SULPHA SCHIFF BASES AS CORROSION INHIBITORS FOR MILD STEEL IN 1M SULPHURIC ACID

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Abstract: Sulphanilic acid and sulphanilamide schiff bases have been synthesised and evaluated as inhibitors for mild steel corrosion in 1M H_2SO_4 by electrochemical and non electrochemical techniques. The inhibition efficiency (IE) increases with inhibitor concentration and decreases with temperature. The adsorption of the inhibitors on the mild steel surface obeys Langmuir and Temkin adsorption isotherms. Potentiodynamic polarization studies show that the inhibitors behave as mixed inhibitors. Addition of halide ions enhances the inhibition efficiency.

Keywords: Sulpha Schiff bases, potentiodynamic polarization, A.C. impedence, corrosion inhibitors

1. Introduction

The inhibition efficiency of organic compounds depends upon the structure and their functional group, nature of metal and aggressive solution. The corrosion inhibitors in acidic solution can interact with metal and affect the corrosion reaction in a number of ways. Most of well-known acid inhibitors are organic compounds containing heteroatom like, nitrogen, sulphur and oxygen atoms [1–4]. For a given metal, the efficiency of inhibitor depends on the stability of the formed chelate, and the inhibitor molecule should have centers, which are capable of forming bonds with the metal surface via an electron transfer. Generally a strong co-ordination bond causes higher inhibition efficiency, the inhibition increases in the sequence O<N<S<P [5]. Several Schiff bases have also been investigated as corrosion inhibitors for different metals and alloys in acidic media [6]. Due to the presence of the C=N group, an electron cloud on the aromatic ring, the electronegative nitrogen, oxygen and sulphur atoms in the molecule, Schiff bases should be good corrosion inhibitors. The aim of this study is to investigate the corrosion of mild steel in 0.1M H_2SO_4 by the newly synthesized Schiff bases, and to observe any relation between molecular structure of these substances and their inhibitive action under various conditions.

2. Experimental

2.1. Material preparation

The mild steel specimens containing 0.084% C, 0.369% Mn, 0.129% Si, 0.025% P, 0.027% S, 0.022% Cr, 0.011% M, 0.013% Ni, and the remainder iron were used. Specimens were mechanically polished and degreased with trichloroethylene before use.

Solutions used were made from analar grade H_2SO_4 and the appropriate concentrations of the acid were prepared using double distilled water.

2.2. Synthesis of inhibitors

The sulpha Schiff bases S.B-1 to S.B-6 (Table 1) have been synthesised by condensation of aromatic aldehydes with sulfanilic acid and sulfanilamide.

2.3. Weight loss method

Mild steel specimens of size 5cm × 2cm × 0.05cm were used for weight loss method . The mild steel specimens were polished successively using emery sheets of 1/0, 2/0, 3/0 and 4/0 grades to remove adhering impurities degreased with acetone and dried using a drier. The specimens in triplicate were hanged into the experimental solution with the help of glass hooks. The volume of the solution was 200ml. The experimental solutions contained 1M H_2SO_4 and different concentrations of inhibitors. The initial weights of the specimen were noted and were completely immersed into the experimental solution. The duration of the experiment was 3 hours. After 3 hours the specimens were removed, washed with running water, dried and the final weight was noted. From the initial and final weights of the specimen, the loss in weights was calculated and the inhibitor efficiency was calculated using the formula,

$$\text{Efficiency of inhibitors (\%)} = \frac{WL_w - WL_i}{WL_w} \times 100 \quad (1)$$
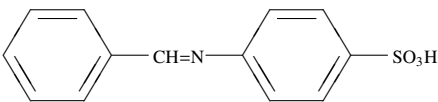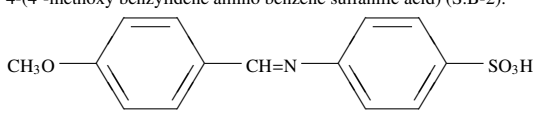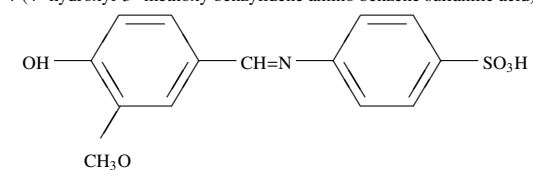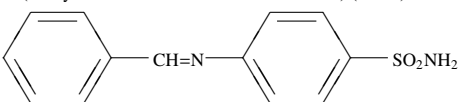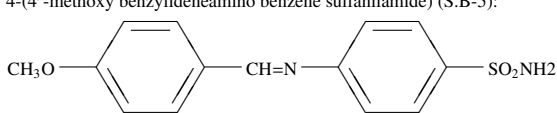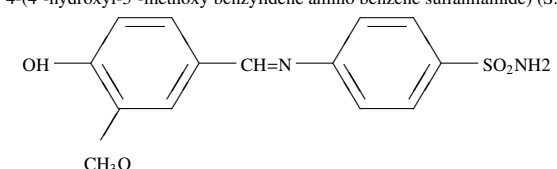
where WL_w is the weight loss without inhibitor and WL_i is the weight loss with inhibitor.

