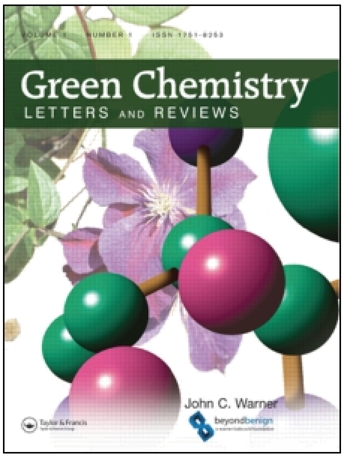

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Nnabuk O. Eddy[a]; Steven A. Odoemelam[b]; Anduang O. Odiongenyi[b]
[a] Department of Chemistry, Ahmadu Bello University, Zaria, Kaduna State, Nigeria [b] Department of Chemistry, Michael Okpara University of Agriculture, Umudike, Abia State, Nigeria

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RESEARCH LETTER

Inhibitive, adsorption and synergistic studies on ethanol extract of *Gnetum Africana* as green corrosion inhibitor for mild steel in H_2SO_4

Nnabuk O. Eddy[a]*, Steven A. Odoemelam[b] and Anduang O. Odiongenyi[b]

[a]*Department of Chemistry, Ahmadu Bello University, Zaria, Kaduna State, Nigeria;* [b]*Department of Chemistry, Michael Okpara University of Agriculture, Umudike, Abia State, Nigeria*

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The inhibitive and adsorptive characteristics of ethanol extract of *Gnetum Africana* for the corrosion of mild steel in H_2SO_4 solutions have been studied using weight loss, gasometric, thermometric, and IR methods of monitoring corrosion. Ethanol extract of *Gnetum Africana* is a good adsorption inhibitor for the corrosion of mild steel in H_2SO_4. The inhibitive property of the extract is attributed to the presence of alkaloid, saponin, tannin, terpene, anthraquinone, cardiac glycoside, and alkaloid in the extract. The adsorption of the inhibitor on mild steel surface is exothermic, spontaneous and is consistent with the mechanism of physical adsorption. In addition, Langmuir and Temkin adsorption isotherms best described the adsorption characteristics of the inhibitor. Efforts to improve the adsorption of the inhibitor through synergistic combinations with halides indicated that only KCl may enhance the efficiency of the inhibitor. The study provides information on the use of ethanol extract of *Gnetum Africana* as a corrosion inhibitor for mild steel.

Keywords: green corrosion inhibitors; mild steel; *Gnetum Africana*; H_2SO_4

Introduction

Industrial processes such as acid prickling, acid cleaning, and etching often involve contact between mild steel and an acidic medium, which implies that the use of inhibitors is necessary (*1–3*). Most corrosion inhibitors are either synthesized from cheap raw materials or are chosen from organic compounds containing electronegative functional groups and π-electrons in triple or conjugated double bonds. The presence of aromatic rings and hetero atoms (such as S, N, O, and P) are the major adsorption centers for these inhibitors (*4–6*). It has been found that the level of protection of organic corrosion inhibitors depends on the nature of the adsorption of the inhibitor on the metal surface, the nature, and surface charge of the metal, the mode of adsorption of the inhibitor, chemical structure of the inhibitor, the type of aggressive solution, etc. (*7*,*8*).

Despite the broad spectrum of organic compounds available as corrosion inhibitors, the successful utilization of most corrosion inhibitors has been hindered by their toxic nature (*9*). Green corrosion inhibitors are biodegradable and do not contain heavy metals or other toxic compounds. The successful use of naturally occurring substances to inhibit the corrosion of metals in acidic and alkaline environments has been reported by some research groups (*10–20*). However, to our knowledge, literature is scanty on the use of ethanol extract of *Gnetum Africana* as an inhibitor for the corrosion of mild steel in H_2SO_4 solution. Therefore, our present focus is aimed at investigating the adsorption and inhibitive properties of ethanol extract of *Gnetum Africana* (as a green corrosion inhibitor) for the corrosion of mild steel in H_2SO_4 solutions.

Gnetum Africana (Afang) occurs naturally in the humid forest zones within Nigeria, Central African Republic, and Angola. Fresh leaves of Afang are widely used as vegetable and for the treatment of piles, high blood pressure, enlarged spleen, sore throat, and as a purgative. Afang contains C-glycosylflavones, including 2-xylosylisoswertisin and 2-glucosylisoswertisin compounds. According to Ekop (*21*), the proximate composition of Afang includes moisture (37.39%), ash (4.72%), oil (14.2%), protein (10.18%), and some amino acids.

