Corrosion inhibition of Urena lobata leave extract on mild steel corrosion in H_2SO_4 acid

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ABSTRACT
Inhibitive potential of Urena lobata leaves extract was studied using gravimetric technique. The study revealed that inhibition efficiency increased with increase in inhibitor's concentration up to 86.39% at 24 hrs for 1.0g/L and decreased with temperature rise. The inhibition process followed physical adsorption mechanism with Langmuir R-squared value of 0.9984 at 303 K. The half-life of the inhibitor decreases with both increase in inhibitor concentration and temperature. Thermodynamic parameters showed that the adsorption of inhibitors on the mild steel surface was feasible, spontaneous, exothermic with increase in disorderliness.
Keywords: Corrosion, inhibition, Urena lobata, weight loss, adsorption.

I.   INTRODUCTION

Appearance and strength of metallic materials are affected adversely when in contact with acidic, and alkaline environment leading to degradation of materials. This results in time and economic losses due to breakdown of refineries, power stations and industrial plants (Amabogha, 2013). Besides, corrosion products act as poisonously contaminants in drugs, dyes, paints, food processing and other fine chemical production industries (Satapathy et al., 2009). As a result of consequences of corrosion effects, many researchers have shown interest in metal protection.

Whenever mild steel is in acidic medium, it experiences excessive corrosive attack (Noor and Al-moubaraki, 2008). One of the commonest protection methods for metallic materials is the use of inhibitors. Inhibitors are substances which when added in small amount retards the corrosion process. They help to isolate the metals from the corrosive media thereby reducing the rate of corrosion reaction (Oguzieet al., 2007; Quraishi et al., 2010).

Initially, inorganic and organic inhibitors were used for inhibition of corrosion. Their contaminations and toxicity in the environment hindered their continuous usage. This resulted in a concerted research by scientists to the development of cheap and environmentally benign inhibitors. It has been reported that organic molecules containing-π-bond and heteroatoms like nitrogen, oxygen, phosphorus,and sulphur show good inhibition properties for mild steel corrosion (Emregul and Hayyali, 2004; Oguzie and Ebenso, 2006).

Natural organic inhibitors from plant origin have been recognized as cheap, viable, non-toxic, and environmentally friendly inhibitors(Martinez 2002, Olusegun et al., 2016). Many plant extracts such as Vernonia amygdalina (Eddy et al., 2009), fruit peel (Da Rocha et al.,2010), Khaya ivorensis gum (Ameh, 2012), Boscia senegalensis (Awe et al., 2015), Azadirachta indicia (Ajanaku et al.,2015), Euphoria royleana (Thapa et al., 2019),etc have been used as efficient inhibitors for steel in harsh environment because of the presence of phytochemical molecules in the extracts.Inhibition of extracts on the metal corrosion is because of adsorption of the molecules onto the metal surface, increasing/decreasing anodic and cathodic reactions, decreasing the diffusion rate of the to the surface of the metal and formation of a protective barrier film (Amitha Rani and Bashu 2012, Ben Hmamouet al., 2012).

II. EXPERIMENTAL

2.1 Material Preparation

Mild steel (MS) of weight % as C (0.2300), Mn (0.2654), Cr (0.0528), Co (0.0313), Ni (0.2205), Cu (0.2120), Mo (0.0330), Si (0.0547) and the rest Fe, was used for the study. The mild steel was mechanically cut to coupons of dimension 2 x 3 x 0.14 cm. All reagents used for the study were analar grade and distilled water was used for the preparation

2.2 Extraction of plant leaves

The leaves of the plant were gathered within Aluu, Port Harcourt, Nigeria. It was dried and pulverized. Methanolic extraction was done by soaking 100 g of dry leaves sample for 3 days in a beaker containing 500 cm^3 of methanol. The mixture was filtered, and the solvent evaporated using rotary evaporator at 60°C to concentrate the extracts. The stock solution of the extracts so obtained were used in preparing different concentrations of the extracts.

2.3 Weight loss

The polished pretreated mild steel coupons were immersed in 250 ml 0.5M H$_2$SO$_4$ solution in the presence and absence of Urena lobata leaves extract in an open beaker. The beaker was inserted into a water bath maintained at a temperature of 303K. After every 24 hrs, each coupon sample was withdrawn from the test solution, washed under running water with brush, rinsed in ethanol, and dried in acetone. The difference in weight for a period of 168 hrs was taken as the total weight loss. From the average weight loss (mean of three replicate analyses) results, the inhibition efficiency (%I) of the inhibitor, the degree of surface coverage (θ) and the corrosion rate of mild steel(CR) were calculated using equations 1-3

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

$$\theta = \left(1 - \frac{W_1}{W_2}\right) \tag{2}$$

$$CR = \frac{\Delta W}{At} \tag{3}$$

Where, CR is the corrosion rate of mild steel in g/cm^2/h, W$_1$ and W$_2$ are the weight loses (g) for mild steel in the presence and absence of the inhibitor, θ is the degree of surface coverage of the inhibitor, A is the area of mild steel coupon (in cm^2), t is the period of immersion (in hours) and ΔW is the weight loss of mild steel after time, t.

