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Synthesis of Some Schiff Bases and Investigation of their Corrosion Inhibition Efficiencies for Aluminum in Acidic Media.

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Abstract: *In this study, the effectiveness of some synthesized Schiff bases as corrosion inhibitors was investigated using gravimetric and FTIR methods. The Schiff ba: the Schiff bases were characterized using Fourier transform infrared spectroscopy (FTIR). The Schiff bases having different ligands ((namely, ligand (L), copper ligand (CuL) and iron ligand (FL))) were investigated for their efficiencies towards the inhibition of the corrosion of aluminum in HCl (0.1 M) and H_2SO_4 (0.05 M) respectively using gravimetric method. The results show good inhibition efficiencies that increase with an increase in the concentration of the inhibitor but decrease with a temperature rise and with the period of contact. Langmuir adsorption isotherm best fitted the adsorption characteristics of the inhibitors and the adsorption was spontaneous, endothermic, ordered and consistent with the mechanism of physical adsorption. The synthesized Schiff bases are therefore considered as useful products for the inhibition of the corrosion of aluminum in HCl and H_2SO_4 media. However, efficiency towards inhibition is better in H_2SO_4 than in HCl due to passivation sustenance.*

Keywords: *Corrosion, aluminum, acid media, inhibition, Schiff bases,*

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1.0 Introduction:

Industrial development is vital in the history of any developed country. However, most industries use various types of metals or metallic alloys in some of their functional operations such as acid cleaning, etching, descaling, etc. Such operations often create the susceptibility of the metal to corrosion (Awe *et al*., 2015; (Ita, 2004a; Li *et* al., 2014; Oguzie *et al*., 2004; 2007; Okafor *et al*., 2007; Patel *et al*.,2013; Umoren *et al*., 2009). The protection of metals against corrosion has a significant impact on the life span of metals in some industries such as iron metallurgical, fertilizer, oil and gas, etc. Corrosion is an electrochemical reaction that can be controlled by either modifying the anodic, cathodic or both anodic and cathodic reactions (Eddy and Ameh, 2021). The use of measures such as cathodic and anodic protection, electroplating, oiling and coating are useful in the protection of metals against corrosion (Eddy *et al*., 2008). Also, corrosion inhibitors are those substances that are needed in minute concentration for the retardation of the corrosion of metals (Eddy and Odoemelam, 2008; Nebaida, 2018). Extensive works have been carried out on the chemistry of corrosion inhibitors and research data have revealed that effective corrosion inhibitors Results of corrosion cost studies for Africa are not readily available, however as of 2017 South Africa reportedly spent about $83 million to combat corrosion. In Nigeria, it was estimated that the oil and gas industry loses up to $765 million yearly to the corrosion of oil pipelines (Muanya, 2017). Similarly, the annual cost of all forms of corrosion to the oil and gas industries in the US is $13.4 billion (Adekunle, 2019). Given the above, industrial facilities exposed to corrosion are often protected against corrosion by adopting several options including painting, oiling, cathodic and anodic protection, etc. However, the use of inhibitors is one of the best options available for the protection of metals against corrosion (Awe *et al*., 2015; Eddy, 2010; Eddy and Awe, 2018).

The starting mechanism for corrosion inhibition is the adsorption of the inhibitor on the surface of the metal (Awe *et al*., 2015). The adsorption may involve the transfer of charge from the charged inhibitor to the charged metal surface or the transfer of electrons in the same direction. However, the back transfer is also possible, that is from the metal to the inhibitor, which often leads to the formation of a feedback bond (Eddy *et al.,* 2015, 2010). Several corrosion inhibitors are effective for the inhibition of the corrosion of metals (Eddy *et al*., 2015, Awe *et al*., 2015, Eddy and Awe, 2018). However, the current challenges in the corrosion inhibition industries is not effectiveness but other requirements such as eco-friendliness, cost-effectiveness, biodegradability and ease of access (Awe *et al*., 2018; -Eddy *et al*., 2015; Momoh-Yahaya *et al*, 2012). The fundamental role in assessing compounds for their corrosion inhibition potentials include the presence of heteroatoms, multiple bonds, pi-electron, conjugated system, aromatic system and high molecular weight (Awe, 2019; Ameh and Eddy, 2019; Anand and Chitra, 2020; Chahul *et al,* 2017; Essien *et al*., 2021; Iroha and Ukpe, 2020)

Consequently, several Schiff bases have been investigated for their corrosion inhibition efficiency due to the presence of >C=N– groups which allow the corresponding Schiff bases to get adsorbed on the surface of metals and their alloys to form a monolayer on the surface spontaneously. Therefore, the present study is aimed at elucidating the chemical structures of the Schiff base to investigate their corrosion potentials for aluminum in acid media.

