

Corrosion Inhibition and Adsorption Behaviour of Methanol Extract of *Spondias cytherea* leaves on Mild Steel Corrosion in 5.0M H₂SO₄

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ABSTRACT: The corrosion inhibition of mild steel in 5.0M sulphuric acid solutions by crude extracts of *Spondias cytherea* leaves (S.C) were studied using gravimetric and gasometric techniques. The results showed that S.C. can serve as a corrosion inhibitor in acid medium. Inhibition efficiencies (%I) of the S.C extract were found to reach 67.72 % and 72.00% for gravimetric and gasometric analyses respectively. The corrosion inhibition efficiency increased with increase in the concentration of the extracts and decreased with the increase in time and temperature. The corrosion inhibition efficiency exhibited by the S.C leaves extract is attributed to the stronger adsorption of the phytochemicals present in the plant extracts onto the mild steel. The mechanism of adsorption proposed for the S.C extract is physical adsorption. The corrosion inhibition of the leaves' extracts followed Langmuir and Freundlich isotherm.

KEYWORDS: Mild steel, corrosion, sulphuric acid, *Spondias cytherea* leaves extract, gravimetric, gasometric.

I. INTRODUCTION

Organic compounds have been studied and analysed to investigate their potential as corrosion inhibitors. Most of these studies revealed that almost all organic molecules showing significant inhibition efficiency contained heteroatoms such as nitrogen, sulphur, phosphorous and oxygen [1-3]. Despite of these promising findings about possible corrosion inhibitors, most of these substances are expensive, toxic and non-biodegradable thus causing pollution problems [2]. Hence, there is need to search for alternative, cheap and eco-friendly corrosion inhibitors. The use of plant extracts have continued to attract patronage as alternative corrosion inhibitors as evident in published papers [4]. Plants have been recognized as sources of naturally occurring compounds with complex molecular structures, having varying physical, chemical and biological properties [5-7]. The use of naturally occurring compounds are of interest, because of their cost effectiveness, abundant availability and more importantly their eco-friendly nature.

Spondias cytherea belongs to the Anacardiaceae family [8]. It is mainly cultivated in the Caribbean region, Asia, Central America, South America and, in a lesser proportion, in Africa. The fruit is very important as food for animals and human beings when ripe. The *Spondias* spp., have shown to possess some pharmaceutical properties. *Spondias cytherea* have been identified to contain several phytochemicals which are responsible for the pharmaceutical properties [9-11].

In view of the continued interest on the application of plant extracts for metal corrosion control, this study investigates inhibitive effects of *Spondias cytherea* leaves extracts on the corrosion of mild steel in 5.0M Sulphuric acid (H₂SO₄) solution.

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(An ISO 3297: 2007 Certified Organization)

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II. MATERIALS AND METHOD

Material collection and preparation

The mild steel coupons used in this work were obtained from Urata market, Aba in Abia State, Nigeria. The mild steel sheets used were mechanically cut into 5.0cm x 1.5cm coupons. The surfaces of the mild steel coupons were mechanically polished with series of emery paper (sand paper) of variable grades – 220, 320, 400 and 800 and 1000. These polished mild steel coupons were then washed with distilled water, respectively degreased with methanol and acetone, air-dried and weighed using a digital weighing balance. The weights were recorded and the weighed coupons were used for corrosion study.

Preparation of plant extracts

The *Spondias cytherea* leaves (S.C) were collected from Amune Ovim, Isuikwuato L.G.A., Abia State, Nigeria. They were dried under room temperature away from direct sun light. A mechanical grinder was used to grind the dried leaves into powder form. 100 g of the leave powders were extracted with 4:1 methanol:water (80% methanol) using a soxhlet extractor for 24 hours. The extracts were distilled at 60 °C in a water bath until almost the entire methanol evaporated. 4.0 g of the methanol extracts were digested in 1000 ml of 5.0M H₂SO₄ and left to cool for 24 hours. The resultant solution was filtered and stored in a 1.0 L volumetric flask as the stock solution. The leave extract test solutions of concentration of 0.5 g/L, 1.0 g/L, 2.0 g/L, and 4.0g/L were prepared from the stock solution. These solutions were used for the corrosion inhibition test.

