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Review Article

Green Corrosion inhibition behavior of *Spondias cytheria* leaves extract and its Synergism with Chloride Ions on Mild Steel in H_2SO_4

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Abstract

The synergistic inhibition behavior of *Spondias cytheria* and chloride ions on mild steel in H_2SO_4 solution were studied using the gravimetric and gasometric techniques. From the investigation the results showed that *Spondias cytheria* and KCl are good corrosion inhibitors. The leaf extract showed maximum inhibition efficiency of 88.52% and 81.42% for gravimetric and gasometric analysis respectively. The leaf extracts + KCl showed maximum inhibition efficiency of 91.49% and 83.76% for gravimetric and gasometric analysis respectively. It was generally observed that the addition of KCl improved the inhibition of the leaf extract. Considering the E_a, K_{ads}, ΔG_{ads}, Q_{ads}, the process of corrosion inhibition is attributed to be by physical adsorption. Comparing the R^2 values, it could be observed that the data fitted well to Freundlich for the leaf extract and Langmuir for the leaf extract + KCl.

Keywords: Corrosion Inhibition, Synergism, Mild steel, plant extract.

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INTRODUCTION

The industrial and domestic application of mild steel is extensive and cannot be over emphasized. (Oguzie *et al.*, 2006). Mild steel is used in the construction of 90% of the hardware used in production in various industries. The conditions under which these hardware operates tend to become more corrosive hence requiring a more effective corrosion control management strategy. This has been a major issue globally in the industrial sector, the issue of corrosion keeps growing resulting to billions of dollars being spent by various companies annually on corrosion management (Obike *et al.*, 2018). Corrosion and material scientists are relentless in seeking better and more efficient ways of combating the corrosion of metals (Orubite *et al.*, 2007). Inhibitors from studies has proven to be the most practical methods for protection against corrosion especially in acid solutions to prevent metal dissolution and acid consumption (Chauhan, and Gunasekaran, 2007; Obike *et al.*, 2016).

The choice of the appropriate inhibitor for a particular application is restricted by several factors. One factor is the vast variety of possible corrosion systems coupled with the specificity of action of most acid inhibitors which often necessitates the use of a combination of additives to provide multiple effects required for effective corrosion inhibition. Synergism is an effective method to improve the inhibitive performance, to decrease the amount of usage, to diversify the application of inhibitor in acidic media (Abdel *et al.*, 2008). Here present the effect chloride ions on the inhibition behaviour of Spondias cytheria in a corrosive media of conc H_2SO_4.

EXPERIMENTAL MATERIAL PREPARATION

Metal Specimen

The mild steel coupons of 5.0cm x 1.5cm x 0.8cm were obtained from Urata market, Aba in Abia State, Nigeria. 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, degreased with methanol, dried with acetone and weighed using a digital weighing balance respectively. The weights were recorded and the weighed coupons were used for the corrosion studies.

Reagent and plant extracts

The leaves and stem bark of Spondias cytherea were collected from Amune Ovim, in Isuikwuato L.G.A of Abia State, Nigeria and identified by a botanist from the department of Plant Science and Biotechnology of Abia State University, Uturu. They were dried under room temperature away from direct sun light. A mechanical grinder was used to grind the dried plant materials into powder form. 100 g of the powders was extracted with methanol using a soxhlet extractor for 24 hours. The extracts were distilled at 333 K in a water bath until almost the entire methanol evaporated. 4.0 g of the methanol extracts was digested in 1.0 L of 1.0 M H_2SO_4 and left to cool for 24 hours. The resultant solution was filtered and stored in a 1.0 L volumetric flask. The extracts' test solutions of 1.0 g/L, 2.0 g/L, and 3.0 g/L concentrations were prepared as above. Furthermore, 4.0 g of the methanol extracts was digested with 0.4 g of KCl in 1.0 L of 1.0 M H_2SO_4 and left to cool for 24 hours. The resultant solution was filtered and stored in a 1.0 L volumetric flask. The extracts' test solutions of "1.0 g/L + 0.1g KCl", "2.0 g/L + 0.2 g KCl", and 3.0 g/L + 0.3g KCl" concentrations were prepared as above. Likewise, KCl test solutions of 0.1 g/L, 0.2 g/L, 0.3 g/L and 0.4 g/L were also prepared. These solutions were then used for the corrosion inhibition test in acidic medium.

