

CORROSION INHIBITION OF PIPELINE STEEL IN 0.5 M H₂SO₄ SOLUTION USING SEED PERICARP OF *CHRYSOPHYLLUM ALBIDUM*

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Received: 04-12-19

Accepted: 09-12-19

ABSTRACT

The corrosion inhibitory characteristics of Chrysophyllum albidum seed pericarp extract was studied on pipeline steel corrosion in sulphuric acidic environment using gravimetric and electrochemical techniques at varied temperatures (30, 40 and 60°C). Fourier transformed infra red spectroscopy (FTIR) and Scanning Electron Microscope (SEM) were used to study metal's surface film and surface morphology respectively. The phyto-constituents were identified using standard functional group analysis. The results obtained showed that corrosion inhibition efficiency increased with increase in extract concentration but decrease with increase in temperature. This is attributed to the identified phytochemical constituents of the extract adsorbed and desorbed on the surface of the pipeline steel. The adsorption data fitted into Langmuir adsorption model better. The data obtained from electrochemical analysis shows that the inhibitor functioned as a mixed type. The kinetics studies revealed that the extract molecules got physically adsorbed spontaneously on pipeline steel, while the thermodynamic data showed an exothermic process.

Keywords: Corrosion Inhibition, *Chrysophyllum albidum*, Surface coverage, Activation energy, Pipeline steel, Weight loss.

INTRODUCTION

Corrosion is of immense threat to the integrity of metallic equipments and structures. There have been lots of investments to corrosion mitigation. The application of corrosion inhibitors which are usually added in small quantities, to inhibit the corrosion of the metallic structures or metallic alloy is one of such mitigation processes (Ejikeme *et al.*, 2015). Plants extracts have been found useful in this regard. They are ecological friendly, cheap and readily available. Extraction techniques such as maceration, soxhlet, supercritical liquid extraction, etc are often used to obtain extracts from these plants (Abiodun & Oladapo, 2011). Plant extracts are known to be good corrosion inhibitors

due to presence of compounds with hetro-atoms (alkaloids, tannins, saponins, etc)

Corrosion is a major threat to steel, especially pipeline steel which is fore mostly used for mechanical construction and fabrication of metallic structures because of their minimal cost, better mechanical properties compared with other oilfield alloys and metals. The cost of corrosion is further increased especially in the petroleum industry due to deeper well, ever increasing aggressive environment and the use of acidic oil field chemical. The application of corrosion inhibitors for Pipeline steel corrosion is less expensive and suitable due to peculiar nature of pipeline operations, (Zhang & Hua, 2009).

The present work evaluates the corrosion inhibiting properties of *Chrysophyllum albidum* (CA) seed pericarp extract applying gravimetric and electrochemical techniques. *Chrysophyllum albidum* is a forest fruit tree popular across Africa. The fruit known as “African star apple” is called “agbalumo” in South Western Nigeria and “Udara” in South Eastern Nigeria. The plant fruit season is between December and April. A typical fruit consists of a gummy skin a succulent pulp in which the seeds are embedded. The seeds are bean shaped with a soft cotyledon encased in an outer pericarp. Agbalumo has immense economic, potential and health benefits. Its rich sources of natural antioxidants have been established to promote health by acting against Oxidative stress related diseases such as diabetes, Cancer and Coronary heart diseases, (Chris *et al.*, 2016). Jams obtained from the fruit-pulp could compete with raspberry jams and jellies while the oil from the seed has been used for diverse purposes. This work uses *chrysophyllum albidum* seed pericarp which is often discarded as waste for corrosion inhibition studies of pipeline steel in acidic medium.

MATERIALS AND METHODS

Materials

The Pipeline steel was purchased from System Metals Industries Limited, Port-Harcourt, Nigeria. They were cut into coupons of dimensions 2cm by 3cm, polished successively with emery paper of 100 to 2000 grits and rinsed with ethanol. Acetone was applied to remove residue from polishing and there after air dried. The initial weight was obtained and they were stored in desiccators free of moisture. These coupons were same as characterized previously by Ngobiri *et al* (2015). For gravimetric test, the coupons were perforated at the edge and hanged with a polymeric thread. All other reagents used were of analytical grade.

Weight Loss Measurements

The weighed pipeline coupons were totally immersed in six 150 ml beakers labelled accordingly using a masking tape as 1 g/dm³, 2 g/dm³, 3 g/dm³, 4 g/dm³, 5 g/dm³ and 0 g/dm³ (blank) of the extracts in 0.5 M H₂SO₄ solution at 30, 40 and 60 °C using polymeric thread tied round the coupons in beakers labelled A, B, C, D, E, F respectively. They were retrieved after 24 hours of exposure, washed with distilled water, brushed, rinsed in ethanol, dried in acetone and then weighed again to obtain the weight loss. The weight loss was taken as the difference in weight of the coupon before and after immersion, (equation 1). All tests were conducted in triplicate and the mean value obtained for reproducibility. The experiment was repeated at a higher temperature in a J.P. SELECTA 6001197 thermostated water bath.

$$\text{Weight loss} = (W_i - W_f) \text{ g} \quad (1)$$

Where: W = weight loss of pipeline steel coupon, and W_i = Initial weight of the pipeline steel coupon, W_f = Final weight loss of pipeline steel coupon, and all unit in grammes.

