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Adsorption and Inhibitive Properties of *Delonix regia* leaves for the Corrosion of Mild Steel in H_2SO_4

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Abstract: **The inhibitive and adsorption properties of ethanol extracts of *Delonix regia* leaves were studied using gravimetric method of monitoring corrosion. Phytochemical analysis was carried out using fat free samples of the respective extracts. The results obtained indicated that ethanol extract of *Delonix regia* leaves is a good adsorption inhibitor for the corrosion of mild steel in H_2SO_4 solutions. The phytochemical screening proved that the leaf extracts are rich in alkaloids, flavonoids and tannins.The adsorption of the inhibitor on mild steel surface is spontaneous and supports the Langmuir adsorption models. From the values of free energy of adsorption, a physical adsorption mechanism has been proposed for the adsorption of the inhibitor on mild steel surface.**

Keywords: **Adsorption, Corrosion, green inhibitor, *Delonix regia*, Phytochemical.**

1. INTRODUCTION

Corrosion of metals is of fundamental academic and industrial concern that has received considerable attention over the years. It remains a global scientific problem as it affects the metallurgical, chemical, food processing and oil industries. Mild steel is widely use in the manufacturing of installations for the petroleum, fertilizers and other industries. In view of the viability of mild steel, its high cost of production and installation, several steps have been adopted to prolong the lifespan of the metal in industries. However, the most practical and preferred method is the use of inhibitors [1-3]. Most corrosion inhibitors protect the corrosion of metals when they are adsorbed on the surface of the metal [4]. There is increasing concern about the toxicity of most corrosion inhibitors in industries because the toxic effect do not only affect living organism but also poison the earth [5]. This has prompted a search for green corrosion inhibitors. According to Eddy and Ebenso [6], green corrosion inhibitors are biodegradable and do not contain heavy metals or other toxic compounds. Ethanol extracts of some plants have been found to be good corrosion inhibitors. [7 – 10]However, literature is scanty on the use of ethanol extracts of leaves of *Delonix regia* as corrosion inhibitors for mild steel. Therefore the objective of the present study is to investigate the inhibitive and adsorption properties of ethanol extracts of *Delonix regia* leaves for the corrosion of mild steel in H_2SO_4.

2. MATERIALS AND METHODS

2.1 Materials

Materials used for the study were mild steel sheet of composition (wt %, as determined by quantiometric method) Fe% (98.048) C% (0.348), Si % (0.221), Mn% (0.745), P% (0.032), S% (0.035), Cr% (0.033), Mo% (0.017), Ni% (0.128), Cu% (0.316), Co% (0.013), Al% (0.001), V% (0.001), Sn% (0.027), As% (0.014), Ca% (0.001), Sb% (0.017) and Te% (0.003). The sheet was mechanically pressed cut into different coupons, each of dimension, 5 x 4 x 0.11cm. Each coupon was degreased by washing with ethanol, cleaned with acetone and allowed to dry in the air before preservation in a desiccator. All reagents used for the study were analar grade and double distilled water was used for their preparation. Concentration of H_2SO_4 used for weight loss and electrochemical studies was 0.1 M.

2.2 Methods

2.2.1 Preparation of Plant Extract

Leaves of *Delonix regia* were obtained from the premises of Gray's International College in Igabi LGA, Kaduna, Kaduna State. The leaves were washed with water; shade dried, grounded and soaked in a solution of ethanol for 48 hours. After 48 hours, the sample was cooled and filtered. The filtrate was further subjected to evaporation at 338K (65ºC) in other to ensure the sample free of ethanol. The plant extract obtained was used in preparing different concentrations of the extract by dissolving 0.1, 0.2, 0.3, 0.4 and 0.5g of the extract in 250ml of 0.1M H_2SO_4 for the gravimetric analysis.

2.2.2 Phytochemical Analysis of the Plant Extract

Phytochemical screening is the application of simple chemical tests to detect the presence of accumulated natural products such as carbohydrate, cardiac glycosides, flavonoids, tannins etc in the ethanol extract of *Delonix reiga* leaves. These are referred to as secondary metabolites and they are responsible for the therapeutic properties of the plants (Yawas, 2005). This test was carried out in the Sheda Science and Technology Complex, FCT, Abuja.

2.2.2.1Test for Carbohydrates Using Molisch's Test

A few drops of molisch reagent was added to 5 g of extract in a test tube and a small quantity of concentrated sulphuric acid was allowed to run down the side of the test tube. A lower purple to violet colour at the interface indicated the presence of carbohydrate.

2.2.2.2 Test for Cardiac Glycosides Using Keller-Killiani Test

Extract was mixed with glacia acetic acid containing traces of ferric chloride. The test tube was held at angle of 45 degree, 1ml of concentrated sulphuric acid was added down the side. A purple ring colour formed at interface indicated the presence of cardiac glycosides.

2.2.2.3 Test ForAnthraquinone Glycosides Using Borntrager's Test

Small portion of the extract was shaken with 10ml of benzene and filtered. 5ml of 10% of ammonia solution was added to the filtrate and stirred. No pink- red or violet colour was observed which indicates the absence of anthraquinone glycosides.

