

## Investigation of the Inhibitive Properties of *Alchornea laxiflora* leaves on the Corrosion of Mild Steel in HCl: Thermodynamics and Kinetic Study

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**Abstract:** The use of naturally occurring compounds as corrosion inhibitors is of potential interest because of their cost effectiveness, abundant availability and environmental acceptability. Therefore, the study investigates the inhibition efficacy of acid extract of *Alchornea laxiflora* leaves on mild steel in acidic medium using gravimetric method. Experiments were performed by varying the immersion time, concentration of extract and the temperature. The results showed that acid extract of *Alchornea laxiflora* leaves is a potential inhibitor for the corrosion of mild steel in acidic medium. The corrosion rate of mild steel in 1M HCl decreases with increase in the concentration of the extract. The inhibition efficiency increases progressively as the concentration of the extract increases but decreases with rise in temperature and the exposure time. The highest inhibition efficiency observed in the presence of the extract was 96 %. Activation energy was found to be 21.81 kJ mol<sup>-1</sup> for the blank and increases to 82.57 kJ mol<sup>-1</sup> in the presence of the extract. Thermodynamic parameters such as enthalpy change, entropy change and Gibb's free energy were evaluated. Kinetics of the reaction in the presence of the extracts revealed that it follows a first order reaction and the half-lives increase as the concentration of the extract increases. Adsorption studies revealed that Langmuir adsorption isotherm is the best adsorption model applicable to the adsorption of the extract on mild steel surface. Preliminary investigation of the phytochemical constituents showed that the extract contains tannin, flavonoid, terpenoid and some other compounds in trace constituents.

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### 1. Introduction

Corrosion is the gradual destruction of materials, usually metals, by chemical reaction with its environment especially in corrosive media. It can also occur in materials other than metals, such as ceramics or polymers, although in this context, the term degradation is usually employed (Fontana, 1987). Corrosion degrades the useful properties of materials and structures which include their strength, appearance and their ability to contain vessel's contents (Al-Otaibi et al, 2014). Since corrosion results in the deterioration of metal material when the metal reacts with the environment, especially in the presence of oxygen, it is imperative to monitor and prevent the interaction of the metal with the environment. There are several ways of preventing corrosion and the rates at which it can propagate with a view to improving the lifetime of metallic and alloy materials. Examples of such methods are: protective coating, changing interfacial potential, changing the environment, addition of inhibitors and so on (Abiola et al, 2007; Ebenso et al, 2009; Orubite and Oforka, 2004). However, the use of inhibitors for the control of corrosion of metals and alloys which are in contact with aggressive environment is one of the acceptable

practices used to reduce and/or prevent corrosion (Buchweishaija, 2011; Ebenso et al., 2004; Olasehinde et al, 2013).

Corrosion inhibitors can be divided into two broad categories namely those that enhance the formation of a protective oxide film through an oxidizing effect and those that inhibit corrosion by selectively adsorbing on the metal surface and creating a barrier that prevents access of corrosive agents to the metal surface (Daniyan et al., 2011). A number of organic compounds have been studied to investigate their potential as corrosion inhibitors (Kliskic et al, 2000; Ebenso et al, 2004; Abder-Gaber et al, 2006). Both synthetic and natural compounds containing heteroatoms have been found to inhibit corrosion by forming an adsorptive film on the metal surface to prevent direct attack of the corrosion agent on the metal. However, the toxic effects of synthetic corrosion inhibitors have led to the search for more naturally occurring substances which are not only readily available but are also environmentally friendly (Olasehinde et al, 2013; Akbarzadeh et al., 2011 ; Olusegun et al., 2013; Fouda et al, 2014; Devarayan et al, 2012; Chen et al., 2013; Behpour and Mohammadi,

2012; and Negm et al., 2012) with a view to replacing the synthetic inhibitors.

