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Ethanol Stem Extract Of Mucuna Pruriens As Green Corrosion Inhibitor For Corrosion of Aluminium In H_2SO_4

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ABSTRACT

The corrosion inhibition of aluminium by the ethanol extract of stem of Mucuna pruriens in 2 M H_2SO_4 *solution was studied by the weight loss method at temperature range of 301 K to 313 K. The percentage inhibition efficiency, %IE was found to increase with both the inhibitor concentration and temperature. The increase in % IE with rise in temperature is suggestive of chemical adsorption process. The values of* ΔG_{ads} *are all negative an indication of the spontaneity of the adsorption process and below -20 kJ* mol^{-1}. *Values of enthalpy of activation,* ΔH_{ads} *are positive and are lower than that of the blank, signifying inhibition effectiveness increases with increase in temperature. The data obtained there from best fitted the Freundlich, El-Awady, Temkin and Adejo-Ekwenchi isotherm. Due to conflicting figures obtained from the values of activation energy as to which of the mechanism of adsorption the inhibitor followed, the Adejo-Ekwenchi Isotherm was used to resolve the ambiguity and is chemisorption.*

Keywords: Corrosion inhibitor**,** *Mucuna pruriens,* Chemisorption, Adsorption Isotherm.

INTRODUCTION

Aluminium is a very significant metal in industry; this is due to its many excellent qualities including good thermal and electrical conductivities, high ductility, good corrosion resistance and low density. Aluminium is used widely in the aviation and automobile industry, household appliances, electronic devices and containers. It is able to resist corrosion as a result of its ability to form a natural oxide film on its surface in a wide variety of media [1-3]. However, under certain conditions the metal can be susceptible to corrosion. Corrosion of metals poses a major challenge in industry and its control is of extreme importance economically, environmentally, technically and aesthetically. Considering both the ecological and environmental reasons, the use of green inhibitors, which is found to be very effective in the control of corrosion, are imperative. Plant extracts which are organic inhibitors are known to be non-toxic and biodegradable, and are also inexpensive, renewable, and readily available. The presence of alkaloids, tannins, organic amino acids and organic dyes in plants has been found to be responsible for their good anti-corrosive ability. These organic compounds possess components, having atoms such as O, S, P, and N. [4,5], and their presence of the organic molecules of plant extracts make them to either adsorb on the metal surface thereby forming a protective layer on the metal or through the oxidising effect, it may cause the formation of a protective oxide film thereby creating a barrier which prevents access of corrosive agents to the metal surface [6,7].

In this work, the inhibitive properties of ethanol stem extract of *Mucuna pruriens* on corrosion reaction of aluminium in 2 M H_2SO_4 solution were investigated. Adsorption mechanisms were deduced through adsorption isotherms using data obtained there from.

MATERIALS AND METHODS

Materials: Aluminium wires were bought from the open market and taken to the Department of Mechanical Engineering, University of Agriculture Makurdi-Nigeria, where they were melted in a furnace and moulded into sheets of desired sizes. The moulded sheets were later mechanically press-cut into coupons of dimension of 2.1 cm x 2.1 cm and 0.35 cm each, with a tiny hole drilled at the edge of each coupon for suspension in the corrosive. The coupon surfaces were thoroughly polished to mirror finish using abrasive paper of different grades and then degreased in absolute ethanol (BDH Chemicals Limited, pool England), dried in acetone (BDH Chemicals Limited, pool England), and preserved in a desiccator [8]. A 2 M H_2SO_4 (BDH Chemicals Limited, pool England) solution was prepared which served as corrosive. All reagents used were of analytical grade, while doubly distilled water was used for the preparation of the solutions. 20 g of the ground *Mucuna pruriens* stem was soaked in 200 $cm^3$of absolute ethanol for 48 h, with occasional shaking and filtered, thereafter, using a cotton wool and the ethanol evaporated from the filtrate using thermostated water bath (BDH Chemicals Limited, pool England) maintained at 321 K to avoid any possible structural changes to the component of the extract. The residue obtained, there from was then preserved in a desiccator. Stock solutions containing 0.1, 0.2, 0.3, 0.4 and 0.5 g $dm^{3\ -1}$ in the 2 M H_2SO_4 were prepared from the extract [9-11].

