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EVALUATION OF PALM LEAF AS A VIABLE INHIBITOR FOR MITIGATION OF MILD STEEL CORROSION IN HYDROCHLORIC ACID MEDIUM

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Abstract- This work aims to limit mild steel corrosion in HCl solution by employing plant extract as inhibitor, namely leaf extract from a palm tree. It involved extracting and characterizing the extract from palm tree leaves. The assessment of mild steel's ability to resist corrosion and the investigation of the impact of concentration, temperature, and time on the corrosion control procedure came next. Phytochemical results revealed that palm leaf extract has alkaloids in a highly concentrated quantity (185.12 ±0.12 mg/100g), while saponin, and tannins values are in concentrated quantities; 79.55 ±0.02 mg/100g and 97.73 ±0.14 mg/100 g respectively. Result analyses revealed O-H stretch, CH_3 C-H bend, C-H stretch, N-H bend, C=H stretch, C=O stretch and C-F stretch as the functional groups of the palm leaf. The inhibitor influenced weight loss, corrosion rate, inhibition efficiency, and degree of surface coverage. The activation energy (E_a) of the control of mild steel deterioration using palm leaf is 53.70 kJ/mol. It was also revealed that the heat of adsorption's status is negative, indicating that heat moved from the apparently higher temperature of the inhibitor-mild steel contact to the surrounding (supposedly lower temperature environment). On the adsorption mechanism, Langmuir isotherm was displayed as the best-fitting isotherm in this physical adsorption of the inhibitor's molecules. Quadratic model was established as the relationship between the efficacy of inhibitor and the factors of concentration, temperature and time for the corrosion control process.
Keywords: Corrosion Inhibitor, Langmuir Isotherm, Mild Steel, Palm Leaf

1.1 Introduction

Mild steel is a useful metal with a strong tensile strength. It is easily produced and widely available for industrial applications. As such, it is used in a wide range of industries (Singh et al, 2014; Popova et al, 2015; Verma et al, 2016). Its industrial usefulness is hindered by the possibility of the mild steel structure deteriorating because of corrosion. The rate and amount of corrosion are influenced by factors such as temperature, time and aggressive medium. Mild steel corrodes severely when exposed to acid solutions in the course of acid cleaning, acid shipping, acid de-scaling, acid storage, and other chemical operations (Fouda et al, 2021). Corrosion can be referred to as the deterioration of materials due to electrochemical reaction with the surrounding medium. Corrosion has various negative effects, many of which are more harmful than loss of a significant amount of metal (Uwiringiyimana et al., 2019 Mahgoub et al, 2019; Yang et al, 2021). Corrosion is detrimental to the chemical, oil and metallurgical industries. As a result, metal corrosion continues to be a global scientific concern. Studies on corrosion processes and control are currently being conducted by researchers from a range of industrial sectors (Azeez et al, 2018; Deyab, 2020).

Application of inhibitor is said to be one of the best ways to prevent metals from corroding, particularly in environments that are acidic. Most corrosion inhibitors are composed of costly and ecologically unsafe synthetic compounds. Right now, the emphasis is on creating and obtaining corrosion inhibitors that are safe for the environment (Guo et al, 2015; Omotioma and

Onukwuli, 2019). Several studies revealed that biomass extracts contain a variety of interesting phytochemicals, such as triterpenoids, alkaloids, tannins, flavonoids, saponins, amino acids, ascorbic acid, and phenolic acids. These phytochemicals share electronic structures with conventional organic corrosion inhibitors. According to Rajeswari et al (2014), plant extracts have the potential to prevent metals from corroding/ deteriorating. Hence, current research reports examined the application of extracts of plant origin to inhibit mild steel deterioration in a harmful environment (Umoren et al, 2016; Gaya et al, 2019; Bilgiç, 2021; Qurishi et al, 2021). But the functionality of the phytochemical of plant extract has not been fully established. For instance, palm leaf is often discarded as waste instead of testing its ability (in terms of using its phytochemicals) to mitigate the corrosion of metals. This is the research gap intended to be filled by this study. Thus, this study focused on the evaluation of palm leaf as a viable inhibitor for mitigation of mild steel corrosion in hydrochloric acid medium.