The fractional surface coverage (θ) for 0.5 M H₂SO₄ at different concentrations of CA were obtained by using the equation below.

$$\theta = 1 - \frac{W_i}{W_o} \quad (2)$$

Where; W_o = corrosion rates in the absence of inhibitor; W_f = corrosion rates in the presence of the additive.

The inhibition efficiencies were calculated using the formula.

$$\%IE = 1 - \frac{W_i}{W_o} \times 100 \quad (3)$$

Where W_o and W_i is weight loss in grams of metal coupon in the presence and absence of various concentration of the extract.

The total surface area of pipeline steel coupon immersed in the solution, was calculated as followed:

$$A=2KM + Kt + 2Mt + 2\pi[r^2 - 2\pi r^2] \quad (4)$$

Where A = Total surface area of the coupon immersed in solution, K = length of coupon, M = Width of coupon, T=Thickness of coupon, R = Radius of the hole drilled on coupon.

The corrosion rate, half-life and rate constant was obtained as showed below.

$$\text{Corrosion rate (mpy)} = \frac{87.6 \Delta w}{DAT} \quad (5)$$

Where; ΔW = change in weight loss , D = density of specimen (g/cm^3), A = surface area of specimen (cm^2) and T = exposure time (hours).

Half life:

$$t_{1/2} = \frac{0.693}{k} \quad (6)$$

Rate constant (k)

$$K = \frac{2.303}{\text{Time}} \log \frac{w_1}{w_2} \quad (7)$$

Surface Morphological Analysis

Pipeline steel coupons were immersed in 0.5 M H_2SO_4 containing CA extract. The coupons were retrieved after 24 hours and the surface film subjected to SEM and FT-IR analysis.

FT-IR Analysis

Specimen of the steel coupons were immersed in a solution of 0.5 M H_2SO_4 containing 1 g/L CA extract for 24 hours. The coupons were retrieved dried and the surface film powered carefully scrapped of the surface with sterile blade. The powered was submitted for FT-IR analysis usinr the KBr disc.

RESULTS

The results of the experimental are presented as follows

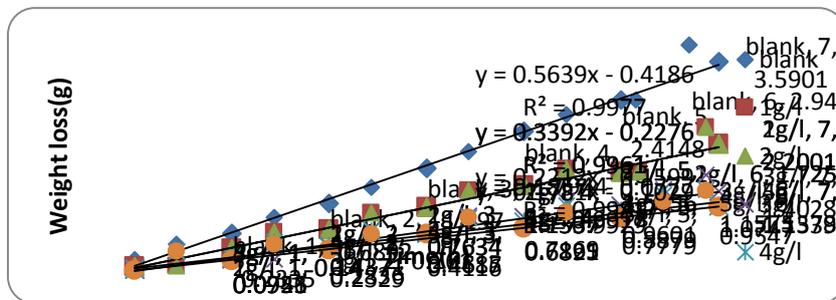


Figure 1: Plot of weight loss against time for pipeline in 0.5 M H_2SO_4 containing different concentration of CA pericarp at 303 K.

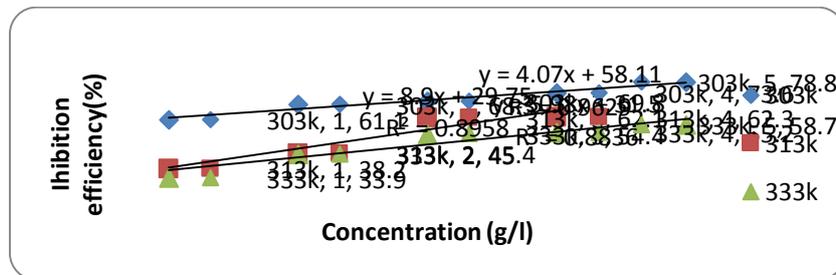


Figure 2: Plot of corrosion inhibition efficiency against concentration of *chrysophyllum albidum* seed pericarp in 0.5 M H_2SO_4 at different temperatures.

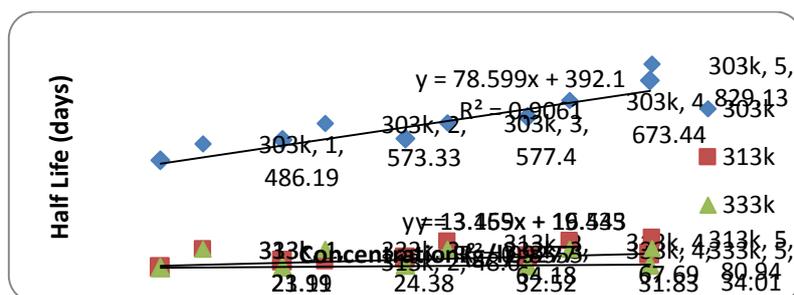


Figure 3. Variation of half-life with concentration of seed pericarp extract in 0.5MH₂SO₄

Table 1. Corrosion rate, Inhibition efficiency and degree of surface coverage deduced from weight loss measurements in H₂SO₄ for pericarp extracts.

