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Studies on the Corrosion Inhibition of Ethanol Extract of *Newbouldia Laevis* Stem Bark and Leaf on Mild Steel in Acidic Medium

Agbenu C. Adah, Joseph A. Gbertyo, Ajeh, G. Otokpa

Department of Chemistry, Benue State University, Makurdi-Nigeria christieadah@gmail.com

Abstract Numerous failures and eventual losses in the chemical industry have been traced to corrosion processes. Failures resulting from the inability of metals to support designed load requirements because of losses imposed by corrosion effects can be combated economically through the use of chemical inhibitors. Despite the fact that inhibitors are effective, they are associated with problems of toxicity, disposal, litigation and enormous costs. In order to protect the environment and reduce cost, the use of green inhibitors as substitutes and partial replacement of chemical inhibitors have become a method of choice. In this study, ethanol extracts of stem bark and leaf of *Newbouldia laevis* were employed on mild steel samples corroded in 0.5M hydrochloric acid solution in the absence and presence of varying concentrations of the extracts (inhibitors). Weight loss data was recorded after six hours out of which the highest inhibition efficiency was 85 % at 309K for stem bark extract and 76.70% at 309 K for the leaf extract. The trend depicted chemisorption process and fits into Langmuir, Temkin and Adejo-Ekwenchi adsorption isotherms.

Keywords weight loss method, green corrosion inhibitors, Newbouldia laevis

Introduction

Hydrochloric acid solution is commonly used for the removal of undesirable scale and rust in metalworking processes, cleaning of boilers, and heat exchangers [1, 2]. To prevent the metal dissolution and acid consumption during these processes, inhibitors are often use.

Metal depletion due to corrosion has become an essential engineering problem. Perhaps, no other source of waste except that disturbing human life is of greater concern to all. It has well been said that only through the elimination of waste caused by corrosion and the increase in our national efficiency we can hope to lower the cost of living on one hand, and raise our standard of living on the other. The elimination of waste is a total asset; it has no liabilities [3-5].

Corrosion studies might give insight into its kinetics so as to enable the prediction of the service life of equipment thereby indicating the environmental control for which a particular metal may be satisfactorily used [6, 7]. The present work was designed as a contribution to the growing interest on environmentally benign corrosion inhibitors to study (i) corrosion inhibition of mild steel in acid media using weight loss technique at a temperature range of 301 K to 317 K, (ii) to evaluate the activation energy, heat of adsorption process as well as determining the best adsorption isotherm for the adsorption of the extracts of *Newbouldia laevis* stem bark and leaf on mild steel surface.



Materials and Methods

Materials

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Mild steel rods were procured and taken to the Department of Mechanical Engineering, University of Agriculture, Makurdi-Nigeria, where they were mechanically press-cut to form different coupons, each of dimensions $2.0 \times 2.0 \times$ 0.35 cm with a tiny hole drilled at the edge of each for the purpose of suspension in the corrodant. The surfaces of the coupons were thoroughly polished to mirror finish using sand paper, degreased by washing with ethanol, dried with acetone, and preserved in a desiccator. Subsequently, the coupons were weighed and made ready for analysis. All reagents used were of analytical grade and the water used for preparation was distilled.

Methods

Newbouldia laevis stem bark and leaf were harvested, shed-dried for four weeks, pounded with mortar and pestle and soaked in absolute ethanol for 48 hours, with occasional shaking and filtered, thereafter, using a cotton wool. The ethanol was evaporated from the filtrate in a thermostated water bath at about 321 K and the residue preserved in a desiccator. A 0.5 M HCl was prepared and used as blank. The extract so obtained was then used to prepare solutions of 0.1, 0.2, 0.3, 0.4, and 0.5 gdm^{-3} in the 2 M HCl, respectively [8-15].

Weight Loss Measurement

The method adopted was that reported by scholars in [5,16].