2.1 Materials and Method

Materials used for this project included filter cloths, spectrophotometers, stopwatches, and palm leaves. Other supplies included mild steel coupons, glass measuring cylinders, electronic weighing balances, conical flasks, corks, emery papers, water baths, retort stands, beakers, spatulas, volumetric flasks, distilled water, ethanol, acetone, $FeCl_3$, HCl, H_2SO_4, NH_4OH, filter cloths, ethanol, ethanol, and acetone.

2.2 Experiment Method

2.2.1 Mild Steel Preparation

To conduct this experiment, mild steel was partitioned into 3cm x 3cm corrosion coupons. Different grades of emery paper were used to make the specimens' surface mechanically polished. After washing with distilled water, the surfaces were decreased using acetone.

2.2.2 Characterization of the palm leaf in Terms of Functional Groups

The palm leaf was analyzed using the Fourier transform infrared (FTIR) spectrophotometer to determine the functional groups. Using the Br pellet approach, the FTIR spectrum was acquired in transmission mode. The approach was used to transform the unprocessed data into a true spectrum (with several peaks). In line with method used by Omotioma et al (2024), FTIR-generated peaks were used to determine the associated functional groups.

2.2.3 Phytochemical analysis of the palm leaf extract

Standard operating protocols from previous authors (Mayuri, 2012; Mada et al, 2012; Omotioma and Onukwuli, 2019) were applied for the phytochemical analysis of the palm leaf.

a. Alkaloids

20ml of ethanol and 10% acetic acid were combined with 1g of the substance. 20ml of ethanol and 10% acetic acid were combined with 1g of the substance. The mixture was shaken and allowed to settle for four hours. There was a screening after that. The filtrate lost about 25% of its initial volume after evaporation. One drop at a time, ammonium hydroxide solution was added. The precipitate that had formed was filtered using a weighted filter paper. To dry, the filter paper was put in an oven set at 60°C. The mixture was shaken and allowed to settle for four hours. There was a screening after that. The filtrate lost about 25% of its initial volume after evaporation. One drop at a time, ammonium hydroxide solution was added. Once the filter paper had dried to a consistent weight, it was weighed.

b. Cardiac glycosides

For fifteen minutes, a gram of the material was baked at 100°C. Glacial acetic acid (2 ml) and a drop of FeCl3 were combined with 1 ml of the sample and 5 ml of water. 1 ml of conc. H2S04 was also added. At 410 nm, the resultant solution's absorbance was measured.

c. Flavonoids

A 2% $AlCl_3$ methanol solution was combined with 0.05 ml of the sample solution. After an hour at room temperature, a yellow tint appeared, indicating the presence of flavonoids. It was determined how much flavonoids there were in mg/g of quereetin.

d. Phenols

Two minutes were given for the 0.2% formic acid to settle into the 2g of sample. After then, it was filtered. 2 milliliters of the filtrate and

0.5 milliliters of the folin-ciocalteau reagent were added to a test tube using a pipette. It was given twenty minutes to acquire its color. The concentration for a standard graph was discovered after the absorbance at 765 nm was read. The gallic acid equivalent, or GAE/g, formula is used for it.

e. Phytate

A test tube containing the sample and 0.5 milliliter of ferric ammonium sulphate was added. For thirty minutes, the test tube was heated in a water bath. After cooling, a centrifuge was used. 1.5 ml of the 2,2-bipyridine solution and 1 ml of the supernatant were mixed together. Pure water was used as the blank for the measurement, which was performed at 519 nm.

f. Saponins

15 ml of ethanol was added to 1 g of the sample, which was then incubated for 4 hours at 55 °C in a water bath. Twice after filtration, the remaining material was cleaned using 20% ethanol. The sample was reduced to around 5 milliliters in the oven. The condensed sample was placed into a separating funnel and five milliliters of petroleum ether were added. After adding three milliliters of butanol, the petroleum ether layer was disposed of. It was washed with five milliliters of 5% sodium chloride. The residue was weighed after baking until it was fully dry.

g. Tannins

Over the course of five hours, one gram of the material was extracted using 25 milliliters of a solvent mixture that contained 80:20 acetone: 10% glacial acetic acid. Following filtering, the absorbance at 500 nm was computed. The reagent blank's absorbency was also measured. Tannic acid concentrations of 10, 20, 30, 40, and 50 mg/100g were used to construct a typical graph. After accounting for any dilution impact, the tannin concentration was determined.

