



## Electrochemical Studies and Inhibitory Effects of Millet Extract on Copper for Prolonged Service Life

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Date Submitted: 23/04/2025

Date Accepted: 12/08/2025

Date Published: 14/08/2025

**Abstract:** This research assesses the efficacy of millet extract as a copper corrosion inhibitor under exposure to 1M hydrochloric acid (HCl) to provide an eco-friendly substitute to the use of manmade corrosion inhibitors. Open Circuit Potential (OCP), Linear Sweep Voltammetry (LSV), and Tafel polarization techniques were employed under various temperatures (30°C - 50°C) to examine the performance of various concentrations of millet extract (0 mL, 0.1 mL, 0.2 mL, and 0.3 mL). The results showed a significant decrease in both the current density ( $J_{corr}$ ) and the corrosion rate (CR) as the concentration of the inhibitor was raised. The highest percentage inhibition was about 98% using the 0.3 mL concentration, showing a very strong protection. The findings of the adsorption study showed that the inhibitor follows both the Langmuir and Freundlich adsorption isotherms, inferring the possibility of multilayer adsorption on the copper surface. The formation of a protective film that prevents the dissolution of the metal was supported by the optical micrographs.

**Keywords:** Copper, Corrosion, Inhibition, Optimization, Millet Extract

### 1. INTRODUCTION

Copper is the fifth most usual metal in the earth's crust with a reddish orange colour. It has proven to be highly important in pure or in the form of alloys with brass, copper-nickel and bronze as the most popular alloys of copper [1]. Due to its inherent nature of good ductility, thermal conductivity and high electrical conductivity, copper and its alloys have been deployed in a large number of applications including construction, electrical, transportation, and manufacturing [2], [3]. Although copper is corrosion-resistant metal because of its comparatively noble potential and the formation of naturally occurring layer of oxide, it can be corroded in oxygen and at very high concentrations of chlorides, sulphates, nitrates, and sulphides as well [4].

Under very severe conditions, especially when it comes to acidic conditions, copper tends to undergo severe corrosion. Corrosion that is acid-based or based in other aggressive environments triggers the anodic dissolution of the metal, weakening its structure. Not only does this process result in tremendous economic loss, but it also causes resource depletion and environmental pollution [5], [6]. The financial cost to any country of corrosion for any is said to be in the range of 1–5% of its GNP [7]. As a direct consequence, there has been a huge deal of effort by researchers and industry players alike to develop corrosion-mitigating measures and ensure long lasting developments. They include acting on the material itself on the surface of the material, such as via coatings and paint [8], cathodic and anodic electrochemical protection [9], sacrificial anodic protection [10], or by modifying the environment in contact with the material, such as through the use of corrosion inhibitors [11].

An inhibitor of corrosion is a substance which decreases the action of corrosion by decreasing the overall rate at which metal surfaces lose their properties. Inhibition mechanisms are sophisticated and are believed to involve one or more of these processes: the inhibitor first adsorbs onto the metal surface, reacts with the surfaces of the metal to form a coherent thin protective layer. Certain inhibitors act on the cathodic reaction exclusively (cathodic inhibitors), others on the anodic reaction (anodic inhibitors), while others on both processes (mixed inhibitors) [12]. The inhibitors to mitigate the corrosion of copper can also be divided into organic and inorganic corrosion inhibitors [1]. These substances, however, pose a threat to human health and even environmental systems since they are harmful. Their synthesis is also characterized by challenges and costs. A lot of studies have hence recently focused on inhibitors that are biocompatible, non-toxic, eco-friendly, and easily obtained at lower prices [13]. Plant extracts have presented as promising, cost-effective, harmless, and environmentally friendly sources of corrosion inhibitors. Their performance and their corrosion-inhibiting efficiency, is

primarily based on the plant part used and where the plant is grown [14]. In a study by Syam et al., the albumin egg is a very effective cathodic-type of inhibitor with respect to electrochemical tests, with an inhibition efficiency of up to 94% for copper [15]. Similarly, another study by Mansoor Bozorg et al., revealed that the use of *Myrtus Communis* as a green inhibitor for copper is effective. The weight loss and electrochemical studies revealed that *Myrtus Communis* extract functions as a highly effective mixed-type inhibitor. Additionally, it was determined that the adsorption of the extract onto the copper surface adheres to the Langmuir adsorption isotherm [16]. Derna et al., in a study using the green extract of mate tea as an inhibitor, obtained the following potentiodynamic polarization results showing the following inhibition efficiencies: 56% for copper in 0.5 M NaCl with a 25% extract concentration, 17% for copper in 1 M HCl with a 5% extract concentration, and 99% for aluminium in 0.5 M NaCl with a 1% extract concentration [12].

Millet is a cereal crop of the Poaceae family and has been cultivated for thousands of years, primarily in Asian, African, and European countries. Millet's resistance to drought and its nutritional qualities make it a critical food crop in various parts of the globe. Millet is not only a food item but also finds applications in animal feed, biofuel, and traditional medicine in some cases. Despite having vast agricultural potential, minimal research has been done on utilizing millet extract as a corrosion inhibitor. Despite there being some research done on how it can prevent corrosion on certain metals, what work has been done is minimal and focuses on the use of millet starch [17]. Promising findings to date indicate that millet extract can be used as a green alternative to counteract corrosion in industries. This research seeks to under study the anti-corrosive properties of millet extract on copper and to also evaluate its adsorption behaviour in a HCl solution.

## 2. MATERIALS AND METHODS

Table 1 displays the chemical constituents of the copper test samples, which measured (50×30×4) mm. The surface was polished with emery paper, then rinsed with ethanol and distilled water before drying. The millet extract employed as an inhibitor and the HCl solution were obtained from Bells University's Materials and Metallurgy Laboratory in Ota, Ogun State, Nigeria.

