Inhibition of Corrosion of Mild Steel in Hydrochloric Acid Solution by two Schiff Bases Derived from Benheric and Linoleic Acids

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Abstract The inhibition efficiencies of the two Schiff bases 2[2-diethylamino) ethyl methyl amino)-4-methy1-5-3 (3-methyl sulfanyl propy1 amino) methyldiene cyclohexdien-1-one (DEMS) and [1-(azepan-1-yl)2-2-[4-(2-tert-butyl sulfanyl ethyl piperazin-1-yl] ethanone (ATSP), synthesized from linoleic and benheric acids on the corrosion of mild steel in 1 M HCl solutions were investigated using gravimetric and potentiodynamic polarization techniques. The results obtained from both methods indicated that inhibition efficiencies increased with increase in concentration of the Schiff bases and decreased as the temperature increased. Potentiometric polarization data indicated decrease in polarization resistance (and a corresponding decrease in corrosion current) with concentration, which indicated a progressive retardation of corrosion strength with increasing concentration. Displacement potential from the reference blank and equality of extension of cathodic and anodic polarization curves confirmed that DEMS and ATSP are mixed type inhibitor. Mechanism of adsorption of the inhibitors was confirmed to be physiosorption based on calculated values of activation energy and the changes in free energy of adsorption. The adsorption of the inhibitors is spontaneous, exothermic and followed the Langmuir adsorption model.

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List of symbols

CR	Corrosion rate
%I	Inhibition efficiency
ΔW	Weight loss
i_{corr}	Corrosion currents in the absence of the inhibitor
i_{Corr}^0	Corrosion current in the presence of inhibitor
R_{ct}	Charge transfer uninhibited system
$R_{ct(inh)}$	Charge transfer for inhibited system
E_a	Activation energy
R	Gas constant
T	Temperature
ΔS_{ads}^*	Standard entropy change
ΔH_{ads}^*	Standard enthalpy change
ΔG_{ads}^*	Standard free energy change
θ	Surface coverage
C	Concentration of the inhibitor
K_{ads}	Equilibrium constant of adsorption

1.0   Introduction

Metals are valuable materials in most industrial fabrications but exposure to aggressive medium often leads to corrosion, which define an electrochemical degradation process that tends to return metals to their natural state (Ameh & Eddy, 2018). Corrosion is a serious environmental problem in metallurgical, fertilizers, oil and gas and other industries because they are processes within this industries (such as etching, acid cleaning, fluid transportation, etc) that involves contact between a metal and aggressive medium (which could be salt, acid, base, some gases, etc)(Musa *et al., 2010*). In

view of this, most industries are suffering huge lost due to corrosion (Librini *et al., 2010*). Hence measures that can contribute to reduce the rate of corrosion of metals are significant requirement for the success of such industries. Some of the options that are currently implemented in corrosion control are painting, oiling, cathodic protection, anodic protection, the use of corrosion inhibitors, etc (Domer *et al*., 2011). Several researchers have confirmed that the use of corrosion inhibitors is one of the best preferences. Consequently, several researches on choice of corrosion inhibition have been directed to some factors including inhibition efficiency, mechanisms of action, environmental requirements, cost, ease of availability, thermodynamic/adsorption as well as kinetic behaviour and other factors (Eddy and Ita, 2011a).

The corrosion industries are flexible in accepting new inhibitors that meets most of these requirements. Structurally, a good corrosion inhibitor is proposed when it possess aromatic or long carbon chain, hetero atoms (which can act as a centre for adsorption) and π-electrons.. In line with these requirements, choice of corrosion inhibitors has progresses from inorganic inhibitors to organic base inhibitors because of toxicity of most inorganic corrosion inhibitors. Green corrosion are those corrosion inhibitors that meet the structural and environmental requirements for corrosion inhibition (Eddy *et al*., 2011). Several green corrosion inhibitors that have been tested and implemented are extracts of living things and some natural polymers (Ameh and Eddy, 2014) that are less toxic. According to Eddy and Ita (2011b), the major problem in the use of plant or animal extracts for corrosion inhibition is that it is difficult to study the mechanism of inhibition because the inhibition process is induced by synergistic combination of several compounds that are present in the extract, some of which, their chemical structures have not been properly elucidated . Detail study of mechanism of any chemical; reaction requires knowledge of the chemical structure. In view of this and other developments, several other synthesized compounds such as drugs (Eddy and Ebenso, 2010) have been tested and confirmed as good corrosion inhibitors (Eddy and Awe, 2018).