Gravimetric technique

The weighed polished mild steel coupons were suspended in beakers containing 100 mL of the acidic test solutions (1.0 M H_2SO_4) at room temperature. The mild steel coupons were immersed completely in the test solutions and retrieved every day for five days. 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 experiments were carried out using the test solutions of the extracts, extracts + KCl and KCl at ambient temperature and in triplicates. From the weight loss data, the corrosion rates (CR) was calculated using equation 1:

$$CR = \frac{WL}{A.t} \qquad (1)$$

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 will be determined using equation 2:

$$I\% = \frac{CR_{blank} - CR_{Inh}}{CR_{blank}} \times 100 \qquad (2)$$

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

Gasometric Technique

In the gasometric measurements the volume of hydrogen evolved was determined following procedures previously describe (Umoren *et al*., 2011., Obike *et al*., 2016). 100 mL of the test solutions (corrodants) was introduced into a 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 closed quickly 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 333 K. The same experiments were repeated using the test solutions of the extracts, extracts + KCl and KCl at elevated temperature of 353 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 (mL) versus time (min) and the inhibition efficiency, I (%) determined using equation 3:

$$I\ (\%) = \frac{Rh_{blank} - Rh_{Inh}}{Rh_{blank}} \times 100 \qquad (3)$$

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

Kinetics studies

In this study, log (W_o/W_f) was plotted against time. This conforms to the first-order reaction kinetics, formulated as:

$$\text{Log } W_f = \text{Log } W_o - kt \qquad (4)$$

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

The values of the rate constants, k, were obtained from the slopes of the plots of log (W_o/W_f) against time.

From the rate constant values, the half-life values, $t_{1/2}$, of the metal in the test solutions were calculated using equation 5:

$$t_{1/2} = \frac{0.693}{k} \qquad (5)$$

Synergistic effect

The synergistic parameter (S_1) was calculated by using the relation given by Murakawa *et al*., (1967) for describing the combined inhibition behaviour of amines and halide ions. Generally, for the interaction of inhibitors A and B, this synergism parameter (S_1) is defined as follows:

$$S_1 = \frac{1 - \eta_A - \eta_B + \eta_A \eta_B}{1 - \eta_{AB}} \qquad (6)$$

Whereη_A and η_B are the inhibition efficiencies observed with compound A and B, respectively, acting alone, η_{AB} is the experimentally observed inhibition efficiency for the combination of A and B.

Thermodynamic Studies and Adsorption Parameters

In examining the effect of temperature on the corrosion process, the activation (E_a) was calculated using the condensed Arrhenius equation (equation 7):

$$E_a = 2.303R\left[\text{ Log}\left[\frac{R_2}{R_1}\right] \times \frac{T_1T_2}{T_2 - T_1}\right] \qquad (7)$$

Where R_1 and R_2 are the corrosion rates at temperatures T_1 and T_2 respectively, and R is the molar gas constant.

An estimate of the heat adsorption will be obtained from the trend of surface coverage (θ) with temperature as follows:

$$Q_{ads} = 2.303R\left[\text{Log}\left[\frac{\theta_2}{1-\theta_1}\right] - \text{Log}\left[\frac{\theta_1}{1-\theta_1}\right] \times \left[\frac{T_1T_2}{T_2-T_1}\right]\right] \qquad (8)$$

Where θ_1 and θ_2 are the degrees of surface coverage at temperature T_1 and T_2 respectively.

The Langmuir, Fruendlich and Temkin adsorption isotherms were tested for fit with the experimental data.

Langmuir, Fruendlich and Temkin isotherms are given by the expressions in equation 9, 10 and 11 respectively:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad (9)$$

$$\text{Log}\ \theta = \text{Log}\ K_f + n\text{Log}\ C \qquad (10)$$

$$-\ln C = 2a\theta - \text{In}K_{ads} \qquad (11)$$

Where θ is the surface coverage, C is the concentration, K_{ads} and K_f are the equilibrium constant of adsorption process from Langmuir and Freundlich isotherm respectively.