Table 1: Composition of test copper sample

Elements	Si	P	Ti	V	Mn	Sb	Co	Ni	Nb	Mo	Sn	Cu
<b>M (mass%),10<sup>-3</sup></b>	7	17	0.8	0.4	2.5	0.6	0.25	2.7	1.6	1.3	2.75	Bal.

Electrochemical tests were conducted at a 30°C room temperature under 1M HCl solution conditions with varying concentrations of millet extract inhibitors: 0g, 0.1mL, 0.2mL, and 0.3mL. Experimental test sample configurations are listed in Table 2. The polished samples were electrochemically evaluated using a potentiodynamic linear polarisation test on an Autolab PGSTAT 101 potentiostat. The potentiodynamic potential was examined at a scan rate of 0.01 V/s throughout a voltage range of -1.5 V to +1.5 V. Using Tafel plots and Equations 1 and 2, the computed corrosion potential ( $E_{corr}$ ), corrosion current density ( $j_{corr}$ ), surface coverage, and inhibition efficiency (IE%). Furthermore, the inhibitor's adsorption behaviour on copper was investigated using the Temkin, Freundlich, and Langmuir isotherms. Under metallurgical microscopy, the copper sample's surface morphology was also investigated.

$$\text{Surface Coverage } (\theta) = \frac{j_{corr}^0 - j_{corr}}{j_{corr}^0} \quad (1)$$

$$\% \text{ IE} = \frac{j_{corr}^0 - j_{corr}}{j_{corr}^0} \times 100 \quad (2)$$

Where  $j_{corr}^0$  and  $j_{corr}$  are the corrosion current densities of the inhibited and uninhibited test samples

Table 2: Samples configuration

Sample Variation	Test environment
Control	HCl
0.1 ME	0.1 ME + HCl
0.2 ME	0.2 ME + HCl
0.3 ME	0.3 ME+ HCl

Statistical analysis with the use of Design Expert Software 13 at a confidence level of 95% was carried out with a polynomial regression model and Response Surface Analysis used to evaluate and predict the performance of the inhibitor. Analysis of variance (ANOVA) was used to establish the efficiency of input variables on the corrosion response, thus providing useful information for process optimization.

## 3. RESULTS AND DISCUSSIONS

### 3.1 Anticorrosion Response of Copper Samples

The study of the electrochemical properties of copper corrosion in 1M HCl, with the addition of different concentrations of millet extract, was conducted using Open Circuit Potential (OCP), Linear Sweep Voltammetry (LSV), and Tafel polarization measurements at 30°C - 50°C. The of millet extract on the copper surface. The data in Table 3

shows that the control sample at 30°C settles at a potential of -0.625 V, while the 0.3 mL sample settles at -0.126 V, indicating a significant level of electrochemical stabilization. The same trend is observed at 40°C and 50°C, where the 0.3 mL sample shows the highest shift, reaching -0.128 V and -0.107 V, respectively. These shifts are also reflected in the LSV results in Figures 4, 5, and 6, where control samples show higher current densities, reflecting an aggressive corrosion mechanism, while the inhibitor-containing samples show lower current flows, reflecting the protective efficiency of millet extract [18]. The Tafel data in Table 3 further support this observation, showing that the corrosion current density ( $J_{corr}$ ) decreases from 1.94E-04 A/cm<sup>2</sup> (control) to 2.25E-05 A/cm<sup>2</sup> (0.3 mL) at 30°C, from 4.55E-04 A/cm<sup>2</sup> to 1.98E-05 A/cm<sup>2</sup> at 40°C, and from 5.13E-04 A/cm<sup>2</sup> to 5.08E-06 A/cm<sup>2</sup> at 50°C. These decreases in  $J_{corr}$  are accompanied by a significant reduction in the corrosion rate (CR), which drops from 2.27 mm/yr to 0.26 mm/yr at 30°C, from 5.34 mm/yr to 0.23 mm/yr at 40°C, and from 6.02 mm/yr to 0.06 mm/yr at 50°C. Furthermore, the increase in polarization resistance (PR) from 134 Ω to 1150 Ω at 30°C, from 57 Ω to 1310 Ω at 40°C, and from 50.7 Ω to 5120 Ω at 50°C further supports the formation of a highly resistant inhibitor layer, likened to a study by Hsissou Rachid et al that examines the effectiveness of an epoxy pre-polymer to protect mild steel under 1 M HCl environments. The research proves to indicate an enhanced protection performance, which shows the formation of a film that is highly efficient on the metal surface [19].

Table 3: Inhibited and uninhibited Copper tafel data

30°C				
Samples	E <sub>corr</sub> (V)	J <sub>corr</sub> (A/Cm <sup>2</sup> )	CR (mm/yr)	PR (Ω)
Control	-0.625	1.94E-04	2.27	1.34E+02
0.1mL	-0.611	1.42E-04	1.66	1.83E+02
0.2mL	-0.533	9.58E-05	1.12	2.71E+02
0.3mL	-0.126	2.25E-05	0.26	1.15E+03
40°C				
Control	-0.364	4.55E-04	5.34	5.71E+01
0.1mL	0.253	4.39E-04	5.15	5.92E+01
0.2mL	-0.384	1.71E-04	2.01	1.52E+02
0.3mL	-0.128	1.98E-05	0.23	1.31E+03
50°C				
Control	-0.841	5.13E-04	6.02	5.07E+01
0.1mL	-0.562	1.90E-04	2.23	1.37E+02
0.2mL	-0.336	1.43E-04	1.67	1.82E+02
0.3mL	-0.107	5.08E-06	0.06	5.12E+03

