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The inhibition effect of Schiff bases on corrosion of Mild steel in HCl and their characterization

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Abstract
The inhibition effect of schiff base compounds for mild steel in HCl solution were analyzed by weight loss method, potentiodynamic polarization, UV-vis, IR and Electrochemical impedance spectroscopy (EIS). Potentiodynamic polarization study revealed that the schiff bases acted as mixed type inhibitors. The EIS parameter is indicative of adsorption of schiff bases on mild steel surface leading to formation of protective layer. The weight loss study showed that the inhibition efficiency of these compounds increases with increase in concentration and vary with immersion time. Adsorption of schiff bases obeyed the Langmuir adsorption isotherm. In the UV-visible and IR spectral data gave the exact structure of the Schiff bases.

Keywords: Greener Synthesis of Schiff Bases, Mild steel, Potentiodynamic Polarization, EIS.

1. Introduction

Schiff base compounds are of most interest in researchers due to its numerous practical applications [1-2]. Schiff bases possess excellent characteristics, structural similarities with natural biological substances and synthetic procedure is relatively simple [2, 3]. Schiff bases and their complexes have a variety of applications in biological, clinical, and analytical fields [4-6]. Unfortunately most Schiff base compounds are chemically unstable and also involved tautomeric interconversions, hydrolysis and formation of ionized species [7, 8]. Hence the Schiff base compounds required a careful study of its characterization. Imines are prepared by condensation of aromatic aldehydes/ketones with aromatic mono/di amines. An interesting application of Schiff bases is their use as an effective corrosion inhibitor, which is based on their ability to spontaneously form a monolayer on the surface to be protected. Many commercial inhibitors include aldehydes or amines, but presumably due to the C=N bond the Schiff bases function more efficiently in many cases [9]. The principal interaction between the inhibitor and the metal surface is chemisorption [10]. The inhibitor molecule should have centers capable of forming bonds with the metal surface by electron transfer. In such cases the metal acts as an electrophile and the inhibitor acts as a Lewis base. Many biologically important Schiff base compounds are possessing antimicrobial, anticonvulsant, anti HIV, anti-inflammatory and antitumor activities [11-13]. In this present work has been initiated to study the corrosion rate of copper, using the two green synthesized organic inhibitors (p-chloro salicylic aldehyde aniline and p- chloro cinnamaldehyde aniline) in hydrochloric acid medium (1M, 2M, 3M HCl). Weight loss method is used to study corrosion rate values with and without the addition of various concentrations of organic inhibitors and also at different time durations. The inhibition efficiency is calculated from both weight loss method and electrochemical methods (potentiodynamic polarization and electrochemical impedance spectroscopy). The surface coverage (θ) of the adsorbed inhibitor is calculated and the suitable plot of surface coverage with the concentration of the inhibitor is made to understand the nature of mechanism involved in the corrosion inhibition.

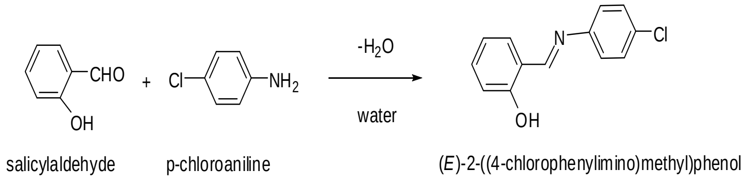
2. Experimental

2.1 Materials

Salicylaldehyde, Cinnamaldehyde, ethylenediamine and p-Chloroaniline were purchased from Merck (AnalaR grade). Ethanol, hexane (HPLC grade) was also purchased from Merck and Deionized water has been used as solvents for purification, recrystallization and solution makings.

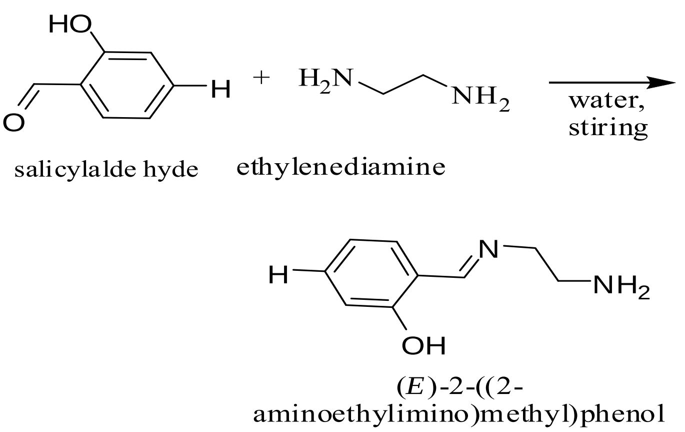
2.1.1 Greener synthesis of Schiff Base 1 (SB 1)

A mixture of liquid salicylaldehyde (1.745 g, 0.014402 mmol) and p-chloroaniline (2 g, 0.015748 mmol) was stirred in a small amount of water (5 ml) at room temperature for 30 minutes. The crystalline powder formed was collected by filtration and washed with water and dried in desiccator to give p-chloro salicylaldehyde aniline (0.50 g, 99% yield). The crude crystals thus obtained were recrystallized from ethanol to give pure compound as yellow prisms. (Melting point: 80-85 °c)



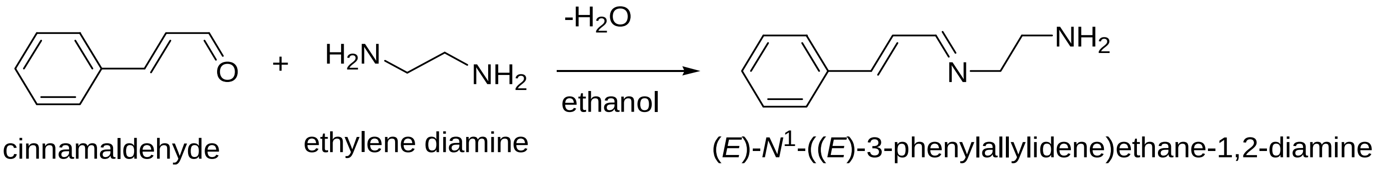
2.1.2 Greener synthesis of Salicylaldehyde Ni Ethylenediamine (SB 2)

