Research Article

Corrosion Behaviour of Mild Steel in the Presence of Schiff Bases Derived from 2-Acetyl Pyridine in 0.5M H_2SO_4

Binsi M. Paulson, Joby Thomas K.

Department of Chemistry, St. Thomas College (Autonomous), Thrissur, Kerala

Corresponding author's email address: drjobythomask@gmail.com

ABSTRACT

Two heterocyclic Schiff bases namely (E)-2-(1-(2-phenylhydrazono)ethyl)pyridine (or 2- acetyl pyridine phenyl hydrazone) (2APPH) and (E)-2-(1-triazylidineethyl)pyridine (or 2-acetyl pyridine semicarbazone) (2APSC) were synthesized, characterized and their corrosion inhibition behaviour as well as mechanism of inhibition were investigated by different techniques. Structural characterization includes NMR, Mass, IR and UV-visible spectroscopy and elemental analysis. Corrosion inhibition behaviour of aforesaid compounds on mild steel in 0.5M sulphuric acid was examined by electrochemical methods including potentiodynamic polarization analysis, electrochemical impedance spectroscopy and noise studies. The mechanism of corrosion inhibition was explored and supplemented by adsorption and surface morphological studies.

Keywords: Schiff base, Corrosion Inhibitor, Polarization, Impedance, Adsorption

1. INTRODUCTION

The study of Schiff bases was considered as predominant research area to prevent the metal corrosion, since it is an important threat which can affect directly or indirectly on the economy[1-4]. Corrosion is the deterioration of metals and alloys by chemical or electrochemical means. Corrosion is considered as a universal phenomenon, omnipresent and omnipotent. In industrial fields, de-scaling, pickling etc. commonly employed for the metal surface cleaning, demands enormous amount of HCl and H_2SO_4 and leads to metal disintegration. Many organic compounds containing azomethine (C=N) linkage act as corrosion inhibitor in acid media[5-10]. Schiff bases are organic compounds containing azomethine linkage and have many applications in corrosion science. The hetero atoms present in these molecules are responsible for the corrosion inhibition property for mild steel (MS), aluminium, copper and zinc in acid media[11-18].

The present investigation was done to examine the corrosion inhibition efficiency and mechanism of two novel heterocyclic Schiff base molecules derived from 2-acetyl pyridine on MS in 0.5M sulphuric acid solution. The experimental study was performed by different corrosion monitoring methods such as electrochemical investigations including polarization analysis, electrochemical impedance spectroscopic method and noise studies. The corrosion inhibition mechanism of the compounds was confirmed by adsorption studies and surface morphological analysis.

2. EXPERIMENTAL ANALYSIS

Synthesis

Heterocyclic hydrazones were synthesized by the condensation reaction of equimolar amounts of 2-acetylpyridine and amino compounds such as phenyl hydrazine and semicarbazide in ethanol medium. The resulting solutions were concentrated and the separated crystals were collected and washed with ethanol. Recrystallization from methanol gave the desired products. The products were characterized by elemental (Vario EL III Element Analyzer) analysis, mass (Shimadzu,QP 2010 GCMS), nmr (Bruker Avance III HD, CDCl3 solvent), IR (Shimadzu affinity-1, KBr pellet method) and UV spectroscopic analyses

Physicochemical Investigations

C H N analysis: Carbon, hydrogen and nitrogen percentage of the Schiff bases were measured by microanalysis using Elementar make Vario EL III model CHN analyzer. Infrared spectra: The infrared spectra of the Schiff base compounds were recorded by KBr pellet method in the range of 4000-400cm^{-1} on Shimadzu model FT-IR Spectrometer (Model IR affinity-1). The infrared spectra contain the characteristic stretching frequencies which help to predict the functional groups present in the compounds. Electronic spectra: The electronic spectra of the Schiff bases were recorded on a Shimadzu UV-Visible-1800 Spectrophotometer with DMSO as solvent. The obtained data gives additional evidence for the molecular structure of Schiff bases. Mass spectra: The mass spectral studies were carried out only after the removal of the impurities present in trace amount by chromatographic method. Mass spectra were recorded using Gcms Qp-2010 plus model. NMR spectra: ^{1}Hnmr and ^{13}Cnmr studies of the compounds were recorded in $CDCl_3$ and DMSO solvent using Bruker Avance lll, 400MHz model.