2.4. Gasometric method

The efficiency of the inhibitors was also determined by gasometric technique from the volume of gas collected in the absence and presence of inhibitors at 30±1°C. The volume

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Table 1: Molecular structure and name of the inhibitors

4-(benzylidene amino benzene sulfanilic acid) (SB-1):

4-(4′-methoxy benzylidene amino benzene sulfanilic acid) (S.B-2):

4-(4′-hydroxyl-3′-methoxy benzylidene amino benzene sulfanilic acid) (S.B-3):

4-(benzylidene amino benzene sulfanilamide) (S.B-4):

4-(4′-methoxy benzylideneamino benzene sulfanilamide) (S.B-5):

4-(4′-hydroxyl-3′-methoxy benzylidene amino benzene sulfanilamide) (S.B-6):

of hydrogen liberated from the metal decreases progressively with increase in inhibitor concentration and it can be calculated by using the formula:

$$\text{Inhibitor efficiency (\%)} = \frac{V_B - V_I}{V_B} \qquad (2)$$

where V_B and V_I are the volumes of hydrogen gas evolved in the absence and presence of inhibitors.

2.5. Electrochemical method

Both cathodic and anodic polarization curves were recorded potentiodynamically at a scan rate of 1mV/s using Solartron (model-1280B). A platinum electrode as counter electrode and calomel electrode as reference electrode were used. The mild steel rod embedded in Teflon with an exposed area of 0.785 cm^2 electrode was placed in test solution before electrochemical measurement. Experiments were carried out at the open circuit potential for the frequency range

of 10kHZ to 0.01HZ with signals amplitude of 10mV. Double layer capacitance (C_{dl}) and charge transfer resistance values (R_t) were obtained using AC impedance measurements .

3. Results and Discussion

3.1. Weight loss and gasometric measurements

The inhibition efficiencies obtained from weight loss and gasometric measurements are given in Table 2. Inhibition efficiency increases with increase in concentration of inhibitors. The inhibition efficiency reached a maximum of 89-96% at a concentration of 7.5mM of the Schiff bases.

Table 2: Inhibition efficiencies of various concentrations of inhibitor for the corrosion of mild steel in 1M H$_2$SO$_4$ obtained by weight loss and gasometric measurements at 303±2K

Inhibitor	Inhibitor concentration (mM)	Inhibitor Efficiency (%) Weight loss method	Inhibitor Efficiency (%) Gasometric method
S.B-1	0.25	17.09	18.10
	5.00	70.47	72.57
	7.50	89.70	88.48
S.B-2	0.25	25.12	26.58
	5.00	82.19	81.43
	7.50	94.87	95.78
S.B-3	0.25	35.76	34.59
	5.00	89.87	88.18
	7.50	93.54	94.98
S.B-4	0.25	27.02	28.27
	5.00	86.00	86.07
	7.50	95.02	93.00
S.B-5	0.25	37.55	38.39
	5.00	92.12	91.51
	7.50	96.63	97.89
S.B-6	0.25	29.82	28.56
	5.00	88.08	89.15
	7.50	94.85	95.35

The inhibition efficiency of the compounds follows the increasing order as:

Sulphanilic acid Schiff bases : S.B-2 > S.B-3 > S.B-1

Sulphonamide Schiff bases : S.B-5 > S.B-6 > S.B-4

There is a good agreement between the values of inhibition efficiency obtained by weight loss and gasometric measurements.