A mixture of liquid salicylaldehyde (1.745 g, 0.01428 mmol) and N, N'-ethylenediamine (0.9546 g, 0.01428 mmol) was stirred in a small amount of water (5 ml) at room temperature for 30 minutes. The crystalline powder formed was collected by filtration and washed with water and dried in desiccator to give Salicylaldehyde Ni Ethylenediamine (1.3 g, 92% yield). The crude crystals thus obtained were recrystallized from ethanol to give pure compound as yellow prisms. (melting point:80-83 °c)



2.1.3 Greener synthesis of Schiff Base 3 (SB 3)

A mixture of liquid cinnamaldehyde (1.90114 g, 0.01440 mmol) and N, N'-ethylenediamine (2.22469 g, 0.03701 mmol) was stirred in a small amount of water (5 ml) at room temperature for 30 minutes. The crystalline powder formed was collected by filtration and washed with water and dried in desiccator to give cinnamaldehyde ethylenediamine (1.3 g, 92% yield). The crude crystals thus obtained were recrystallized from ethanol to give pure compound as yellow prisms. (melting point: 80-83 °c).



2.2 Selection of sample and Preparation of specimen, medium

Here we choose an alloy (Mild steel, Brass) and Copper. The corrosion rate of the above specimens studied in acid condition (1M, 2M, 3M HCl) using weight loss method. Rectangular samples were cut from different metal plates. The samples are mechanically polished and numbered by punching before using. The specimens were polished by using emery papers with 80 grades then the samples are degreased by acetone. Then a line is drawn with known area to have same amount of corrosion possibilities. The area of the sample which we have taken is 1x1 $cm^{2.}$

Hydrochloric acid medium (1M, 2M, and 3M) is used as the medium for immersion studies. The solutions are prepared using AnalaR grade chemicals with deionized water.

2.3 Methods

The UV-visible absorption spectral measurements have been carried out using spectrophotometer JASCO V-530 with constant time (in secs) over a range of 200-800 nm. The infrared spectra have been recorded in a JASCO FT-IR-410 spectrophotometer, in a solid phase using KBr pellets. These weight loss values are used to calculate the corrosion rate and inhibition efficiency.

3. Results and discussion

3.1 UV-visible spectral study

All the UV-visible absorption spectral measurements have been carried out using spectrophotometer JASCO V-530 with constant time (in secs) over a range of 200-800 nm. The Schiff base (1) absorbs at λ_{max}= 380 nm (Fig 1) in 100% CH_3CN medium and change of solvent from acetonitrile to ethanol the absorption of Schiff base (SB1) shifts from λ_{max}= 380 nm to λ_{max}= 390 nm (red shift) and the λ_{max}= 330nm (blue shift) in ethyl acetate as a solvent. The Schiff base (SB 2) shows the absorption at λ_{max}= 358nm in 100% CH_3CN medium and change of solvent to ethanol the absorption of Schiff base (3) shift from λ_{max}=358 nm to λ_{max}= 396 nm (red shift) and the λ_{max}= 345 nm (blue shift) in ethyl acetate as a solvent. The same trend was observed for all the three schiff base compounds. This result attributed that in the presence of a polar solvent the energy required for π-π^* electronic transition is affected by solvent polarity. In the presence of a polar solvent, the more polar π^*orbital of the LUMO will be better stabilized than the π orbital of the HOMO leading to a net decrease in the transition energy. Hence the absorption maximum shifted from lower wavelength region to higher wavelength region for more polar solvent (ethanol) (red shift) and the absorption maximum (λ_{max}) shifted from higher wavelength region to lower wavelength region for less polar solvent (ethyl acetate) (blue shift). When the solvent medium is changed from acetonitrile to aqueous acetonitrile and Ethanol to aqueous Ethanol the absorption maximum (λ_{max}) shifts to higher wavelength side are shown in Fig 2 and Fig 3. This absorption maximum is sensitive to the nature of the substituent in the Schiff bases.

From the data provided in Table 2, it is obvious that the same trend of shifting of λ_{max} to higher wavelengths is observed for all Schiff bases (SB 1- SB 3).

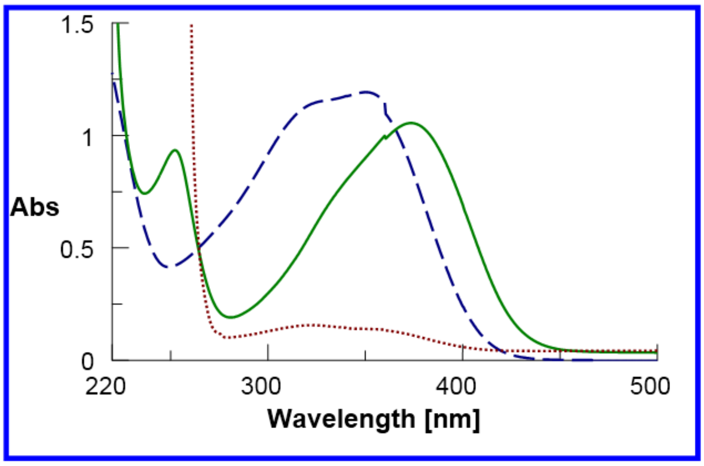


Fig 1: Absorption Spectral changes of SB 3 in (----) 100% acetonitrile, (…..)100% ethylacetate and (…..) 100% ethanol at the concentration. [SB 3]= 2x10 $^{-4}$ M.