Weight Loss Measurement Method: 50 mL of the 2 M H_2SO_4, which served as the corrosive, was taken in a 100 mL beaker and placed in an unstirred thermostated water bath maintained at 301 K. The initial weight of the coupon was taken using an ADAM Electric Weighing Balance (ADAM PW& PGW series; Max: 180g, d = 0.0001 g) [9]. The coupon was then wholly immersed in the corrosive with the aid of a thread tied to a retort stand and left for 8 h. At retrieval, the coupon was immediately dipped into a saturated solution of ammonium acetate (BDH Chemicals Limited, pool England) to quench the corrosion reaction, and carefully washed in absolute ethanol, rinsed in distilled water and then kept in the desiccator to cool to ambient temperature to a constant weight and re-weighed [12]. The weight loss of the coupon was obtained as the difference in the weight before and after the immersion. This procedure was repeated at 305 K, 309 K and 313 K which served as the blank at each of these temperatures. Triplicate results were used in each case and the mean value reported. The same procedure was repeated after the introduction of various concentrations of the extract into the corrosive maintained at the same temperatures.

The weight loss was calculated thus;

$$W_L = W_i - W_f \qquad (1)$$

where W_L is the weight loss of the coupon, W_i is the initial weight and W_f is the weight losses after retrieval. The percentage inhibition was then obtained through equation (2) [13,14].

$$\%IE = [1 - \left(\frac{W_{inh}}{W_{blank}}\right) X\ 100 \qquad (2)$$

where W_{inh} and W_{blank} are the weight losses in grams of the coupon in the presence and absence of the inhibitor, respectively.

The degree of surface coverage θ was obtained through by equation (3) [15].

$$\theta\ = 1 - W_{inh}/W_{blank} \qquad (3)$$

The corrosion rate of the metal was determined for the immersion period from weight loss through equation (4).

$$\text{Corrosion rate } (\text{mgcm}^{-2}\text{h}^{-1}) = WL/At \quad (4)$$

where WL is the weight loss in milligrams (mg), A the coupon surface area in cm^2 and t is the immersion time in hours [16].

RESULTS AND DISCUSSION

Effects of Concentration and Temperature on the Corrosion of Aluminium in Acid solution : Table1 shows the results of weight loss for aluminium corrosion in 2 M H_2SO_4 in the absence and presence of various concentrations of *Mucuna pruriens* stem extract at different temperatures. The decrease in weight loss in the presence of the extract in comparison to the blank signifies that *Mucuna pruriens* stem extract inhibited the corrosion of aluminium in the acid solution. The weight loss decreased with increase in the extract concentration, but increased with rise in temperature. The corrosion rate that was observed to be high in the blank came down considerably as the inhibitor was introduced.

Table 1. Weight loss (WL) and inhibition efficiency (%IE) for the inhibition of aluminium corrosion bystem extract of *Mucuna pruriens* at various temperatures.

Conc.	WL (g)				Corrosion Rate (gcm^{-2} h^{-1})			
g/dm^3	301K	305K	309K	313K	301K	305K	309K	313K
Blank	0.0084	0.0092	0.0194	0.0240	0.000100	0.000110	0.000230	0.000290
0.1	0.0082	0.0088	0.0167	0.0191	0.000099	0.000110	0.000200	0.000230
0.2	0.0080	0.0080	0.0123	0.0167	0.000096	0.000096	0.000150	0.000200
0.3	0.0069	0.0064	0.0111	0.0135	0.000083	0.000077	0.000130	0.000160
0.4	0.0065	0.0061	0.0091	0.0110	0.000078	0.000073	0.000100	0.000130
0.5	0.0061	0.0058	0.0086	0.0089	0.000073	0.000069	0.000100	0.000110

The evaluated inhibition efficiency % IE (Table 2) was found to increase with both increase in the inhibitor concentration and temperature. This increase in %IE with rise in temperature is suggestive of chemical adsorption (chemisorption) mechanism as the mechanism of adsorption of the inhibitor onto the surface of the metal [11]. The degree of surface coverage, θ (Table 2) increased with both inhibitor concentration and temperature. [12]. Table 2 reveals that as the inhibitor concentration was increased the corrosion rate decreased, while the corrosion rate increased with an increase in the temperature. [13,14].