2.0 Materials and Method

2.1 Materials

The aluminum sheet with dimension, used for the study was obtained from Ameh Technical Company, Makurdi. The sheet was cut into different coupons each of dimension, 5cm × 4cm × 0.15 cm. The coupons were polished with different grades of abrasive paper (#400 to #1200), washed with distilled water, rinsed with absolute ethanol, cleaned in acetone and allowed to dry in the air. 4-amimophenol and 4-bromobenzaladhyde were obtained from Zayo-sigma Chemicals Limited while the other reagents including Hydrochroric acid, sulphuric acid, citric acid, acetone, ethanol, potassium hydroxide, Co(II), Ni(II), Cu(II), Ti(II), Cd(II) and Zn(II) salts were obtained from Emole Nig Ltd and the Chemistry Laboratory of NDA Kaduna, UAM Makurdi and ABU Zaria. Acid concentrations of 0.1 M HCl and 0.05 M H_2SO_4 were used for gravimetric (weight loss) while the concentration for the inhibitors was 0.1g/L, 0.2g/L, 0.3g/L, 0.4g/L and 0.5g/L respectively (Eddy *et al*, 2010; Momoh-Yahaya, 2014).

2.2 Spectroscopy analysis

The synthesized inhibitor and corrosion material were characterized with FTIR and UV vis spectrophotometer (Perkin-Elmer Lambda 35 UV-Vis spectrophotometer).

2.3 Synthesis of Schiff bases

2.3.1 Synthesis of Schiff base ligands (L)

The Schiff base ligand was prepared by taking 20 mmol of 3,5-diiodosalicyaldehyde and 20mmol of 4,4’-diaminodiphenylamine in the ratio of 1:1 in a mortar. 5 drops of citric acid were added to adjust After the completion of the reaction, cold water was added and the collected precipitate was filtered and dried (Devidas *et al, 2011;* Veni *et al*, 2014).

2.3.2 Synthesis of metal complex

This was achieved by grinding a mixture of the Schiff base ligand and potassium hydroxide using mortar and pestle for 20 minutes. The entire mixture was blended sich that the ratio of the ligand, KOH and metal (II) salts was 2:2:1 for the synthesis of the metal (II) complexes (Devidas *et al, 2011;* Veni *et al*, 2014).

2.4 Gravimetric measurement

The gravimetric study was conducted using the standard protocol for weight loss measurements. The respective test solution was made to come in contact with the aggressive solutions (acid media) respectively. The experiments were conducted at different temperatures. Each metal coupon was allowed to remain in the corrodent for a specified time before it was removed, washed (to remove the corrosion product) and re-weighed. The difference in weight before and after immersion was recorded as the weight loss. The corrosion rate, degree of surface coverage and inhibition efficiency were calculated using equations 1, 2 and 3 respectively.

$$\frac{weight\ loss}{A} \qquad (1)$$

$$\frac{Weight\ loss\ of\ metal\ coupon}{Weight\ of\ metal\ coupon\ before\ immersion} \qquad (2)$$

$$\frac{Weight\ loss\ of\ metal\ coupon}{Weight\ of\ metal\ coupon\ before\ immersion} \times \frac{100}{1} \qquad (3)$$

where A is the surface area of the metal coupon

2.4.1 Theoretical models

A modified form of the Arrhenius-type relationship between the corrosion rate (*CR*) of a metal in acidic media and temperature (*T)* is used in the determination of the activation energies (E_a).

$$log\left(\frac{CR_2}{CR_1}\right) = \frac{E_a}{2.303R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \qquad (4)$$

where R is molar gas constant, T_1 = 303K and T_2 = 333K.

The heat of adsorption of the inhibitors can be calculated using equation 5 (Momoh-Yahaya *et al, 2014*),

$$Q_{ads} = \left(\frac{\theta_2}{1-\theta_2} - \frac{\theta_1}{1-\theta_1}\right) \neq \left(\frac{T_2 T_1}{T_2 - T_1}\right) \qquad (5)$$

The enthalpy and entropy of activation of the corrosion process can be evaluated from the Transition state plot according to equation 6

$$\log\left(\frac{CR}{T}\right) = \left\{\log\left(\frac{R}{hN_A}\right) + \frac{\Delta Sa}{2.303R}\right\} - \frac{\Delta Ha}{2.303RT} \qquad (6)$$

where CR is the corrosion rate at temperature T, R is the molar gas constants, n is the Avogadro’s constant, 6.0225×10^{23}, and h is the Planck’s constant. A plot of $Log\ {CR}/{T}$ versus ${1}/{T}$ will be a straight line graph with a slope of ($-\ {\Delta H}/{2.303RT}$) and an intercept $\left[Log\left({R}/{nh}\right) + {\Delta S}/{2.303R}\right]$ from which the values of ΔH and ΔS can be calculated (Olasehinde *et al*, 2013).