The Gibbs free energy was calculated using equation 12:

$$\Delta G^\circ_{ads} = -2.303RT\text{Log}\,(55.5K_{ads}) \qquad (12)$$

RESULTS AND DISCUSSION

Gravimetric Analysis

Table-1: Corrosion rates (mg/cm^{2}hr) and inhibition efficiencies (%) of S.P leaf, KCl and S.P + KCl for mild steel in 1.0M H_2SO_4

	Time	**Corrosion rate(mg/cm^{2}hr)**					**Inhibition Efficiency (%)**			
	(Hours)	**Blank**	**1.0g/L**	**2.0g/L**	**3.0g/L**	**4.0g/L**	**1.0g/L**	**2.0g/L**	**3.0g/L**	**4.0g/L**
SP leaf + KCl 1.0M	4 hours	2.8055	0.9352	0.6234	0.3117	0.3117	66.6700	77.7800	88.8900	88.8900
	8 hours	3.6627	1.0910	0.7014	0.4676	0.3117	70.2100	80.8500	87.2300	91.4900
	12 hours	3.1692	0.9871	0.8832	0.5715	0.3117	68.8500	72.1300	81.9700	90.1600
	16 hours	4.9356	2.0781	1.0910	0.7273	0.5715	57.8900	77.8900	85.2600	88.4200
	20 hours	4.2394	1.7768	0.9040	0.7170	0.6234	58.0900	78.6800	83.0900	85.2900
SP Leaf 1.0M	4 hours	0.4676	0.6105	0.5282	0.6170	0.7066	50.0000	72.2200	72.2200	77.7800
	8 hours	0.0037	0.0015	0.0012	0.0007	0.0005	59.5700	68.0900	80.8500	85.1100
	12 hours	0.0032	0.0014	0.0009	0.0006	0.0004	57.3800	72.1300	80.3300	88.5200
	16 hours	0.0049	0.0020	0.0015	0.0009	0.0006	60.0000	69.4700	81.0500	88.4200
	20 hours	0.0042	0.0017	0.0010	0.0008	0.0006	58.8200	77.2100	81.6200	86.7600
KCl	4 hours	0.4676	0.2598	0.2078	0.1818	0.1559	44.4400	55.5600	61.1100	66.6700
	8 hours	0.6105	0.2857	0.2338	0.1818	0.1299	53.1900	61.7000	70.2100	78.7200
	12 hours	0.5282	0.2598	0.2165	0.1732	0.1299	50.8200	59.0200	67.2100	75.4100
	16 hours	0.6170	0.3117	0.2273	0.1559	0.1169	49.4700	63.1600	74.7400	81.0500
	20 hours	0.7066	0.3325	0.2494	0.1766	0.1455	52.9400	64.7100	75.0000	79.4100

Inspection of Table 1 indicated that inhibition efficiency and corrosion rate increased and decreased respectively with increase in extracts concentration. Samuel *et al*., (2015) in their investigation of the inhibitory action of plant extract observation which is in line with the increase and decrease of the inhibition efficiency and corrosion rate respectively as seen in the data presented in table 1. However, it was observed that the inhibition efficiency apparently decreased over time similarly because the inhibitor is being used up. The desorption of the plant constituents from the metal surface can be possibly attributed to the decreased in inhibition of efficiency and increase in corrosion rate of the plant extract on the metals coupons (Saedah, 2013). At 4.0 g/L and 8 hours for S.P. leaf extract + KCl, a maximum inhibition efficiency of 91.49% was recorded. At 4.0 g/L and 12 hours for S.P leaf extract, a maximum inhibition efficiency of 88.52% was recorded. At 4.0 g/L and 16 hours for the KCl, a maximum inhibition efficiency of 81.05 % was recorded. It was generally observed that the inhibition efficiency followed the trend S.P leaf extract + KCl > S.P leaf > KCl.