Weight loss technique

The weighed polished mild steel coupons were suspended in beakers containing 100ml of the acidic test solutions (5.0M H₂SO₄) at room temperature. The mild steel coupons were completely immersed in the test solutions and retrieved every hour for 1-5hours. The retrieved coupons were washed, scrubbed with bristle brush under fast flowing water, rinsed in methanol, dried using acetone, and re-weighed. The weight loss of the mild steel was evaluated as the difference in the initial and final weight of the coupons. The experiment was carried out for *Spondias cytherea* leaves (S.C) extract using concentrations of 0.5g/L, 1.0g/L, 2.0g/L and 4.0g/L at ambient temperature and in triplicates. From the weight loss data, the corrosion rates (CR) were calculated using equation (1):

$$CR = \frac{WL}{A \cdot t} \quad (1)$$

Where WL is weight loss in mg, A is the metal surface area and t, the time of immersion in hours. From corrosion rate, the percentage inhibition efficiencies of the plant extracts (I%) were determined using equation (2):

$$I\% = \frac{CR_{blank} - CR_{inh}}{CR_{blank}} \times \frac{100}{1} \quad (2)$$

Where CR_{blank} and CR_{inh} are the corrosion rates in the absence and presence of the plant extracts respectively.

Gasometric technique

The gasometric technique was conducted using a gasometric assembly consisting of a reaction chamber, connected to burette that contained paraffin oil through a reservoir. In determining the rate of hydrogen evolution per surface area, the contribution of other gases, including water vapour at higher temperature is assumed to be insignificant.

In the gasometric measurements the volume of hydrogen evolved was determined following procedures previously describe [12-14]. 100 mL of the corrodant (acidic test solution) was introduced into the reaction chamber, connected to a burette through a delivery tube. A mild steel coupon was dropped into the acidic test solution in the chamber and the reaction vessel was quickly closed to avoid any escape of hydrogen gas. The volume of the hydrogen gas evolved from the reaction was monitored by the depression (in mL) in the level of paraffin oil. This depression in the paraffin oil level was recorded every minute for a maximum of 15 minutes at 303K. The same experiment was repeated in the presence of the inhibitor, *Spondias cytherea* (S.C) leaves extract, with concentrations of 0.5g/L, 1.0 g/L, 2.0 g/L, 4.0g/L. The same procedure was repeated at elevated temperatures of 313, 323 and 333 ± 2 K. The rate of evolution of the hydrogen gas (Rh) was determined from the slope of the graph of volume of the hydrogen gas evolved (VHE) against time (t) and the inhibitors efficiencies (I%) determined using equation (3):

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$$I\% = \frac{R_{h_{blank}} - R_{h_{inh}}}{R_{h_{blank}}} \times \frac{100}{1} \quad (3)$$

Where $R_{h_{blank}}$ and $R_{h_{inh}}$ are the corrosion rates in the absence and presence of the plant extracts respectively.

Activation and adsorption parameters

The activation parameters, activation energy (E_a), change in entropy of activation (ΔS^\ddagger), and change in enthalpy of activation (ΔH^\ddagger) were determined from the slopes and intercepts of the plot of $\ln k$ and $\ln k/T$ versus $1/T$. The values of k were taken to be equal to the rate of hydrogen evolution.¹⁴ The logarithm of corrosion rate (k) is a linear function of the reciprocal of the absolute temperature ($1/T$) (Arrhenius equation) as given in the equation (4)

$$\ln k = \frac{-E_a}{RT} + \frac{\ln A}{1} \quad (4)$$

where E_a is the apparent effective activation energy, T the absolute temperature, R the universal gas constant, and A is Arrhenius pre-exponential factor. An alternative formulation of the Arrhenius equation is the transition state equation as given in equation (5). In this case, logarithm of corrosion rate/absolute temperature (k/T) is a linear function of the reciprocal of the absolute temperature ($1/T$).