Gravimetrical measurements

The gravimetric measurements were carried out using ASTM standard method. Mild steel metal specimens of approximate composition C,0.42%; Mn,0.05%; P,0.01%; S,0.016%; Si,0.025% and rest Fe were used for the studies. The weight loss happened for the metal specimens were calculated for 24 hours. A blank measurement was also taken without inhibitor addition. From weight loss measurements corrosion rate and percentage of corrosion inhibition efficiency was determined. The corrosion rate and the inhibition efficiency of compounds were calculated using Eq. (1) and Eq. (2), respectively.

$$\text{Rate of corrosion W} = \frac{\text{K}\times\text{wt.loss in grams}}{\text{Area in sq.cm}\times\text{time in Hrs}\times\text{Density}} \quad (1)$$

where ‘K’ = 87600 (This is a factor used to convert cm/hour into mm/year), density of MS specimen = 7.88g/cc and the density of Cu = 8.76g/cc. Percentage of inhibition or the inhibition efficiency (η) is given by

$$\eta = \frac{W - W'}{W} \times 100 \quad (2)$$

where W’ & W are the corrosion rate of the MS specimen with and without the inhibitor respectively

Adsorption isotherms

The mechanism of corrosion inhibition by the organic molecules on the surface of metal is not yet completely disclosed. However it is strongly convinced by the scientific researchers that the corrosion inhibition mainly occurred due to the adsorption of the molecules on the metal surface. Physical, chemical or both type adsorptions may happen. Adsorption isotherms have an important role in determining the corrosion inhibition mechanism. The common adsorption isotherms are Langmiur, Temkin, Freundlich and Frumkin adsorption isotherms[19-23]. All the above mentioned isotherms were tried with the help of equations given below to fit the best adsorption model.

Langmiur adsorption isotherm $\frac{C}{\theta} = \frac{1}{K_{ads}} + C$ (3)

Freundlich adsorption isotherm $\theta = K_{ads}C$ (4)

Temkin adsorption isotherm $ef\theta = K_{ads}\,C$ (5)

Frumkin adsorption isotherm $\frac{\theta}{1-\theta}\exp(f\theta) = K_{ads}C$ (6)

where C is the inhibitor concentration, θ is the fractional surface coverage, f is the molecular interaction parameter and K_{ads} is the adsorption equilibrium constant. Among the tried isotherms, most suitable one which has the highest correlation co-efficient value (R_2) was considered for explaining the adsorption mechanism. The relation between adsorption equilibrium constant K_{ads} and the standard free energy of adsorption ΔG^0_{ads} is given by the equation,

$$\Delta G^0_{ads} = -RT \ln (55.5\ K_{ads}) \quad (7)$$

where the constant value 55.5 is the molar concentration of water, R is the ideal gas constant and T is the temperature in Kelvin. The value of K_{ads} and ΔG^0_{ads} were obtained from different adsorption isotherms.

Electrochemical measurements

The steel samples used for the electrochemical studies were abraded with different grades of silicon carbide paper, washed and dried. Different inhibitor solutions were prepared in the concentrations range of 0.2–1.0mM in 0.5M H_2SO_4 medium. Electrochemical studies were carried out using three electrode cell assembly consisting of MS as working electrode (exposed area $1cm^2$), platinum electrode as counter electrode ($1cm^2$) and saturated calomel electrode (SCE) as reference electrode at 30^0C. For acid corrosion the working area of metal specimens were exposed to the electrolyte for 30 min prior to the experiment. Ivium Compact state electrochemical system together with Iviumsoft software package was used to perform the experiments.

Electrochemical Impedance Spectroscopic studies (EIS)

EIS measurements are were taken at constant potential in the frequency range from 1KHz to 100 mHz with amplitude of 10 mV as excitation signal. The percentage of inhibition calculated by the following equation,

$$\eta_{R_p}\,\% = \frac{R'_p - R_p}{R'_p}\ X100 \quad (8)$$

where R’p and Rp are the polarization resistance with and without the inhibitor respectively.

Electrochemical systems are attributed with respect to the equivalent electrical circuits. A simple system shown in the Figure 1, where R_s is the solution resistance, R_{ct} is the charge transfer resistance and C_{dl} is the double layer capacitance which forms due to the interaction of charged metal surface and the opposite charged layer distributed by the ions in the solution]. From R_{ct} values, calculation of electrochemical reaction rates became possible.

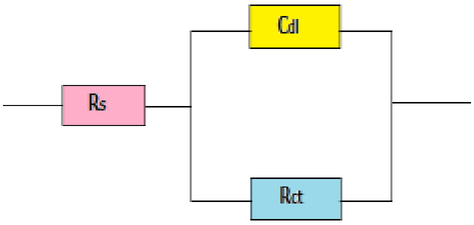

Figure 1: Equivalent circuit model

Potentiodynamic Polarization studies

A potential range of +250 to -250 mV with a sweep rate of 1mV/s was used for the study. Slope analysis of Tafel curves gave the corrosion current densities and the inhibition efficiency was calculated by the following equation

$$\eta_{pol}\,\% = \frac{i_{corr} - i'_{corr}}{i_{corr}}\ X100 \qquad (9)$$

where i’corr and icorr are corrosion current densities with and without the inhibitors, respectively.