Table 2 Degree of Surface Coverage (θ) and Corrosion rate(CR) gcm-1h^{-1}

Conc (g/dm^3)	IE %				Degree of Surface Coverage (θ)			
Temp.	301 K	305 K	309 K	313 K	301 K	305 K	309 K	313 K
0.1	2.38	4.35	13.92	20.41	0.0240	0.0435	0.1392	0.2042
0.2	4.76	13.04	36.60	30.42	0.0480	0.1304	0.3660	0.3042
0.3	17.86	30.44	42.78	43.75	0.1786	0.3043	0.4274	0.4373
0.4	22.62	33.70	52.58	54.17	0.2262	0.3369	0.5309	0.5417
0.5	27.38	36.96	56.67	62.90	0.2738	0.3696	0.5567	0.6292

To further demonstrate that the extract was adsorbed onto the metal surface for the inhibition process, a plot of inhibition efficiency against the logarithm of concentration was carried out at the 301 K. The s-shaped curve as depicted by figure 1 is an indication of formation of a protective barrier by the inhibitor molecules on the aluminium surface, and signifying inhibitive action of the extract [11].

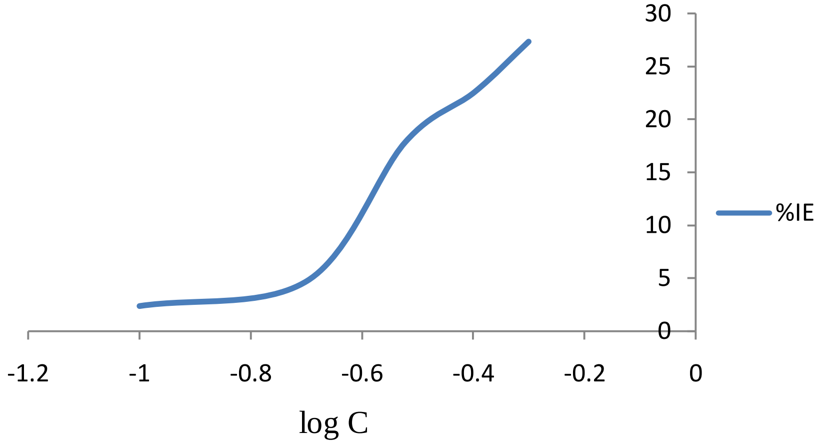

Figure 1. Relationship between %IE and log C of inhibitor at 301 K.

Activation energy and thermodynamic parameters for the inhibition process : Table 3 shows the values of activation energy, E_a and some thermodynamics parameters. The value of apparent activation energy, E_a was calculated using the Arrhenius-type equation as given by equation (5) [6].

$$CR = A_{exp}{}^{-Ea/RT} \qquad (5)$$

where CR is the corrosion rate of aluminium, A is Arrhenius constant or pre-exponential factor, R is the universal gas constant and T is absolute temperature. Linearization of equation (5) gives equation (6).

$$lnCR = lnA - \frac{Ea}{RT} \qquad (6)$$

A plot of $lnCR$ against $\frac{1}{T}$ gives a slope of $-\frac{Ea}{R}$, from where the values of apparent activation energy were evaluated (table 3). The apparent activation energy value for the blank was found to be higher than in the inhibited solution and it decreased with increase in the extract concentration. The decrease in Ea with increase in concentration is suggestive of chemisorption [17-19]. The values of heat of adsorption, Q_{ads} of *Mucuna pruriens* stem extract adsorption onto the aluminium surface were obtained through equation (7) [12].

$$\log\left(\frac{\theta}{1-\theta}\right) = logA + logK - Q_{ads}/2.303R\left(\frac{1}{T}\right) \qquad (7)$$

where θ is the degree of surface coverage, T is the temperature and A is temperature independent constant. Values of heat of adsorption were obtained from the slope ($-Q_{ads}/2.303R$) of a plot of $\log(\theta/1-\theta)$ against 1/T. Obtained values of Q_{ads} are all positive, indicative of endothermal nature of the dissolution process of the metal sample in the acid solution [9]. The apparent enthalpy of activation, ΔH_{ads} and entropy of activation, ΔS_{ads} values were obtained through the linearised transition-state theory equation given thus;

$$\ln\left(\frac{CR}{T}\right) = \ln\left(\frac{R}{Nh}\right) + \frac{\Delta S}{R} - \frac{\Delta H}{RT} \qquad (8)$$