3.2. Effect of temperature

The temperature performance (303-333K) of the compounds at the best inhibitor concentration of 7.5mM for the corrosion inhibition of mild steel in 1M H$_2$SO$_4$ solution are given in Table 3 and the corresponding Arrhenius plots are depicted in Figure 1. It can be seen from the table that inhibition efficiency decreased with increase in temperature. This may be due to the fact that the inhibitive film formed on the metal surface was less protective in nature at higher temperatures because of desorption of the inhibitor molecules from the metal surface. Like most chemical reaction, the corrosion of mild steel in aqueous acid solution increased with increase in temperature.The diminished effectiveness of the tested inhibitors under the influence of increasing temperature may be attributed to diminished coverage by the inhibitor. This is based on the assumption that metal dissolution occurs on that part of the surface, free of adsorbed molecules [7].

Table 3: Inhibition efficiencies at 7.5 mM concentration of inhibitors for the corrosion of mild steel in 1M H_2SO_4 obtained by weight loss measurements at different temperatures

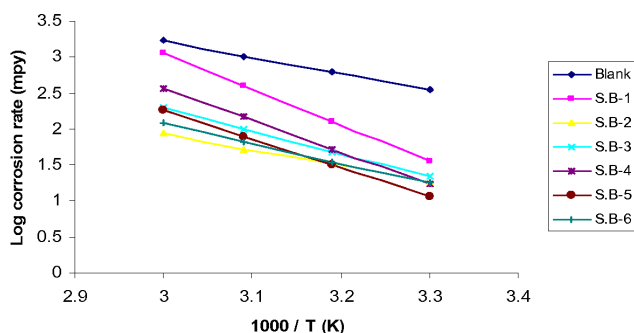| Inhibitor	Inhibition efficiency (%)			
	303K	313K	323K	333K
S.B-1	89.78	80.05	60.22	29.90
S.B-2	94.89	94.78	94.67	94.76
S.B-3	93.61	92.44	90.02	88.05
S.B-4	95.01	91.69	84.87	77.27
S.B-5	96.67	94.99	92.08	88.59
S.B-6	94.89	94.53	93.40	92.46

Figure 1: Arrhenius plot for the corrosion of mild steel in 1M H_2SO_4 solution in the absence and the presence of inhibitors at 7.5mM concentration

The free energies of adsorption (Goads) are calculated from the equilibrium constant of adsorption at different temperatures using the following equation:

$$\Delta G^o_{ads} = -RT \ln(55.5K) \tag{3}$$

where K is given by $K = \theta/C(1 - \theta)$, C is the molar concentration of the solution and θ is the surface coverage.

The activation energy,E_a, values of the uninhibited solutions obtained from the slopes of the Arrhenius plots were higher than those of the inhibited solutions(Table 4). The adsorption of inhibitor molecule causes an increase in the activation energy of the process.This is because the organic compounds have reaction centres that can block the active sites for corrosion resulting in an increase in activation energy [8]. The negative free energy of adsorption indicates interaction of the inhibitor molecule and spontaneous adsorption on the metal surface [9].

Table 4: Activation energies (E_a) and free energies of adsorption (ΔG^o_{ads}) for the corrosion of mild steel in 1mM H_2SO_4 at selected concentration of the inhibitors

Inhibitor	E_a	ΔG^o_{ads} at various temperatures(kJ)			
	(kJ)	303K	313K	323K	333K
Blank	38.29	-	-	-	-
S.B-1	95.74	-10.5	-8.82	-8.31	-3.18
S.B-2	46.91	-12.39	-12.75	-13.09	-13.55
S.B-3	59.16	-11.8	-11.72	-11.27	-11.06
S.B-4	83.48	-12.45	-11.45	-9.97	-8.90
S.B-5	76.56	-13.52	-12.83	-11.96	-11.2
S.B-6	46.91	-12.39	-12.61	-12.48	-12.47

3.3. Adsorption isotherms

Adsorption isotherms are very important in determining the mechanism of organo-electrochemical reactions. The most frequently used isotherms are Langmuir, Tempkin and Frumkin. A linear behaviour has been noticed in all the cases when the graph is plotted between C/θ and C (Figure 2) and Vs log C (Figure 3) this shows that the adsorption of all these compounds obeys the Langmuir and Tempkin adsorption isotherm relationship.
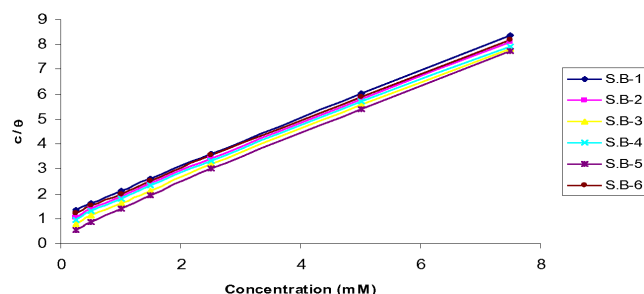

