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# Adsorption and adsorption isotherm: application to corrosion inhibition studies of mild steel in 2 M HCl

**F. E. Abeng**[1,2,*], **V. D. Idim**[2]**, O. E. Obono**[1] and **T. O. Magu**[1]

[1]Corrosion and Electrochemistry Research Group, Department of Pure and Applied Chemistry, Universityn of Calabar, PMB 1115, Calabar, Nigeria

[2]Department of Chemical sciences, Cross River university of technology, P.M.B 1123, Calabar, Nigeria

*E-mail address: fidelisabeng@yahoo.com

*Tel: +2348035664813

**ABSTRACT**

Ethanollic extract of Phyllanthus amarus (EEPA) was tested as corrosion inhibitor for mild steel in 2 M HCl solution using gravimetric and gasometric methods at 303, 313, 323 K and 333 K. The results revealed that the corrosion rate increases with temperature and time. Addition of the concentration of ethanollic extract of Phyllanthus amarus to the corroding environment lowered the corrosion rate of mild steel and increased inhibition efficiency (IE %) of ethanollic extract. Inhibition efficiency was found to decreased with temperature. Fitting of the experimental data to the Arrhenius and Transition state equations revealed that the Organic constituents of the extract were physically adsorbed on the corroding surface of the steel and adsorption of ethanollic extract of Phyllanthus amarus (EEPA) on mild steel surface was found to obey the Langmuir adsorption isotherm and Freundlich adsorption isotherm and sign of the Gibb's fee energy of the adsorption obtained suggested that inhibitor molecules have been spontaneously adsorbed onto the mild steel surface. Positive values obtained for enthalpy change indicated that the adsorption of inhibitor is endothermic.

***Keywords*****:** inhibition mechanism, Adsorption isotherm, mild steel, thermodynamic parameters

## 1. INTRODUCTION

Adsorption is the attachment of substance on the surface of a solid or liquid. Thus adsobate are substances that attach onto surfaces while adsorbent is the solid to which it is attached. They are two types of adsorption, the physical adsorption (Physi-sorption) and chemical adsorption (chemisorptions). In physi-sorption, molecules or atoms are attach to surfaces via van der waals interaction between the adsorbate and adsorbent, van der waals interaction have a long range but weaker forces, at this process the attachment is loss and separation could be made. By thermodynamic evaluation of adsorption, stipulated values in the range of -20 KJ $mol^{-1}$. The enthalpy of physi-sorption can be measured by monitoring the rise in temperature of a sample. Thus this small enthalpy change could lead to bond breaking. Whereas in chemisorptions, molecules or atoms stick to the surfaces by forming covalent bond and tend to find sites that maximize their coordination number with the adsorbent. The enthalpy of chemisorptions is very much greater than that of physi-sorption because it's values is in the region of -40 KJ $mol^{-1}$ and above [1,2]. Chemical adsorption must be exothermic and spontaneous that is

$$\Delta G < 0 \tag{1}$$

At this point the translational freedom of the adsorbate is reduced when it is adsorbed and at such

$\Delta S$ and $\Delta H$ becomes negative

$$\Delta G = \Delta H + T\Delta S \tag{2}$$

where: $\Delta G$ is the change in free energy, $\Delta H$ is the change in enthalpy, $\Delta S$ is the change in entropy and T is the absolute temperature. The free energy in equ. 2 also becomes negative which is the same as equ. 1, this process is exothermic. Exception may occur if the adsorbate (molecules) dissociates and has high translational mobility on the surface. Then at that process $\Delta S$ is positive.

### 1. 1. Adsorption isotherms

Adsorption isotherms is the a model use in representing the relationship between the amount of adsorbate (molecules) adsorbed by the given amount of adsorbent (steel surface) at constant temperature, many adsorption isotherms have been derived to explain the relationship between the surface coverage $\theta$ and the pressure of the adsorbed molecules [1-3] In this study two distinct isotherms namely, Langmuir, and Freundlich isotherms shall be apply in the study of corrosion inhibition.

### 1. 2. Corrosion inhibition

Corrosion inhibition is the process whereby metals are control or prevented from been corroded through addition of chemical substances such as inhibitors, when this chemical substance are added in a small concentration into the environment where the metal reside it decrease the rate of attack of the metal. Inhibitors are of two types; the inorganic and organic inhibitors. Most of this inhibitors are toxic to the environment, expensive and not accessible. But this present study is focus towards ecofriendly, cheap and accessible inhibitors for the control of corrosion of metals. Many authors have reported on plants extracts as corrosion inhibitors [4-11], but non have been reported on ethanolic extract of *Phyllanthus amarus*. Some plants extracts contain a mixture of polar functional group and thus possess multiple active centre's. Generally the inhibitive effect of plants extract is attributed to the adsorption of organic substances on the metal surface by blocking active sites or forming a protective barrier on steel surfaces [12].