Results and discussions

Effect of ethanol extract of Gnetum Africana on the corrosion of mild steel

Figures 1 and 2 show the variation of volume of hydrogen gas evolved with time for the corrosion of

*Corresponding author. Email: nabukeddy@yahoo.com

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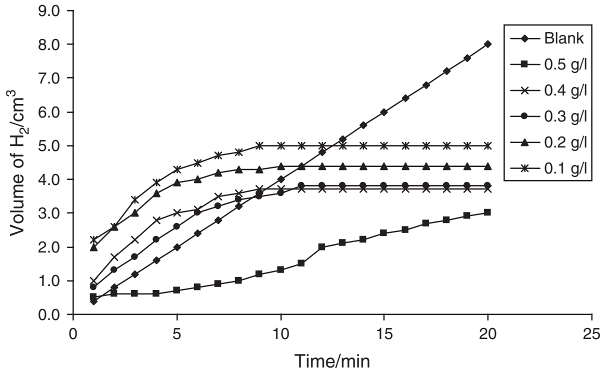

Figure 1. Variation of volume of hydrogen gas evolved with time for the corrosion of mild steel in 2.5M tetraoxosulphate (VI) acid containing various concentrations of ethanol extract of *Gnetum Africana* at 303 K.

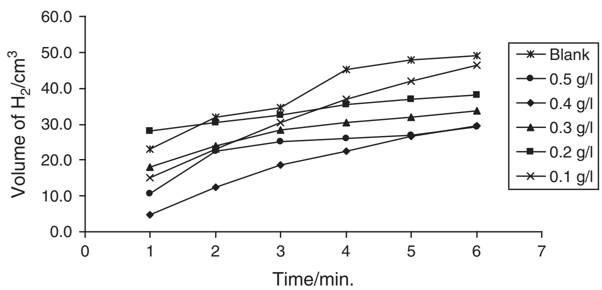

Figure 2. Variation of volume of hydrogen gas evolved with time for the corrosion of mild steel in H_2SO_4 containing various concentrations of ethanol of *Gnetum Africana* at 333 K.

mild steel in 2.5M H_2SO_4 containing various concentrations of ethanol extract of *Gnetum Africana* at 303 and 333 K, respectively. From the figures, it was found that the volume of hydrogen gas evolved increases with increase in temperature, but decreases with increasing concentration of ethanol extract of *Gnetum Africana,* indicating that:

1. the rate of corrosion of mild steel increases with increase in temperature, but decreases with increase in the concentration of ethanol extract of *Gnetum Africana*; and
2. ethanol extract of *Gnetum Africana* inhibited the corrosion of mild steel in H_2SO_4.

Figure 3 shows the variation of weight loss of mild steel with time for the corrosion of mild steel in 0.1M H_2SO_4 containing various concentrations of ethanol extract of *Gnetum Africana* at 303 K. From the figure, it was found that weight loss of mild steel increases with increase in the period of contact, but decreases with increase in the concentration of ethanol extract of *Gnetum Africana* implying that ethanol extract of *Gnetum Africana* is an adsorption inhibitor for the

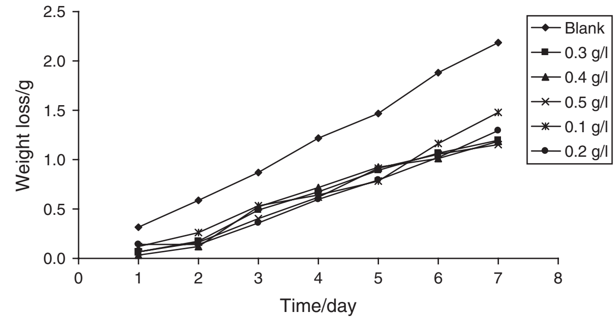

Figure 3. Variation of weight loss with time for the corrosion of mild steel tetraoxosulphate (VI) acid containing various concentrations of ethanol extract of *Gnetum Africana*.

corrosion of mild steel in H_2SO_4 and that the corrosion rates of mild steel increases with increase in the period of contact.

Values of the corrosion rates of mild steel in H_2SO_4 and inhibition efficiencies of *Gnetum Africana* calculated from gasometric, thermometric, and gravimetric data are presented in Table 1. The results indicated that the corrosion rate of mild steel increases with increase in temperature while the inhibition efficiency of ethanol extract of *Gnetum Africana* increases with increasing concentration of ethanol extract of *Gnetum Africana*. This confirms that ethanol extract of *Gnetum Africana* is an adsorption inhibitor for the corrosion of mild steel in H_2SO_4.

