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Electrochemical Study of Corrosion Inhibition of Mild Steel in Acidic Solution Using *Gnetum africana* Leaves Extracts

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Authors' contributions

This work was carried out in collaboration between all authors. Author IOO designed the study. Authors LAN performed the experimental, statistical analysis and literature searches all with the supervision of author IOO. All authors read and approved the final manuscript.

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ABSTRACT

Weight loss and electrochemical (open circuit potential (OCP), linear sweep voltammetry (LSV) and Potentiodynamic polarization (PDP)) techniques were used to assess the effectiveness of *Gnetum africana* leaves extracts as corrosion inhibitor for mild steel in 1.0 M hydrochloric acid (HCl) solution at 30-45°C. It was found that *Gnetum africana* leaves extracts retarded the dissolution of mild steel in 1.0 M HCl solution. The inhibition efficiency increased with increase in extract concentration and did reasonably well at increased temperature, which is suggestive of physical and chemical adsorption mechanism. Open circuit potential shows reduction of resistance polarization R_p with the addition of the *Gnetum africana* extract. Potentiodynamic polarization result suggests that *Gnetum africana* extracts functioned as mixed-type inhibitor. The adsorption of *Gnetum africana* extracts onto the mild steel surface followed Langmuir and Temkin adsorption isotherm models. The mechanism of physisorption of the extracts onto the mild steel surface is proposed from the trend of inhibition efficiency with temperature which is corroborated by the values of activation parameters obtained from the experimental data.

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1. INTRODUCTION

Acid solutions are generally used for removal of undesirable scale and rust on the metals, cleaning of boilers and heat exchangers, oil-well acidizing in oil recovery, and so on [1–4]. HCl solution is one of the most widely used agents for these goals. However, iron and its alloys could be corroded during these applications which result in a waste of resources. Corrosion prevention systems favour the use of chemicals with low or no environmental impacts. The reduction in the corrosion rate of metals has numerous advantages such as saving of resources, economic benefits during the industrial applications, increasing the lifetime of equipment and decreasing the dissolution of toxic metals from the components into the environment. Therefore, the prevention of metals against corrosion is vital and must be dealt with. Commercial inhibitors are generally inorganic and some kind of organic compounds. However, the usage of some of them has been restricted due to the toxicity of their insufficient inhibitory efficiencies at low dosages [5-7]. In recent years, considerable amount of efforts have been devoted to find novel, healthy and efficient corrosion inhibitors.

The use of organic inhibitors is one of the most practical methods for protection of metals against corrosion, and is becoming increasingly popular according to recent studies. The existing data show that organic inhibitors act by adsorption and a film formation on the surface of metals. The adsorption of organic inhibitors on the metal surface can change the corrosion resistance properties of metals. Earlier studies have shown that organic compounds bearing heteroatom's with high electron density such as phosphorus, sulphur, nitrogen, oxygen or those containing multiple bonds, which are considered as adsorption centres, are effective inhibitors for the corrosion of metals [8-15]. The organic inhibitors are generally adsorbed on the metal surface through physical adsorption or chemical adsorption, which reduces the reaction area susceptible to corrosive attack [16,17]. In this work, *Gnetum africana* leaves extract was used to inhibit corrosion of mild steel in 1.0 M HCl. *Gnetum africana* leaves have been found to contain phytochemical components which are rich in adsorption heteroatoms including alkaloids, tannin, saponin, flavonoids and phenol [18-21]. The present work reports on the inhibitive effect of *Gnetum africana* leaves extracts for mild steel in hydrochloric acid solution using chemical and electrochemical techniques in furtherance of our continuous quest to explore green corrosion inhibitors.

2. EXPERIMENTAL DETAILS

2.1 Materials Preparation

A flat sheet of mild steel 0.05 cm in thickness with the following composition: C = 0.05%; Mn = 1.13%; Si = 0.05%; P = 0.91%; S = 0.85%; Cu = 0.09%; Pb = 0.15%; Va 0.13%; Mo = 0.08%; and the balance Fe was used in the study. The mild steel was mechanically press-cut into coupons of 2 cm × 2 cm dimensions for weight loss measurements and 1 cm × 1 cm for electrochemical measurements. The coupons were ground with different grades (# 400, 600, 800 and 1000) silicon carbide paper, degreased in absolute ethanol, dried in warm air and stored in moisture-free desiccators prior to use. The aggressive medium was 1 M HCl prepared from 98% analytical grade supplied by Sigma-Aldrich. Deionised water was used for the preparation of all reagents.