## 2. MATERIALS AND METHODS

The mild steel sheets used in this present study have the composition as those reported previously in [5,8]. The test coupons were prepared, degreased and cleaned as previously described by [5]. All chemical used were of Annular grade.

### 2. 1. Preparation of plant extracts

**Figure 1.** Sample of *Phyllanthus amarus* plant

The sample of *Phyllanthus amarus* plant was obtained within the premises of the department of Pure and Applied Chemistry, University of Calabar, Nigeria and identify in the Department on Botanic, University of Calabar. The plant leaves and stems were dried in an memmert ovun at 50 °C and grounded to powder form, extracted in a soxhlet extractor using ethanol as a solvent. The sample of the plant is shown in Figure 1 The clear dark brown ethanollic concentrated solution was dried under vacuum to get a semi solid liquid [5], 10 g of semisolid liquid that is ethanollic extract was dissolved in 2 M HCl solution and kept for 24 hours. The resulting solution were filtered and stored. From the stock solution (10 g /L ) inhibitor test solutions of concentration; 0.1, 0.5, 1.0, 2.0 and 4.0 g /L were prepared using serial dilution method ($C_1V_1 = C_2V_2$). These solutions were then used for the corrosion test.

### 2. 2. Gravimetric measurements

Mild steel specimens were first weighed using a digital weighing balance ADAM[R] and suspended in the test solution (2 M HCl) with and without inhibitor by means of glass rod at room temperature (303 K). Each mild steel specimen was withdrawn from test solution at one hour interval washed in distilled water for several times degreased in ethanol and dried in acetone. Further drying of the mild steel specimen was done by means of a dryer and then reweighed in order to determine the weight loss of the mild steel (coupon). The corrosion rate (CR) was calculated by using equ. 3 [5].

$$CR = \frac{WL}{At} \times 1000 \text{ (Mg cm}^{-2}\text{ hr}^{-1}\text{)} \tag{3}$$

where: WL is weight loss in mg, A is the specimen surface area, *t* the immersion period in hours (5hrs) from the corrosion rate, the surface coverage (θ) and inhibition efficiencies (IE %) were determined using equ. 4 and 5 respectively [9].

$$\theta = \frac{CR_{blank} - CR_{inh}}{CR_{blank}} \tag{4}$$

$$IE\ \% = \frac{CR\ blank - CR\ inh}{CR\ blank} \times 100 \tag{5}$$

where: $CR_{blank}$ and $CR_{inh}$ are the corrosion rate in absence and presence of the inhibiting molecules respectively.

### 2. 3. Gasometric mearsurement

Gasometric method were carried out at 303, 313, 323 and 333 K as described in [10-14]. The rate of evolution of the gas ($V_H$) is determined from the slope of the graph of volume of gas evolved (V) versus time (t) and the degree of surface coverage (θ) and inhibition efficiency (IE %) were calculated using equation 6 and 7 respectively.

$$\theta = 1 - \frac{V1Ht}{V0Ht} \tag{6}$$

$$IE\% = \left(1 - \frac{V1Ht}{V0Ht}\right) \text{ x } 100 \tag{7}$$

where $V_{1Ht}$ is the volume of hydrogen evolved at time t for inhibited solution and $V_{0Ht}$ is the volume of hydrogen evolved at time t for uninhibited solution.

## 3. RESULTS AND DISCUSSION

We have employed gravimetric and gasometric techniques to study the corrosion inhibition of mild steel in 2 M HCl solution in the absence and presence of ethanollic extract of *Phyllanthus amarus* (EEPA)

### 3. 1. Gravimetric measurement

The results of gravimetric measurements for mild steel in acidic media in the absence and presence of inhibitor concentration of ethanollic extract of *Phyllanthus amarus* (EEPA) at 303 K are listed in Table 1.

**Table 1.** Calculated values of corrosion rate, inhibition efficiency, rate constant and half life for mild steel coupon 2 M HCl solution containing ethanollic extract of *Phyllanthus amarus* (EEPA)

| System | Con g/L | Corr Rate mgcm$^{-3}$ hr$^{-1}$ | IE % | Rate const. (hr$^{-1}$) × $10^{-3}$ | Half life |
|---|---|---|---|---|---|
| | Blank | 0.468 | | 3.39 | 0.20 |
| | 0.1 | 0.292 | 37.9 | 2.07 | 0.46 |
| EEPA | 0.5 | 0.163 | 65.7 | 1.17 | 0.59 |
| | 1.0 | 0.161 | 66.4 | 1.12 | 0.61 |
| | 2.0 | 0.150 | 67.9 | 1.08 | 0.64 |
| | 4.0 | 0.014 | 96.8 | 0.10 | 6.93 |

According to these results and Figure. 1 weight losses increases with increased in time but decreased with increase in concentration of EEPA. The decrease is due to the inhibitive effects of EEPA [14-16]. The result also revealed that corrosion rate decreases with increase in the concentration of the EEPA [17-19]. The ethanollic extract show a significant inhibitive effect on mild steel in HCl solution that reaches up to 96.8 % at concentration of 4.0 g/L as shown in Table. 1 [20]. In the present study of corrosion of mild steel in HCl solution, weight at time t (after post treatment of coupons) is designated $w_f$. When In $(w_f/w_o)$ was plotted against time (in hour), linear variation was observed in Figure 3 which confirms a first order reaction kinetics. Formulated as [1-3]

$$\text{In}\,[w_f/w_o] = -kt \quad (8)$$

where: $w_o$ is the initial weight before immersion. k is the rate constant and t is time.
The values of the rate constant k obtained from slope of the plot in Figure 3 are presented in Table 1.

