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Adsorption and Thermodynamics Study of the Inhibition of Corrosion of Mild Steel in H_2SO_4 Medium Using *Vernonia amygdalina*

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ABSTRACT

In this work, adsorption and thermodynamics study of the inhibition of corrosion of mild steel in H_2SO_4 medium using *Vernonia amygdalina* was carried out. The inhibitive and adsorptive properties of ethanol extract of *Vernonia amygdalina* for the corrosion of mild steel in 0.2 M H_2SO_4 solutions was investigated using weight loss technique. The result has proved that that the extract is a good inhibitor of corrosion of mild steel in H_2SO_4. The inhibition efficiencies ranged from 23.37% to 38.59% and from 22.45% to 35.78% at 303 and 323 K respectively. The inhibition efficiency of the extract decreased as temperature and time of immersion increased but increased with increase in concentration of extract. The adsorption of the inhibitor on surface of mild steel was found to be exothermic, spontaneous and consistent with the mechanism of physical adsorption as the value for heat of adsorption ranged from –2.12 to –4.87 $KJ \cdot mol^{-1}$. The adsorption data fitted well to Langmuir, Temkin, Frumkin and Flory-Huggins adsorption isotherms.

Keywords: Adsorption; Corrosion; Mild Steel; Inhibition; *Vernonia amygdalina*

1. Introduction

Mild steel is the most commonly used engineering material [1]. Oftentimes, mild steel is exposed to the attack of acid solutions during industrial processes such as acid cleaning, pickling and descaling. This results to easy corrosion of the mild steel, thereby necessitating the use of inhibitor [2-5]. There are numerous methods for controlling the corrosion of metals but the use of inhibitors is still one of the best methods of protecting metals against corrosion [6-8]. Many organic compounds are used as corrosion inhibitors for mild steel in acidic environments. Such compounds usually contain nitrogen, oxygen or sulphur in a conjugated system and function via adsorption of the molecules on the metal surface, creating a barrier to the corrodant attack [3,5,8,9]. However, because of the Sequel to toxic nature, high cost and increasing awareness and strict environmental regulations of some of these compounds, the use of natural product of plant origin as corrosion inhibitor is receiving attention [10, 11]. Corrosion inhibitors derived from plant extract are biodegradable and do not contain heavy metals or other toxic compounds [12].

Several researches have been done on the use of extract of plant as inhibitor for metals against corrosion in different aggressive media [13,14]. However, not much has been reported on the use of the extract of *Vernonia amygdalina* plant as inhibitor against corrosion of mild steel in H_2SO_4. *Vernonia amygdalina* is a member of the Asteraceae family which is a small shrub that grows in the tropical Africa. *Vernonia amygdalina* is also called bitter leaf because of its bitter taste. The leaves are used either as a vegetable (macerated leaves in soups) or aqueous extracts for treatment of various illnesses [15]. Therefore, the present work is aimed at investigating the potential of ethanol extract of *Vernonia amygdalina* leaves as corrosion inhibitor of mild steel in sulphuric acid.

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2. Materials and Methods

2.1. Materials Preparation

Mild steel of composition (wt%) Mn (0.6), P (0.36), C (0.15) and Si (0.03) and Fe (98.86) were used for the study. The sheet was cut to form different coupons of dimension, 4 × 3 × 0.1 cm. Each coupon was degreased by washing with ethanol, rinsed with acetone and allowed to dry in the air before preservation in a dessicator. All reagents used for the study were of analytical grade and double distilled water was used in this study.

2.2. Preparation of Plant Extract

Stock solutions of the plant extract were prepared by soaking known amount of the dried and ground leaves of *Vernonia amygdalina* in solution of ethanol. The sample was filtered after 48 hours and the filtrate was heated so as to remove ethanol from the sample. From this stock solution, test concentrations of 0.1, 0.2, 0.3, 0.4 and 0.5 g/l were prepared by diluting with 0.2 M H_2SO_4.

2.3. Chemical Analysis

Phytochemical analysis of the ethanol and aqueous extracts of *Vernonia amygdalina* was carried out according to the method reported by Onyeka and Nwabekwe [16].