2.2 Preparation of the Leaf Extracts of *Gnetum africana*

The procedure for the preparation of the leaf extracts is similar to that reported recently by [22]. *Gnetum africana* leaves were collected from Abiriba, Abia State, Nigeria. They were dried in an N53C-Genlab Laboratory oven at 50ºC and ground to powder form. 10 g of the powder was digested in 1 litre of 1 M HCl solution. The resultant solution was kept for 24 h, filtered and stored. From the stock solution, test solutions of the leaf extracts were prepared at concentration range of 0.1 – 0.5 g/l using excess acid as solvent at room temperature and 45℃ using water baths.

2.3 Gravimetric Experiment

The cleaned and dried specimens were weighed before immersion into the respective test solutions of 1 M HCl using JA 1003A electronic weighing balance with accuracy of ±0.005. Tests were conducted with different concentrations of inhibitor. At the end of the tests, the specimens were carefully washed in absolute ethanol having used nitric acid to quench further corrosion from taking place and then reweighed. Triplicate experiments were performed in each case and the mean values reported. From the weight loss values, corrosion rates were computed using the expression:

$$CPR = \frac{K\Delta W}{DAt} \quad (1)$$

Where CPR is the corrosion rate in mm/yr, ΔW is the weight loss (mg)

D = metal density (g/cm^3)
A = area of the coupon (cm^2)
t = the time of exposure (h)
K = constant (87.6)

The inhibition efficiency (I %) was computed using Eq. (2)

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

Where CPR_{blank} and CPR_{inh} are the corrosion rates in the absence and presence of the inhibitor respectively in 1 M HCl at the same temperature.

2.4 Electrochemical Measurements

The electrochemical experiments were performed using a VERSASTAT 400 complete dc voltammetry and corrosion system, with V3 Studio software. A conventional three-electrode Pyrex glass cell was used for the experiments. Test coupons with 1 cm^2 exposed areas were used as working electrode and a graphite rod as counter electrode. The reference electrode was a saturated calomel electrode (SCE), which was connected via a Luggin's capillary. The test electrolyte was 1 M solution of HCl. All experiments were undertaken in stagnant aerated solutions at 30±1℃. The working electrode was immersed in a test solution for one hour until a stable open circuit potential was attained. The linear sweep voltammetry (LSV) tests were made for corrosion potential (E_{corr}) over a frequency range of 100 kHz to 100 MHz, with a signal amplitude perturbation of 2 mV. The potentiodynamic polarization study was from cathodic potential of -250 mV to anodic potential of +250 mV with respect to the corrosion potential at a sweep rate of 1 mV/s. The linear Tafel segments of the anodic and cathodic curves were extrapolated to corrosion potential to obtain the corrosion current densities (i_{corr}). Each experiment was carried out three times to estimate reproducibility and average values of the electrochemical parameters are reported.

2.5 EDS and SEM Surface Morphology

The analysis of the morphology of the mild steel surface was carried out using scanning electron microscopy (SEM), operated in the contact mode under ambient conditions using JSM-6010LA analytical scanning electron microscope (JEOL Technologies, USA). Images of the specimens were recorded after 12 h exposure time in 1 M HCl without and with various concentrations of the extract inhibitor. The EDS of the extract inhibitor was performed so as to ascertain the compound composition. The result of the EDS micrograph confirmed the presence of the heteroatoms of oxygen and phosphate which authenticates the leaves extract to be a good and efficient inhibitor.