According to the results obtained, the rate constant decreases with increase in EEPA concentration. The half life values $t_{1/2}$ of the metal in the test solution were calculated from the constant values using the equation 9.

$$t_{½} = \frac{0.693}{K} \tag{9}$$

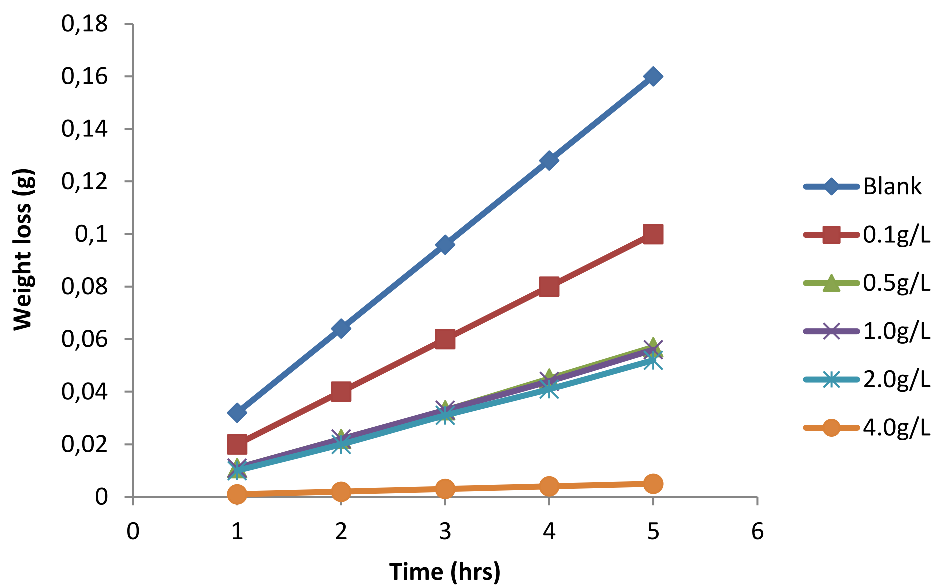


**Figure 2.** Variation of weight loss with time for the corrosion of mild steel in solution of HCl containing various concentrations of ethanollic extact of *Phyllanthus amarus* at 303 K

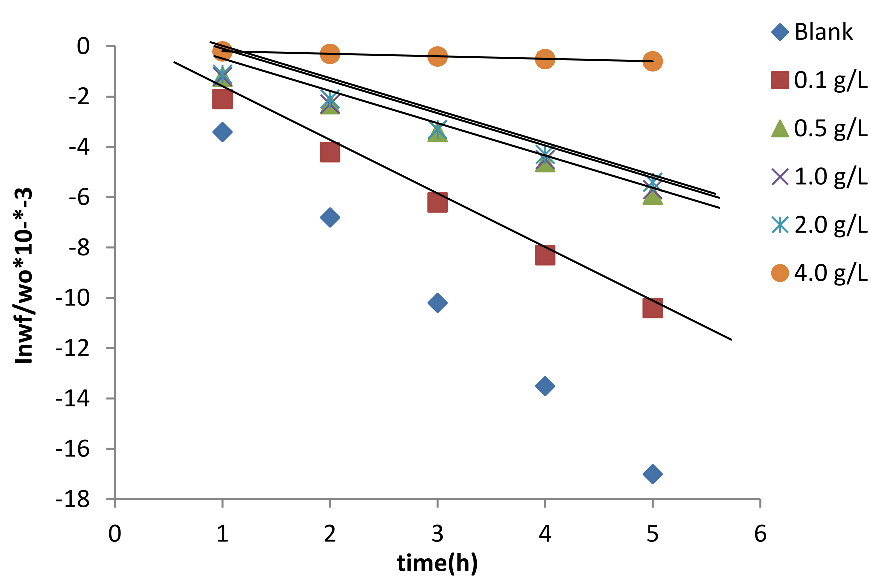


**Figure 3.** Variation of In($w_f$/$w_o$) with time for the corrosion of mild steel in 2 M HCl containing Various concentration of EEPA at 303 K.

And the deduced data are also presented in Table 1. The half life values were observed to increase with increase in concentration of the EEPA, indicating decrease in the dissolution rate of the metal in the solution with increase in EEPA concentration and also is an indication of prolonging the life span the metal [14].

