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Comparism of Corrosion Inhibitory Effect of 4-[(3,4-Dimethoxybenzylidene)Amino]-1,5-Dimethyl-2-Phenyl-1*H*-Pyrazol-3(2*H*)-One in H_2SO_4 and HCl Environment.

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***KEYWORDS*:** corrosive environments, temperature, corrosion, Schiff base, mild steel

ABSTRACT: The inhibition ability of Schiff base, 4-[(3,4-Dimethoxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one on the corrosion of mild steel in 2M H_2SO_4 and HCl was studied by weight loss method.The inhibition efficiency of the Schiff base on mild steel metal was investigated at four temperatures and concentrations. The highest concentration $2x10^{-2}$M of the Schiff base gave inhibition efficiencies of 91%, 89%, 88%,and 60% at temperatures; 30°C, 40°C, 60°C,and 80°C respectively in H_2SO_4 medium, the same concentrations under the same conditions gave inhibition efficiencies of 94%, 95%, 97%,and 98% at temperatures; 30°C, 40°C, 60°C, and 80°C respectively in HCl medium, suggesting that the inhibitor performs better in HCl medium than in H_2SO_4 medium at high temperatures. © JASEM

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One of the factors that affects corrosion of metals is the environment. The more acidic an environment becomes, the higher the rate of corrosion of metals found in it. Corrosion is the deterioration of materials usually metals by chemical interaction with the environment. It is a spontaneous destruction of metals due to heterogeneous chemical reactions (Suraj et al; 2012). When metals are refined they become unstable due to the energy input during refining as a result of that, it tends to undergo a natural process of reverting back or corroding to the original form, known as ore. Corrosion therefore, is one of the most important problems in the industries and the environment since installations are made up of refined metals such as mild steel or alloy materials that corrode. The threat is from gradual to total breakdown of the installations if left unchecked.

Metallic installations or equipment are often exposed to acidic environments in a lot of ways: for example in oil industries, acids such as hydrochloric acid (HCl) and sulphuric acid (H_2SO_4) are used in a process called acidization to loosen hard formulations such as cement or clay which tend to block the flow of crude oil in the pipeline, the acids end up causing corrosion of the equipment to which it is applied. Other processes that bring industrial installations in contact with acids include: acid pickling, and other industrial acid cleaning, acid de-scaling and presence of naphthenic acids which comes with crude oil.

Studies have shown that metal corrosion can be prevented or reduced by using some chemical compounds called inhibitors such as Schiff bases, (Chitra et al., 2010). Corrosion inhibitors are of great practical importance, being extensively employed in minimizing metallic waste in engineering materials (James et al., 2007).

Schiff base, named after Hugo Schiff, is a compound with a functional group that contains a carbon-nitrogen double bond moiety (-CH=N-), with the nitrogen atom connected to an aryl or alkyl group, not hydrogen. (Nic et al., 2006). Schiff bases are typically formed by the condensation of a primary amine and an aldehyde.

The inhibitory efficiency of most inhibitors decreases with increase in temperature (Orubite et al., 2007). The aim of this study is to investigate the corrosion inhibitory efficiency of the studied Schiff base on mild steel in two different acidic media at high temperatures.

Experimental Methods: Synthesis of 4-[(3,4-Dimethoxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Schiff base)

A combination of stirring and reflux method was used to achieve maximum result. A mixture of 3,4-dimethoxybenzaldehyde (57.77mmol, 9.6g) and the corresponding (equimolar) 4-aminoantipyrine (57.77mmol, 11.74g) in an anhydrous methanol

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(60ml) was refluxed at 50^oC for five hours with continuous stirring in the presence of few drops of acetic acid. At completion the solution was cooled, and a heavy yellowish precipitate was obtained. This was collected by filtration and purified by recrystalization from methanol. The product was dried, broken into powder and was weighed (Zeliha et al., 2010). The percentage yield was calculated, and 99% yield was obtained.