The plant *Alchornea laxiflora* belongs to the family of Euphorbiaceae. It is a shrub up to six metre height. It is widely spread in Nigeria as well as in West Cameroon. In Nigeria, it bears different names: Edo (Uwenuwen), Igbo (Ubuho), Urhobo (Urievwu), Yoruba (ijàn, ijàn funfun or pépé) and in Western Cameroon, it is known as Longoso. The leaves are often used to preserve the moisture of kola nuts during packaging. The stems, and especially the branchlets, are used in Nigeria as chewing sticks. Decoction of the leaves are used in the treatment and management of inflammatory and infectious diseases as well as an important component of herbal antimalarial formulations (Farombi et al, 2003).

To the best of our knowledge, the use of this plant as an inhibitor for corrosion mitigation in corrosive media has not been reported. Therefore, the aim of this paper is to investigate the inhibitive properties of *Alchornea laxiflora* extracts on mild steel in acid medium from thermodynamic and kinetic point of view.

## 2. Material and Methods

### 2.1 Extract Preparation

The leaves of *Alchornea laxiflora* were obtained from the vicinity of the Federal University of Technology, Akure and were authenticated at Crop Science and Production Department, Federal University of Technology, Akure. The leaves were washed, air-dried at room temperature, ground and sieved through a 850 micron mesh. Acid extraction method was carried out by soaking 50 g of the pulverized leaves in 100 mL of 1M HCl and boiled in a thermostated water bath at 90 °C for 3 hours. After this pre-set time, the mixture was allowed to cool overnight, after which it was filtered through Whatman filter paper. The filtrate was then kept as the stock solution. Working solutions of different concentrations ranging from 0.1 to 1 % (v/v) were prepared from the stock solution.

### 2.2 Specimen Preparation:

Materials used for the study was mild steel of composition (wt %); C (0.34); Si (0.16); S (0.05); P (0.05); Mn (0.81); Cr (0.18); Mo (0.02); V (0.005); Cu (0.36); W (0.002); As (0.005); Sn (0.04); Co, (0.009); Al, (0.002); Fe (98.83). The steel was mechanically cut to coupons of dimension 1.8 × 1.4 × 0.4 cm. Each coupon was degreased by washing with ethanol, cleaned with acetone and allowed to dry in the air before preservation in a desiccator. Accurate weights of the coupons were determined using an analytical balance of 0.0001 mg accuracy. All reagents used for the study were analar grade and distilled water was used for the preparation.

### 2.3 Weight loss method

The weight loss method employed in this study has been described elsewhere (Olusegun et al, 2013; Olasehinde et al., 2012, 2013). Briefly, a previously weighed mild steel coupon was completely immersed in 100 mL of 1M HCl in the absence and presence of different concentrations (0.1%, 0.2%, 0.3%, 0.5%, 1%,,) of the inhibitor with the aid of glass hooks at different temperatures (303, 313, 323 and 333 K) for 4 hours. The thermostated water bath was set to the appropriate temperature and after 4 hours of immersion, the mild steel was withdrawn from the test solution and the corrosion product was removed by washing each coupon in distilled water, rinsed in acetone and dried in the air completely before re-weighing. From the initial and final weights of the mild steel, the weight loss, corrosion rate ( $C_R$ , g hr<sup>-1</sup>cm<sup>-2</sup>) in absence and presence of inhibitors, inhibition efficiency (I.E %) of the inhibitors and the degree of surface coverage ( $\theta$ ) were calculated using equations 1, 2 and 3 respectively (Eddy, 2009).

$$\text{Corrosion rate} = \frac{\Delta W}{At} \quad (1)$$

$$\text{Inhibition Efficiency} = 1 - \left( \frac{C_{R2}}{C_{R1}} \right) \times 100 \quad (2)$$

$$\text{Surface coverage } (\theta) = 1 - \left( \frac{C_{R2}}{C_{R1}} \right) \quad (3)$$

where  $\Delta w$  is the weight loss in grammes,  $C_{R1}$  and  $C_{R2}$  are the corrosion rates of the mild steel strip coupons in absence and presence of inhibitor,  $A$  is the cross-sectional area in cm<sup>2</sup> and  $t$  is the exposure time in hours.

### 2.4 Chemical Analysis

Phytochemical analysis of *Alchornea laxiflora* extract was carried out according to the method reported by Onyeka and Nwabekwe, 2007.

