

PARKIA BIGLOBOSA (AFRICAN LOCUST BEAN) PULP AS A GREEN INHIBITOR FOR MILD STEEL CORROSION

ABSTRACT

This work investigated corrosion inhibition potential of *Parkia biglobosa* pulp on mild steel in 1M HCl and 0.1M NaOH aqueous solutions at 301 and 321 K, aimed at converting a cellulosic renewable waste into an environmentally friendly answer to the challenge of mild steel corrosion, especially in chemical industries. Weight loss of mild steel coupons over a period of 120 hrs. were measured at varying concentrations (0.0-7.5 g/L) of *P. biglobosa* pulp. The data didn't strictly fit the Langmuir isotherm, having a slope greater than unity and suggesting that adsorbing species from *P. biglobosa* pulp occupied more than one active site. This was collaborated by the quantum chemical parameters evaluated. Results of the investigations showed that *P. biglobosa* pulp is a good green inhibitor for mild steel corrosion in both acidic and alkaline media. An optimum dosage of 4.5g/L resulted to 85.73 % inhibition efficiency (η) at 301 K while at 321 K, the optimum dosage was 1.0 g/L of the pulp with an inhibition efficiency \approx 50 % and 55 % in the acidic and alkaline medium respectively. The η values decreased with increase in temperature and were strongly collaborated by values ($-0.86 \text{ Lcm}^{-2}\text{h}^{-1}$ at 301 K and $-0.498/0.55 \text{ Lcm}^{-2}\text{h}^{-1}$ at 321 K) of order (b) of the reaction. The corrosion reaction showed a large rate constant ($2.66 \times 10^3 \text{ Lcm}^{-2}\text{h}^{-1}$ and $5.33 \times 10^3 \text{ Lcm}^{-2}\text{h}^{-1}$ in acidic and alkaline medium respectively) at ambient temperature; reflecting the fast nature of the process. Values of the standard free energy of adsorption (ΔG_{ads}^o) were negative for the process both at ambient and elevated temperature indicative of a spontaneous process and stable layer of adsorption. These values were less than -40 kJmol^{-1} , suggestive of a physisorption mechanism. This was collaborated by higher adsorption activation energy (E_a) in presence of the inhibitor than in its absence.

Keywords: Weight loss, *Parkia biglobosa*, isotherm, corrosion, corrosion inhibitor, quantum chemical parameters

INTRODUCTION

Metallic materials constitute the most widely used group of materials particularly in both mechanical and the transportation industry. They are also used in electronics and increasingly in the construction industry. Mild steel is the most commonly used engineering material [1]. It is cheap, readily available in a wide range of standard forms and sizes, and can easily be worked upon and welded. It has good tensile strength and ductility. However, the usefulness of mild steel, and indeed, metals and alloys; is constrained by one common challenge known as corrosion. Corrosion is a naturally occurring phenomenon simply defined as deterioration of metal surfaces caused by reaction with the surrounding environmental conditions. Acid solutions (basic ones in some cases), commonly used in the chemical industry to remove mill scales from metallic surfaces; constitute one of such environments that breed corrosion. Hydrochloric acid is widely used in removing scales and salts from steel surfaces, cleaning tanks and pipelines and steel alloys [2]. Corrosion can cause disastrous damage to metal and alloy

structures; causing economic consequences in terms of repair, replacement, product losses, safety and environmental pollution. Due to these harmful effects, corrosion is an undesirable phenomenon that ought to be prevented.

Several ways of preventing corrosion and the rates at which it can propagate with a view of improving the lifetime of metallic and alloy materials are in practice. The use of inhibitors for the control of metals and alloys in contact with aggressive environment is one among the acceptable practices used to reduce and/or prevent corrosion. A corrosion inhibitor is a substance which when added in small concentration to an environment, effectively reduces the corrosion rate of a metal exposed to that environment [3] The inhibitors can be broadly classified into two categories namely those that enhance the formation of a protective oxide film through an oxidizing effect and those that inhibit corrosion by selectively adsorbing on the metal surface and creating a barrier that prevent access of corrosive agents to the metal surface. Several organic

compounds have been studied and analyzed to investigate their potential as corrosion inhibitors [4,5]. Most of these studies reveal that almost all organic molecules containing heteroatoms such as nitrogen, sulphur, phosphorous and oxygen show significant inhibition efficiency [4,5,6,7]. Despite these promising findings about possible corrosion inhibitors, most of these substances are not only expensive but also toxic [5] and non-biodegradable; thus, raising pollution issues. These deficiencies have prompted the search for their replacements.