Weight loss was evaluated using,

$$W = W_i - W_f$$
 (1)
where W is the weight loss of the coupon, W_i the initial weight and W_f the weight after retrieval. Each reading
reported is an average of three experimental readings recorded to the nearest 0.001 g. The inhibition efficiency was
calculated using the formula,

% I.E =
$$[1 - \frac{w_1}{w_2}] \ge 100$$
 (2)
ere $W_1 and W_2$ are the weight losses in grams of mild steel coupon in the presence and absence of the inhibitor

Whe in the acid solution at the same temperature respectively. The degree of surface coverage, θ , was evaluated by the equation (3) [5, 17]

$$\theta = 1 - \frac{W_1}{W_2} \tag{3}$$

The corrosion rate of the mild steel coupons was determined for the immersion period from weight loss using equation (4)

Corrosion rate (mg/cm²/h) = $\frac{W_L}{At}$ (4)

where W_L is the weight loss in milligrams (mg), A, the coupon surface area in cm² and t the immersion time in hours [6]

Using the Arrhenius equation (equation 5), values of Ea were obtained [3, 18].

$$lnCR = \ln A + \frac{Ea}{RT}$$
(5)

An alternative formula of Arrhenius equation is: (())

$$\ln\left(\frac{CR}{T}\right) = \ln\left(\frac{R}{Nh}\right) + \frac{\Delta S^*}{R} - \frac{\Delta H^*}{RT}$$
(6)
ere *h* is plank's constant, *N* the Avogadro's number, ΔH^* is the enthalpy of activation and ΔS^* the entrop

Wh py of activation. A plot of $ln\left(\frac{c\kappa}{T}\right)$ versus $\frac{1}{T}$ gave a straight line with a slope of $-\frac{\Delta u}{RT}$ and an intercept of $ln\left(\frac{\kappa}{Nh}\right) + \frac{\Delta s}{R}$ from where the values of ΔH^* and ΔS^* were calculated.

The values of heat of adsorption Q_{ads} were evaluated using equation 7 below;

$$\log\left(\frac{\theta}{1-\theta}\right) = \log A + \log K - \frac{Q_{ads}}{2.303 R} \left(\frac{1}{T}\right)$$
(7)



(1)

where θ is the degree of surface coverage, R is the molar gas constant, T is the absolute kelvin temperature, and A is a temperature independent factor. Values of heat of adsorption were obtained from the slope $\left(\frac{-Q_{ads}}{2.303R}\right)$ of a plot of log

$$\left(\frac{\theta}{1-\theta}\right)$$
 against $\frac{1}{T}$.

Values of ΔG_{ads} were evaluated using equation 3.8, where $K = \frac{\theta}{(1-\theta)C}$ $\Delta G = -RTln(55.5K)$



Results and Discussion



Figure 1: Weight loss (g) in the presence and absence of (a) New bouldia laevis stem extract (b) New bouldia laevis leaf extract

Figure 1 (a) and (b) depicts the results of weight loss for the corrosion of mild steel in acid medium in the absence and presence of different concentrations of *Newbouldia laevis* stem bark and leaf extracts (inhibitor) with time at different temperatures respectively. The results show that both stem bark and leaf extracts of the plant inhibited the corrosion of mild steel in acidic medium since there was a general decrease in weight loss in comparison with the blank. It was observed that the inhibitors minimized the loss in weight of mild steel coupons and the reduction was proportional to both inhibitor concentration and temperature. At each studied temperature, the weight loss of the metal without the inhibitor was highest.

Tables 1 and 2 show that inhibition efficiency for both extracts increased with increase in the concentration of the inhibitor and temperature; the highest inhibition efficiency was 96.30 % for stem bark and 94.67 % for leaf extracts obtained at concentration of 0.5 g/dm³ and at 317 K each, while the least value of 8.66 % was found for leaf and 48.91 % for stem bark at 0.1 g/dm³ and at 317 K. From the Tables, it can be seen that there is a significant difference between values of inhibition efficiency of the extract of stem nark and leaf at 301 K and 317 K. This increase in inhibition efficiency with rise in temperature suggest the inhibitor molecules were chemically adsorbed onto the coupon surface [19].