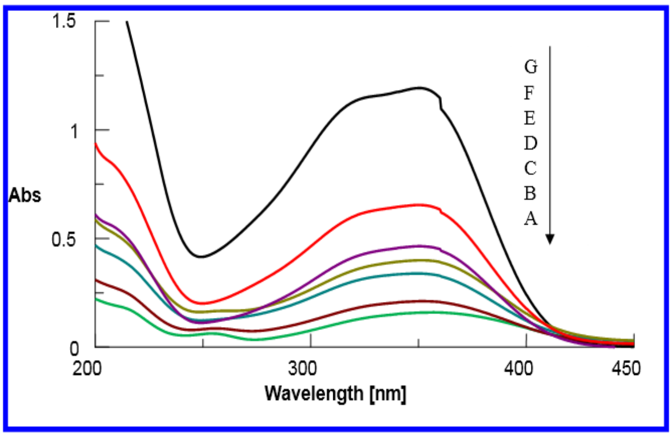


Fig 2: Absorption Spectral changes of SB 3 with the change of medium from acetonitrile to aqueous acetonitrile. [SB 3]=2x10 $^{-4}$ M. Acetonitrile / H_2O (v/v) Solvent ratio A). 50:50 B).60:40 C). 70:30 D). 80:20 E). 90:10. F). 100:0.

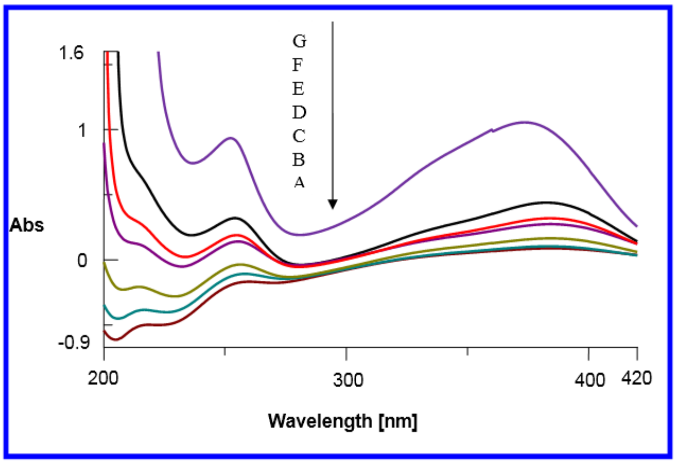


Fig 3: Absorption Spectral changes of SB 3 with the change of medium from Ethanol to aqueous Ethanol. [SB 3]=2x10 $^{-4}$ M. Ethanol/ H_2O (v/v) Solvent ratio A). 50:50 B).60:40 C). 70:30 D). 80:20 E). 90:10. F). 100:0.

Table 1: Yield (in %) and Spectral data [UV-vis] for Schiff bases (1-5) in acetonitrile, ethanol and ethyl acetate at constant temperature T=298K.

Schiff Bases	Yield (in %)	UV-vis Spectral data		
		λ_{max}(nm) in CH_3CN	λ_{max}(nm)in C_2H_5OH	λ_{max} (nm) in $C_2H_5OOCCH_3$
1	96.2	380	313, 390	298, 330
2	92.1	358	318, 396	260, 345
3	92.0	362	340. 406	348

Table 2: Spectral data [UV-vis] for Schiff bases (SB 3) in acetonitrile+water and ethanol+water at constant temperature T=298K.

Solvent ratio	UV-vis Spectral data	
	λ_{max}(nm)in CH_3CN+ H_2O	λ_{max} in C_2H_5OH +H_2O
90:10	358	252.5, 373.5
80:20	361	254, 382.5
70:30	363	255, 384
60:40	365	256, 384.5
50:50	366	258, 390

3.2 Infra Red spectral study

The infrared spectra of all Schiff bases (**SB 1- SB 3**) have been recorded in a JASCO FT-IR-410 spectrophotometer, in a solid phase using KBr pellets. The IR spectra shows the stretching frequency (ν) of Schiff base (**SB 1**) for $\nu_{C=N}$ is1635 cm^{-1} and ν_{O-H} is 3461cm^{-1}. In the case of Schiff base (**SB 2**) the stretching frequency (ν) $\nu_{C=N}$ is 1610 cm^{-1} , ν_{N-H} is 2925 cm^{-1} and ν_{O-H} is 3438 cm^{-1}. In the Schiff base (**SB 3**) the stretching frequency (ν) $\boldsymbol{\nu}_{\mathbf{C-N}}$ is 1189 cm^{-1}, $\boldsymbol{\nu}_{\mathbf{C=N}}$ is 1625 cm^{-1}, ν_{N-H} is 3081 cm^{-1} and $\boldsymbol{\nu}_{\mathbf{O-H}}$ is 3457 cm^{-1}. The IR spectra show the stretching frequency (ν) of Schiff bases are shown in **Table 3**. Infrared spectra of all the three Schiff base compounds are shown in **Figs 4, 5 & 6**.

3.3 Weight loss method

Corrosion of mild steel in 3M HCl medium was studied at fixed time intervals with difference in the concentration of HCl. The time duration as 1, 2, 3, 4 and 5 hours were fixed and HCl concentrations 1, 2 and 3M were used. In all the studies it was found that with the increase in the concentration of hydrochloric acid the corrosion rate increases substantially. The corrosion rate and inhibition efficiency (IE %) of the Schiff bases in the corrosion of mild steel in HCl medium, the values were given in Table 4. It is very interesting to observe that the corrosion rates were substantially decreases as compared with the corrosion rate without inhibitor and also the inhibition efficiency for mild steel (IE %) were found to be good and it starts from 54.54% for 1mM of SB 1 and goes upto 83.33% for 0.02M of SB 1. The inhibition efficiency of mild steel was studied by using another Schiff bases (SB 2 and SB 3) are given in Table 4. With increase in the concentration of Schiff bases the inhibition efficiency (IE %) is increasing and also decreases corrosion rate (Fig 7 & 8). The work has repeated at fixed the concentration of Schiff bases and increase the time (hours). With increase in the immersion time at fixed concentration of Schiff bases the inhibition efficiency (IE %) is increasing and also decreases the corrosion rate (Fig 9 & 10). The values are given in Table 5.