2.4 Scanning Electron Microscopy

Surface morphological examinations of mild steel in 0.5 M H$_2$SO$_4$ were done by Scanning electron microscopy (SEM). Mild steel specimens were immersed in 0.5 M H$_2$SO$_4$ for 168 hours with and without 1.0 g/L U. lobata. The specimens were taken out, washed well and dried. SEM images of the specimen were recorded usingModel-PHENOM ProXScanning Electron Microscope (SEM)

2.5 FT-IR Spectroscopy

FT-IR spectra of U. lobata and that of the film formed on the surface of mild steel after immersion in 1.0g/L U. lobata solution for 168 hours were recorded using FTIR-8400S FourierTransform InfraredSpectrophotometer in the frequency range of 4000-450cm^{-1}.

III. RESULTS AND DISCUSSION

3.1 Gravimetric Analysis

In weight loss measurement, the effect of immersion time up to 168 hrsand effect of concentration of inhibitor's extract on the inhibition of corrosion of mild steel were studied. The variation of weight loss with time of immersion of MS on different concentrations of inhibitors were shown in Figures 1-4. The plots reveal that the plant extract significantly retards the corrosion of mild steel in acidic solution at all concentrations. Weight loss and corrosion rate were found to increase with increase in temperature and decrease with increase in inhibitors concentration while inhibition efficiency decreased with increase in temperature. This suggest that the adsorption of U. lobata leaves extract on mild steel surface was consistent with physical adsorption mechanism. Table 1 presents the calculated inhibition efficiency and corrosion rate as a function of both immersion time and inhibitor concentration. It also revealed that Corrosion rate decreases with increase in inhibitor's concentration and increases with temperature. Increase in corrosion rate with temperature was due to the desorption of already adsorbed inhibitor molecules at higher temperature (Bentisset al.,2009).
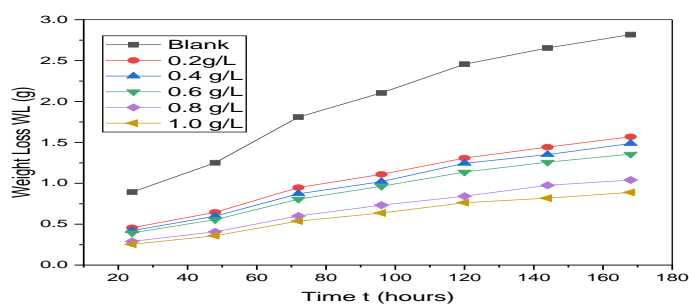
Figure 1: Variation of weight loss with time of MS in 0.5M H_2SO_4 at 303K in the absence and presence of different concentration of U. lobata leaves extract
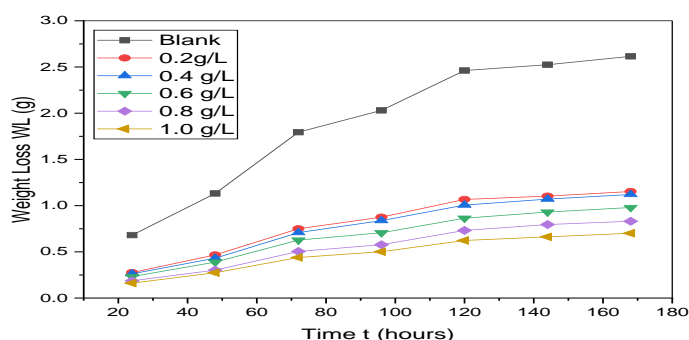

Figure 2: Variation of weight loss with time of MS in 0.5M H_2SO_4 at 313K in the absence and presence of different concentration of U. lobata leaves extract
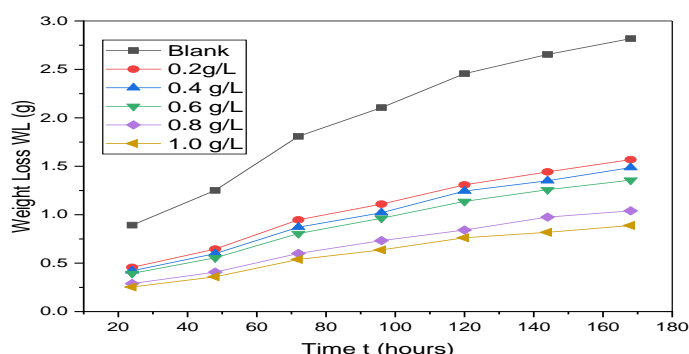