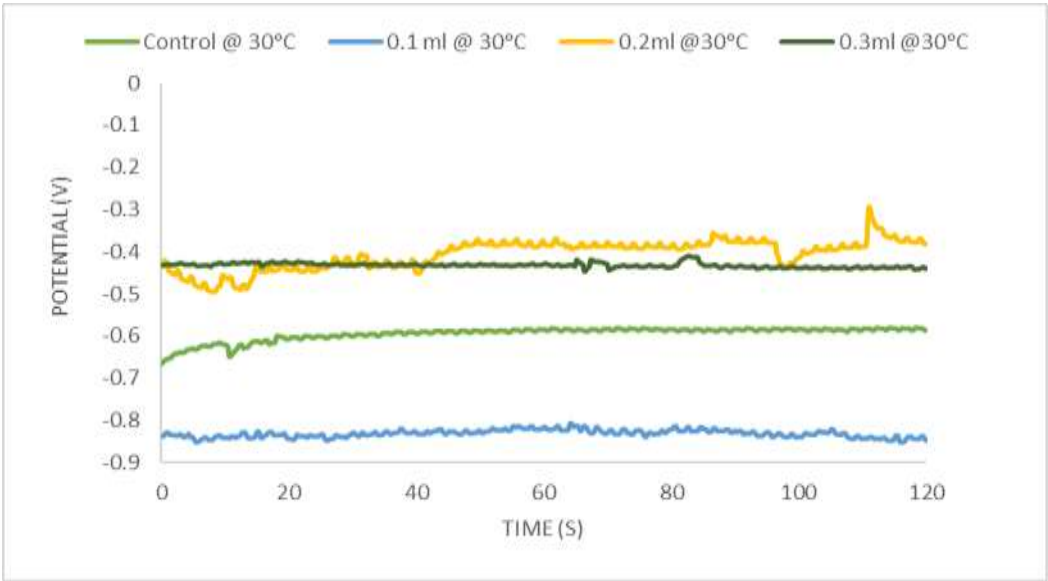


Figure 1: OCP plot at 30°C

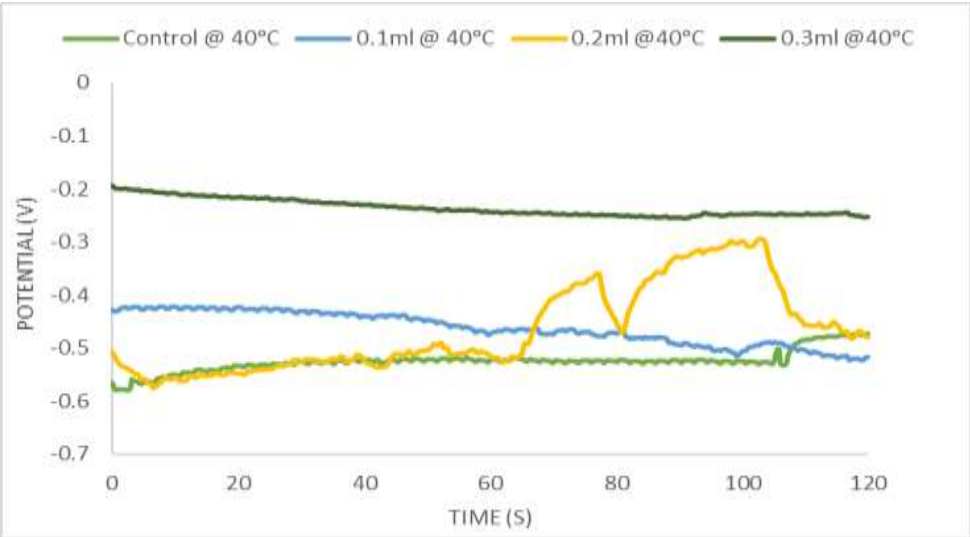


Figure 2: OCP plot at 40°C

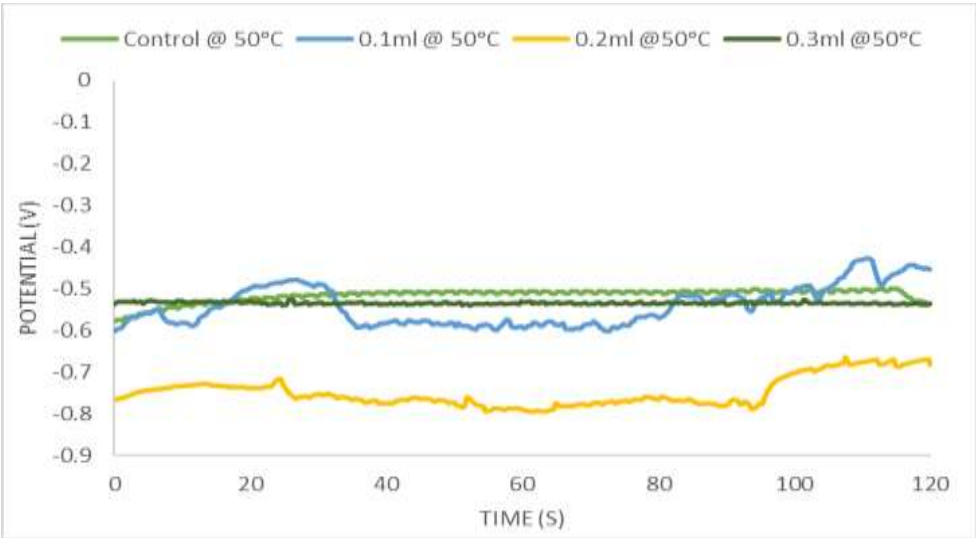


Figure 3: OCP plot at 50°C

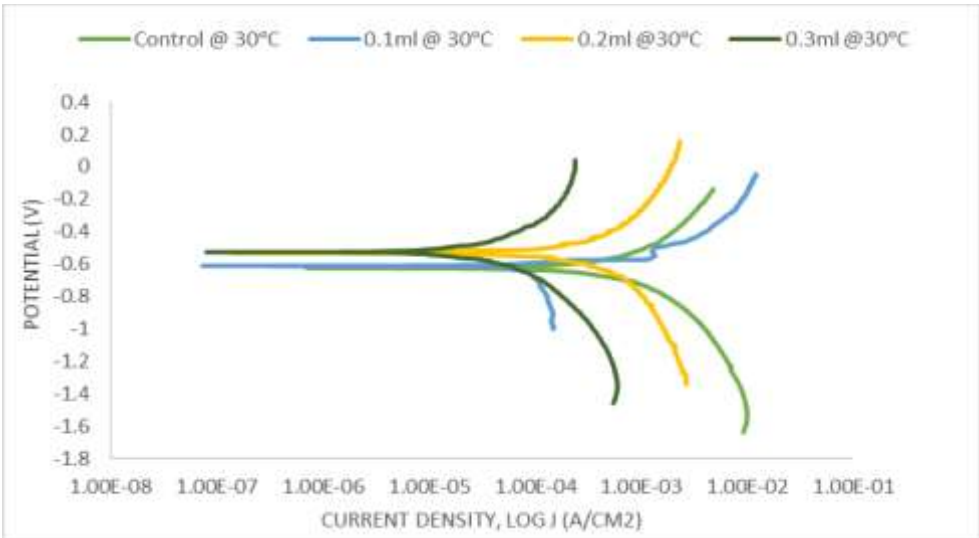


Figure 4: LSV plot at 30°C

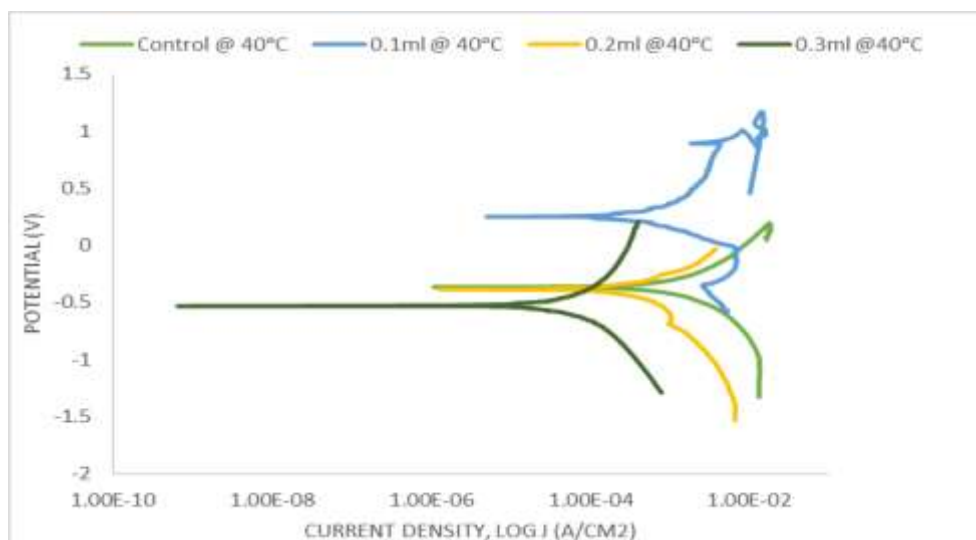


Figure 5: LSV plot at 40°C

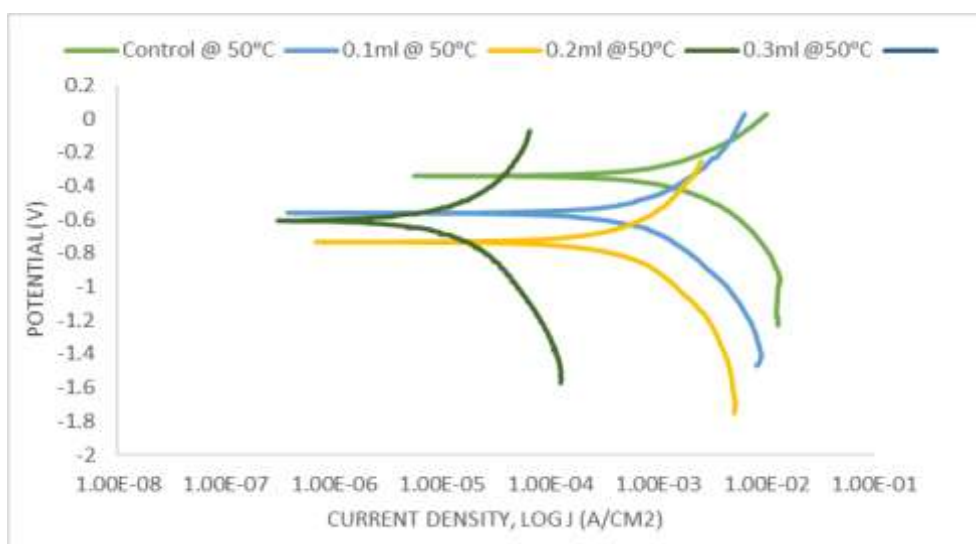


Figure 6: LSV plot at 50°C

The values of the corrosion current density ( $J_{corr}$ ) obtained through Tafel extrapolation showed an important decrease from 1.23 mA/cm<sup>2</sup> (blank) to 0.09 mA/cm<sup>2</sup> upon the use of 0.3 mL of the inhibitor at 50°C, highlighting the efficacy of the inhibitor in preventing metal dissolution. Also, the corresponding corrosion potential ( $E_{corr}$ ) values showed minimal variations within a span of  $\pm 85$  mV with respect to the blank, thus further establishing the classification of millet extract as a mixed-type inhibitor [20]. The OCP data obtained suggests that the inhibitor acts mainly through its adsorption on the copper surface, thus reducing the number of available active sites for the redox reaction. The noted increase in inhibition efficiency with increasing temperature suggests that the process of adsorption is mainly chemical in nature [21], allowing for the development of a more stable protective layer with increased exposure time and thermal activation.