### 3. 2. Gasometric results

**Table 2.** Values of inhibition Efficiency (IE %) and corrosion rate (CR) at 303, 313, 323 K and 333 K

| Con g/L | IE % 303 K | IE % 313 K | IE % 323 K | IE % 333 K | CR ($cm^3$/min) 303 K | CR ($cm^3$/min) 313 K | CR ($cm^3$/min) 323 K | CR ($cm^3$/min) 333 K |
|---|---|---|---|---|---|---|---|---|
| 0.0 | | | | | 0.0153 | 0.0187 | 0.0327 | 0.0380 |
| 0.1 | 41.2 | 35.8 | 44.9 | 20.5 | 0.0090 | 0.0120 | 0.0180 | 0.0350 |
| 0.5 | 47.7 | 41.1 | 48.1 | 31.6 | 0.0080 | 0.0110 | 0.0170 | 0.0260 |
| 1 | 48.3 | 51.9 | 57.2 | 47.4 | 0.0079 | 0.0090 | 0.0140 | 0.0200 |
| 2 | 54.3 | 73.3 | 59.6 | 52.6 | 0.0070 | 0.0050 | 0.0132 | 0.0180 |
| 4 | 67.3 | 83.9 | 66.4 | 68.4 | 0.0050 | 0.0030 | 0.0110 | 0.0120 |

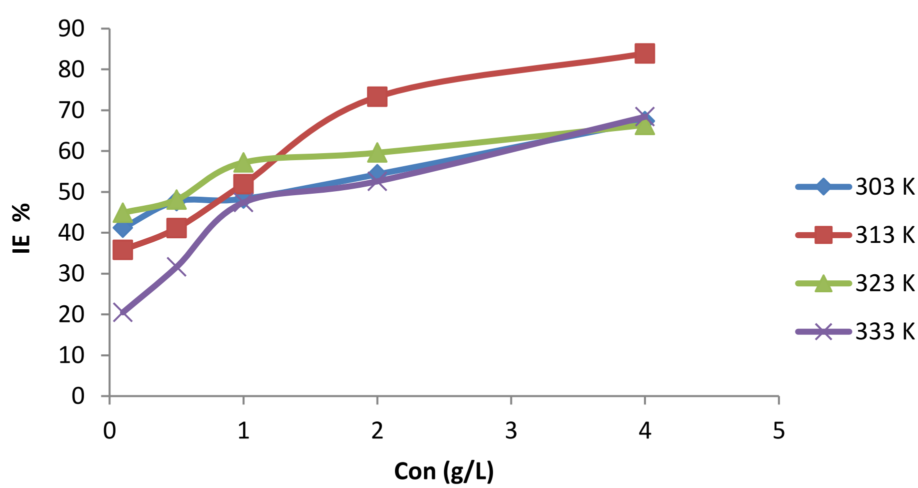


**Figure 4.** Variation of inhibition efficiency against different concentration of EEPA in 2 M HCl at 303, 313, 323 K and 333 K

Table 2, shows values of corrosion rate (CR) and the inhibition efficiency (% I) of ethanollic extract on mild steel in 2 M HCl at 303 313, 323 K and 333 K the results obtained show that the corrosion rate of mild steel in 2 M HCl decreases as the concentration of ethanollic extract of *Phyllanthus amarus* (EEPA) increases with increased temperature while inhibition efficiency increase with concentration of EEPA and decreased with increased in temperature.

Figure 4, shows the variation of inhibition efficiency against the different concentration of EEPA at both 303, 313, 323 K and 333 K. The significant difference between values of inhibition efficiency of EEPA obtained at the studied temperature suggested that the mechanism of adsorption of inhibitor on mild steel surface is by physical adsorption. For a physical adsorption mechanism, inhibition efficiency of the inhibitor increases with decrease in temperature which mean physical adsorption mechanism occurred on the mild steel surface [21-22]. Values of inhibition efficiency obtained at 303 K from gravimetric and gasometric method are in agreement.

### 3. 3. Effect of temperature

In order to study the effect of temperature on the corrosion reaction of mild steel in the presence of EEPA as inhibitor, Arrhenius- type relationship between the corrosion rate (CR) of mild steel in acidic media and temperature (T) as often expressed by Arrhenius equation used to determine the activation energies ($E_a$).

$$\log CR = \log A - \frac{Ea}{2.303RT} \qquad (10)$$

where A is Arrhenius constant, $E_a$ is activation energy ,R is the gas constant T is absolute temperature and CR is corrosion rate [13]. The variation of logarithm of corrosion rate with reciprocal of absolute temperature is shown in Figure 5 for mild steel corrosion in 2 M HCl containing EEPA.

