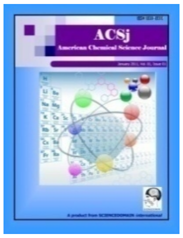

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Corrosion Inhibition Effect of Phenolic Extract of *Gnetum africana* on Copper in 1.0 mol/dm^3 Ammonium Hydroxide

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Authors' contributions

This work was carried out in collaboration between both authors. Both authors read and approved the final manuscript.

Original Research Article

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ABSTRACT

Corrosion Inhibition of phenolic extract of *Gnetum africana* on copper in 1.0 mol/dm^3 ammonium hydroxide has been studied using gravimetric (weight-loss) and potentiodynamic polarization techniques. Inhibition efficiency increases with increase in inhibitor concentration, while corrosion rate decreases with increment of *Gnetum africana* extract concentration. The inhibition was chemisorptions in nature and the absorption the inhibitor on copper surface obeys Langmuir adsorption isotherm. Potentiodynamic polarization suggests that *Gnetum africana* extract acts as mixed type inhibitor, attending a maximum Inhibition efficiency of 84.21 % as against weight-loss method with 87.83 %.

Keywords: Copper; corrosion inhibitor; weight loss; potentiodynamic polarization; inhibition efficiency; Gnetum africana.

1. INTRODUCTION

Copper and its alloys are metals that have a wide range of applications due to their good properties. They are widely used in many environments and applications because of their excellent corrosion resistance, which is coupled with combinations of other desirable properties, such as in electronic industries and communications as a conductor in electrical

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power lines, pipelines for domestic and industrial water utilities including sea water, heat conductors, heat exchangers, etc. [1-2]. Copper and its alloys are resistant toward the influence of atmosphere, many saline solutions, alkaline solutions, organic chemicals, and many other chemicals. However, it is known that in aggressive media, (oxidizing acids, oxidizing heavy-metal salts, sulfur, ammonia (NH_3), and some sulfur and ammonia compounds) copper is susceptible to more rapid attack [3]. The possibility of the copper corrosion prevention has attracted the interest of many researchers and to this effect numerous possible inhibitors have been investigated. Amongst them are organic corrosion inhibitors. It is noticed that presence of heteroatom such as nitrogen, sulphur, phosphorous in the organic compound molecule improves its action as copper corrosion inhibitor [4-6]. Organic inhibitor compounds can be classified as anodic, cathodic, or both. In most cases, it appears that the inhibitors affect both the anodic and cathodic processes, although in many cases the effect is unequal [6-7]. Most organic inhibitors contain at least one polar group with an atom of nitrogen, oxygen or sulphur through which they can be absorbed into the metal surface and suppress metal dissolution and reduction reactions usually by chemisorptions [8].

However, the efficiency of these inhibitors depends on the presence of vacant d orbital in copper atom that forms coordinative bonds with atoms able to donate electrons, interaction with rings containing conjugated bonds and also the presence of π electrons [9]. The mechanism of inhibition follows a transition of metal/solution interface from a state of active dissolution to the passive state which is attributed to the adsorption of the inhibitor molecules on the metal surface, forming a protective film (metal complex) and hence, the reactive metal surface is shield from the aggressive environment [10]. The inhibition mechanism is a separation process involving (i) the inhibitor is adsorbed on the surface of the metal forming a compact protective thin layer and (ii) the inhibitor forms a precipitate on the surface of the metal, acting on the aggressive media to form protective precipitates or remove the aggressive agents. Adsorption, on the other hand can be described by two main types of interaction, which are physisorption and chemisorptions, where:

1.1 Physisorption

Involves electrostatic forces between ionic charges or dipoles on the adsorbed species and the electric charge at the metal/solution interface. The heat of adsorption is low, thus, this adsorption is only stable at relatively low temperature.

1.2 Chemisorptions

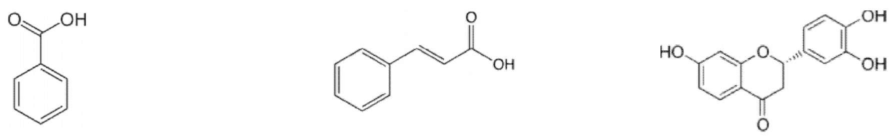
Involves charge transfer or sharing from the inhibitor molecules to the metal surface to form a coordinate type bond. This type of adsorption is known to have much stronger adsorption energy compared to the other mode of adsorption. Thus, such bond is more stable at higher temperature [10].

$$Cu_{(s)} + Inh \rightarrow Cu(Inh)_{ads} \quad (1)$$

$$Cu(Inh)_{ads} \rightarrow Cu^{2+} + 2e^{-} + Inh \quad (2)$$

Where, Cu (Inh) and Inh are the Copper complex and the inhibitor [11].