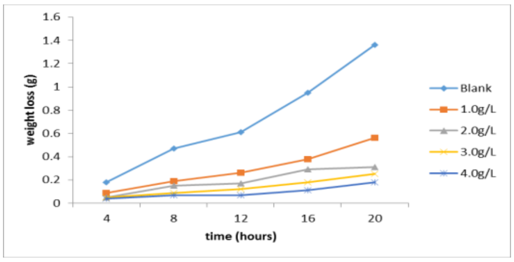

Fig-1: Variation of weight loss against time for Spondias cytherea leave extract in 1.0M H_2SO_4

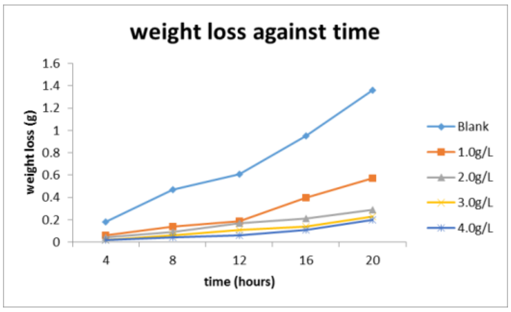

Fig-2: Variation of weight loss against time for Spondias cytherea + Potassium Chloride in 1.0M H_2SO_4

Figures 1 and 2, showed that weight loss of mild steel increased with time which indicated that the corrosion of mild steel in 1.0M increased with time, a confirmation that corrosion increases over time. But the weight loss was reduced in the presence of the inhibitors as compared to the acid solution (Blank). This is an indication that the extracts inhibited corrosion (Obike *et al*., 2016). The inhibitive action of leaf extract towards the acid corrosion of mild steel can be attributed to the adsorption of the leaf and stem extract components onto the steel surface. Corrosion inhibition is initiated by the displacement of adsorbed water molecules by the inhibitor species leading to specific adsorption on the metal surface (Solmaz *et al*., 2008).

Synergism between Spondias cytherea and KCl

The inhibition efficiency of the Spondias cytherea leaf extract increased on the addition of KCl and could be ascribed to a synergistic effect (Eduok *et al*., 2010). It is generally accepted that the presence of halide ions in acidic media synergistically increases the inhibition efficiency of some organic compounds. It is thought that the anions are able to improve adsorption of the organic cations in solution by forming intermediate bridges between the metal surface and the positive end of the organic inhibitor. Corrosion inhibition synergism then results from increased surface coverage arising from ion-pair interactions between the organic cations and the anions (Eduok *et al*., 2010).

Table-2: Synergism parameter (S1) for different concentrations of S.P leaf + KCl

	S1			
	1.0g/L	2.0g/L	3.0g/L	4.0g/L
1day	1.0526	1.2170	1.1264	1.1264
2days	1.0922	1.0800	1.1234	1.0706
3days	1.0967	1.1843	1.1620	1.1470
4days	1.1851	1.0821	1.0749	1.0843
5days	1.1030	1.1609	1.0806	1.0872

GASOMETRIC ANALYSIS

Table-3: Rate of hydrogen evolution and inhibition efficiency for S.P leaf extract and S.P leaf + KCl in 1.0 M H_2SO_4

		Rate of hydrogen evolution					**Inhibition Efficiency (%)**			
	Temp. (K)	**Blank**	**1.0g/L**	**2.0g/L**	**3.0g/L**	**4.0g/L**	**1.0g/L**	**2.0g/L**	**3.0g/L**	**4.0g/L**
S.P Leaf	**333**	1.66	0.93	0.69	0.4	0.31	43.90	58.69	75.83	81.42
	353	4.28	3.22	2.47	2.23	1.52	24.74	42.34	48.04	64.57
S.P. Leaf + KCl	**333**	1.66	0.45	0.33	0.32	0.27	73.00	79.92	80.82	83.76
	353	4.28	1.52	1.39	1.22	1.02	64.57	67.46	71.50	79.14

The values in the Table 3 showed that the rate of hydrogen evolution increased with increase in temperature and reduced with increase in the concentration of the plant extracts. The decrease was also dependent on the concentration of the plant extract and temperature (Obike *et al*., 2016). It was observed that the inhibition efficiency increased with increase in the concentration of the plant extracts and decreased with increase in temperature. This trend as observed earlier may be an implication that adsorption and surface coverage increases with the increase in the inhibitors concentration, thus, separating the surface of the metal from the medium (Abdallah *et al*., 2006; Rehim *et al*., 2008; Zhao and Mu, 1999).

The maximum inhibition efficiency for S.P leaf extract was observed to be 81.42 % at 4.0g/L at 333K. The maximum inhibition efficiency observed for the S.P leaf + KCl is 83.76% at 4.0 g/L and 333K. This also points to the fact that this leave extract inhibits corrosion but the addition of KCl improved the inhibition of the leaf extract.