Schiff bases are compounds whose general chemical structure is $R_2CH=NR$ and can be defined as

macrocyclic or macroacylic ligands that contain heteroatoms (such as nitrogen and oxygen) and are polydented in coordination ability (Burn and Royers, 2003). The role of Schiff base on the inhibition of the corrosion of metal is attributed to several properties including polydentated nature (that enable effective adsorption between the Schiff base and the metal surface), possession of hetero atoms, which can donate electrons and become adsorbed and the less toxicity of most of them (Burn and Royers, 2003; Ju *et a.,* 2008' Lashgari *et al*., 2010). Several studies on the effectiveness of Schiff base for the inhibition of the corrosion of metals have been documented. For example, Aouniti *et al*. (2015) found that Schiff's base derived from 2-acetyl thiophene was a very good corrosion inhibitor for mild steel in acidic medium and obtain inhibition efficiency that were above 80 %. Binsi *et al*. (2020) deduced similar finding in using Schiff bases derived from 2-acetyl pyridine for the inhibition of the corrosion of mild steel in hydrochloric acid. . Study conducted by Ahmad *et al*. (2010) gave good hope on the use of several Schiff bases for the inhibition of the corrosion of mild steel in hydrochloric acid solution. In all these and other studies, literature is scanty on the use of Schiff bases derived from benheric and linoleic acid for the inhibition of the corrosion of mild steel in acidic medium. Therefore, the present study is aimed at synthesizing benheric and linoleic acid derived Schiff base for the inhibition of the corrosion of mild steel in acidic medium.

2.0   Materials and Methods

The mild steel used for the study had the following composition (% weight): Mn (0.65), P (0.36), C (0.16), Si (0.04) and Fe (98.78). The sheet was mechanically press-cut into coupons of dimension, 3 x 3 cm. Each coupon was cleaned using different grades of emery papers until a mirror appearance was observed. The coupons were degreased by successive washings with ethanol, de-ionized water and acetone and air-dried before preservation in a desiccator. A solution of 1 M HCl was used as the test solution. Different concentrations of the inhibitors (0.0005, 0.001, 0.002, 0.003, and 0.006 mol/dm^3) were employed for the inhibition studies. These were prepared by dissolving the required amount of each Schiff base in 100 ml of 1 M HCl and at room temperature.

2.1 Preparation of fatty acid hydrazine

Fatty acid hydrazine was prepared using the method reported by Toliwal et al., (2010). 0.1 M fatty acid ester was measured into 150 ml ethanol in a refluxing flask followed by addition of 0.2 M of hydrazine hydrate (95%). The reaction mixture was refluxed for 4 hours and cooled. The solid part was collected, washed and recrystallized from ethanol. Recrystallize

2.2 Preparation of thiosemicarbazide

A solution of 0.02 M fatty acid hydrazide in 150 ml methanol was prepared according to the method of Toliwal et al., (2010). A solution of 0.03 M potassium thiocynate and 3 ml of concentrated HCl were mixed with constant stirring in a round bottom flask. The mixture was evaporated to dryness on a steam bath. This was reconstituted in 150 ml ethanol and heated for an additional one hour. The resulting solid was mixed with 10 ml of water and then recrystallized from 20 ml ethanol.

2.3 Preparation of Schiff base

The Schiff bases were prepared according to the method reported by Yaseen et al. (2003). 0.02 M thiosemicarbazide was added to a solution of 0.02 M acetaldehyde in absolute alcohol and was refluxed for 7 h on a water bath. The mixture was cooled and the solid was filtered and recrystallized from alcohol. The Schiff base was characterized using FTIR spectrophotometry.