The Langmuir adsorption isotherm was established by plotting log(C/θ) against logC according to equation 7

$$log\left(\frac{C}{\theta}\right) = logC - logk_{ads} \qquad (7)$$

where C is the concentration of the inhibitor in the bulk electrolyte, θ is the degree of surface coverage and k_{ads} is the Langmuir adsorption constant. The Langmuir model applies to a single adsorption layer (monolayer) process and expects the slope value to be unity for ideal fitting. From the Langmuir adsorption constant, the free energy of adsorption of the inhibitor was evaluated using the Gibb Helmholtz equation, that is equation 8

$$\Delta G_{ads} = -RT\ln k_{ads} \tag{8}$$

Adsorption is spontaneous if the free energy change is negative. Free energy values are also an index for predicting the mechanism of adsorption. Values of ΔG_{ads} between 0 and -20 kJ/mol are typical for the physisorption mechanism. Above 40 kJ/mol, the values interpreted the chemisorption mechanism but both mechanisms for ΔG_{ads} values between -20 and -40 kJ/mol.

3.0 Results and Discussion

3.1 Spectroscopy study

The IR data of the ligand and all the complexes were recorded within the range, 0 to 4000 cm^{-1} presented in Table 1. The preliminary identification of the Schiff base was obtained from the disappearance of the characteristic band attributed to the aldehyde stretching ν(C=O). The strong band observed at 1644.21 cm^{-1} in the spectra of the Schiff base can be assigned to -C=N stretching vibration (Sani *et. al.,* 2018). There is a shift in the bands of the complexes that range from 1578.58 to 1599.40 cm^{-1} when compared with the free ligand. This shows that there is a coordination of the metal center with azomethine nitrogen (Iorungwa *et. al.,* 2020). The spectrum of the Schiff base also shows strong absorption at 3436.33 cm^{-1} which can be assigned to O-H stretching vibration and 1409.33 cm^{-1} which can be assigned to the aromatic C-C stretching vibration (Sani *et. al.,* 2018). The phenolic C-O stretching frequency of the Schiff base is seen at 1245 cm^{-1} which is within the acceptable range found in the literature (Vadivela,and Dhamodaranba 2015). This phenolic C-O band of the Schiff base was shifted in the spectrum of the complexes and may be due to deprotonation and coordination of the hydroxyl oxygen to the metal ion. (Sani *et. al.,* 2018). New bands found in the region 403.41 - 460.95 cm^{-1} are assigned to M-O and those between 507.70 – and 599.76 cm^{-1} are assigned to M-N vibrations in the complexes which were not present in the Schiff base ligand. (Sani *et. al.,* 2018; Iorungwa *et. al.,* 2020). The appearance of M-N and MO vibration supports the involvement of N and O atoms in the complexation with metal ions (Tella *et al*., 2011, Veni *et al*., 2014).

Table 1: Infrared data of the synthesized complexes

Compounds	ν(C=N)	ν(O-H)	ν(C-O-C)	ν(C-C)	ν(M-O)	ν(M-N)
Ligand	1644.21	3436.33	1047.00	1245.00	-	-
Ni(II) complex	1585.20	3414.00	1066.45	1249.56	403.41	517.87
Co(II) complex	1583.09	3435.00	1015.19	1248.48	407.61	513.47
Zn(II) complex	1578.58	3410.00	1019.93	1249.00	460.95	525.65
Cu(II) complex	1599.40	3432.32	1043.84	1331.65	425.39	507.70
Fe(III) complex	1589.00	3263.00	1020.67	1226.31	443.19	599.76

3.2 Corrosion inhibition studies

Tables 2 and 3 respectively presents the inhibition efficiencies and corrosion rates of aluminum in various acid media at various temperature. The results presented indicate that the rate of corrosion of aluminum in HCl and H_2SO_4 solution increases with an increase in time but decreased with an increase in the concentration of the Schiff base (Awe and Eddy, 2018; Oguzie *et al*., 2012a). We also noted that the corrosion rate of aluminum in HCl was higher than the rate in H_2SO_4. Aluminum has a high resistance to corrosion due to the formation of oxide film on the surface which usually protects it in air or neutral solutions (Eddy *et al*., 2015; Emregul, 2006; Poubaix, 1974). The sustenance of this passivation layer determines how long the metal can remain in an aggressive solution without corrosion attack. Sulphutiacid is a dibasic acid and has higher acidic strength than HCl. However, H_2SO_4 can form a relatively stable complex with the passivating aluminum surface while HCl dissolves it faster, hence the observed variation in the rate of aluminum corrosion in the two aggressive solutions.

