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G. I. Udom¹, G. A. Cookey^{1*} and A. A. Abia²

¹Department of Chemistry, Rivers State University, Port Harcourt, Rivers State, Nigeria. ²Department of Pure and Industrial Chemistry, University of Port Harcourt, Port Harcourt, Rivers State, Nigeria.

Authors' contributions

This work was carried out in collaboration among all authors. Author GIU performed the experiment and statistical analyses, wrote the protocol and the first draft of the manuscript. Author GAC supervised the experiments and statistical analyses of the study and edited the manuscript. Author AAA designed the study and edited the manuscript. All authors read and approved the final manuscript.

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Original Research Article

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ABSTRACT

The inhibiting action of *Myrianthus arboreus* leaves extract on the corrosion of copper in sulphuric acid solution was assessed using weight loss method at various temperatures. It was observed that the extract inhibited the acid-induced corrosion of copper and the corrosion rate decreased in the presence of the inhibitor as compared to the blank. Inhibition efficiency was found to increase with increasing concentration of the extract and temperature. Kinetic study of the data followed a first-order reaction. Thermodynamics study revealed that the corrosion inhibition might be due to spontaneous adsorption of the leaves constituents on the metal surface. The adsorption of the extract on copper surface was found to obey Freundlich and Temkin adsorption isotherms.

*Corresponding author: E-mail: cookey.grace@ust.edu.ng;



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1. INTRODUCTION

Copper is an important industrial metal due to its excellent properties [1]. It is known to be susceptible to corrosion in aggressive media especially in industries where acids are extensively used for cleaning, removal of rust, acid pickling and de-scaling processes [1]. Corrosion is an electrochemical process in which metal surfaces interact with aggressive environments, converting the metal atoms to ions thus causing the breakdown of physical and protective layers of the metal [2]. Acids aggressively enhance metal corrosion. Therefore it is essential to employ corrosion inhibitors to prevent the dissolution of metal materials such as copper [3]. For economic and environmental benefits, it is vital to use extracts of cheap, readily available biomass as corrosion inhibitors to prevent the problems of metal dissolution [4].

The inhibition abilities of plant parts extract usually are due to the presence of heterocyclic constituents such as flavonoid, tannin, alkaloids, nitrogenous bases, proteins and carbohydrates which usually contain heteroatom like Oxygen, Sulphur or Nitrogen as well as triple or conjugated double bonds with aromatic rings in their molecular structures which are the main adsorption centre [5,6].

Some reports have been highlighted on the effective application of plants extracts as corrosion inhibitors for copper in different media, such as the inhibitive action of Morinda tinctoria leaves extracts in H₂SO₄ [7]. The influence of aqueous extract of Cerdtonia siliguaon corrosion of copper and brass in aqueous 1M nitric acid has been examined [4]. The use of ethanolic extract of Capparis decida seeds in controlling the corrosion of copper in HCI acid has also been reported [3]. Hart et al. [8] reported the inhibitive action of xanthosoma Spp leaf extracts (XLE) on the corrosion of copper in seawater. It has been shown that the extract of Azadirachta indica is a suitable inhibitor for copper corrosion in Nitric acid medium [9]. Adsorption and inhibitive properties of methanol extract of Eeuphorbia heterophylla for the corrosion of copper in Nitric acid solutions have been investigated [6]. Abd-El-Nabev et al. [10] have reported that aqueous extract of Alhagimaurorum is a good inhibitor for copper in H₂SO₄ acid medium. Savita et al. [11] found that fruit extracts of citrus aurantium,

Moringa oleifera and capsicum annuum are good corrosion inhibitors for copper in Nitric acid solutions. The inhibitive efficacy of Emblica officinalis leaves extract on the corrosion of copper and its alloy (Cu-27Zn) in natural seawater environment has been studied [12]. Inhibition efficiency (%IE) was observed to be markedly higher in natural seawater with the addition of the extract. Nkuzinna et al. [13] found that acid extracts of Gnetum Africana and Musa acuminate peel have the potential of inhibiting the corrosion of copper in Nitric acid. Aloe vera Barbadensis Gel as active corrosion inhibitor for copper in HCI acid has been reported by [14]. Inhibitive action of cannabis plant extract on the corrosion of copper in H₂SO₄ acid has been carried out by [15]. Corrosion behaviour of copper in phosphoric acid containing sodium chloride and its inhibition by Artemisia oil extract has been investigated by [16]. Inhibitive effect of Trigonella stellate as a suitable corrosion inhibitor for copper in HNO₃ solution was studied by [17]. Swati et al. [18] investigated the inhibition potential of Azardiracha indica fruit extract on the corrosion of copper in HCI acid solution. Fadel et al. [19] carried out an investigation on the corrosion inhibition of copper by Capparis spinosa L. extracts in strong acidic medium. It was observed that the extract inhibited the acid-induced corrosion to maximum IE (%) of 82.7%.