$$k = \left(\frac{RT}{Nh}\right) \exp\left(\frac{\Delta S^\ddagger}{R}\right) \exp\left(\frac{\Delta H^\ddagger}{RT}\right) \quad (5)$$

The standard free energy of adsorption, ΔG_{ads}° , which can characterize the interaction of adsorption molecules and metal surface, was calculated by equation (6).

$$\Delta G_{ads}^\circ = -RT \ln (K_{ads} \times 55.5) \quad (6)$$

Where K_{ads} is the equilibrium constant of adsorption from Langmuir isotherm.

The Langmuir and Freundlich adsorption isotherms were tested for fit with the experimental data. Langmuir isotherm is given by the expression:

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

The Freundlich isotherm is given by the expression:

$$\text{Log } \theta = \text{Log } K + n \text{Log } c \quad (8)$$

Where θ is the surface coverage, c is the concentration, K_{ads} is the equilibrium constant of adsorption process.

Preliminary phytochemical screening

Phytochemical screening was carried out on the leaves following the methods described by Kumar *et al.*, Parekh and Chanda, and Onwukaeme *et al* [15-17]. The leaves were screened for alkaloids, saponins, tannins, flavonoids, steroids.

III. RESULTS AND DISCUSSION

Weight loss measurement

Different concentrations of the inhibitor were used to gain insight on the corrosion behaviour of mild steel. The Corrosion rates ($\text{mg}/\text{cm}^2\text{hr}$) and inhibition efficiencies (%) for weight loss of mild steel in 5.0M H_2SO_4 in the absence and presence of different concentrations of S.C are shown in Table 1.

Table 1: Corrosion rates ($\text{mg}/\text{cm}^2\text{hr}$) and inhibition efficiencies (%) for weight loss of mild steel in 5.0M H_2SO_4

	Corrosion rate ($\text{mg cm}^{-2}\text{hr}^{-1}$)					Inhibition efficiency (%)			
	Blank	0.5g/L	1.0g/L	2.0g/L	4.0g/L	0.5g/L	1.0g/L	2.0g/L	4.0g/L
1hr	26.39	18.29	16.00	11.43	8.52	30.71	39.37	56.69	67.72
2hr	29.51	22.44	18.60	14.13	11.01	23.94	36.97	52.11	62.68
3hr	26.95	21.75	20.85	17.25	12.12	19.28	22.62	35.99	55.01
4hr	25.82	24.21	21.15	16.57	12.21	6.24	18.11	35.81	52.72
5hr	27.10	21.11	20.70	16.00	13.09	22.09	23.62	40.95	51.69

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Inspection of Table 1 showed that the corrosion rate decreases with increase in the extract concentration while inhibition efficiency increased with increase in extract concentration, an indication that the extracts inhibited corrosion of mild steel in 5.0M H₂SO₄. However, the inhibition efficiency decreased over time. This observed trend may result from the fact that adsorption and surface coverage increase with the increase in concentration; thus, the surface is separated from the medium [4]. The S.C. extract showed significant inhibition efficiency on mild steel in 5.0M H₂SO₄ solutions that reached up to 67.72% for extract concentration at 4.0g/L.

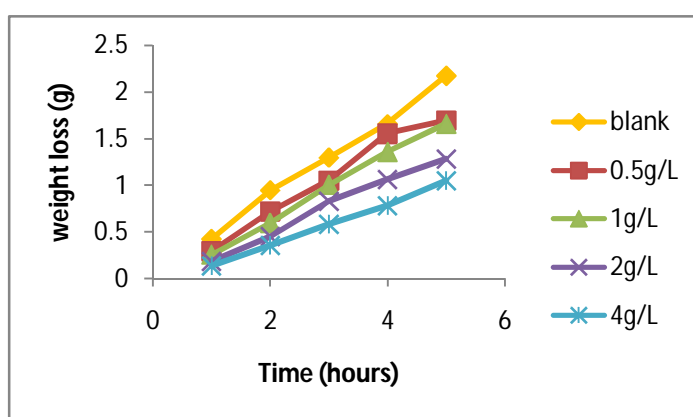


Figure 1: Variation of weight loss with time for the corrosion of mild steel in 5.0 M H₂SO₄ solution in the absence and presence of different concentrations of S.C

From Figure 1 it is seen that weight loss of mild steel increased with time, an indication that the rate of corrosion of mild steel in acid medium increases with time.