Plants have been recognized as sources of naturally occurring compounds, some with rather complex molecular structures and having varying physical, chemical and biological properties [7,8,9,10]. Most plant extracts are traditionally used as pharmaceuticals and biofuels [11]. The use of these phytochemicals is of interest because of their low cost, abundant availability and more importantly their environmentally benign status. As a result of these advantages, extracts of some common plants and plant products have been tried as corrosion inhibitors for metals and alloys under different environments [12, 13, 14, 15, 16]. Plants have, therefore, become an important source of a wide range of green corrosion inhibitors.

Parkia biglobosa (African locust beans) is a perennial, leguminous tree of the family *Fabacea* native, majorly, to the savannah region of West African countries stretching from Senegal to Cameroun and extending eastwards to Uganda. Annual world production of the fruit from the tree is estimated at over 100 million metric tons (MT) and has not been utilized beyond the natives where such trees occur, in spite of its medicinal and nutritional values [17]. According to the report, about 18 million MT of seed and 25.7 million MT of the fruit pulp are produced annually in Nigeria. The sweet, yellow floury pulp is eaten fresh (licked) by rural dwellers, which indicates its edibility and nontoxicity. The pulp is, therefore, virtually not placed for any commercial value and constitutes solid waste with its attendant challenges.

This study, therefore, seeks to investigate the corrosion inhibition potential of the *P. biglobosa* pulp on mild steel in 1M HCl and 0.1M NaOH aqueous solutions at ambient and elevated temperatures.

METHODOLOGY

2.1 Materials' Collection and Preparation

Dry *P. biglobosa* pods were collected from naturally growing *P. biglobosa* trees within the campus of Bingham University, Karu, Nigeria and air-dried for 336 hrs. The seeds were removed from the dry pods and pounded lightly to loosen the pulp-coating from the seeds. The pulp was then sifted off and collected and stored in a polyethylene bag. Mild steel AISI 1018 (low carbon steel) with composition as shown in Table 1 was collected from and cut into 4x3x0.3cm coupons at Nasco Household Products Limited, Jos, Nigeria.

Table 1: Composition of Mild Steel (AISI 1018)

Parameters	% Composition
Carbon	0.2 max.
Manganese	0.9 max.
Phosphorus	0.04 max.
Sulphur	0.05 max.
Iron	98.81 min.

2.2 Weight-Loss Measurements

A hole of about 0.2 cm diameter was drilled towards the edge of each of the metal coupon. The coupons were mechanically polished with silicon carbide abrasive paper, degreased with ethanol, washed with deionized water, rinsed with acetone and dried. Each of the coupons was weighed and suspended in a 250 mL glass beaker containing 200 mL of 1 M HCl and 0.1 M NaOH respectively. The corrosion vessels were charged with different concentrations (0.0 –7.5 g/L) of the pulp (inhibitor) at 301±1 K, allowed to stand for 120 hr and covered with paraffin to prevent contamination and evaporation of solution. The steel coupons were retrieved, dipped in distilled water and immersed in saturated Na₂CO₃ solution, scrubbed with bristle brush to remove residual acids and sodium hydroxide. They were washed thoroughly (and rinsed) with distilled water, dried in acetone and reweighed. Triplicate measurements were made. This procedure was repeated at 320±1 K for 24 hr progressively for 120 hr.

2.3 FT-IR Analysis

Fourier transform infrared spectrophotometer (Agilent Technologies) was used for identification of active surface functional groups of the *P. biglobosa* pulp.

2.4 Quantum Chemical Parameters

Quantum chemical parameters were evaluated using SPARTAN'14 V112 semi-empirical program using AMI method.