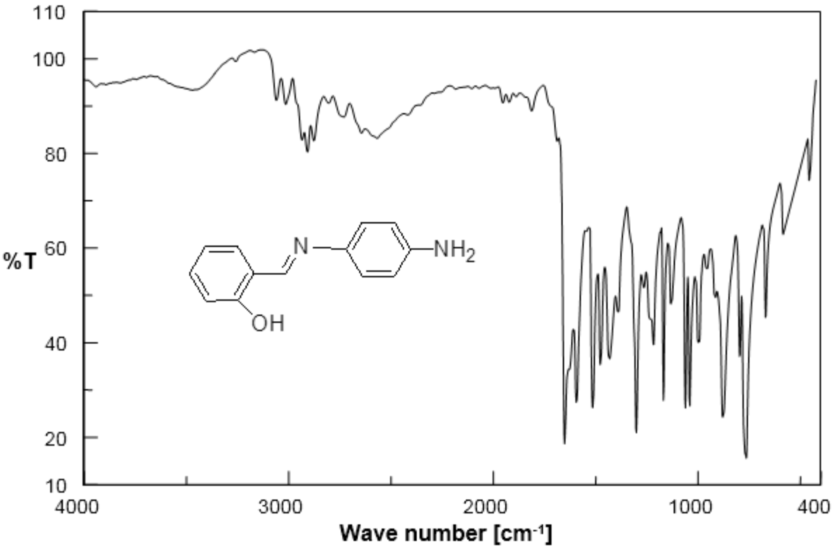


Fig 4: Infrared spectrum of **SB 1**

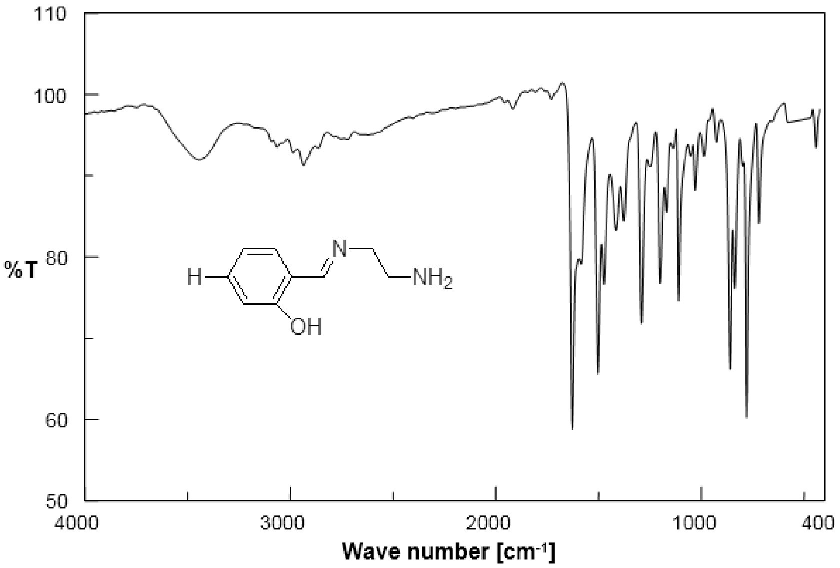


Fig 5: Infrared spectrum of **SB 2**

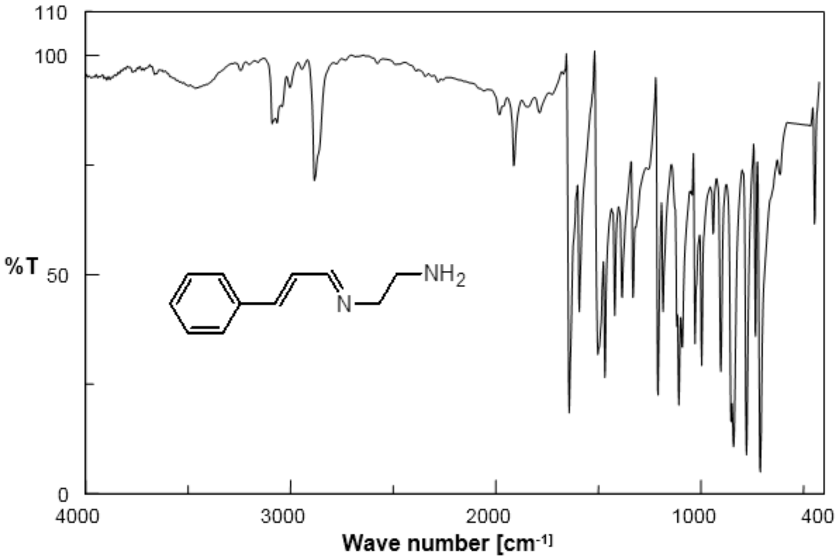


Fig 6:
Infrared spectrum of **SB 3**

Table 3: IR stretching frequencies of Schiff Bases (**1-3**)

Name of the inhibitor	IR stretching frequencies, cm $^{-1}$					
	C-N	C=N	C=C	O-H	N-H	C-X
SB 1		1635		3461		775
SB 2		1610		3438	2925	
SB 3	1189	1625		3457	3081	

Table 4: Corrosion rate of copper in HCl (3M) with SB 1 as a function of variation of concentration of Inhibitor (M)

Time in (hours)	**[SB 1] M**	**W_1 gm**	**W_2 gm**	**W gm**	**Corrosion rate x10^{-4} mmpy**	**IE%**	**θ**
3	0.001	0.4476	0.4466	0.0010	7.4394	54.54	0.5454
3	0.005	0.4617	0.4609	0.0008	5.9515	63.63	0.6363
3	0.01	0.5276	0.5269	0.0007	5.2076	68.18	0.6818
3	0.015	0.4545	0.4539	0.0006	4.4636	72.72	0.7272
3	0.02	0.4660	0.4659	0.0001	2.2318	83.33	0.8333

Table 5: Corrosion rate of copper in HCl (3M) with SB 2 as a function of variation of exposure time (hours)

ime in (hours)	[SB 2] M	W_1 gm	W_2 gm	W gm	Corrosion rate x10^{-4} mmpy	IE%	ɵ
3	0.001	0.4733	0.4731	0.0002	4.4636	66.66	0.6666
3	0.005	0.4614	0.4596	0.00018	4.0173	70.00	0.7000
3	0.01	0.5109	0.5094	0.00015	3.3477	75.00	0.7500
3	0.015	0.4561	0.4548	0.00013	2.9014	78.33	0.7833
3	0.02	0.5217	0.5215	0.0002	2.2318	85.71	0.8571