2.2.2.4 Test for Saponins Using Frothing Test

Small quantity of the extract was added to 10ml of distilled water in a test tube. This was shaken vigorously for 30 seconds and was allowed to stand for 10 minutes. No frothing observed indicates the absence of Saponins

2.2.2.5 Test of Flavonoids

Few drops of aqueous NaOH were added to 5g of extract. A yellow colour observed indicated the presence of flavonoid.

2.2.2.6 Test for Tannin

About 0.5ml of extract was stirred with 10ml of distilled water, then filtered. Few drops of ferric chloride reagent added to the filtrate. A blue-black precipitate formed indicated the presence of tannins.

2.2.2.7 Test for Akaloids

2 drops of dilute hydrochloric acid was added to 3g of the extract in a test tube, then followed by dragendorff's reagent. A red precipitate was formed which indicate the presence of alkaloids.

2.2.2.8 Test for Steroid and Triterpenes Using Liebermann-Burchards Test

Equal volume of acetic anhydride was added to the extract. 1ml of concentrated sulphuric acid was added down side the tube. Red colour was observed and no blue-green or blue was observed. Red colour indicates the presence of triterpenes and the absence of blue or blue-green indicates the absence of steroid.

2.3 Corrosion Inhibition Studies

2.3.1 Weight loss method (Gravimetric analysis)

A previously weighed metal (mild sheet) was completely immersed in 250 ml of the test solutions containing 0.1M H_2SO_4 and varied masses (0.1, 0.2, 0.3, 0.4, 0.5g) of the *Delonix regia* leaves extract (DRLE) in open beakers. The beakers were inserted into a water bath maintained at a temperature of 30 °C. Similar experiments were repeated at 40, 50 and 60°C and for test solution without DRLE as inhibitor. In each case, the weight of the sample before immersion was measured using Scaltec high precision balance (Model SPB31). After every 24 hours, each sample was removed from the test solution, washed in a solution of NaOH containing zinc dust and dried in acetone before re-weighing. The difference in weight for a period of 168 hours (7 days) was taken as total weight loss. The inhibition efficiency (% I) for each inhibitor was calculated using equation 1.

$$\%I = \left(1 - \frac{W_1}{W_2}\right) \times 100 \qquad (1)$$

where W_1 and W_2 are the weight losses (g/dm^3) for mild steel in the presence and absence of inhibitor in 0.1M H_2SO_4 solution respectively. The degree of surface coverage θ is given by the equation 2:

$$\theta = (1 - \frac{W_1}{W_2}) \qquad (2)$$

The corrosion rates for mild steel corrosion in different concentrations of the acid was determined for 168 hours immersion period from weight loss using equation 3:

$$Corrosion\ rate(mpy) = \frac{534W}{DAT} \qquad (3)$$

where W = weight loss (mg); D = density of specimen (g/cm^3), A = area of specimen (square inches) and T = period of immersion (hour).

3. RESULTS AND DISCUSSIONS

3.1 Phytochemical Screening

Table 4.1 presents the result obtained from the phytochemical screening of the *Delonix regia* leaves extract (DRLE). It can be seen from the results that the plant extract is rich in carbohydrates, alkaloids, flavonoids and tannins. These compounds contain oxygen, nitrogen and sulphur atoms which possess lone pair of electrons that may facilitate the formation of dative bonds acting as center for adsorption, thus creating a barrier between the steel surface and the corrosive media. Umoren et al., (2008) have reported that saponins, tannins and alkaloids are active constituents of most green inhibitors. Hence, the inhibition efficiency of the *Delonix regia* leaves extract observed may be due to the presence of some or all of the listed phytochemical constituents in Table 1 below.

Table 1: Phytochemical Screening of *Delonix regia* Leaves Extracts

S/N	Chemical constituent	Observation / inference
1	Tannins	+
2	Flavonoid	+
3	Alkaloid	+
4	Saponins	-
5	Carbohydrates	+
6	Anthraquinone	-
7	Cardiac glycoside	+
8	Steroid	-
9	Triterpene	+

3.2 Corrosion studies

3.2.1 Weight loss study (Gravimetric analysis)

Fig. 1 – 4 shows the variation of weight loss with time for the corrosion of mild steel in 0.1 M H_2SO_4 containing various concentrations of the plant extract at 303, 313, 323 and 333 K. A closer look at the figures reveal that the introduction of the DRLE resulted in the reduction of the rate of weight loss of the mild steel as compared to the blank, indicating that ethanol extract of *Delonix regia* leaves retarded the rate of corrosion of mild steel in H_2SO_4 and that the extract is an inhibitor for the corrosion of mild steel.

Table 2 and 3 respectively present the corrosion rates and inhibition efficiencies of mild steel in various media at 303 to 333 K. The results obtained reveal that the corrosion rate of mild steel decreases with increase in the concentration of the *Delonix regia* leaves extract (DRLE) while the inhibition efficiency increases with increasing concentration of the extract. The inhibition efficiency was also found to decrease with increase in temperature. This may be attributed to the decrease in the protective nature of the inhibitive film formed on the metal surface (or desorption of the inhibitor molecules from the metal surface) at higher temperatures [5]. This, according to Eddy et al [11], suggests physical adsorption mechanism. Physical (electrostatic) adsorption takes place when inhibition efficiency decreases with increase in temperature whereas chemical adsorption takes place when inhibition efficiency increases with increase in temperature [12]. The inhibitive action of the *Delonix regia* leaves extract is due mainly to the presence of carbohydrates, alkaloids, flavonoids and tannins present in the plant extracts. These compounds contain heteroatoms such as oxygen, nitrogen and aromatic rings with π bonds in their molecules, which serve as centers of adsorption onto the metal surface.