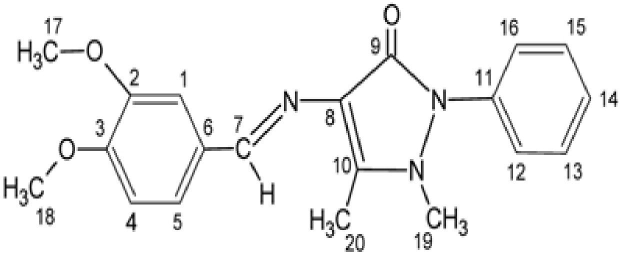

Fig 1: Structure of 4-[(3,4-Dimethoxybenzylidene)amino]-1,5-dimethyl-2-phenyl-1*H*-pyrazol-3(2*H*)-one (Schiff base); (Zeliha et al.,2010).

Preparation of the Mild Steel Coupons **:** Rectangular specimens of mild steel of approximate composition: 0.120% C, 0.900% Mn, 0.066% S, 0.050% P, 0.100% Si, and 98.314% Fe, were cut into dimensions: 2.5cm x 2.0cm x 0.05cm with a small hole of about 2mm in diameter near the upper edge were taken. The hole was to allow passage of rubber thread for suspension of the metal into acid solution. The specimens were cut from the centre of the steel sheet and thoroughly cleaned, buffed and rubbed with emery paper to obtain a mirror-like spotless surface. Then washed in absolute ethanol, degreased by rinsing in acetone, dried and stored in a desiccator to prevent contact with moisture before use.

*Experimental set-up of Corrosion weight loss measurements***:** The corrosion test was carried out using the weight loss method also called total immersion test method (James et al., 2006). Initial weights of the metal coupons were taken. The experiment was set up by transferring 100ml of each of the inhibitor solution of different concentrations into beakers. Seven beakers were set up for inhibitor solutions prepared with 2M H_2SO_4, with one of the beakers bearing blank 2M H_2SO_4 (without inhibitor), this serves as control experiment. The remaining six beakers had inhibitor solution of concentrations; 2 x 10^{-2}M, 1 x 10^{-2}M, 1 x 10^{-3}M, 1 x 10^{-4}M, 1 x 10^{-5}M, 1 x 10^{-6}M. Same was set for inhibitor solutions prepared with 2M HCl.

Finally, the metal coupons were immersed into the solutions and appropriate labeling was made both on the dangling rubber thread fixed to the metal and on the solution container for proper recognition. The metal coupons were retrieved after six hours, dipped into NaOH, washed with ethanol, brushed off the corrosion products on the metal with iron brush, rinsed in acetone, dried, reweighed and dipped once again into the corrodent for the next six hours. This experiment was repeated for seven replicate values of the weight loss. Monitoring the rate at which the metal was corroding in the blank acid solution vis-à-vis the solution of the different concentrations of the inhibited corrodents. This experiment was repeated at room temperature (30^oC – 31^oC) and further performed at temperatures; 40^oC, 60^oC and 80^oC in a thermostatic water bath. The observations and results were recorded, analyzed and presented subsequently.

RESULTS AND DISCUSSIONS

UV-visible Spectra of the Schiff base: The absorption spectra of the compound was obtained in methanol at room temperature. The spectra exhibited three broad absorption bands in the region of 200 nm to 400nm, and could be attributed to n- π* or π-π* transition. The bands are; λ_1 = 235.0 nm which was assigned to -C=C-, λ_2 = 350.0 nm attributed to -CH=N- and λ_3 = 385.0 nm which could be due to charge transfer Adem et al., (2011).

Infrared spectra analysis: FT-IR spectral data of the compound showed strong intensity absorption bands, the most important band is 1649.48cm^{-1} which belongs to –CH=N- (functional group) stretching mode of the Schiff base. Similar 1647cm^{-1} band was reported by Zeliha et al., (2010), Muhammed et al., (2009), Zhaoqi and Pinhua, (2006), Alikabar et al., (2011), Srivestava et al.,(2010). Other observed vibrations are shown in Table 1.