	Corrosion Rate			Inhibition Efficiency(%IE)			Degree of surface coverage(θ)		
	30°C	40°C	60°C	30°C	40°C	60°C	30°C	40°C	60°C
Blank	4.38	5.95	8.62						
1	4.21	5.62	7.15	65.4	64.3	62.4	0.654	0.643	0.624
2	3.62	4.82	6.37	76.1	74.2	66.9	0.761	0.742	0.669
3	3.48	4.01	5.57	78.4	76.3	72.5	0.784	0.763	0.725
4	2.98	3.83	4.67	81.7	79.7	73.7	0.817	0.797	0.737
5	2.43	2.54	4.27	84.6	81.2	80.3	0.846	0.812	0.803

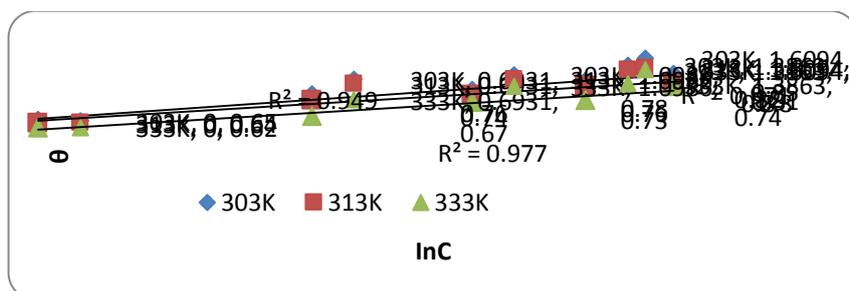


Figure 4: Temkin adsorption isotherm for pipeline steel corrosion in 0.5M H₂SO₄ at 30 °C, 40 °C and 60 °C for CA corrosion inhibition.

Table 2: Some parameters of the linear regression between θ and ln C for Temkin adsorption isotherm.

Temperature (K)	H ₂ SO ₄ PERICARP		
	K _{ads}	a	R ²
303	0.762	0.742	0.949
313	0.895	0.864	0.971
333	0.982	0.981	0.977

Where a is the molecular interactions of the adsorption layer and heterogeneity of the coupons; K_{ads} is the equilibrium constant.

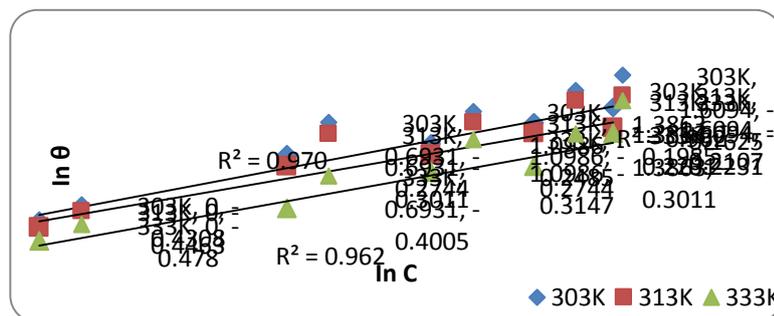


Figure 5: Freundlich adsorption isotherm for pipeline steel corrosion pericarp in 0.5 M H₂SO₄ at 30 °C,40 °C and 60 °C

Table 3: Calculated parameters from Freundlich adsorption isotherm plot for pipeline steel in 0.5 M H₂SO₄ of the extract.

Temp(K)	H ₂ SO ₄ PERICARP			
	K _{ads}	ΔG _{ads}	n	R ²
303	0.958	-10.4087	0.463	0.962
313	0.892	-10.975	0.496	0.962
333	0.795	-10.986	0.503	0.970

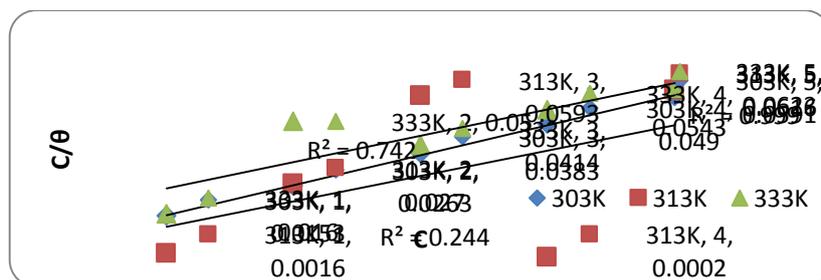


Figure 6: Langmuir adsorption isotherm for pipeline steel corrosion pericarp in 0.5 M H₂SO₄ at 30 °C,40 °C and 60 °C

Table 4. Calculated parameters from Langmuir adsorption isotherm plot for pipeline steel in 0.5 M H₂SO₄ of the extract.