Over the years, different organic compound and its derivatives [12-19], have been reported as good inhibitors for copper or copper-based alloys corrosion in different corrosive media. Due to the toxicity of most of these widely used corrosion inhibitors and the ever tightening environmental regulations surrounding their use and disposal, there is great interest in replacing harmful inhibitors with environmental friendly, abundant availability, cheap source organic corrosion inhibitor as alternatives. Over the past two decades, extensive research and development have led to increased interest in the assessment of natural product of plant origin as efficient organic corrosion inhibitor for metal and alloy in different environment [18, 20-22]. *Gnetum africanum* locally known in Nigeria, as ukasi by the Igbo and afang by (Efik/Ibibio tribes), Southern Nigeria are lianas or climbing plant native to most humid tropical forests of Africa [23], with its leaf measuring about 10-13 cm long, 3.5-5 cm broad. Research have shown that it is a significant source of flavonoids like (flavanones, flavanols and flavones) and Phenolic compounds such as benzoic acid, and cinnamic acid [24], most of these phytochemicals contains heteroatoms with lone pair of electrons which can serve as centre of adsorption. Some of the phytochemicals that are contained in *Gnetum africana* leaf are shown below in scheme 1:



Benzoic acid Cinnamic acid Butin (flavanones)

Scheme 1. Some phytochemicals contained in *Gnetum africana* leaf

These factors favour the interaction of *Gnetum africana* with copper metal as a corrosion inhibitor. The aim of this paper is to investigate the effect of phenolic extract of *Gnetum africana* as an effective eco-friendly, cheap source organic corrosion inhibitor for the corrosion inhibition of copper in 1.0 mol/dm^3 NH_4OH using weight-loss and electrochemical methods at room temperature (30ºC).

2. EXPERIMENTALS

2.1 Extraction Phenolic Compounds in Plant Leaf (*Gnetum africana*)

Leaves of *Gnetum africana* (ukazi or afang) were collected from small farmers at the eke Ukwu markets at Owerri municipal council of Imo state within South Eastern Nigeria. The samples were cleaned after removing manually the inedible parts with sharp knife, dried, pulverized and stored in air tight containers for further processes. Extraction of the *Gnetum africana* leaves was done by using cold extraction method [25]. 20 g of the pulverized leaf sample was soaked in 800 cm^3 of acetone solvent and shaken vigorously for 5 - 10 min and left for 72 h (3 days) after which the aqueous solution was filtered using an already weighed clean dried filter cloth to obtain a pure acetone solution containing phenolic compounds extract and an extract residue. The filter cloth containing the residue was sun dried and the weight recorded.

2.2 Confirmation of Phenolic Compound in *Gnetum african* Leaf Extract

2.2.1 Ferric Chloride Test

1 cm^3 extracts were treated with 3-4 drops of ferric chloride solution. Formation of bluish black colouration indicates the presence of phenolic compound [26].

2.2.2 Alkaline reagent test

1 cm^3 extracts were treated with few drops of sodium hydroxide solution. Formation of intense yellow colouration, which becomes colourless on addition of dilute acid HCl, indicates the presence of flavonoids [27].

2.3 Preparation of Sample/ Test Solution

Copper specimen having composition (wt) of Cu 99.3%, Ag 0.68%, Bi 0.001%, Sb 0.002%, As 0.002%, Fe 0.005 %, Pb 0.005% and S 0.005%.Copper sheets with purity level of 99.30% were used in this study. Each sheet was mechanically press-cut into coupons of dimension 30 × 20 × 0.25 mm. A tiny hole of diameter 3.5 mm was drilled on each coupon close to its upper edge so as to suspend it in the corrosive medium. They were however degreased in absolute ethanol, dried in acetone, weighed and stored in moisture-free desiccators prior to use [27].An aqueous solution of 1.0 mol/dm^3 NH_4OH was calculated to be 7.265 mol/dm^3 based on density of 0.90 g/cm^3, weight percent of 56.6% and molecular weight of 35.05 g/mol. NH_4OH solution with the concentration above was obtained by accurately measuring 68.85 cm^3 of NH_4OH with measuring cylinder and pour in 1000 cm^3 standard flask and make up to mark with distilled water. The NH_4OH stock solution was used as a bank solution. *Gnetum africana* extract was added to the alkaline in concentrations ranging from 200- 800 mg/dm^3.