Kinetic consideration and effect of temperature on the corrosion of mild steel

Data obtained from weight loss measurements were used to test curves for the order of the corrosion of mild steel in various media. The tests revealed that the plots of –log (weight loss) versus time were linear which indicated that a first order kinetic is applicable to the corrosion of mild steel in the absence and presence of ethanol extract of *Gnetum Africana*. In addition, the half lives of the corrosion of mild steel in various media were calculated using the relation:

$$t_{1/2} = 0.693/k_1, \quad (1)$$

where k_1 is the rate constant and $t_{1/2}$ is the half life in days (*23–25*). Values of rate constant (k_1) and half life ($t_{1/2}$) calculated through the slopes of the kinetic plots are recorded in Table 2. The results revealed that the rate constant and half life of mild steel corrosion do not vary significantly with the concentration of the extract indicating that the assumptions of the rate models is applicable to the kinetic of the corrosion of mild steel in the absence and presence of ethanol extract of *Gnetum Africana*.

Table 1. Inhibition efficiencies of various concentrations of ethanol extract of *Gnetum Africana* for mild steel corrosion in H_2SO_4.

C (gL^{-1})	WL	T	G (303 K)	G (333 K)	CR (303 K)	CR (333 K)	Extract + 0.06M KBr (WL)	Extract + 0.06M KI (WL)	Extract + 0.06M KCl (WL)
Blank	–	–	–	–	0.38	2.94	–	–	–
0.1	52.77	37.15	43.33	60.53	0.15	1.16	6.63	13.85	9.28
0.2	61.59	37.76	54.32	61.84	0.14	1.12	12.75	15.00	26.20
0.3	64.38	39.13	62.34	63.16	0.14	1.08	12.80	23.46	26.93
0.4	75.51	46.01	68.43	67.11	0.13	0.97	13.63	26.06	33.88
0.5	79.01	46.89	72.12	67.11	0.12	0.97	20.03	27.02	36.31

Note: WL = results from weight loss method; T = results from thermometric method; and G = results from gasometric method. CR (303 K) and CR (333 K) are the corrosion rates of mild steel (in cm^3min^{-1}) at 303 and 333 K.

In order to study the effect of temperature on the corrosion of mild steel in the absence and presence of ethanol extract of *Gnetum Africana*, the Arrhenius Equation (2) was used (*24*):

$$\log\frac{CR_2}{CR_1}=\frac{E_a}{2.303R}\left(\frac{1}{T_1}-\frac{1}{T_2}\right), \tag{2}$$

where CR_1 and CR_2 are the corrosion rates of mild steel at 303 K (T_1) and 333 K (T_2). Values of the activation energy calculated from Equation (2) are recorded in Table 2. The activation energies were found to increase with increase in the concentration of ethanol extract of *Gnetum Africana* indicating that there is an increasing ease of adsorption of the inhibitor as its concentration increases. In addition, the activation energies were lower than the threshold value of 80 $kJmol^{-1}$ indicating that the adsorption of ethanol extract of *Gnetum Africana* on the mild steel surface occurred according to the mechanism of physical adsorption.

Thermodynamic/adsorption considerations

The heat of adsorption (Q_{ads}) of ethanol extract of *Gnetum Africana* on the surface of mild steel was calculated using the following equation (*25*):

$$Q_{ads}=2.303R[\log(\theta_2/1-\theta_1)-\log(\theta_1/1-\theta_1)] \times (T_1\times T_2)/(T_2-T_1). \tag{3}$$

Values of Q_{ads} calculated from Equation (3) are recorded in Table 2. These values are negative and ranged from –18.9897 to –63.7138 $kJmol^{-1}$ indicating that the adsorption of the extract is exothermic.

In order to study the adsorption characteristics of ethanol extract of *Gnetum Africana* on mild steel surface, data obtained for degree of surface coverage were used to fit curves for different adsorption isotherms including Langmuir, Temkin, Flory-Huggins, Frumkin, Freundlich, and Brockris Swinkel adsorption isotherms. The tests indicated that Langmuir and Temkin adsorption isotherms best described the adsorption characteristics of ethanol extract of *Gnetum Africana* on mild steel surface.

The assumptions of Langmuir adsorption isotherm can be written as follows (*26*):

$$\log(C/\theta)=\log C-\log K, \tag{4}$$

where K is the adsorption equilibrium constant and θ is the degree of surface coverage of the inhibitor. Using Equation (4), the plots of $\log(C/\theta)$ versus $\log C$ were linear confirming the application of Langmuir adsorption isotherm to the adsorption of ethanol extract of *Gnetum Africana* on mild steel surface (Figure 4). Values of adsorption parameters deduced from Langmuir adsorption isotherm are presented in Table 3. The results indicated that the slopes and R^2 values were close to unity which also indicated strong adherence to

Table 2. Kinetics and thermodynamic parameters for the adsorption of ethanol extract of *Gnetum Africana* on mild steel surface.