2.4. Gravimetric Experiment

Mild steel coupon was weighed and immersed in 100 ml of the test solution in an open beaker. The beaker was placed into a water bath maintained at 303 K. Each sample of mild steel was withdrawn from the test solution after every 24 hours, washed with washing liquor, rinsed in acetone and dried in air before reweighing. The difference in weight after 120 hours was taken as the total weight loss. The experiment was repeated at 323 K. From the weight loss results, the inhibition efficiency (% I) of the inhibitor, degree of surface coverage (θ) and corrosion rates (*CR*) were determined using Equations (1)-(3) respectively.

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

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

$$\mathrm{CR}\left(\mathrm{g}\cdot\mathrm{cm}^{-2}\cdot\mathrm{h}^{-1}\right) = \frac{W}{At} \quad (3)$$

where W_1 and W_2 are the weight losses (g/dm^3) for mild steel in the presence and absence of inhibitor, A is the area of the mild steel coupon (cm^2), t is the time of immersion (hours) and W is the weight loss of mild steel after time t.

3. Results and Discussion

3.1. Phytochemical Analysis

The phytochemical constituents of aqueous and ethanol extract of *Vernonia amygdalina* are shown in **Table 1**. The result reveals that saponnin, tannin, alkaloids, cardiac glycoside, flavanoid, anthraquinone are present in the ethanol extract of *Vernonia amygdaliana* but absent in the aqueous extract of *Vernonia amygdalina*. This indicates that the inhibition efficiency of the extract is due to the presence of the phytochemical constituents [17].

3.2. Effects of Ethanol Extract of *Vernonia amygdalina* on the Corrosion of Mild Steel

The variation of weight loss of mild steel with time for the corrosion of mild steel in 0.2 M H_2SO_4 in the absence and presence of various concentration of ethanol extract of *Vernonia amygdalina* at 303 K is shown in **Figure 1**. The weight loss of mild steel in H_2SO_4 increased with increase in time of immersion, but decreased with increase in the concentration of ethanol extract of *Vernonia amygdalina*. This shows that ethanol extract of *Vernonia amygdalina* inhibited the corrosion of mild steel in H_2SO_4. Odiongenyi and his co-workers [17], in their study on corrosion inhibition and adsorption properties of ethanol extract of *Vernonia amygdalina* for the corrosion of mild steel in H_2SO_4, obtained similar result.

The corrosion rate of mild steel in the absence and presence of *Vernonia amygdalina* extract and inhibition efficiencies of various concentrations of *Vernonia amygdalina* extract is presented in **Table 2**. The result shows that the rate of corrosion of mild steel decreased as the concentration of ethanol extract of *Vernonia amygdalina* increased but increased with increase in temperature, while the inhibition efficiency of the extract of *Vernonia amygdalina* decreased with increase in temperature, indicating that the adsorption of extract of *Vernonia amygdalina* on mild steel surface is physical adsorption as reported by other researchers [3,18-21].

3.3. Effect of Temperature

The effect of temperature on the rate of corrosion of mild steel in H_2SO_4 containing various concentrations of ethanol extract of *Vernonia amygdalina* was investigated us ing Arrhenius equation as given in Equation (4) [4,22-24].

Table 1. Phytochemical constituents of aqueous and ethanol extracts of *Vernonia amygdalina*.

Phytochemicals	Aqueous extract	Ethanol extract
Tannins	-	+++
Saponins	-	++
Alkaloids	-	++
Cardiac glycosides	-	++
Flavanoid	-	+
Anthraquinones	-	+++

Table 2. Values of corrosion rate and inhibition efficiency at various temperatures in the absence and presence of ethanol extract of vernonia amygdalina.

Concentration (g/l)	Corrosion rate (g·h^{-1}·cm^{-2}) ×10^{-7}		Inhibition efficiency (%)	
	303 K	323 K	303 K	323 K
Blank	6.0	6.8		
0.1	5.0	5.3	23.37	22.45
0.2	4.6	4.9	27.72	26.53
0.3	4.4	4.7	31.52	30.63
0.4	4.1	4.4	35.33	33.71
0.5	3.9	4.2	38.59	35.78

$$CR = A\exp\left(-Ea/RT\right) \quad (4)$$

where CR is the corrosion rate of mild steel, A is Arrhenius or pre-exponential factor, Ea is the activation energy, R is the gas constant and T is the temperature. the corrosion rates of mild steel at 303 K (T_1) and 323 K (T_2) denoted as CR_1 and CR_2. Equation (4) becomes

$$\log\left(\frac{CR_2}{CR_1}\right) = \frac{Ea}{2.303R}\left(1/T_1 - 1/T_2\right) \quad (5)$$