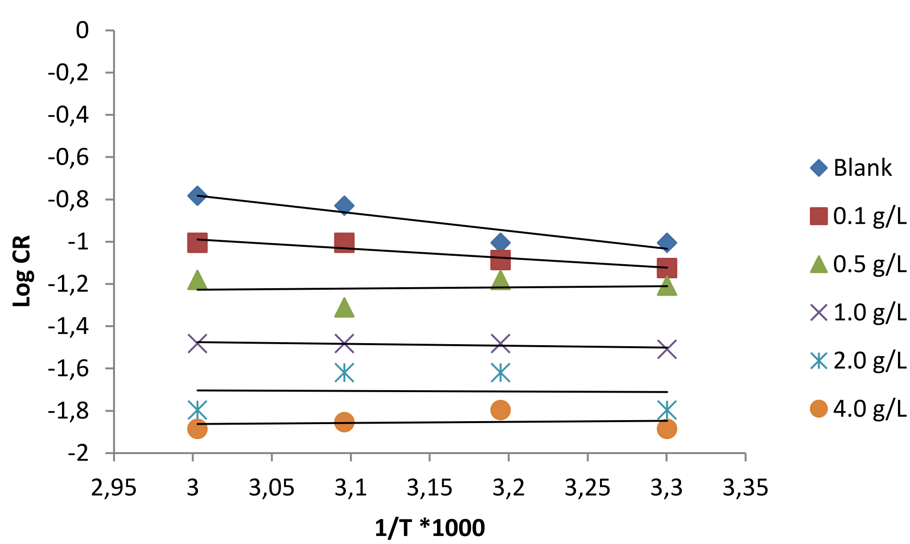


**Figure 5.** Variation logarithm of corrosion rate with reciprocal of absolute temperature for mild steel corrosion in 2 M HCl containing EEPA.

The calculated values of $E_a$ are given in Table 3. Addition of the EEPA can be seen to increase $E_a$ for the corrosion reaction, implying that the extract would be more effective at lower temperature, which is in agreement with the observed trend of inhibition efficiency with temperature as well as the proposed physisorption mechanism for the adsorption of the plant extract [13,12].

**Table 3.** Some thermodynamic parameters for the adsorption of ethanol extract of *Phyllanthus amarus* (EEPA) on mild steel surface.

| Con g/L | Ea $KJmol^{-1}$ | $\Delta H^0$ $KJmol^{-1}$ | $\Delta S^0$ $Jmol^{-1}K^{-1}$ | $R^2$ |
|---|---|---|---|---|
| Blank | 27.32 | 27.26 | -8.21 | 0.94 |
| 0.1 | 36.86 | 36.86 | -35.00 | 0.94 |
| 0.5 | 32.24 | 32.89 | -21.06 | 0.98 |
| 1.0 | 23.4 | 23.2 | -10.62 | 0.87 |
| 2.0 | 28.99 | 37.05 | -32.09 | 0.94 |
| 4.0 | 37.58 | 47.79 | -63.66 | 0.98 |

**Table 4.** Langmuir and Freundlich adsorption parameters for Ethanol Extract of *Phyllanthus amarus* at 303, 313, 323 K and 333 K.

| Isotherms | Temperature K | slope | $b_{ads}$ | $R^2$ | $\Delta G^0$ $KJmom^{-1}$ |
|---|---|---|---|---|---|
| | 303 | 1.42 | 1.98 | 0.96 | -11.84 |
| Langmuir | 313 | 1.08 | 1.85 | 0.97 | -12.05 |
| | 323 | 1.75 | 6.13 | 0.98 | -15.65 |
| | 333 | 2.-04 | 1.74 | 0.98 | -12.65 |
| | | | | | |
| Freundlich | 303 | 0.33 | 0.63 | 0.97 | -8.95 |
| | 313 | 1.25 | 0.14 | 0.98 | -5.33 |
| | 333 | 0.13 | 0.40 | 0.87 | -8.58 |
| | | | | | |

### 3. 4. Thermodynamic and adsorption considerations

Thermodynamic parameters such as enthalpy change $\Delta H_{ads}$ and entropy change $\Delta S$ were obtained from the Eyring transition state equation [13,15]:

$$CR = \frac{RT}{Nh} exp\left(\frac{\Delta S}{R}\right) \exp\left(-\frac{\Delta H}{RT}\right) \tag{9}$$

where: R is molar gas constant T is absolute temperature N is Avogadro's number h is Planck constant [15].

Straight lines were obtained from the Erying plots in Figure.6 with slope $\Delta H^*/R$ and intercept $\left[In\left(\frac{R}{NAh}\right) + \Delta H*/R\right]$. The calculated values of $\Delta H^0_{ads}$ and $\Delta S^0$ obtained from these plots are also given in Table. 3 the positive values of $\Delta H^0_{ads}$ both in absence and presence of inhibitor reflect the endothermic nature of the mild steel dissolution process, supporting proposed inhibition mechanism. Large negative values of entropy imply that the activation complex in the rate determining step represents an association rather than a dissociation step, meaning that decrease in the disordering takes place on going from reactants to the activated complex [13].