where h is Planck's constant and N is Avogadro's number. A plot of $\ln\left(\frac{CR}{T}\right)$ versus $\frac{1}{T}$ will give a straight line and the slope is equal to $-\Delta H/R$ with intercept$\ln\left(\frac{R}{Nh}\right) + \frac{\Delta S}{R}$, and from where the values of ΔH and ΔS were calculated and are presented in table 3. The values of enthalpy of activation, ΔH_{ads} are positive and are lower than that of the blank, signifying inhibition effectiveness increases with increase in temperature. The average difference in value of the $E_a - \Delta H_{ads}$ (equation 9) was found to be 2.48 kJ mol^{-1}, almost same as the value of RT (2.55 kJ mol^{-1}), where T is the average of the temperatures (307K) at which the work was carried out. This implies that the corrosion process of this metal in the acid medium is a unimolecular reaction [13].

$$Ea - \Delta H_{ads} = RT. \qquad (9)$$

The negative values of entropy of activation, ΔS_{ads} show that the activated complex in the rate determining step represents an association, rather than dissociation [12], that is a decrease in disorderliness on going from reactants to the activated complex. And the value was observed to increase with increase in concentration, showing increase in disorderliness in that direction too. The values of free energy of adsorption of *Mucuna pruriens* stem onto the aluminium surface were calculated using the equation (10) [21].

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

where K = θ / [(1- θ) C], C is the inhibitor concentration and 55.5 the molar concentration of water. The values of ΔG are all negative, a clear indication that adsorption of the inhibitor onto the metal surface is spontaneous and the increase in values with rise in temperature shows that the spontaneity of the adsorption increased as temperature was raised. However, the values are below -20 kJ mol^{-1}, the threshold value for physical adsorption mechanism. Generally, values of ΔG_{ads} up to -20 kJ mol^{-1} are supportive of electrostatic interaction between charged molecules and a charged metal (which indicates physical adsorption), while those higher than -40 kJ mol^{-1} involves charged sharing or transfer from the inhibitor molecules to the metal surface to form a coordinate type of bond (which indicates chemisorption) [11].

Table 3. Values of activation energy, E_a, and thermodynamic parameters for extract of *Mucuna pruriens* stem as inhibitor for aluminium corrosion.

Conc. (g/dm^3)	E_a kJ/mol (kJ/mol)	Q_{ads}	$+\Delta H_{ads}$ (kJ/mol)	$-\Delta S_{ads}$ (J/mol)	$-\Delta G_{ads}$(kJ/mol) 301 K	305 K	309 K	313 K
Blank	74.26	72.19		82.55				
0.1	59.97	161.68	57.87	130.08	6.54	8.08	11.55	12.41
0.2	50.96	153.06	48.39	163.13	6.60	9.45	13.04	12.49
0.3	48.25	84.89	45.68	172.35	9.25	11.29	12.66	12.93
0.4	35.92	92.59	33.35	213.74	9.27	10.79	12.99	13.27
0.5	28.53	102.02	25.96	238.38	9.35	10.59	12.69	13.63

Adsorption Considerations: The inhibition of metal corrosion by organic additives has been attributed to their adsorption onto metal surface. The coefficient of determination, R^2, value is usually used to test the fitness of experimental data to an adsorption isotherm. The linearised forms of some isotherm equations are given below.

Langmuir $\frac{C}{\theta} = \frac{1}{K} + C$ (11)

Freundlich $\log\theta = \log K_F + n_f \log C$ (12)

Flory-Huggins $\log \frac{\theta}{C} = \log K + x\log[1 - \theta]$ (13)

Temkin $-2\alpha\theta = \ln KC$ (14)

Frumkin $\log\frac{\theta}{[(1 \ \theta)]C} = \log K + \frac{2a\theta}{2.303}$ (15)

El-Awady $\log \frac{\theta}{1-\theta} = \log K' + y\log[C]$ (16)

Sips $\log \frac{\theta}{1-\theta} = \log A + x\log[C]$ (17)

Adejo – Ekwenchi $\log\frac{1}{(1-\theta)} = \log K_{AE} + b\log[C]$ (18)

The data obtained fitted the El-Awardy, Freundlich, Temkin and Adejo-Ekwenchi adsorption isotherms, judging from the value of the coefficient of determination, R^2, being close to unity in each case (Table 4). From the El-Awardy isotherm, y<1 implies single inhibitor molecule occupies more than one active sites on the metal surface and y>1 means a formation of multilayer [13]. Values of y (Table 4) are 1.854, 1.674, 1.269, and 1.177 for 301 K, 305 K, 309 K and 313 K respectively, which are greater than unity implying formation of multilayer.