The results obtained also indicate that the Schiff bases are adsorbed on the surface of the aluminum in both media and consequently inhibit the corrosion of aluminum. As expected, the inhibition efficiencies of the Schiff bases for aluminum in H_2SO_4 are better than the efficiency in HCl.= (Tables 2 and 3). The inhibition efficiencies of the investigated Schiff bases decreased with an increase in temperature indicating that the adsorption of the extracts supports the mechanism of physical adsorption (Awe and Eddy, 2018; Ihebrodike *et al*., 2012)

Table 2. Inhibition efficiencies and corrosion rate of aluminum of the various inhibitors in 0.1 M HCl at 303K and 333K respectively

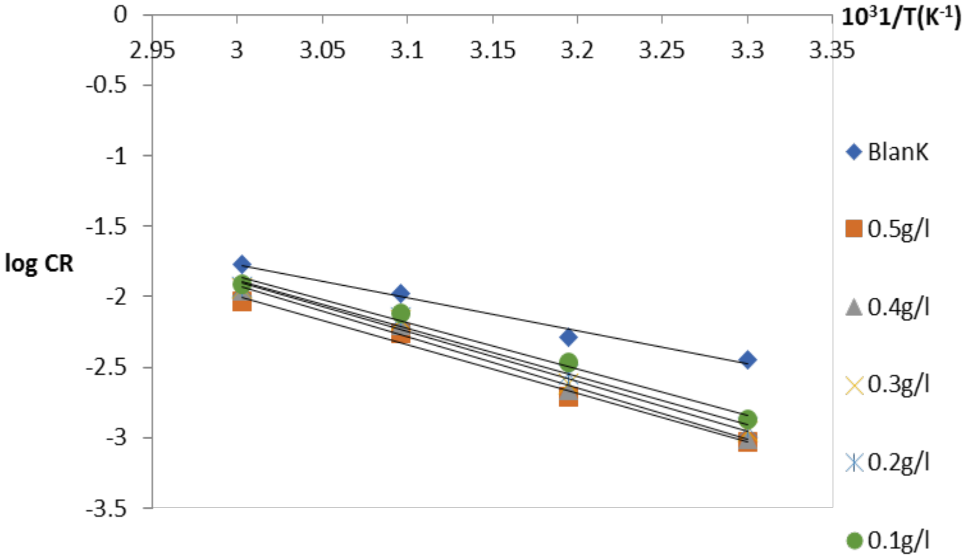
System	Inhibition efficiency (%)			CR ($gh^{-1}cm^{-2}$)		
at 303K	**L**	**CuL**	**FL**	**L**	**CuL**	**FL**
Blank	-	-	-	0.00355	0.00355	0.00355
0.1g/l	62.12	56.56	64.78	0.00134	0.00154	0.00125
0.2g/l	66.66	64.63	67.76	0.00118	0.00126	0.00114
0.3 g/l	68.85	66.19	68.85	0.00111	0.00120	0.00111
0.4 g/l	72.61	68.85	69.01	0.00097	0.00111	0.00110
0.5 g/l	73.55	69.17	70.10	0.00094	0.00109	0.00106
at 333K						
Blank	-	-	-	0.01686	0.01686	0.01686
0.1 g/l	26.85	26.26	12.65	0.01233	0.01243	0.01473
0.2 g/l	30.18	33.44	17.13	0.01177	0.01122	0.01397
0.3 g/l	32.12	35.55	21.21	0.01144	0.01087	0.01328
0.4 g/l	35.12	36.93	22.43	0.01094	0.01063	0.01308
0.5 g/l	45.50	40.42	22.50	0.00919	0.01004	0.01307

Table 3. Inhibition efficiencies and Corrosion rate of aluminum of the various inhibitors in 0.05 M H_2SO_4 at 303K and 333K respectively