In this study of corrosion of mild steel in H₂SO₄ solutions, log (W_o/W_f) was plotted against time and a linear variation was observed. This confirmed first-order reaction kinetics with respect to mild steel in H₂SO₄ solutions, formulated as:

$$\text{Log } W_f = \text{Log } W_o - kt \tag{8}$$

Where W_o is the initial weight before immersion, W_f weight after immersion at time t, k is the rate constant and t is time.

Table 2: Values for the rate constant and half life

	k	t _{1/2}
Blank	0.199	3.487
0.5g/l	0.072	9.625
1.0g/l	0.061	11.323
2.0g/l	0.055	12.600
4.0g/l	0.043	16.116

The values of the rate constants, k, obtained from the slopes of the plots in Figures 2 and the half-life, t_{1/2}, are presented in Table 2. The values obtained for the rate constant are approximately 0.1 in all cases, an indication that it is a first order reaction [18]

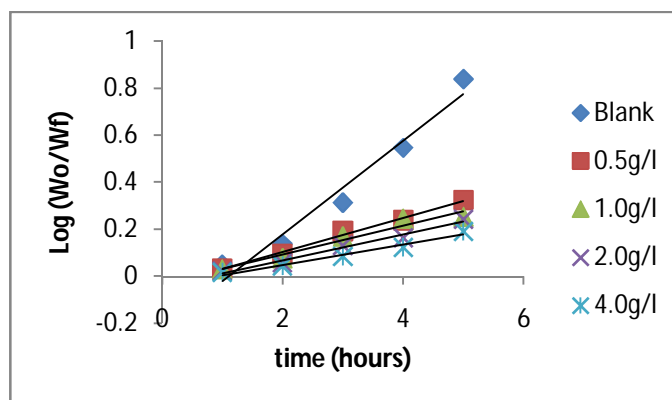


Figure 2: Plot of $\text{Log}(W_o/W_t)$ against time for S.C leaves extract in 5.0M H_2SO_4

The results obtained revealed that the rate constant decreases with increase in the concentration of the leave extracts. From the rate constant values, the half-life values, $t_{1/2}$, of the metal in the test solutions were calculated using equation 9:

$$t_{1/2} = \frac{0.693}{k} \quad (9)$$

The half-life values are also presented in Table 2. The half-life values were observed to increase with increase in concentration of the leave extracts, indicating decrease in the dissolution rate of the metal in the solutions with increase in the leave extract concentration [18].

Gasometric results

The rate of evolution of the hydrogen gas was determined from slope of the linear portion of the graph of volume of hydrogen gas evolved versus time. The values for the rate of hydrogen evolution and inhibition efficiency are shown in Table 3.

Table 3: Gasometric data for rate of hydrogen evolution (mL/min) and inhibition efficiency (%) on mild steel corrosion in 5.0M H_2SO_4 solution.

Temperature(K)	Rate of hydrogen evolution(mL/min)					Inhibition efficiency (%)			
	Blank	0.5g/L	1.0g/L	2.0g/L	4.0g/L	0.5g/L	1.0g/L	2.0g/L	4.0g/L
303	2.92	1.45	1.36	1.37	1.07	50.23	53.27	53.07	63.37
313	6.69	3.27	2.78	2.62	1.87	51.16	58.49	60.81	72.00
323	8.82	6.73	6.28	4.37	3.33	23.73	28.82	50.49	62.23
333	11.25	10.10	9.11	7.25	5.33	10.22	19.03	35.52	52.58