2.4 Weight loss measurement

Weight loss measurements were carried out according to the method reported elsewhere (Eddy and Awe, 2018). Pre-weighed metal coupon was immersed in the test solution and was allowed to stand under thermostated condition (maintained by a water bath). After every three hours, each coupon was withdrawn from the test solution, washed with distilled water containing 50 % of zinc dust. The washed coupon was rinsed in acetone and allowed to dry in the air before it was re-weighed. The experiment was repeated for different systems at different concentrations and different temperatures. From the results obtained from weight loss results, corrosion rate of the metal (CR), surface coverage (θ) and inhibition efficiency (I %) were calculated using the relations express in the equations (Eddy and Awe, 2018).

$$CR = \frac{\Delta W}{At} = \frac{w_1 - w_1}{At} \qquad (1)$$

$$\theta = \left(\frac{CR_0 - CR_1}{CR_0}\right) \qquad (2)$$

$$I\% = \left(\frac{CR_0 - CR_1}{CR_0}\right) x100 \qquad (3)$$

where, CR is the corrosion rate, CR_0 and CR_1 are corrosion rates in the absence and presence of inhibitor, respectively. w_1 and w_2 are weight loss of the metal before and after immersion respectively, A is the area of the specimen and t is the period of immersion.

2.5 Potentiodynamic polarization measurements

The potentiodynamic current-potential curves were recorded by changing the electrode potential (E_{corr}) automatically with a scan rate 0.33 mV/s from a low potential of −800 to −300 mV (SCE). Before each run, the working electrode was immersed in the test solution for 30 min to reach steady state. The corrosion rate of the inhibitor was calculated through corrosion current density l_{corr}, which was obtained by extrapolating the linear Tafel segments of the anodic and cathodic curves. The inhibition efficiency (l%) was calculated using equation 4, (Ameh and Eddy, 2016).

$$\%I = \frac{i_{Corr}^0 - i_{Corr}}{i_{Corr}^0} \times \frac{100}{1} \qquad (4)$$

where i_{Corr} and i_{Corr}^0 are the corrosion current for uninhibited and inhibited systems respectively.

3.0 Results and Discussion

Results obtained from gravimetric and potentiodynamic polarization studies are discussed in this section

3.1 Gravimetric study

Figs. 1 and 2 are plots indicating the patterns of variation of weight loss with time for the corrosion of mild steel in solution of HCl containing various concentrations of DEMS and ATSP at 303 K respectively. The two plots reveal some common features. First, weight loss of mild steel increase with increase in the period of contact but decreased with increasing concentration of the respective inhibitor. The observed trend indicates that the corrosion of mild steel increase with time but it is retarded by various concentration of the DEMS and ATSP. Therefore, DEMS and ATSP are inhibitors for the corrosion of mild steel and since their inhibition efficiency increase with concentration, they are adsorption inhibitors.
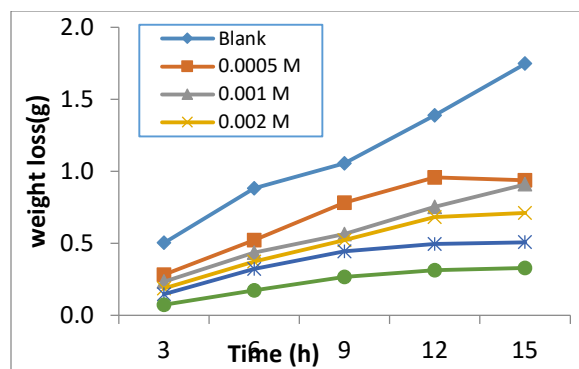
Fig.1: Variation of weight loss of mild steel with time for the corrosion of mild steel in solution of HCl containing various concentrations of DEMS at 303 K
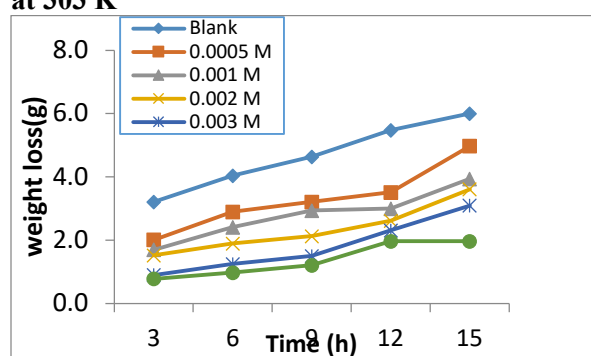