The parameter n of the Freundlich isotherm is related to intensity of adsorption and it varies with heterogeneity of the material [11], and its value is always positive, but not an integer, with typical value of 0.6 [12]. The average value of n is 1.15, a value far from 0.6, an indication that this adsorption process cannot be reasonably modelled by this isotherm, in spite of the seemly good values of R^2.

The adsorption process also followed the Temkin adsorption isotherm which is a feature of chemisorption [12]. The adherence of this adsorption process to Temkin adsorption isotherm is suggestive of molecular interaction within the adsorption layer species, and the negative values of 'a' are indication of repulsive interaction in the adsobed layer [10]. The values of ΔG_{ads} are all negative and below -20kJ/mol. The values of R^2 are good, but ΔG_{ads} values are very low for chemisorption. The repulsion might have accounted for the decrease in the values of ΔG_{ads}

The b parameter (intercept) of the Adejo-Ekwenchi isotherm is used to determine the mode of adsorption of an inhibitor on the metal surface, whether it follows the physical adsorption mechanism or the chemical adsorption mechanism, most especially if the activation energy, R^2 and the free energy of adsorption (ΔG_{ads}) cannot be used to due to conflicting figures [21,22]. Decrease in the value of *b* of the isotherm with rise in temperature indicates the mode of adsorption to be physisorption, while a fairly constant or an increase in its value with rise in temperature is a pointer towards chemisorption. From Table 4 the values of b increases with a corresponding increase in temperature, and therefore it can be concluded, unambiguously, that the mode of adorption is chemisorption.

Table 4.Values of parameters of linearised various adsorption isotherms for adsorption of the extract onto aluminium surface.

Isotherm	Temp(K)	R^2	Slope	Intercept	K_{ads}		ΔG_{ads} (KJ/mol)
El-Awardy						y	
	301	0.943	1.854	0.171	1.483	1.854	-11.039
	305	0.962	1.674	0.368	2.333	1.674	-12.335
	309	0.961	1.269	0.540	3.467	1.269	-13.515
	313	0.974	1.177	0.536	3.436	1.177	-13.666
Freundlich						n	
	301	0.948	1.639	-0.022	0.951	1.639	-9.927
	305	0.956	1.396	0.083	1.211	1.396	-10.672
	309	0.928	0.866	0.068	1.169	0.866	-10.721
	313	0.992	0.716	0.011	1.026	0.716	-10.520
Temkin						a	
	301	0.901	2.386	-0.942	0.114	-2.747	-4.617
	305	0.949	1.890	-1.032	0.093	-2.176	-4.163
	309	0.983	1.638	-1.246	0.057	-1.886	-2.959
	313	0.955	1.565	-1.246	0.057	-1.802	-2.997
Adejo-Ekwenchi						b	
	301	0.885	0.519	1.495	31.261	0.519	-18.669
	305	0.936	0.825	1.827	67.14	0.825	-20.855
	309	0.970	1.570	2.692	492.04	1.570	-26.247
	313	0.847	1.940	3.003	1006.9	1.940	-28.451

APPLICATIONS

The corrosion inhibition of aluminium by the ethanol extract of stem of Mucuna pruriens in 2 M H_2SO_4 solution was studied by the weight loss method at temperature range of 301 K to 313 K. From the results it was found that the percentage inhibition efficiency, %IE was found to increase with both the inhibitor concentration and temperature. The increase in % IE with rise in temperature is suggestive of chemical adsorption process. So it is useful for this purpose.

CONCLUSIONS

This work has shown that ethanol extract of stems of *Mucuna pruriens* inhibited the corrosion of Aluminium in 2 M H_2SO_4. The data obtained there from best fitted the Freundlich, El-Awady, Temkin and Adejo-Ekwenchi isotherm on the account of R^2. The ambiguity of the adsorption model was resolved using the Adejo-Ekwenchi Isotherm, to be chemisorption. As the adsorption has been resolved to be chemisorption, it can only be monolayer and not fit into El-Awady as given above. Therefore, the data best fitted the Temkin and Adejo-Ekwenchi isotherm models.

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