C (gdm^{-3})	E_a ($kJmol^{-1}$)	Q_{ads} ($Jmol^{-1}$)	K_1	$t_{1/2}$ (day)	R^2
Blank	22.42	–	0.2	4	0.9998
0.1	67.63	−63.71	0.4	2	0.9487
0.2	71.80	−34.81	0.4	2	0.9487
0.3	72.80	−30.32	0.4	2	0.8588
0.4	74.76	−33.42	0.4	2	0.8043
0.5	77.38	−18.99	0.4	2	0.8910

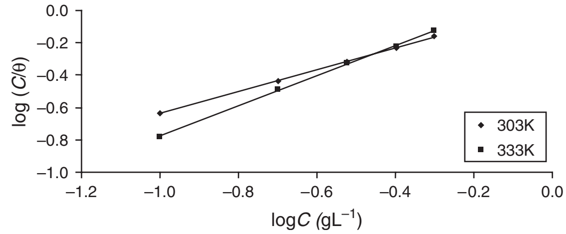

Figure 4. Langmuir isotherm for the adsorption of ethanol extract of *Gnetum Africana* on the surface of mild steel.

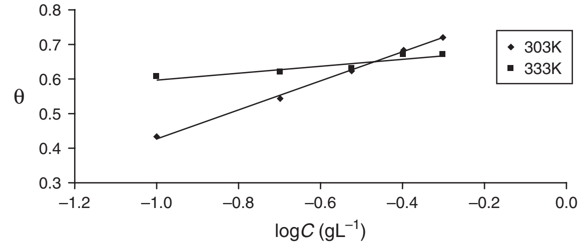

Figure 5. Temkin isotherm for the adsorption of ethanol extract of *Gnetum Africana* on the surface of mild steel.

the assumptions of Langmuir adsorption isotherm. Therefore, there is no interaction between the adsorbed species.

According to Temkin adsorption isotherm, the degree of surface coverage (θ) is related to the inhibitor's concentration (*C*) as follows (*27*):

$$\theta = \frac{-\ln k}{2a} - \frac{\ln C}{2a}, \quad (5)$$

where *K* is the equilibrium constant of adsorption and "a" is the attractive parameter. From Equation (5), a plot of θ versus log*C* should be linear provided the assumptions of Temkin isotherm are valid. Figure 5 shows Temkin plots for the adsorption of ethanol extract of *Gnetum Africana* on the surface of mild steel. Values of adsorption parameters deduced from Temkin plots are also presented in Table 3. From the results, the attractive parameters were found to be positive indicating the attractive behavior of the inhibitor.

The equilibrium constant of adsorption of ethanol extract on the surface of mild steel is related to the free energy of adsorption as follows (*28*):

$$\Delta G_{ads} = -2.303RT\log(55.5K), \quad (6)$$

where *R* is the gas constant in $kJmol^{-1}$, T is the temperature in Kelvin, *K* is the equilibrium constant of adsorption and 55.5 is the molar concentration of H_2SO_4 in water. Values of *K* deduced from Langmuir and Temkin plots were used to calculate ΔG_{ads} using Equation (6). Calculated values of ΔG_{ads} are recorded in Table 3. The results indicated that ΔG_{ads} values are negatively less than the threshold value of -40 $kJmol^{-1}$ required for chemical adsorption. Therefore, the adsorption of ethanol extract of *Gnetum Africana* on the surface of mild steel is spontaneous and supported the mechanism of physical adsorption.

Phytochemical constituents of ethanol extract of Gnetum Africana

Table 4 shows the phytochemical constituents of ethanol extract of *Gnetum Africana*. The constituents analyzed were saponin, terpenes, tannins, flavonoid, phlobatanins, anthraquinones, cardiac glycosides, and alkaloids. The results indicated that these phytochemicals are present only in ethanol extract of *Gnetum Africana* and not in the aqueous extract (except saponin and terpenes), which justified the choice of ethanol extract of the leaves for the study. We therefore concluded that the inhibitive and adsorption properties of ethanol extract of *Gnetum Africana* is due to the presence of saponin, terpenes, tannins, flavonoid, phlobatanins, anthraquinones, cardiac glycosides, alkaloids, and some amino acids in the extract of the plants. These phytochemicals and amino acids are rich in electronegative functional groups and π-electrons in triple or conjugated double bonds hence they are expected to be efficient corrosion inhibitors (*29–32*).