2.2.4 Weight Loss Method

The weight loss (gravimetric) strategy used in Omotioma et al (2024) was applied in this investigation. The elemental compositions of the mild steel are Cu = 0.01%, Mn = 0.11%, Si = 0.02%, Ni = 0.02, P = 0.02%, Cr = 0.01%, and Fe = 99.56 percent. Equations (1), (2), (3) and (4), were employed to calculate the weight loss (Δw), corrosion rate (CR), inhibition efficiency (IE) and degree of surface covering in that order:

$$\Delta w = w_i - w_f \quad (1)$$

$$CR = \frac{w_i - w_f}{At} \quad (2)$$

$$IE\% = \frac{\omega_0 - \omega_1}{\omega_0} * 100 \quad (3)$$

$$\Theta = \frac{\omega_0 - \omega_1}{\omega_0} \quad (4)$$

2.2.5 Determination of Effects of Corrosion Control Variables

The impacts of three corrosion control variables (inhibitor concentration, temperature and time) on the weight reduction, corrosion rate, and inhibition efficiency were examined.

2.2.6 Determination of thermodynamic properties of the corrosion control process

The activation energy of the corrosion control approach was found using the linear form of the Arrhenius-model (Equation 5).

$$\text{In}(\text{CR}) = \text{InA} - \left({E_a}/{R}\right)\left(\frac{1}{\text{T}}\right) \quad (5)$$

Equation (6), as applied by Onukwuli and Omotioma (2024), was used to determine the heat of adsorption, Qads (kJmol-1).

$$Q_{ads} = 2.303R\left[\log\left(\frac{\theta_2}{1-\theta_2}\right) - \log\left(\frac{\theta_1}{1-\theta_1}\right)\right] * \frac{T_2 . T_1}{T_2 - T_1} \quad (6)$$

2.2.7 Consideration of the adsorption isotherms

Equations (7), (8), (9), and (10) assessed the applicability of Langmuir, Frumkin, Temkin, and Flory-Huggins, respectively, using data on the degree of surface covering (Equation 4), which is directly connected to efficiency (Equation 3), at different inhibitor doses. The Gibb's energy (ΔGads) was then calculated using Equation (11), which is dependent on the gas constant (R), absolute temperature (T), and adsorption equilibrium constant (K) (Omotioma and Onukwuli, 2017; Anadebe et al, 2019).

$$log\frac{C}{\theta} = \log C - logK \quad (7)$$

$$\log\left((C) * \left(\frac{\theta}{1-\theta}\right)\right) = 2.303logK + 2\alpha\theta \quad (8)$$

$$\theta = -\frac{2.303logK}{2a} - \frac{2.303logC}{2a} \quad (9)$$

$$\log\left(\frac{\theta}{C}\right) = \log K + xlog(1-\theta) \quad (10)$$

$$\Delta G_{ads} = -2.303RTlog(55.5K) \qquad (11)$$

3.1 Characteristics of the Palm Leaf

3.1.1 Functional groups of the palm leaf

An FTIR spectrum of palm leaf is shown in Figure 1. Predominant functional groups of the palm leaf were revealed as O-H stretch, CH_3 C-H bend, C-H stretch, N-H bend, C=H stretch, C=O stretch and C-F stretch. Heteroatoms of N and O were present, which is an indication that it is a potential corrosion inhibitor (Omotioma et al, 2024).