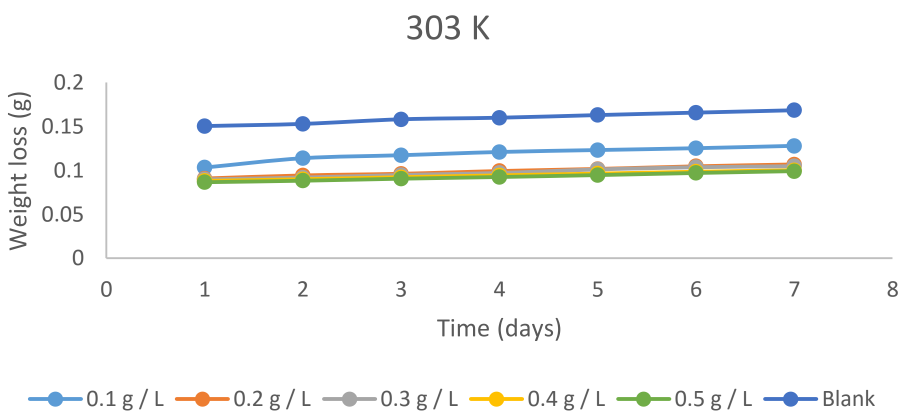

Fig. 1: Variation of weight loss with time for the corrosion of mild steel in 0.1 M H_2SO_4, containing various concentrations of DRLE as an inhibitor at 303 K

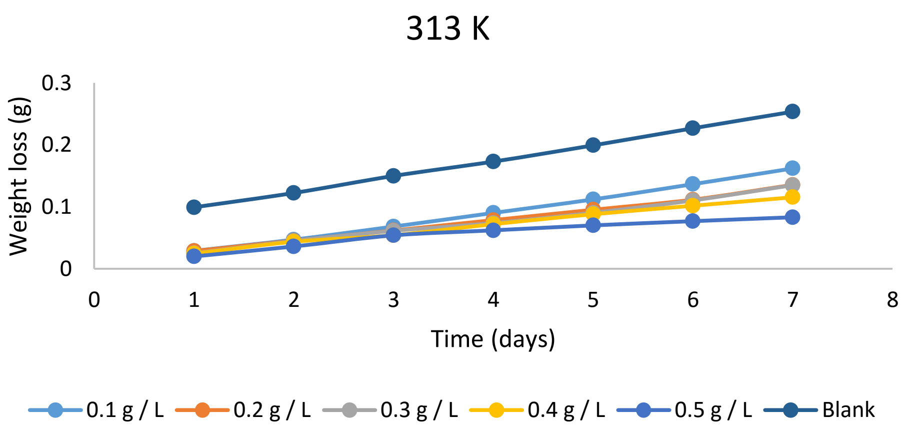

Fig. 2: Variation of weight loss with time for the corrosion of mild steel in 0.1 M H_2SO_4, containing various concentrations of DRLE as an inhibitor at 313 K

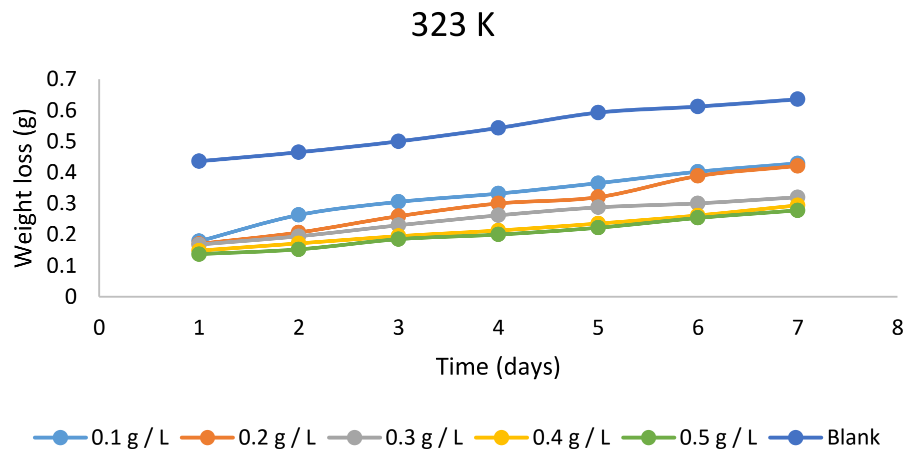

Fig. 3: Variation of weight loss with time for the corrosion of mild steel in 0.1 M H_2SO_4, containing various concentrations of DRLE as an inhibitor at 323 K