Table 3. Langmuir and Temkin adsorption parameters for the adsorption of ethanol extract of *Gnetum Africana* on the surface of mild steel.

Langmuir	Temperature (K)	log*K*	Slope	ΔG_{ads} ($kJmol^{-1}$)	R^2
	303	0.0401	0.6783	−10.33	0.9997
	333	0.1541	0.9308	−12.08	0.9991
Temkin	**Temperature (K)**	**log*K***	**a**	**ΔG_{ads} ($kJmol^{-1}$)**	**R^2**
	303	0.6989	1.196	−14.99	0.9967
	333	0.5448	4.916	−15.50	0.8579

Table 4. Phytochemical constituents of ethanol and aqueous extract of *Gnetum Africana*.

Phytochemicals	Aqueous extract	Ethanol extract
Saponins	++	++
Terpenes	++	++
Tannins	–	++
Flavonoid	–	–
Phlobatannins	–	++
Anthraquinones	–	++
Cardiac glycoside	–	+++
Alkaloids	–	+++

Note: +++, highly present; ++, moderately present; –, absent or presence in negligible quantity.

IR study of the inhibitor and the corrosion product of mild steel

Figures 6 and 7 show the IR spectra of the corrosion product of mild steel in the absence and presence of ethanol extract of *Gnetum Africana*, respectively. Figure 8 shows the IR spectrum of ethanol extract of the inhibitor. From Figure 6, it can be seen that the corrosion product of mild steel in the absence of the inhibitor is not IR active. Table 5 shows the frequencies of adsorption of IR by ethanol extract of *Gnetum Africana* and the corrosion product of mild steel when the extract was used as an inhibitor.

Comparing Figures 6 and 7, it was found that the –OH stretch frequencies (3416.46 and 2997.23 cm^{-1}) were shifted to 3434.30 cm^{-1}, the N–H bend at 1536.41 cm^{-1} was shifted to 1639.18 cm^{-1} and the C–O stretch at 1241.16 and 1080.62 cm^{-1} were shifted to 1045.81 and 1130.91 cm^{-1}, respectively. These shifts in frequencies indicated that there is an interaction between the inhibitor and the surface of mild steel (*33,34*). However, the C–H stretch frequencies (at 878.42 and 633.85 cm^{-1}), the carbon–carbon triple bond frequency (2105.78 cm^{-1}) and the C–H scissoring and bending frequency (1403.93 cm^{-1}) were missing in the spectrum of the corrosion product indicating that these bonds might have been used for bonding between the vacant d orbital of Fe and the inhibitor (*35*).

Synergism

Synergism is a combined action of a compound greater in total effect than the sum of individual effects. It has become one of the most important factors in inhibition process and serves as a basis for all modern corrosion inhibitor formulation. Synergism of corrosion inhibitors is either due to interaction between components of the inhibitors or due to interaction between the inhibitor and one of the ions present in aqueous solution (*36*).