Surface Morphological analysis

The analysis of changes observed in the surface morphology of the metal surface helps to explain the inhibition mechanism by which the Schiff bases decrease the corrosion rate. This was carried out by recording scanning electron micrographs (SEM) of the surfaces of bare metal, metal immersed I acid solution (48 h) and metal immersed in inhibitor solution (24 h). It was taken by the model JEOL model JSM-6390LV in the resolution value 2.00 x.

3. RESULTS AND DISCUSSION

Physicochemical discussions

2APPH: Elemental analytical data calculated: C, 73.9; H, 6.16; N, 19.90%. Found: C, 73.6; H, 6.12; N, 19%. Melting point: 220°C. The 1Hnmr spectrum of the Schiff base 2APPH exhibited nine clear peaks. The methyl protons displayed a peak at 2.42δ. A weak broad peak exhibited at 10.48 δ was due to the NH proton and the broadness of the peak can be explained by the effects of H-bonding and quadrupole broadening. The peaks appeared in the range 7.78- 8.72

δ were assigned to the pyridine ring aromatic protons. In the ^{13}Cnmr spectrum a peak appeared at 12.1ppm assigned to methyl carbon atom and all 10 different type sp^2 hybridized carbon atoms on aromatic ring exhibited their signals in the range 114-149ppm. Mass: Base peak at 211 (M+). The clear signals appeared at m/z 196, 194 and 106 were assigned due to the fragments $[C_{12}H_{10}N_3]^+$, $[C_{12}H_8N_3]^+$ and $[C_6H_6N2]^+$ respectively. IR: 1606 cm^{-1} (C=N), 3343 cm^{-1} (-N-H). UV-vis: 28901cm^{-1} (n-π*), 32680cm^{-1}(π-π*) and 39216cm^{-1} (π-π*).

2APSC: Elemental analytical data calculated: C, 53.93; H, 5.61; N, 31.46. Found: C, 53.46; H, 5.45; N, 31.39. Melting point: 200°C. The eight distinct non equivalent protons of 2APSC exhibited eight clear peaks on 1Hnmr spectrum. The three protons on sp^3 hybridized carbon atom (CH_3) showed its characteristic peak at 2.31δ. A broad peak observed at 3.9 δ is assigned to the NH_2 protons and the broadness of the peak can be explained by the effects of H-bonding and quadrupole broadening. The NH proton exhibited a peak at 10.167 δ and the peaks appeared in the range 7.91- 8.80 δ were assigned to the aromatic protons. A very weak peak appeared at 6.18 δ can be assigned to the –OH proton, which generates by the mechanism of tautomerism. In the ^{13}Cnmr spectrum the peaks appeared at 12.02ppm and 156ppm were displayed by methyl carbon atom and carbonyl carbon atom respectively. Mass: m/z at 178 (M+). The base peak observed at m/z 134 is assigned to the fragment $[C_7H_8N_3]^{+,}$ which was generated the removal of amide group from the molecular ion. The clear signals appeared at m/z 106, 78 and 51 were assigned due to the fragments $[C_7H_8N]^+$, $[C_5H_4N]^+$and $[C_4H_3]^+$ respectively. IR: 1608 cm^{-1} (C=N), 3176 cm^{-1} (-N-H). UV-vis: 29142cm^{-1} (n-π*), 34013 cm^{-1} (π-π*) and 39370cm^{-1} (π-π*).

Gravimetric analysis

Weight loss analysis of MS coupons were performed by immersing metal coupons of 1cm^2 area in 0.5M H_2SO_4 solution for 24 hours in the presence and absence of the prepared Schiff bases and analysing them for the study of effectiveness of Schiff bases on corrosion inhibition. The corrosion rate of the immersed MS is calculated in mmy^{-1}. Efficiency of inhibition of the prepared inhibitor molecules 2APPH and 2APSC are recorded in the Table 1 and compared with the plot of inhibition efficiency vs concentration in the Figure 1. These experimental analysis and data clearly indicate that the corrosion rate of MS specimen in sulphuric acid medium in the presence of the synthesised Schiff base inhibitors decreases significantly. In contrary to the expected results based on the previous experiment with HCl, 2APSC exhibited more corrosion inhibition efficiency in H_2SO_4 whereas 2APPH showed higher corrosion efficiency in HCl

Table 1: Corrosion rates and inhibition efficiency of MS in the presence and absence of 2APPH and 2APSC in 0.5M H_2SO_4 at 24hrs

Conc (mM)	2APPH		2APSC	
	Corrosion rate (mmy^{-1})	Inhibition Efficiency (%)	Corrosion rate (mmy^{-1})	Inhibition Efficiency (%)
0	16.26	-	16.26	-
0.2	10.19	37.32	8.14	49.89
0.4	8.19	49.59	6.11	62.41
0.6	6.79	58.18	4.67	71.24
0.8	5.54	65.91	3.58	77.93
1	5.02	69.12	3.50	78.42