2.4 Preparations of Metal Sample for Potentiodynamic Polarization Studies

The copper sample used was same as the one used for weight loss experiment. The metal samples were machined into test electrode (working electrode) of dimension 20 × 10 mm by use of hand cutting machine and a hole of 2.0 mm was drilled at the upper part of the coupon to enable a coated copper wire of about 30 cm in length, whose both ends were made bare to be fixed to the electrode, and the other end to be connected to one of the three terminals of the potentiostat. The test electrode (working electrode) was then covered with a polytetrafluoroethylene (PTFE) ribbon and an epoxy resin such that an area of 1 cm^2 was left exposed at one side for electrochemical measurements. The exposed surface was wet polished with an emery paper (grit No.1200), degreased with acetone, rinsed in distilled water and dried in the air [28].

2.5 Gravimetric Studies (Weight Loss Measurement)

The gravimetric method (weight loss) is known as the most widely used method of monitoring inhibition efficiency [29]. The weight loss experiments were carried out using duplicates copper coupons under total immersion conditions in the test solutions, The cleaned copper coupon was weighed and suspended with the aid of nylon thread in a beaker containing 200 cm^3 solution of 1.0 mol/dm^3 NH_4OH without and with different concentrations of investigated *Gnetum africana* extract for exposure period 24 hours. At the end of tests, the coupons were rinsed with distilled water, dried and re-weighed. The efficiency percentage of inhibitors (IE %) was determine using the following equation.

$$IE\% = \left[1 - \left(\frac{\Delta W_{inh}}{\Delta W_{blank}}\right)\right]100 \quad (3)$$

Where, ΔW_{inh} and ΔW_{blank} are the average weight loss values in the presence of inhibitor and absence of inhibitor.

2.6 Electrochemical Technique (Potentiodynamic Polarization)

Electrochemical experiments were conducted at room temperature in a conventional three-electrode glass cell of capacity 300 cm^3 using an EG & G Princeton Applied Research Potentiostat/ Galvanostat Model 263 electrochemical workstation with power suit software. A saturated calomel electrode (SCE) was used as reference electrode and platinum foil as counter electrode, respectively. Before Polarization scan, the working electrode was subjected to open circuit potential (OCP) scan (electrode potential versus time) for 1800 s. The polarization curves were then conducted from cathodic potential of -0.25 V to anodic potential of +0.25 V with respect to the open circuit potential at a sweep rate of 1 MV/s. The linear Tafel segments of the anodic and cathodic curves were extrapolated to corrosion potential (Ecorr) to obtain the corrosion current densities (Icorr) from the power suit software [28].

The inhibition efficiency (IE %) were calculated by following equation below:

$$IE\% = \left[1 - \left(\frac{I_{corr}\,(\text{Inh})}{I_{corr}\,(\text{blank})}\right)\right]100 \quad (4)$$

Where, $I_{corr\ (\text{Inh})}$ and $I_{corr\,(\text{Blank})}$ are corrosion current density in the presence of inhibitor and Corrosion current density in the absence of inhibitor respectively.

3. RESULTS AND DISCUSSION

3.1 Effect of Inhibitor Concentration

Weight loss measurements were carried out for the different concentrations of inhibitor. The trend of the inhibition efficiency and corrosion rate with respect to the *Gnetum africana* extract concentration (200, 400,600, 800mg/dm^3) was plotted in Fig. 2.1(A, B) to show the trend of increment of the inhibition efficiency and the decrease of corrosion rate. It is evident from Table 2.1 and Fig. 2.1(A, B) that increment of *Gnetum africana* extract concentration increases with the inhibition efficiency and decreases corrosion rate of the copper sample in 1.0 mol/dm^3 NH_4OH solution. This indicates that the inhibitors were adsorbed onto the metal surface and thereafter, inhibits the corrosion process and reduce corrosion rate [29].