Fig. 2: Variation of weight loss (g) of mild steel with time for the corrosion of mild steel in solution of HCl containing various concentrations ATSP at 303 K
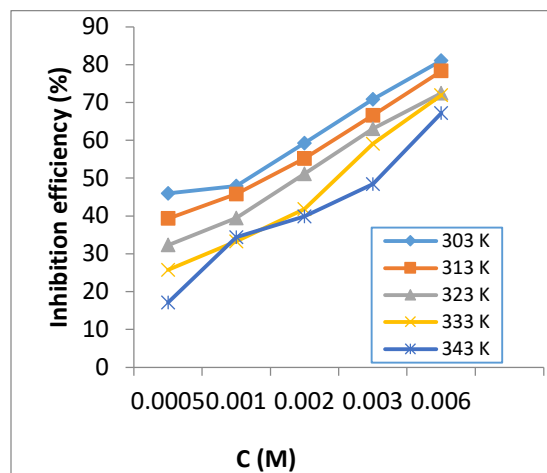

Fig.3: Variation of inhibition efficiency with concentration at various temperatures
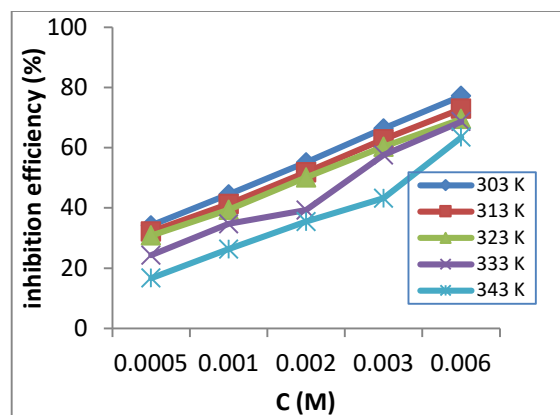

Fig. 4: Variation of inhibition efficiency with concentration at various temperatures.

Calculated inhibition efficiencies of DEMS and ATSP were strongly dependent on concentration and temperature. As shown in Figs. 3 and 4, the inhibition efficiency increases with concentration but decreased with increase in temperature.

This suggests that the mechanism of adsorption of the two inhibitors is physiosorption, which involves charge transfer from charged inhibitor to charged metal surface as opposed to chemisorption that involves the transfer of electron from the inhibitor to the metal surface.

3.2   *Potentiodynamic polarization measurement*

Figs. 5 and 6 represent potentiodynamic polarization plots for the corrosion of mild steel in 1 M HCl in the absence and presence of different concentrations of DEMS and ATSP, respectively.
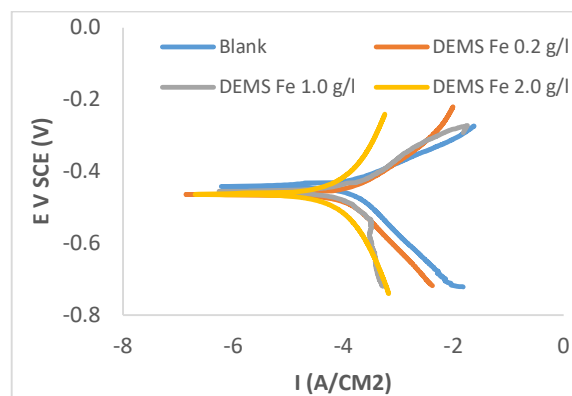

Fig. 5 : Potentiodynamic polarization plots for the corrosion of steel in 1 M HCl containing various ~~with~~ different concentrations of DEMS at 303 K
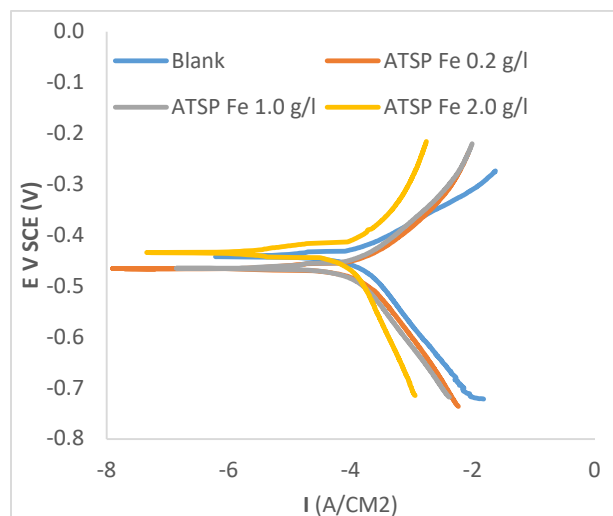
Fig. 6: Polarization plots for the corrosion of mild steel in 1 M HCl containing various concentrations of ATSP at 303 K

The polarisation curves show cathodic and anodic polarization with well-defined regions. The pattern of the polarization curve (Tafel plot) remained the same in the absence and presence of the inhibitors but the curves shifted towards lower current density in the presence of the inhibitors. This shows that the inhibitor molecules retarded the corrosion process and the inhibition increases with increasing concentration without changing the mechanism of the corrosion process since the plots are almost parallel to each other. It is also evident from the plots that the inhibitor molecules affected both cathodic and anodic reactions almost equally, leading to a small displacement in E. This suggest that the inhibitors are mixed type inhibitors (Eddy et al., 2015).In Table 1, polarization parameters for the corrosion and inhibition of mild steel corrosion by DEMS and ATSP are presented.