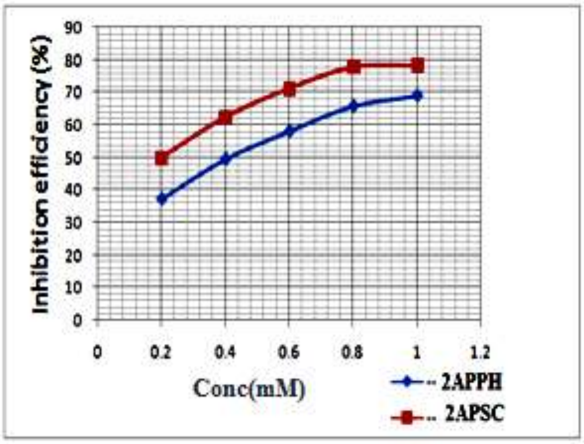

Figure 2: Comparison of corrosion inhibition efficiency of MS with different concentration of Schiff bases 2APPH and 2APSC in 0.5M H_2SO_4 for 24 hours

According to the data, 2APSC exhibited higher inhibition efficiency, compared to that of 2APPH and achieved a maximum efficiency of 78% at 1mM concentration. The inhibition of corrosion on MS surface by Schiff bases in H_2SO_4 medium is comparatively low than in HCl medium, which can be explained by the mechanism that at initial stages, the inhibitor molecules get strongly adsorbed on the metallic surface which are surrounded by a number of hydronium ions due to their polar nature. It is leading to the hydrolysis of the molecules which ultimately results in their desorption from the metal surface. Generally, the interaction of hydronium ions with metal atoms is facilitated only by the replacement of adsorbed water molecules from the metal surface. However, here, the inhibitor molecules facilitate higher possibility for the hydronium ions to reach the metal surface which leads to the reduction of the corrosion inhibition efficiency of these bases in H_2SO_4 medium.

Adsorption studies

Corrosion inhibition mechanism of Schiff bases on metallic surface is mainly explained by the adsorption process. The mechanism of adsorption and surface modifications by the organic compounds can be easily described by invoking suitable adsorption isotherms from which a best fit isotherm model is selected with the assistance of correlation coefficient (R_2) and the thermodynamic parameters evaluated. Figures 3 and 4 represent the adsorption isotherms for 2APPH and 2APSC in 0.5M H_2SO_4 respectively. From attempts on various isotherms, the adsorption behaviour of 2APPH and 2APSC can be described by Langmuir isotherm with regression coefficient values 0.995 and 0.998 respectively. The adsorption equilibrium constant K_{ads}, which is a measure of adsorption for 2APSC, is comparatively higher than 2APPH, suggesting the more efficient adsorption of 2APSC on the metallic surface. Both compounds have ΔG^0_{ads} value between -34 and -33 kJmol^{-1} which indicate the involvement of both physisorption and chemisorption and the negative sign explains the spontaneity of the process.

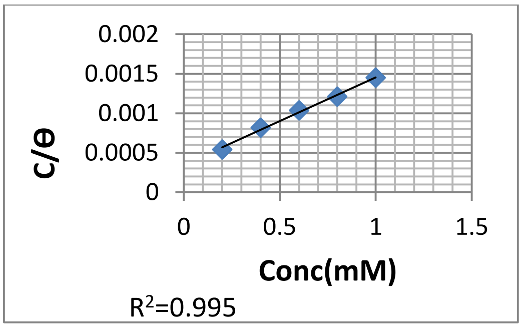

Figure 3: Langmuir adsorption isotherm for 2APPH on MS in 0.5M H_2SO_4

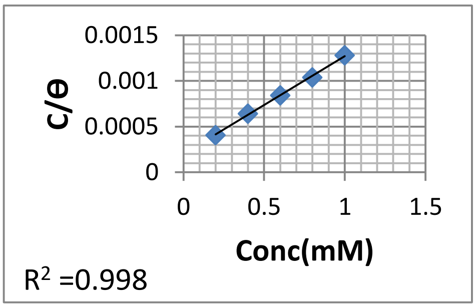

Figure 4: Langmuir adsorption isotherm for 2APSC on MS in 0.5M H_2SO_4

Table 2: Adsorption parameters of Schiff bases for the adsorption on MS surface in 0.5M H_2SO_4

Adsorption Parameter	2APPH	2APSC
K_{ads}	10000	20000
ΔG^0_{ads}(kJ/mol)	-33.12	-34.85

Electrochemical corrosion investigations

Electrochemical investigations were carried out using a three electrode system, which consists of saturated calomel electrode (SCE) as reference electrode, platinum electrode as counter electrode and MS specimen with 1cm^2 area as working electrode. Electrochemical investigations including AC impedance analysis and potentiodynamic polarization studies were performed by Ivium Compactstat-e electrochemical system.