RESULTS AND DISCUSSION

3.1 Weight-loss Measurements

The weight loss data was used to calculate the corrosion rate (v), percentage inhibition efficiency (η) and degree of surface coverage (θ) in accordance with ASTM G1-72, 1990 [18]:

$$v = \frac{\Delta w}{At} \quad (1)$$

where Δw = average weight loss, A = exposed surface area of the mild steel coupons, and t = time of immersion in the solution.

$$\% \eta = \frac{v_o - v_i}{v_o} \times 100 \quad (2)$$

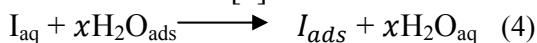
where v_o = corrosion rate in absence of inhibitor, v_i = corrosion rate in presence of inhibitor.

$$\theta = \frac{\% \eta}{100} \quad (3)$$

A clear concentration dependence of the corrosion rate and, consequently, inhibitor efficiency and fractional surface coverage, was observed as shown in Figures 1-3.

The corrosion rate decreased to a minimum value at inhibitor concentration of 4.5g/L of the corrosion medium. This change in corrosion rate had a very strong impact on the inhibition efficiency, which increased with increasing concentration of the inhibitor to reach a maximum value of 85.73% at 4.5g/L concentration of inhibitor. This shows *P. biglobosa* pulp as an effective green inhibitor for mild steel. Similar impact was observed for fractional surface coverage.

Inhibitors inhibit the corrosion process by adsorbing on the metal surface; the adsorption of inhibitor (I_{ads}) is always a displacement reaction involving removal of x number of the adsorbed water molecules from the metal surface due to differential in interaction energy between water and inhibitor molecules [3]:



The adsorption depends on the structure of inhibitor, the type of the metal and the nature of its surface, pH of the corrosion medium,

temperature and the electrochemical potential of the metal-solution interface [3].

According to Aoun [19], the observed trends (Figures 1-3) are so because increasing concentration of the inhibitor reduces the steel

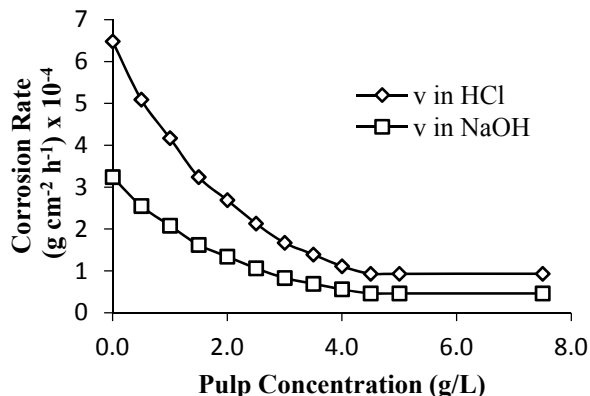


Figure 1: Corrosion rate versus pulp concentration at 301±1 K

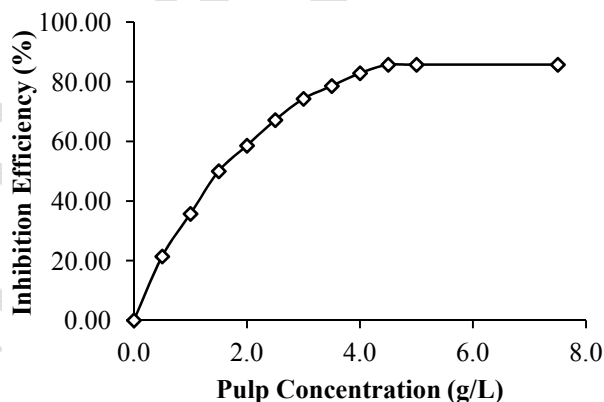


Figure 2: Inhibition efficiency versus pulp concentration 301±1 K

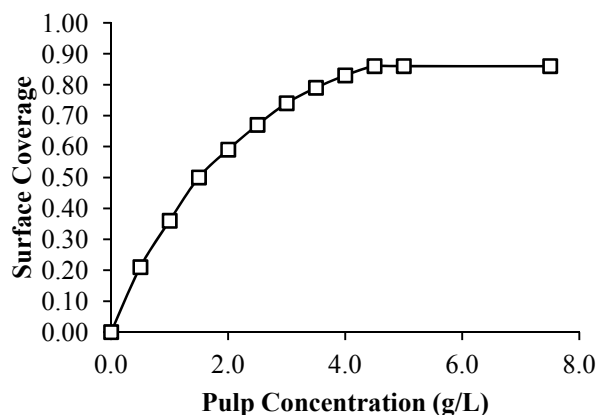


Figure 3: Surface coverage versus pulp concentration 301±1 K

surface area exposed to the corrosion media through the progressive increase in number of adsorbed inhibitor molecules on its surface, which

hinders direct attack of the corrosion agent on the metal surface.