The successful results obtained from previous studies on naturally occurring substances to inhibit the corrosion of metals in acidic and alkaline environments have motivated this investigation on methanol extract of myrianthus arboreus as corrosion inhibitor for copper. Presently, and to the best of our knowledge, there is no reported work in the open literature on myrianthus arboreus leaves extract as corrosion inhibitor of copper. This study investigated the adsorption and inhibitive properties of methanol extract of myrianthus arboerus leaves for the corrosion of copper in H₂SO₄ acid solutions using weight loss technique. Myrianthus arboreus (giant yellow mulberry or bush pineapple) which is of the family Ceropiaceae, is a shrub found in the forest zone of tropical Western Africa. Extracts of the leaves and leafy shoots are used in the treatment of dysentery, diarrhoea, boils, dysmenorrhoea, incipient hernia and vomiting. The bark decoction is used to treat malaria, fever, cough and muscular pains.

2. MATERIALS AND TEST SOLUTION PREPARATION

The sheet of copper used for this study was obtained from engineering workshop of the University of Port Harcourt, Rivers State, Nigeria. The copper sheet of 0.1 cm thickness and 99.5% purity was mechanically press-cut into 5.0 x 2.5 cm coupons. The coupons were polished with different grades of emery paper to obtain a clean shiny surface, washed in deionized water, degreased in absolute ethanol, dried and then stored in a moisture free desiccator prior to use. 2M H_2SO_4 solution used as corrodent was prepared from pure H_2SO_4 purchased from Sigma-Aldrich. Deionized water was used for the preparation of all reagents used for the study.

2.1 Preparation of Extract

Fresh leaves of *Myrianthus arboreus* plants were obtained from Ukanafun in Akwa Ibom State, South-South Nigeria. The leaves were washed thoroughly and rinsed with deionized water to remove impurities; sun-dried to constant weight and pulverized using a blender. The extract was obtained using 99.8% methanol in a Soxhlet extractor. The resulting solution was evaporated to dryness in an oven at 40°C, weighed and stored in sample container before being used for phytochemical analysis. Different concentrations of the extract were prepared by dissolving 0.5g, 0.4 g, 0.3 g, 0.2 g and 0.1 g of the dry *Myrianthus arboreus* in 2M H_2SO_4 acid solution for weight loss measurements.

2.2 Weight Loss Method

A known weight of copper coupon was suspended in 60ml of the test solution in an open beaker in the absence and presence of the extract (inhibitor) between the temperatures of 303–333K. Thermostated water bath was used to regulate the temperatures. After every 2 hours progressively for 18 hrs, each sample was retrieved from the test solution, washed with deionized water, rinsed in acetone, dried in air and reweighed. The difference in weight after immersion and drying was taken as the total weight loss of each copper coupon. This procedure for weight loss determination was similar to that reported by [20] and stated in Equation 1.

$$\Delta W = W_i - W_f \tag{1}$$

where ΔW is the weight loss of the copper coupons, W_i is weight before immersion, while W_f is the weight after immersion in the test solution.

From the weight loss results, the percentage inhibition efficiency (%IE), corrosion rate (CR) and surface coverage (θ) were calculated using Equations, 2, 3 and 4 respectively.

$$IE\% = \left(\frac{\Delta W_B - \Delta W_{inh}}{\Delta W_B}\right) x100$$
 (2)

Where, ΔW_B , ΔW_{inh} are the weight losses of copper in absence and presence of *Myrianthus arboreus* leaves extract in H₂SO₄ acid solution at a particular temperature.