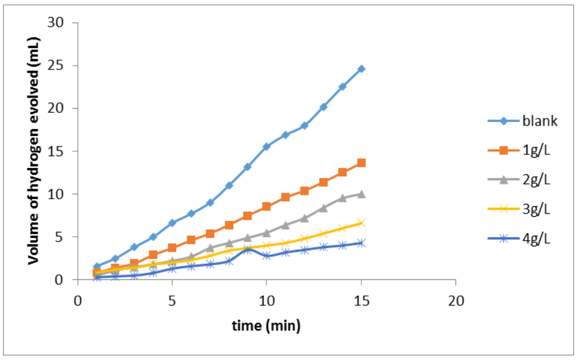

Fig-3: Plot of volume of hydrogen evolved (mL) against time (min) for Spondias cytherea leave extract in 1.0M H_2SO_4 at 333K

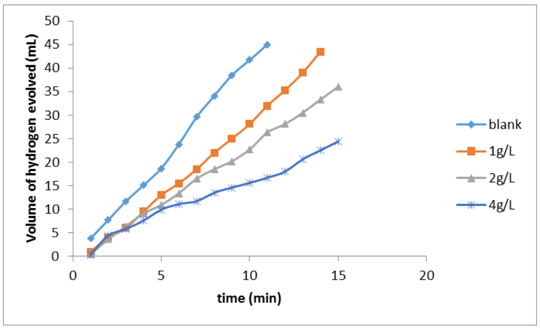

Fig-4: Plot of volume of hydrogen evolved (mL) against time (min) for Spondias cytherea leave extract in 1.0M H_2SO_4 at 353K

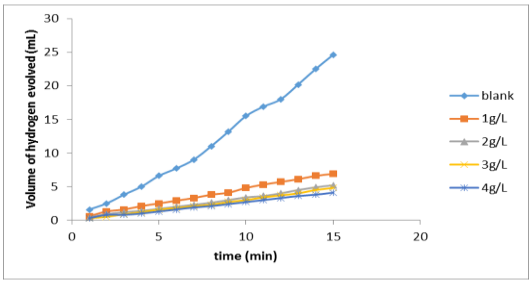

Fig-5: Plot of volume of hydrogen evolved (mL) against time (min) for Spondias cytherea + potassium chloride in 1.0M H_2SO_4 at 333K

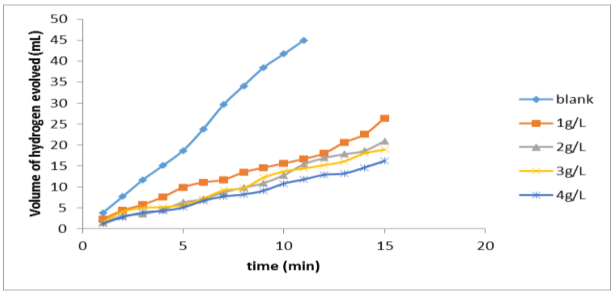

Fig-6: Plot of volume of hydrogen evolved (mL) against time (min) for Spondias cytherea + Potassium Chloride in 1.0M H_2SO_4 at 353K

Figures 3 to 6 showed the variation of volume of Hydrogen evolved (mL) with time (min) for mild steel in 1.0M H_2SO_4 at 333K and 353K respectively using the leaf extract and the leaf + KCl. It could be observed that the volume of hydrogen evolved increases with increase in time, an indication that corrosion rate increases over time.

THERMODYNAMIC PARAMETERS

Table-4: Calculated values of E_a and Q_{ads} for Spondias cytherea leaf extract and Spondias cytherea leaf extract + KCl in 1.0M H_2SO_4.