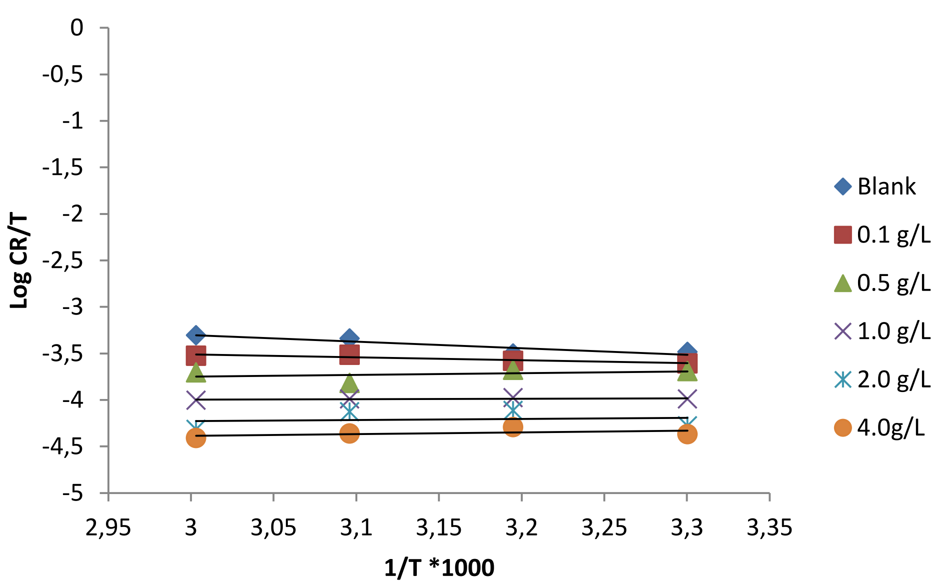


**Figure 6.** Eyring plots for mild steel corrosion in 2 M HCl without and with EEPA.

To describe the adsorption behaviour of the extract, several adsorption isotherms have been tested and best results judged by the correlation coefficient $R^2$, the Langmuir and Freundlich kinetic thermodynamic model fits the experimental data well. Langmuir and Freundlich isotherms are given by equ. 10 and 11. By the application of this isotherms, the free energy of adsorption $\Delta G_{ads}$ of ethanollic extract of *Phyllanthus amarus* (EEPA) on the mild steel surface were calculated from the plot of the isotherms in Figure 7 and 8. Using equ. 12 the calculated values of $\Delta G_{ads}$ are listed in Table. 4 these values are negative and are -11.84 KJ/mol at 303 K and -12.65 KJ/mol at 333 K for the Langmuir isotherm plot while for Freundlich isotherm are -8.95 KJ/mol at 303 K and -8.58 KJ/mol the values obtained indicated adsorption of EEPA to be spontaneous and occur as physical adsorption mechanism. Generally, values of $\Delta G_{ads}$ up to -20 KJ/mol signifies physical adsorption while values more than -40 KJ/mol signifies chemical adsorption [23].

$$\frac{C}{\theta} = \frac{1}{bads} + C \tag{10}$$

$$\frac{\theta}{1-\theta} = C + b_{ads} \tag{11}$$

$$b_{ads} = \frac{1}{55.5} \exp\left(-\Delta Gads|RT\right) \tag{12}$$

where: θ is degree of surface coverage, C is the molar inhibitor concentration and $b_{ads}$ is the equilibrium constant of the adsorption process.