## 3. Results

### 3.1 Effect of extract concentrations on corrosion rate

Figure 1 shows the effect of the extract concentrations on the corrosion rate of mild steel in 1 M HCl. The corrosion rate was monitored by varying the temperature of the medium from 303 k to 343 k. The result shows that the corrosion rate of mild steel in 1M HCl decreases with increase in the concentrations of the extract at all temperature values. This is expected because as the concentration of the extract increases, there is an increase in the rate at which the phytochemical constituents are adsorbed on the surface of the mild steel thereby creating a barrier for charge and mass transfer which results into a decrease in the interaction between the metal and the corrosive medium and hence, reduces the corrosion

rate. This result is in agreement with the reports of the previous investigators (Okafor et al, 2008; Olasehinde et al, 2012).

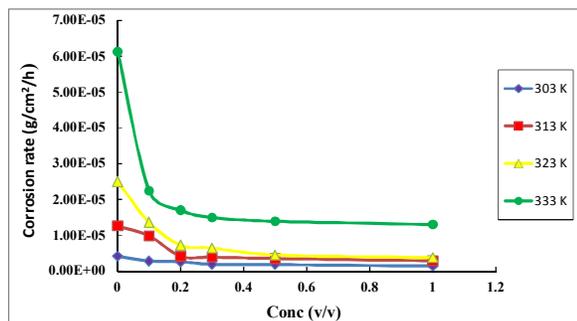


Figure 1: Effect of extract concentration on corrosion rate of mild steel

### 3.2. Effect of extract concentrations on inhibition efficiency

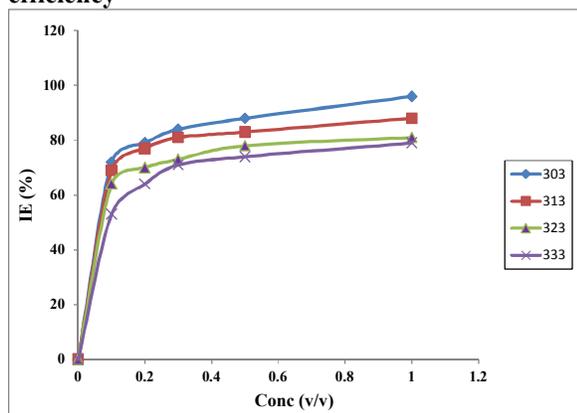


Figure 2: Effect of extract concentrations on inhibition efficiency

As shown in Figure 2, the inhibition efficiency of the extract increases as the concentration of the extract increases due to the increase in the fraction of the surface of the mild steel covered by the absorbed molecules of the extracts at higher concentration. The inhibition efficiency increases progressively as the concentrations of the extract increase up to about 0.3 v/v, above which, further increase in the extract concentration did not cause any significant change in the inhibition efficiency of the extract which might indicate that the reaction of the inhibitor on the surface of the mild steel has reached the state of equilibrium. The maximum percentage inhibition of 96 % was recorded at the highest concentration studied at 303 K.

### 3.3 Effect of temperature on corrosion rate of mild steel

The effect of temperature on the corrosion rate of mild steel in blank solution and in the presence of different concentrations of the inhibitors was

studied in the temperature range 303 K – 343 K as shown in Figure 3. It was found that the rate of corrosion of mild steel in the blank and inhibited acid solution increases with increase in temperature. However, the corrosion rate is much retarded in the solution containing the inhibitor than the blank solution. This is plausible because as temperature increases, the rate of corrosion of mild steel also increases as a result of increase in the average kinetic energy of the reacting molecules. The decrease in the corrosion rate in the solution containing the inhibitor is as a result of the mitigating effect of the phytochemical constituents of the extracts on the corrosion rate of the mild steel.