Figure 3: Variation of weight loss with time of MS in 0.5M H_2SO_4 at 323K in the absence and presence of different concentration of U. lobata leaves extract
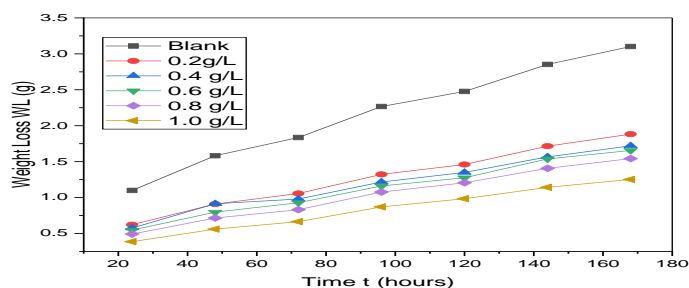

Figure 4: Variation of weight loss with time of MS in 0.5M H_2SO_4 at 333K in the absence and presence of different concentration of U. lobata leaves extract

Table 1: Corrosion rate (CR) of mild steel and inhibition efficiency (%I) of methanolic extract of U. lobata

Concentration (g/L)	CR mpy				I (%)			
	303K	313K	323K	333K	303K	313K	323K	333K
Blank	80.39	115.93	126.91	142.80	-	-	-	-
0.2	31.31	49.06	66.84	83.06	62.39	59.46	48.83	43.32
0.4	28.04	46.62	62.19	77.75	66.46	61.43	52.81	47.52
0.6	23.86	40.80	57.70	73.04	71.73	65.83	55.91	50.24
0.8	21.60	33.39	43.19	66.64	74.94	72.53	67.57	55.20
1.0	16.89	28.70	37.85	53.25	86.39	76.28	71.54	64.87

3.2 Adsorption Kinetic Study

As depicted in Figures 5 - 8, the corrosion data fit the rate law for first order reaction as expressedby Abiola and Otaigbe, (2008) in Equation 4

$$-\log(Wi - Wt) = \frac{k_1 t}{2.303} + Wi \quad (4)$$

Where, Wi is the initial weight of mild steel coupon, [Wi-Wt] is the weight loss of mild steel at time t and k is the first-order rate constant.

The obtained linear plots with correlation coefficient of almost 1, confirm a first-order kinetics for the corrosion of mild steel in 0.5M H_2SO_4 solution without and with the U. lobata extract.The corrosion rate k and half -life ($t_{\frac{1}{2}}$) increases and decreases with temperature respectively as presented in Table 2.

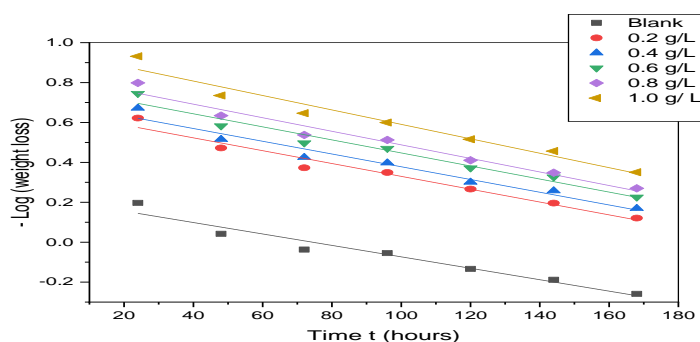

Figure 5: Variation of – log (Weight Loss) with time for MScorrosion in 0.5M H_2SO_4 containing various concentrations of U. lobata leaves extract at 303K
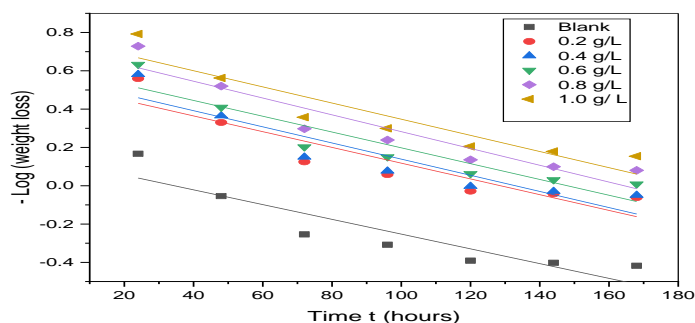