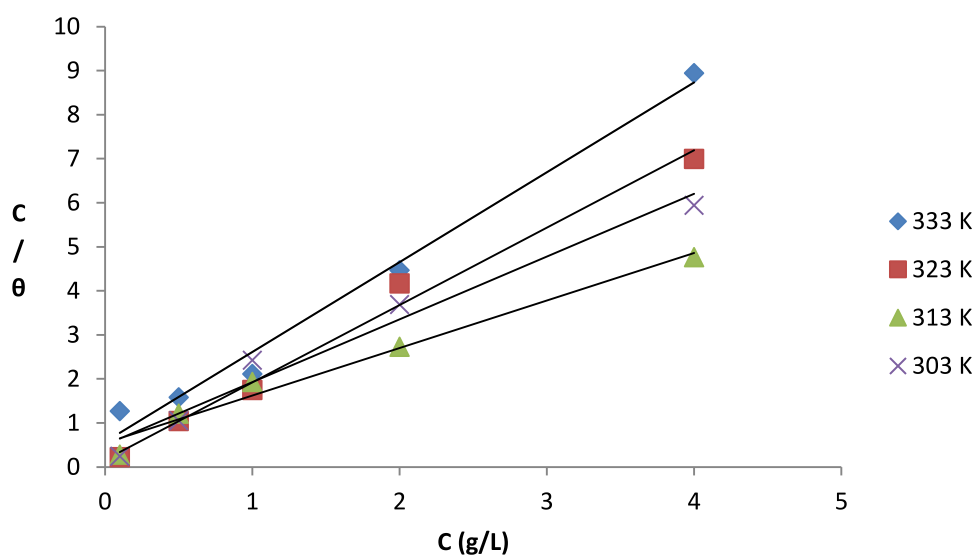


**Figure 7.** Langmuir isotherm for the adsorption of EEPA on the surface of mild steel at 303 K to 333 K.

The organic components present in the plant extracts is attributed to the adsorption on the metal surface. There is a barrier for mass and charge transfer in the adsorbed layer that lead to a reduction in the rate of corrosion of the metal. The fraction of surface coverage by the adsorbed molecule is an indication of the effectiveness of the adsorbed species and is directly proportional to the inhibition efficiency [23]. But the adsorption characteristics of EEPA on the mild steel surface at 303 K in HCl solution clearly shows a departure from a type.1 adsorption isotherm and assuming a more type II. Adsorption isotherm Figure. 9 the surface energy conditions of the metal can be said to change with decrease in temperature making the adsorption to shift towards a multilayer coverage at low inhibitor concentration. As the inhibitor concentration increases a flat region appear indicative of monolayer coverage [23].

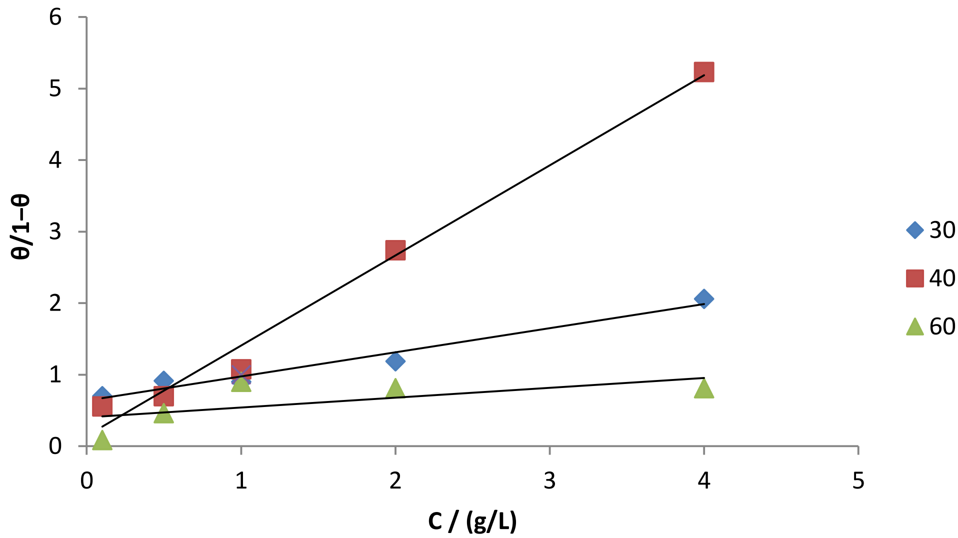


**Figure 8.** Langmuir isotherm for the adsorption of EEPA on the surface of mild steel at 303 K to 333 K.

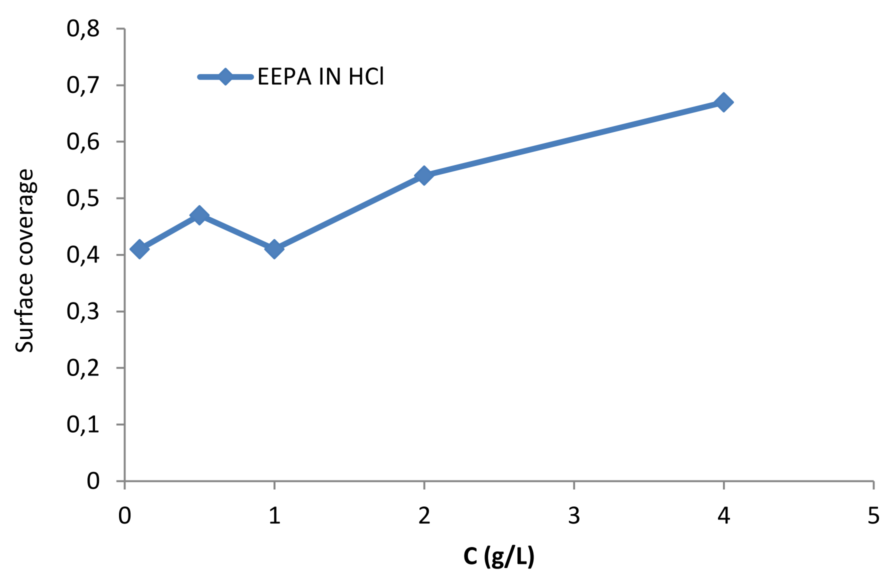


**Figure 9.** Type II adsorption isotherm depicting the adsorption characteristics of EEPA on mild steel surface in HCl solution at 303 K.