3.2 Adsorption Study

Adsorption studies were made using the surface coverage at different concentrations to fit curves for Langmuir and El Awady isotherms [20] in their linear forms.

Langmuir isotherm:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (5)$$

where C is the concentration of the pulp in the corrosion medium and K_{ads} is the adsorption equilibrium constant. A plot of C/θ against C , which represents the Langmuir isotherm, showed linearity (Figure 4) with a very strong correlation coefficient (R^2) equal to 0.99 and slope equal to 1.351 for the process at ambient temperature (Figure 4a). Correlation of the linearity of the isotherm for the process at elevated temperature (Figure 4b) was not very strong ($R^2 = 0.77$), an indication that the isotherm may not strictly apply to the process at 321 K.

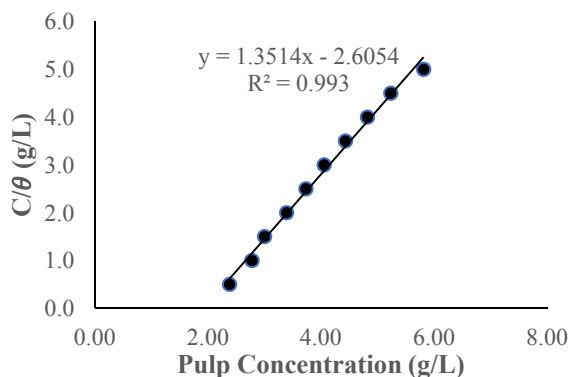


Figure 4a: Langmuir adsorption isotherm at 301 K

The strongly correlated linearity suggests adherence of the experimental data for *P. biglobosa* pulp to the Langmuir adsorption isotherm. The about 35% deviation of the slope from unity could be attributed to molecular interactions between adsorbed species from the pulp, which could result to variation in the free energy of adsorption at the adsorption sites and multiple adsorption, a phenomenon assumed to be nonexistent at conceptualization of the Langmuir model. Similar results have been reported [20].

As a check on this deviation, the data was also fitted (Figure 5) into the El Awady isotherm:

$$\log\left(\frac{\theta}{1-\theta}\right) = \log K_{ads} + y \log C \quad (6)$$

where y is the number of active sites and the adsorption equilibrium constant, $K_{ads} = K^{1/y}$. From this isotherm, $(1/y) < 1$ suggests multilayer adsorption while $(1/y) > 1$ suggests the adsorbate occupies more than one adsorption site.

The values for K_{ads} and $1/y$ calculated from Figure 5 were as presented in Table 2. $(1/y) = 2.297$, is