Figure 6: Variation of – log (Weight Loss) with time forMScorrosion in 0.5M H_2SO_4 containing various concentrations of U. lobata leaves extract at 313K
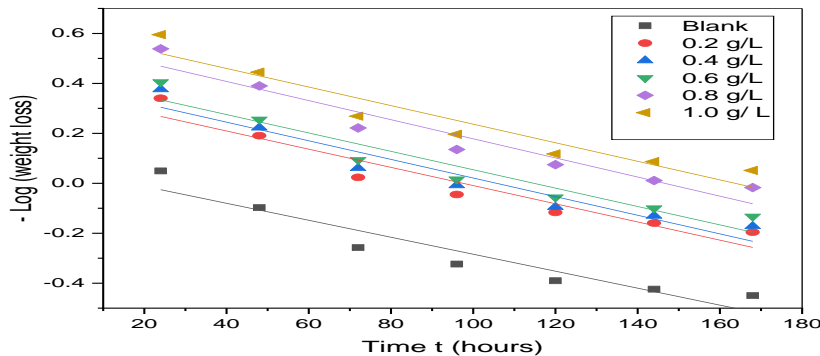
Figure 7: Variation of – log (Weight Loss) with time for MScorrosion in 0.5M H_2SO_4 containing various concentrations of U. lobata leaves extractat323K
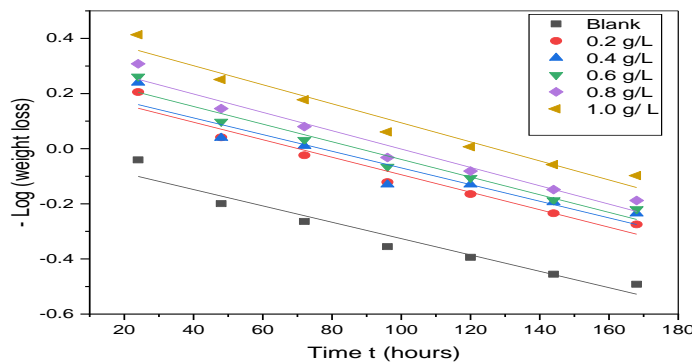

Figure 8: Variation of – log (Weight Loss) with time for MScorrosion in 0.5M H_2SO_4 containing various concentrations of U. lobata leaves extract at 333K

Table 2: Kinetic parameters for the inhibition of the corrosion of mild steel in 0.5M H_2SO_4solution containing various concentrations ofU. lobata leaves extract at different temperatures.

Concentration (g/L)	k (hrs⁻¹)				$t_{1/2}$(hrs)				R^2			
	303K	313K	323K	333K	303K	313K	323K	333K	303K	313K	323K	333K
Blank	0.00661	0.00684	0.00783	0.00887	104.84	101.32	88.51	78.13	0.9560	0.9476	0.9073	0.8368
0.2	0.00742	0.00730	0.00838	0.00947	93.40	94.93	82.70	73.18	0.9693	0.9556	0.9232	0.8485
0.4	0.00735	0.00696	0.00859	0.00972	94.29	99.57	80.68	71.30	0.9652	0.9431	0.9300	0.8621
0.6	0.00753	0.00737	0.00848	0.00949	92.03	94.03	81.72	73.02	0.9658	0.9648	0.9263	0.8657
0.8	0.00783	0.00767	0.00882	0.01011	88.51	90.35	78.57	68.55	0.9677	0.9791	0.9314	0.8834
1.0	0.00834	0.00795	0.00857	0.00972	83.09	87.17	80.86	71.30	0.9590	0.9584	0.9140	0.8681

3.3 Effect of Temperature

The Arrhenius equation (5) and Transition state equation (6) were used in calculating activation parameters such as Activation Energy Ea, Activation enthalpy change $\Delta H^*$and activation entropy change ΔS^* for the corrosion process.

$$\log CR = \log A + \left(-\frac{Ea}{2.303RT}\right) \tag{5}$$

$$\log \frac{CR}{T} = -\frac{\Delta H^*}{2.303RT} + \left(\frac{\Delta S^*}{2.303R} + \log \frac{R}{Nh}\right) \tag{6}$$

Where, T is the temperature in kelvin, R is the universal gas constant (8.314 J/mol/K), N is the Avogadro's number (6.02×10^{23} mol^{-1}), and h is the Planck's constant (6.626×10^{-34}Js). For Arrhenius equation, a plot of $\log CR$ against $\frac{1}{T}$ gives a straight line with intercept $(\log A)$ and slope $\left(-\frac{E_a}{2.303R}\right)$ while for Transition state equation, a plot of $\log \frac{CR}{T}$ against $\frac{1}{T}$ gives a straight line with intercept $(\frac{\Delta S^*}{2.303R} + \log \frac{R}{Nh})$ and slope $(-\frac{\Delta H^*}{2.303R})$
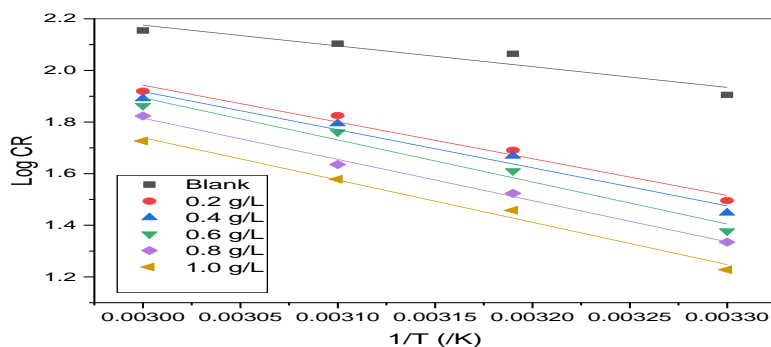