Ea values, calculated from Equation (5), are presented in **Table 3**. These values ranged from 2.37 to 3.70 $KJ \cdot mol^{-1}$ and are lower than the threshold value of 80 $KJ \cdot mol^{-1}$ required for chemical adsorption. This shows that the adsorption of ethanol extract of *Vernonia amygdalina* on mild steel surface is physical adsorption [2,4,21].

3.4. Thermodynamic/Adsorption Parameters

The heat of adsorption Q_{ads} of ethanol extract of *Vernonia amygdalina* on the surface of mild steel was calculated using Equation (6) [4,22,23,25,26].

$$Q_{ads} = 2.303R\left[\log\left(\frac{\theta_2}{1-\theta_2}\right) - \log\left(\frac{\theta_1}{1-\theta_1}\right)\right] \times \frac{(T_1 \times T_2)}{(T_2 - T_1)} KJ \cdot mol^{-1} \quad (6)$$

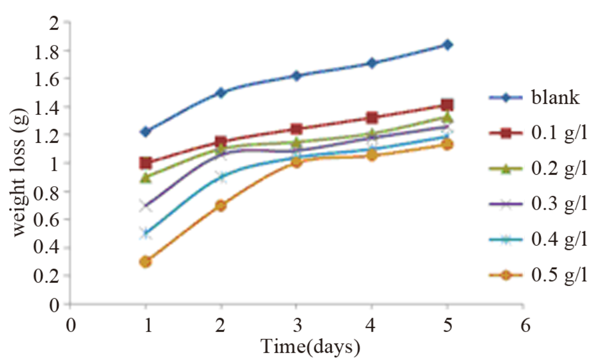

Figure 1. Variation of weight loss with different concentrations of *Vernonia amygdalina* extract for the corrosion of mild steel in 0.2 M H_2SO_4 at 303 K.

Table 3. Thermodynamic parameters for adsorption of ethanol extract of *Vernonia amygdalina* on surface of mild steel.

Concentration (g/l)	Activation energy ($KJ \cdot mol^{-1}$)	Heat of adsorption ($KJ \cdot mol^{-1}$)
Blank	5.20	
0.1	2.42	–2.12
0.2	2.63	–2.44
0.3	2.74	–2.69
0.4	2.94	–2.90
0.5	3.70	–4.87

where R is the gas constant, θ_1 and θ_2 are the degree of surface coverage at temperatures, T_1 and T_2 respectively. Calculated values of activation energy and heat of adsorption are shown in **Table 3**. The values ranged from –2.12 to –4.87 $KJ \cdot mol^{-1}$, indicating that the adsorption of ethanol extract of *Vernonia amygdalina* on mild steel surface is exothermic [3,18,19,26].

Data obtained for the degree of surface coverage were used for the evaluation of different adsorption isotherms (Langmuir, Frumkin, Temkin and Flory-Huggins).

3.4.1. Langmuir Isotherm

Langmuir adsorption isotherm is expressed according to Equation (7) [27,28].

$$\frac{C}{\theta} = \frac{1}{K} + C \quad (7)$$

where C is the concentration of the inhibitor, K is the adsorption equilibrium constant and θ is degree of surface coverage of the inhibitor. Taking logarithm of both sides of Equation (7) yields Equation (8).

$$\log\left(\frac{C}{\theta}\right) = \log C - \log K \quad (8)$$

Plotting $\log\left(\frac{C}{\theta}\right)$ against $\log C$ gave a linear relationship as shown in **Figure 2**. The parameters of Langmuir isotherm are presented in **Table 4**. The R^2 values of 0.998 and 0.999 indicate strong adherence to Langmuir adsorption isotherm [29]. The application of Langmuir isotherm to the adsorption of extract of *Vernonia amygdalina* on surface of mild steel indicated that there is no interaction between the adsorbate and adsorbent [30].