Table 1. Observed Vibrational Frequencies of the Studied Schiff base

Band (cm^{-1})	Functional Group Vibrations
3411.42	O - H of enol
2937.14	C - H of Aromatic
1649.48	C = N of azomethine
1492.74 – 1253.36	C – O
1139.37 – 501.03	Other vibrations on the benzene ring

Corrosion Studies: Mild steel corrodes in 2M sulphuric acid and hydrochloric acid. This was noticed from the loss in mass of the metal in both solutions using the weight loss method, Figures 2 and 3 showed the trend obtained. This was attributed to the presence of hydrogen ion H^+ from acids. H^+ takes up electrons from the mild steel metal to form hydrogen gas (H_2), it was also attributed to increase in the rate of diffusion and ionization of the active species in the corrosion reaction (James and Akaranta, 2009) and (Orubite and Oforka, 2004). Mild steel donates electrons as follows:

$Fe \longrightarrow Fe^{2+} + 2e^-$, $Fe^{2+} \longrightarrow Fe^{3+} + e^-$ forming the anode while H^+ form the cathode, $2H^+ + 2e^-$ $H_2(g)$ accepting electrons, thus electrochemical cell was set up. As such processes continued the metal weared away (corroded) thus the loss in weight.

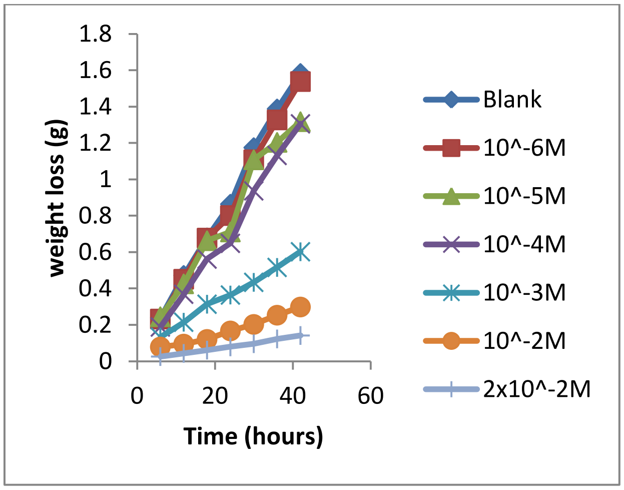

Fig 2: Weight Loss (g) of Mild Steel in Various Concentrations of the studied Schiff Base inhibitor in 2M H_2SO_4 as time progressed.

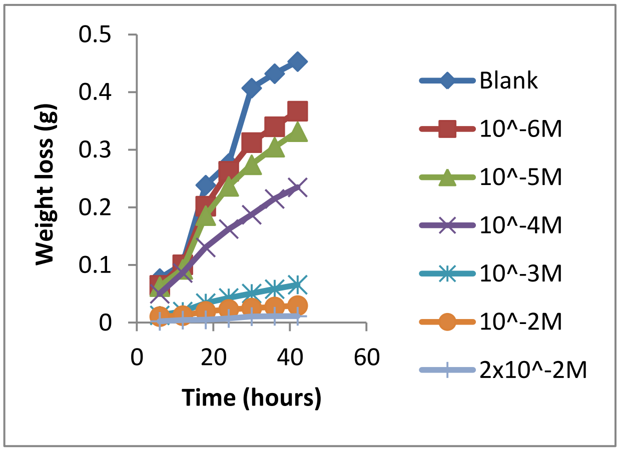

Fig3: Weight Loss (g) of Mild Steel in Various Concentrations of the studied Schiff Base inhibitor in 2M HCl as time progressed

Using the following equations the thermodynamic parameters were calculated to show the different performances of the Schiff base in the two acids. The parameters are:

Rate constant (k) at 30°C, 40°C, 60°C and 80°C for the reaction was calculated using $K = \frac{2.303}{Time} log \frac{wi}{wf}$ (1)

Whre wi and wf are the initial and final weights of metal coupons.