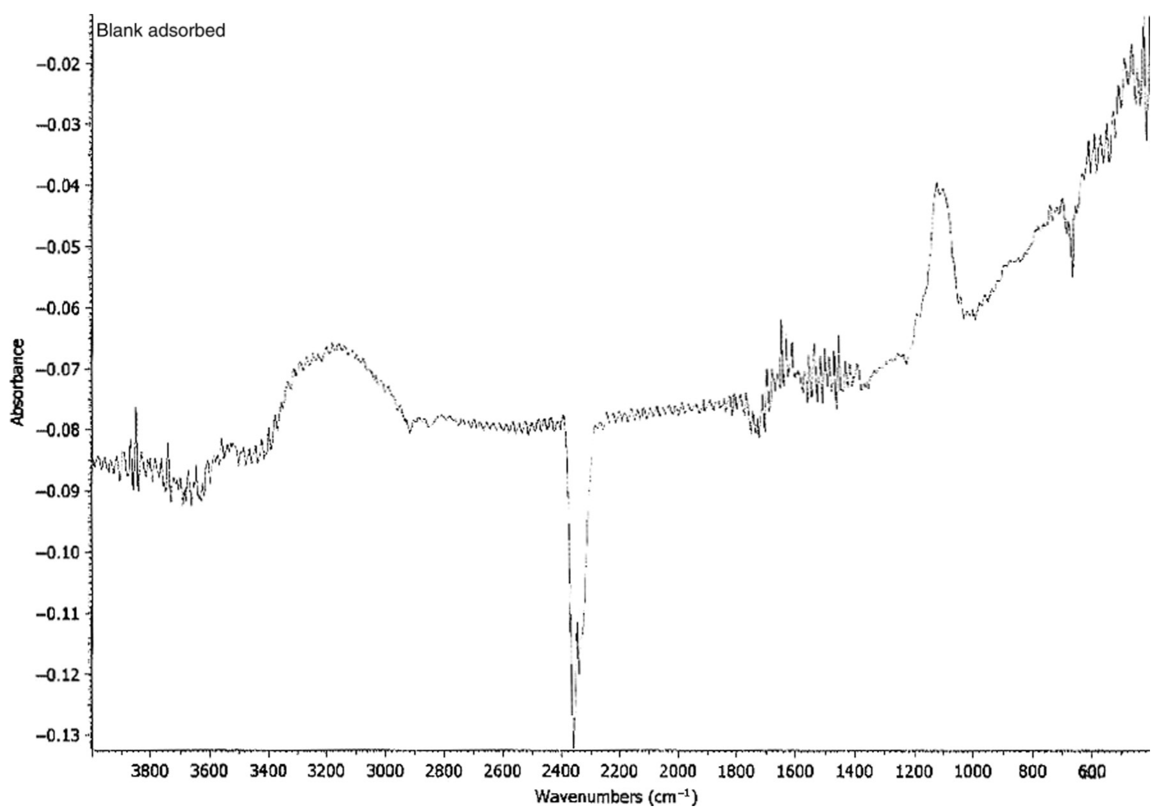

Figure 6. IR spectrum of the corrosion product of mild steel (without inhibitor).

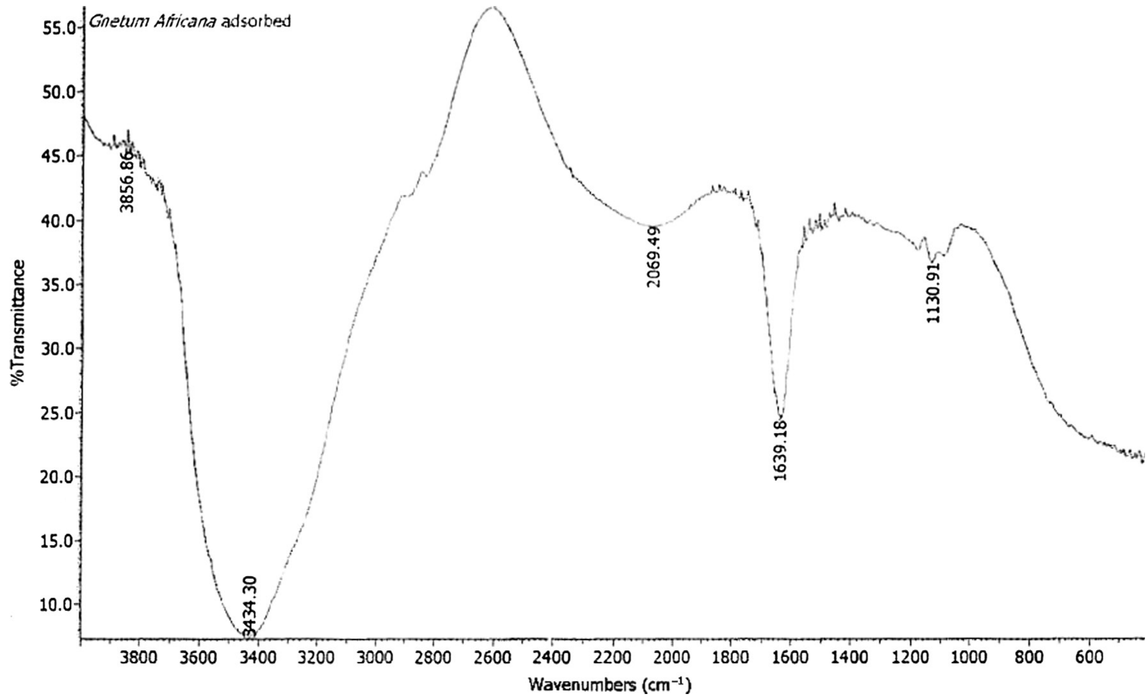

Figure 7. IR spectrum of the corrosion product (with the inhibitor).