Temp(K)	H ₂ SO ₄ PERICARP			
	K _{ads}	ΔG _{ads}	Slope	R ²
303	1.162	-10.580	0.98	0.999
313	1.126	-10.669	0.98	0.742
333	0.886	-10.796	0.99	0.244

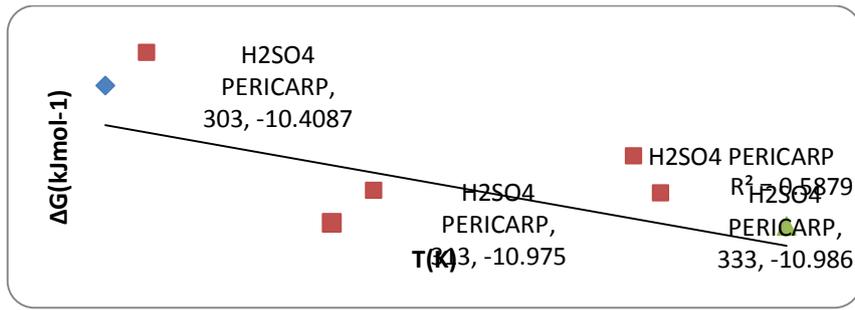


Figure 7. Frundlich plot of ΔG°_{ads} against T(K) for the adsorption of CA extracts in acid medium

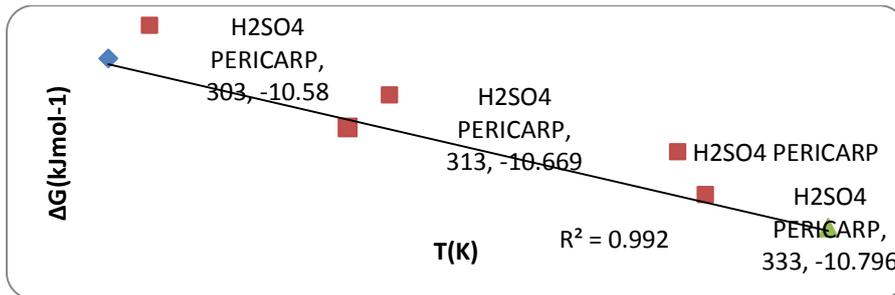


Figure 8: Langmuir plot of ΔG°_{ads} against T(K) for the adsorption of CA extracts on pipeline steel in acid medium

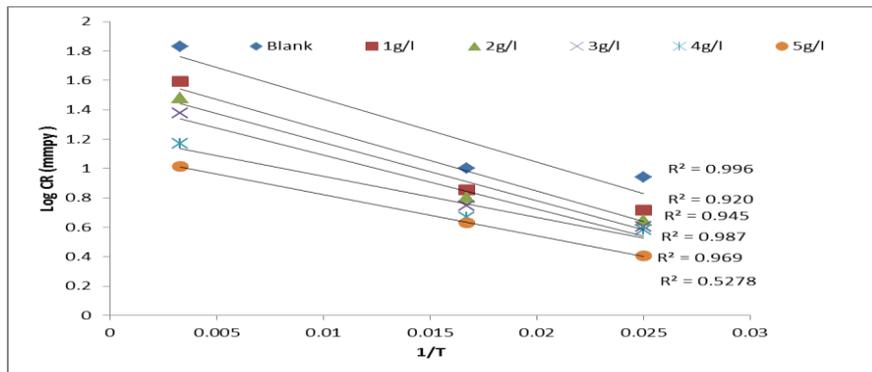


Figure 9: Arrhenius plot in the absence and presence of different concentration of the pericarp extract in H₂SO₄