Half life ($t_{1/2}$) at 30°C, 40°C, 60°C and 80°C was calculated using $t_{1/2} = \frac{0.693}{k}$.(2)

The Activation Energy (Ea) ($KJmol^{-1}$) of the Reaction

$E_a = \frac{2.303RT_1T_2(logk_2/k_1)}{T_2 - T_1}$.(3)

Where T_1 and T_2 are the initial and final temperatures respectively, while K_1 and K_2 are the rate constants at T_1 and T_2 respectively. R is the gas constant = 8.314 $Jmol^{-1} K^{-1}$.

Surface Coverage (θ) of the Schiff base on mild steel surface $\theta = \frac{\Delta W_b - \Delta W_i}{\Delta W_b}$.(4)

Heat of adsorption (ΔHads) $\Delta Hads = InKadsRT$ (5)

Where Kads is the coefficient of adsorption, T= temperature.

Free Energy of adsorption (ΔGads) of the reaction $\Delta Gads = -RTIn(55.5Kads)$.(6)

Entropy adsorption (ΔSads) of the Reaction System was obtained from; $\Delta G° = \Delta H° - T\Delta S$ (7)

$$\Delta S^\circ = \frac{-\Delta G^\circ - \Delta H^\circ}{T} \quad (8)$$

Adsorption coefficient (Kads) $Kads = {}^{\theta}/_{c(1-\theta)}$ (9) Where C is the concentration of the Schiff base

Table 2: Summary of the Thermodynamic Parameters of the studied Schiff base at different Concentrations in 2M H_2SO_4 Solution

Inhibitor Concentra-tion(M)	Rate constant (K) (min^{-1}) x 10^{-3}				Activation energy (KJmol^{-1})		Average activation energy (KJmol^{-1})	
	30°C	40°C	60°C	80°C	30°C-40°C	60°C-80°C	30°C-40°C	60°C-80°C 42.79
1.0 x 10^{-6}	4.65	17.19	18.65	26.15	103.11	16.52	107.19	
1.0 x 10^{-5}	3.92	15.06	16.92	24.34	106.15	17.77		
1.0 x 10^{-4}	3.83	14.53	15.42	23.20	105.15	19.96		
1.0 x 10^{-3}	1.78	12.64	13.56	21.76	154.59	23.11		
1.0 x 10^{-2}	0.80	2.37	3.45	20.82	85.65	87.85		
2.0 x 10^{-2}	0.42	1.29	2.09	13.60	88.50	91.54		

Table 3: Summary of the Thermodynamic Parameters of the studied Schiff base at different Concentrations in 2M HCl Solution

Inhibitor Concentrations(M)	Rate constant (K) (min^{-1}) x 10^{-3}				Activation energy (KJmol^{-1})		Average activation energy (KJmol^{-1})	
	30°C	40°C	60°C	80°C	30°C-40°C	60°C-80°C	30°C-40°C	60°C-80°C
1.0 x 10^{-6}	1.00	2.22	3.70	4.20	62.89	6.20	66.58	16.44
1.0 x 10^{-5}	0.95	2.35	2.97	3.62	71.43	9.67		
1.0 x 10^{-4}	0.62	2.03	2.72	3.01	93.54	4.95		
1.0 x 10^{-3}	0.19	0.71	0.92	1.02	103.96	5.04		
1.0 x 10^{-2}	0.08	0.12	0.20	0.36	31.98	28.73		
2.0 x 10^{-2}	0.07	0.11	0.13	0.32	35.65	44.02		

The average activation energy of the reaction decreased in both reaction media with respect to temperature Tables 2 and 3. This was not surprising since reaction rate generally increases with increased in temperature which suggested that the reaction barrier was on the decrease as temperature increased. This observation could be buttressed by the relationship between temperature and activation energy in the equation; In K = In A – Ea / RT (Anusiem, 2004), analyzing the equation by making Ea the subject of the formula we have; -Ea = RT(InK – InA), same as; 1/Ea =RT(InK – InK), showing that Ea is inversely proportional to temperature while the rate constant is directly proportional to temperature due to the conventional increase in reaction rate with temperature.