	SP			SP + KCl		
	E_a	Average E_a	Q_{ads}	E_a	Average E_a	Q_{ads}
Blank	46.2476			46.2476		
1g/L	60.6023	71.1474	11.9849	59.5346	65.0053	-48.6053
2g/L	62.5410		-17.1608	69.8307		-67.4868
3g/L	83.6482		-55.8674	65.6003		-70.2802
4g/L	77.7979		-72.1998	65.0558		-80.1791

The results showed that E_a values in the presence of the leaf extracts and leaf extract + KCl are higher than that of the blank which suggests that the process of adsorption may be by physical adsorption. Except for 1.0g/L of leaf extract, the negative values of Q_{ads} observed for the leaf extracts and the leaf extract + KCl were an indication that the processes were exothermic. The negative Q_{ads} values indicated that the degree of surface coverage decreased with rise in temperature in the presence of extract supporting physisorption mechanism (Umoren *et al*., 2006). The average E_a values in both cases were found to be 71.14 kJ/mol and 65.00 kJ/mol for the leaf extract and leaf extract + KCl respectively. These values confirmed the assertion that the process of inhibition is by physical adsorption (Atkin and de Paula, 2006).

Adsorption isotherm

Table-5: Values of K_{ads}, R^2, ΔG_{ads} and ΔS_{ads} from Langmuir, Freundlich and Temkim Isotherms for Spondias cytherea leaf extract and leaf extract + KCl in 1.0M H_2SO_4

		Langmuir			Freundlich			Temkim		
	Temp. (K)	K_{ads}	ΔG_{ads}	R^2	K_f	ΔG_{ads}	R^2	K_{ads}	ΔG_{ads}	R^2
S.P Leaf	**323K**	0.6557	-9.6542	0.9840	0.4375	-8.5674	0.9870	1.5311	-11.9318	0.9780
	333K	0.3026	-7.8114	0.8820	0.2518	-7.3024	0.9760	1.2674	-11.7779	0.9490
S.P. Leaf + KCl	**323K**	4.2918	-14.7002	0.9990	0.7362	-9.9651	0.9550	2.0834	-12.7591	0.9580
	333K	2.4752	-13.6313	0.9950	0.6383	-9.8783	0.9240	1.8870	-12.8800	0.9120

Basic information on the interaction between the inhibitor and the mild steel surface can be provided by adsorption isotherm. The Langmuir, Freundlich and Temkim isotherms were employed to fit the experimental data and the plots are shown in Figures 7 to 12. Table 4 showed the linear correlation coefficient R^2 and K_{ads} of the fitted data.