3. RESULTS AND DISCUSSION

3.1 Fourier Transform Infrared (FTIR) of *Gnetum africana*

Fourier transform infrared (FTIR) is a powerful technique that is always used to determine the type of bonding for organic inhibitors adsorbed on the metal surface [23-25]. Fig. 1 shows the FTIR spectrum of the *Gnetum africana* powder. The peak at 3520 cm-1 is attributed to N-H or O-H stretching vibration and that at 2880 cm-1 is related to C-H stretching vibration. The strong band at 1643 cm-1 is assigned to C=C and C=O stretching vibration. Owing to the conjugation effect of flavonoids of *Gnetum africana*, the C=O peak shifts from about 1700 cm-1 to lower wave number (approximately 1644 cm-1), C=C and C=O stretching vibration bands are superposition [26]. The adsorption bands at 1452 and 1122 cm^{-1} could be assigned to the framework vibration of aromatic ring. These results indicate that *Gnetum africana* contains O and N atoms in functional groups (O-H, N-H, C=C, C=O) and aromatic ring, which meets the general structural consideration of the corrosion inhibitor.

3.2 Weight Loss Measurements

We had previously reported the gravimetric technique results of *Gnetum africana* leaves extracts of mild steel corrosion inhibition after 9 h exposure in acidic solution. The present study is considering the 12 h exposure within the environment. The assessment of the effectiveness of a metal corrosion inhibitor can be done using chemical and electrochemical methods. For the chemical techniques, weight loss measurements have been extensively employed for long-term immersion test. Corroborative results between weight loss and other corrosion monitoring techniques have been reported by some researchers [27-29]. The variation in weight loss against time for mild steel corrosion in 1 M HCl without and with different concentrations of *Gnetum africana* leaves extracts at different temperatures (30-45℃). Fig. 3 shows the plot of corrosion rate versus extract concentration for *Gnetum africana*. Inspection of the figures revealed that the corrosion rate of mild steel in 1 M HCl solution was reduced upon the introduction of *Gnetum africana* leaves extracts into the corrosive medium. The extent of reduction in corrosion rate is seen to increase with increase in the concentration of the *Gnetum africana* extracts. The plot of the inhibition efficiency against extract concentration from weight loss experiments is shown in Fig. 4. Close examination of Fig. 4 showed that the leaves extracts of *Gnetum africana* afforded protection for mild steel in 1 M HCl solution which could be attributed to the adsorption of the components of the extracts on the mild steel surface. The figure further revealed that the inhibition efficiency increased with increase in the concentration of *Gnetum africana* leaves extracts but decreased as the temperature was raised after 0.4 g/l cocentration. The decrease in the inhibition efficiency of *Gnetum africana* leaves extracts as the temperature increases may be attributed to desorption of the adsorbed components of the extracts on the mild steel surface. It is therefore pertinent to conclude that the high inhibition efficiency of the extracts observed in the present study could be attributed to high phytochemical constituents of tannins, flavonoids and saponins. Also, the presence of oxygen heteroatoms as revealed by the EDS micrograph in Fig. 2 serve as the adsorption sites of the extracts onto the mild steel surface and thus enhances protection of the mild steel from corrosive agents present in the aqueous medium.

3.3 Adsorption Considerations

Inhibition by organic compounds is, mainly, due to their ability to adsorb onto a metal surface to form a protective film. The establishment of isotherms that describe the adsorption behaviour of corrosion inhibitor is important as they provide relevant clues about the nature of metal-inhibitor interaction. It has been reported that the adsorption of the inhibitor molecules depends on a variety of factors such as the presence of functional groups (either electron donating or withdrawing), steric factors, charge distribution at the donor atom, the π orbital character of donating electrons, the nature of substrate metal and the type of interaction between organic molecules and the metallic surface as well [30,31]. Langmuir adsorption isotherm was used to deduce whether there is formation of layer of insoluble complex of the metal on the surface which acts as a barrier between the metal surface and the corrosive medium - usually termed physisorption and Temkin isotherm was used to determine if the extract adsorption on the metal surface is via chemisorption which involves displacement of water molecules from the metal surface and the sharing of electrons between oxygen atom and iron. Langmuir isotherm plot describes the relationship between the surface coverage and inhibition concentration of a material as:

$$\frac{C}{\theta} = \frac{1}{k} + C \tag{3}$$

Temkin isotherm plot is expressed as:

$$\theta = \frac{1}{f}\ln\left(k_{ads}C\right) \tag{4}$$

Where C is the concentration of inhibitor, θ is the surface coverage, f determines the adsorbent-adsorbates interaction and k is the equilibrium constant employed in calculating the Gibb's free energy (Equation 5) [32-34]. However, Temkin adsorption isotherm assumes a uniform distribution of adsorption energy, which decreases with the increase of the value of surface coverage, θ. It focuses on the chemisorption aspect of corrosion inhibition of organic plants on metals. Fig. 5 shows Langmuir adsorption isotherm plot with a straight line curve with the slopes (1.0108 and 1.0290) and correlation coefficients (0.9999 and 0.9956) for 30℃ and 45℃ respectively. Fig. 6 also shows a straight line curve for the Temkin adsorption isotherm plot with the slopes vanishing (0.0098 and 0.0743) and correlation coefficients (0.6049 and 0.7902).

$$\Delta G^{\circ}_{ads} = -RT\ln(55.5k) \quad (5)$$

Where R is the gas constant (8.314 kJ/mol) and T is the temperature in Kelvin. 55.5 is the concentration of water in solution in mol/l. The negative value of ΔG^{o}_{ads} indicates spontaneity in the inhibitor adsorption on mild steel surface.