EIS analysis

The corrosion behaviour of MS in 0.5M H_2SO_4 in the presence and absence of inhibitor was examined using impedance spectroscopic analysis at 300C. Figures 5 and 6 represent the Nyquist plots and Bode plots of two Schiff bases 2APPH and 2APSC respectively. The impedance parameters such as solution resistance (R_s), capacitance of double layer (C_{dl}) and percentage of inhibition efficiency (η_{EIS}%) were evaluated from the charge transfer resistance (R_{ct}) values and are noted in the Table 3.

Table 3: Electrochemical impedance data of MS corrosion in the presence and absence of Schiff bases 2APPH and 2APSC in 0.5M H_2SO_4

Schiff base	Conc (mM)	R_{ct} (Ωcm^2)	C_{dl}	η_{EIS}%
2APPH	0	13.3	67.1	
	0.2	56.1	70.8	76.29
	0.4	57.8	76.2	76.98
	0.6	65	81.2	79.53
	0.8	65.1	79.3	79.56
	1	98.9	63	86.5
2APSC	0.2	20.1	48.8	33.83
	0.4	25.5	52.9	47.84
	0.6	28	50.8	52.50
	0.8	43.1	51.2	69.14
	1	84.9	43.3	84.33

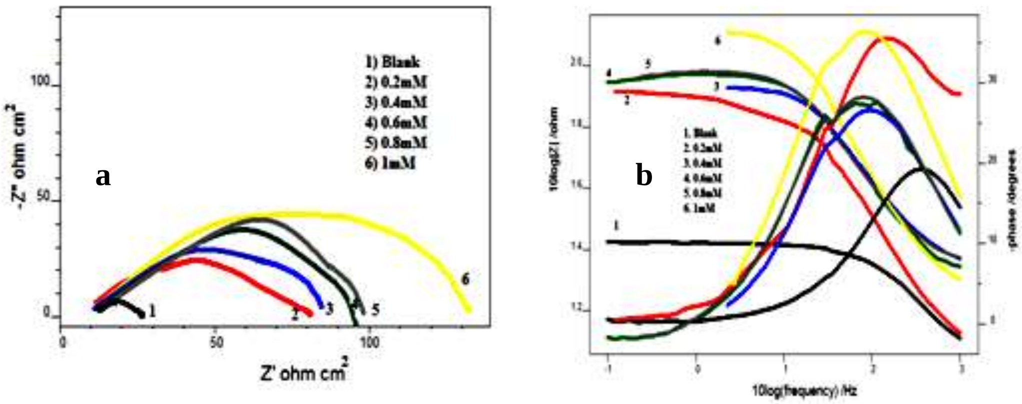

Figure 5: a) Nyquist plots and **b)** Bode plots of MS corrosion in the presence and absence of 2APPH in 0.5M H_2SO_4

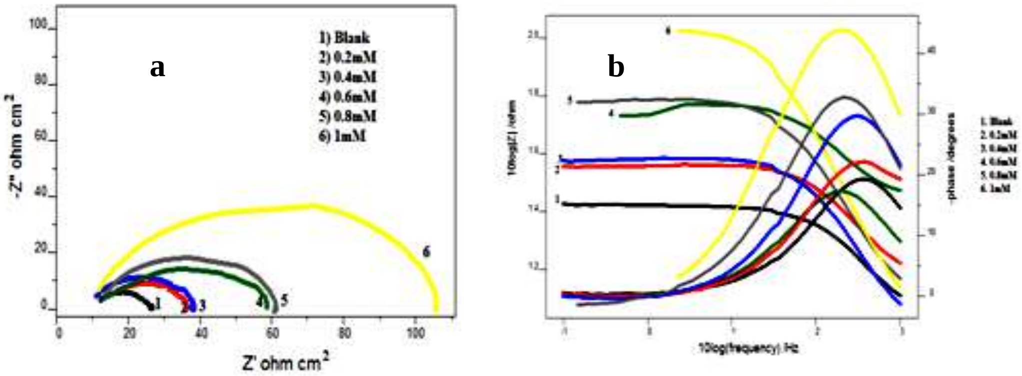

Figure 6: a) Nyquist plot and **b)** Bode plot of MS corrosion in the presence and absence of 2APSC in 0.5M H_2SO_4

From the experimental and analytical data it was observed that:

- Among the Schiff bases studied, 2APPH showed comparatively good efficiency at all concentrations and a maximum efficiency of 86.5% was obtained at 1mM concentration.
- 2APSC exhibited a minimum efficiency at lower concentrations but acted as a good inhibitor at higher concentrations achieving 84% efficiency at 1 mM concentration.
- Both Schiff bases 2APPH and 2APSC exhibited less inhibition efficiency when compared to those in HCl medium in 30 minutes.

Potentiodynamic polarization analysis

Tafel extrapolation analysis and linear polarization studies were conducted to establish the impact of Schiff base compounds towards the polarization of metal specimens by the determination of corrosion current density, polarization resistance and the percentage of inhibition efficiencies. The percentage of inhibition efficiency was obtained from the values of polarization resistance.

