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Corrosion Inhibition of Mild Steel in 1 M HCl Using Schiff Base with Pyrazoline Ring

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Abstract: In the present research, the use of organic compound (Schiff base with parazoline heterocyclic moiety) as corrosion inhibitor for mild steel in 1 M HCl was studied. The adsorption of the inhibitor on the surface of the mild steel has been studied. The study involved weight loss, electrochemical, surface (SEM and EDX) measurements. The obtained results indicated that the investigated organic compound (SB) exhibited excellent inhibition efficiency against the corrosion of mild steel in 1 M HCl solution.

Key words: Corrosion, inhibitors, Schiff, steel, pyrazoline, weight loss

INTRODUTION

Metal corrosion is one of the most safety and economic issues in different industrial processes. The cost effectiveness and good mechanical properties of mild steel make this metal to be prevalently used in different industries (Saha *et al*., 2016; Ulaeto *et al*., 2012; Fuente *et al*., 2011). Acidic solutions, particularly hydrochloric acid solution are widely used in variety of techniques such acid descaling, cleaning, rust removal, oil well acidizing and other industries and petroleum processes, corrosion is one of the drawbacks of such processes (Verma *et al*., 2015; Jeeva *et al*., 2015; Ekanem *et al*., 2010; Gopiraman *et al*., 2012). There are different methodologies that are implemented to overcome the corrosion problems, the use of organic compounds as corrosion inhibitors is one of the solutions and now it has become interesting area of scientific research (Obot and Obi-Egbedi, 2010; Finsgar and Jackson 2014; Jeeva *et al*., 2015; Saha *et al*., 2015). The concentrations of inhibitors are small addition to corrosion media lead to decrease or prevent the reaction of the metal with the media (Al-Sultani, 2012). Schiff bases and heterocyclic compounds are among the classes of the organic compounds which are used as inhibitors for mild steel corrosion (Verma *et al*., 2015a, b, 2016; Singh *et al*., 2016; Ji *et al*., 2016). The heterocyclic compounds with π-electrons in form of multiple (double or triple) bonds and polar functional groups (-OH, $-NH_2$, -CN, -C = C-, -N = N-) are approved to have high adsorption capability on the surface on the metals forming a protecting shield, the adsorption happens through the interaction of the metal surface with the aromatic rings, conjugated double (and/or) triple bonds and unshared pairs of electrons (Zheludkevich *et al*., 2005; Aljourani *et al*., 2009). Different compounds with such structural properties are readily synthesized, therefore, they are used as metal corrosion inhibitors (Obot *et al*., 2015; Amin *et al*., 2009). The nature of the structure, availability of functional groups and the density of the electrons at the heteroatoms in the organic compounds, all of these are factors influence the adsorption tendency on the metal surface (Qu *et al*., 2007; Bentiss *et al*., 2002).

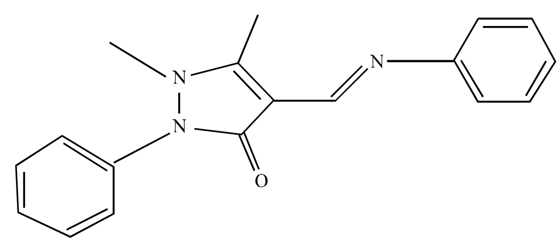

Fig. 1: Molecular structure of SB

In the present research, we report the use organic compound with aromatic rings, heterocyclic system and -C = N- functional group (Schiff base) in its structure as corrosion inhibitor for mild steel in 1 M HCl. Figure 1 shows the structure of the inhibitor (SB).

MATERIALS AND METHODS

Preparation of mild steel samples: The mild steel having the composition (wt. %): C, 0.056; Si, 0.034; Mn, 0.303; P, 0.0008; S, 0.007; Cr, 0.018; Al, 0.049; V, 0.0005; Cu, 0.016; Mo, 0.002; Ni, 0.010 and balance Fe. Samples have been

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cut by CNC machine into 1.5×1 cm and made into working electrode for electrochemical measurements. For weight loss studies, samples were made with 0.2×0.2×0.4 cm and having 2 mm hole. Prior to all the corrosion experiments, the surface of mild steel has been abraded with grade (#80-2000). Al_2O_3 study in double disc metallurgical sample grinder polisher machine with diameter of 200-230 mm. The samples were polished with 1 μm cloth diamond paste, afterward the samples were degreased and rinsed with double distilled water and ethanol, then dried.