The degree of the adsorbent surface covered by the adsorbate (θ) was calculated using Equation 3.

$$\theta = \left[1 - \frac{\Delta W_{inh}}{\Delta W_B}\right] \tag{3}$$

Corrosion rates $(gcm^{-2}h^{-1})$ of copper in different corrosion media were determined using Equation 4

$$CR = \frac{\Delta W}{At} \tag{4}$$

Where, ΔW = weight loss (g), *A*, surface area (cm²) of adsorbent and t, time of immersion (hours).

3. RESULTS AND DISCUSSION

3.1 Effect of Time and Extract Concentration on Weight Loss

The variation of weight loss of copper in the 2M H_2SO_4 acid medium in the absence and presence of different levels of the *Myrianthus arboreus* leaves to extract as a function of time at temperatures of 303–333K is shown in Fig. 1.

Results obtained depict an increase in weight loss with immersion time and a decrease in concentration of the extract, with highest values of weight loss for the blank which probably was due to increase in corrosion rate. The reduction observed in weight loss with an increase in the

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concentration of the extract suggests an increase in surface coverage on the metal and the adsorption of the extract constituents on the metal surface which may have created a barrier between the metal and the acidic medium. Similar observations have also been reported [2,21].

3.2 Effect of Extract Concentration and Temperature on Inhibition Efficiency

The inhibition efficiency of copper exposed to different levels of *Myrianthus arboreus* leaves extract in 2M H_2SO_4 acid solutions at 303 to 333K is shown in Fig. 2.



Fig. 1. Variation of weight loss (g) with time (hrs) for Copper coupons in $2M H_2SO_4$ at different concentrations of extract and temperatures



Fig. 2. Variation of Percentage inhibition efficiency (%) with inhibitor Concentration (g/l) for the corrosion of copper coupons in 2M H₂SO₄ at different temperatures

The inhibition efficiency values were observed to increase with an increase in the concentration of *Myrianthus arboreus* extract from 0.1g/l to 0.5g/l. The maximum percentage inhibition of 97.67% was recorded at the highest level of 0.5g/l at 333K. Hence, increase in the frequency of the inhibitor appears to have significantly increased the adsorption of its molecules on the metal surface and subsequently retarded the dissolution of the metal in acidic medium, as has also been reported elsewhere [22,23].

The effect of temperature on the corrosion rate of copper in the absence and presence of different concentrations of Myrianthus *arboreus* leaves extract was carried out at the temperature range of 303 -333K as shown in Table 1.

It was observed that the corrosion rate of copper increased with increase in temperature for the uninhibited but decreased with increase in temperature for the inhibited systems. The similar result has been reported by Nwaugbo, and James [24] on the corrosion inhibition of mild steel in sulphuric acid solution by flavonoid separated from Nypa fruticans wurmb leaves extract. The effect of temperature on corrosion rate and corrosion inhibition efficiency was attributed to a change in adsorption type from physisorption to chemisorption as temperature increased [25].

We observed from Fig. 2 and Table 1 that, as the reaction temperature is increased from 303K to 333K, the inhibition efficiency increases. Hence, it is appropriate to say that increase in temperature favours the corrosion inhibition efficiency of *Myrianthus arboerus* on copper in H_2SO_4 acid solution. Similar observations have been reported [26,27].

3.3 Kinetic and Mechanism of Corrosion Inhibition Studies

To investigate the order of reaction, the weight loss the experiments were carried out at various inhibitor concentrations of 0.1g/l to 0.5g/l in 2M H_2SO_4 . The order of response was determined from the plot of log ($W_i - W_f$) = log(ΔW) against time in the absence and presence of *Myrianthus arboreus* leaves extract at 303K (Fig. 3).

The plots of log (ΔW) against time (hrs) at 303K in Fig. 3 showed a linear variation and the values of the constant rate k were obtained from the slope of the graph. A first order reaction kinetics concerning the corrosion of copper in 2M H₂SO₄

solution was confirmed. The half-life $t_{\frac{1}{2}}$ was calculated using Equation 5.

$$t_{\frac{1}{2}} = \frac{0.693}{k}$$
(5)

Table 2 indicates that the rate constant of the corrosion process decreases while the half-life increases with increase in the inhibitor concentration. As the $t_{\frac{1}{2}}$ increases, the rate constant decreases, which indicates that more protection of copper coupons by *Myrianthus arboreus* leaves extract has been established. This is also observed in literature [21,28,23].