The rate of corrosion that was observed to be high in the blank came down significantly with the introduction of the inhibitors into the corrodent as seen in tables 5 and 6. Generally, it is clear that the rate of corrosion of mild steel was affected by temperature and concentration of these inhibitors [20].

The evaluated values of enthalpy of activation for both stem bark and leaf are positive and are lower than that of the blank as shown in Table 7 and 8, respectively, signifying that inhibition effectiveness increased with increase in temperature [21]. The positive values of enthalpy imply that the inhibition process was endothermic. The average difference in value of the, $E_a - \Delta H^*$ which equates to RT as shown in equation 9 for both extracts was found to be 2.560 kJ/mol almost same as the value of RT (2.570 kJ/mol). This proposes that the corrosion process of this metal in the acid medium is a unimolecular reaction [22].

$$E_a - \Delta H^* = RT$$



(9)

	Conc. g/dm ³	Percentage inhibition efficiency (IE%)							
	Blank	301 K	305 K	309 K	313 K	317 K			
	0.1	48.91	59.88	68.73	68.00	69.86			
	0.2	49.15	60.27	73.09	75.62	73.21			
	0.3	49.87	60.83	77.04	78.95	80.48			
	0.4	50.60	61.22	80.98	85.25	91.22			
	0.5	51.09	66.73	85.77	87.04	96.30			
Т	able 2: V	alues of	inhibitior	n efficien	cy (IE%)	of leaf ex			

 Conc.
 Percentage inhibition efficiency (IE%)

able 2: Values of inhibition efficiency (IE%) of leaf extract										
Conc.	Percentage inhibition efficiency (IE%)									
g/dm ³										
Blank	301 K	305 K	309 K	313 K	317 K					
0.1	8.669	3.593	5.907	45.89	46.99					
0.2	16.00	10.78	12.24	57.17	62.59					
0.3	18.67	19.76	22.79	66.99	74.14					
0.4	27.33	23.95	29.54	73.43	88.35					
0.5	32.67	28.74	28.27	78.10	94.67					

Tables 3 and 4 showed that corrosion rate decreased with concentration of the inhibitor but was high at higher temperatures while surface coverage increased progressively with rise in both inhibitor concentration and temperature. **Table 3:** Values of corrosion rate and surface coverage of *stem bark* extract

Conc.	nc. Corrosion Rate (mgcm ⁻² h ⁻¹)					Surface coverage (θ)					
g/dm ³	301 K	305 K	309 K	313 K	317 K	301 K	305 K	309 K	313 K	317 K	
	$\times 10^{-4}$	×10 ⁻⁴	$\times 10^{-4}$	$\times 10^{-4}$	×10 ⁻⁴						
Blank	8.56	10.96	14.79	39.4	18.04	-	-	-	-	-	
0.1	4.37	4.40	4.630	12.6	5.440	0.4890	0.5989	0.6873	0.6800	0.6986	
0.2	4.35	4.35	3.980	9.60	4.830	0.4915	0.6027	0.7310	0.7562	0.7321	
0.3	4.29	4.29	3.400	8.29	3.520	0.4987	0.6084	0.7704	0.7895	0.8049	
0.4	4.23	4.25	2.810	5.81	1.580	0.5061	0.6122	0.8099	0.8524	0.9122	
0.5	4.19	3.65	2.100	5.10	0.670	0.5109	0.6673	0.8577	0.8704	0.9630	