Table 4: Surface coverage(θ) and Half life($t_{1/2}$) of the studied Schiff base at different concentrations in 2M H_2SO_4 Solution

Inhibitor Concentration (C) (M)	Log C	Surface Coverage (θ)				Half life ($t_{1/2}$) (min) x 10^{2}			
		30°C	40°C	60°C	80°C	30°C	40°C	60°C	80°C
Blank	-	-	-	-	-	-	-	-	-
1.0 x 10^{-6}	-6	0.03	0.14	0.12	0.10	1.49	0.40	0.37	0.27
1.0 x 10^{-5}	-5	0.17	0.17	0.16	0.14	1.77	0.46	0.41	0.28
1.0 x 10^{-4}	-4	0.18	0.20	0.18	0.17	1.81	0.48	0.45	0.30
1.0 x 10^{-3}	-3	0.62	0.25	0.24	0.22	3.89	0.55	0.51	0.32
1.0 x 10^{-2}	-2	0.81	0.80	0.80	0.45	8.66	2.92	2.01	0.33
2.0 x 10^{-2}	-1.7	0.91	0.89	0.88	0.60	16.50	5.37	3.32	0.51

The surface coverage increased with increase in concentration of the inhibitor in both reaction media which means that more of the inhibitor was adsorbed at high concentration. Comparing Tables 4 and 5 showed that in HCl medium the surface coverage increased with temperature unlike H_2SO_4, indicating more protection of the metal at high temperature in HCl than in H_2SO_4 which showed a decrease due to the presence of acidic conjugate base $HSO4^-$ from the partial ionization of H_2SO_4.The observation was further supported by the longer half life of the inhibitor in HCl than in H_2SO_4 medium as shown in Tables 4 nad 5.

Table 5: Surface coverage(θ) and Half life($t_{1/2}$) of the studied Schiff base at different concentrations in 2M HCl Solution

Inhibitor Concentration (C) (M)	Log C	Surface Coverage (θ)				Half life ($t_{1/2}$) (min) x 10^2			
		30°C	40°C	60°C	80°C	30°C	40°C	60°C	80°C
Blank	-	-	-	-	-	-	-	-	-
1.0 x 10^{-6}	-6	0.19	0.19	0.20	0.25	6.93	3.12	1.87	1.65
1.0 x 10^{-5}	-5	0.27	0.19	0.21	0.30	7.29	2.95	2.33	1.91
1.0 x 10^{-4}	-4	0.48	0.33	0.42	0.57	11.17	3.41	2.55	2.30
1.0 x 10^{-3}	-3	0.85	0.74	0.80	0.86	36.47	40.76	7.53	6.79
1.0 x 10^{-2}	-2	0.93	0.95	0.95	0.97	86.63	57.75	34.65	19.25
2.0 x 10^{-2}	-1.7	0.94	0.95	0.97	0.98	99.00	63.00	53.31	21.66

Table 6: The average change in heat of adsorption Hads, free energy of adsorption ΔGads, entropy of adsorption ΔSads, and equilibrium constant of adsorption Kads of the inhibitor in H_2SO_4 at different temperatures used.

Temperature (°C)	ΔHads (KJMol^{-1})	ΔGads (KJMol^{-1})	ΔSads (KJmol^{-1})	Kads (M^{-1})
30	3.55	-13.67	16.24	4.10
40	4.14	-14.59	17.88	4.90
60	4.61	-15.73	18.56	5.28
80	7.29	-19.08	26.07	12.00

Table 7: The average change in heat of adsorption Hads, free energy of adsorption ΔGads, entropy of adsorption ΔSads, and equilibrium constant of adsorption Kads of the inhibitor in HCl at different temperatures used.