Time in (hours)	[SB 2] M	W_1 gm	W_2 gm	W gm	Corrosion rate x10^{-4} mmpy	IE%	ɵ
1	0.001	0.4193	0.4178	0.0015	8.3694	50.00	0.5000
2	0.001	0.4696	0.4684	0.0012	6.6955	60.00	0.6000
3	0.001	0.5165	0.5155	0.0010	5.5796	66.00	0.6600
4	0.001	0.4991	0.4983	0.0008	4.4636	73.33	0.7333
5	0.001	0.4717	0.4711	0.0001	3.3477	80.00	0.8000

Time duration (hours)	[SB 3] M	W_1 gm	W_2 gm	W gm	Corrosion rate x10^{-3} mmpy	IE%	ɵ
1	0.01	1.8155	1.8130	0.0025	2.787	87.30	0.8730
2	0.01	1.8592	1.8554	0.0038	2.120	90.45	0.9045
3	0.01	1.7942	1.7900	0.0042	1.562	93.53	0.9353
4	0.01	1.6398	1.6350	0.0048	1.339	94.42	0.9442
5	0.01	1.7448	1.7392	0.0056	1.249	94.89	0.9489

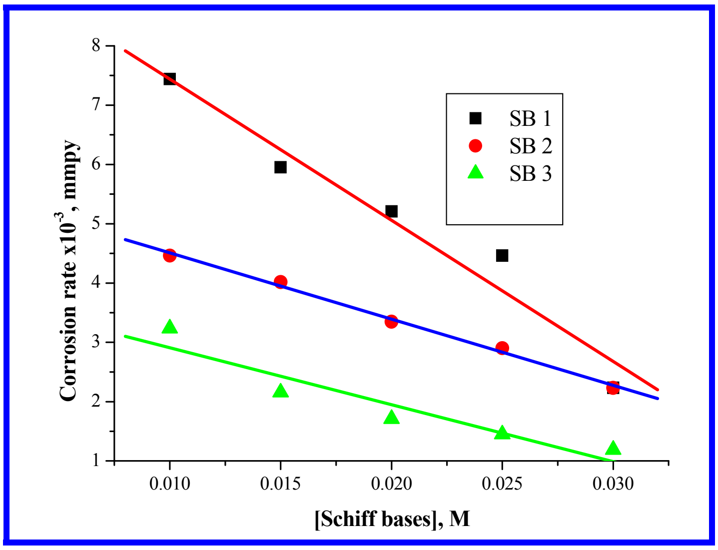


Fig 7: Corrosion rate of mild steel in HCl (3M) with **SB 1, SB 2** and **SB 3** for the variation of Inhibitor concentration.

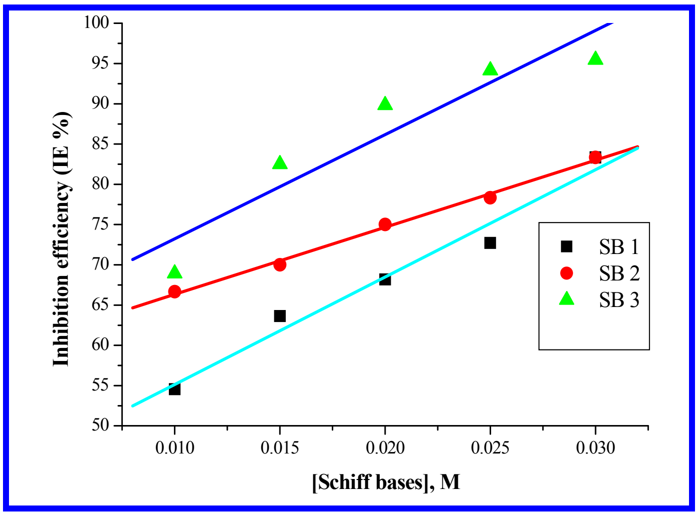


Fig 8: Inhibition Efficiency (%) of mild steel in HCl (3M) with **SB 1, SB 2** and **SB 3** for the variation of Inhibitor concentration.

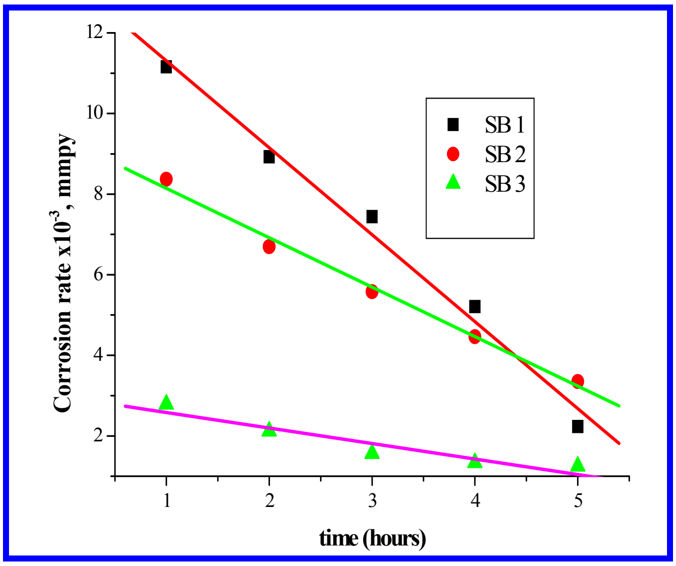


Fig 9: Corrosion rate of mild steel in HCl (3M) with **SB 1, SB 2** and **SB 3** for the variation of time in hours.