3.4 Thermodynamics and Adsorption Studies

The values of activation parameters such as activation energy Ea, for corrosion of copper in the absence and presence of the extract at different concentrations over the working temperature range (303 – 333K) were obtained using Arrhenius equation (6).

$$\log CR = \log A - \left(\frac{E_a}{2.303RT}\right) \tag{6}$$

Where, CR is the corrosion rate at absolute temperature T, R is the universal gas constant, E_a is the apparent activation energy and A, the Arrhenius pre-exponential factor. The Arrhenius plot of log CR vs 1/T is shown in Fig. 4 for the corrosion of copper in the absence and presence of different concentrations of *Myrianthus arboreus*.

Linear curves were obtained from the Arrhenius plots with the slope as – ($E_a/2.303RT$). The values of E_a calculated from the slopes of the linear plots are given in Table 3.

Table 3 shows higher values of E_a in the presence of the extract when compared to that in the absence of the extract. The higher costs of E_a for the inhibited system which increase with increasing concentration of the extract suggest the strong inhibitive action of the extracts by increasing energy barrier for the corrosion process. Thus indicating a robust electrostatic character of the inhibitor's adsorption on the copper surface. The enthalpy and entropy of the

adsorption process were calculated using an alternative formula to Arrhenius equation, which is sometimes referred to as Transition state Equation (7), according to [29].

$$\log\left(\frac{CR}{T}\right) = \left[\left(\log\frac{R}{Nh}\right) + \frac{\Delta S}{2.30\Re}\right] - \frac{\Delta H}{2.30\Re T}$$
(7)

Where, h is the plank's constant, N, Avogadro's number, R, universal gas constant and T the absolute temperature. ΔH and ΔS are enthalpy and entropy of activation respectively.

Fig. 5 shows the plot of log (^{CR}/_T) vs 1 /_T for the corrosion of copper in 2M H₂SO₄ at the different concentrations studied.

Table 1. Corrosion rate (CR gcm⁻²hr⁻¹x10⁻³), inhibition efficiency (%IE) of extract in 2M H₂SO₄ acid solution at different temperatures

Temperature (K)	CR (gcm ⁻² hr ⁻¹) x10 ⁻³			IE (%)				
	303	313	323	333	303	313	323	333
Extract Conc.(g/l)								
Blank	18.05	24.24	28.07	31.89	-	-	-	-
0.1	9.52	9.56	8.86	4.36	53.51	60.42	68.51	86.38
0.2	8.92	8.81	8.02	3.39	56.49	63.55	71.48	89.43
0.3	8.29	8.09	7.17	2.38	59.47	66.48	74.62	92.57
0.4	7.69	7.58	6.54	1.48	62.47	68.62	76.72	95.40
0.5	7.25	7.08	5.97	0.75	64.58	70.67	78.74	97.67



Fig. 3. The variation of log (Δ W) with time (hrs) for the corrosion of copper coupons in 2M H₂SO₄ solution with and without the leaves extract at 303K

Table 2. Rate const	ant (k) and half-lif	e parameters at various	concentrations of the extrac
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Temp.(K)	k (hr ⁻¹) x 10 ⁻³				Half-life (hr ⁻¹)			
	303	313	323	333	303	313	323	333
Extract Conc.(g/l)								
Blank	3.29	3.71	4.44	4.37	210.91	186.67	156.14	158.57
0.1	1.53	1.58	1.42	0.57	452.38	438.91	489.23	1210.20
0.2	1.43	1.44	1.27	0.45	483.23	479.98	545.49	1539.11
0.3	1.34	1.29	1.14	0.32	517.98	535.76	609.44	2177.53
0.4	1.23	1.21	1.04	0.19	563.37	571.64	663.79	3512.59
0.5	1.16	1.13	0.95	0.05	596.08	613.11	731.57	6595.60



Fig. 4. Arrhenius plot of log CR vs 1/T at different extract concentrations for the corrosion of copper coupons in 2M H₂SO₄ solution



Fig. 5. Plots of log (CR/T) vs 1/T for the corrosion of copper in $2M H_2SO_4$ at different concentrations of the extract

Table 3. The Activation Energy (E _a) values at	ļ
the various concentrations of the extract	

Extract Concentration(g/l)	E _a (kJ/mol)
Blank	12.31
0.1	20.43
0.2	24.92
0.3	32.17
0.4	42.33
0.5	58.01

Straight line graphs were obtained with a slope of -(Δ H/2.303R) and an intercept of [log (R/Nh) + Δ S*/(2.303R)], from where the values of Δ H and Δ S were calculated and shown in Table 4.