3.1.2 Qualitative and Quantitative Analyses of the Phytochemicals of Palm Leaf

Table 1 provide the findings of qualitative and quantitative phytochemical studies of leaf extract from palm trees The symbols for the phytochemicals' qualitative effects are +++ (very concentrated), ++ (concentrated), + (in traces), and - (absent). Plants and plant-derived extracts often include these substances. The extract's ability to reduce corrosion is demonstrated by the phytochemicals it contains (Omotioma et al, 2019). The quantitative results showed that the alkaloids in palm leaf extract are highly concentrated at 185.12 ±0.12 mg/100g, while the values of saponin and tannins are also concentrated at 79.55 ±0.02 mg/100g and 97.73 ±0.14 mg/100g, respectively. As a result, the extract has strong anti-corrosion qualities.

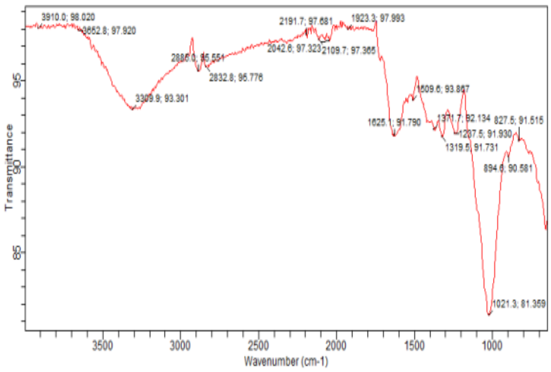

Figure 1: Spectrum of the palm leaf

Table 1: Phytochemical Analysis of the palm leaf extract

Phytochemicals	Qualitative results	Quantitative results
Alkaloids (mg/100g)	+++	185.12 ±0.12
Cardiac glycosides (mg/100g)	-	13.42 ±0.07
Flavonoids (mg/100g)	+	48.72 ±0.33
Phenolics (GAE/g)	+	37.41 ±0.01
Phytates (mg/100g)	+	68.32 ±0.013
Saponins (mg/100g)	++	79.55 ±0.02
Tannins (mg/100g)	++	97.73 ±0.14

–. (too little to be observed qualitatively), + (in traces), ++ (concentrated) and +++ (highly concentrated)

3.2 Effect of Process Factors on Corrosion Control of Mild Steel

Tables 2 to 4 show the impact of inhibitor concentration, temperature, and duration on mild steel corrosion inhibitors in HCl-solution. Tables 2 demonstrate that weight loss and the rate of corrosion decreased as the inhibitor's concentration increased. Conversely, at the maximal concentration, both the level of surface covering, and the inhibitory efficacy increased. At 0.8g/L concentration, the inhibitory efficiency of palm leaf, respectively, peaked at 94.44%. This is because the inhibitor molecules are more electrostatically attracted to the mild steel surface. As the concentration increased from 0.8g/L to 1.0g/L, the inhibition efficiency showed a little decline beyond the maximum point, moving from 94.44% to 92.47%. The decrease in the force of attraction between the inhibitor's molecules and the mild steel surface is the cause of the observed retardation in each case (Ezeugo et al, 2017; Paul et al, 2020; Qurishi et-al, 2021). Tables 3 demonstrate how the temperature-dependent weight loss and corrosion rate of the mild steel increased. As a result, the effectiveness of palm leaf as inhibitors is reduced by temperature increases. This is explained by the fact that a rise in temperature weakens the interaction between the mild steel and inhibitor molecules. Each inhibitor's efficiency rose with an increase in time, as seen in Tables 4. It is consistent with findings from earlier studies (Singh et al., 2014; Popova et al., 2015; Umoren et al, 2016; Gaya et al, 2019; Bilgiç, 2021; Qurishi et al, 2021) that showed inhibitor efficiency increases with time. The existence of live alkaloids and other phytochemicals in palm leaf may be the cause of their high levels of inhibition efficiency, as demonstrated by the effects of concentration, temperature, and time.