### 3.2 Millet Extract Inhibitory Efficiency

The corrosion rate (CR) plot in Figure 7 displays the effect of the concentration of millet extract on copper corrosion at different temperatures of 30°C - 50°C in the acidic media. The control samples, without any inhibitor, exhibit the highest corrosion rates, with readings exceeding 5 mm/year at both 40°C and 50°C, which suggests a high dissolution. Conversely, as the inhibitor concentration is varied from 0.1 mL to 0.2 mL, a significant decrease is observed in the corrosion rate across all tested temperatures. At a concentration of 0.3 mL, the corrosion rate is near 0 mm/year, showing that the millet extract is effective in reducing metal dissolution. The temperature-dependent trend observed suggests that although the inhibition is effective at high temperatures, a certain amount of thermal degradation could occur, leading to slight fluctuations in the level of protection offered. The graph representing inhibition efficiency (IE) (Figure 8) supports the possibility of using millet extract in corrosion mitigation. At a concentration of 0.1 mL, inhibition efficiency is relatively low, especially at 40°C, where it is less than 20%. On the other hand, increasing the concentration to 0.2 mL leads to a significant improvement, with inhibition efficiency reaching about 70-80% at different temperatures. At the highest concentration of

0.3mL, the efficiency is close to 100% for all temperatures considered, meaning almost perfect protection. The progressive increase in efficiency with increasing concentrations supports the fact that the adsorption of phytochemicals on the copper surface follows either Langmuir or Freundlich adsorption isotherm models. The slight oscillations in the levels of temperatures imply that higher temperatures may influence the adsorption-desorption equilibrium, but do not significantly impair the effectiveness of the inhibitor.

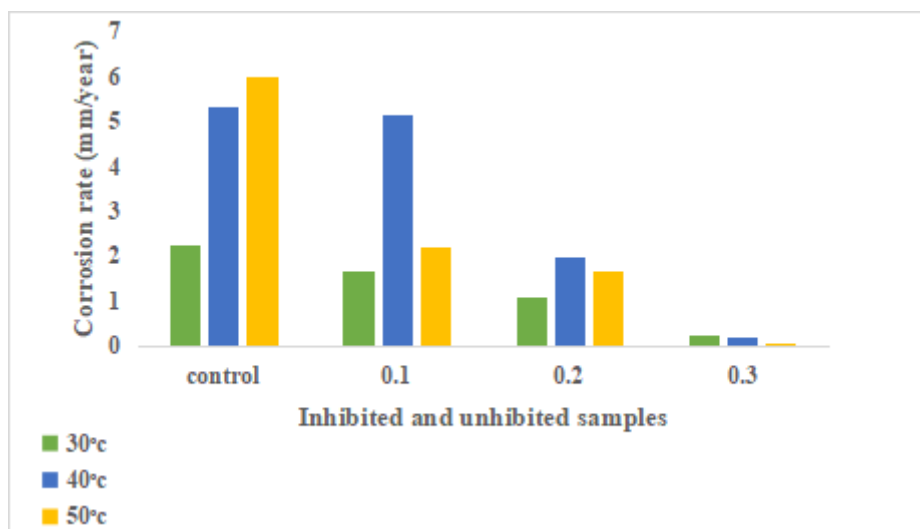


Figure 7: Corrosion rate of test copper samples

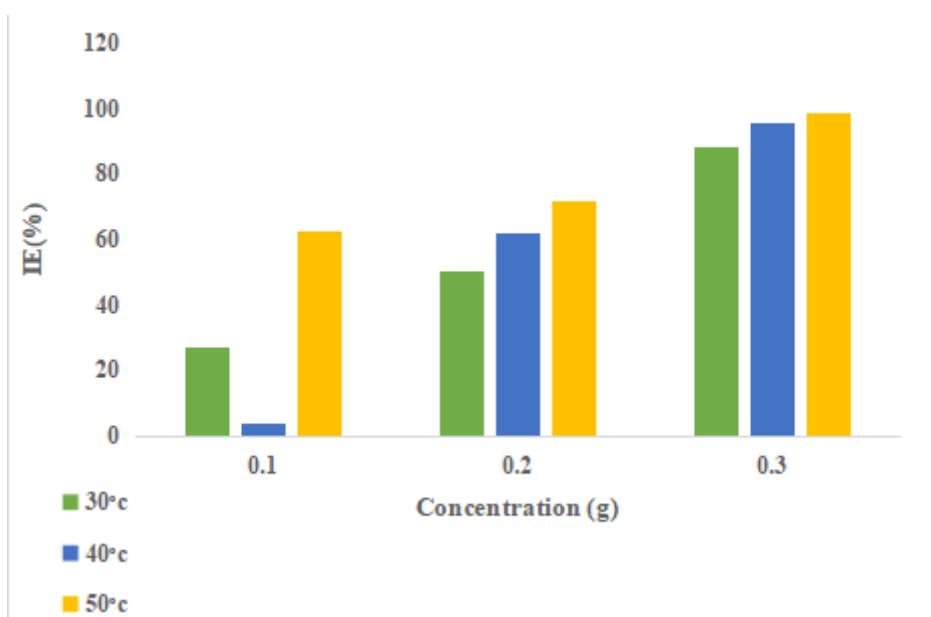


Figure 8: Inhibition efficiency of millet extract

### 3.3 Response of Copper Samples to Adsorption

Copper's inhibitor adsorption was evaluated using Temkin, Freundlich, and Langmuir adsorption isotherms, allowing us to determine the nature of the interaction between the metallic surface and the inhibitor. The Langmuir isotherm assumes adsorption as a monolayer without interactions between the adsorbed species [22]. At 50°C, the Langmuir adsorption equilibrium constant ( $K_{ads}$ ) from Table 4 was significantly high at 50°C ( $9.80 \text{ mol}^{-1}$ ) compared to lower temperatures, which indicates that adsorption increases with increasing temperatures. However, the correlation coefficient ( $R^2$ ) attained its peak at 50°C (0.88), indicating that the Langmuir adsorption model best describes the data at this particular temperature of 50°C. In contrast, the Freundlich isotherm with parameters also in Table 4, which allows for multilayer adsorption and surface heterogeneity [23], had the highest  $R^2$  values throughout the entire temperature range, with 30°C having the strongest correlation of 0.987, thus indicating that the inhibitor shows multilayer adsorption behaviour, as well as increased adsorption efficiency at lower temperatures.