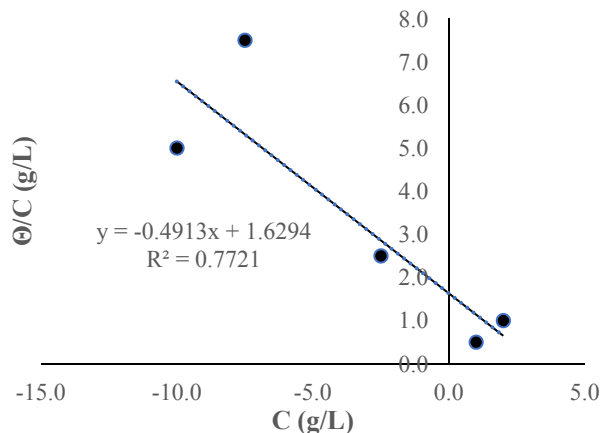


Figure 4b: Langmuir adsorption isotherm at 321 K

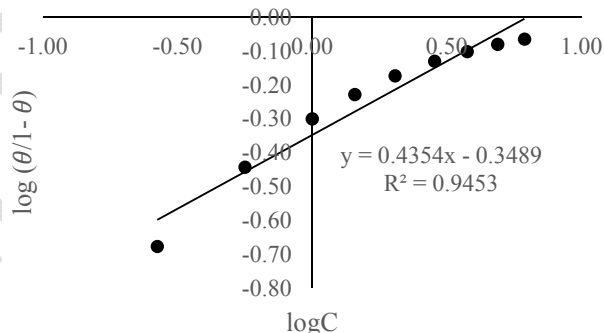
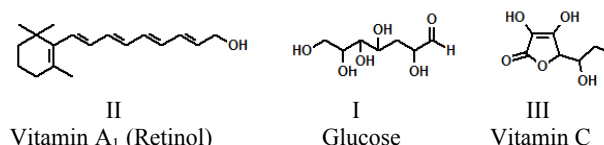


Figure 5: El Awady adsorption isotherm at 301 K

greater than 1 and shows that the adsorbed specie(s) from the *P. biglobosa* pulp occupy more than one active site, which corroborates the deviation from unity of the slope of the Langmuir isotherm.

Omojola and Ihegwau [17] reported that *P. biglobosa* pulp is very rich in glucids (80%), vitamins A, B and C; and a small quantity of gummy matter:



This composition suggests that adsorption of the adsorbate species from the pulp on the mild steel surface could be through π -electrons of aromatic ring, lone pairs of electrons of the heteroatoms, and protonated imine groups ($-N = C -$) [21]. Figure 7 shows the FT-IR results and confirms the

strong presence of an out-of-plane bending C-H bond for alkene (987.7 cm^{-1}), a C=O bond (1736.9 cm^{-1}), a broad absorbance at 2922.2 cm^{-1} and 3268.9 cm^{-1} for O-H bond for carboxylic acids and alcohols respectively. Adsorption of the adsorbate species from the pulp on the mild steel surface is schematically represented in Scheme 1.

3.3 Kinetic and Thermodynamic Study

The corrosion rates calculated for this process were fitted (Figure 6) into the pseudo first order kinetic model:

$$\log v = \log k + b \log[I] \quad (7)$$

where k = rate constant ($\text{g cm}^{-2}\text{ h}^{-1}$), b = order of reaction with respect to inhibitor, which is a measure of the inhibitor efficiency in this case and $[I]$ = concentration of the inhibitor (pulp).

Scheme 1: Interaction between steel surface and pulp molecules

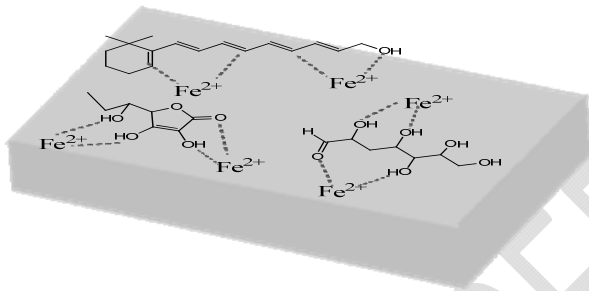


Table 2: Calculated parameters from Langmuir and El Awady isotherms

Parameters	301 K		321 K
	Langmuir Isotherm	El Awady Isotherm	Langmuir Isotherm
Slope	1.351	0.435	-0.491
R^2	0.993	0.945	0.772
K_{ads}	0.384	0.448	0.614
$1/y$	N/A*	2.297	N/A*

*N/A = not applicable

Figures 6a and 6b give a good linearity ($R^2 = 0.93$ and 0.95) for the corrosion process in these media. The values k (derived from the y-intercept) and b (calculated from the slope) are presented in Table 3. The near-unity values of b , especially at 301 K suggests that the process obeys pseudo first order kinetics.