Table 4: Values of corrosion rate and surface coverage of leaf extract

Conc.	Corrosion Rate (mgcm ⁻² h ⁻¹)				Surface coverage (θ)					
g/dm ³	301 K	305 K	309 K	313 K	317 K	301 K	305 K	309 K	313 K	317 K
	$\times 10^{-4}$	×10 ⁻⁴	×10 ⁻⁴	$\times 10^{-4}$	×10 ⁻⁴					
Blank	5.00	11.46	13.15	14.44	21.70	-	-	-	-	-
0.1	4.25	6.48	7.15	11.90	15.04	0.150	0.4345	0.4564	0.1819	0.4699
0.2	3.56	6.40	6.21	9.45	12.27	0.2875	0.4418	0.5277	0.3496	0.6259
0.3	5.15	5.54	5.71	7.83	9.95	0.3708	0.5164	0.5658	0.4613	0.7413
0.4	2.67	4.83	5.10	6.92	8.19	0.4667	0.5781	0.6117	0.5244	0.8835
0.5	2.29	4.17	3.06	6.67	7.04	0.5417	0.6364	0.7670	0.5415	0.9467

The negative values of entropy of activation, ΔS_{ads}^* , as shown in Tables 7 and 8 indicated that the activated complex in the rate-determining step represented an association, rather than dissociation, that is a decrease in disorderliness in going from reactants to the activated complex [22]. Heats of adsorption for both inhibitors were observed to be all positive indicating that the processes were endothermic.



 ΔG_{ads} values also presented in Table 5 and 6 are all negative and increased with rise in temperature indicating that spontaneity of the adsorption process and stability of absorbed layer increased with rise in temperature for both extracts. However, the values are in the physical adsorption mechanism range as they are all below -20.0

Conc	Ea	- ΔS^*_{ads}	$-\Delta \mathbf{H^*}_{ads}$		Δ			
g/dm ⁻³	(kJ/mol)	(kJ/mol)	(J/mol)	301 K	305 K	309 K	313 K	317 K
Blank	66.70	303.81	64.14	-	-	-	-	-
0.1	62.05	281.52	59.49	-15.70	-17.03	-18.25	-16.73	-17.49
0.2	56.00	261.80	53.58	-13.99	-15.32	-17.02	-15.16	-16.25
0.3	47.79	232.93	44.95	-13.05	-14.35	-16.52	-15.33	-15.56
0.4	44.99	224.23	42.41	-12.40	-13.67	-16.39	-14.66	-15.08
0.5	40.74	209.41	38.18	-11.90	-13.71	-16.71	-14.15	-15.16

Table 5: Evaluated Parameters of Kinetic energy and thermodynamics of Stem bark extract

Table 6: Evaluated Parameters of Kinetic energy and thermodynamics of *leaf*

							-	
Conc	Ea	- ΔS^*_{ads}	$-\Delta \mathbf{H^*}_{ads}$		Δ (G _{ads} (kJ/m	ol)	
g/dm ⁻³	(kJ/mol)	(kJ/mol)	(J/mol)	301 K	305 K	309 K	313 K	317 K
Blank	63.19	290.13	60.61	-	-	-	-	-
0.1	62.16	283.64	59.59	-11.47	-15.36	-15.78	-12.53	-14.50
0.2	56.63	264.91	54.06	-11.80	-13.67	-14.73	-13.02	-14.13
0.3	52.61	250.20	50.04	-11.74	-13.40	-14.09	-13.18	-14.19
0.4	51.75	246.20	49.18	-12.01	-13.30	-13.84	-13.09	-14.32
0.5	53.84	251.30	51.27	-12.20	-13.36	-15.16	-12.69	-14.35

Table 7: Evaluated Parameters of some linearized adsorption isotherms for stem bark extracts