Table 1. Potentiodynamic polarisation data for the corrosion of mild steel electrode in 1 M HCl containing various concentrations of DEMS and ATSP at 303 K

Inhibitors	C (M)	E_{corr} (mV/SCE)	I_{corr} (μAcm^{-2})	β_c (mVdec^{-1})	β_a (mVdec^{-1})	R_p	I%
	Blank	-442	570	331	615	0.164	
	0.2	-465	319	398	628	0.332	50.57
DEMS	1.0	-455	186	318	641	0.496	66.96
	2.0	-465	76	365	670	1.35	87.86
	0.2	-466	375	343	646	0.259	36.81
ATSP	1.0	-465	202	321	630	0.457	64.14
	2.0	-443	158	335	643	0.605	72.92

Corrosion potential (E_{Corr}) and corrosion current (I_{corr}) were obtained by extrapolation of the plots at the point of intersection to the respective axis. Tafel constants (β_a and β_b)were obtained as the slope of the respective cathodic and anodic arms of the plot s The results obtained, indicates that the corrosion current decreases with concentration of the inhibitor while the polarization resistance increases in the same direction. This indicates an increase retardation to corrosion of mild steel (coupled with reduce dissolution rate, since dissolution rate increases with increase in corrosion rate) with increasing concentration, hence increase in inhibition efficiency with increasing concentration.

respectively. However, the polarization resistance was calculated using the following equation (Eddy *et al.*, 2015)

$$R_p = \frac{\beta_a \beta_b}{2.303(\beta_a + \beta_b)} \times \frac{1}{R_p} \qquad (5)$$

The observed displacement in E_{corr} was less than ± 85 mV/SCE with respect to corrosion potentials of the blank and for various concentrations of the inhibitors. Hence DEMS and ATSP are mixed type inhibitors (Singh, 2012).

3.3   Kinetic study

The minimum energy needed for the corrosion reaction in the presence and absence of the inhibitors (i.e the activation energy) were calculated using the Arrhenius relationship

between corrosion rate and temperature, which can be written according to equation 6 (Eddy *et al.*, 2014)

$$CR = A exp\left(\frac{-E_a}{RT}\right) \qquad (6)$$

where A is the Arrhenius or pre-exponential constant, R is the gas constant and E_a is the activation energy. Arrhenius plots for the corrosion and corrosion inhibition reactions of mild steel in 1 M HCl are not shown but calculated activation energy are recorded in Table 2. The activation energies for the inhibited systems are higher than that of the blank and tend to increase with increase in concentration. This indicate that the corrosion of mild steel in 1 M HCl is retarded by DEMS and ATSP and the ease of inhibition increases with concentration. Also, the observed activation energies were less than the threshold value that is required to confirm the mechanism of physical adsorption. Therefore, the adsorption of DEMS and ATSP on mild steel surface supports physisorption mechanism (Li and Fu, 2011)

Table 2: Kinetic and thermodynamic parameters for the corrosion of mild steel in 1 M HCl containing various concentrations of DEMS and ATSP

C (M)	E_a (J/mol)	ΔH_t (kJ/mol)	ΔS_t (J/mol/K)
Blank	3697.34	30.79	-98.95
0.0005 of DEMS	4849.1	39.54	-75.11
0.001 of DEMS	4430.42	36.36	-85.99
0.002 of DEMS	4813.83	39.27	-78.39
0.003 of DEMS	5153.66	41.86	-72.73
0.006 of DEMS	5216.14	42.33	-74.62
0.0005 of ATSP	4849.1	39.54	-75.11
0.001 of ATSP	4430.42	36.36	-85.99
0.002 of ATSP	4813.83	39.27	-78.39
0.003 of ATSP	5153.66	41.86	-72.73
0.006 of ATSP	5216.14	42.33	-74.62