From Table 3 it was observed that the presence of S.C extract decreased the rate of hydrogen evolution, and consequently decreased the corrosion rate of the mild steel in 5.0 M H_2SO_4 solutions compared to the blank. The decrease was also dependent on the concentration of the S.C extract and temperature. Further inspection of Table 3 showed that the rate of hydrogen evolution increased with increase in temperature and decreased with increase in the concentration of the plant extracts. From the rate of hydrogen evolution, the inhibition efficiencies were determined and are also presented in Table 3. It was observed that, except for 303K, the inhibition efficiency increased relatively with increase in the concentration of the plant extracts and decreased relatively with increase in temperature. The maximum inhibition efficiency observed was 72.00%. These observations are indication that the leaves extract inhibited corrosion of mild steel in 5.0M H_2SO_4 .

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Activation parameters

Table 4: Calculated values of activation energy (E_a), change in enthalpy of activation (ΔH^\ddagger), change in entropy of activation (ΔS^\ddagger), Average E_a and $E_a - \Delta H^\ddagger$

Extract concentration (g/L)	E_a	Average E_a	ΔH^\ddagger	$E_a - \Delta H^\ddagger$	ΔS^\ddagger	ΔG_{ads}°
Blank	36.58		33.94	2.64	-122.85	-12.41
0.5	55.06		53.84	1.22	-63.80	-12.61
1.0	54.79	50.36	52.15	2.64	-70.08	-9.14
2.0	46.29		43.65	2.64	-98.17	-7.02
4.0	45.32		42.68	2.64	-103.65	-12.41

The values of E_a , ΔS^\ddagger , ΔH^\ddagger , Average E_a and $E_a - \Delta H^\ddagger$ for the corrosion of mild steel with S.C leaves extract in 5.0M H_2SO_4 are presented in Table 4. The values of k were taken to be equal to the rate of hydrogen evolution [12, 19].

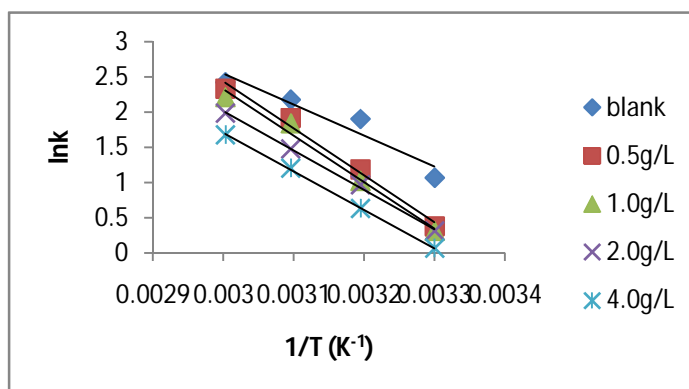


Figure 3: Variation of $\ln k$ vs. $1/T$ for mild steel corrosion in the presence of different concentrations of S.C extracts in 5.0M H_2SO_4

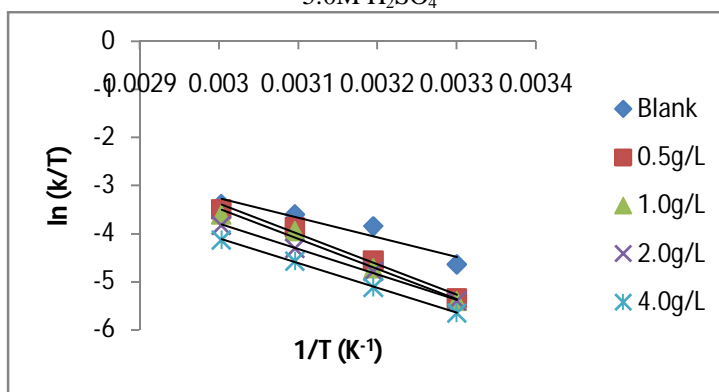


Figure 4: Variation of $\ln(k/T)$ vs. $1/T$ for mild steel corrosion in the presence of different concentrations of S.C extracts in 5.0M H_2SO_4