3.4.2. Temkin Isotherm

For Temkin adsorption isotherm, the degree of surface

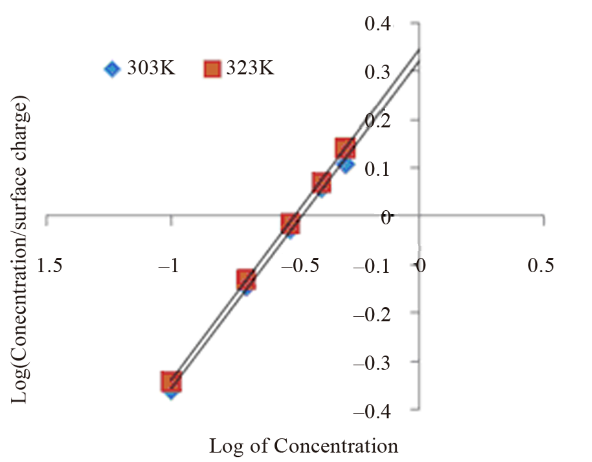

Figure 2. Langmuir isotherm for adsorption of ethanol extract of *Vernonia amygdalina* on mild steel surface.

Table 4. Adsorption parameters for adsorption of ethanol extract of *Vernonia amygdalina* on mild steel surface.

Isotherm	Temperature	R^2	log K	ΔG_{ads} KJ/mol	
Langmuir	303 K	0.998	−0.32	−8.25	
	323 K	0.999	−0.35	−8.64	
		R^2	log K	ΔG_{ads} KJ/mol	α
Frumkin	303 K	0.984	−1.29	−2.67	3.23
	323 K	0.995	−1.33	−2.55	3.52
		R^2	log K	ΔG_{ads} KJ/mol	a
Temkin	303 K	0.973	2.004	−21.75	−5.21
	323 K	0.993	2.069	−23.58	−5.7
		R^2	log K	ΔG_{ads} KJ/mol	x
Flory-Huggins	303 K	0.984	−1.29	−2.67	4.67
	323 K	0.995	−1.33	−2.55	5.51

coverage (θ) is related to inhibitor concentration (C) according to Equation (9) [27,31,32].

$$\exp(-2a\theta) = KC \quad (9)$$

where K is the adsorption equilibrium constant and a, is the attractive parameter. Rearranging and taking logarithm of both sides of Equation (9) gives Equation (10).

$$\theta = \frac{-2.303\log K}{2a} - \frac{2.303\log C}{2a} \quad (10)$$

Plots of θ against log$C$, as presented in **Figure 3**, gave linear relationship, which shows that adsorption data fitted Temkin adsorption isotherm. Adsorption parameters obtained from Temkin adsorption isotherms are recorded in **Table 4**. The values of attractive parameter (a) are negative in all cases, indicating that repulsion exists in the adsorption layer.

3.4.3. Flory-Huggins Isotherm

Flory-Huggins adsorption isotherm can be expressed according to Equation (11) [33].

$$\log\left(\frac{\theta}{C}\right) = \log K + x\log(1-\theta) \quad (11)$$

where x is the size parameter and is a measure of the number of adsorbed water molecules substituted by a given inhibitor molecule. The plots of $\log\left(\frac{\theta}{C}\right)$ against $\log(1-\theta)$ are shown in **Figure 4** which the data conformed to Flory-Huggins isotherm. The values of the size parameter x are positive as shown in **Table 4**. This indicates that the adsorbed species of ethanol extract of *Vernonia amygdalina* is bulky since it could displace more than one water molecule from the mild steel surface.

3.4.4. Frumkin Isotherm

Frumkin adsorption isotherm is given by Equation (12) [4,27].

$$\log\left\{[C]\times\left(\frac{\theta}{1-\theta}\right)\right\} = 2.303\log K + 2\alpha\theta \quad (12)$$

where K is the adsorption-desorption constant and α is the lateral interaction term describing the interaction in adsorbed layer. Plots of $\log\{[C]\times(\theta/1-\theta)\}$ versus θ as presented in **Figure 5** were linear which shows the applicability of Frumkin isotherm. The values for Frumkin adsorption parameters were recorded in **Table 4**. This shows that values of the adsorption parameter α are positive suggesting the attractive behavior of the inhibitor on the surface of mild steel [11,34].