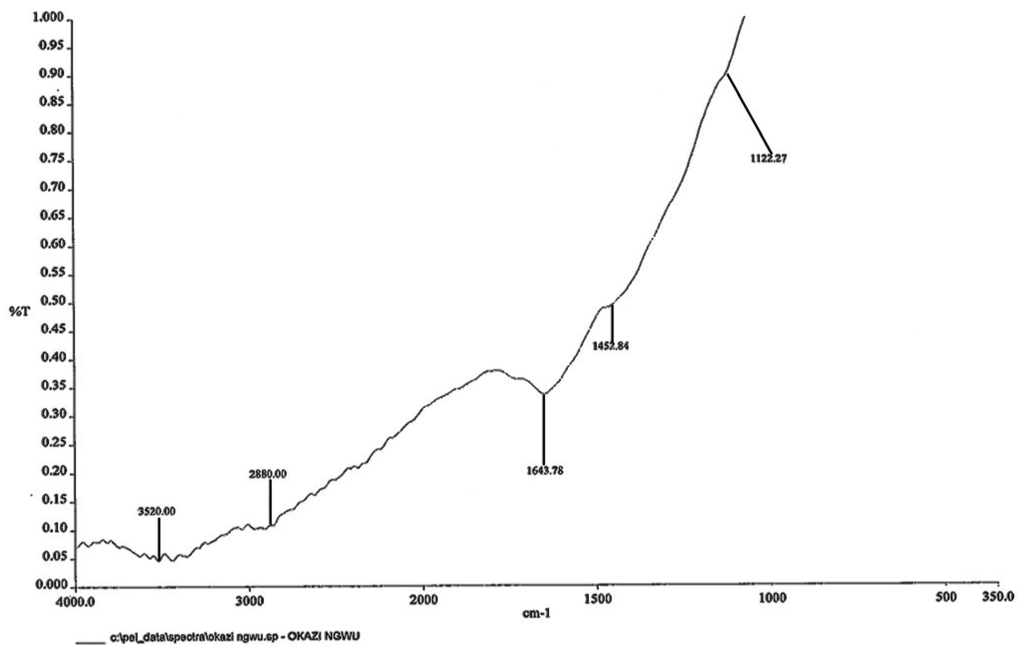


Fig. 1. FTIR of Ground *Gnetum africana* Leaves

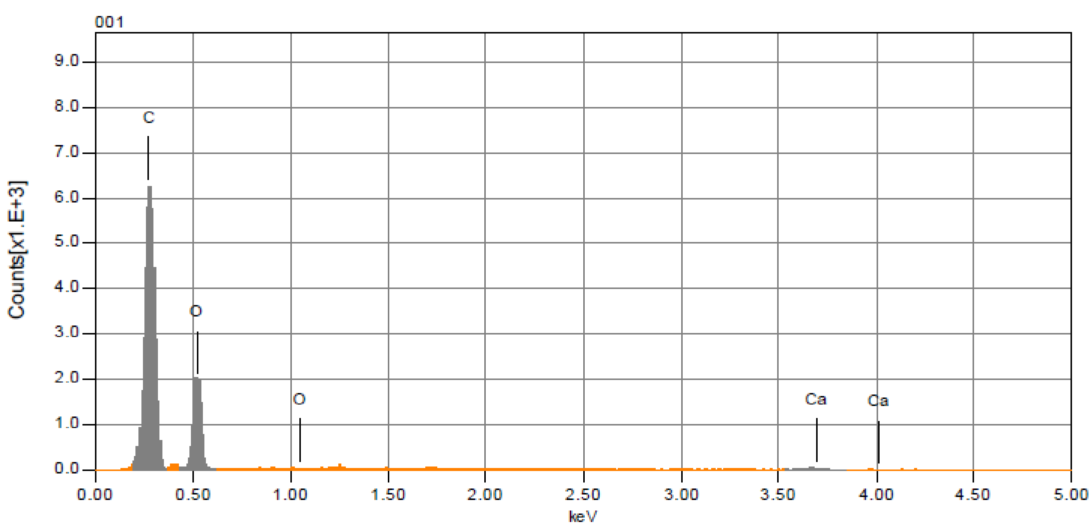
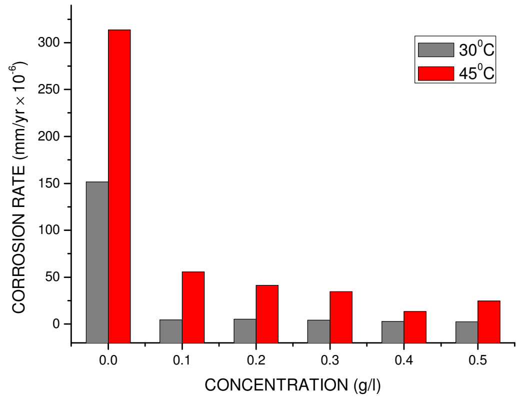


Fig. 2. EDS Micrograph of Ground *Gnetum africana* Leaves

A



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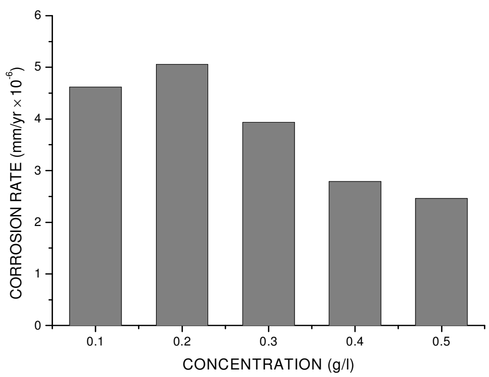

Fig. 3. Corrosion rate values of mild steel in 1.0 M HCl in the (A) absence and presence of different concentrations of *Gnetum africana* leaves extract after 12 h exposure time at 30℃ and 45℃ (B) Presence of Different Concentrations of *Gnetum africana* Leaves Extract After 12 h Exposure Time at 30℃

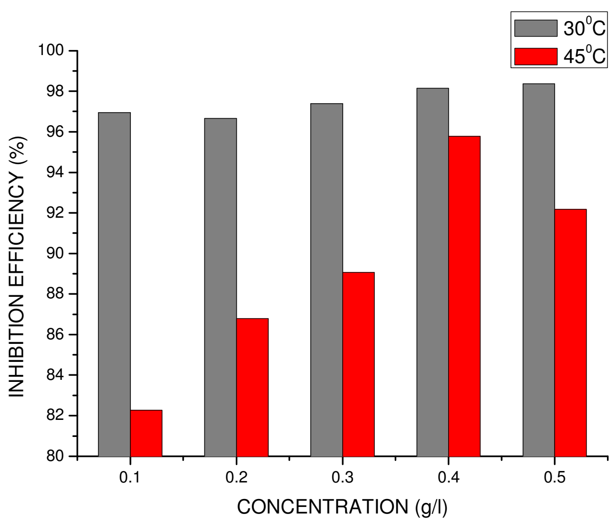

Fig. 4. Inhibition efficiency *Gnetum africana* Leaves extract in the corrosion of mild steel in 1.0 M HCl after 12 h exposure time