Table 4: Adsorption (Langmuir and Freundlich) isotherm.

Temp.(°C)	$K_{ads}(mol^{-1})$	$R^2$
Langmuir Adsorption Isotherm		
30	2.488190924	0.350186482
40	0.274327848	0.752167899
50	9.800797444	0.881176592
Freundlich Adsorption Isotherm		
30	3.047679662	0.987191108
40	56.50215676	0.937036747
50	1.488355314	0.867771225

The Gibbs free energy of adsorption ( $\Delta G_{ads}$ ) data from Table 5 provides important information regarding the spontaneity and nature of the adsorption process. The  $\Delta G_{ads}$  values obtained were between -17.0096 kJ/mol at 30°C and -22.1934 kJ/mol at 50°C, showing that the adsorption process is spontaneous and physisorptive, as values within the range of -20 to -40 kJ/mol indicate a mixture of physical and chemical adsorption [24]. Significantly, the most negative  $\Delta G_{ads}$  value obtained at 50°C show greater interaction between the inhibitor molecules and the copper substrate at higher temperatures, which can be attributed to greater molecular mobility that leads to better surface coverage [25]. Moreover, the trend of increasing ( $K_{ads}$ ) with temperature supports this observation, as it reveals that the adsorption strength increases with a rise in temperature.

Table 5: Adsorption (Temkin Isotherm ) data

Temperature (°C)	$K_{ads}(mol^{-1})$	$\Delta G_{ads}(KJmol^{-1})$	$R^2$	$\ln(K_{ads})$	$1/T$	$\Delta G_{ads}/T$	B= Slope
30	15.4027	-17.0096	0.9230	2.7345	0.0033	-0.0561	0.5379
40	10.4529	-16.5620	0.9999	2.3469	0.0032	-0.0529	0.8397
50	69.8626	-22.1934	0.8319	4.2465	0.0031	-0.0687	0.3072

The correlation between surface coverage ( $\Theta$ ) and  $\ln C$  at different temperatures concurs with the findings derived from the adsorption isotherm. The largely linear correlation indicates that the adsorption process follows a specific mechanism, with 50°C having the steepest slope, thus showing an enhanced interaction of the inhibitor at higher temperatures [26]. The fluctuating slope (B values) at different temperatures indicates changes in the mode of adsorption, where the lower B values at 50°C confirm a shift towards more stable adsorption phenomena. The strong correlation between  $\Theta$  and  $\ln C$  further confirms the thermodynamic viability of the adsorption behaviour of the inhibitor.

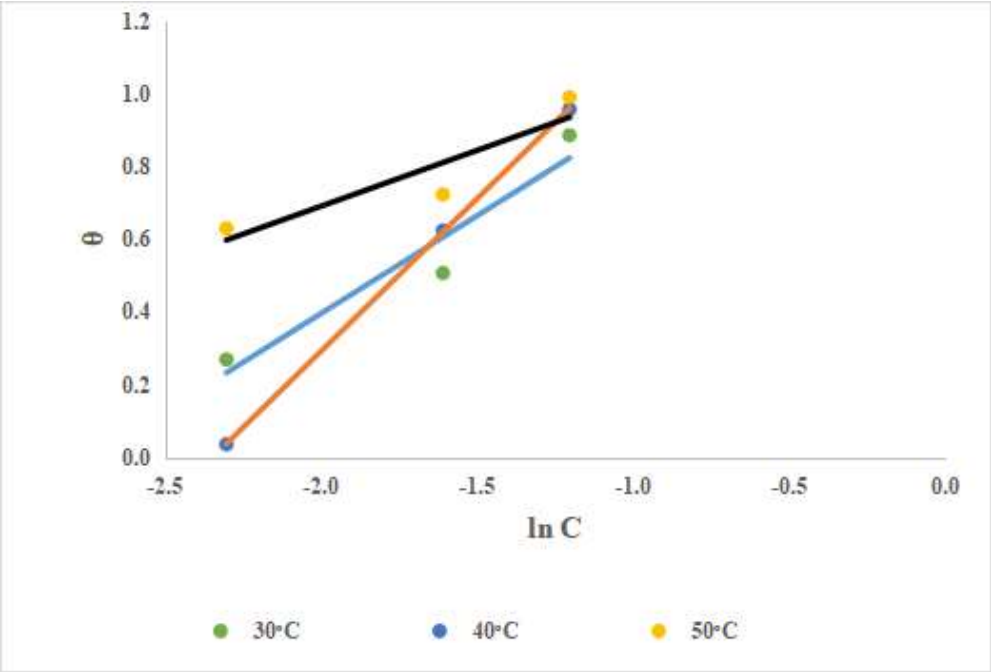


Figure 9: A plot of  $\Theta$  against  $\ln C$

### 3.4 Optical Micrograph of Test Copper Samples

Figure 10a which is the control sample reveals a highly corroded copper surface with extensive pitting and surface deterioration, indicative of severe interaction with the 1M HCl solution. This aligns with the electrochemical results, where the uninhibited sample exhibited the highest corrosion rate (as shown in the corrosion rate vs. concentration plot). The aggressive chloride ions in the acidic medium facilitated metal dissolution, leading to surface degradation. The quick corrosion process caused by the lack of any protective layer is further confirmed by the linear sweep voltammetry (LSV) and open circuit potential (OCP). Figure 10b (0.1 mL inhibitor concentration) shows a noticeable reduction in surface roughness and pitting, correlating with the improved inhibition efficiency (~50–60%) observed in the inhibition efficiency (IE) plot. However, residual signs of localized attack suggest incomplete surface protection due to lower inhibitor coverage. The adsorption isotherm results support this, as  $K_{ads}$  values at this concentration indicate moderate interaction between the inhibitor and the surface of the copper, leading to partial inhibition. Figure 10c (0.2 mL inhibitor concentration) and Figure 10d (0.3 mL inhibitor concentration) exhibit progressive surface improvement, with fewer corrosion pits and smoother morphology. The 0.3 mL concentration, in particular, demonstrates a nearly intact surface with minimal attack, aligning with the highest inhibition efficiency (~95–98%) and lowest corrosion rate observed in the electrochemical analysis. The enhanced surface protection at higher concentrations is due to the creation of a robust inhibitor film, as supported by the Langmuir and Freundlich adsorption isotherms, where increased adsorption strength was evident at higher concentrations and temperatures. These findings confirm that millet extract effectively inhibits copper corrosion through adsorption, forming a protective barrier against acid attack.