Preparation of SB solutions: The organic inhibitor SB was synthesized according to literature procedure (Solankee *et al*., 2011). In 1 M HCl as solvent, solution of 200 mg/L was prepared and other concentrations (50, 75, 100, 125, 150 and 175 mg/L) were prepared by dilution using the same solvent.

Immersion test: Immersion test were performed in order to determine the corrosion rate and the percent of the inhibition efficiency. Immersion test for polished mild steel samples 0.2×0.2×0.4 cm in 1 M HCl in absence and presence of different SB concentrations 50-175 mg/L were carried out at room temperature. The samples have been immersed in 70 mL of 1 M HCl with and without inhibitor for duration of 3 h. Before immersion in HCl solution, weights of the polished, cleaned and dried samples were measured. After 3 h, all the samples were taken out, washed thoroughly with double distilled water and ethanol to remove the corrosion products, dried with a hot air stream and weighed. The corrosion rate was calculated according to Eq. 1 (Perumal *et al*., 2017):

$$CR_{mpy} = 534\times\frac{W}{\rho}\times A\times T \quad (1)$$

Where:
CR = Corrosion Rate (mpy)
W = Weight loss (mg)
ρ = Density of mild steel (g/cm^3)
A = Area of sample (in^2)
T = Exposure time (h)

The percentage of the corrosion inhibition efficiency was calculated according to Eq. 2 (Kumar and Yadav, 2013):

$$\eta(\%) = \frac{((CR_{blank}-CR_{inh})\times 100)}{CR_{blank}} \quad (2)$$

The degree of surface coverage (θ) was calculated according to Eq. 3 (Momin *et al*., 2016):

$$\theta = \frac{\eta(\%)}{100} \quad (3)$$

Tafel polarization measurements: A three-electrode cell has been employed in all electro-chemical measurements with a Saturated Calomel Electrode (SCE) reference electrode and a platinum electrode as the auxiliary electrode. The surface area exposed to HCl is 1.3253 cm^2 and the working electrode has been immersed in 70 mL for 1 M HCl for 30 min to a establish steady-state (open circuit potential) corresponding to the corrosion potential (E_{corr}) of the working electrode. After measuring the E_{ocp}, the polarization measurements were performed. The corrosion rate was calculated according to Eq. 4 (Finsgar and Jackson, 2014):

$$CR = \frac{(K\times I_{corr})}{(\rho\times A\times\sum(nw/A_i))} \quad (4)$$

Where:
I_{corr} = Corrosion current (A)
K = Constant
ρ = Density (g/cm^3)
A = The sample Area (cm^2)
n = The valence of the alloying element (equivalent/mole)
w = The mass fraction of the alloying element
A_i = The Atomic mass of the element (g/mol)

Polarization resistance is (Sastri, 2012):

$$R_p = \frac{(\beta_a\times\beta_c)}{2.3\ I_{corr}(\beta_a\times\beta_c)} \quad (5)$$

Where:
R_p = Polarization Resistance (Ω cm^2)
β_a and β_c = The measured Tafel slopes of the anodic and cathodic reactions (mV/Dec), respectively

All the tests were completed in freshly prepared aerated solution at room temperature. The open-circuit voltage were measured for 30 sec to get a stable voltage, then the device was set to scan over and the stable voltage with ±200 mV.

Atomic absorption spectroscopy: Atomic absorption spectroscopy has been used to determine the concentration of iron (Fe^{2+}) in 1 M HCl.

SEM and EDX analysis: To study the morphology and elemental composition of the mild steel samples, scanning electron microscope equipped with Energy-Dispersive X-ray (EDX) detector have been used. Non-immersed (blank), immersed in 1 M HCl and immersed in 1 M HCl containing SB concentration 125 mg/L samples were subjected to the analysis, the immersion time was 3 h. The samples have been coated with gold using sputter coating.