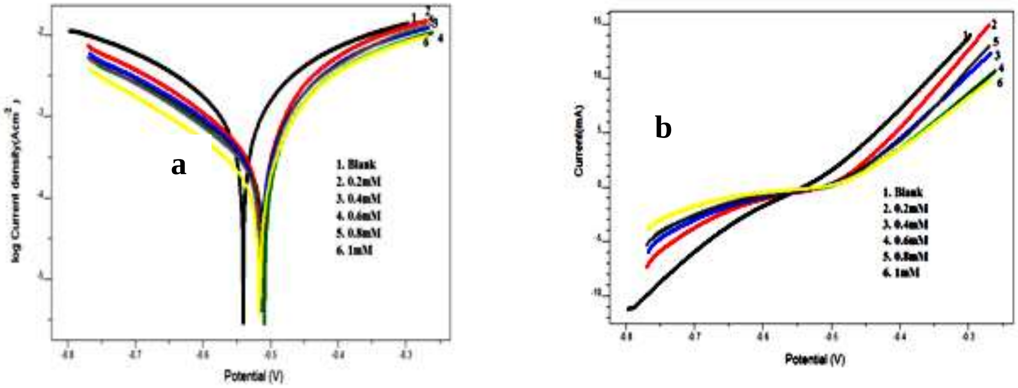

Figure 7: a) Tafel plots and **b)** linear polarization curves for MS corrosion in the presence and absence of 2APPH in 0.5M H_2SO_4

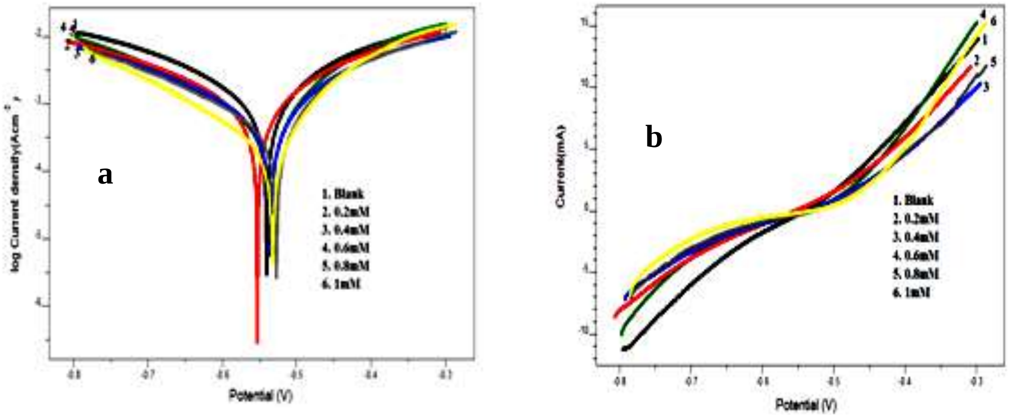

Figure 8 a) Tafel plots and **b)** linear polarization curves for MS corrosion in the presence and absence of 2APSC in 0.5M H_2SO_4

Tafel curves and linear polarization curves obtained for the different Schiff base concentrations are represented in the Figures 7 and 8 and their corrosion parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}), polarization resistance (R_p) and inhibition efficiency percentage (η_{pol} %) are listed in the Table 4.

The Tafel data analysis revealed moderate inhibition efficiency of both compounds in 0.5M H_2SO_4 at all inhibitor concentrations on the MS surface and a maximum efficiency of 73.1% was achieved by 2APSC at 1mM concentration which is slightly different from the data obtained in

EIS measurements. In the cases of both Schiff bases 2APPH and 2APSC, there was no appreciable change in cathodic and anodic slopes which is a clear evidence for the action of these inhibitor molecules on both the cathodic and anodic sites. Furthermore, the E_{corr} value was not altered (>85) with respect to E_{corr} of blank experiment in both cases suggesting that they act as mixed type inhibitors for MS in 0.5M H_2SO_4

Table 4: Polarization data for MS corrosion in the presence and absence of 2APPH and 2APSC in 0.5M H_2SO_4

			Tafel Data				Polarization Data	
Schiff Bases	Conc (mM)	E_{corr} (mV/SCE)	I_{corr} (μA/cm^2)	b_a (mV/dec)	$-b_c$ (mV/dec)	η_{pol}%	R_p (ohm)	η_{Rp} %
	0	-560	1468	236	232	-	34.62	-
2APPH	0.2	-572	841	216	192	42.7	52.45	-34
	0.4	-570.4	694	214	200	52.7	64.67	46.4
	0.6	-568	611	221	202	58.3	72.97	52.5
	0.8	-576.4	586	206	189	60.1	74.98	54
	1	-586.2	445	210	187	69.6	96.85	64.2
2APSC	0.2	-563.3	1040	217	244	29	47.92	27.7
	0.4	-559.5	783	211	225	46.6	52.38	34
	0.6	-566.4	778	181	195	47	60.42	42.7
	0.8	-571	712	215	205	51.5	63.98	46
	1	-532.9	395	156	169	73.1	89.12	67.2