Temperature (°C)	ΔHads (KJmol^{-1})	ΔGads (KJMol^{-1})	ΔSads (KJmol^{-1})	Kads (M^{-1})
30	2.61	-12.73	12.82	2.82
40	2.26	-12.71	11.27	2.38
60	1.05	-12.17	06.86	1.46
80	0.06	-11.73	03.49	0.98

The negative free energy of adsorption (ΔGads) implies the spontaneous adsorption of the inhibitor, while the positive values of both ΔSads and ΔHads could be due to displacement of water molecules by the Schiff base inhibitor. The constant or thresh hold requirement for adsorption called equilibrium constant of adsorption Kads decreased with temperature in HCl while reverse was the case in H_2SO_4 medium Tables 6and 7. All together making adsoption of the inhibitor faster and easier in HCl than in H_2SO_4.

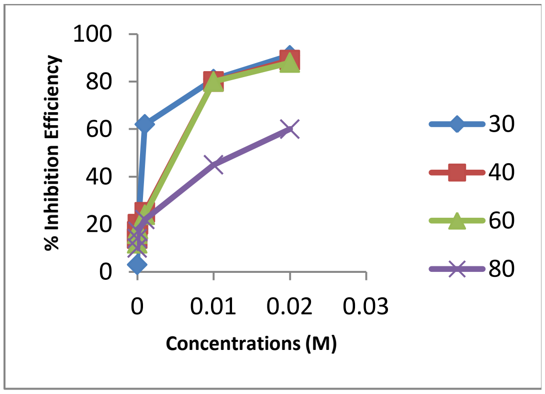

Fig 4: Inhibition Efficiency of Mild Steel in 2M H_2SO_4 for Various Concentrations of Schiff

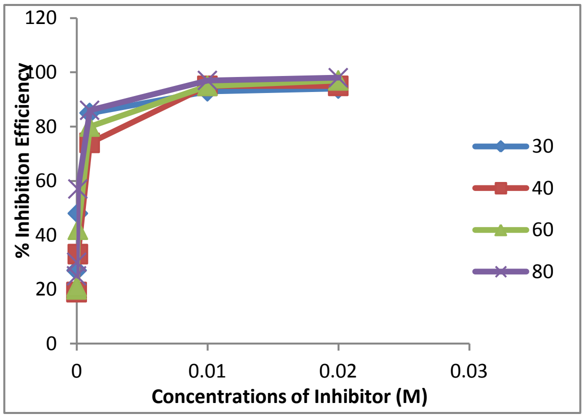

Fig 5: Inhibition Efficiency of Mild Steel in 2M HCl for Various Concentrations of Schiff Base at Different Temperatures (°C)

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Base at Different Temperatures (^{o}C).

The trend of inhibition efficiency calculated for both acids as the concentrations of the Schiff base increased showed that the percentage inhibition efficiency of both acids increased with increase in the additive concentrations (Figures. 4 and 5) though the observation was stronger in HCl which maintains close uniformity for the four temperatures than H_2SO_4. Similar trend was recorded by Chitra et al., 2010, Ashish and Quraishi, (2012). This observation could be due to the fact that the more the inhibitor molecules present in the solution the more they are properly adsorbed or the better the coverage they could make on the surface of the metal.