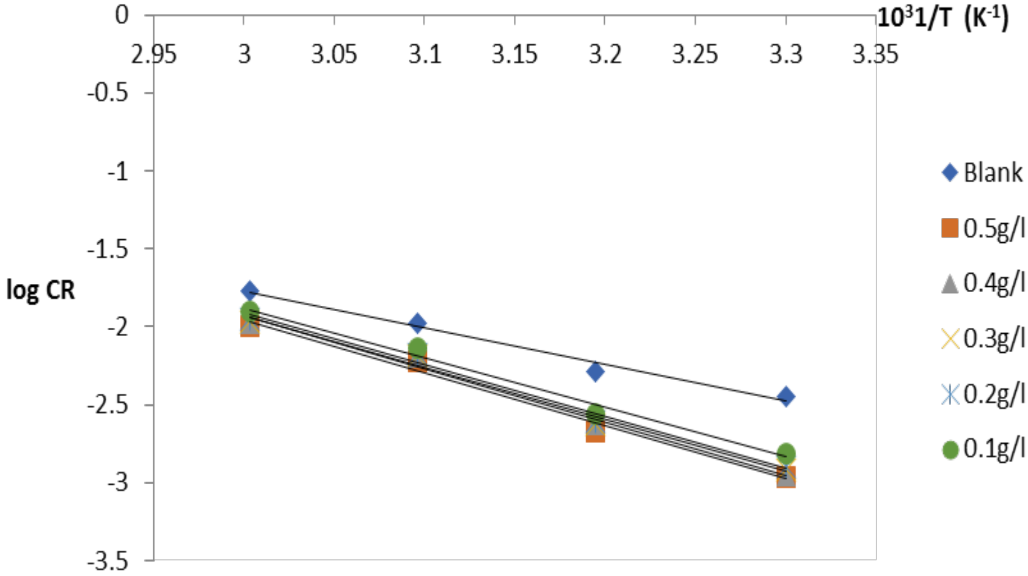
System	Inhibition efficiency (%)			Corrosion rates ($gh^{-1}cm^{-2}$)		
at 303K	L	CuL	FL	L	CuL	FL
Blank	-	-	-	8.33E-05	8.33E-05	8.33E-05
0.1g/l	20.00	20.00	-6.66	6.67E-05	6.67E-05	8.89E-05
0.2g/l	20.00	20.00	6.66	6.67E-05	6.67E-05	7.78E-05
0.3g/l	20.00	20.00	6.66	6.67E-05	6.67E-05	7.78E-05
0.4g/l	20.00	20.00	6.66	6.67E-05	6.67E-05	7.78E-05
0.5g/l	40.00	40.00	6.66	0.00005	0.00005	7.78E-05
at 333K						
Blank				0.00016	0.000483	0.000483
0.1g/l	-5.74	-33.33	-12.64	0.00016	0.000644	0.000544
0.2g/l	-4.59	-33.33	-5.74	0.00016	0.000644	0.000511
0.3g/l	-4.59	-28.73	-3.44	0.00016	0.000622	0.000500
0.4g/l	-2.29	-10.34	-5.74	0.0001	0.000533	0.000511
0.5g/l	-1.14	-9.19	-4.59	0.000133	0.000528	0.000506

3.3 Effect of temperature

The effect of temperature on the corrosion of aluminum in the absence and presence of various concentrations of the inhibitors was investigated using the Arrhenius state equations and the plots obtained are shown in Figs. 1 and 2 for the different media.



(a)