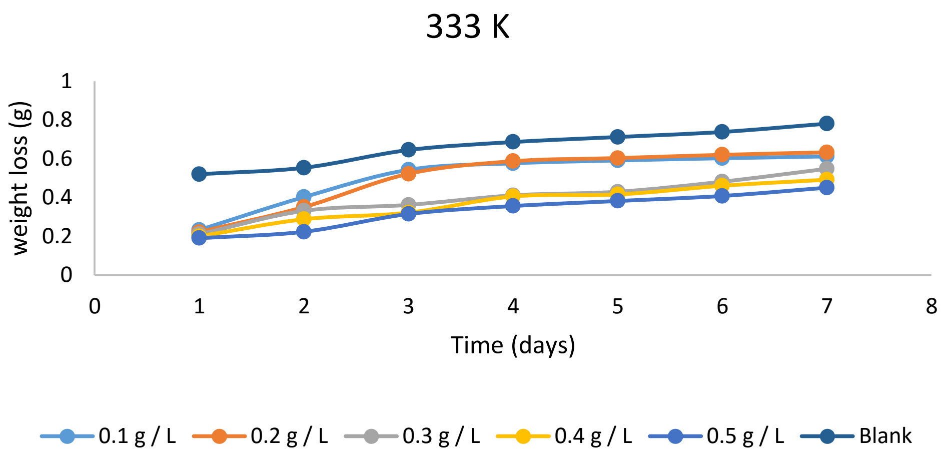

Fig. 4: Variation of weight loss with time for the corrosion of mild steel in 0.1 M H_2SO_4, containing various concentrations of DRLE as an inhibitor at 333 K

Table 2: Corrosion rate (CR x 0.0001) of mild steel in solutions of 0.1M H_2SO_4 containing various concentrations of *Delonix regia* leaves extracts at 303 to 333 K

C (g/L)	303K	313K	323K	333K
Blank	1.65	1.99	2.22	2.44
0.1	0.99	1.33	1.49	1.78
0.2	0.90	1.25	1.39	1.57
0.3	0.80	1.14	1.28	1.50
0.4	0.68	1.03	1.18	1.38
0.5	0.58	0.94	1.12	1.32

Table 3: Inhibition efficiency of plant extracts for the corrosion of mild steel in solutions of 0.1M H_2SO_4 at 303 to 333 K

C (g/L)	303K	313K	323K	333K
0.1	46.93	42.54	26.04	23.01
0.2	50.44	45.84	31.99	35.56
0.3	53.26	50.24	36.01	34.06
0.4	58.95	54.09	42.46	39.26
0.5	62.45	57.55	47.25	45.06

In Table 4, values of degree of surface coverage (θ) calculated from equation 2 are presented. The results show trend similar to those of inhibition efficiencies recorded in Table 3. This is because the degree of surface coverage is directly proportional to the inhibition efficiency.

Table 4: Degree of surface coverage (θ) of DRLE for the corrosion of mild steel in solutions of 0.1M H_2SO_4 at 303 to 333 K

C (g/L)	303K	313K	323K	333K
0.1	0.4693	0.4254	0.2604	0. 2301
0.2	0.5044	0.4584	0.3199	0.3556
0.3	0.5326	0.5024	0.3601	0.3406
0.4	0.5895	0.5409	0.4246	0.3926
0.5	0.6245	0.5755	0.4725	0.4506

3.3 Kinetic Study

In order to study the kinetic of the corrosion of mild steel in 0.1 M H_2SO_4 containing various concentrations of the *Delonix regia* leaves extract (DRLE), data obtained from weight loss measurements were used to fit curves for different orders of reaction. The test indicated that the corrosion of mild steel in the absence and presence of DRLE is first order.

First order kinetic rate law for the corrosion of metal can be written as follows [13 – 14],

$$-\log(\text{weight loss}) = k_1 t/2.303 \quad (4)$$

where k_1 is the first order reaction rate constant and t is the time in day. From equation 4, the plots of –log(weight loss) versus time (Fig 5 - 8)were linear for the corrosion of mild steel in the absence and presence of the plant extract, indicating that the reaction is first order. Values of rate constant and degree of fitness (R^2) of lines on the plots are recorded in Table 5. From the results, it is found that the rate constant decreased with increase in the concentration of plant extract. Also, the R^2 values were very close to unity indicating a high degree of fitness of the plots; hence obedient to equation 4.

For a first order reaction, the rate constant is related to the half-life according to equation 5 [13 – 14].

$$t_{1/2} = 0.693/k_1 \quad (5)$$

Values of $t_{1/2}$ calculated from equation 5 were also recorded in Table 5. These values were found to increase with increasing concentration of the plant extract as shown in Fig. 4.4. Also values of the half-life in the presence of DRLE were higher than the value obtained for the blank indicating that the inhibitor increased the half-life of mild steel in 0.1M H_2SO_4.