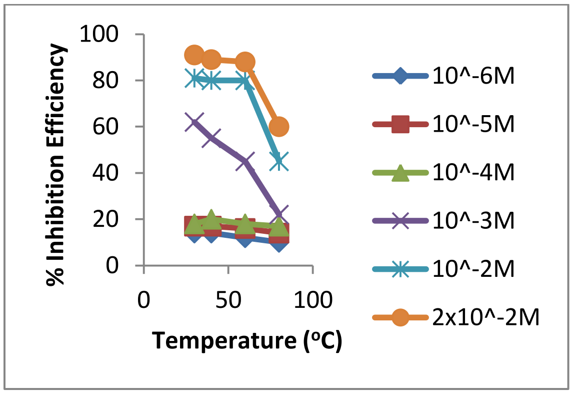

Fig 6: Inhibition Efficiency of Mild Steel in 2M H_2SO at Different Temperatures

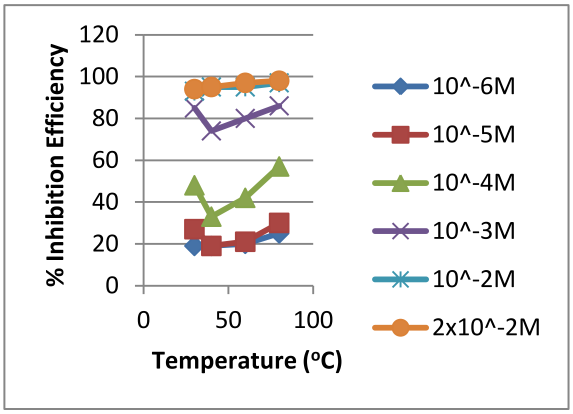

Fig 7: Inhibition Efficiency of Mild Steel in 2M HCl at Different Temperatures

In Figure 6, the graph indicated that as the temperature of the reaction system was increased there was a significant reduction in the inhibition efficiency from 91% to 60%. The reason is obvious, increase in temperature means increase in the reaction rate (Anusiem, 2004). The donation of H^+ and subsequent attack of the mild steel was more favoured than the adsorption of the Schiff base inhibitor on the mild steel surface in H_2SO_4 medium.

On the other hand, using similar conditions, increase in temperature rather showed a remarkable increase in the inhibition efficiency of mild steel corrosion in 2M HCl. From the plot in Figure 7, it was clear that for every increase in temperature there was proportional increase in the inhibition efficiency except for concentrations of 10^{-5}M, 10^{-4}M and 10^{-3}M that recorded drops at temperature of 40^{o}C before rising again, similar drop was recorded by Orubite et al., 2007.

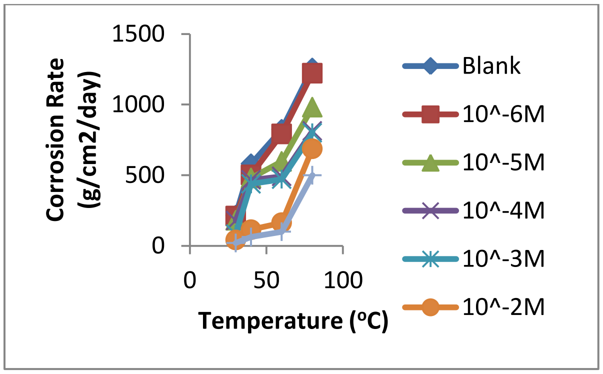

Fig 8: Corrosion Rate of Mild Steel in 2M H_2SO_4 for Various Concentrations of Schiff Base at Different Temperatures

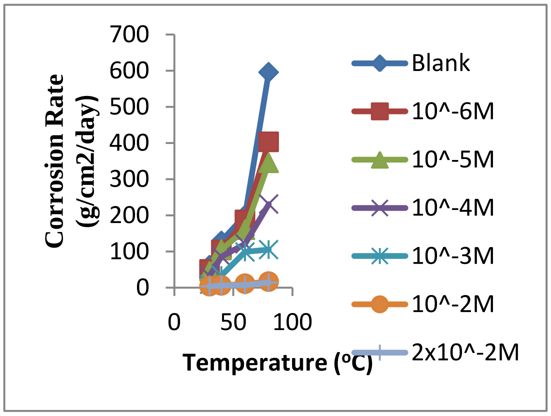

Fig 9: Corrosion Rate of Mild Steel in 2M HCl for Various Concentrations of Schiff Base at Different Temperatures

In this experiment, corrosion rate increased with increase in temperature as shown in Figure9. This situation is generally true for most chemical reactions; that is, increase in temperature increases rate of chemical reactions. The trend is consistent with the observations of previous authors (Okafor et al; 2010) who reported that the tendency for partial desorption of the inhibitor from the metal surface and the metal dissolution increases with temperature. But this observation was resisted by strong adsorption in the HCl medium as seen above. Figure 9