RESULTS AND DISCUSSION

Weight loss, corrosion rate and inhibition efficiency have been calculated from immersion test for mild steel samples without and with different concentrations of the organic inhibitor SB in 1 M HCl, Table 1 shows the obtained results.

It was found that SB reduces the weight loss significantly. Weight loss of immersed mild steel sample 1 M HCl was 16 mg while the sample immersed in 1 M HCl containing SB (50-175) mg/L, the higher weight loss was 3.8 mg. Figure 2 illustrates weight loss in absence and presence of various concentrations of SB in 1 M HCl. The higher corrosion inhibition efficiency of SB was 87.36% at concentration 125 mg/L as shown in Fig. 3.

Langmuir adsorption isotherm is the best description of the adsorption behavior of the organic inhibitors on the mild steel surface. This isotherm can be represented by the Eq. 6 (Ghames *et al.*, 2017):

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads} + C_{inh}} \tag{6}$$

Where:

C_{inh} = Concentration of the inhibitor (mg/L)
θ = The fractional surface coverage
K_{ads} = The equilibrium constant for adsorption process $(mg/L)^{-1}$

There is a good correlation between adsorption equilibrium constant (K_{ads}) and standard free energy of adsorption ($\Delta G°_{ads}$) which Eq. 7 (Khadiri *et al.*, 2016):

$$K_{ads} = \frac{1}{55.5\exp-\dfrac{\Delta G°_{ads}}{RT}} \tag{7}$$

Where:

55.5 = Constant value of concentration of water in solution
R = The gas constant
T = The Temperature

The surface coverage values (θ) according to Langmuir Model for different concentrations of SB have been calculated by immersion test measurements. Plots of SB Concentration versus (C/θ) are shown in Fig. 4. The standard free energy of adsorption ($\Delta G°_{ads}$) values for the investigated inhibitor are reported in Table 2. These results reveal that the negative value of ($\Delta G°_{ads}$) indicates a spontaneous adsorption process and strong interaction between the SB molecules and mild steel surface, the free energy in SB is <-20 kJ/mol, this infers a physical adsorption process occurs by an electrostatic interaction between the charged SB molecules and the charged mild steel surface. The solid surface consists of a fixed number of adsorption sites and each site hold one adsorbed

Table 1: Immersion test results

Concentrations of SB (mg/L)	Weight loss (mg)	Corrosion rate (mpy)	Inhibition efficiency (%)
0	16	224.0019	-
50	2.1	29.5844	86.79
75	2.1	30.3139	86.46
100	3.8	54.1963	75.80
125	2.0	28.3100	87.36
150	2.5	36.2153	83.83
175	2.1	29.9860	86.61.3

Tabe 2: Equilibrium constants and standard free energy values of mild steel in 1 M HCl with various concentrations of SB

Concentrations of inhibitor (mg/L)	θ	C/θ (mg/L)	K_{ads} $(mg/L)^{-1}$	ΔG_{ads} (kJ/mol)
50	0.8679	57	0.1428	-5.12
75	0.8646	86	0.0909	-4
100	0.7580	131	0.0322	-1.43
125	0.8736	143	0.0555	-2.78
150	0.8383	178	0.0357	-1.69
175	0.8661	202	0.037	-1.78