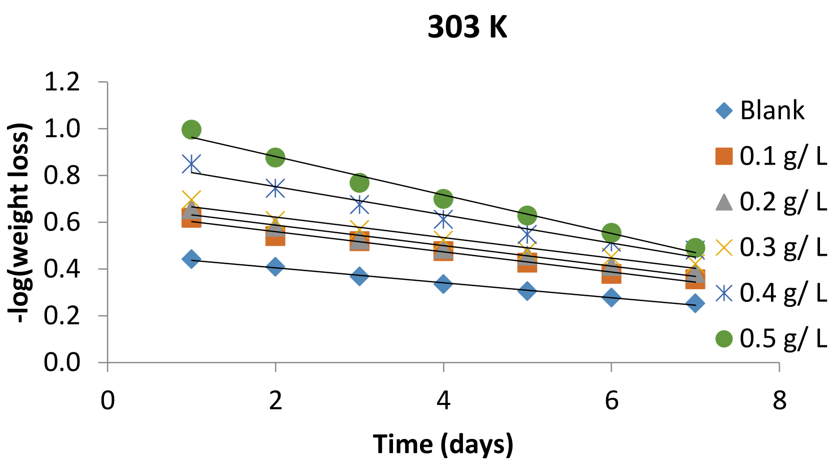

Fig. 5: Variation of –log(weight loss) with time for the corrosion of mild steel in 0.1 M H_2SO_4 containing various concentrations of the inhibitor (DRLE) at 303 K

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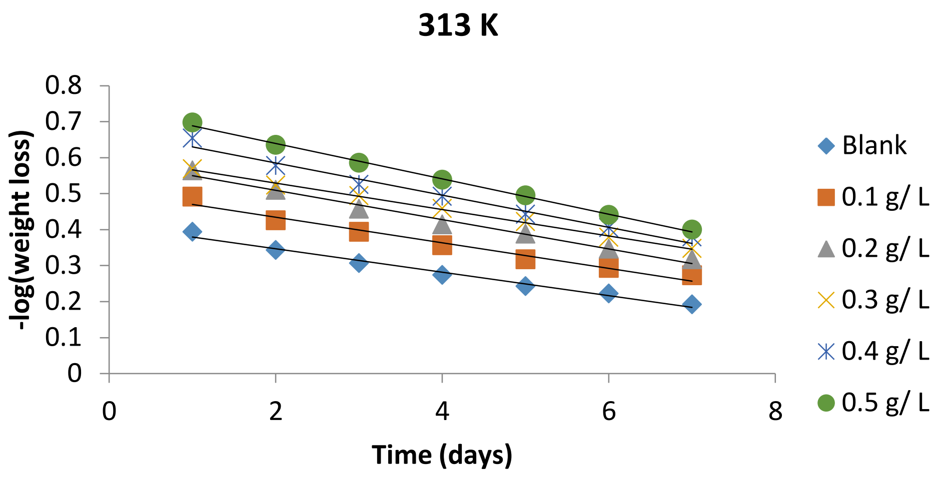

Fig. 6: Variation of –log(weight loss) with time for the corrosion of mild steel in 0.1 M H_2SO_4 containing various concentrations of plant extract (DRLE) at 313 K

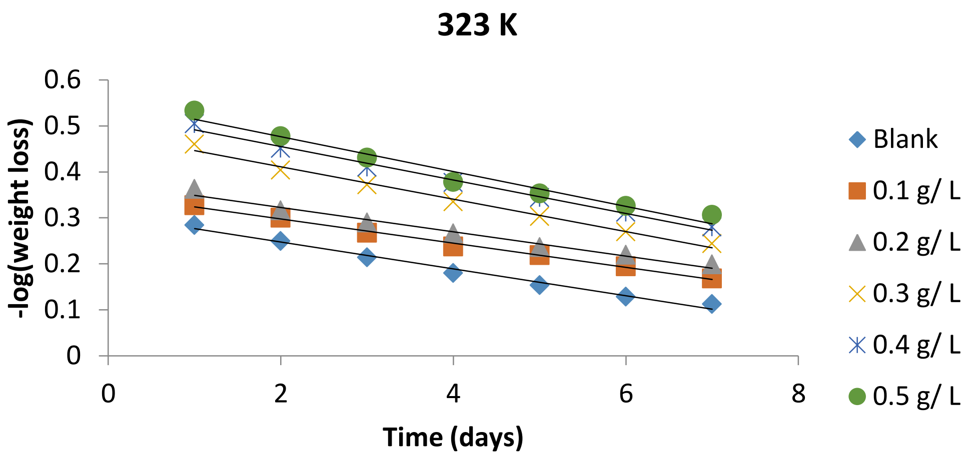

Fig. 7: Variation of –log(weight loss) with time for the corrosion of mild steel in 0.1 M H_2SO_4 containing various concentrations of plant extract (DRLE) at 323 K

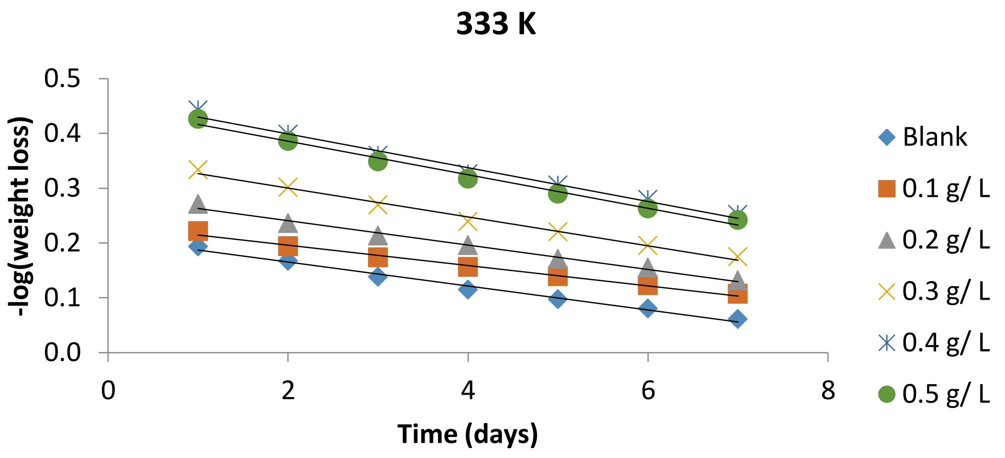

Fig. 8: Variation of –log(weight loss) with time for the corrosion of mild steel in 0.1 M H_2SO_4 containing various concentrations of plant extract (DRLE) at 333 K