Electrochemical noise analysis

Electrochemical noise (ECN) measurements were done using a three-electrode cell system, which consists of two carbon steel electrodes of area 1cm2 used as working electrode and as counter electrode and SCE as reference electrode. All ECN analyses were performed for a period of 1200 sec using Ivium Compactstat-e electrochemical system controlled by Iviumsoft software. Figure 9 represents the noise current of metal specimen dipped in acid solution in the presence and absence of Schiff base compounds. From the figure it is evident that the noise current in the presence of Schiff base compounds have very low value than the blank metal specimen, which indicates the corrosion protective power of Schiff base compounds. The frequency domain analysis of noise measurement gave the PSD (Power Spectral Density) of different systems, which is represented in the Figure 10. On close examination of PSD plots, it is understandable that at all frequencies the values of current noise are comparatively large for blank metal specimen than the metal dipped in the acid solution in the presence of 2APPH and 2APSC which is a clear implication of the occurrence of localized corrosion on MS surface in the absence of Schiff base compounds.

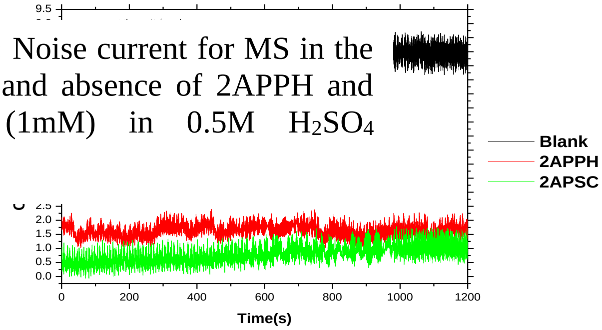

Figure 9: Noise current for MS in the presence and absence of 2APPH and 2APSC (1mM) in 0.5M H_2SO_4 solution.

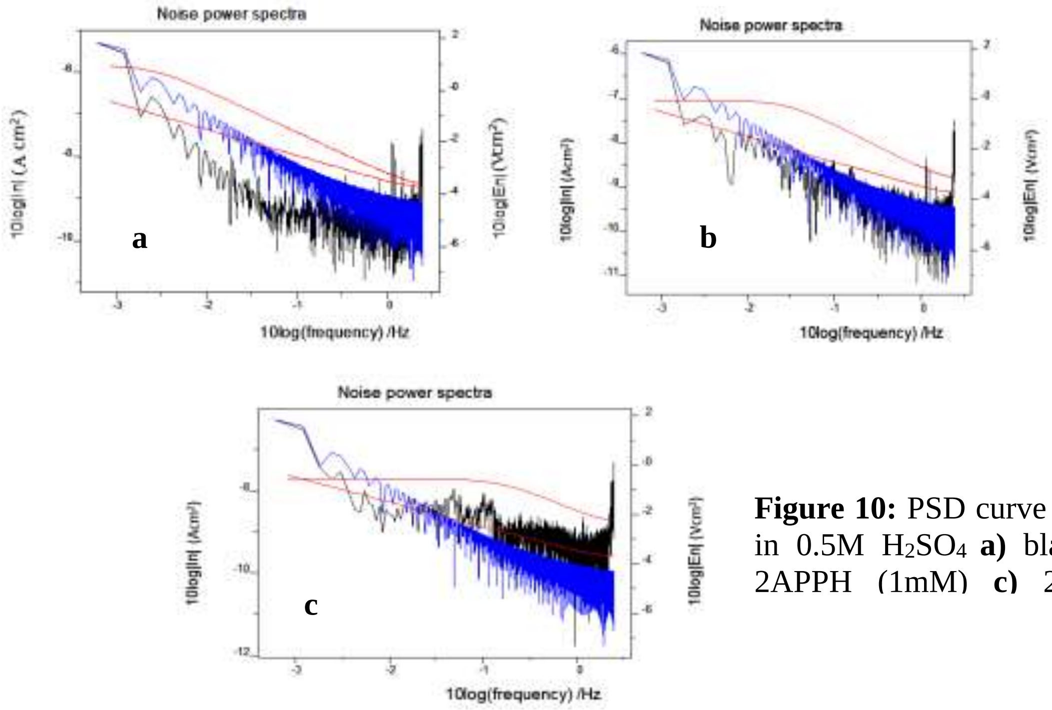

Figure 10: PSD curve of MS in 0.5M H_2SO_4 **a)** blank **b)** 2APPH (1mM) **c)** 2APSC

The measurement of the resisting power to localised pitting corrosion is indicated as pitting index or Pitting Resistance Equivalent Number (PREN). On analysing the pitting index curves represented in the Figure 11, obviously established that the amplitude of pitting index curves corresponding to the blank metal specimen is lower than the metal specimen treated with Schiff base containing solution indicating the higher resistance of Schiff base adsorbed surface towards pitting corrosion.