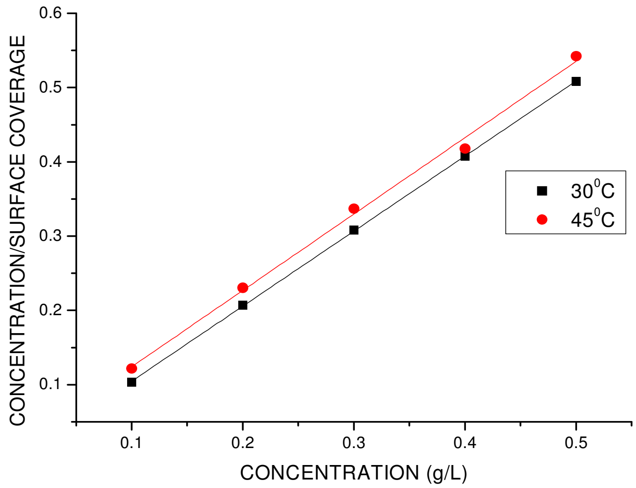

Fig. 5. Langmuir isotherm plots of corrosion of mild steel in 1.0 M HCl in the presence of extract *Gnetum africana* leaves extract after 12 h exposure time at 30℃ and 45℃

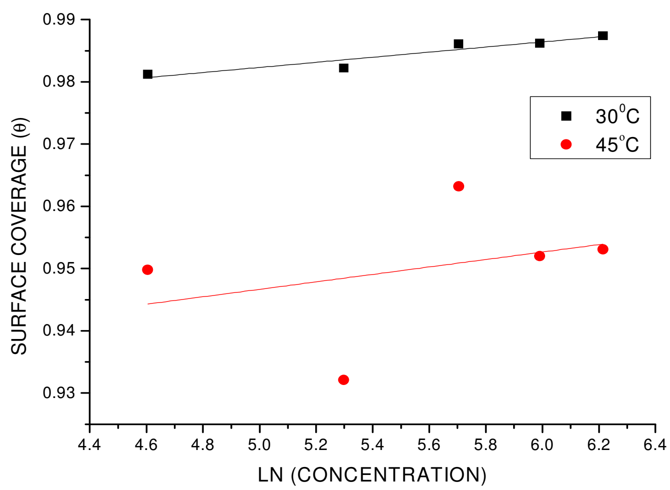

Fig. 6. Temkin isotherm plots of corrosion of mild steel in 1.0 M HCl in the presence of extract *Gnetum africana* leaves extract after 12 h exposure time at 30℃ and 45℃

3.4 Effect of Temperature

The influence of temperature on the corrosion behaviour of mild steel in the absence and presence of *Gnetum africana* leaves extracts in 1 M HCl solution was investigated at temperature range of 30-45℃. Analysis of the temperature dependence of inhibition efficiency as well as comparison of corrosion apparent activation energies in absence and presence of inhibitor gives some insight into the possible mechanism of inhibitor adsorption.

A decrease in inhibition efficiency with the rise in temperature with analogous increase in corrosion activation energy in the presence of inhibitor compared to its absence is frequently interpreted as being suggestive of formation of an adsorption film of physical (electrostatic) nature. The reverse effect, corresponding to an increase in inhibition efficiency with rise in temperature and lower activation energy in the presence of inhibitor, suggests a chemisorption mechanism [35-37].

3.5 Electrochemical Measurements

3.5.1 Open circuit potential

The variation of the open circuit potential with time for mild steel corrosion in 1 M HCl solution without and with selected concentrations of *Gnetum africana* leaves of extracts is shown in Fig. 7. It is clear from the curves that the corrosion potential of the blank solution increased steadily and reached a maximum value -0.41871 V. The potential decreased reasonably upon application of the leaves extract of *Gnetum africana* compared to the blank. However, the 0.1 g/l concentration did not do well, compared to other concentrations of 0.3 g/l and 0.5 g/l.