Table 2: Effect of concentration on corrosion control of mild steel in HCl with palm leaf at 313K

Inh. Conc. (g/L)	ΔШo (g)	CR_0 (mg/cm^2hr)	$d\hat{w}_1$ (g)	C_{R1} (mg/cm^2hr)	IE (%)	ϴ
0.0	0.558	20.67				
0.2			0.188	6.963	66.31	0.6631
0.4			0.146	5.407	73.84	0.7384
0.6			0.102	3.778	81.72	0.8172
0.8			0.031	1.148	94.44	0.9444
1.0			0.042	1.556	92.47	0.9247

Table 3: Effect of temperature on corrosion control of mild steel in HCl with palm leaf

Temp. (K)	ΔШo (g)	CR_0 (mg/cm^2hr)	$d\hat{w}_1$ (g)	C_{R1} (mg/cm^2hr)	IE (%)	ϴ
303	0.58	21.48	0.045	1.667	92.24	0.9224
313	0.558	20.67	0.031	1.148	94.44	0.9444
323	0.679	25.15	0.097	3.593	85.71	0.8571
333	0.762	28.22	0.155	5.741	79.66	0.7966
343	0.801	29.67	0.187	6.926	76.65	0.7665

Table 4: Effect of time on corrosion control of mild steel in HCl with palm leaf

Time (hr)	ΔШo (g)	CR_0 (mg/cm^2hr)	$d\hat{w}_1$ (g)	C_{R1} (mg/cm^2hr)	IE (%)	ϴ
1.0	0.129	14.33	0.025	2.778	80.62	0.8062
2.0	0.283	15.72	0.029	1.611	89.75	0.8975
3.0	0.558	20.67	0.031	1.148	94.44	0.9444
4.0	0.553	15.36	0.036	1.000	93.49	0.9349
5.0	0.665	14.78	0.049	1.089	92.63	0.9263

3.3 Activation energy of the corrosion control

Table 5 provides the value of the activation energy (Ea in kJ/mol) of the corrosion control utilizing palm leaf, while Figure 2 is the graph for the determination of activation energy of the corrosion control using palm leaf. It was determined by using the Arrhenius model to fit the experimental data. The Ea for controlling the deterioration of mild steel employing leaf of the palm is 38.35 kJ/mol. The number falls below the 80 kJ/mol threshold value. This observation is consistent with earlier research findings (Omotioma and Onukwuli, 2017; Anadebe et al, 2019) in this area, where decisions on the classification of the adsorption process were facilitated by Ea < 80kJ/mol. The study's findings show that Ea value of 38.35 kJ/mol suggests that the control process using palm leaf is not chemisorption, but a physical adsorption process.

Table 5: Activation energy of the corrosion control using palm leaf

T	K	1/T	Ln k	E_a (kJ/mol)
303	1.667	0.0033	0.51103	38.35
313	1.148	0.0032	0.13802	
323	3.593	0.0031	1.27899	
333	5.741	0.003	1.74763	
343	6.926	0.0029	1.93528	

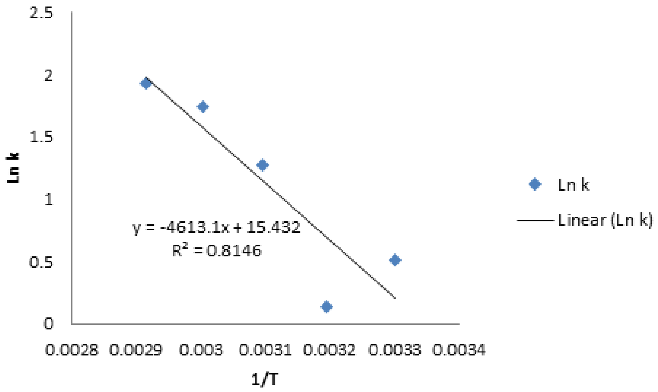

Figure 2: Graph for the determination of activation energy of the corrosion control using palm leaf

3.4 Heat of Adsorption for the Corrosion Control Process

Table 6 displays the adsorption heat (Qads) for the corrosion control employing leaf from palm tree. The Qads for the process has negative outcome for each concentration. This implies that heat and energy were released when the molecules of the palm leaf were adsorbed on the mild steel. This assertion is consistent with the research conducted by Verma et al (2016) and Omotioma et al (2024), which showed that an exothermic process is indicated by a negative heat of adsorption value.