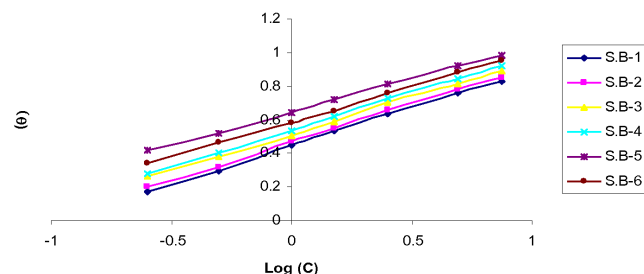Figure 2: Langmuir adsorption isotherm for the inhibitors at 303K

Figure 3: Temkin adsorption isotherm for the inhibitors at 303K

3.4. Potentiodynamic polarization studies

Polarization studies on mild steel have been made for the inhibitors in 1M H_2SO_4. Corrosion kinetic parameters such as corrosion potential E_{corr}, corrosion current I_{corr}, anodic and cathodic Tafel slopes (b_a and b_c) have been derived from these experiments and they have been summarized in Table 5. Typical polarization curves presented in Figure 4 indicates that E_{corr} values are only slightly shifted in the presence of the inhibitors towards the negative side, I_{corr} values decrease with increase in the concentration of the inhibitors. The Tafel constants ba and bc are both affected . Hence it can be concluded that all the inhibitors behave as mixed type inhibitors.
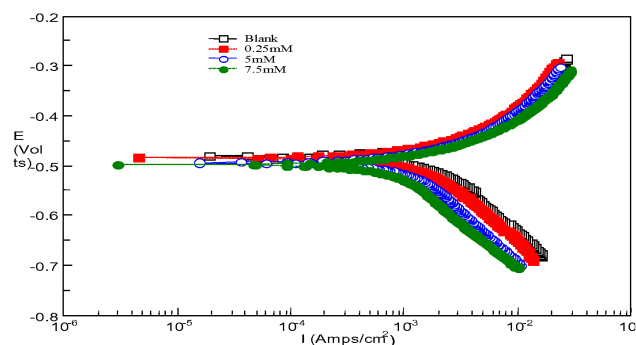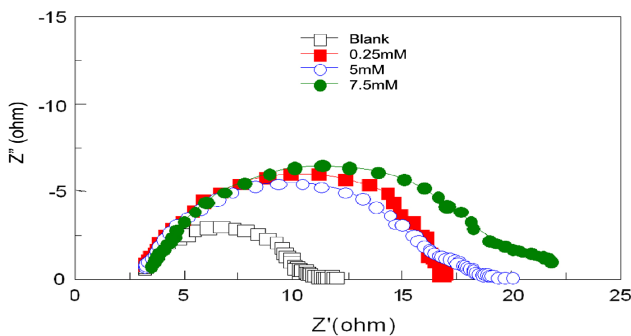

Figure 4: Polarization curves for mild steel in 1M H_2SO_4 the absence and presence of inhibitors at selected concentrations

Table 5: Corrosion parameters for mild steel with selected concentration of the inhibitors in 1mM H_2SO_4 by potentiodynamic polarization method

Inhibitor	Inhibitor Conc.(mM)	Tafel slopes (mV)		E_{corr} (mV)	I_{corr} ($\mu A/cm^2$)	Inhibition Efficiency (%)
		b_a	b_c			
Blank	-	98.33	157.63	-481.5	431	-
S.B -1	0.25	69.49	156.8	-486.22	358.8	16.74
	5.00	79.91	156.06	-497.5	315.9	26.68
	7.50	46.25	155.38	-488.26	277.2	35.56
S.B -2	0.25	79.51	137.83	-500.21	241.4	44.07
	5.00	69.25	148.06	-494.87	145.9	66.13
	7.50	64.72	158.17	-502.67	114.7	73.36
S.B -3	0.25	78.06	179.06	-484.5	330	23.43
	5.00	81.26	104.15	-494	297	31.09
	7.50	83.09	105.84	-498.6	213.3	50.39
S.B -4	0.25	83.34	194.14	-487.33	419.3	2.70
	5.00	86.35	101.11	-488.94	193.1	55.9
	7.50	87.61	126.36	-483.6	145.9	66.12
S.B -5	0.25	81.19	113.15	-489.66	166.2	61.43
	5.00	83.74	149.39	-506.04	898.7	79.14
	7.50	85.34	135.56	-502.24	367.5	91.42
S.B -6	0.25	65.8	156.96	-493.7	239.4	44.43
	5.00	83.48	147.07	-498.4	187.3	56.53
	7.50	89.19	190.93	-485.6	180.3	58.12