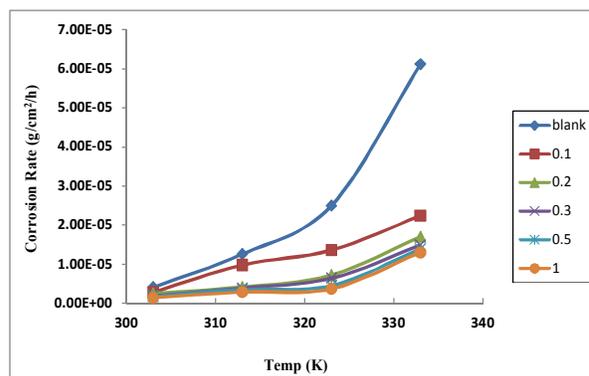


Figure 3: Effect of temperature on corrosion rate of mild steel

### 3.4 Effect of temperature on inhibition efficiency

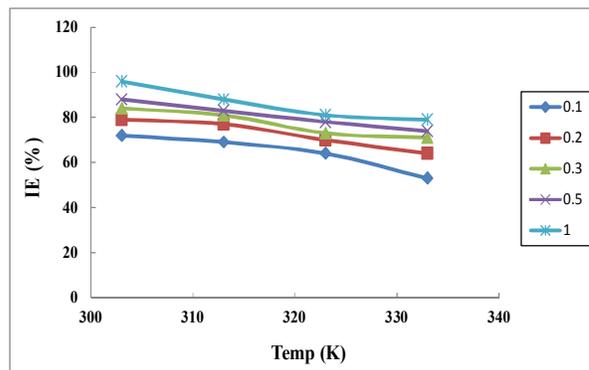


Figure 4: Effect of temperature on the inhibition efficiency.

The effect of temperature on the inhibition efficiency of *Alchornea laxiflora* extract on mild steel is as shown in Figure 4. It is evident from the Figure that as the reaction temperature increases from 303 - 333K, the inhibition efficiency decreases. This is expected because increase in the reaction temperature might result into increase in the rate of dissolution process of the mild steel and partial desorption of the

inhibitor from the metal surface (Blaedel and Meloche, 1963). This phenomenon is consistent with the mechanism of physical adsorption as reported by (Eddy et al., 2009; Ebenso et al, 2003, 2004).

### 3.1.5. Effect of immersion time

In order to assess the stability of inhibitive behaviour of the extract on a time scale, weight loss measurement was performed in 1M HCl in absence and presence of the extract at 0.1– 0.6% (v/v) concentration for 7 days at room temperature. The plot of weight loss against time (Figure 5) shows that weight loss increases as the immersion time increases. However, the weight loss is much reduced in the presence of the inhibitor compared to the blank solution. The decrease in weight loss in the presence of inhibitor may be due to the adsorption of the phytochemical constituents in the extract on the surface of the mild steel.

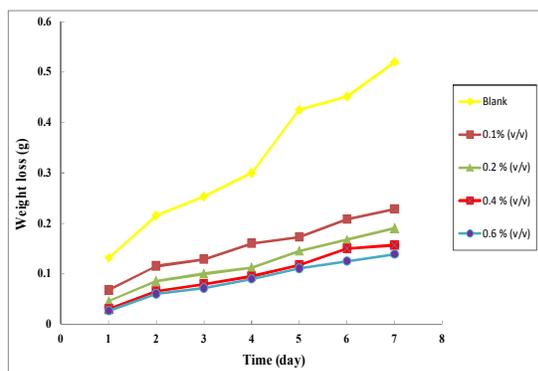


Figure 5: Effect of immersion time on the weight loss of mild steel at room temperature.

### 3.2. Kinetic Study

The kinetics of the corrosion process acquires the character of a diffusion process, in which at higher temperature, the amount of inhibitor present on the metal surface is much reduced than that present at lower temperature (Ekpe et al. 2004). It is on this basis that kinetic analysis of the data is considered necessary. In this present study, the initial weight of mild steel coupon at time  $t$ , is designated  $W_i$ , the weight loss is  $W_L$  and the weight change at time  $t$ , ( $W_i - W_L$ ) while  $k$ , is the first order rate constant.

$$\ln (W_i - W_L) = -k_1t + \ln W_L \quad (4)$$

According to equation 4, the plots of  $\ln (W_i - W_L)$  against time (days) at 303 K showed a linear variation (Figure 6) and the first order reaction rate constants ( $k$ ) calculated from the slope of the graph were presented in Table 1.