3.5.2 Potentiodynamic polarization measurements

Potentiodynamic polarization measurements were undertaken mainly to gain an insight into the possible influence of addition of different concentrations of *Gnetum africana* leaves extracts on the anodic dissolution of mild steel and the corresponding cathodic reduction of hydrogen ion of the corrosion process. Typical anodic and cathodic polarization curves obtained for the corrosion of mild steel in 1 M HCl solution in the absence and presence of selected concentrations of *Gnetum africana* leaves extracts are presented in Fig. 8. The curves showed that the introduction of the additives to the blank solution has modest influence on both anodic and cathodic half reactions, although cathodic influence appeared much pronounced. The potentiodynamic parameters obtained by extrapolation of the linear Tafel segments of the anodic and cathodic curves are presented in Table 2. The results in the table indicate that the introduction of the various concentrations of *Gnetum africana* leaves extracts did not remarkably shift Ecorr /SCE. For instance, the difference between the Ecorr /SCE of the blank solution and that of the highest concentration of *Gnetum africana* leaves extract is 8.0 mV. It could be inferred that the extracts acted as a mixed-type inhibitor and the inhibition is due to simple geometric blocking mechanism [38]. Also, the corrosion current densities of the additives decreased significantly compared to the blank. It is clear also from the result Table 2 that the β_a and β_c values for the various concentrations of the extracts reduced remarkably compared to the blank indicating that the additive simultaneously modified both the anodic and cathodic reactions thus supporting the assertion that the extract is a mixed-type inhibitor. The corrosion inhibition efficiency of *Gnetum africana* leaves extracts was calculated using the relation:

$$\eta\% = \left(1 - \frac{i_{corr}}{i^{\circ}_{corr}}\right) \times 100 \tag{6}$$

where i^{o}_{corr} and i_{corr} are the corrosion current densities in the absence and presence of the extracts, respectively, and the values obtained are given in Table 1. From the result in the table, it is clear that the *Gnetum africana* leaves extracts significantly inhibit the corrosion of mild steel in 1 M HCl solution with the optimum value of 95.5 % obtained for the highest concentration of the extract used.

The SEM image (Fig. 9) clearly shows that the corrosion reaction does not take place homogenously over the surface of mild steel in 1.0 M HCl solution. However, the surface is remarkably protected by *Gnetum africana* leaves extracts, in comparison to the inhibitor-free solution.

Table 1. Activation energy and heat of adsorption parameters for the corrosion of mild steel in 1 M HCl at 30°C and 45°C in the absence and presence of *Gnetum aficana* leaves extract

Inhibitor concentration (g/l)	Corrosion rate (mm/yr)	Inhibition efficiency (%)	Corrosion rate (mm/yr)	Inhibition efficiency (%)	Activation energy (kJ/mol)	Heat of adsorption (kJ/mol)
	30°C		45°C			
0	151.39	0.00	313.76		-38.93	
0.1	4.62	96.95	55.64	82.27	-132.92	-102.79
0.2	5.06	96.66	41.44	86.79	-112.33	-79.20
0.3	3.94	97.40	34.31	89.06	-115.60	-81.53
0.4	2.79	98.15	13.23	95.78	-83.14	-45.35
0.5	2.46	98.37	24.58	92.17	-122.95	-87.31

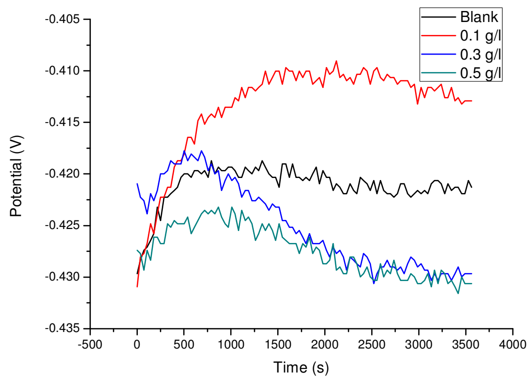

Fig. 7. OCP curve of the corroded mild steel in 1.0 M HCl in the absence and different concentration of *Gnetum africana* leaves extract