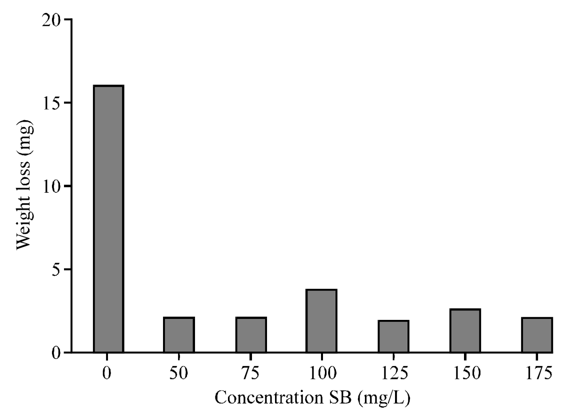

Fig. 2: Weight loss obtained from immersion test in 1 M HCl

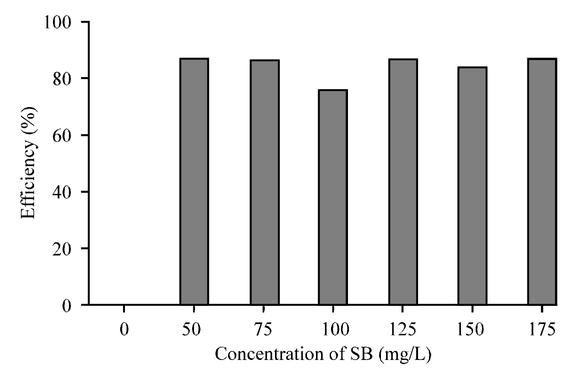

Fig. 3: Inhibition efficiency obtained from immersion test for 1 M HCl

Table 3: Polarization parameters for mild steel in 1 M HCL

Conc. of SB (mg/L)	i_c (μA)	i_c (μA/cm^3)	CR (mpy)	η (%)	OCP (mV)	E (mV)	R_p (Ωcm^2)	βa (mV/Dec)	βc mV/Dec
0	402.89	303.99	140.525	-	-466	-475.9	0.04	57.3	-88.3
50	50.43	38.05	17.5893	87.48	-467	-466.6	0.54	88.2	-103.8
75	49.71	37.5	17.3352	87.66	-461	-471.4	0.56	88.5	-108.7
100	44.40	33.50	15.4861	87.97	-455	-466.0	0.11	16.8	-18.7
125	40.82	30.80	14.2379	89.86	-460	-467.4	0.45	57.9	-73.4
150	39.83	30.05	13.8912	90.11	-464	-472.5	0.43	52.9	-71.1
175	30.52	23.02	10.6414	92.42	-464	-415.7	0.19	21.3	-20.7

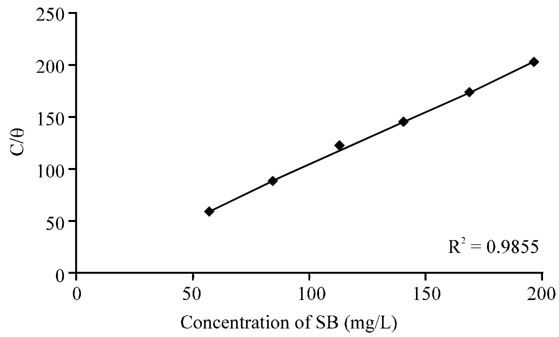

Fig. 4: Langmuir adsorption isotherm of SB on mild steel in 1 M HCl

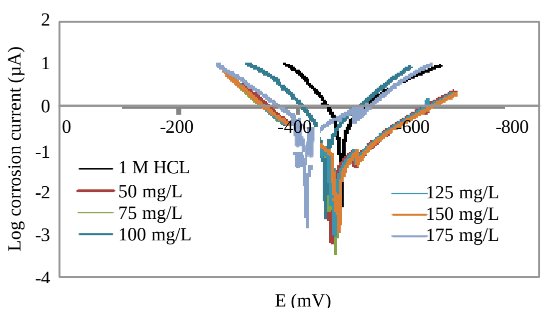

Fig. 5: Tafel polarization curves of mild steel

species. A large (K_{ads}) relative to the interaction of the inhibitor with the mild steel surface (Khan *et al.*, 2017).

Tafel polarization measurements: Tafel polarization measurements (Fig. 5) provide valuable information about the kinetics of anodic and cathodic reactions. Table 3 shows the potentiodynamic polarization parameters. The corrosion current decreases with increasing concentrations of SB. There is no noticeable shift in corrosion potential (E_{corr}) values with increasing SB concentrations. No observable change in (E_{corr}) upon the addition of SB can be interpreted as inhibition of both anodic and cathodic processes (Sastri, 2012). SB can be considered as mixed type inhibitor which inhibits the corrosion process by geometric blocking of both cathodic and anodic surface active sites of the mild steel (Ituen *et al.*, 2017). The highest inhibition efficiency of SB

Table 4: AAS results for (Fe^{2+}) with and without SB in 1 M HCL

Concentrations of SB (mg/L)	Concentration of (Fe^{2+}) dissolved in HCl within and without SB (mg/L)
0	71.7
50	25.8
75	24.8
100	30.8
125	24
150	30
175	30.8

