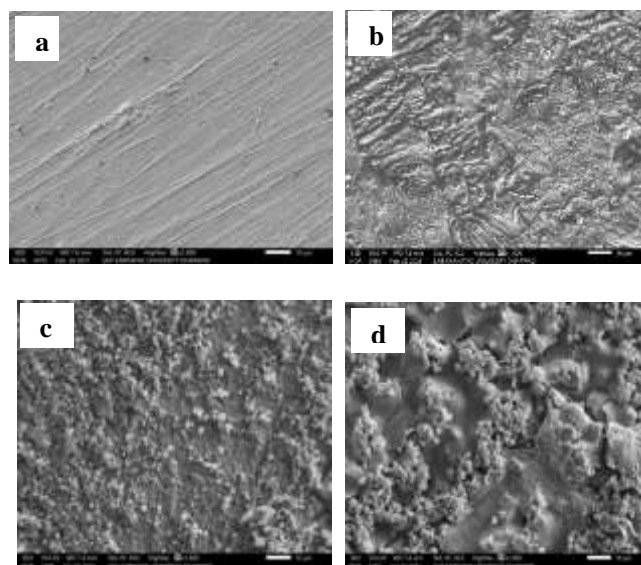


Fig. 7 SEM images of corroded mild steel surfaces dipped in 2 h at 303 K. a) Polished, b) 2M HCl, c) 2M HCl + 0.0002 M *FPH* and d) 2M HCl + 0.0007 M *FPH*.

### Atomic Force Microscope Analysis (AFM)

It is one of the most preferred tools while observing the mild steel surface morphology at nano level in corrosion field. Fig. 8a, b & c depicted the 3-D AFM images of polished, uninhibited and inhibited at higher concentration of *FPH* in 2M HCl [37]. In Fig. 8b AFM image visualized that, in absence of *FPH* the mild steel surface was strongly

damaged and shows rough structures with deep large pits due to the aggressive corrosion attack with calculated average roughness value of 443 nm.
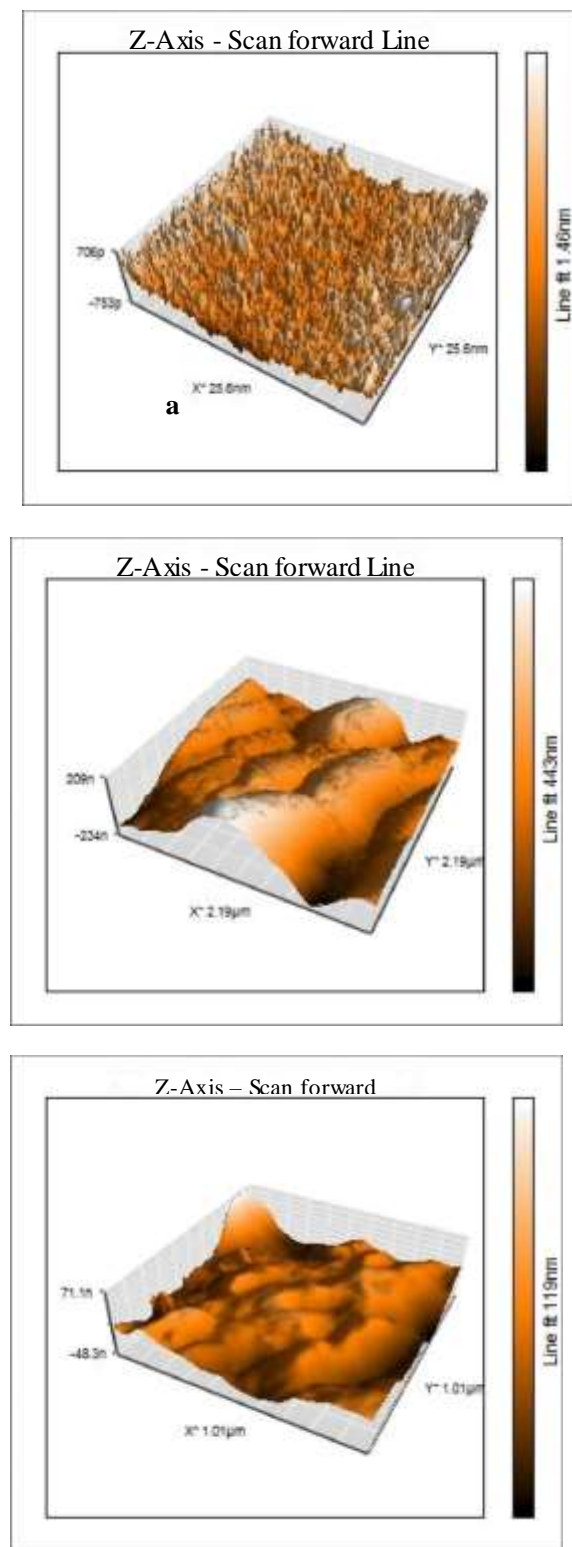
### Z-Axis - Scan forward Line

a

### Z-Axis - Scan forward Line

### Z-Axis – Scan forward

Fig. 8 AFM images of mild steel surfaces dipped in 2 h at 303 K.

a) Uninhibited, b) 2M HCl alone and c) 2M HCl + 0.0007 M *FPH*

With addition of *FPH* inhibitor, the AFM image shows mild steel surface become smoother, less damaged with calculated average roughness value found decreased to 48.60 nm (Fig. 8c) and indicating the corrosion rate of the mild steel specimen was markedly less [6].In the case of polished mild steel specimens (Fig. 8a), the average surface roughness was found to be 2.099 nm. Thus from above observation indicating mild steel protected by *FPH* inhibitor and thus could effectively reduce the metal surface dissolution [38].

**CONCLUSION**

The chemical and electrochemical experiments revealed that the prepared *FPH* exhibited an active corrosion inhibitor for mild steel in 2M HCl solution. The *FPH* molecule possessed heterocyclic ring, oxygen and nitrogen hetero atoms and imine group; these are act as active centers and effectively interact on the mild steel surface to form a strong barrier between metal surface and corrosive medium. The corrosion retardation of mild steel was explained based on the adsorption of *FPH* molecules (Chemisorptions) on the metal surface and obeyed the Langmuir adsorption model. The SEM and AFM images of corroded surfaces noticed that the specimen was least affected by the acid in presence of *FPH* inhibitor and appeared to have a smooth surface. FT-IR studies supported that active centers of the *FPH* molecules chemically interacted on metal surfaces and controlled the corrosion in acid medium. The synthesized *FPH* compound soluble in aqueous medium and can be used as corrosion inhibitor for mild steel in HCl solution.

Declaration of Conflict of Interest

The authors declare that they have no known conflict of interests.

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