Table 6: Heat of adsorption for the corrosion control using palm Leaf

Concentration (g/L)	Heat of adsorption, Q_{ads} (J/mol) (Palm Leaf)
0.2	-25632.61031
0.4	-28761.71393
0.6	-52487.94285
0.8	-87512.07011
1.0	-77183.92432

3.5 Adsorption Parameters for the Corrosion Control

Table 7 displays the adsorption parameters' result having Figures 3 – 6b as their respective plots. Comparatively, the best fitted isotherm is Langmuir. This claim was supported by the highest recorded average coefficient of determination (R^2) value, which was 0.9945 at 313K and 0.9758 at 323K, respectively, with an average of 0.9925. The value is closest to the critical value of 1 (one) when compared to the R^2 values of the other isotherms (Frumkin, Temkin, and Flory-Huggins). At 313K and 323K, respectively, the attractive parameter (a) values on Temkin analysis are negative, with values of -2.7574 and -3.0447. It demonstrates that the inhibitor and mild steel did not interact chemically. At 313K and 323K, values of the lateral interaction term (α) are 2.7616 and 2.3254 respectively. Each of them has a positive sign, which implies that there was a discernible attraction between the mild steel surface and the molecules of the palm leaf. Positive values of the size attribute (x) were discovered during the analysis of the Flory-Huggins isotherm. This implies the mild steel had a sufficient layer of the inhibitor attached to it. Gibb's free energy has been measured to be less than -40.00 kJ/mol. As a result, the inhibitor's molecules physically absorbed rather than chemisorbing. The findings of the study by Azeez et al (2018) are supported by this investigation.

Table 7: Adsorption parameters for the corrosion control using palm leaf

Adsorption Isotherm	Temperature (K)		K	ΔG_{ads} (J/mol)	Isotherm property	
Langmuir Isotherm	313	0.9945	0.9406	-10294.2		
	323	0.9904	0.8410	-10322.6		
Temkim isotherm	313	0.9212	177.42	-10366.8	a	-2.7574
	323	0.874	163.83	-10407.3		-3.0447
Frumkin Isotherm	313	0.9977	0.0173	-10294.2	α	2.7616
	323	0.978	0.0401	-10322.6		2.3254
Flory-Huggins Isotherm	313	0.7257	4.3371	-10366.8	x	0.5339
	323	0.6993	4.9204	-10407.3		0.9114

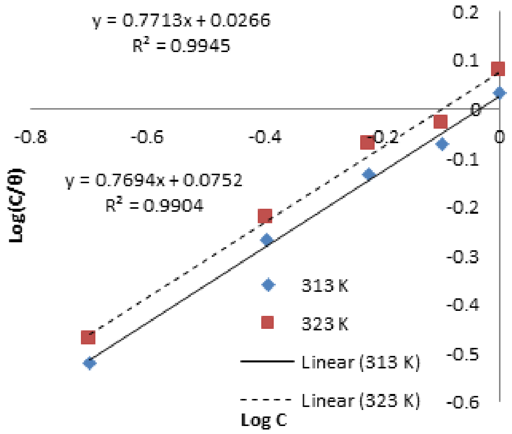

Figure 3: Langmuir plot of the corrosion control of mild steel in HCl using palm leaf