3.4   Thermodynamic/adsorption considerations

Basic thermodynamics parameters that can furnish information on corrosion and corrosion inhibition are standard enthalpy (ΔH_{ads}^*), entropy (ΔS_{ads}^*) and free energy (ΔG_{ads}^*) changes of adsorption. Enthalpy and entropy changes in the inhibitors 'adsorption process were estimated through the slope and intercept of the Transition state plots

which is according to equations 6 and 7 (Li and Deng, 2012)

$$CR = \frac{RT}{Nh} exp\left(\frac{\Delta S_{ads}^*}{R}\right) exp\left(\frac{-\Delta H_{ads}^*}{RT}\right) \qquad (6)$$

$$log\left(\frac{CR}{T}\right) = \frac{R}{Nh} + \frac{\Delta S_{ads}^*}{R} - \frac{\Delta H_{ads}^*}{RT} \qquad (7)$$

where N is the Avogadro's number, h is the Planck constant while other parameters are as defined before. Application of equation 7 yielded straight-line plots when $log\left(\frac{CR}{T}\right)$ were plotted against 1/T as shown in Fig. 5 and 6. Slopes and intercepts of the plots were used to calculate standard changes in enthalpy (ΔH_{ads}^*) and entropy (ΔS_{ads}^*) of adsorption and the results obtained were also recorded in Table 2, Hence, the adsorption of the inhibitors is endothermic and ooccured with increasing degree of orderliness because enthalpy (ΔH_{ads}^*), entropy (ΔS_{ads}^*) changes are positive and negative respectively,

Data obtained from weight loss measurements were used to test for the best fitted adsorption isotherm and the isotherm that described the adsorption of DEMS and ATSP best was the Langmuir adsorption isotherm, which align with the expression of equation 8 (Okafor, 2007).
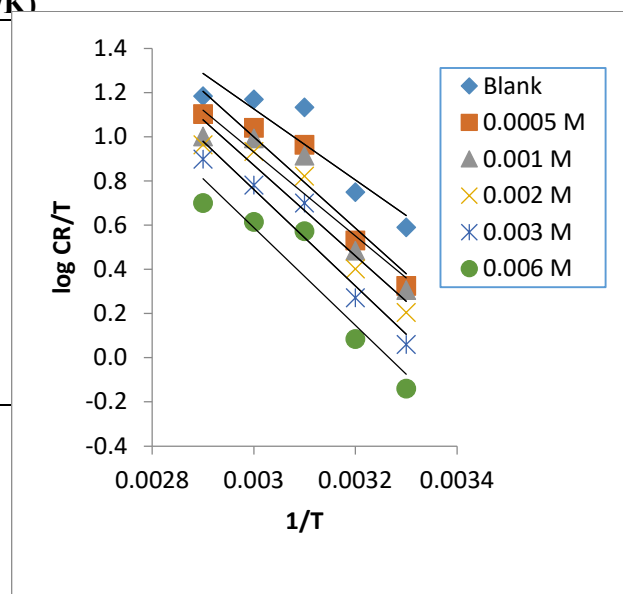
$$\frac{C}{\theta} = k_{ads} - logC \qquad (8)$$

Fig. 5. Variation of log (CR/T) with 1/T for the corrosion of mild steel in 1M HCl containing various concentrations of DEMS
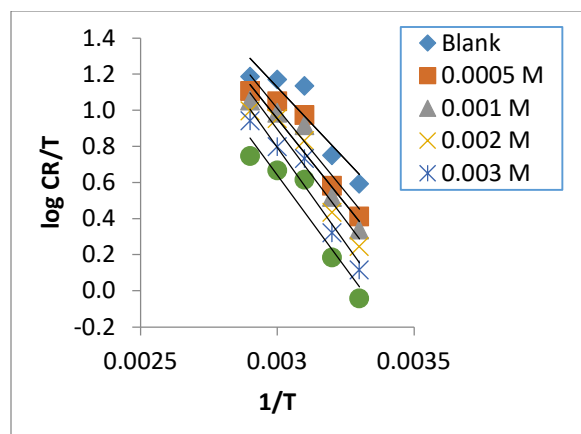
Fig. 6. Plots of log (CR/T) versus 1/T for the corrosion of mild steel in 1M HCl containing various concentrations of ATSP

Fig.8. Langmuir isotherm for the adsorption of ATSP on the surface of mild steel.