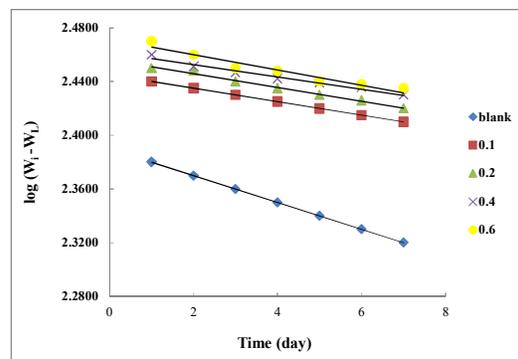


Figure 6: Variation of  $\ln (W_i - W_L)$  with time (days) for mild steel coupons in 1M HCl solution containing *Alchornea laxiflora* extract at 303K.

The half-life ( $t_{1/2}$ ) was calculated from the relation below:

$$t_{1/2} = 0.693 / k_1 \quad (5)$$

The increase in half-lives ( $t_{1/2}$ ) in the presence of the extract compared to the blank further corroborates our earlier results that corrosion rate decreases in the presence of the extract compared to the blank solution. It should also be noted that as the concentration of the extract increases, the half-life also increases which results into a decrease in the corrosion rate suggesting that more protection of the mild steel by the *Alchornea laxiflora* extract has been established.

Table 1: Half-life parameters at various concentrations

Extract Conc. (% v/v)	Rate Constant ( $\text{day}^{-1}$ )	Half life (days)
Blank	0.01	69.3
0.1	0.005	138.6
0.2	0.0034	203.8
0.4	0.0028	247.5
0.6	0.0017	407.6

### 3.3. Thermodynamic Study

#### 3.3.1 Determination of Activation Energy ( $E_a$ )

The adsorption of the organic compounds can be described by two main types of interactions: physisorptions and chemisorption. They are influenced by the nature of the charge of the metal, the chemical structure of the inhibitor, pH, the type of the electrolyte and temperature. Thus, in order to elucidate the inhibitive properties of the inhibitor and the temperature dependence on the corrosion rates, the apparent activation energy ( $E_a$ ) for the corrosion process in the absence and presence of the inhibitor was evaluated from Arrhenius equation:

$$\ln (k_2/k_1) = E_a/R (1/T_1 - 1/T_2) \quad (6)$$

Where  $k_1$  and  $k_2$  are the corrosion rates at temperature  $T_1$  and  $T_2$  respectively,  $E_a$  is the apparent activation

energy and R is the molar gas constant. The estimated values of  $E_a$  for mild steel corrosion in the presence of *Alchornea laxiflora* extract in 1 M HCl are presented in the Table 2. Activation energy  $E_a$  was found to be 21.81 kJ mol<sup>-1</sup> for 1M HCl and increases to 82.57 kJ mol<sup>-1</sup> in the presence of the extract which shows that the adsorbed organic matter has provided a physical barrier to charge and mass transfer, leading to reduction in corrosion rate (Abiola et al, 2007; Ebenso et al, 2003).

As observed from Table 2, the  $E_a$  increased as the concentration of the extract increases, and all the values of  $E_a$  in the range of the studied concentration were higher than that of the uninhibited solution. The increase in the activation energy values with increasing concentration of the extract further corroborates the fact that inhibition efficiency increases with increasing concentration of the extract. Previous investigators have established that the value of  $E_a > 80$  kJ/mol indicates chemical adsorption whereas  $E_a < 80$  kJ/mol infers physical adsorption (Vijayalakshmi et al, 2011). In the present study, a physical adsorption mechanism is proposed since the values of  $E_a$  are lower than 80 kJ mol<sup>-1</sup>. The increase in activation energy in the presence of plant extracts can be attributed to an appreciable increase in the adsorption of the inhibitor on the mild steel

surface which resulted into decrease in the corrosion rate of the mild steel.