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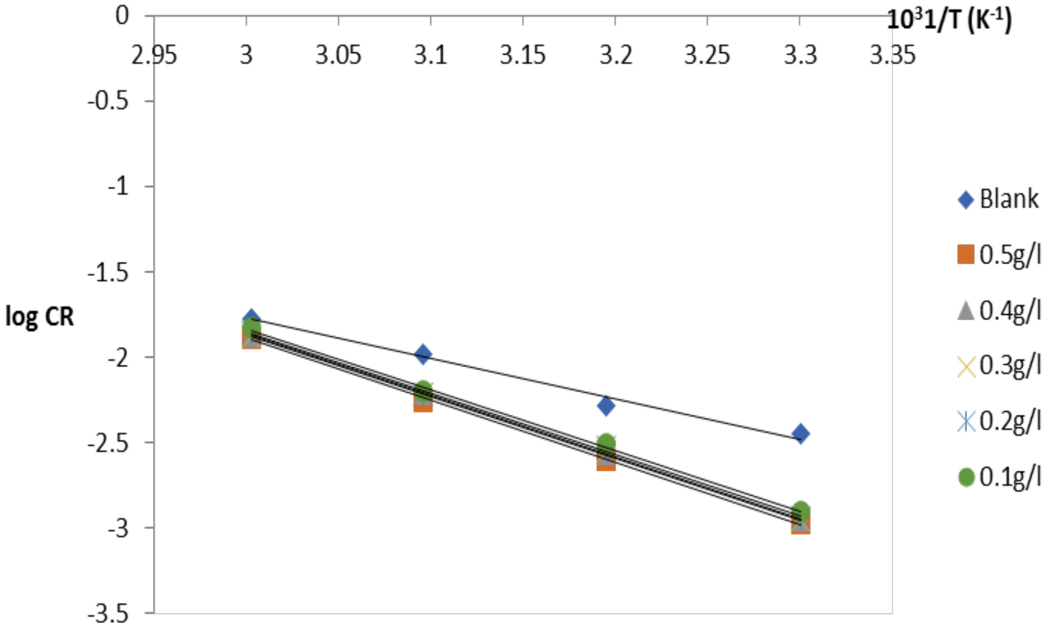
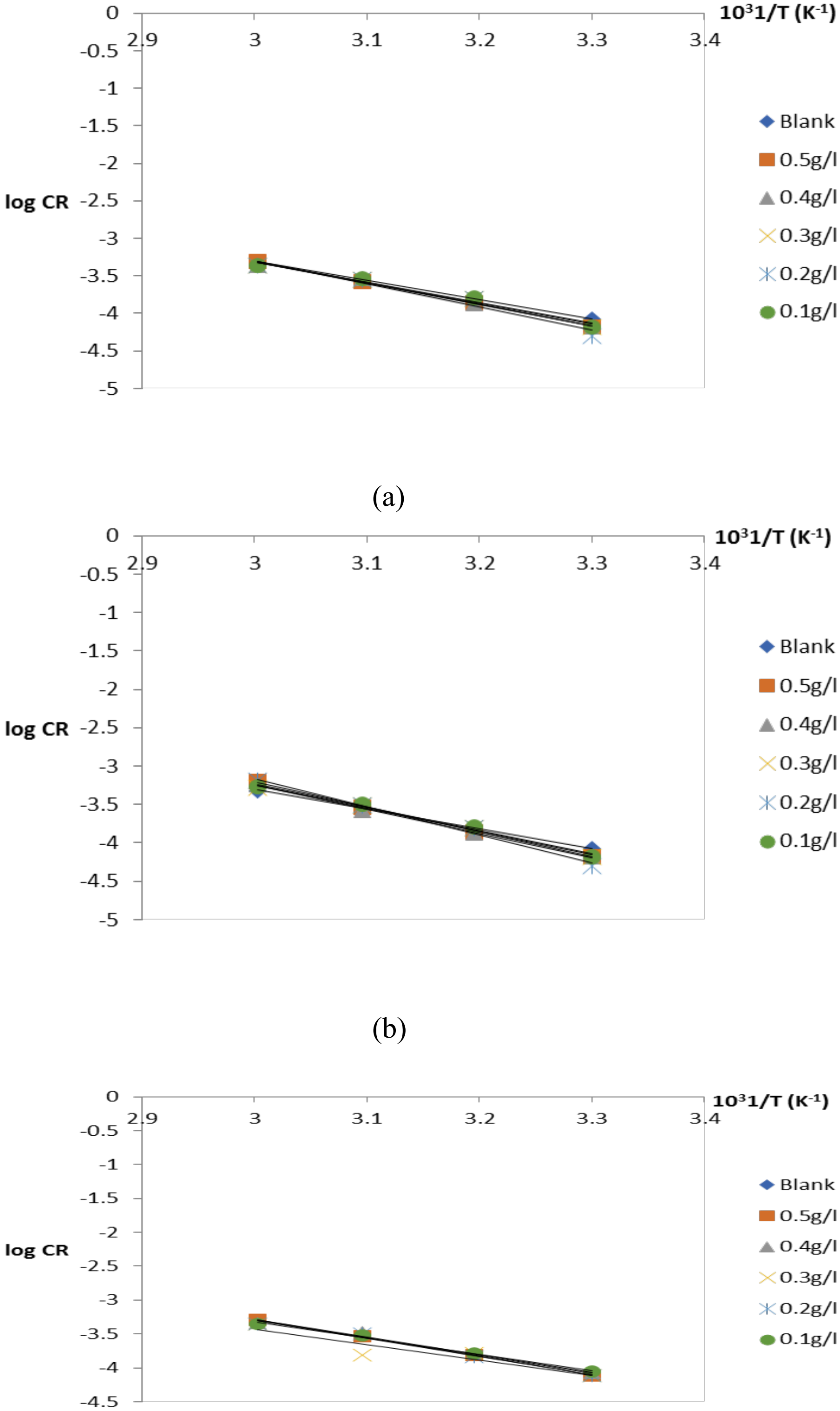


(c)

Fig. 1: Variation of log (corrosion rate) of aluminium with inverse temperature in 0.1 M HCl containing various concentration of a) L b) CuL and (c) FL



(a)

(b)

(c)

Fig. 2. Variation of log (corrosion rate) of aluminium with inverse temperature in 0.05 M H_2SO_4 containing various concentration of a) L b) CuL c) FL

. From the calculated activation energies (Tables 4 and 5, it is obvious that the presence of inhibitor increases the activation energy of the system and thus present a greater energy barrier against further corrosion (Awe and Eddy, 2018; Oguzie *et al*., 2012c). This tendency toward corrosion retardation is observed to increase with the inhibitor's concentration as reported elsewhere (Eddy *et al*., 2010; Oguzie *et al*., 2012b; Ihebrodike *et al*., 2012; Awe and Eddy, 2018). Also, the evaluated values of the activation energies are within the limits expected for the mechanism of physical adsorption (i.e $E_a < 80$ kJ/mol) (Eddy *et al*., 2010, 2011).

The enthalpy and entropy of adsorption were calculated from the slope and intercept of the Transition state plots are presented in Tables 6 and 7, Evaluated values of the entropy are negative and suggest that there is an ordered adsorption process between the inhibitors and the metal surface (Lebrini *et al*., 2010).. However, the adsorption is endothermic because enthalpies are positive.