where k_{ads} and θ are the Langmuir equilibrium constant of adsorption and surface coverage respectively. Fig. 7 and 8 are the Langmuir plots for the adsorption of DEMS and ATSP on mild steel surface. Adsorption parameters calculated from the plots are recorded in Table 3. The high degree of age agreement between the adsorption data with the Langmuir model is confirmed by the excellent degree of fitness as expressed by R^2 (Table 3). Values of k_{ads} were found to decrease with increasing temperature, indicating that the stability of the adsorption layer decreases with increase in temperature, a phenomenon, which also confirms the existent of physisorption mechanism.
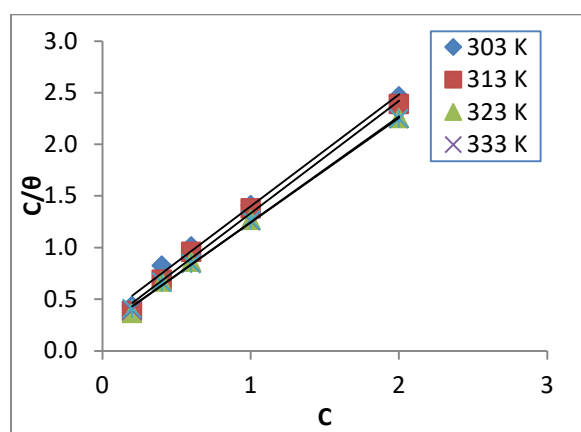
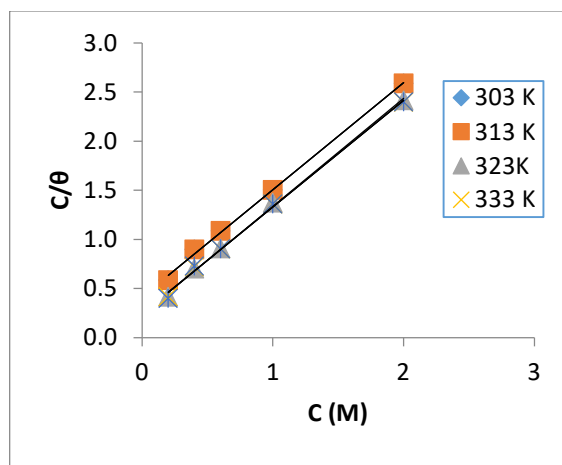**Table 3: Langmuir parameters for the adsorption of DEMS and ATSP on mild steel surface**

System	K_{ads}	G_{ads}	R^2
DEMS at 303 K	3.126	-12.989	0.9925
DEMS at 313 K	2.611	-12.5355	0.994
DEMS at 323 K	2.1404	-12.0349	0.9944
DEMS at 333 K	1.3602	-10.8928	0.9702
DEMS at 343 K	1.3841	-10.9318	0.9992
ATSP at 303 K	2.3918	-12.3146	0.9979
ATSP at 313 K	2.2168	-12.1232	0.9973
ATSP at 323 K	2.1533	-12.05	0.9972
ATSP at 333 K	1.5883	-11.2834	0.9974
ATSP at 343 K	0.936	-9.9517	0.9824

Fig. 7. Langmuir isotherm for the adsorption of DEMS on the surface of mild steel.

The Langmuir adsorption equilibrium constanr, , k_{ads} is related to free energy of adsorption according to the following equation (Mohmoh-Yahaya et al., 2012),

$$\Delta G_{ads}^* = -2.303RTlog(55.5k_{ads}) \qquad (9)$$

where 55.5 is the molar concentration of water in the electrolyte. Values of ΔG^*_{ads} upto -20 kJ/mol and below are spontaneous and consistent with the mechanism of physiosorption while those above -40 kJ/mol and above supports chemisorption. Therefore, the adsorption of DEMS and ATSP followed physiosorption mechanism (Jacob and Parameswaran, 2010).

4.0   Conclusion
The results and findings deduced from this study indicate that DEMS and ATSP are good adsorption corrosion inhibitors for the corrosion of mild steel in solution of HCl. The inhibitor's efficiencies increase with concentration and decreased with temperature while the mechanism of adsorption is physical adsorption that obeys the Langmuir adsorption model. The adsorption of the inhibitors on the surface of the metal is exothermic, spontaneous and occurred with increasing degree of orderliness.

5.0   References

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