The values of ΔH in the absence and presence of the extract are presented in Table 4.

Table 4. The Enthalpy (Δ H) and Entropy (Δ S)
values of corrosion at the various
concentrations of the extract

Extract	$\Delta \mathbf{H}$			
Conc.(g/l)	(kJ/mol)	(J/mol)		
Blank	11.69	72.68		
0.1	22.72	99.51		
0.2	27.54	100.61		
0.3	34.78	101.53		
0.4	44.94	112.94		
0.5	60.63	160.49		

The positive values of ΔH in the absence and presence of the extract reflects endothermic nature of the copper dissolution process [30]. It is evident that the activation enthalpies vary in the same trend as the activation energies. The negative and significant values of ΔS in both solutions indicate that the activated complex of the adsorbate and adsorbent in the rate

determining steps represents association rather than dissociation step.

This suggests a decline in disorderliness ongoing from reactant to activated complex under some findings in the literature [31,32] and [17]. A negative value of entropy also signifies no significant change in the internal structure of the copper surface during the adsorption process.

3.5 Adsorption Consideration

Adsorption isotherms are very important in obtaining necessary information on the interaction between the inhibitor and the metal mechanism surface. The of organoelectrochemical reactions is also determined through adsorption isotherm [33,34]. The inhibiting process of the corrosion of copper in 2M H₂SO₄ medium at different concentrations of the extract can be explained concerning the adsorption of the components of the extract on a metal surface. Three adsorption isotherms were used to describe the adsorption mechanisms [21] of Myrianthus arboreus extract on copper coupons in 2M H₂SO₄ solutions. They are Freundlich (equation 8), Temkin (equation 9) and Langmuir (equation 10). The linearity of curves is an indication that a given isotherm obeys an adsorption process.

$$\log \theta = \log K_f + \frac{1}{n} \log C \tag{8}$$

Where K_f and C, represents the equilibrium constant of adsorption and extract concentrations respectively, $\frac{1}{n}$, the adsorption intensity and θ , the surface coverage. The continuous K_f is an approximate indicator of adsorption capacity while $\frac{1}{n}$ is a function of the strength of adsorption in the adsorption process. The values of $\frac{1}{n}$ and K_f can be evaluated from the slope and intercept of such plots respectively. If $\frac{1}{n} = 1$ then the partition between the two phases are independent of the concentration and if the value

of $\frac{1}{n}$ is below one, it signifies normal adsorption [21,35].

The Temkin isotherm is given as:

$$exp\left(-2a\theta\right) = K_{ad}C\tag{9}$$

The Langmuir is given as:

$$C/_{\theta} = \left(\frac{1}{K_{ad}}\right) + C \tag{10}$$

Where, *a* is molecular interaction parameter. The values of *a* and K_{ad} can be evaluated from the slope and intercept of such plots respectively. Linear plots in Figs. 6 and 7 suggested that the experimental data fits Freundlich and Temkin adsorption isotherm. The estimated values for K_{f} , $\frac{1}{n}$, *a* K_{ad} and R^2 for the two isotherms at various temperatures are shown in Table 5.

Considerable K*f* value indicates greater adsorption capacity, low amounts of $\frac{1}{n}$ obtained from Freundlich plot shows normal adsorption. The negative values of "*a*"derived from Temkin plot showed that repulsion exists in adsorption layer. The benefits of *a* and *K*_{ads} obtained signify strong adsorbate – adsorbent molecular attraction and great binding strength [36,37] and [38].

The equilibrium constant of adsorption (K_{ad}) obtained from the intercepts of θ vs log C plot is related to the free energy of adsorption (ΔG_{ads}). The (ΔG_{ads}) which characterizes the interaction of adsorption between the molecules of leave extracts and metal surface was calculated using Equation 11 and also presented in Table 6.

$$\Delta G_{ads} = -2.303 RT \log(55.5 K_{ad}) \tag{11}$$

where, R is the gas constant, T, temperature, K_{ad} , equilibrium constant of adsorption and 55.5 is the molar concentration of water in solution.