Table 6: A.C. Impedance parameters for corrosion of mild steel for selected concentrations of the inhibitor in 1M H_2SO_4

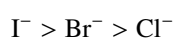| Inhibitor	Inhibitor conc. (mM)	R_t (ohm cm^2)	C_{dl} (μA / cm^2)	Inhibition Efficiency (%)
Blank	-	7.3287	21.175	-
S.B -1	0.25	13.414	20.29	45.36
	5.00	14.201	18.59	48.39
	7.50	16.242	17.604	54.87
S.B -2	0.25	18.03	19.53	59.35
	5.00	21.47	15.63	65.86
	7.50	33.83	15.49	78.34
S.B -3	0.25	9.093	20.802	19.4
	5.00	15.032	18.59	51.24
	7.50	20.337	17.604	63.96
S.B -4	0.25	15.267	21.097	51.99
	5.00	24.145	19.004	69.64
	7.50	29.009	16.184	74.47
S.B -5	0.25	14.048	20.965	47.83
	5.00	36.253	20.818	79.78
	7.50	70.045	19.856	89.53
S.B -6	0.25	16.26	21.089	54.92
	5.00	16.55	20.623	55.71
	7.50	22.9	20.174	67.99

3.5. A.C. impedance measurements

Typical Nyquist plots obtained in the absence and presence of different concentrations of S.B-1 are shown in Figure 5 and the values R_t, C_{dl} derived for all the inhibitors from nyquist plots are presented in Table 6 which clearly indicate that the presence of inhibitors enhances the value of R_t in the acidic solution. Values of double layer capacitance C_{dl} were brought down to the maximum extent in the presence of inhibitors. The decrease in C_{dl} may be due to the adsorption of the compounds on the metal surface leading to the formation of a film [10]. The inhibition efficiency almost follows the same order as that of weight loss and gasometric measurements.

Figure 5: Nyquist plots for mild steel in 1M H_2SO_4 in the presence of inhibitors at different concentrations

3.6. Synergism

The synergistic effect provided by the addition of halide ions I^-, Br^- and Cl^- to the solutions containing 1M H_2SO_4 and the Schiff bases S.B-1, S.B-2, S.B-4, S.B-5 was studied by weight loss method and the data are presented in Table 7. Analysis of the data reveals that the synergistic influence of halide ions follows the order:

$$I^- > Br^- > Cl^-$$

Table 7: Synergistic effect of 1mM KCl /KBr/ KI on the inhibition efficiency of S.B-1, S.B-2, S.B-4, S.B-5 and synergism parameter for the inhibitors

Inhibitor	Conc. (mM)	Inhibition Efficiency(%) / S Value					
		Without KCl, KBr and KI	1mM KCl	S_{KCl}	1mM KBr	S_{KBr}	1mM KI	S_{KI}
	0.25	17.09	29.29	1.04	46.00	1.09	58.13	1.15
	0.5	21.24	31.8	1.03	46.88	1.05	66.46	1.23
	1	30.42	44.41	1.06	48.8	1.1	70.32	1.59
S.B-1	1.5	42.05	54.46	1.3	66.78	1.33	70.74	1.69
	2.5	52.56	68.33	1.42	73.75	1.48	84.68	1.78
	0.25	25.12	40.37	1.12	52.52	1.31	76.14	1.4
	0.5	31.41	52.35	1.28	67.13	1.48	77.56	1.57
	1	48.22	64.21	1.29	76.44	1.56	81.8	1.64
S.B-2	1.5	61.13	72.81	1.37	79.29	1.63	85.07	1.7
	2.5	74.29	80.21	1.46	86.86	1.78	87.18	1.81
	0.25	27.02	34.24	1.00	45.52	1.03	49.68	1.1
	0.5	32.08	41.86	1.04	47.93	1.19	49.77	1.14
	1	47.23	48.8	1.06	67.65	1.09	69.13	1.23
S.B-4	1.5	60.04	58.73	1.08	78.74	1.17	79.56	1.27
	2.5	70.46	83.73	1.25	87.67	1.31	89.76	1.33
	0.25	37.55	38.26	1.08	46.82	1.12	51.56	1.14
	0.5	48.76	51.57	1.09	57.34	1.16	59.44	1.18
	1	61.12	63.76	1.13	68.71	1.19	71.56	1.22
S.B-5	1.5	68.67	77.04	1.21	79.74	1.23	80.1	1.26
	2.5	82.87	87.24	1.26	89.00	1.29	90.84	1.34