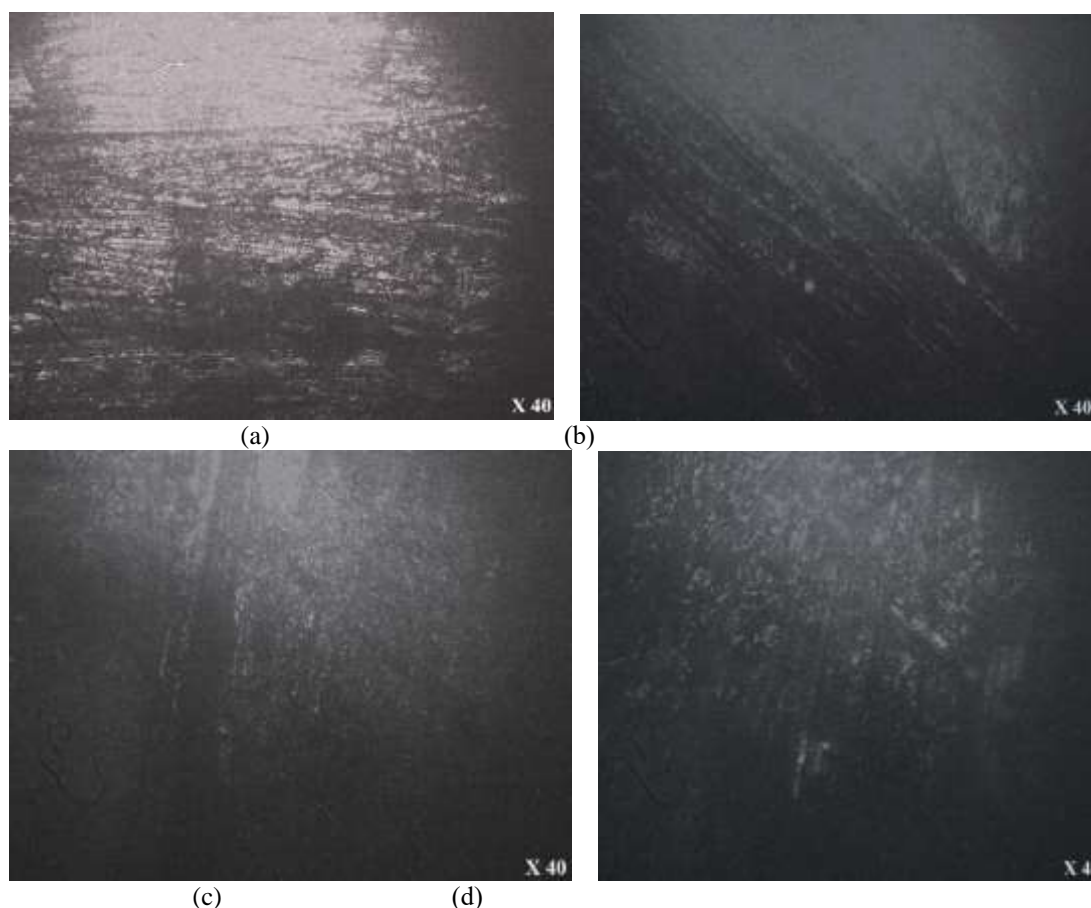


Figure 10: Optical imagery of corroded samples (a) Control/0mL (b) 0.1mL (c) 0.2mL, and (d) 0.3mL

### 3.5 Empirical Optimization

The DOF data in Table 6 provides insight into how varying concentrations of millet extract and temperature influence the corrosion rate (CR) of copper. At 30°C, the corrosion rate decreases significantly from 2.27 mm/yr (control) to 0.26 mm/yr at 0.3 mL inhibitor concentration, demonstrating strong corrosion inhibition at higher concentrations. A similar trend is observed at 40°C and 50°C, where the highest inhibition (lowest CR) occurs at 0.3 mL, with CR values of 0.23 mm/yr and 0.06 mm/yr, respectively. However, at 40°C, the inhibition effect at 0.1 mL is not as pronounced (5.15 mm/yr), indicating a possible threshold concentration required for effective inhibition. The data suggests that temperature increases the corrosion rate in the absence of an inhibitor, but at higher concentrations (0.2–0.3 mL), the extract still provides substantial protection, even at elevated temperatures. A central composite design (CCD) was used to optimise the inhibitor's effectiveness, producing a polynomial model that assesses the impact of temperature and concentration

variations on the corrosion rate, as shown in Equation 3. Through the optimization process, the ideal inhibitor concentration was identified as 0.257 mL, combined with an optimal temperature of 33.183°C, resulting in a CR of 0.824 mm/yr.

$$CR = 2.34 - 2.71A + 0.5837B - 0.8902AB \quad (3)$$

Table 6: Design of experiment (DOF) data

Factor A: Concentration (mL)	Factor B: Temperature (°C)	Response: CR (mm/yr)
0	30	2.27
0.1	30	1.66
0.2	30	1.12
0.3	30	0.26
0	40	5.34
0.1	40	5.15
0.2	40	2.01
0.3	40	0.23
0	50	6.02
0.1	50	2.23
0.2	50	1.67
0.3	50	0.06