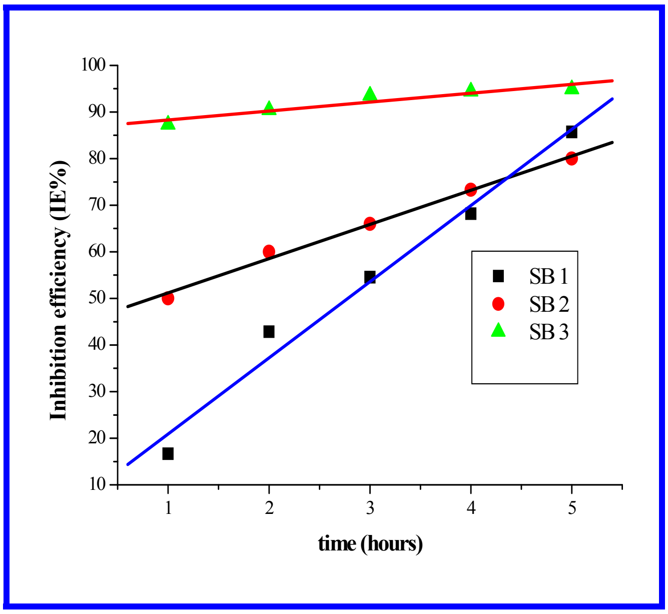


Fig 10: Inhibition Efficiency (%) of mild steel in HCl (3M) with **SB 1, SB 2** and **SB 3** for the variation of time in hours.

3.4 Tafel Polarisation Studies

The potentiodynamic polarization studies of the corrosion of mild steel in 3M HCl medium in the absence and in the presence of inhibitors were carried out .The Figs **11 & 12** shows the anodic and cathodic polarization behavior in the absence of and in the presence of inhibitors of various concentrations Electrochemical parameters such as corrosion potential (E_{corr}) ,anodic Tafel slope (β_a), cathodic Tafel slope (β_c),corrosion current density (I_{corr}) and percentage inhibition efficiency (IE%) are given in **Table 6**. It is observed that the corrosion rate is decreased and inhibition efficiency IE(%) is increased with increase in inhibitor concentration. The same trend is observed both inhibitors. This result suggests that the addition of the preparation of the prepared inhibitors reduces anodic dissolution and also cathodic reaction. These inhibitors cause change in the anodic and cathodic Tafel slopes and no definite trend was observed in the shift of E_{corr} values in the presence of different concentrations of the added inhibitors. The results show the Schiff base compounds act as mixed type (anodic/cathodic) inhibitors.

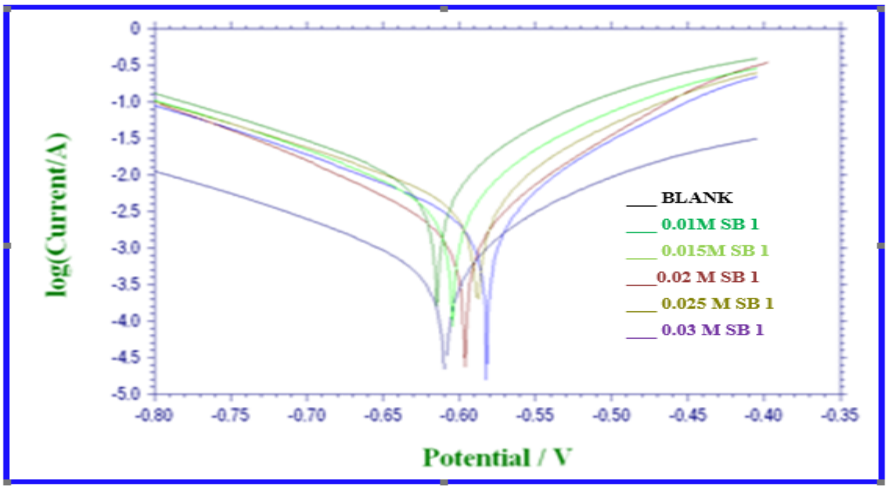


Fig 11: Potentiodynamic polarization behavior of mild steel in (3M) HCl with **SB 1**

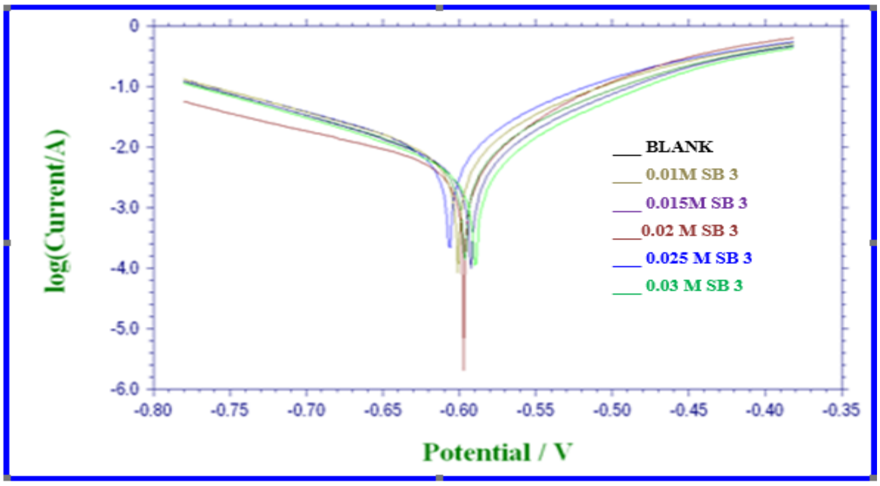


Fig 12: Potentiodynamic polarization behavior of mild steel in (3M) HCl with **SB 3**

Table 6: The polarization parameters for mild steel in HCl (3M) in the presence of SB1, SB2 and SB3.