Table 2: Activation energy values with the various concentrations of the extract

Extract Conc. (% v/v)	Activation Energy (kJ mol <sup>-1</sup> )
Blank	21.81
0.1	29.09
0.2	34.66
0.3	50.04
0.5	65.38
1.0	82.57

### 3.3.2. Determination of $\Delta H$ and $\Delta S$

Other thermodynamic parameters such as enthalpy change ( $\Delta H$ ) and entropy change ( $\Delta S$ ) of activation process were evaluated from the effect of temperature on the corrosion rate mild steel in 1 M HCl using equation 7.

$$\log \left( \frac{CR}{T} \right) = \log \left( \frac{R}{nh} \right) + \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT} \quad (7)$$

where CR is the corrosion rate at temperature T, R is the molar gas constant, n is Avogadro's constant, and h is the Planck's constant. A plot of  $\log \left( \frac{CR}{T} \right)$  vs  $1/T$  is a straight line graph (Figure 7) with a slope of  $(-\frac{\Delta H}{2.303R})$  and an intercept of  $(\log \left( \frac{R}{nh} \right) + \frac{\Delta S}{2.303R})$ .

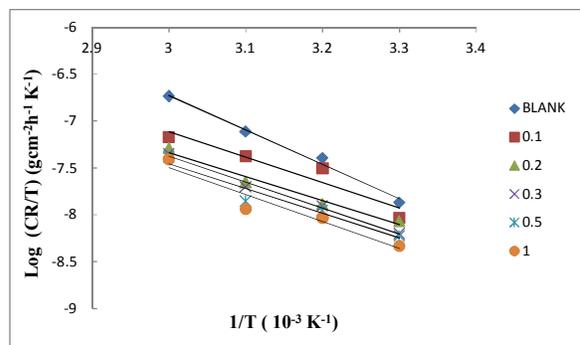


Figure 7: Eyring Transition State plot for mild steel in 1M HCl in the blank and presence of the extract

Thus,  $\Delta H$  and  $\Delta S$  can be evaluated from the slope and intercept of the graph, respectively. The results presented in Table 3 show that  $\Delta H$  values are all positive. The positive signs of the enthalpies reflect the endothermic nature of the mild steel dissolution process. Also,  $\Delta S$  values are negative indicating that the activation complex in the rate-determining step represents an association rather than dissociation step. These results are in excellent agreement with the reports of Fouda et al, 2009.

Table 3: Enthalpy and Entropy change of the reaction process with various concentrations of the extract.

Extract Conc. (% v/v)	$\Delta H$ (kJ/mol)	$\Delta S$ (kJ/mol/k)
Blank	104.9	-0.35
0.1	112.2	-0.40
0.2	117.8	-0.42
0.3	133.2	-0.47
0.5	148.51	-0.52
1.0	155.6	-0.55

### 3.3.3 Determination of Free Energy

The free energy of adsorption values  $\Delta G^{\circ}_{ads}$  were evaluated using equation 6:

$$\Delta G^{\circ}_{ads} = -2.303RT \log(55.5 K_{ads}) \quad (6)$$

The values of  $\Delta G^{\circ}_{ads}$  calculated are presented in Table 4. The results indicate that the values of  $\Delta G^{\circ}_{ads}$  are negative in all cases, showing that the reaction is spontaneous and feasible (Olasehinde, et al, 2012; Abiola, et al, 2007), and that the *Alchornea laxiflora* extracts are strongly adsorbed on the medium carbon steel surface by physical adsorption mechanism (Ebenso et al, 2003). Previous researchers have established that the values of  $\Delta G^{\circ}_{ads}$  up to -20 kJmol<sup>-1</sup> are consistent with electrostatic interaction between charged molecules and a charged metal indicating physisorption, while those more negative than -40 kJmol<sup>-1</sup> involve charge sharing or transfer

from the inhibitor molecules to the metal surface to form a co-ordinate type of bond otherwise known as chemisorption. In the present study, the values of  $\Delta G^{\circ}_{ads}$  are less than  $-40 \text{ kJ mol}^{-1}$  which indicate that the adsorption of the inhibitor on the metal surface confirms a physical adsorption mechanism (Vimala et al, 2011).