Synergistic studies were carried out on the combination of the inhibitor with 0.06M KI, KCl, and KBr, respectively. Synergism parameter (*S*) of the inhibitor due to halide was calculated using Equation (7):

$$S = \frac{1 - I_A - I_B + I_A I_B}{1 - I_{AB}}, \qquad (7)$$

where I_A and I_B are inhibition efficiencies of the inhibitor and halide, respectively, and I_{AB} is the inhibition efficiency of the inhibitor, when it is combined with halide. Values of *S* calculated from Equation (7) are recorded in Table 6. These values were found to be less than unity (except for KCl) indicating that the adsorption of KI and KBr on the surface of mild steel antagonizes the adsorption of

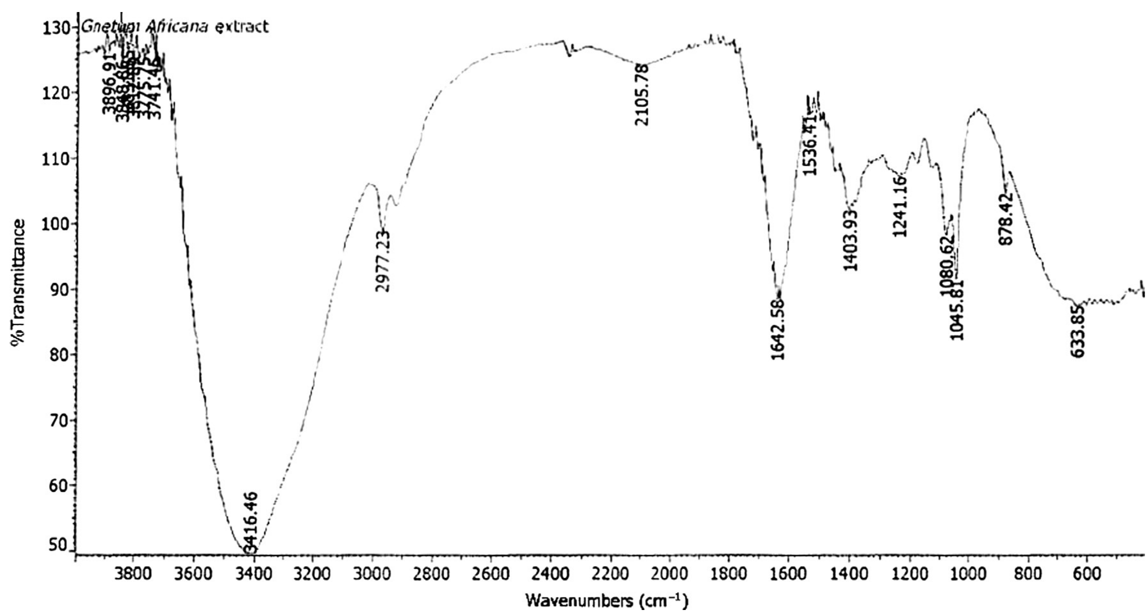

Figure 8. IR spectrum of the inhibitor.

Table 5. Frequencies and peaks of adsorption of IR by ethanol extract of *Gnetum Africana*.

Pure extract			Extract adsorbed on mild steel		
Frequency (cm^{-1})	Height (cm)	Assignment	Frequency (cm^{-1})	Height (cm)	Assignment
3416.46	49.339	−OH stretch	3434.30	7.301	O–H stretch
2977.23	98.714	−OH stretch	2069.49	39.440	
2105.78	124.299	—C≡C—	1639.18	24.257	N–H bend, NO_2 asymmetrical stretch
1642.58	88.295	C=C	1130.91	36.616	C–N stretch, C–O stretch
1536.41	116.494	N–H bend			
1403.93	101.956	C–H scissoring and bending			
1241.16	107.148	C–O stretch, C–N stretch			
1080.62	98.048	C–O stretch, C–N stretch			
1045.81	91.392	CO stretch, C–N stretch			
878.42	104.556	C–H stretch			
633.85	87.335	C–H bend			

the inhibitor and that the performance of ethanol extract of *Gnetum Africana* as an inhibitor can be enhanced by synergistic combination of KCl and not KI and KBr.

Experimental

Materials

Materials used for the study were mild steel sheet of composition (wt%, as determined by quantiometric method) Mn (0.6), P (0.36), C (0.15), and Si (0.03) and the rest Fe. The sheet was mechanically pressed and cut into different coupons, each of dimension, $5 \times 4 \times 0.11$ cm. Each coupon was degreased by washing with ethanol, cleaned with acetone and allowed to dry in the air before preservation in a desiccator. All reagents (H_2SO_4, KI, KCl, and KBr) used for the study were analar grade and double distilled water was used for their preparation. Concentrations of H_2SO_4 used for gasometric, thermometric, and weight loss studies were 2.5, 2.5, and 0.1M, respectively.

Table 6. Synergistic parameters of ethanol extract of *Gnetum Africana* combined with 0.06M KBr, KI, and KCl, respectively.