Figure 9: Arrhenius plots in the presence and absence of different concentrations of U. lobata leaves extract on MS inhibition
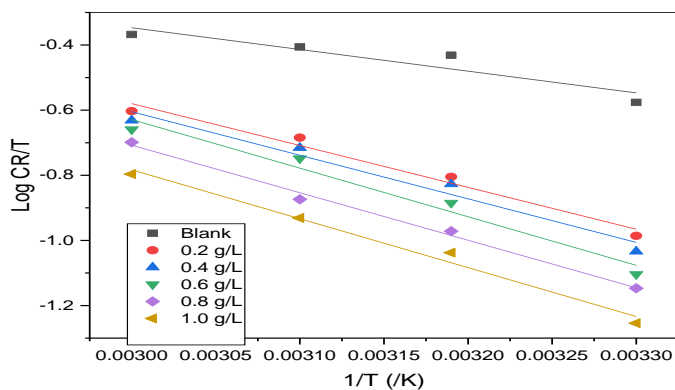

Figure 10: Transition state plots in the presence and absence of different concentrations of U. lobata leaves extract on MS inhibition

Table 3: Corrosion activation parameters for MS in 0.5M H_2SO_4 in the absence and presence of different concentrations of the inhibitor.

Inhibitor Concentration (g/L)	E_a (kJ/mol)	ΔH^* (kJ/mol)	ΔS^* (J/mol/K)
Blank	15.40	12.76	-165.93
0.2	27.25	24.62	-134.82
0.4	28.24	25.62	-132.31
0.6	31.21	28.57	-123.91
0.8	30.59	27.94	-127.29
1.0	31.38	28.75	-126.33

From Table 3, activation energyE_a values for inhibited systems were higher than those for uninhibited system. This could be due to adsorption of inhibitor's molecules on the surface of mild steel which increases the energy barrier of the corrosion process thereby retarding the corrosion reaction (Ebenso, 2003; Umoren and Obot, 2008). Higher values of E_a in the presence of inhibitor can be correlated with increasing thickness of double layer which enhances the E_a of the corrosion reaction (Singh et al., 2008). Because the values of E_a obtained in this work is less than 80kJ/mol, it represents physical adsorption (Obot and Obi-Egbedi, 2008). The positive values of activation enthalpy ΔH*indicate that the adsorption of inhibitor molecules was endothermic. Higher values of ΔH* in the presence of inhibitor makes the mild steel dissolution difficult because of the protection offered by the adsorbed inhibitor (Ebenso and Obot, 2010). Activation entropy being negative indicates non-spontaneous process. Since the values of ΔS* in the presence of inhibitor increases than in blank solution, it shows increase in disorderliness in going from reactants to activated complex (Shukla andEbenso, 2011).

3.4 Thermodynamics and Adsorption Considerations

The adsorption characteristics of the inhibitor was studied by fitting the data obtained for degree of surface coverage of the inhibitor into adsorption isotherms like Freundlich, Langmuir,Frumkin, Temkin and Henry (Figures 11 – 15). The correlation coefficient (R^2) was used to determine the best fitted isotherm (Table 4). From Table 4, it can be seen that the R^2 values for all the isotherm models are close to unity and the order of fitting of the data to the isotherms is as follows: Langmuir > Henry >Freundlich > Frumkin >Temkin.

The equilibrium constant of adsorption obtained from the intercept of Langmuir adsorption isotherm whichwas related to the Gibb's free energy of adsorption was calculated using equation 7 and the results presented in Table 4.

$$\Delta G°_{ads} = -2.303 \, RT\log(55.5K_{ads})(7)$$

From the results obtained, the free energies were negatively less than -40 kJ/mol which was the threshold for chemical adsorption mechanism (Abdallah,2009). The negative values of $\Delta G°_{ads}$ indicates the spontaneity of the adsorption process and the stability of adsorbed species on the mild steel surface (Fouda et al., 2009). Hence, the adsorption of U. lobata leaves extract on the mild steel surface was spontaneous and support physical adsorption mechanism.