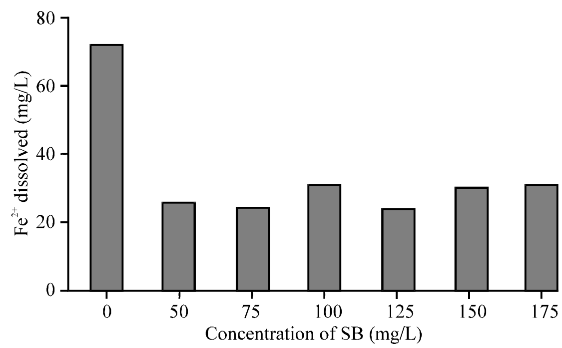

Fig. 6: A_{as} results for Fe^{2+} dissolved with and without SB in 1 M HCl

(92.24 %) with concentration of 175 mg/L. The increase in polarization Resistance (R_p) value can be attributed to the formation of protective film on the metal/solution interface. The increase in values of R_p with increasing the concentration, also, indicate that the SB acts as primary interface inhibitor and controls the corrosion of mild steel under (OCP) (Daoud *et al.*, 2014).

Atomic Absorption Spectroscopy (AAS): AAS results obtained shown in Table 4could explain the dissolution concentration of (Fe^{2+}) in 1 M HCl for SB. After immersion test, inspect the solution in which the mild steel were immersed in HCl in absence and present SB as in Table 4 and Fig. 6.

Results obtained indicate that SB reduced the dissolved amount of Fe^{2+}. In 1 M HCl solution, dissolved is Fe^{2+} form mild steel sample was found of 71.7 mg/L. While in the presence of SB, this value was reduced 24 mg/L with concentration of 125 mg/L (Fig. 6). This could be attributed to the adsorption of SB on the mild