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Figure 3 showed the plot of $\ln k$ versus $1/T$ for mild steel corrosion in 5.0M H_2SO_4 with different concentrations of the S.C leaves extract. Figure 4 showed the plot of $\ln(k/T)$ versus $1/T$ for mild steel corrosion in 5.0M H_2SO_4 with different concentrations of the S.C leaves extract. The activation parameters, activation energy (E_a), entropy of activation (ΔS^\ddagger), enthalpy of activation (ΔH^\ddagger) were determined from the slopes and intercepts of the plot of $\ln k$ and $\ln k/T$ versus $1/T$. As seen, the plots in Figures 3 and 4 gave straight lines.

The results showed that E_a values in the presence of S.C extract were higher than that of the blank, which is typical of physical adsorption [20]. Inspection of the results as given in Table 4 revealed that E_a and ΔH^\ddagger values decreased as the concentration of the extract increased, meaning a lower protection efficiency was attained due to increase in the energy barrier for the reaction. The positive values of ΔH^\ddagger was an indication that the processes were endothermic. The average values for the activation energy of the inhibitors reported was $50.36 \text{ kJ mol}^{-1}$ (Table 4). This value confirmed the assertion that the inhibition of mild steel corrosion by the extract is by physical adsorption mechanism, which is in agreement with those of Barrow (1983) [21]. According to the author, the activation energy for physical adsorption mechanism should be less than 80 kJ mol^{-1} . The higher E_a values in the presence of inhibitor compared to the blank solution indicated that the inhibitor would be effective at low temperatures, but efficiencies would be diminished at higher temperatures [22]. The higher values for ΔH^\ddagger in the presence of the plant extracts were indicative of the higher protection efficiency observed for the system [23]. The values of ΔG_{ads}° obtained are presented in Table 4. The negative values of ΔG_{ads}° ensure the spontaneity of adsorption process and stability of the adsorbed layer on the mild steel surface. The results obtained for ΔG_{ads}° are negative in all cases and are less than 20 kJ/mol . This further showed that the adsorption process is by physical adsorption. Generally, the values of ΔG_{ads}° around -20 kJ/mol or lower are consistent with physisorption, while those around -40 kJ/mol or higher involve chemisorptions [24]. This implies that the plant extracts adheres on the surface of the corroding system and so gives a very strong inhibitor. It was found that the difference, $E_a - \Delta H^\ddagger$, (Table 4) except in 0.5 g/L , were 2.64 kJ/mol which is very close to the value of RT (2.6390 kJ/mol) at the average temperature (T) studied (318 K). This result indicated that the corrosion process is a unimolecular reaction characterized by equation (10) [25-26].

$$E_a - \Delta H = RT \tag{10}$$

In addition, the entropies of the activation in the presence of the inhibitors were negative. Negative values of entropies of activation implied that the activated complex in the rate determining step represents an association rather than a dissociation step, meaning that a decrease in disordering takes place on going from reactants to the activated complex [20].

Adsorption considerations

The experimental data were subjected to Langmuir and Freundlich adsorption isotherm equations. The Langmuir and Freundlich plots for S.C. in 5.0M H_2SO_4 are shown in Figures 9 and 10 respectively. The adsorption parameters, K_{ads} , K and R^2 are presented in Table 5.

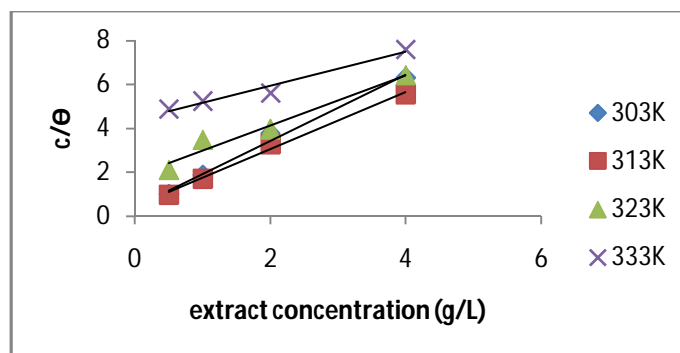


Figure 9: Langmuir plot for S.C in 5.0M H_2SO_4

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From Figure 9, linear plots were obtained with good correlation coefficient which seemed to suggest that adsorption of the extracts followed Langmuir adsorption isotherms.