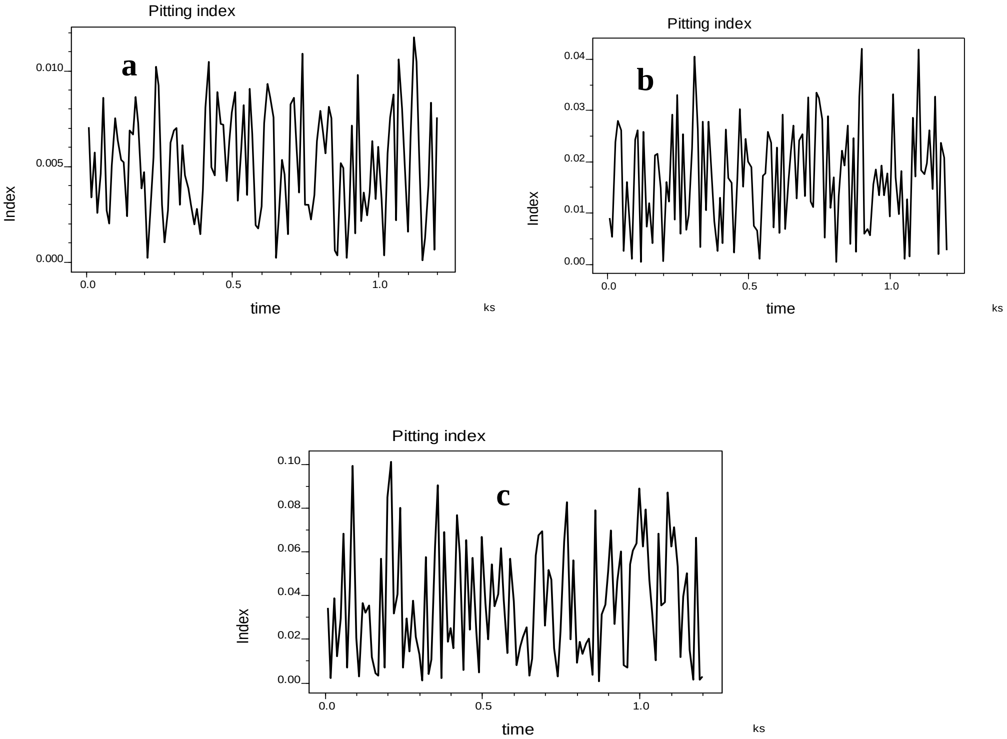

Figure 11: Pitting index curves of MS in 0.5M H_2SO_4 **a)** blank **b)** 2APPH (1mM) **c)** 2APSC (1mM)

Surface morphological analysis

To verify the inhibition mechanism of investigated Schiff base compounds on the MS surface, morphological studies were conducted by taking SEM images of steel surfaces. Figure 12 represents the SEM images of bare sample, metal immersed in 0.5M H_2SO_4, and metal immersed in 0.5M H_2SO_4 containing 1mM 2APPH. On close examination it was evident that the MS surface was highly corroded in blank H_2SO_4 solution. Small cracks and pits on the bare metal surface generated by the surface polishing were totally disappeared on the metal surface dipped in acid solution in the absence of Schiff base due to the occurrence of corrosion. The comparison of figures established that the surface damaging was appreciably decreased in the presence of 2APPH, which indicates that the corrosion tendency was considerably suppressed due to the formation of a protective film of 2APPH through adsorption.

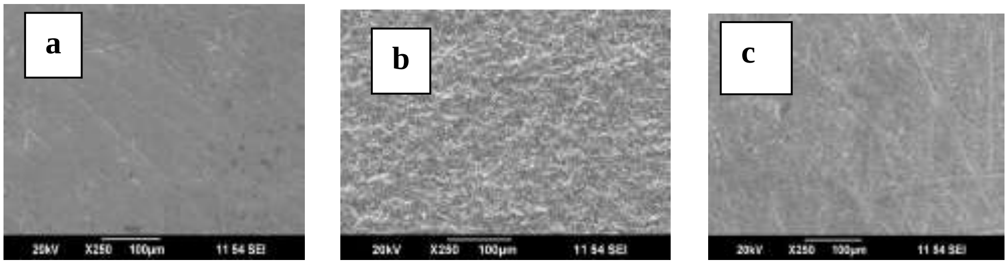

Figure 12: SEM images of **a)** bare sample **b)** metal immersed 0.5M H_2SO_4 **c)** metal immersed in 0.5M H_2SO_4 containing 1mM 2APPH

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