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Table 5: Kinetic parameters for the inhibition of the corrosion of mild steel in 0.1 M H_2SO_4 by plant extract (DRLE)

T (K)	System	Slope	K_1	$t_{1/2}$ (day)	R^2
303	**Blank**	0.0270	0.1280	5.414063	0.9965
	0.1 g/L	0.0405	0.1045	6.631579	0.9933
	0.2 g/L	0.0355	0.0962	7.203742	0.9840
	0.3 g/L	0.0364	0.0935	7.411765	0.9886
	0.4 g/L	0.0545	0.0877	7.901938	0.9730
	0.5 g/L	0.0468	0.1094	6.334552	0.9720
313	**Blank**	0.0261	0.0974	7.114990	0.9758
	0.1 g/L	0.0359	0.0907	7.640573	0.9924
	0.2 g/L	0.0300	0.0852	8.133803	0.9768
	0.3 g/L	0.0284	0.0796	8.706030	0.9904
	0.4 g/L	0.0275	0.1034	6.702128	0.9963
	0.5 g/L	0.0261	0.0971	7.136972	0.9755
323	**Blank**	0.0232	0.08448	8.203125	0.9890
	0.1 g/L	0.0335	0.0783	8.850575	0.9888
	0.2 g/L	0.0309	0.0769	9.011704	0.9879
	0.3 g/L	0.0284	0.1027	6.747809	0.9950
	0.4 g/L	0.0268	0.0737	9.402985	0.9995
	0.5 g/L	0.0244	0.0716	9.678771	0.9912
333	**Blank**	0.0200	0.0727	9.532325	0.9818
	0.1 g/L	0.0283	0.0684	10.13158	0.9894
	0.2 g/L	0.0273	0.0631	10.98257	0.9930
	0.3 g/L	0.0255	0.0585	11.84615	0.9845
	0.4 g/L	0.0240	0.0559	12.39714	0.9825
	0.5 g/L	0.0229	0.0536	12.92910	0.9830

3.4 Effect of temperature

The dependence of corrosion rate on temperature can be expressed by the modified Arrhenius equation [15]

$$\ln(CR) = \ln A - \frac{E_a}{RT} \qquad (6)$$

where Ea is the apparent effective activation energy, R the general gas constant and A the Arrhenius pre-exponential factor. A plot of the logarithm of the corrosion rate, (CR) vs. 1/T gave a straight line as shown in Figure 9, with slope of -Ea/2.303R. The calculated activation energies for the corrosion process in 0.1 M H_2SO_4 was found to be 13.36 kJ mol^{-1} and values obtained in the presence of 0.1, 0.2, 0.3, 0.4 and 0.5 g /L were, 11.34, 10.42 , 10.21, 9.99, 6.93 kJ mol^{-1} respectively. The plant extract (DRLE) thus reduces the corrosion activation energies for mild steel in 0.1 M H_2SO_4.

Standard enthalpy and entropy changes due to the adsorption of the plant extract (DRLE) was calculated from alternative formulation of Arrhenius equation (the Transition state equation), which can be written according to equation 7 [4].

$$log\left(\frac{CR}{T}\right) = log\left(\frac{R}{Nh}\right) + \frac{\Delta S^0_{ads}}{2.303R} - \frac{\Delta H^0_{ads}}{2.303RT} \qquad (7)$$

where CR is the corrosion rate at the temperature, T, R is the universal gas constant, N is the Avogadro's number, h is the Planck constant, ΔS^0_{ads} is the standard entropy change of adsorption and ΔH^0_{ads} is the standard enthalpy of adsorption of the inhibitor.

Figure 10 shows a plot of log CR/T against reciprocal of absolute temperature (transition state equation) which gave a straight line in form of a linear graph with slope equal to -ΔHa/2.303R and the intercept equal [log(R/Nh)+ΔS^0_{ads}

/2.303R]. The enthalpy of activation (-ΔH_a) and the entropy of activation (ΔS_a) were calculated and tabulated in Table 7. The entropy of activation is positive in both absence and presence of inhibitor, the increase in entropy implies disordering took place on going from reactants to the activated complex. The negative sign of enthalpy of activation reflect the exothermic nature of steel dissolution process. The increase of ΔH_a accompanying the increase in the inhibitor concentration is explained by an increase of the energy barrier of the corrosion reaction.