3.4 Adsorption isotherm

Best suited isotherms established (after a series of tests for several isotherms) for the adsorption of the inhibitors on the aluminum surface was the Langmuir isotherm, shown in Figure 4, values of adsorption free energy estimated from the Helmholtz equation were negatively less than -40 kJ/mol. This confirms that the mechanism for the adsorption of the inhibitors on the surface of the metal is physical adsorption (Awe and Eddy., 2018; Eddy *et al*., 2015 and Oguzie *et al*., 2012c)

Table 4. Thermodynamic parameters for the adsorption of various concentration of the inhibitor on aluminium surface in 0.1 M HCl

	System	Ea(kJmol^{-1})	Qads (kJmol^{-1})
	Blank	44.94	-
L Al	0.1g/l	62.61	-41.87
	0.2g/l	65.67	-42.84
	0.3g/l	67.72	-39.35
	0.4g/l	69.96	-48.19
	0.5g/l	66.25	-33.65
CuL Al	0.1g/l	60.75	-36.36
	0.2g/l	63.43	-36.11
	0.3g/l	64.03	-35.43
	0.4g/l	65.29	-37.15
	0.5g/l	64.64	-33.44
FL Al	0.1g/l	68.16	-71.09
	0.2g/l	69.10	-64,86
	0.3g/l	69.06	-58.88
	0.4g/l	69.01	-57.08
	0.5g/l	69.79	-58.43

Table 5. Thermodynamic parameters for the adsorption of various concentration of the inhibitor on mild steel and aluminum surface in 0.05 M H_2SO_4

		Al		
	System	**E_a(kJmol^{-1})**	**Q_{ads} (kJmol-1)**	
	Blank	49.46		
	0.1g/l	51.91	-90.92	
	0.2g/l	59.22	-93.20	
L	0.3g/l	52.64	-76.34	
	0.4g/l	54.03	-71.83	
	0.5g/l	55.64	-70.17	
	0.1g/l	58.32	-31.61	
	0.2g/l	70.17	-39.31	
CuL	0.3g/l	58.02	-40.53	
	0.4g/l	60.08	-33.18	
	0.5g/l	62.30	-32.31	
	0.1g/l	46.11	-54.61	
	0.2g/l	51.03	-66.82	
FL	0.3g/l	43.08	-68.22	54.91
	0.4g/l	49.44	-73.87	62.02
	0.5g/l	52.35	-75.07	67.74

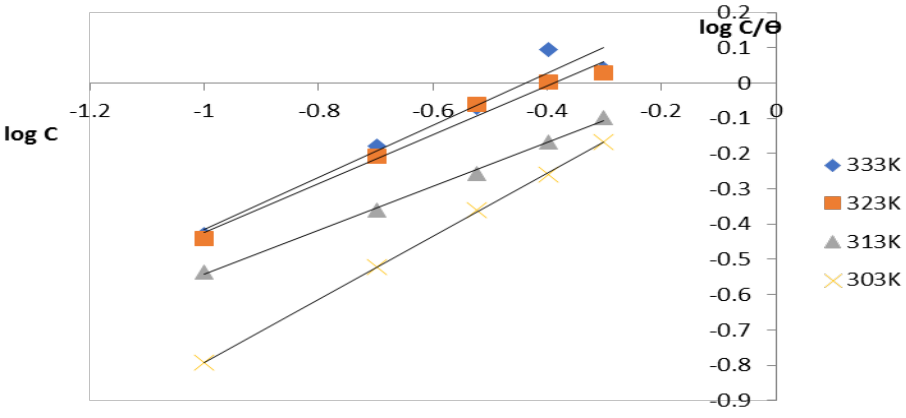
Table 6. Thermodynamic parameters for the enthalpy and entropy of activation of various concentrations of the inhibitor on the aluminum surface in 0.1 M HCl

		Al	
		ΔH_a (kJ/mol)	**ΔS_a (kJ/mol)**
	Blank	42.32	-0.152
	0.5g/l	63.61	-0.093
L	0.4g/l	67.32	-0.081
	0.3g/l	65.08	-0.087
	0.2g/l	63.03	-0.093
	0.1g/l	59.97	-0.102
	0.5g/l	62.00	-0.097
	0.4g/l	62.65	-0.095
CuL	0.3g/l	61.39	-0.099
	0.2g/l	60.81	-0.100
	0.1g/l	58.11	-0.108
	0.5g/l	67.15	-0.081

	0.4g/l	66.36	-0.083
FL	0.3g/l	66.44	-0.083
	0.2g/l	66.46	-0.082
	0.1g/l	65.52	-0.065

Table 7. Thermodynamic parameters for the enthalpy and entropy of activation of various concentrations of the inhibitor on the aluminium surface in 0.05 M H_2SO_4

		Al	
		ΔH_a (kJ/mol)	**ΔS_a (kJ/mol)**
	Blank	46.81	-0.168
L	0.5g/l	53.02	-0.150
	0.4g/l	51.39	-0.155
	0.3g/l	50.01	-0.159
	0.2g/l	56.58	-0.139
	0.1g/l	49.27	-0.162
CuL	0.5g/l	59.66	-0.128
	0.4g/l	57.44	-0.136
	0.3g/l	55.37	-0.142
	0.2g/l	67.53	-0.104
	0.1g/l	55.68	-0.142
FL	0.5g/l	49.71	-0.160
	0.4g/l	46.81	-0.168
	0.3g/l	40.44	-0.190
	0.2g/l	48.38	-0.164
	0.1g/l	43.46	-0.179