The values of k at 301 K are very large compared to that at 321 K. Large values of k show that the

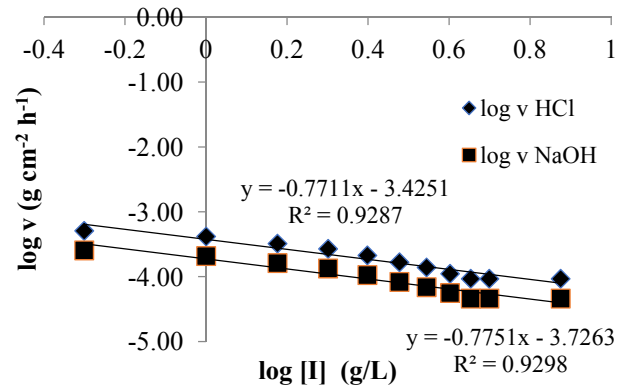


Figure 6a: Variation of $\log v$ with $\log[I]$ at 301 K

corrosion of mild steel is a fast reaction at ambient temperature but very slow at elevated temperatures. The k values also show that mild steel would corrode faster in alkaline (NaOH) environment than in acidic (HCl) environment. This, seemingly, contradicts the observation that weight loss in the acidic medium was more than that in the basic medium, more so that k is independent of concentration. However, mild steel resists corrosion in certain specific environments such as H_2SO_4 and NaOH solutions [22], which might account for the less weight loss in NaOH. The negative value of b reflects the inverse-proportionality of the corrosion rate to the inhibitor concentration, and its near-unity value reflects good inhibitory properties of the *P. biglobosa* pulp ($\eta = 85.76 \approx 86\%$) at ambient temperature.

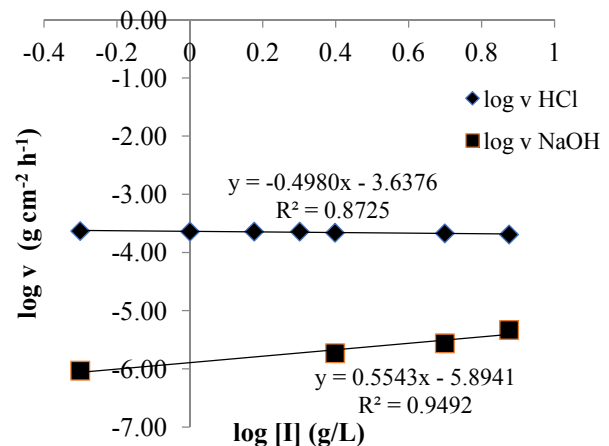


Figure 6b: Variation of $\log v$ with $\log[I]$ at 321 K

The adsorption equilibrium constant (K_{ads}) for the corrosion process calculated from the Langmuir and El Awady isotherms is related to the standard free energy of adsorption (ΔG_{ads}^0) by the expression,

$$\Delta G_{ads}^o = -2.303RT \log(55.5K_{ads}) \quad (8)$$

Table 3: Kinetic and thermodynamic parameters

Parameters	Temperature (K)			
	301		321	
	1.0M HCl	0.1M NaOH	1.0M HCl	0.1M NaOH
k (Lcm ⁻² h ⁻¹)	2.66x10 ³	5.33x10 ³	2.30x10 ⁻⁴	1.28x10 ⁻⁶
b (Lcm ⁻² h ⁻¹)	-0.86	-0.86	-0.498	0.55
ΔG_{ads}^o (kJmol ⁻¹)	-7.66 Langmuir -8.04 Awady		-9.42 Langmuir	

Table 4: Quantum parameters of glucose, vitamin A₁ and vitamin C

Molecule	Molecular Weight (g)	E_{Homo} (eV)	E_{Lumo} (eV)	$E_{Homo}-E_{Lumo}$ (eV)	Dipole Moment (D)	CPK Volume (Å)	Polarizability	log P
I	194.183	-10.58	-0.74	9.76	1.84	182.29	53.50	-2.94
II	258.405	-8.21	-0.58	7.71	2.05	316.06	65.21	4.34
III	174.152	-9.68	-0.56	9.12	3.13	161.85	52.35	-1.51