Table 4: Free Energy of adsorption at various temperatures

Extract Conc. (% v/v)	Activation energy ( $\text{kJ mol}^{-1}$ )
303	-7.68
313	-8.64
323	-9.32
333	-9.80

### 3.4. Adsorption Isotherm

Adsorption isotherms are very important to understand the mechanism of heterogenous organo-electrochemical reactions. The establishment of adsorption isotherms that describe the adsorption of a corrosion inhibitor can provide important information on the nature of the metal-inhibitor interaction (Onuegbu et al, 2013; Obot et al, 2010). Adsorption of the organic molecules occurs as the interaction energy between molecule and metal surface is higher than that between the  $\text{H}_2\text{O}$  molecule and the metal surface. In adsorption isotherm, the degree of surface coverage ( $\theta$ ) is very useful. The degree of surface coverage values for different concentrations of *Alchornea laxiflora* obtained at different temperatures from the weight loss measurement was calculated using Equation 3. Attempts were made to fit the experimental data obtained from weight loss measurements into different isotherms. The test indicates that Langmuir adsorption isotherm model best described the adsorption characteristics of *Alchornea laxiflora* extracts on the mild steel. Hence, no further consideration of other models was attempted.

Langmuir adsorption isotherm is the ideal adsorption isotherm for physical and chemical adsorption on a smooth surface (Bilgic and Caliskan, 2001). The Langmuir isotherm is valid for monolayer adsorption onto a surface containing a finite number of identical sites. The model assumes uniform energies of adsorption onto the surface and no transmigration of adsorbate in the plane of the surface. Langmuir adsorption isotherm can be expressed according to Equation (7).

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (7)$$

According to this isotherm,  $C_{inh}$  is the inhibitor concentration,  $\theta$  is the surface coverage and  $K_{ads}$  is the adsorption equilibrium constant. The plot of  $C_{inh}/\theta$  against  $C_{inh}$  is linear as shown in Figure 8. The results show that the slopes and  $R^2$  are very close to unity indicating that the adsorption of the inhibitor on the surface of mild steel is consistent with Langmuir isotherm.

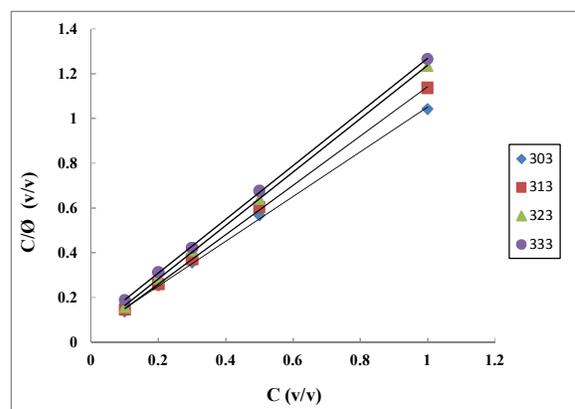


Figure 8: Langmuir adsorption isotherm plot for mild steel corrosion in 1M HCl for *Alchornea laxiflora* extract at different temperatures

### 3.5 Phytochemicals

Preliminary investigation of the phytochemical composition of *alchornea laxiflora* extracts in reo showed that the extract contains saponins, terpenes, tannins, Flavonoid, alkaloid, cardiac glucosides and phlobatannins. Inspection of the chemical structures of these phytochemical constituents reveals that these compounds are easily hydrolysable and the compounds can be adsorbed on the metal surface via the lone pair of electrons present on their oxygen atoms which can cause a barrier for charge and mass transfer leading to decrease in the interaction of the metal with the corrosive environment (Al-Dokheily et al 2014). As a result, the corrosion rate of the metal was decreased. Hence, the inhibition efficiency of the extract may be attributed to the phytochemical constituents of the extract.

### 4.0 Conclusion

The study has revealed that the acid extract of *alchornea laxiflora* is a potential eco-friendly inhibitor for the corrosion of mild steel in acid medium. Inhibition efficiency increases as the concentration of the extract increases but decreases with increase in the temperature. Thermodynamic parameters confirm that the adsorption of the inhibitor on the surface of the mild steel is spontaneous and is consistent with the physical adsorption mechanism. Kinetic studies showed that inhibition efficiency increases with the

concentration of the extract. The inhibition potentials of the inhibitor are attributed to the presence of the phytochemical constituents in the extract.

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