T(K)		Freundlich			Temkin		
	R^2	Kf	1/n	R^2	а	K _{ad}	
303	0.99	0.69	0.02	0.99	-0.02	1.01	
313	0.99	0.75	0.01	0.99	-0.02	1.01	
323	0.99	0.81	0.01	0.99	-0.02	1.01	
333	0.99	1.01	0.01	0.99	-0.02	1.01	

 Table 5. Freundlich and Temkin parameters for the adsorption of

 Myrianthus arboreus leave on a Copper surface at 303 – 333K



Fig. 6. Plot log θ vs log C (Freundlich adsorption) for the inhibition of copper corrosion in 2M H₂SO₄ by different concentrations of extract at temperatures of 303-333K



Fig. 7. Plot θ vs In C (Temkin adsorption) for the inhibition of copper corrosion in 2M H₂SO₄ by different concentrations of extract at temperatures of 303-333K



Fig. 8. Langmuir adsorption plot (C/ θ vs C) for inhibition of copper corrosion in 2M H₂SO₄ by different concentrations of extract at temperatures of 303-333K

Temperature (K)	-∆G _{ads} (kJ/mol)
303	9.15
313	9.68
323	10.27
333	11.15

 Table 6. The calculated values of Free Energy of adsorption at various temperatures

Table 6 shows that the values of ΔG_{ads} are negative at all temperatures, indicating spontaneous adsorption of the leaves extract and good stability of the adsorbed layer on the copper surface [39]. The negative values of ΔG_{ads} for the extract signify that the adsorption mechanism of the extract on copper in 2M H₂SO₄ acid solution at the temperatures studied may be physisorption. Similar results have also been reported [40] on the inhibitive action of *the delonixregra* extract on the corrosion of aluminium in acidic medium.

Literatures reviewed reveal that negative values of ΔG_{ads} up to – 20kJ/mol or lower are consistent with electrostatic interaction between charged molecules and a charged metal (indicating physical adsorption) while those around or more than - 40kJ/mol involve charge sharing or transfer from the inhibitor molecules to the metal surface to form a coordinate type bond (indicating chemisorption) [41,42]. The mechanism of adsorption observed from this study could be attributed to the fact that Myrianthus arboreus leaves extract contains many phyto-constituents in which some can absorb chemically and others physically. Similar observations have been reported [27,21].

3.6 Phytochemical Constituents of *Myrianthus arboreus* Leave Extract

Qualitative phytochemical screenings of Myrianthus arboreus leaves extract revealed the presence of some chemical constituents according to [43,44] and [45]. The chemical structures of most of these phyto-constituents contain electron rich bond or hetero atoms that facilitate their electron donating ability. The presence of secondary metabolites in plants extracts has been identified as the factor responsible for the corrosion inhibitory properties on a metal surface [46]. Hence, the inhibition of the corrosion of copper by methanol extracts of Myrianthus arboreus is attributed to the phytoconstituents of the extract. Some researchers for

their inhibition have reported similar inferences. The corrosion inhibition of copper using three different types of flavonoid was studied, and inhibition efficiency of 92% was obtained. This indicates a suitable corrosion inhibitor [47]. The corrosion inhibition of mild steel in sulphuric acid solution by flavonoid separated from Nypa fruticans wurmb leaves extract was reported to be a useful and efficient corrosion inhibitor [24].

Table 7. The phytochemical Results for
Methanol extract of Myrianthus arboreus
leaves

Phytochemical constituents	Results				
Alkaloid	+++				
Flavonoid	+++				
Saponin	+				
Tannin	+				
Terpenoids	+				
Carbohydrates	+				
Cardiac glycosides	+				
protein	+				
steroids	-				
Anthraquinone	-				
Fixed oils	-				
— =Absent, + = Present,					
++ = moderately present,					

+++ = in high concentration.

4. CONCLUSIONS

This study has shown that the methanol extract of Myrianthus arboreus leave is a good and efficient inhibitor for the corrosion of copper in 2M H₂SO₄ solution, particularly at increased inhibitor concentration. The maximum inhibition efficiency of 97.67% was obtained. The Ea values obtained were found to be higher for the inhibited system than the uninhibited system which shows that the adsorbed organic constituent provided a physical barrier for charge and mass transfer, leading to decrease in dissolution rate of copper. The mechanism of physical adsorption has been proposed, and a first order type of reaction was obtained from the kinetic treatment of the experimental data. The adsorption mechanism was observed to obey Freundlich and Temkin adsorption isotherms at all concentrations and temperature ranges studied.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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