Table 4: Isotherm for adsorption of inhibitors on Mild Steel surface

| Adsorption Isotherm	Temperature (K)	R^2	K_{ads}	ΔG°(kJ/mol)
Langmuir	303	0.9984	0.7625	-9.436
	313	0.9951	0.7267	-9.623
	323	0.9838	0.6681	-9.704
	333	0.9874	0.5744	-9.586
Henry	303	0.9914	0.21750	-6.276
	313	0.9781	0.23175	-6.648
	323	0.9527	0.30425	-7.592
	333	0.9454	0.24335	-7.208
Freundlich	303	0.9531	0.7978	-9.550
	313	0.9938	0.7681	-9.767
	323	0.9125	0.7161	-9.891
	333	0.9224	0.6393	-9.883
Frumkin	303	0.9772	0.0175	-0.074
	313	0.9484	0.0282	-1.166
	323	0.9415	0.0693	-3.618
	333	0.9198	0.0640	-3.510
Temkin	303	0.9359	1390.91	-28.356
	313	0.8754	775.89	-27.772
	323	0.8413	108.94	-23.387
	333	0.8243	160.51	-25.184
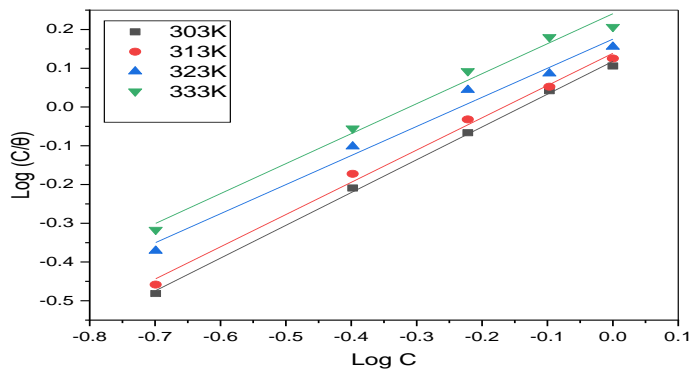
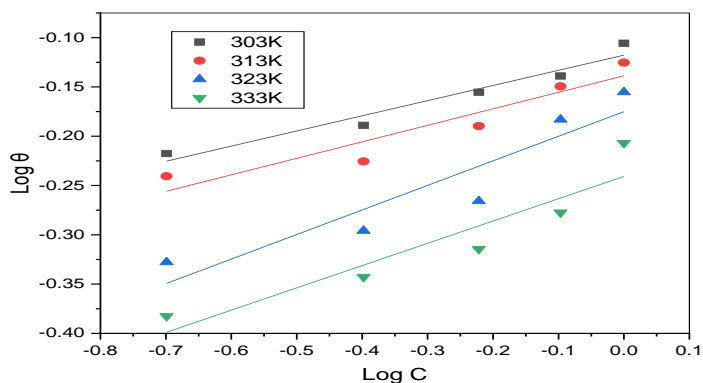Figure 11: Langmuir Isotherm for adsorption of U. lobata on mild steel surface

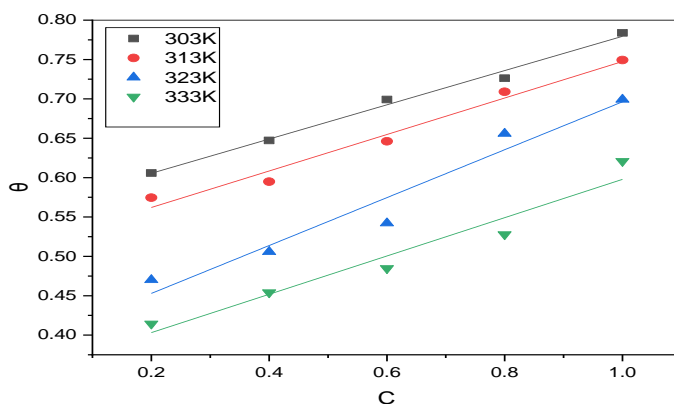Figure 12: Freundlich Isotherm for adsorption of U. lobata on mild steel surface