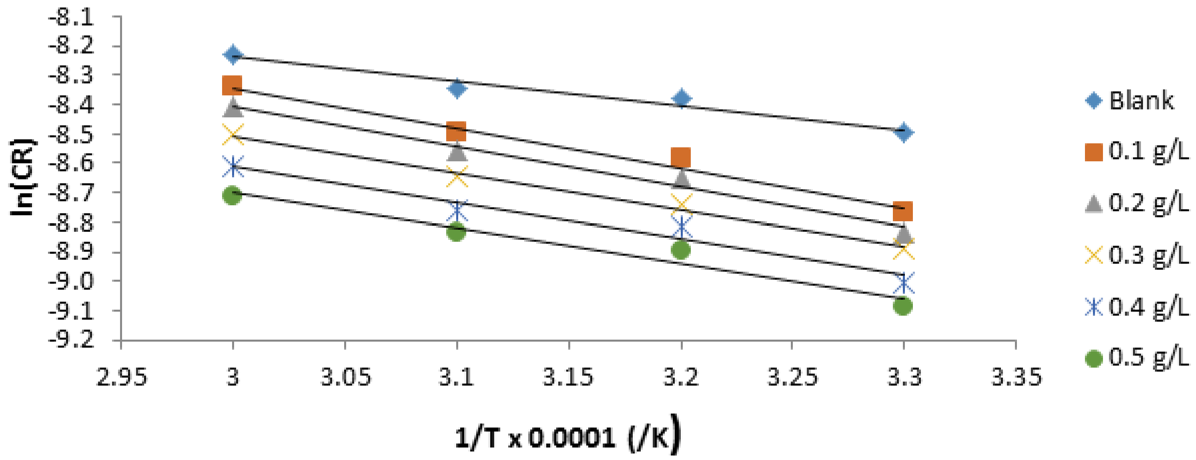

Fig. 9: Arrhenius plots for the inhibition of the corrosion of mild steel in solution of H_2SO_4 by DRLE

Table 6: Arrhenius parameters for the inhibition of the corrosion of mild steel by the plant extract (DRLE)

System	Slope	Intercept	E_a (kJ/mol)	A	R^2
Blank	0.8314	-5.743	13.36	3.22E-06	0.9693
0.1 g/L	1.3680	-4.2411	11.34	1.46E-05	0.9844
0.2 g/L	1.3635	-4.3175	10.42	1.34E-05	0.9865
0.3 g/L	1.2531	-4.7465	10.21	8.69E-06	0.9967
0.4 g/L	1.2277	-4.929	9.99	7.24E-06	0.9593
0.5 g/L	1.1994	-5.1035	6.93	6.09E-06	0.9532

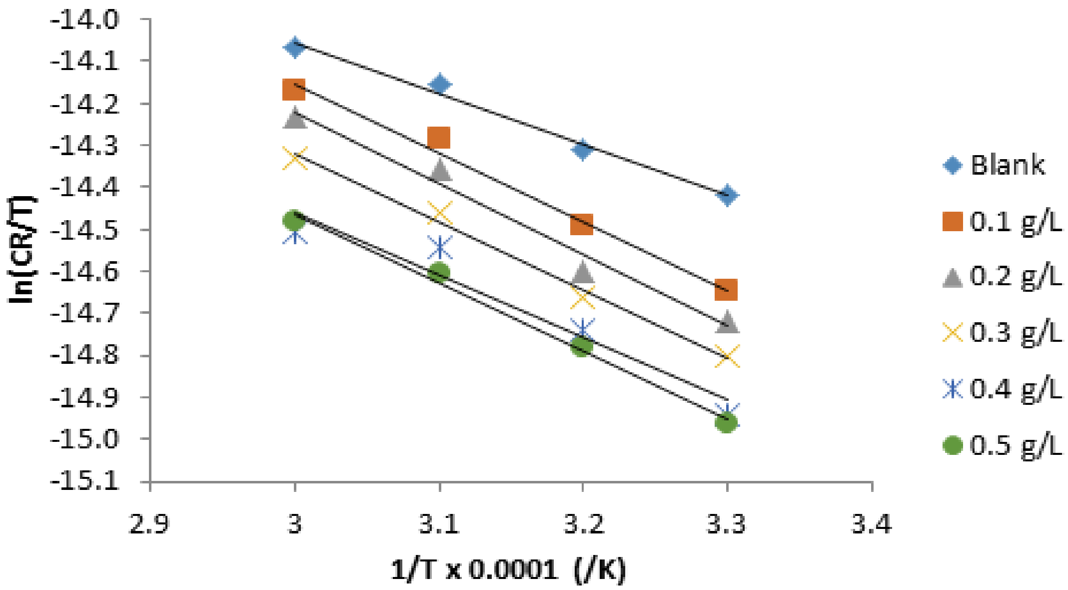

Fig. 10: Transition state plots for the inhibition of the corrosion of mild steel in solution of 0.1M H_2SO_4 by the plant extract (DRLE)

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Table 7: Thermodynamic parameters for the adsorption of various concentration of the inhibitor on mild steel surface

System	Slope	Intercept	$\Delta H^0_{ads}(\frac{kJ}{mol})$	$\Delta S^0_{ads}(\frac{kJ}{mol})$	R^2
Blank	1.2222	-10.4930	-18.22	136.41	0.9873
0.1 g/L	1.7776	-9.0856	-16.56	148.11	0.9823
0.2 g/L	2.0188	-8.4276	-15.92	153.58	0.9672
0.3 g/L	1.8776	-8.9399	-14.88	149.32	0.9781
0.4 g/L	1.8600	-9.1722	-14.46	147.39	0.9972
0.5 g/L	1.8698	-9.2143	-13.55	147.04	0.9995

3.5 Adsorption considerations

The adsorption characteristics of ethanol extract of *Delonix regia* leaves were studied by fitting data obtained from weight loss measurements into different adsorption isotherms. The test revealed that Langmuir adsorption isotherm best described the adsorption characteristics of the *Delonix regia* leaves extract (DRLE).