This observed order suggests that I^- has highest synergistic influence among the halide ions. This may be explained as follows.

The steel surface is originally positively charged in 1M H_2SO_4. When I^- ion are added to the inhibiting solution they are strongly chemisorbed by forming chemical bonds even leading to the formation of iron iodide [11]. This strong chemisorption of I^- ions shift ϕ_n of the metal to more positive potential than in the case of Cl^- and Br^- and renders the surface more highly negatively charged. On the highly negatively charged metal surface, the protonated cationic inhibitor molecules are physisorbed due to electrostatic interaction. This interaction is higher for I^- than for Cl^- or Br^- due to higher magnitude of negative charge on the metal surface. Hence, the observed order $I^- > Br^- > Cl^-$.

3.6.1. Significance of synergism parameter

The synergism parameter S was defined by the relation:

$$S = [1 - I_{1+2}]/[1 - I'_{1+2}] \qquad (4)$$

where I_{1+2} is the measured IE of the anions plus organic cations, I_1 and I_2 is the IE of anions (halides) and of cations (inhibitors), and $I'_{1+2} = I_1 + I_2$.

The significance of synergism parameter S is that a value of $S > 1$ denotes synergism while a value of $S < 1$ denotes antagonism.

The synergism parameter calculated for the three halide ions $Cl^-/Br^-/I^-$ are presented in Table 7. The S values are all greater than unity indicating that the corrosion inhibition by the Schiff bases (S.B-1, S.B-2, S.B-4, S.B-5) has been enhanced by the addition of halide ions which have synergistically influenced the inhibition. Further the 'S' value increases in the order $Cl^-/Br^-/I^-$ giving highest synergistic influence for I^- which is in accordance with the findings noted in literature [12].

3.7. Mechanism of corrosion inhibition-structure and reactivity

The inhibition of corrosion can be explained on the basis of the concept of adsorption of inhibitors on the corroding metal surface. The inhibitive action of these compounds has been attributed to the strong adsorption of these molecules on the metal surface using the electrons of the aromatic ring. The inhibition of corrosion of these compounds may be due to the adsorption of these compounds on the metal surface by the following interactions

- interaction between π-electrons of the benzene ring and the positively charged metal surface and
- interaction between the lone pairs of electrons of heteroatom with positively charged metal surface.

It can be derived that aromatic Schiff bases inhibit corrosion of mild steel in acidic media by adsorption through the interaction of π-electrons of aromatic ring and the lone pair of electrons on the nitrogen atom with the metal surface. All the six Schiff bases taken for study contain two aromatic rings, a -CH=N group, $-SO_3H$ / $-SO_2NH_2$ group and substituents -OH and $-OCH_3$. The Schiff bases are expected to have adsorbed on the metal surface through the combination of three adsorption mechanisms(Figure 6)

- electrostatic interaction between the charged molecules and charged metal surface (Figure 6(a)) (favoured by protonation of N atom of -CH=N group)
- interaction of unshared electron pairs on the hetero atom in the molecule with the metal surface (Figure 6(b)) (-OH, $-OCH_3$, $-SO_3H$,$-SO_2NH_2$)
- interaction of π-electrons of aromatic ring and CH=N-groups with the metal surface (Figure 6(c)). These interactions are facilitated by the flat orientation of the molecule with respect to the metal surface.