Figure 13: Henry Isotherm for adsorption of U. lobata on mild steel surface
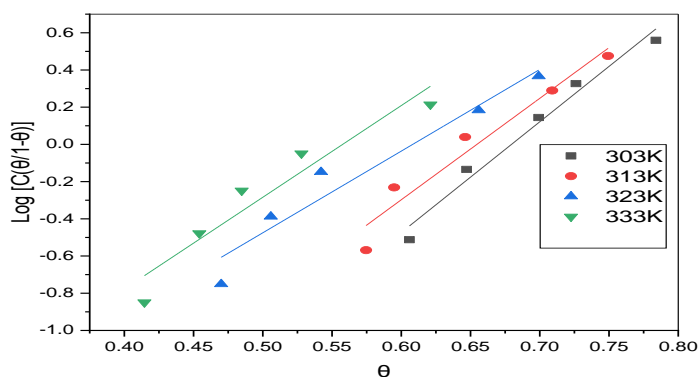
Figure 14: Frumkin Isotherm for adsorption of U. lobata on mild steel surface
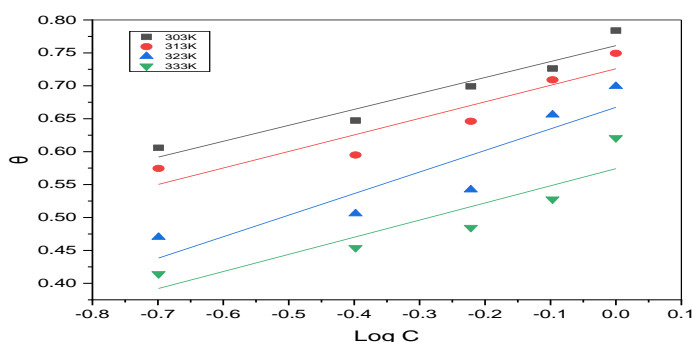

Figure 15: Temkin Isotherm for adsorption of U. lobata on mild steel surface

Corrosion inhibition of mild steel in the presence of U. lobata leaves extract can be better explained using enthalpy of adsorption $\Delta H°_{ads}$ and entropy of adsorption $\Delta S°_{ads}$ which can be calculated from the integrated van't Hoff equation (Shukla and Ebenso, 2011):

$$\ln K_{ads} = -\frac{\Delta H°_{ads}}{RT} + \frac{\Delta S°_{ads}}{R} - \ln 55.5 \qquad (8)$$

A graph of $\ln K_{ads}$ against $\frac{1}{T}$ (Figure 16) with slope$(-\Delta H°_{ads}/R)$ and intercept$(\Delta S°_{ads}/R - \ln 55.5)$ was plotted and the values of $\Delta H°_{ads}$ and $\Delta S°_{ads}$ listed in Table 5.

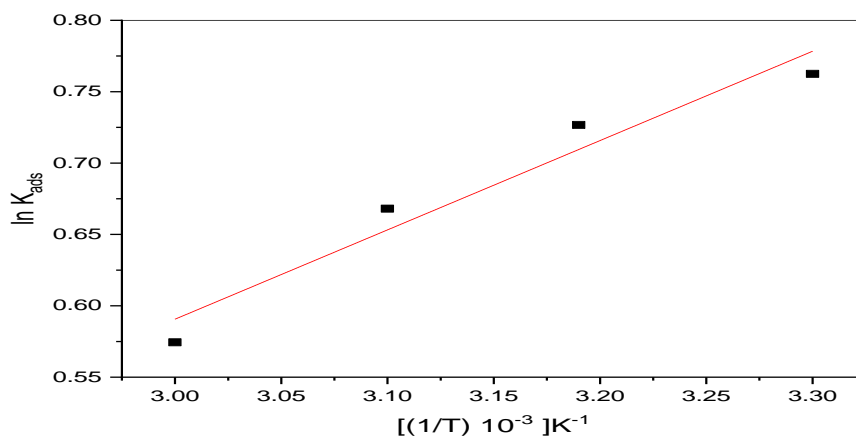

Figure 16: van't Hoff plot of $\ln K_{ads}$ against $\frac{1}{T}$

Table5: Enthalpy and Entropy of adsorption values of U. lobata leaves extract on mild steel from van't Hoff plot

Slope	Intercept	ΔH°_{ads} (kJ/mol)	ΔS°_{ads} (J/mol/K)
0.62572	-1.28653	-5.202	22.696

The enthalpy of adsorptionΔH°_{ads} obtained was negative and less than - 40 kJ/mol indicating exothermic physical adsorption mechanism. ΔS°_{ads} is positive indicating spontaneous process with increase in disorderliness whileΔG°_{ads} is negative showing feasible and spontaneous process.
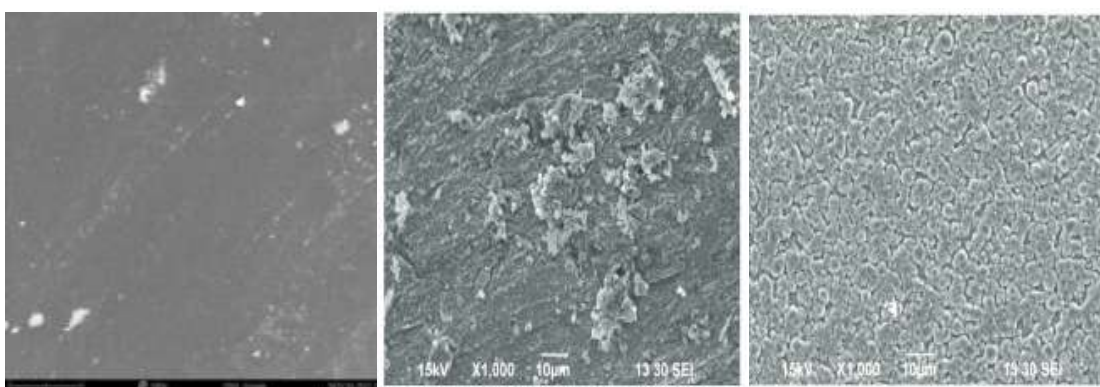
3.6. Scanning Electron Microscopy