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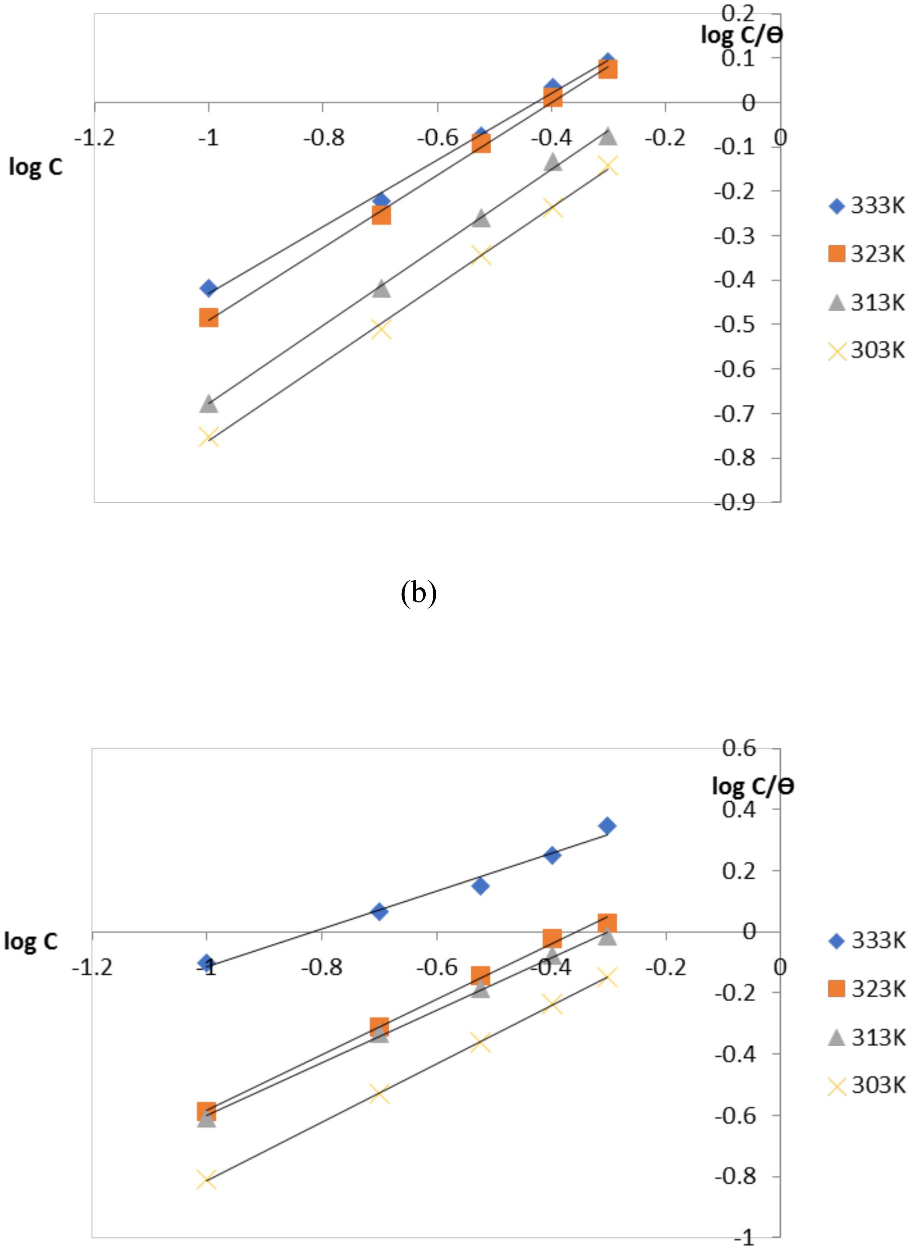

(b)

(c)

Fig. 4 Langmuir isotherm for the adsorption of (a) L (b) CuL and (c) FL on aluminum surface in 0.1 M HCl solution at various temperatures respectively

4.0 Conclusions

The following conclusions are derived from the investigation:

(i) It has been established in this study that the Schiff bases F, CuL and FL respectively exerted an inhibiting effect on the acid corrosion of aluminum.

(ii) It has been established in this study that the rate of corrosion decreases with an increase in concentrations of inhibitors, equally the corrosion rate increased with temperature in the studied media.

(iii) The Schiff base maintained remarkable corrosion inhibition efficiency even with prolonged exposure time.

(iv) The adsorption of the extracts on the aluminum surface in 0.1 M HCl and 0.05 M H_2SO_4 solutions respectively obeys Langmuir and Freundlich isotherm.

(v) The presence of this Schiff base in the acid media increases the activation energy of the corrosion process which indicates physical adsorption.

5.0 Acknowledgment

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