Table 2.1. Corrosion Rate (R_{corr}) and inhibition efficiency (I.E %) of copper in 1.0 mol/dm^3 NH_4OH for 24 Hours in different concentrations of the inhibitor (mg/dm^3)

Inhibitor conc. (mg/dm^3)	R_{corr} (mg/cm^2-hr)	(I.E %)
Blank	7.814X10^{-5}	-----
200	1.885X10^{-5}	75.93
400	1.580X10^{-5}	79.76
600	1.053X10^{-5}	86.65
800	0.951X10^{-5}	87.83

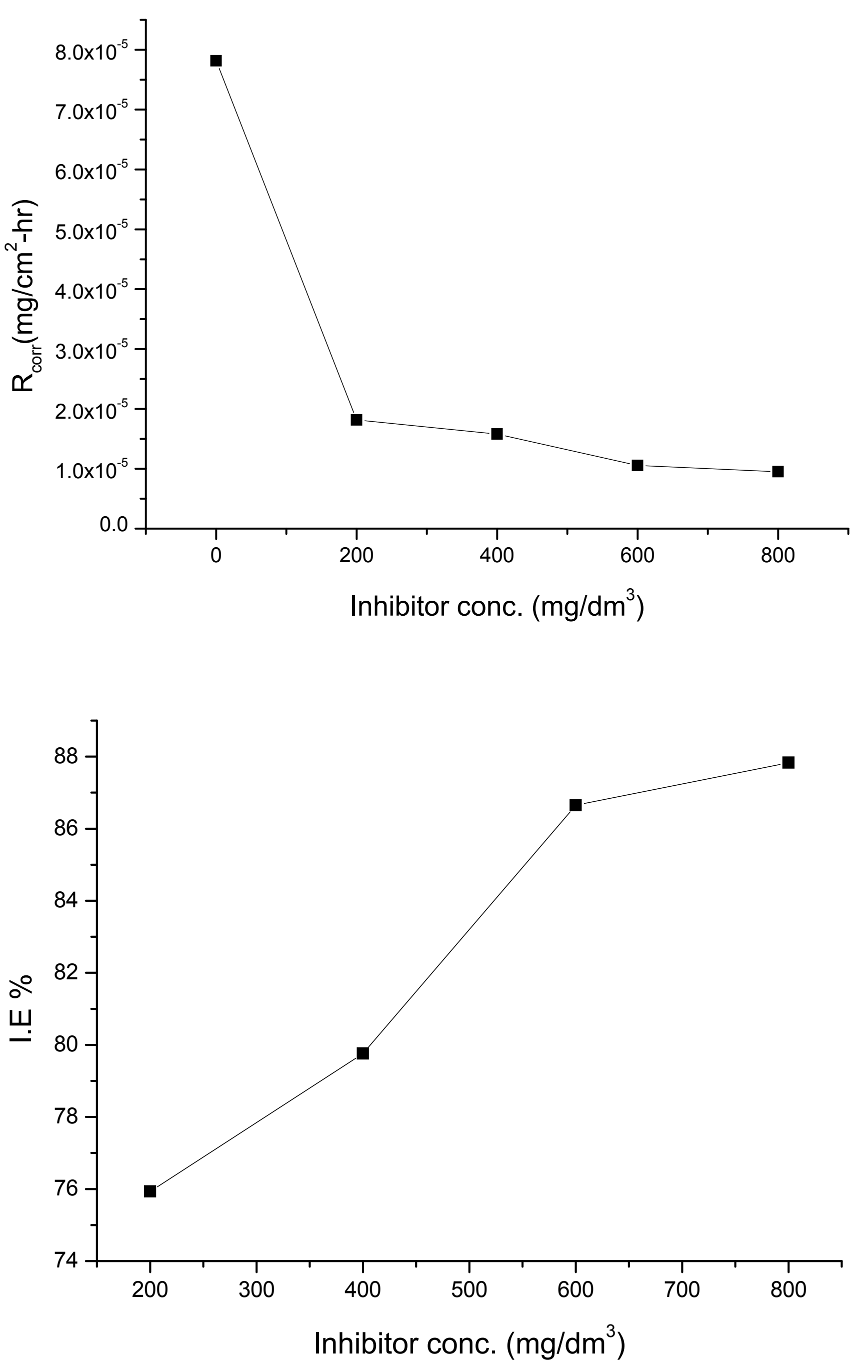


Fig. 2.1(A and B). Variation of Inhibitor Concentration in 1.0 mol/dm^3 NH_4OH on Cu sample with (A) Corrosion Rate and (B) Inhibition Efficiency, using weight loss data

3.2 Adsorption Isotherm

Adsorption isotherm study describes the adsorptive behaviour of organic inhibitors to know the adsorption mechanism. The most usually used adsorption isotherms are Langmuir, Temkin, freundlich, Frumkin and some other various isotherms [30]. The surface coverage (θ) values were calculated using weight loss data.