C (gL^{-1})	Extract + 0.06M KBr	Extract + 0.06M KI	Extract + 0.06M KCl
0.1	−32.23	−49.20	86.03
0.2	−15.71	−45.92	28.74
0.3	−16.22	−29.69	28.96
0.4	−17.89	−31.41	26.92
0.5	−12.11	−30.85	25.55

Extraction of plants

Samples of *Gnetum Africana* were obtained from Modern Botanical garden located at Chubb Road, Ikot Ekpene, South South Nigeria. The leaves were sun-dried, ground, and soaked in a solution of ethanol for 48 hours. After 48 hours, the samples were cooled and filtered. The filtrates were subjected to evaporation at 352 K in order to leave the sample free of the ethanol. The stock solutions of the extract so obtained were used in preparing different concentrations of the extract by dissolving 0.1, 0.2, 0.3, 0.4, and 0.5 g of the extract in 1 L solution of 2.5 or 0.1M of H_2SO_4, respectively.

Chemical analysis

Phytochemical analysis of ethanol and aqueous extract of the sample was carried out according to the method reported by Odiongenyi et al. (*17*). For the identification of saponin, frothing, and Na_2CO_3 tests were adopted. For the identification of tannin, bromine water, and ferric chloride tests were used. For the identification of cardiac glycodises, Leberman's and Salkowski's tests were adopted and for the identification of alkaloid, Dragendorf, Hagger, and Meyer reagents were used.

Infrared analysis

IR analysis of the corrosion product of mild steel and ethanol extract of *Gnetum Africana* were carried out using BUCK model 500M infrared spectrophotometer. The sample was prepared using KBr and the analysis was done by scanning the sample through a wave number range of 400–4000 cm^{-1}.

Gasometric method

Gasometric methods were carried out at 303 and 333 K as reported elsewhere (*22*). From the volume of hydrogen gas evolved per minute, inhibition efficiency (%I), degree of surface coverage (θ), and corrosion rate were calculated using Equations (8–10), respectively:

$$\%I = \left(1 - \frac{V^1_{Ht}}{V^0_{Ht}}\right) \times 100, \quad (8)$$

$$\theta = \%I/100 = 1 - \frac{V^1_{Ht}}{V^0_{Ht}}, \quad (9)$$

$$CR = (V^0_{Ht} - V^1_{Ht})/t, \quad (10)$$

where V^1_{Ht} and V^0_{Ht} are the volumes of H_2 gas evolved at time t for inhibited and uninhibited solutions, respectively. CR is the corrosion rate of mild steel in cm^3min^{-1}.

Thermometric method

This was also carried out as reported elsewhere (*23*,*24*). From the rise in temperature of the system per minute, the reaction number was calculated using Equation (11):

$$RN(^\circ C\ min^{-1}) = \frac{T_m - T_i}{t}, \quad (11)$$

where RN is the reaction number in °C/min, T_m and T_i are the maximum and initial temperatures attained by the systems, respectively, and "t" is the time (min) taken to attain the maximum temperature. From the reaction numbers, the %I of the inhibitor was calculated using the following equation:

$$\%I = \frac{RN_{aq} - RN_{wi}}{RN_{aq}} \times 100, \quad (12)$$

where RN_{aq} is the reaction number in the absence of inhibitors (blank solution) and RN_{wi} is the reaction number of 2M H_2SO_4 containing studied inhibitors.

Gravimetric analysis

In the gravimetric experiment, a previously weighed mild steel coupon was completely immersed in 250 ml of the test solution (in an open beaker). The beaker was inserted into a water bath maintained at 303 K. After every 24 hours, each coupon was withdrawn from the test solution, washed in a solution containing 50% NaOH and 100 g/L of zinc dust. The washed steel coupon was rinsed in acetone and dried in air before re-weighing. The difference in weight for a period of 168 h was taken as total weight loss. The inhibition efficiency (%I) of the inhibitor was calculated using the formula:

$$\%I = (1 - W_1/W_2) \times 100, \quad (13)$$

where W_1 and W_2 are the weight losses (in grams) for mild steel in the presence and absence of inhibitor in H_2SO_4 solution, respectively.

Conclusions

From the results and findings of the study, the following conclusions are made:

1. Ethanol extract of *Gnetum Africana* is a good inhibitor for the corrosion of mild steel in acidic medium.
2. The inhibition efficiency of the extract is due to the presence of saponin, terpenes, tannins, flavonoid, phlobatanins, anthraquinones, cardiac glycosides, and alkaloids in the extract.
3. The inhibitor is an adsorption inhibitor and the mechanism of physical adsorption is applicable to the adsorption of the inhibitor.
4. The adsorption characteristics of the inhibitor are consistent with the assumptions of Langmuir and Temkin adsorption isotherms and cannot be enhanced by synergistic combination of potassium halides (except KCl).

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