[SB 1] M	**Corrosion potential E_{corr} mV/sec**	**Corrosion current density I_{corr} µA/ cm^2**	**Anodic Tafel slope βa mV/dec**	**Cathodic Tafel slope βc mV /dec**	**IE (%)**
Blank	-0.616	1.119	9.176	5.782	_
0.01	-0.628	1.087	8.008	6.473	85.00
0.015	-0.622	1.985	8.107	6.380	72.62
0.02	-0.618	1.698	8.424	6.506	76.57
0.025	-0.615	1.367	8.889	6.820	81.14
0.03	-0.611	1.071	9.471	7.251	85.22

[SB 2] M	Corrosion potential E_{corr} mV/sec	Corrosion current density I_{corr} $\mu A/cm^2$	Anodic Tafel slope βa mV/dec	Cathodic Tafel slope βc mV /dec	IE (%)
Blank	-0.617	6.583	10.167	6.870	_
0.01	-0.601	3.802	11.010	8.751	90.92
0.015	-0.598	4.425	10.649	8.285	93.89
0.02	-0.584	5.187	10.370	7.616	92.85
0.025	-0.576	5.421	10.865	6.988	92.52
0.03	-0.572	4.550	11.445	7.150	93.72
[SB 3] M	**Corrosion potential E_{corr} mV/sec**	**Corrosion current density I_{corr} $\mu A/cm^2$**	**Anodic Tafel slope βa mV/dec**	**Cathodic Tafel slope βc mV /dec**	**IE (%)**
Blank	-0.560	5.337	12.904	7.392	---
0.01	-0.576	9.426	9.536	7.524	86.99
0.015	-0.573	8.061	9.801	7.812	88.88
0.02	-0.559	8.022	9.798	7.024	88.93
0.025	-0.557	5.201	10.891	7.648	92.82
0.03	-0.556	3.590	11.541	8.253	95.04

5. Electrochemical Impedance Spectroscopy (EIS)

The corrosion of mild steel in 3M HCl medium was investigated by electrochemical impedance spectroscopy method. The Nyguist plots in uninhibited and inhibited acidic solutions containing different concentrations of Schiff bases are shown in **Figs 13 & 14**. The analysis of the impedance spectra containing a single capacitive semicircle, where the circuit is composed of a solution resistance component (Rs), a polarization resistance component (Rp) and a from

concentrations of inhibitors. Since Rp is inversely proportional to the corrosion current it can be used to calculate the corrosion efficiency

results show that the Rp values are increasing with increasing (IE %)

capacitance component (C_{dl}). These values are given in **Table 7**.

the relation. The trend in the values of C_{dl} in the adsorption of the inhibitor on the metal surface which leads to the formation of surface film in the acidic medium. The impedance study also gave the same efficiency trend as given in Tafel polarization study.

$$IE\ (\%) = \frac{R_p - R_p^0}{R_p} \ \text{X}\ 100$$

The

Where,

R_p and R_p^o are the polarization resistance in the presence and absence of inhibitors respectively.

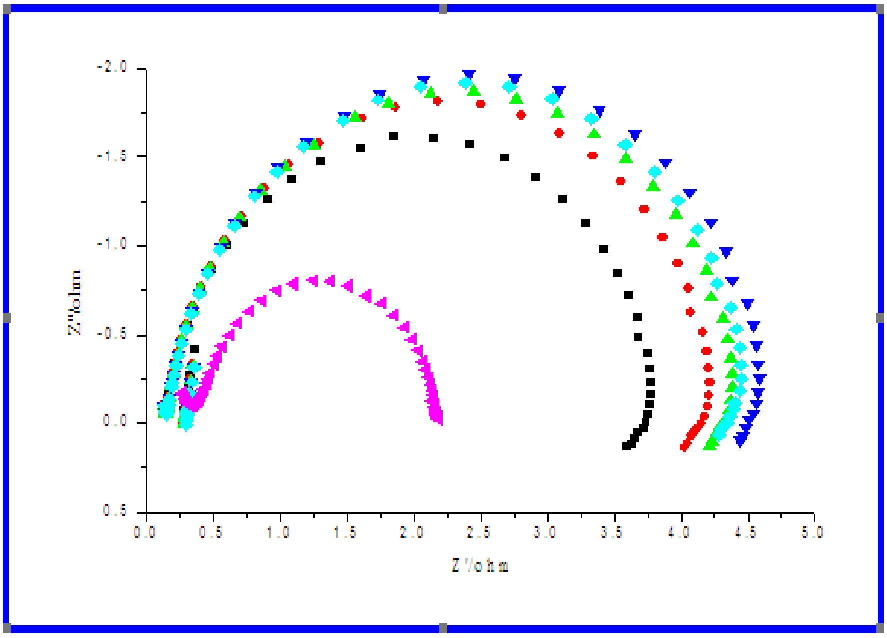


Fig 13: Nyquist plot for the behavior of mild steel in (3M) HCl with concentration of **SB 1 a). Blank b). 0.01M c). 0.015 M d). 0.02M e). 0.025 M f). 0.03M.**

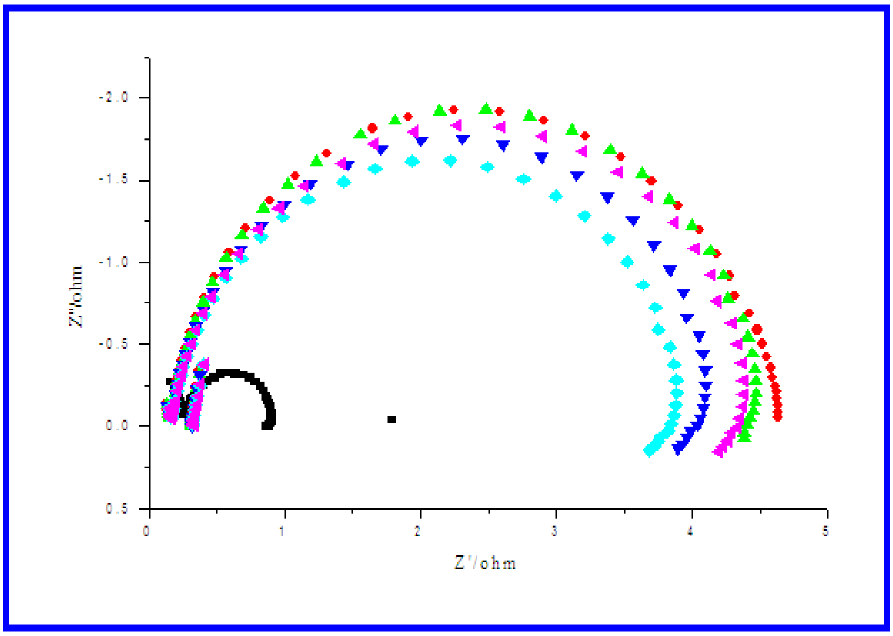


Fig 14: Nyquist plot for the behavior of mild steel in (3M) HCl with concentration of **SB 3 a). Blank b). 0.01M c). 0.015 M d). 0.02M e). 0.025 M f). 0.03M.**