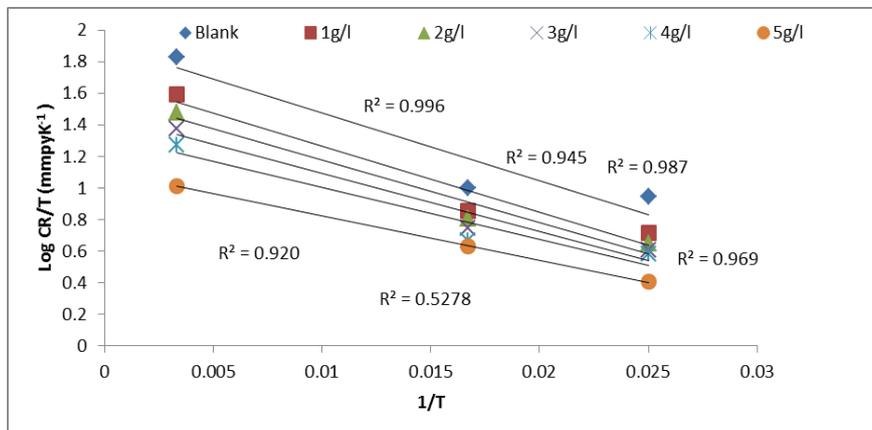
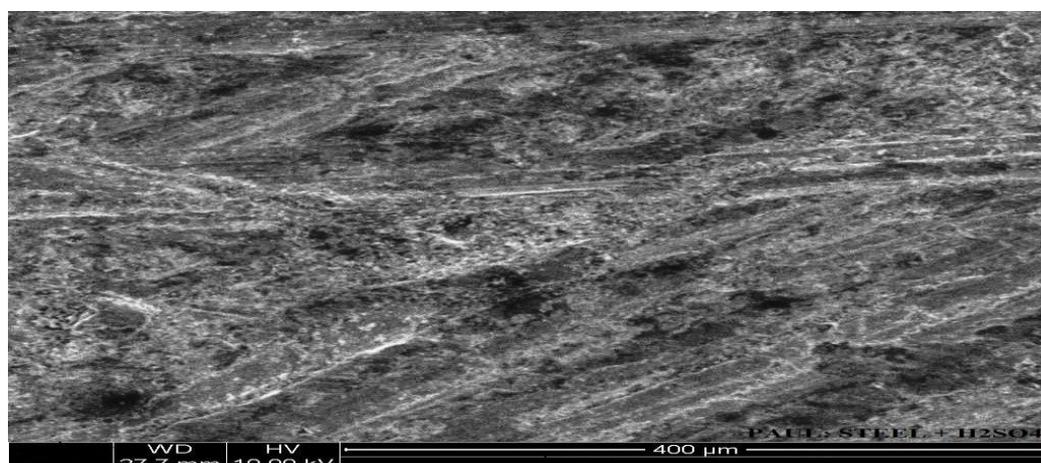
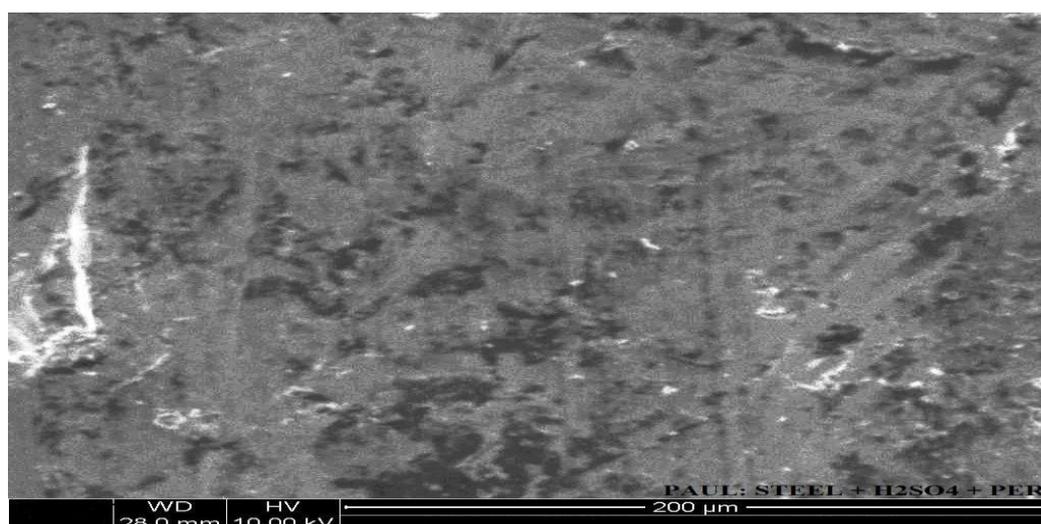


Figure 10. Transition state plots for pipeline steel in 0.5 M H₂SO₄ in the absence and presence of different concentrations of pericarp extract

Table 6. Activation parameters for pipeline steel in 0.5M H_2SO_4 of the extract

Conc(g/ cm ³)	H_2SO_4	PERICARP	E_a (kJmol ⁻¹)	ΔH^* (KJmol ⁻¹)	ΔS^* (Jmol ⁻¹)	R^2 (Arrhenius)	R^2 (Transition)
Blank			43.60	40.34	-102.24	0.920	0.912
1			44.90	43.52	-100.23	0.996	0.995
2			46.09	45.46	-96.42	0.945	0.955
3			47.65	48.97	-93.71	0.969	0.929
4			50.96	50.46	-90.46	0.987	0.982
5			53.96	51.21	-84.81	0.952	0.892

Figure 11a: SEM image of pipeline steel in 0.5 M H_2SO_4 without CA extractFigure 11b: SEM image of pipeline steel in 0.5 M H_2SO_4 with CA pericarp extract