The strong adsorption of the inhibitor in HCl compared to H_2SO_4 could be attributed to the nature of the conjugate bases of the two acids. The conjugate base of H_2SO_4 is HSO_4^- which is still an acid ready to donate more H^+, and more H^+ means more electrons required from the Fe (mild steel) thus more corrosion. In addition, HSO_4^- destabilizes the formation of protective film during adsorption. A protonated Schiff base stabilizes the adsorption process by electrostatic attraction, when it finds itself in between the positive metal and the negative conjugate base. In view of this, it forms a more stable synergistic adsorption with the chloride ion Cl^- than the HSO_4^- which has to disintegrate again to give out H^+ in order to transform into a more stable conjugate $SO4^{2-}$ before a stable protective film would be reestablished.

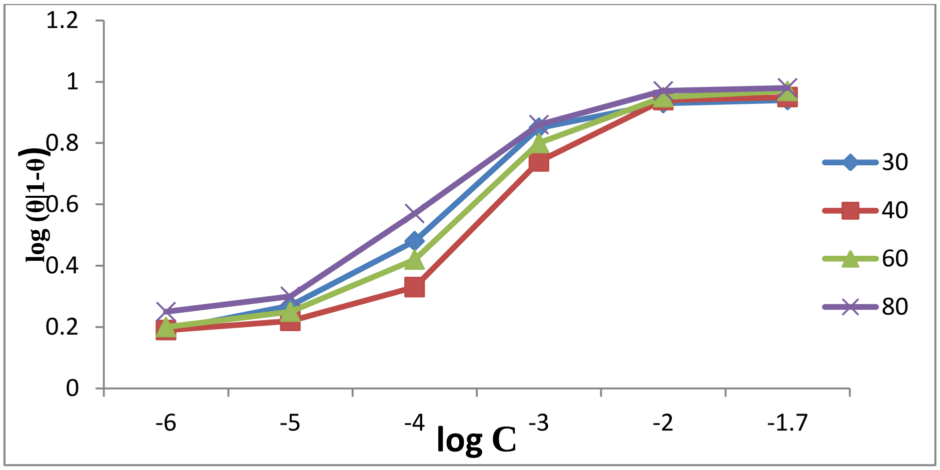

Fig 10: Langmuir adsorption isotherm plot for the Schiff base adsorption on mild steel in HCl medium at different temperatures (^oC).

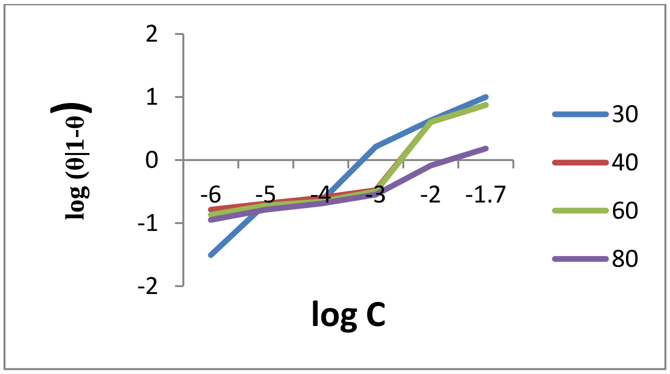

Fig 11: Langmuir adsorption isotherm plot for the Schiff base adsorption on mild steel in H_2SO_4 medium at different temperatures (^oC).

No unity gradient in all the isotherm plots at all the temperatures for the two acids though that of HCl has more unified gradients showing less interactions of the molecules of the inhibitor in this medium relative to H_2SO_4 medium. This also confirmed a better performance of the inhibitor in HCl medium.

Conclusion: The studied Schiff base inhibited corrosion of mild steel more at high temperatures in HCl than in H_2SO_4 media under the same experimentalconditions

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