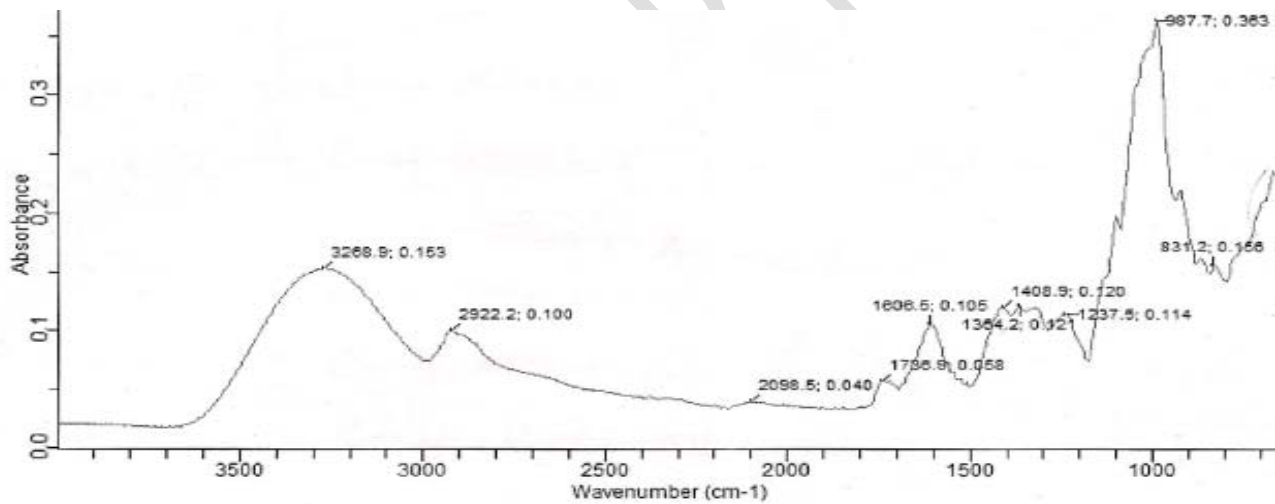
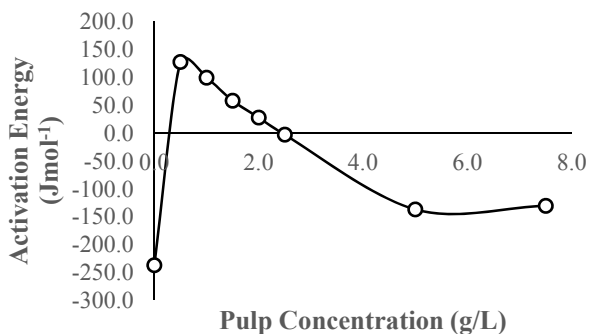


Figure 7: FT-IR spectrum of *Parkia biglobosa* pulp.



Figures 8: Activation energy profile in presence of *P. biglobosa* pulp

where R is the gas constant, T is the thermodynamic temperature and 55.5 is the molar concentration of water in the solution.

Activation energy (E_a) of the process was evaluated using modified Arrhenius equation to relate corrosion rate and temperature,

$$\log\left(\frac{v_2}{v_1}\right) = \frac{E_a}{2.303R} \left(\frac{1}{T_1} - \frac{1}{T_2}\right) \quad (9)$$

where v_1 and v_2 are the corrosion rates at two temperatures T_1 and T_2 respectively and R is the gas constant.

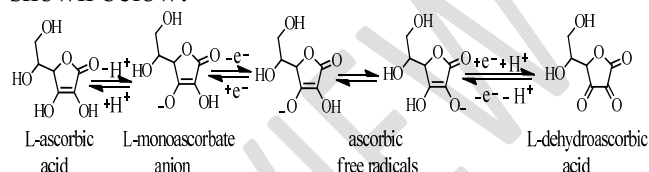
From Table 3, the constant b , which is also a measure of the efficiency of an inhibitor, has best values of 86 % and 55 % at 301 K and 321 K respectively. This shows that the inhibition efficiency of the *P. biglobosa* pulp decreases with increase in temperature. It's been reported [20] that for an inhibitor whose inhibition efficiency decreases with increase in temperature, the value of the activation energy (E_a) for the process would be greater than that for the uninhibited process.

Figure 8 presents the activation energy profile for this process; which shows that the activation energy for the inhibited process (127.3 Jmol^{-1}) is greater than that for the uninhibited process ($-237.29 \text{ Jmol}^{-1}$) and decreases with increase in concentration of the *P. biglobosa* pulp to fairly steady at a value (-134.0 Jmol^{-1}), still higher than that of the uninhibited process. Higher values of activation energy (in the presence of inhibitor) suggests that physisorption (physical adsorption) dominates in this process. This is collaborated by the standard free energy of adsorption (ΔG_{ads}^o).