Figure 17: SEM image of (a) Polished Mild steel before corrosion (b) mild steel in 0.5M H_2SO_4 in the absence of ULLE for 168 h (c) mild steel in 0.5 M H_2SO_4 in the presence of 1.0g/L ULLE for 168 h

The SEM analysis was carried out on polished mild steel surfaces immersed for 168 h in 0.5M H_2SO_4 solution in the presence and absence of leaf extract as presented in Figure 17. By comparing the SEM images, (Figure 17b) shows that the mild steel surface is strongly damaged in the absence of extract inhibitor, which results in a heterogeneous and rough surface due to corrosive nature of the acid. On the other hand, the inhibited solution (Figure 17c) reveals smooth surface morphology because of formation of extract protective layer on the surface of the mild steel thereby, inhibiting the corrosion process.
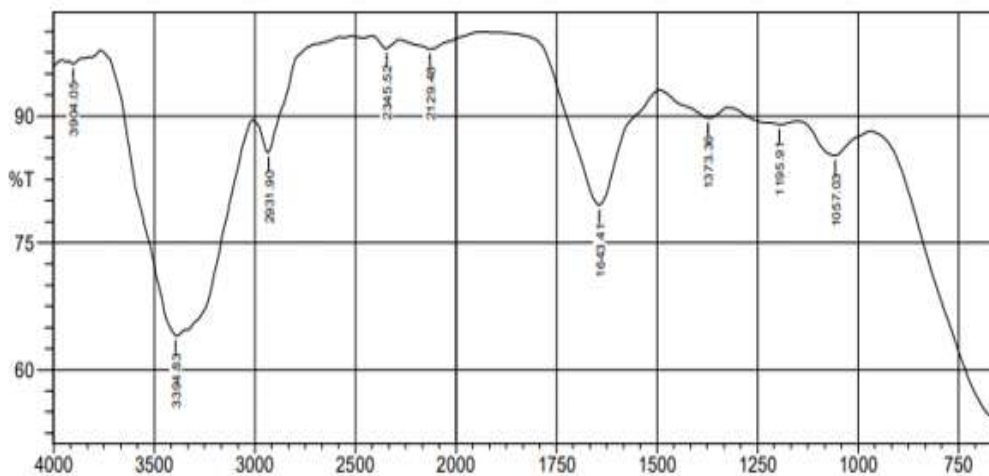
3.7 FT-IR Spectroscopy

Figure 18: FTIR of U.lobata leaves extract

Table 6: FTIR of the detected functional group in U. lobata leaves extract

Functional group	Wave number cm^{-1}
O-H stretch of alcohol	3904.05
C≡C-O	3394.83
C-H asymmetric stretching of CH_2 in fatty acid	2931.90
C-O bond	2345.52
C≡C	2129.40
N-H bend from amine/amide	1643.41
C-H bonds bending symmetric CH_2 group	1373.36
C-N bond from amine	1195.91
C-H bending	1067.03

FTIR spectra were used to assess the protective film created on the metal surface to reveal the bonding type for organic inhibitors adsorbed at the surface the metal. Figure 18 and Table 6 revealed the presence of many active functional groups like ketones, aldehydes, amines, amides, alcohols, alkenes, alkynes and aromatics, which have inhibitory properties.

IV. CONCLUSION

Methanolic extract of U.lobata leaves was found to be effective inhibitor for mild steel corrosion in 0.5 M H_2SO_4. The inhibition efficiency of the inhibitor increased with increasing concentration of the extract and decreased with temperature rise. Adsorption of inhibitor molecules on the surface of mild steel was responsible for the protection against corrosion. Apparent activation energy increased with increase in inhibitor concentration revealing physisorption mechanism. The mechanism of adsorption of the inhibitor on mild steel followed first order kinetics with half-life decreasing as temperature increases and R-squaredvalues close to unity.Thermodynamic parameters obtained for inhibitor/metal system showed that the adsorption of inhibitors on the mild steel surface was feasible, spontaneous, exothermic with increase in disorderliness. Adsorption isotherm models –Langmuir, Henry, Freundlich, Frumkin and Temkin isotherm fit well as evident from the correlation coefficient values ($R^2 \approx 1$ in all cases). This proves the applicability of all the models to the process.

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