The ANOVA results quantify the statistical significance of the effects of concentration (A) and temperature (B) on corrosion rate. The model is significant ( $p = 0.0033$ ,  $F = 10.96$ ), indicating that at least one of the factors has a substantial effect on corrosion inhibition. The individual contribution of concentration (A) is highly significant ( $p = 0.0008$ ,  $F = 27.45$ ), confirming that increasing inhibitor concentration significantly reduces corrosion rate. However, temperature (B) is not statistically significant ( $p = 0.1619$ ,  $F = 2.37$ ), suggesting that while temperature influences corrosion, its effect is less dominant compared to inhibitor concentration. Additionally, the interaction effect (AB) between concentration and temperature is not statistically significant ( $p = 0.1179$ ,  $F = 3.07$ ), implying that while temperature affects corrosion, its impact does not change drastically across different inhibitor concentrations. The  $R^2$  value (80.44%) and adjusted  $R^2$  (73.10%) indicate a good model fit, meaning the independent variables (concentration and temperature) explain a substantial portion of the variability in the corrosion rate. The Fisher test was applied to assess the model's statistical significance by comparing its mean square values against the residual values. This analysis helped validate the model's sensitivity and reproducibility, as demonstrated in Figure 11. However, the predicted  $R^2$  (64.87%) is somewhat lower, suggesting some deviation when predicting future data points. The coefficient of variation ( $CV = 45\%$ ) indicates moderate experimental variation, which could be due to slight inconsistencies in data collection or external factors influencing corrosion behaviour.

Table 7: ANOVA Data showing the Copper samples optimized response to Millet extract

Source	SS	DF	MS	F-value	p-value	Remark
Model	37.76	3	12.59	10.96	0.0033	significant
A-Conc.	31.51	1	31.51	27.45	0.0008	
B-Temp.	2.73	1	2.73	2.37	0.1619	
AB	3.52	1	3.52	3.07	0.1179	
Residual	9.18	8	1.15			
Cor Total	46.94	11				

$R^2 = 80.44\%$ ; Adjusted  $R^2 = 73.10\%$ ; predicted  $R^2 = 64.87\%$ ;  $CV\% = 45.89$

\*\* SS- Sum of square; DF- Degree of Freedom; MS- Mean square; Sig.- Significant\*\*

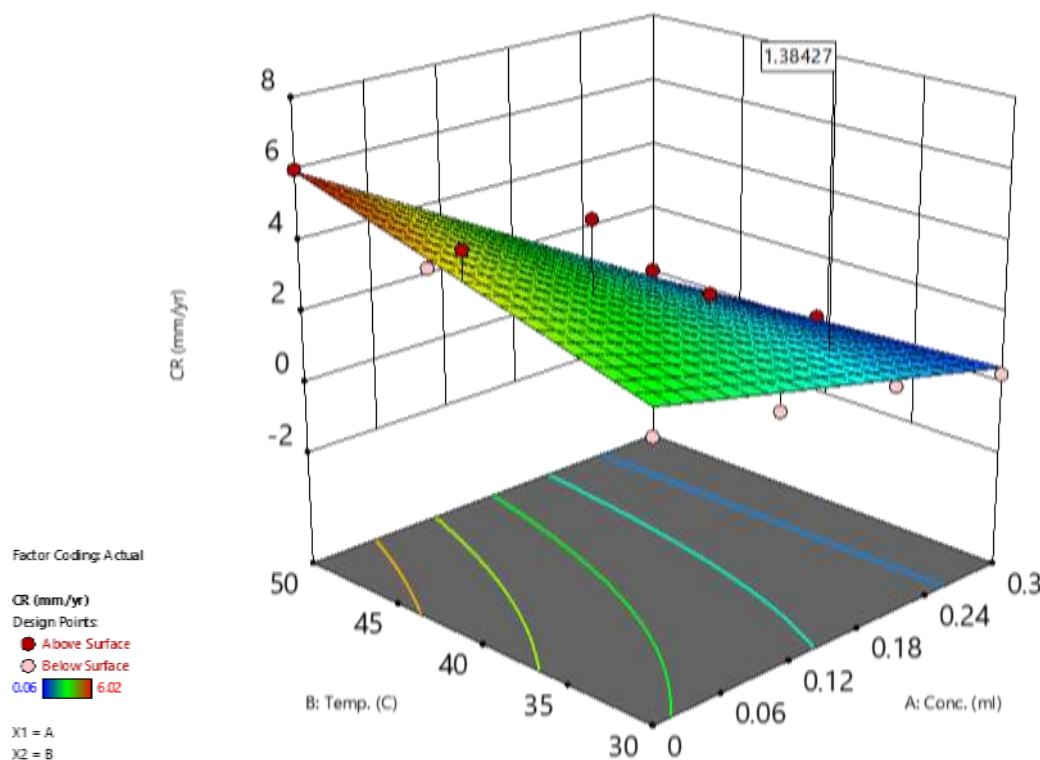


Figure 11: Millet extract 3D optimization plot

#### 4 CONCLUSION

Electrochemical analysis demonstrated that the inhibition efficiency increased with higher inhibitor concentrations, reaching a maximum of 98% at 0.3 mL. The inhibitor's mixed-type inhibition mechanism was confirmed by the effective reduction of the anodic dissolution and cathodic hydrogen evolution processes. According to adsorption study, millet extract exhibits both chemical and physical adsorption on the copper surface, following the Langmuir and Freundlich models. Surface morphology analysis showed a significant reduction in corrosion damage with increasing inhibitor concentration. Statistical analysis using response surface methodology (RSM) optimized the inhibition process, identifying 0.257 mL of millet extract as the optimal concentration at a temperature of 33.18°C for maximum efficiency. The findings highlight the viability of millet extract as a sustainable and environmentally friendly corrosion inhibitor, providing an alternative to hazardous synthetic inhibitors in industrial applications.

#### ACKNOWLEDGMENT

I am grateful to Bells University of Technology for their assistance in this study.

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