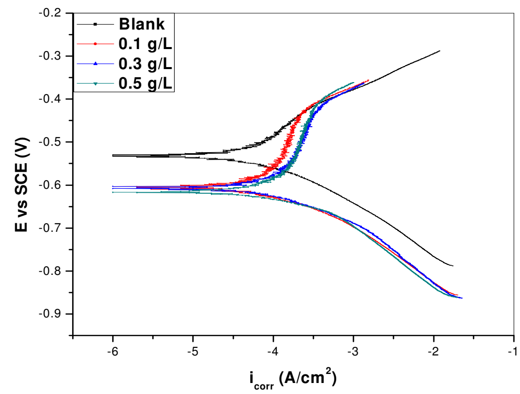

Fig. 8. Potentiodynamic polarization curves of mild steel in 1.0 M HCl without and with different concentrations of *Gnetum africana* leaves extract

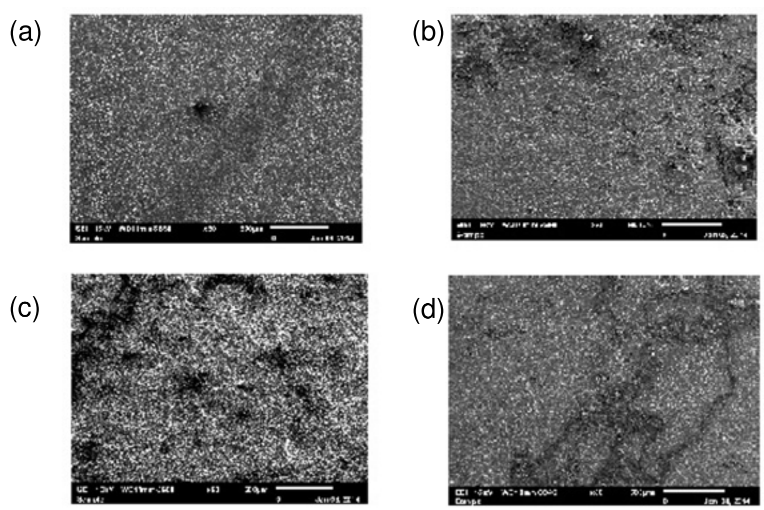

Fig. 9. SEM Characteristics of the corroded mild steel in 1.0 M HCl in (a) the absence of inhibitor at 30℃, (b) the presence of *Gnetum africana* leaves extract at 30℃, (c) the absence of inhibitor at 45℃, (d) the presence of *Gnetum africana* leaves extract at 45℃

Table 2. Potentiodynamic polarization kinetic parameters and inhibition efficiency for mild steel in 1.0 M HCl without and with different concentrations of *Gnetum africana* leaves extract

System	E_{corr} (mV)	I_{corr} ($\mu A/cm^2$)	β_a	β_c	IE (%)
Blank	-531.5	841	5838.9	356.2	
0.1 g/l	-605.4	121.1	522.2	111.6	85.6
0.3 g.l	-605.8	81.2	252.9	102.9	90.3
0.5 g/l	-615.8	37.8	193.8	88.2	95.5

4. CONCLUSION

The following conclusions can be drawn from the work;

1) *Gnetum africana* leaves extracts act as an inhibitor for mild steel corrosion in 1 M HCl solution. Inhibition efficiency increased with increase in the concentration of the inhibitor.
2) Potentiodynamic polarization results indicate that *Gnetum africana* leaves extracts behave as mixed-type inhibitor.
3) Corrosion inhibition is afforded by both physical and chemical adsorption components of *Gnetum africana* leaves extracts on the mild steel surface. Therefore, Langmuir and Temkin adsorption isotherms are obeyed.
4) Results from all the techniques employed are in reasonably good agreements and points to the fact that *Gnetum africana* leaves extracts is a good and efficient inhibitor within the range of concentrations investigated.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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