Langmuir adsorption isotherm were tested and found most appropriate isotherm to explain the experimental data. Langmuir adsorption isotherm is represented by following equations [31]:

$$\frac{C}{\theta} = \frac{1}{QO} C + \frac{1}{QOb} \quad (5)$$

Where Q_O and *b* are Langmuir constants determined from the slope and intercept of the linear plot and C is the concentration of inhibitor used in the corrosive medium.

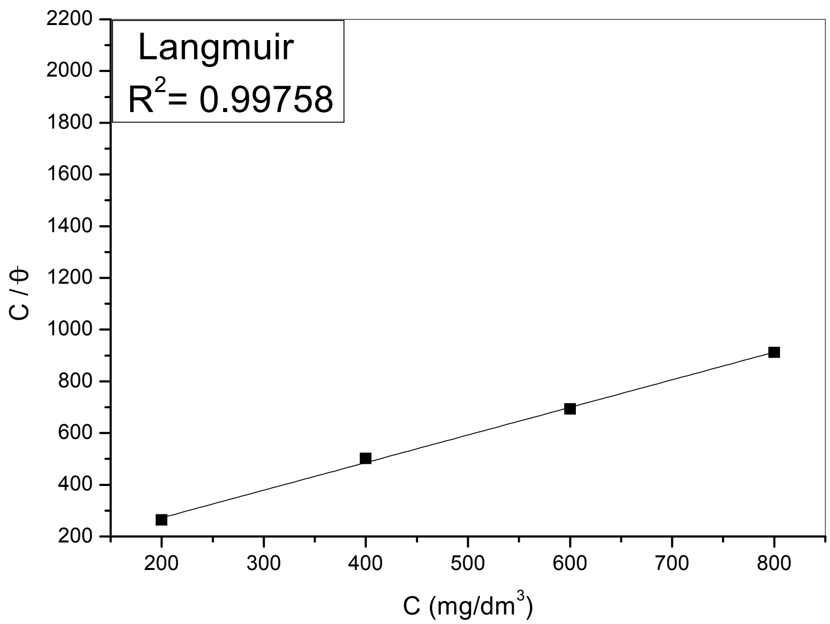


Fig. 2.2. Langmuir isotherm plot of copper in 2.0 mol/dm^3 NH_4OH

Table 2.2. Adsorption parameters data for copper in 1.0 mol/dm^3 in NH_4OH

Inhibitor concentration C (mg/dm^3)	In C	Log C	C/θ	Θ	In θ	Log θ
200	4.605	2.301	263.500	0.759	-0.276	-0.120
400	5.991	2.602	501.800	0.797	-0.227	-0.099
600	6.397	2.778	693.600	0.865	-0.145	-0.063
800	6.685	2.903	911.200	0.878	-0.130	-0.057

Table 2.3. Correlation coefficient R^2 of adsorption isotherm for copper in 1.0 mol/dm^3 NH_4OH

Isotherm	Langmuir	Freundlich	Temkin
Correlation Coefficient R^2	0.99758	0.91747	0.78649

Table 2.2 shows the adsorption parameters "s data for copper in 1.0 mol/dm^3 in HH_4OH while Fig 2.2 shows a straight line obtained by plotting the graph of C verses $\frac{C}{\theta}$ with the R^2 value almost unity (0.99758).This is in accordance to the works of Nnanna et al. [13,33] and Umeron et al. [32] which states that the correlation coefficient (R^2) which gave particular linear plot with highest correlation coefficient close to unity (1) best describes the adsorption type. Table 2.3 showed Langmuir adsorption highest correlation coefficient of 0.99758 suggesting that the Langmuir adsorption isotherm model which favours chemisorptions provides the best description of the adsorption behavior [34].