The inhibition efficiency of the compounds follows the increasing order as
Sulphanilic acid Schiff bases: S.B-2 > S.B-3 > S.B-1
Sulphonamide Schiff bases: S.B-5 > S.B-6 > S.B-4

In both the series of inhibitors, the unsubstituted Schiff bases S.B-1 and S.B-4 have lower inhibition efficiency in comparison to the others. The high inhibition efficiency of
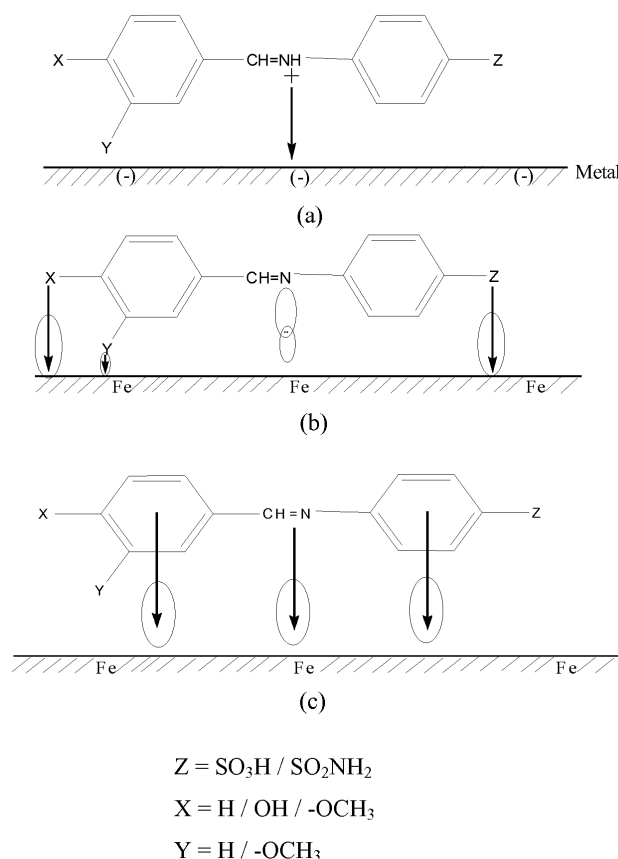

$$Z = SO_3H / SO_2NH_2$$
$$X = H / OH / -OCH_3$$
$$Y = H / -OCH_3$$

Figure 6: Schematic representation of the mode of adsorption of Schiff bases on mild steel surface.(a)electrostatic interaction (b)through lone pair of electron on hetero atoms (c)through π-electron of the rings and the double bond

S.B-2, 3, 5, 6 may be attributed to the presence of electron donating groups -OH and $-OCH_3$. According to Hariharaputhran et al [13] as the number of electron donating substituents on the benzene ring increases, the inhibitive power in general increases. For inhibitors of high molecular weight, a widely spread film is formed on the metal surface which prevents corrosion. Thus the Schiff bases 2, 3, 5 and 6 due to their high molecular weight form a functional blanket preventing the metal from coming into contact with the acid in the corrosive environment. On the basis of the above literature Schiff bases 3 and 6 are expected to have maximum inhibition efficiency due to the presence of electron donating -OH, $-OCH_3$ groups. On the contrary, their inhibition efficiency values are slightly less compared to the inhibition efficiency of Schiff bases 2 and 5 with only $-OCH_3$ groups. This may be due to the greater solubility of the hydroxyl compound in aqueous acid medium leading to the dissolution of the inhibitor film from the metal surface. This is supported by the findings of Quraishi et al. [14], while comparing the inhibition efficiency of the Schiff bases of sulphanilic acid and sulphanilamide, it is clear that all sulphanilamide Schiff bases exhibit a slightly higher inhibition efficiency in comparison to sulphanilic acid Schiff bases. This may be attributed to the presence of a $-NH_2$ group (Nitrogen containing lone pair of electrons) along with the sulphonyl group which also act as an additional anchoring site for adsorption.

4. Conclusion

- All investigated Schiff bases are effective inhibitors for corrosion of mild steel in 1M H_2SO_4.
- They inhibit corrosion by getting adsorbed on the metal surface.
- The inhibition efficiency increases with increase in inhibitor concentration.
- The adsorption of these inhibitors follows Langmuir and Tempkin adsorption isotherms.
- The effect of temperature indicates that the inhibition efficiency decreases with increasing temperature.
- The activation energy (E_a) is higher for inhibited acids then for uninhibited acids.
- The less negative (ΔG^o_{ads}) values indicate spontaneous adsorption of the inhibitors on the metal surface.
- The Tafel constants obtained from potentiodynamic polarization curves indicate that they are mixed type inhibitors.
- Addition of halide ions shows synergism.

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