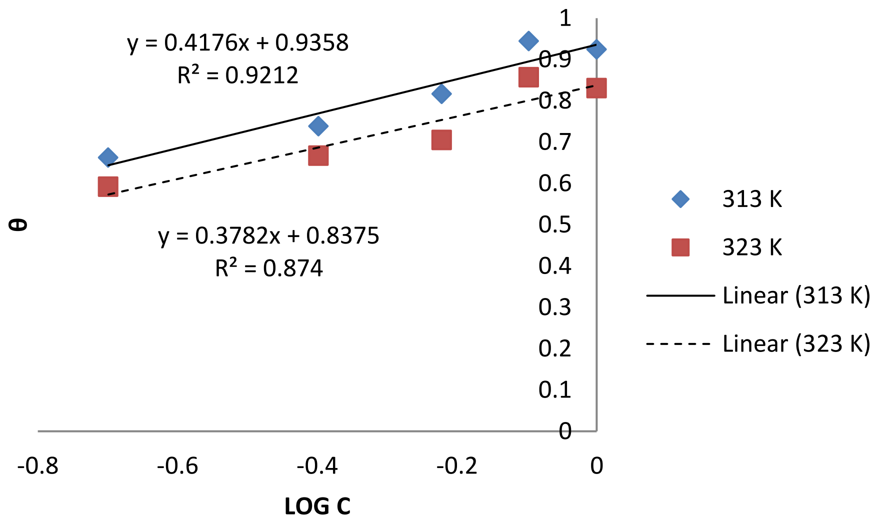

Figure 4: Temkin plot of the corrosion control of mild steel in HCl using palm leaf

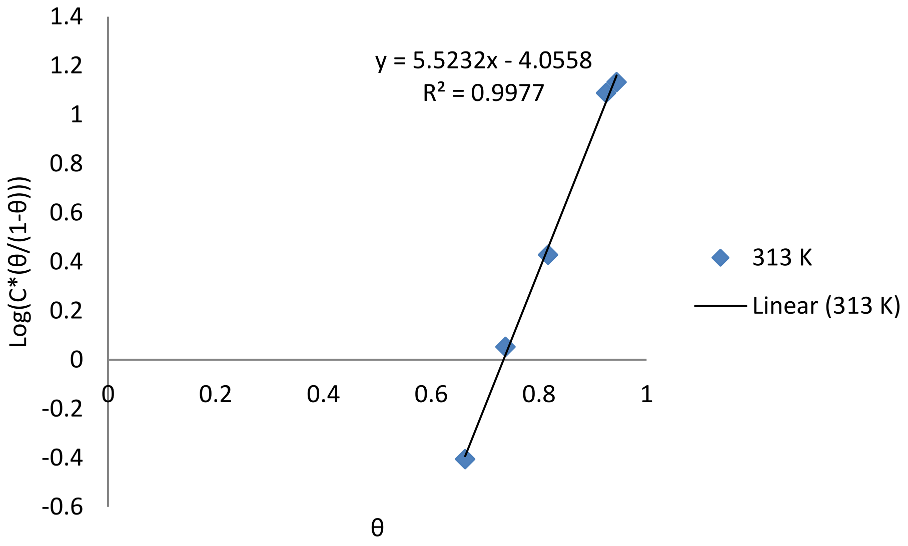

Figure 5a: Frumkin plot (at 313k) of the corrosion control of mild steel in HCl using palm leaf

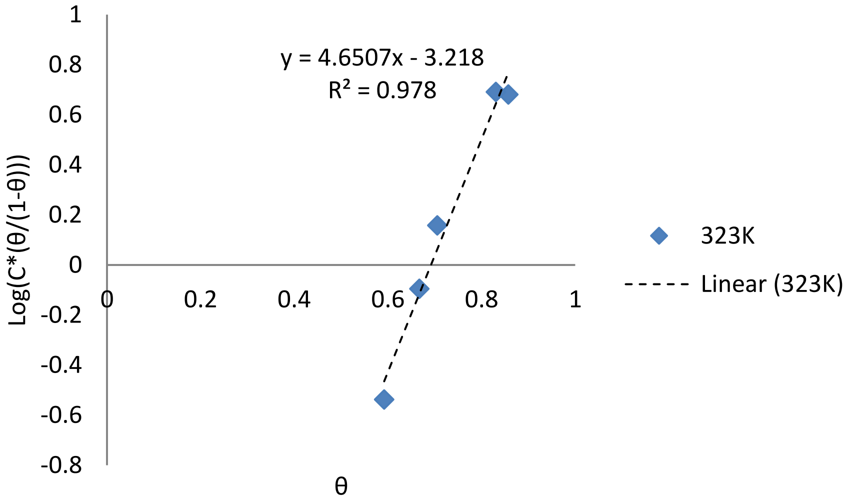

Figure 5b: Frumkin plot (at 323k) of the corrosion control of mild steel in HCl using palm leaf