The assumptions of Langmuir adsorption isotherm can be expressed according to equation 8 which can also be written as equation 9

$C/\theta = 1/k + C$ (8)

$\log(C/\theta) = \log C - \log k$ (9)

From equation 9, a plot of log(C/θ) versus logC should be linear provided the assumptions of Langmuir adsorption isotherm are valid. Though the plot of logC/θ versus logC was linear (Fig.11) (correlation 0.999), the deviation of the slopes from unity (for ideal Langmuir isotherm) can be attributed to the molecular interaction among the adsorbed inhibitor species, a factor which was not taken into consideration during the derivation of the Langmuir equation. The fit of the experimental data to this isotherm provides evidence for the role of adsorption in the observed inhibitive effect of the *Delonix regia* leaves extract (DRLE).

The equilibrium constant of adsorption (k) in the Langmuir equation (Equation 9) is related to the free energy of adsorption (ΔG_{ads}) according to Equation 10 [5, 15].

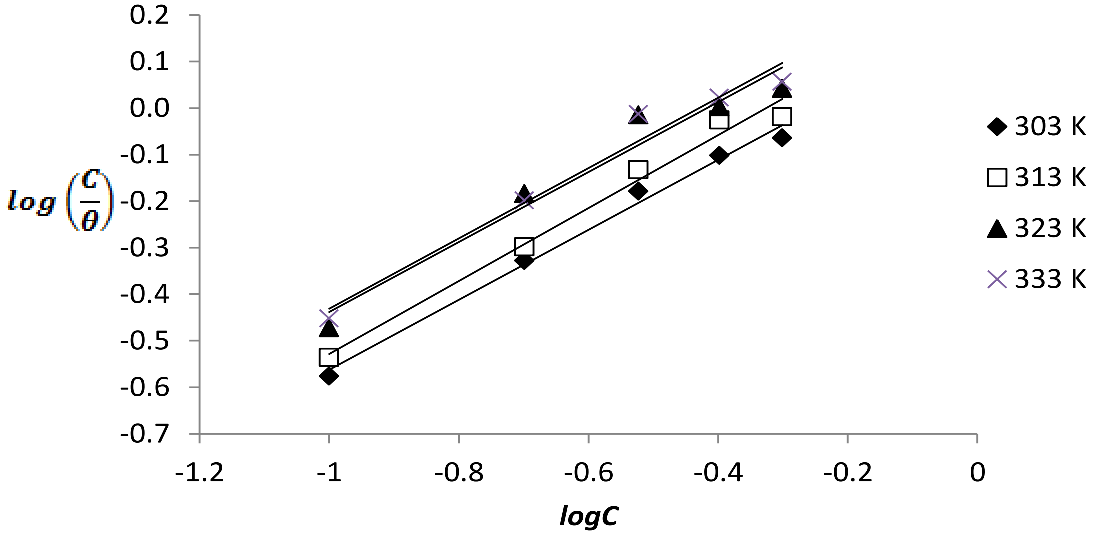

Fig. 11: Variation of log(C/θ) with logC for the adsorption of DRLE on the surface of mild steel

Table 8: Langmuir parameters for the adsorption of plant extract on mild steel surface

T (K)	Slope	Intercept	$\Delta G^0_{ads}(kJ/mol)$	R
303	0.7524	0.1898	-12.20	0.9944
313	0.7842	0.2560	-12.96	0.9922
323	0.7521	0.3141	-13.70	0.9837
333	0.7553	0.3243	-14.16	0.9826

$$\Delta G_{ads} = -2.303RT\log(55.5K_{ads}) \quad (10)$$

Values of ΔG_{ads} calculated from equation 10 was -21.23KJ/mol indicating that the adsorption of the plant extract on the surface of mild steel is spontaneous and occurred according to the mechanism of physical adsorption [5].

4. CONCLUSIONS AND RECOMMENDATIONS

From the results and findings of the study, the following conclusions were drawn.

1. Ethanol extract of *Delonix regia* leaves is an adsorption inhibitor for the corrosion of mild steel in acidic (0.1M H_2SO_4) medium.
2. Inhibition of the corrosion of mild steel by ethanol extract of *Delonix regia* leaves is due to the phytochemical constituents of the plant extract. These phytochemicals enhanced the adsorption behaviour of the inhibitor.
3. The corrosion of mild steel in the presence and absence of ethanol extract of *Delonix regia* leaves is first order and the extract increased the half-life of mild steel in H_2SO_4.
4. The adsorption of ethanol extract of *Delonix regia* is spontaneous and occurred according to the mechanism of physical adsorption.
5. Langmuir adsorption isotherm best described the adsorption characteristics of DRLE on the surface of mild steel.

In view of the above conclusion, the use of ethanol extract of *Delonix regia* leaves as eco-friendly inhibitor for mild steel corrosion is highly recommended.

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