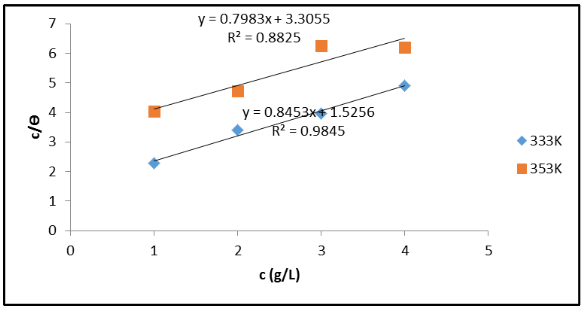

Fig-7: Langmuir plot for Spondias cytherea leave in 1.0M H_2SO_4

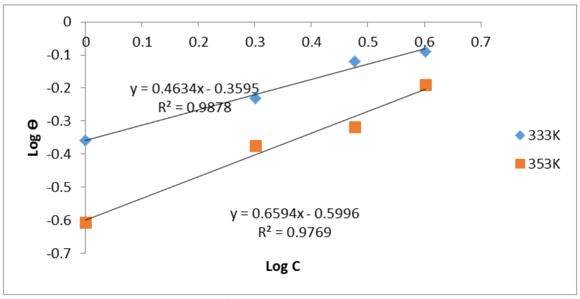

Fig-8: Freundlich plot for Spondias cytherea leave extract in 1.0M H_2SO_4

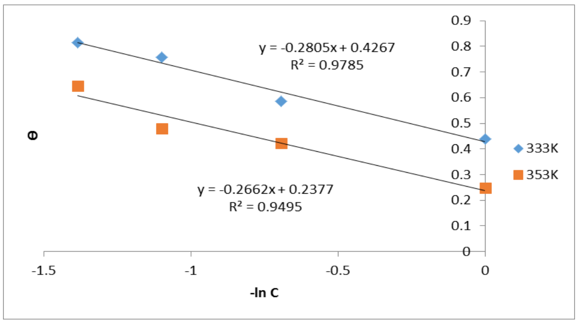

Fig-9: Temkim plot for Spondias cytherea leave extract in 1.0M H_2SO_4

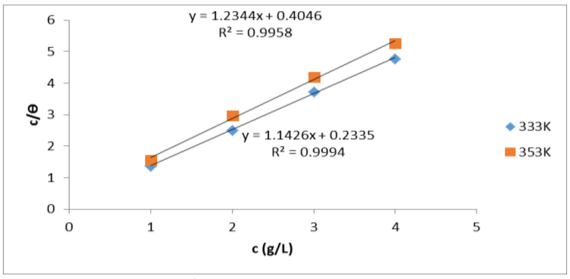

Fig-10:Langmuir plot for Spondias cytherea + Potassium chloride in 1.0M H_2SO_4

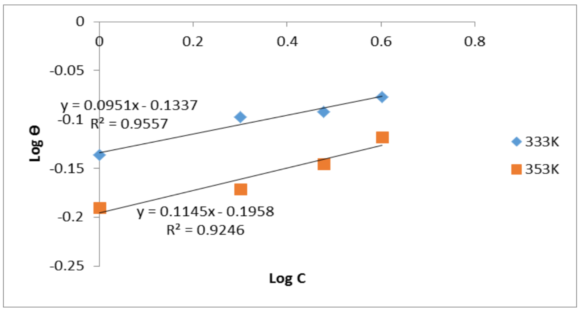

Fig-11: Freundlich plot for Spondias cytherea + Potassium chloride in 1.0M H_2SO_4

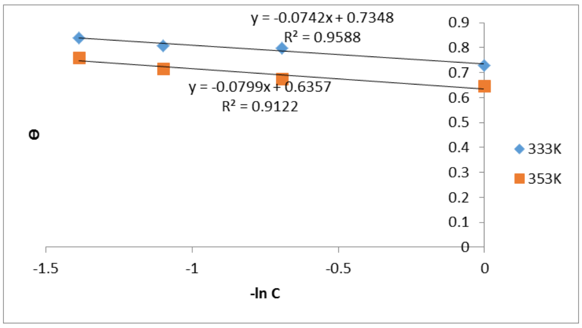

Fig-12: Temkim plot for Spondias cytherea + Potassium Chloride in 1.0M H_2SO_4

The negative values for ΔG_{ads} as seen in Tables 4 indicate that the reaction was spontaneous and feasible (Saedah, 2013). Generally, negative values of ΔG_{ads} up to -20 kJ $mol^{-1}$are consistent with electrostatic interactions between the charged metal which signifies physical adsorption while values more negative than -40 kJ mol^{-1} involved charge sharing or transfer from the inhibitor molecules to the metal surface to form coordinate type of bond which signifies chemical adsorption. It follows therefore that the values from this investigation are indicative of physical adsorption (Umoren *et al*., 2006). For the Langmuir, Freundlich and Temkim isotherms, linear plots were obtained. Comparing the R^2 values, it could be observed that the data fitted well to Freundlich for the leaf extract and Langmuir for the leaf extract + KCl. The values of K_{ads} were seen to decrease as the temperature rises from 333K to 353K in all the cases. This further confirms that the inhibitors were physically adsorbed on the surface of the mild steel (Ikpi *et al*., 2012).

CONCLUSION

Extract of the Spondias cytherea leaves was found to inhibit corrosion of mild steel in 1.0 M H_2SO_4 solution. The amount of weight loss decreased significantly in the presence of leave extract compared to the blank acid solutions indicating that leave extract inhibits the corrosion of mild steel in H_2SO_4. The leaf extract showed maximum inhibition efficiency of 88.52% and 81.42% for gravimetric and gasometric analysis respectively. The leaf extracts + KCl showed maximum inhibition efficiency of 91.49% and 83.76% for gravimetric and gasometric analysis respectively. It was generally observed that the addition of KCl improved the inhibition of the leaf extract. Considering the E_a, K_{ads}, ΔG_{ads}, Q_{ads}, the process of corrosion inhibition is attributed to be by physical adsorption. Comparing the R^2 values, it could be observed that the data fitted well to Freundlich for the leaf extract and Langmuir for the leaf extract + KCl. From this study, extract of Spondias cytherea leaf and KCl were found to act as a good inhibitor for the corrosion of mild steel in H_2SO_4.

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