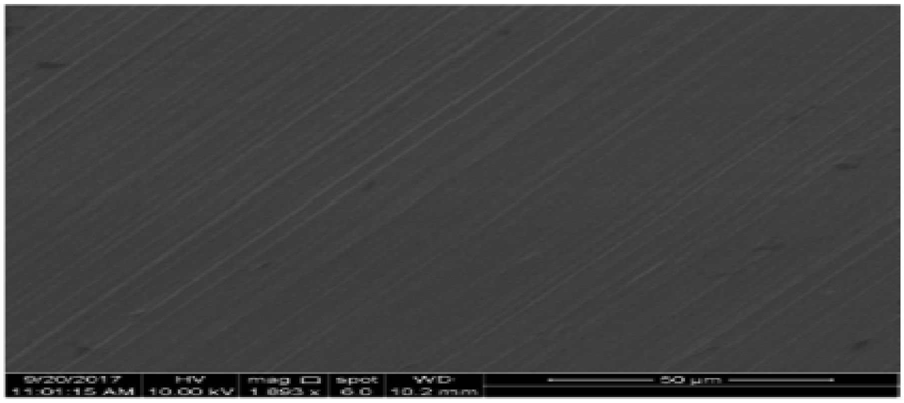

Fig. 7: SEM morphology of a mild steel sample before immersion

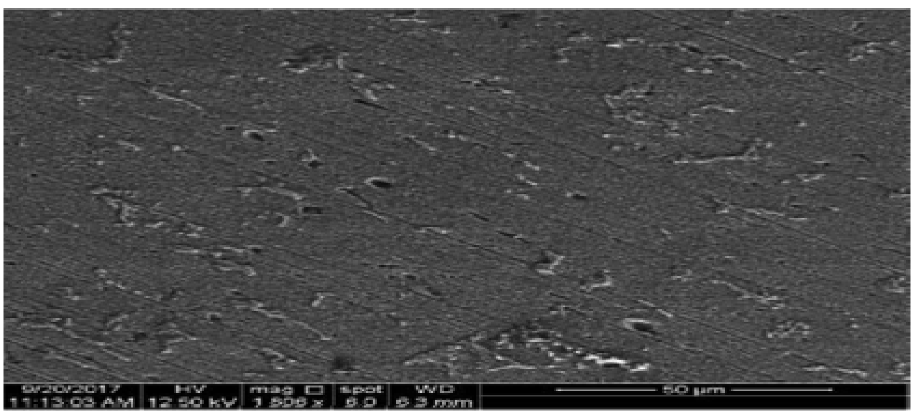

Fig. 8: SEM of sample immersed 1 M HCl

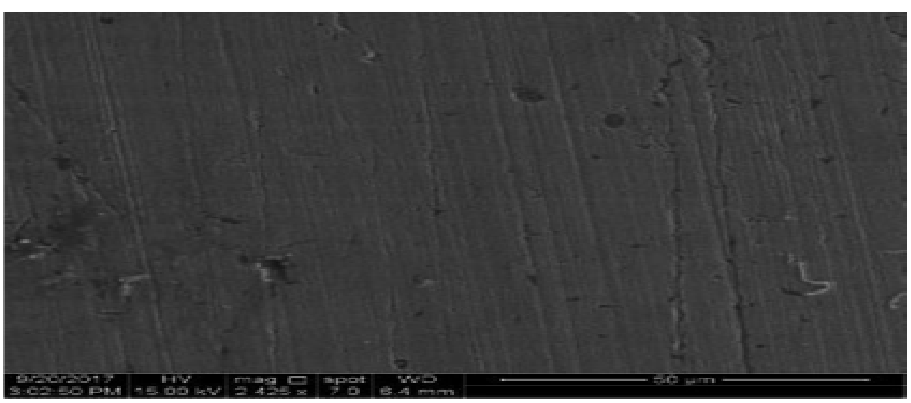

Fig. 9: SEM of sample immersed in 1 M HCl containing SB 125 mg/L

steel surface, producing a barrier which isolates the surface from the corrosion environment (Umoren *et al.*, 2016).

SEM micrographs of the surfaces of mild steel samples were recorded in absence and presence of SB in 1 M HCl. Figure 7 shows the morphology of a mild steel sample before immersion. The effect of exposure of the sample to 1 M HCl for 3 h is shown in Fig. 8, the surface was found to be highly corroded with cracks and pits due to rapid corrosion attack. While in the presence of SB, the surface was observed to be relatively smoother and less corroded as shown in Fig. 9, this is another

Table 5: EDX of surface composition (wt. %) of mild steel samples before immersion and after 3 h of immersion in 1 M HCl within and without SB

Mass (%)	Fe	C	O	Cr
Pure sample	79.09	20.42	0.40	0.09
Blank (HCl)	75.34	20.40	4.26	0.00
SB	68.41	27.71	3.88	0.00

evidence that proofs SB acts as corrosion inhibitor by forming a protective film over on the mild steel surface.

Finally, the EDX analysis for mild steel surface after immersion test showed that Fe and oxygen have been recognized as shown in Table 5 which indicates to the formation passive film (Hassan *et al*., 2017).

CONCLUSION

Schiff base inhibitor has been investigated as corrosion inhibitor using a series of techniques. This organic inhibitor exhibits excellent inhibition performance. The results obtained from immersion test and tafel polarization showed that the inhibiting efficiency increases with increasing the inhibitor concentrations. Langmuir adsorption isotherm has been found to be the preferable description for the studied organic inhibitor and involves physical adsorption mechanism. K_{ads} values show that both molecules tend adsorption towards on the mild steel surface.

The values of ΔG_{ads} could be refering to the adsorption of the inhibitor molecules with the mild steel surface, forming a protective film. From results of Atomic Absorption Spectroscopy (AAS), we concluded the SB produces a barrier which isolates the surface of the mild steel from HCL medium. Results of surface roughness test have been showed that the adsorption forms a continuous layer on surface to covers the scratches and gaps and slows resulting from the corrosion process and reduces the surface roughness of mild steel. The SEM and EDX analysis confirmed that SB can be used as a potential organic inhibitor for mild steel in 1 M HCl. The structural properties of SB including the presence of nitrogen heteroatoms, conjugated π-bonds and aromatic rings made this compound readily interact with metal surface to form the protecting layer and that is why we have chosen this compound for the study.

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