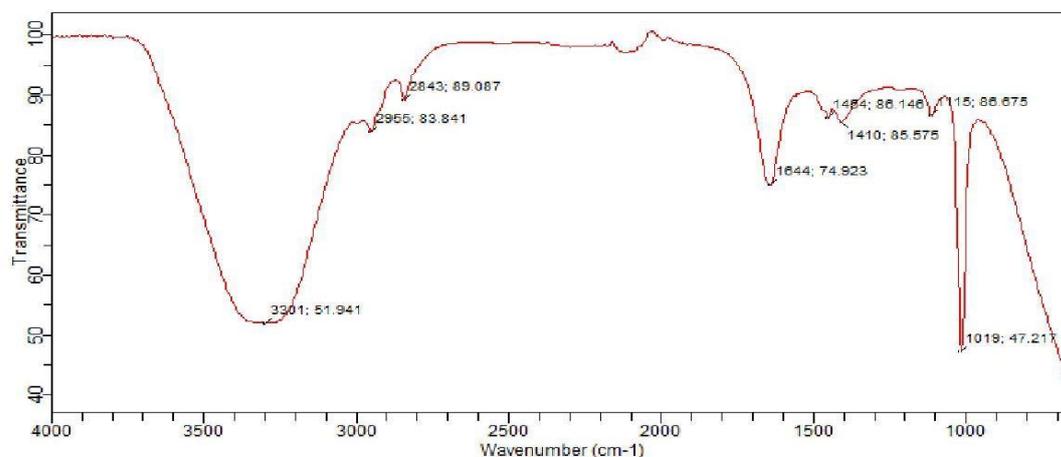
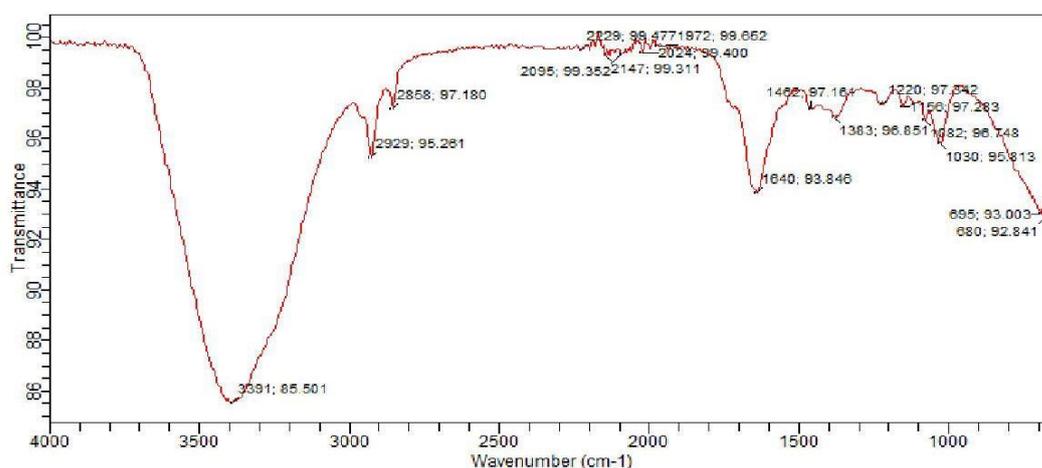


Figure 12a: FT-IR spectra of CA pericarp extract .

Figure 12b: FT-IR spectra formed on the pipeline steel after immersion in 0.5 M H₂SO₄ with CA pericarp extract.

DISCUSSION

Effect of temperature on Inhibition Efficiency of the extract of *Chrysophyllum albidum* seed pericarp.

The effect of different concentration of the extract of *Chrysophyllum albidum* seed pericarp on pipeline steel in 0.5M H₂SO₄ at temperature 30 °C, after 168 hours of immersion period is shown in Figure 1. Similar trends were observed at temperatures 40 and 60 °C

The figure clearly indicates a reduction in weight loss of the metal coupons in the presence of the extract compared to its

blank. They further reveal decreases in weight loss of the pipeline steel as the concentration of CA increases from 1 g/dm³ to 5 g/dm³.

Also a graph of concentration of the extracts versus inhibitor efficiency of pipeline steel in 0.5M H₂SO₄ is presented in Figure 2. It was observed that corrosion inhibition efficiency increased with increase in the concentration and decreased with rise in temperature. The highest corrosion inhibition efficiency of 84.6 % for pipeline steel in H₂SO₄ was obtained at 30 °C in 5g/l of CA. At 40 and 60 °C inhibition efficiencies were 81.2 and 80.3%

respectively for same concentration. Similar behaviours have earlier been reported by (Ejikeme *et al.*, 2015). The obtained values for Corrosion rate, Inhibition efficiency and degree of surface coverage are presented in Table 1.

The impressive action of CA in inhibiting the dissolution of pipeline steel in 0.5M H₂SO₄ medium may be attributed to the adsorption of extract molecules on the surface of the pipe steel. However, it should be noted that since CA contain alkaloids, terpenes, flavonoids, tannins, etc, with fused benzene rings and heteroatoms in the ring, the chemical complexity of the extracts makes it improper to assign the inhibiting action to a particular It is suggestible that the different constituents of the CA extract reacted differently at the surface to cause the inhibition. Increase in corrosion rate as the temperature increased is attributed to increase in kinetic energy of the corrodants at elevated temperature.