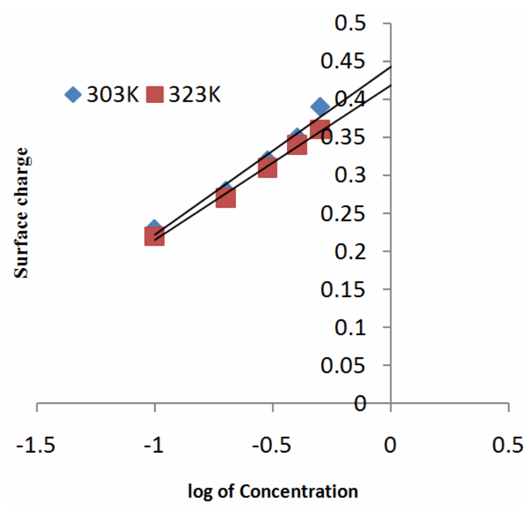

Figure 3. Temkin isotherm for adsorption of ethanol extract of *Vernonia amygdalina* on the surface of mild steel.

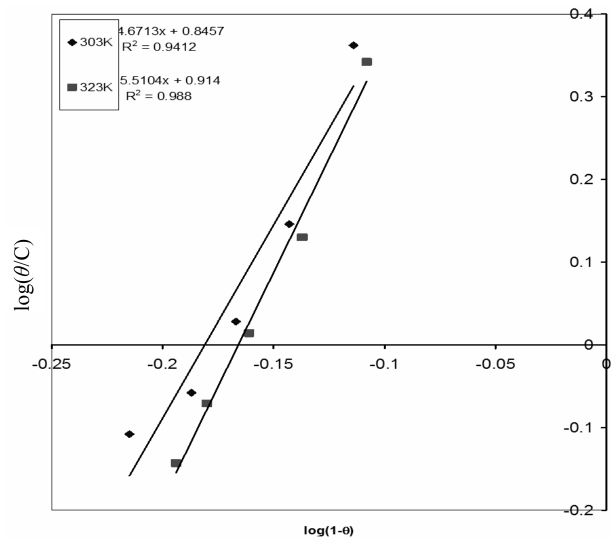

Figure 4. Flory-Huggins isotherm for adsorption of ethanol extract of *Vernonia amgydalina* on the mild steel surface.

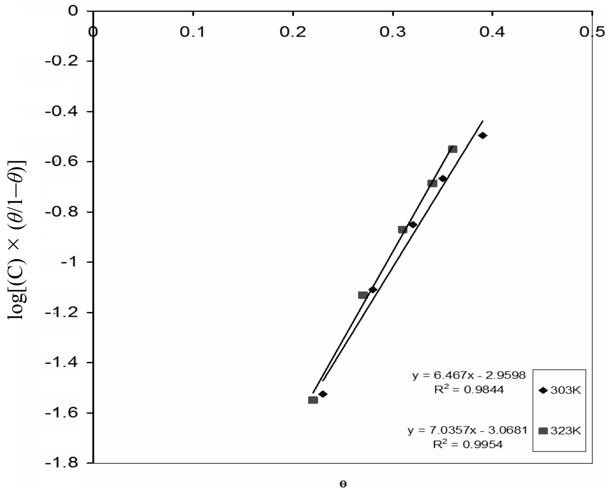

Figure 5. Frumkin isotherm for adsorption of ethanol extract of *Vernonia amgydalina* on the mild steel surface.

3.5. Free Energy of Adsorption

The equilibrium constant of adsorption of ethanol extract of *Vernonia amygdalina* on the surface of mild steel is related to the free energy of adsorption (ΔG_{ads}) according to Equation (13) [26,35-37].

$$\Delta G_{ads} = -2.303RT\log(55.5K) \qquad (13)$$

where R is the gas constant and T is the temperature. The free energy of adsorption was calculated from values of K obtained from Langmuir, Temkin, Flory-Huggins and Frumkin according to Equation (13) and is recorded in **Table 4**. The results show that free energy of adsorption ΔG_{ads} are negative and less than the threshold value of -40 KJ·mol^{-1} required for chemical adsorption, indicating that adsorption of ethanol extract of *Vernonia amygdalina* on mild steel surface is spontaneous and occurred according to the mechanism of physical adsorption [26, 38,39].

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