The ΔG_{ads}^o values, as presented in Table 3, were all negative for the process at both ambient and elevated temperature. This suggests spontaneity of the adsorption process and stability of the adsorbed layer on the steel surface. It has been reported [20] that physisorption processes usually have $\Delta G_{ads}^o < -40 \text{ kJmol}^{-1}$, and above this value, sorption is of the chemisorption mechanism. This suggests, therefore, that the adsorption process here was physical (i.e. physisorption) since the ΔG_{ads}^o values (-7.66 , -8.04 and -9.42 kJmol^{-1}) are less than -20.0 kJmol^{-1} .

The b values, which as stated earlier, is the order of reaction with respect to inhibitor and a measure of the inhibitor efficiency, was observed to be same, 0.86 (86%), for both acid and alkaline medium at 301 K. At 321 K, however, the inhibition efficiency value of the inhibitor is not same for these corrosion media – $0.498 \approx 0.50$ (50%) for acidic medium and 0.55 (55%) for alkaline medium. This could be attributed to the chemical stability of the inhibitor constituents,

which is dependent on combined effects of temperature and pH of the solution [23]. The ionization of ascorbic acid (III), for instance, generates the formation of the L-monoascorbate and ascorbate anions, which are also able to adsorb on the metallic surface or to originate other products that can act in the same way. The L-monoascorbate anion is a very reactive species, which can be easily oxidized to L-dehydroascorbic acid; the main specie believed to be involved in the inhibition process [24] as shown below:



Higher inhibition efficiency would, therefore, be observed at temperature/pH levels where the concentration of L-dehydroascorbic acid is higher than that of ascorbate anions.

3.4 Quantum Chemical Analysis

Better adsorption of inhibitor molecules on the metal surface is what inhibition of corrosion reaction depends on. The effectiveness of a material as a corrosion inhibitor, therefore, strongly depends on its molecular structure. The calculated quantum chemical parameters of the dominant phytochemical content [17] of *P. biglobosa* pulp (molecules I, II and III) were as listed in Table 4. This Table show relatively higher value of HOMO (highest occupied molecular orbital) energy and lower LUMO (lowest unoccupied molecular orbital) energy. This favors bonding with the metal surface since E_{HOMO} is often associated with electron-donating ability of the molecule. A plot of HOMO and LUMO properties of these molecules revealed that the frontier orbital, used in predicting the adsorption centers of the inhibitor molecule, were distributed around the carbonyl/ α -hydroxyl group, conjugate double bond chain and the heterocyclic ring of molecule I, II and III respectively. This suggests that more than one active site could be obtained from the adsorbing species from the *P. biglobosa* pulp.

The energy gap, $E_{HOMO} - E_{LUMO}$, for the molecules I, II, and III is in the order $I > III > II$. A large value of $E_{HOMO} - E_{LUMO}$ indicates low reactivity to a chemical specie since energy gap relates softness or hardness of the molecule [25]. A soft

molecule is more reactive than a hard molecule and from the results, molecule II is softer and likely to show more involvement in the process. The substituent constant, $\log P$, of these molecules were as presented in Table 3. These values have also been reported [25] to have a good relationship with corrosion inhibition efficiency of the inhibitor. It accounts for the hydrophobicity of a molecule, which is related to the mechanism of formation of oxide/hydroxide layer on the metal surface. The corrosion process is substantially reduced by this layer. This implies that the higher the $\log P$ value, the higher the inhibition efficiency of the inhibitor, suggesting that molecule II is likely to provide higher corrosion inhibition efficiency than I & III. This is also collaborated by the polarizability values since the minimum polarizability principle (MPP) requires that the natural direction of any evolving system would be towards a state of minimum polarizability [25].

CONCLUSION

From the foregoing results, *P. biglobosa* pulp, a cellulosic renewable waste, is a good green inhibitor for mild steel corrosion in both acidic and alkaline medium. There is an optimum concentration beyond which the amount of pulp added has no positive effect on the efficiency of inhibition, which decreases with increase in temperature of the corrosion medium. The adsorption of the adsorbate is spontaneous, the process proceeds through physisorption and the adsorbed layer is stable.

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