Figure 3 shows the variation of half-life with concentration for corrosion inhibition of pipeline steel in acid medium. Half-life of the pipeline steel increased at higher concentrations of the extract. Corrosion material half life can be used as indicator for material integrity, (Ngobiri *et al.* 2015).

Adsorption Study

The mode and degree of the contact between the extracts molecules and the coupons were considered by adsorption isotherms. In isotherm studies, the linearity of the plot and good correlation coefficient may be interpreted to suggest that the experimental data for the studied extracts obey a particular adsorption isotherm but consideration deviation of the slope from unity shows that the isotherm may not be strictly applied. Temkin adsorption isotherm assumed a uniform distribution of adsorption energy which increased with increase of the surface coverage is given by equation 8

$$\theta = (1/2a) \text{Log } K_{\text{ads}} - (1/2a) \text{In } C \quad (8)$$

Where: a is the molecular interactions of the adsorption layer and heterogeneity of the surface of the coupons; K_{ads} is the equilibrium constant, C is the concentration of the extracts, θ is the degree of surface coverage. Plots of surface coverage (θ) against In C at 30 °C, 40 °C and 60 °C are shown in figure 4.

Corrosion coefficients of the linear regression (R²) plots greater than 0.835 for pericarp in H₂SO₄ are shown in Table 2 which suggests some measure of conformity of the data to the Temkin adsorption isotherm model, in the acid medium. The values of molecular interactions are all greater than zero (>0) indicates lateral interaction between the pipeline steel and the extracts.

Linear form of Freundlich adsorption isotherm is given by equation 12. Plots of In θ against In C are shown in figure 5 using corrosion data. It is shown from the plots that the data calculated within the temperature range confirmed to the Freundlich adsorption isotherm model better than Temkin model, considering their regression values.

$$\text{In } \theta = \text{In } K + n \text{In } C \quad (9)$$

Where C and K both represent the concentrations of CA extract and equilibrium constant. In both extract seed pericarp extract with the pipeline steel, it was better fitted at 60°C than 30°C with regard to the R² values of the plots for Freundlich adsorption isotherm, in acid medium, shown in Table 3.

$$\frac{C}{\theta} = \frac{1}{K} + C \quad (10)$$

Where: K is the equilibrium constant, C is the concentration of CA extracts and θ is the degree of surface coverage. The *Chrysophyllum albidum* extract also conformed to Langmuir adsorption isotherm for its pericarp extract in the acidic medium with R² values of 0.999 which

shows a better adsorption data than Temkin and Freundlich isotherm.

The data obtained from this model are shown in Table 4. The values of ΔG_{ads} are all negative ranging from -10.580 and -10.796 kJmol⁻¹ for pericarp in H₂SO₄ medium. The values of $\Delta G_{\text{ads}}^{\circ}$ are often used to suggest either chemisorptions, physisorption or both adsorption modes. Physisorption is linked to electrostatic interactions between charged extracts molecules and charged metal surface, while chemisorptions has to do with charge sharing between the metal surface and the extracts resulting in special kind of coordinate bond (Li, *et al.*, 2012). A value of $\Delta G_{\text{ads}}^{\circ}$ around -20 kJmol⁻¹ or less implies physisorption while around -40 kJmol⁻¹ or greater suggest chemisorptions. But the values of $\Delta G_{\text{ads}}^{\circ}$ obtained as showed in Table 4 and are all less than -20 kJmol⁻¹, suggesting physisorption mechanism.

The standard enthalpy ($\Delta H_{\text{ads}}^{\circ}$) and entropy ($\Delta S_{\text{ads}}^{\circ}$) were obtained from the equation below:

$$\Delta G_{\text{ads}}^{\circ} = \Delta H_{\text{ads}}^{\circ} - T\Delta S_{\text{ads}}^{\circ} \quad (14)$$

Plots of $\Delta G_{\text{ads}}^{\circ}$ against T are shown in figure 17-18 from which the $\Delta H_{\text{ads}}^{\circ}$ and $\Delta S_{\text{ads}}^{\circ}$ values were obtained with slope and intercept respectively. $\Delta H_{\text{ads}}^{\circ}$ and $\Delta S_{\text{ads}}^{\circ}$ values were -1.94 and -60 kJmol⁻¹ for pericarp in H₂SO₄. The values obtained confirm exothermic adsorption to be either by physical adsorption or chemical adsorption while endothermic is only by chemical adsorption. Based on numerical values, physisorption only manifest when $\Delta H_{\text{ads}}^{\circ}$ value is lower than -40kJmol⁻¹ while chemisorptions is -100kJmol⁻¹ (Edidong *et al.*, 2018). The values of $\Delta H_{\text{ads}}^{\circ}$ obtained confirmed physisorption mechanism while the negative values of $\Delta S_{\text{ads}}^{\circ}$ implies adsorption of the extracts on the surface of the coupons resulting in a reduction of the entropy, which agreed with basic thermodynamic principle of lower in entropy for exothermic reaction.