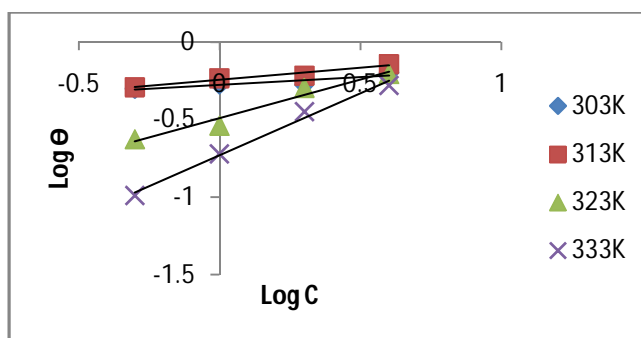


Figure 10: Freundlich plot for S.C extract in 5.0M H₂SO₄

Linear plots were also obtained in Figure 10, with good correlation coefficient which seemed to suggest that a Freundlich adsorption isotherm was followed in the adsorption process.

Figure 9 and 10 suggest that adsorption of the extracts followed Langmuir and Freundlich adsorption isotherms. However, a comparison of the correlation coefficients of the Langmuir and Freundlich isotherms as presented in Table 6 showed that the data fitted more to Langmuir than Freundlich isotherm.

Table 5: Langmuir and Freundlich adsorption isotherm parameters

	Langmuir Isotherm		Freundlich Isotherm	
	R ²	k _{ads}	R ²	K
303K	0.9901	2.4876	0.7871	0.5291
313K	0.9933	2.2894	0.9523	0.5704
323K	0.9634	0.5413	0.9585	0.3221
333K	0.9704	0.2273	0.9897	0.1862

Langmuir isotherm postulate monolayer adsorption of the adsorbate onto the adsorbent which was expected to have a correlation coefficient of unity [27]. The R² value was also an indication that the leaves extract components are approximated by Langmuir adsorption and the monolayer of the extract species must have been attached to mild metal surface without lateral interaction between the adsorbed species. The values of K_{ads} were seen to decrease as the temperature rises from 303K to 333K. This further confirms that the leaves extracts were physically adsorbed on the surface of the mild steel [28].

Preliminary phytochemical results

The results from the phytochemical screening of the extracts are shown in Table 6. It was revealed from the result that the plant extracts contained appreciable quantities of phytochemicals (alkaloids, flavonoid, saponins, and tannins), which might be the reason for the increased inhibitions efficiencies of the plant extracts.

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Table 6: Preliminary Phytochemical screening

Phytochemical	S.C
Alkaloids	+
Flavonoids	+
Steroids	-
Saponins	+
Tannins	+

- = absent
+ = present

IV. CONCLUSION

80% Methanol leaves extracts of *Spondias cytherea* (S.C), was found to inhibit the corrosion of mild steel in 5M H₂SO₄. S.C showed an inhibition efficiency of 67.72 % and 72.00% for gravimetric and gasometric analysis respectively. The corrosion inhibition efficiency increased with increase in the concentration of the extracts and decreased with the increase in time and temperature. The mechanism of adsorption proposed for S.C extracts was physical adsorption as it was evidenced from the activation parameters that the E_a of the blank was lower than the E_a in the presence of the plant extracts. Furthermore, the decreasing values of the k_{ads}, from the Langmuir isotherm, as the temperature increased also asserted that the mechanism of adsorption was by physical adsorption. The inhibitory property of the S.C leaves extract could be attributed to the phytochemicals present as the preliminary phytochemical analysis showed that it contained alkaloids, flavonoids, saponins and tannins. The corrosion inhibition of the extract followed Langmuir and Freundlich isotherm.

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