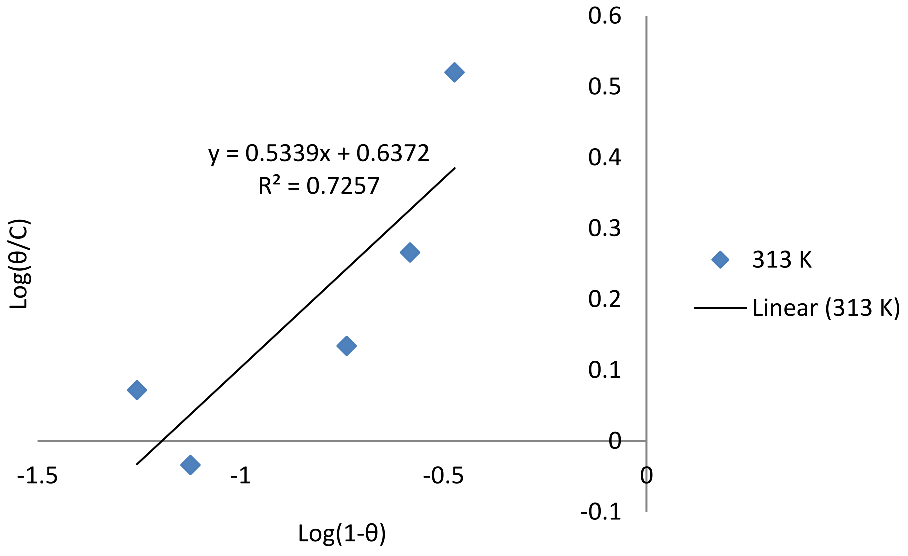

Figure 6a: Flory-Huggins plot (at 313k) of the corrosion control of mild steel in HCl using palm leaf

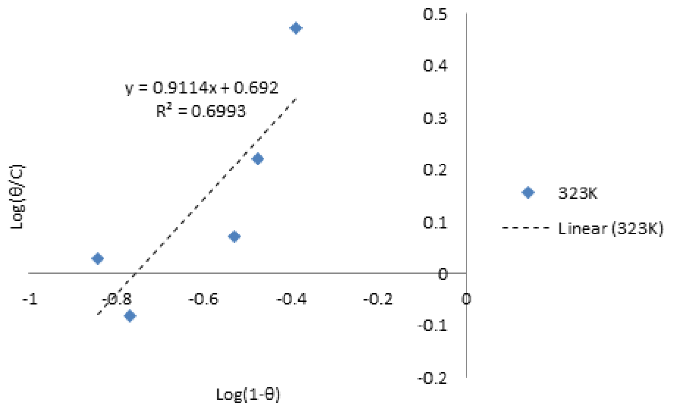

Figure 6b: Flory-Huggins plot (at 323k) of the corrosion control of mild steel in HCl using palm leaf

4.1 Conclusion

The following deductions were made when the experimental findings were analyzed. The phyto-chemical results revealed that the alkaloids in the palm leaf extract were highly concentrated (185.12 ±0.12 mg/100g), while the values of the saponin and tannins were also concentrated (79.55 ±0.02 mg/100g and 97.73 ±0.14 mg/100g, respectively). The extracts have effective anti-corrosion qualities. The core functional groups of the palm leaf were found to be the O-H stretch, CH_3 C-H bend, C-H stretch, N-H bend, C=H stretch, C=O stretch, and C-F stretch. Leaf extract of palm plants contain heteroatoms of nitrogen and oxygen. This implies that it may have anti-corrosion properties. Surface coverage, weight loss, corrosion rate, and inhibition efficiency were all impacted by the inhibitor. Good inhibitory effectiveness was demonstrated by palm tree leaf extracts. It can therefore be used to stop mild steel from corroding in a solution containing HCl. The E_a of the control of mild steel deterioration using palm leaf is 38.35 kJ/mol. The Qads status is negative, indicating that heat moved from the apparently higher temperature inhibitor-mild steel contact to the surrounding. The Langmuir isotherm is the best-fitting isotherm in this physical adsorption of the inhibitor's molecules. Factors of Concentration, temperature and time influenced the efficiency of the palm leaf in a quadratic manner.

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