3.3 Potentiodynamic Polarization

The potentiodynamic polarization measurements were carried out to study the kinetics of the cathodic and anodic reactions. The results of the effect of *Gnetum africana* extract on the cathodic as well as anodic polarization curves of copper in 1.0 mol/dm^3 NH_4OH solution are shown in Table 2.4 and Fig. 2.3 respectively.

Table 2.4. Potentiodynamic polarization of copper immersed in 1.0 mol/dm^3 NH_4OH in the absence and presence of *Gnetum africanium*

Inhibitor conc. (mg/cm^3)	$-E_{corr}$ (mV)	I_{corr} (μA)	IE%
Blank	237.450	0.019	00.00
800	160.570	0.003	84.21

Electrochemical corrosion kinetic parameters namely corrosion potential (E_{corr}) and corrosion current density (I_{corr}) obtained from the extrapolation of the polarization curves are listed in Table 2.4. It was observed that the corrosion current density (I_{corr}) decreased by the increase in the adsorption of the inhibitor with increasing inhibitor concentration and also that the displacement in corrosion potential (E_{corr}) was less than 85 mV with respect to the corrosion potential of blank solution, This indicates that the addition of *Gnetum africana* extract (Inhibitor) primarily affects both cathodic and the anodic processes. This is in accordance with the works of Ferreira et al. [35] and Li et al. [36] that says, if the displacement in corrosion potential is more than 85 mV with respect to the corrosion potential of blank solution, the inhibitor can be consider as a cathodic or anodic type. In present study, displacement was 77 mV with respect to the corrosion potential of the uninhibited sample which indicates that the studied inhibitor is a mixed type of inhibitor. The inhibition efficiency was calculated using equation (4) and listed in Table 2.3 and was seen to increases with an increase in the inhibitor concentration. It is also evident from the Fig. 2.3 that both reactions were suppressed with the addition of the inhibitor. This indicates that the addition of *Gnetum africana* extract (Inhibitor) primarily reduced the anodic dissolution reactions as well as retarded the reactions on the cathodic sites.

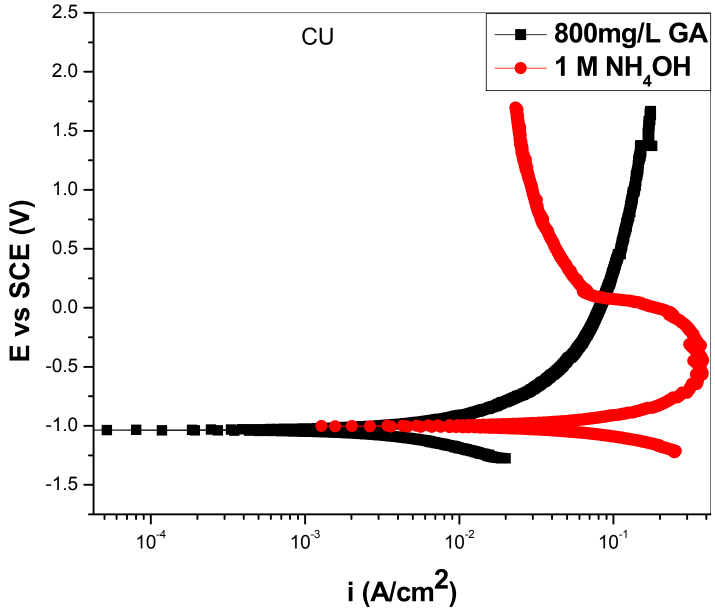


Fig. 2.3. Potentiodynamic polarization curves for Copper electrode in 1.0 mol/dm^3 NH_4OH in absence and presence of 800mg/dm^3 of *Gnetum africana* extract

4. CONCLUSION

Gnetum africana extract was found to inhibit the corrosion of copper in 1.0 mol/dm^3 NH_4OH solution and inhibition efficiency increases with increasing extract concentration. At the highest extract concentration of 800 mg/dm^3, the inhibition efficiency is as high as 87.83 %. The Langmuir adsorption isotherm provides a formal description of the adsorptive behavior of *Gnetum africana extract on* copper surface. The result of Potentiodynamic polarization curves showed that the *Gnetum africana* extract (Inhibitor) acted as a mixed-type inhibitor. The inhibition efficiency (IE %) reached maximum value of 84.21 % at 800 mg/dm^3 *Gnetum africana* extract (Inhibitor) in 1.0 mol/dm^3 NH_4OH solutions, showing that the results obtained from this method is in reasonable agreement with the gravimetric methods.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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