Kinetic and Thermodynamic Parameters

The values were obtained from Arrhenius equation shown below:

$$\log CR = \log A - \left(\frac{E_a}{2.303 RT} \right) \quad (15)$$

$$\log \frac{CR}{T} = \left[\left(\frac{R}{Nh} \right) + \left(\frac{\Delta S^*}{2.303R} \right) \right] - \frac{\Delta H^*}{2.303RT} \quad (16)$$

Where E_a represent activation energy, A frequency factor, R gas constant and T absolute temperature, ΔH^* enthalpy of activation, ΔS^* entropy of activation.

The value of E_a were obtained from the plots of log CR against 1/T as shown in figure 19-20, while ΔS^* and ΔH^* were calculated from the intercepts and slopes of log CR/T against 1/T shown in figures 21-22. Generally, physisorption is noted when E_a and ΔH^* data in the presence of extracts are greater than in the absence of extracts while opposite is for chemisorptions.

From Table 5, it is observed that E_a and ΔH^* data in the presence of extracts are greater than in the absence validating that physisorption occur against chemisorptions on the pipeline surface. The activation energy calculated shows an increase with increase concentration compared to the blank. The value of ΔS^* were noted to be negative both in the absence and presence of the seed pericarp extract in acidic medium. The negative value of energy of activation for uninhibited system has already being reported with hydrogen evolution reaction by Zhang *et al.* 2009. With increase in negative values of the activation energy in the presence of extracts, which increase with increase in concentration sees Table 5. The negative values of ΔS^* signified association than dissociation step of activated complex in the rate determining step, signifying a reduction of degree of disorderliness. (Edidiong *et al.*, 2018).

Surface Morphological Analysis (SEM):

Polished pipeline steel specimens were immersed in 0.5 M H₂SO₄ solution in absence and presence *Chrysophyllum albidum* (CA) for 12 hours, shown in figure 23a-b. From the result obtained it shows that the surface of the coupon in 0.5 M H₂SO₄ in absence of CA extracts was badly corroded by acid attack, but corrosion rate decreases on addition of *Chrysophyllum albidum* extracts as evidence in the smoother surface with less roughness formation on the pipeline surface as shown in figure 23b-c in the acid medium. This is probably due to adsorption of the extract on the pipeline surface forming a protective layer that inhibits corrosion.

FT- IR Studies of CA Seed pericarp In H₂SO₄ surface film on pipeline steel

The FT IR analysis of the surface film of the coupons (fig 12a -12b) showed the functional groups present in the extracts. The spectra show a peak at 3390cm⁻¹ indicating a strong and broad band corresponding to O-H stretching vibration. A peak around 2929cm⁻¹ N-H which shows stretching mode. The peak at 2843cm⁻¹ linked to stretching vibration of C=N. The band at 1644cm⁻¹ indicate the presence of C=O (ketone) or suggest C=O (ester), stretching frequency. The peak at 1019cm⁻¹ confirmed C-N stretch. While for Seed pericarp in H₂SO₄, the peak at 3290cm⁻¹ indicate O-H stretching vibrations, 2929cm⁻¹ can be assigned to N-H bend or C=O vibrations. The peak 1413cm⁻¹ prove the presence of C=C, peak at 1022cm⁻¹ assigned to C-N stretch for pericarp in H₂SO₄. The presence of functional groups of CA extract molecules confirms adsorption of CA on the surface of the pipeline steel.

4. CONCLUSION

Chrysophyllum albidum Seed Pericarp extracts acted as a good inhibitor for corrosion of pipeline steel in 0.5 M H₂SO₄ acid solution. The inhibition efficiency increased with increasing concentration of

Seed Pericarp but showed decreased as the temperature increased.

There was a remarkable reduction in corrosion rate with higher concentrations of CA but corrosion rate increased with increasing temperature. The values of activation energy also decreased with increasing concentration of Seed Pericarp. Protective film formation was confirmed by FT-IR, SEM results revealing functional groups such as O-H, N-H and C=O were adsorbed on the surface of pipeline steel and may be responsible for corrosion inhibition.

The results obtained from adsorption studies showed that adsorption of inhibitor molecules on pipeline steel obeys the Langmuir adsorption isotherm at all temperatures (303K-333K) compared to Temkin, and Freundlich isotherm with a spontaneous exothermic process accompanied by decrease in entropy of reaction, decrease in activation energy in the presence of CA extract which indicate physical adsorption.

The negative values of ΔG suggested that spontaneous adsorption of Seed Pericarp on pipeline steel surface occurred via physical adsorption mechanism. The negative values of ΔH imply exothermic adsorption process for seed pericarp on the metal surface.

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