Isotherm	Temperature (K)	\mathbf{R}^2	Slope	Intercept		-∆G
Freundlich			n			
	301	0.8875	0.0277	-0.2856		8.405
	305	0.5117	0.0498	-0.1803		9.132
	309	0.9567	0.1328	-0.0365		10.10
	313	0.8693	0.1740	-0.1108		9.787
	317	0.9695	0.1424	-0.0984		9.988
Langmuir	301	0 9998	1 932	0.0169		20.26
200080000	305	0.9919	1.486	0.0331		18.82
	309	0.9952	1.095	0.0485		18.09
	313	0.9932	1.339	0.0674		17.47
	317	0.9948	1.272	0.0582		18.08
Temkin					-α	
	301	0.8851	0.0318	0.5178	36.21	103.8
	305	0.5029	0.0722	0.6601	15.94	63.57
	309	0.8087	0.3787	0.8069	3.040	22.92
	313	0.8655	0.2399	0.7566	4.799	29.34
	317	0.9553	0.2132	0.7850	5.401	32.93
Adejo-			b			
Ekwenchi	301	0.8826	0.0276	0.3166		11.87
	305	0.4889	0.0850	0.4684		12.92
	309	0.7017	0.4278	0.6492		14.16
	313	0.8615	0.2642	0.5764		13.91
	317	0.9174	0.2713	0.6332		14.43



Isotherm	Temperature (K)	R ²	Slope	Intercept		-∆G
Freundlich			п			
	301	0.9938	0.7920	-0.0160		9.959
	305	0.8663	0.2304	-0.1555		9.277
	309	0.8708	0.2822	-0.0740		9.880
	313	0.9254	0.6394	-0.0569		10.11
	317	0.9940	0.4863	-0.0200		10.45
Longmuir	201	0.0720	0 6743	0 5000		11 29
Langinun	301	0.9739	1 2065	0.3000		11.50
	303	0.9701	1.3903	0.1339		15.20
	212	0.9207	1.1404	0.1447		12.20
	217	0.91/1	1.1054	0.3710		13.05
Tomlin	517	0.9945	1.0505	0.2320	~	14.45
Temkin	201	0.0920	0 5501	0 (0 17	-u	17 77
	301	0.9850	0.5501	0.0847	2.093	1/.//
	305	0.8538	0.2721	0.6768	4.232	24.71
	309	0.8087	0.3787	0.8069	3.041	22.92
	313	0.9620	0.4702	0.6693	2.448	18.98
	317	0.9921	0.5157	0.8135	2.358	18.45
Adejo-			b			
Ekwenchi	301	0.9511	0.3704	0.4225		12.48
	305	0.8372	0.2478	0.4667		12.91
	309	0.7016	0.4278	0.6492		14.16
	313	0.9731	0.3175	0.4110		12.91
	317	0.9739	0.4461	0.5870		14.15

Table 8: Evaluated Parameters of some linearized adsorption isotherms for leaf extracts

Table 7 shows the results of fitness of the leaf extract of the plant to three isotherms tested, using R^2 as the basis. These are the Freundlich, Langmuir, Temkin, and Adejo-Ekwenchi adsorption isotherms.

Freundlich isotherm, a plot of $\log \theta$ versus logC gave a straight line with slope equal to n and intercept logK. The positive adsorption equilibrium constant, K, values are indication of favourable adsorption. The parameter, n, in the Freundlich isotherm is related to intensity of adsorption and it varies with heterogeneity of the material [24] Values of ΔG_{ads} obtained through this isotherm were all below 20 kJ/mol adhering to physisorption.

From the Adejo-Ekwenchi adsorption isotherm in Table 7and 8for both extracts, it was observed that the values of K_{AE} were fairly rising. The increase in K_{AE} value with temperature implies a favourable adsorption process. The values of b did not follow an ordered trend. This could be categorized as physisorption feature. In general, it can be said that the adsorption processes of the extracts can best be modeled by the Freundlich, Langmuir, Temkin and Adejo-Ekwenchi isotherms [25-29].

Conclusion

Newbouldia laevis stem bark and leaf extract can be used as a replacement for synthetic, toxic, and scarce inhibitors due to environmental friendliness [1-6, 12]. The focus on green corrosion inhibitors is mainly due to economic reasons, strict environmental regulations and renewability.

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