Table 7: ESI parameters of mild steel in HCl (3M) with **SB 3**

[SB 3] X10 $^{-4}$, M	Rct (Ωcm^2)	Cdl (μFcm^2)	IE(%)
Blank	0.8932	1.1973	---
0.01	1.214	0.2725	23.65
0.015	1.544	0.2666	45.9
0.02	2.180	0.3080	57.49
0.025	2.810	0.3696	67.52
0.03	3.578	1.0856	75.03

3.6 Langmuir adsorption isotherm

Corrosion data obtained in this study suggests that the corrosion inhibition is mainly due to the adsorption of the inhibitors on the metal surface. In this study we use three Schiff base compounds (SB 1, SB 2 & SB 3) having oxygen, nitrogen atoms, these are having lone pair of electrons and easily adsorb on the metal surface by formation coordinative covalent bonds. The inhibitors cause the blockage of active sites on the metallic surface, thus causing decrease in corrosion rate. The results proved the formation of monolayer on the metallic surface. Langmuir adsorption isotherm were drawn for the corrosion of mild steel in 3M HCl medium and in the presence of inhibitors are given in **Fig 15**. Normally four types of adsorption may involve between the inhibitor and the metal surface i. electrostatic interaction between charged molecules and the charged metal ii. Interaction of π-electrons with the metal iii. Interaction of uncharged electron pairs in the inhibitor with the metal and iv. A combination of the above.

Based on the present investigation it is attributed that the adsorption of Schiff bases on the metal surface could occur directly on the basis of donor acceptor between the lone pairs of the heteroatom and the extensively delocalized π-electrons of the Schiff base compounds and the vacant d orbitals of the metal. In acidic solution, these Schiff base compounds exist in protonated species. These protonated species may adsorb on the cathodic sites of the metal and decrease the hydrogen evolution. These compounds are able to adsorb on anodic sites through N atoms and aromatic rings which are electron rich. The adsorption of these Schiff bases on anodic sites may decrease anodic dissolution metal. Plot of log θ/1-θ vs. log C gives straight lines as shown in **Fig 15**. the absorption of these Schiff base compounds on the metal surface were found to obey Langmuir adsorption isotherm, which implies that the adsorbed molecules form a unimolecular layer on the surface and the adsorption is uniform all over the adsorbent.

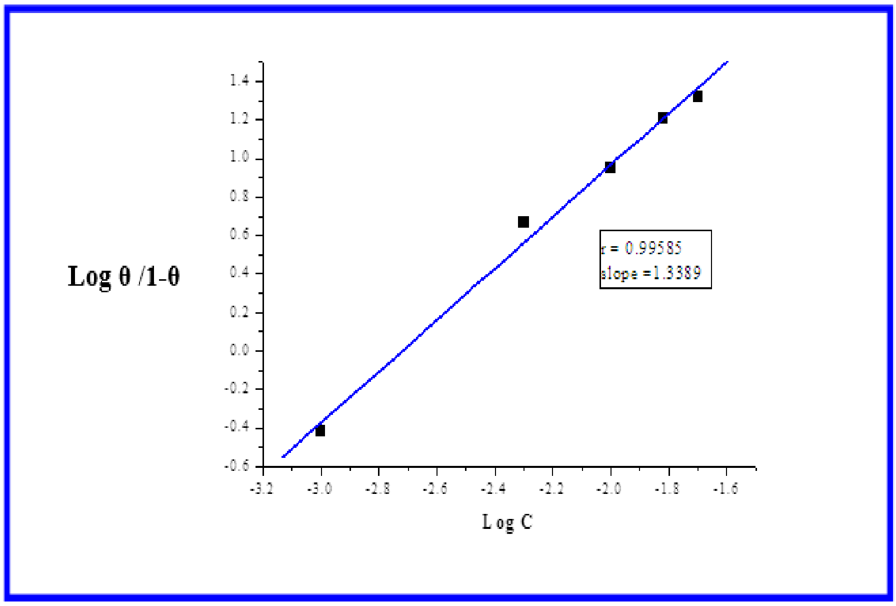


Fig 15: Langmuir adsorption isotherm of mild steel in HCl (3M) with **SB 2**

4. Conclusion

Corrosion of mild steel was studied in 3M HCl and the corrosion rate in the presence of Schiff bases were obtained by weight loss method. The inhibition efficiency of SB 3 is high when compared to SB 1 and SB 2. This may be attributed to the electron withdrawing nature of the chlorine present in the inhibitor SB 1 and also the extending of conjugation present in SB 3.

In the UV-visible spectral studies the wavelength of absorption of all the Schiff bases are reported and the values are in accordance with the structure and polarity of the solvents. When there is more conjugation, there is red shift in λ_{max} values. Likewise with an increase in the polarity of the medium there is red shift in the wavelength absorption values. IR spectral data gave the exact matching frequencies of the groups which are in accordance with the literature values. These values support the exact structures of the Schiff bases.

Weight loss measurements show that the inhibitors reduce the corrosion rate and with increasing of concentration of inhibitors the inhibition efficiency increases. The Tafel polarization studies conclude that the inhibitors behave as mixed type (anodic/cathodic) inhibitors and inhibition efficiency (IE %) increases with increase in concentration of inhibitors. Electrochemical impedance spectroscopy (EIS) studies also prove the same mechanisms. The adsorption of Schiff bases on mild steel in H_2SO_4 medium obeys Langmuir adsorption isotherm.

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6. References

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