### 3. 5. Inhibition mechanism

The phytochemical analysis of *Phyllanthus amarus* carried out by[14] reveal the presence of saponins (24.05%), tannins (17.05%), Oxalate (5.47 %), alkaloid (2.56 %), Cyanogenic glycoside (1.46%), carbohydrates (45.52 %), fibre (24.50 %), protein (34.20 %) and fat (6.03 %) other literature [24] reported same. Organic compound in *Phyllanthus amarus* contain O and N atoms in functional groups such as O-H, N-H, C=O and C-O as well as O- heterocyclic rings in their molecular structure shown in Figure. 10 These properties satisfy the characteristics of corrosion inhibitors: Hence these compounds are responsible for the observed inhibition of mild steel corrosion in the inhibitor- acid solution [23]. Corrosion of mild steel in aqueous solution depend on the concentration of anion in solution [5] A general mechanism for the dissolution of mild steel would be similar to that reported by [5,8, 14]. In order to predict the type of adsorption, the corrosion mechanism of the iron must be known. The dissolution of iron in Hydrochloric acid is described by following equations.

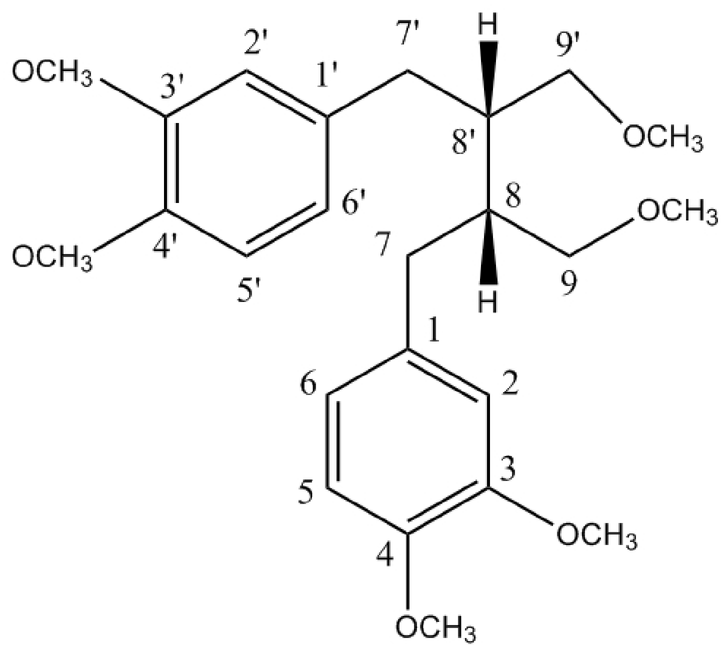


**Figure 10.** Molecular structure of *Phyllanthus amarus*

$$Fe + H_2O \leftrightarrow Fe.H_2O_{ads}$$

$$Fe.H_2O_{ads} + X \leftrightarrow Fe.OH^-_{ads} + H^+ + X^-$$

$$Fe.H_2O_{ads} + X \leftrightarrow Fe.X_{ads} + H_2O$$

$$Fe.OH^-_{ads} \leftrightarrow FeOH_{ads} + e^-$$

$$Fe.Xads \leftrightarrow FX^+_{ads} + e^-$$

$$FeOH_{ads} + FeX^+_{ads} \leftrightarrow Fe.X_{ads} + FeOH^+$$

$$FeOH^+ + H^+ \leftrightarrow Fe^{2+} + H_2O$$

The species X is the inhibitor molecule in this study. This mechanism show that the anodic reaction kinetic is affected by two intermediates that is one involving adsorbed hydroxyl ($FeOH_{ads}$) and the oxide involving the adsorbed inhibitor molecule ($Fex_{ads}$) [14]. The rate of anodic dissolution (step 4) depends on the product of step 2, but the competitive steps 2 and 3 are based on the $FeH_2O_{ads}$. Displacement of adsorbed water molecule by the species X can affect the step 4. The adsorption of ethanol extract of *Phyllanthus amarus* (EEPA) on the metal surface can occur either directly via donor- acceptor interaction between the π-electrons and free pair electrons of the heterocyclic atom and the vacant d orbital of surface iron atoms [10,23]. The inhibitor may be adsorbed on the metal surface in the form of natural molecules. Electrons sharing occur between the oxygen, nitrogen atoms and the metal surface thereby decreasing the corrosion rate of the metal [12,15,25-29].

## 4. CONCLUSIONS

The aim of this research was to apply adsorption isotherms in corrosion inhibition processes and also determine the feasibility of exploiting the Ethanollic extract of *Phyllanthus amarus* (EEPA) for materials corrosion control.

Our findings show that Ethanollic extract of *Phyllanthus amarus* (EEPA) effectively inhibited mild steel corrosion in 2 M HCl. At higher concentration and lower temperature, the extract functioned well as corrosion inhibitor. The experimental data fitted the Langmuir and Freundlich adsorption isotherm.

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