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Research Article

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Electrochemical and Corrosion Inhibition Properties of *Urena Lobata* Leaves Extract on Copper in 1M NaOH

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ABSTRACT

An investigation of the performance of *Urena lobata* leaves extract as a corrosion inhibitor, on copper in 1M NaOH solution was carried out using Gravimetric, Electrochemical and Surface analytical methods. Adsorption of the extract on the surface of the metal was studied using Langmuir and Freundlich isotherm plots. Phytochemical contents of the extract were determined using Gas Chromatography-Mass Spectrometric (GC-MS) technique. Corrosion inhibition of the extract was monitored at different concentration. The results obtained revealed that the leaves contain good phytochemicals with hetero atoms that are rich in electrons. The corrosion inhibition efficiency of the extract gave a maximum value of 94.8% at 2.5g/L extract concentration. It was observed that the corrosion rate of copper decreased with increase in the extract concentration in the basic solution. The adsorption process reduced as temperature increased thereby indicating physisorption. The Langmuir isotherms gave the best fit indicating chemisorption. Physical adsorption was confirmed for the leaves extract from observations of inhibition efficiency with temperature, activation energy and enthalpy of adsorption values, indicating that the extract has mixed properties. The inhibition efficiencies (% IE) were attributed to the phytochemical components (alkaloids, *saponins*, *flavonoids*, tannins, cynagenic glycosides) present in the extract.

Keywords: corrosion inhibitors, corrosion rate, inhibition efficiency *Urena lobata* copper

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INTRODUCTION

Corrosion is a natural process of conversion of refined metals to a stable form such as its oxide, sulphide or hydroxide state which enhances deterioration of the material (Li et al., 2019 Obi-Egbedi et al., 2011). It occurs in both acidic and basic media. Corroded products act as toxic contaminants in food, drugs, paints, dye, etc (Satapathy et al., 2009).

Recent studies have shown that organic compounds are adsorbed effectively on metal surface due to presence of multiple bonds, and heteroatoms such as N, S or O in them (Enyinnaya et al., 2021). These organic compounds are key constituents of corrosion inhibiting materials which can be synthetic or natural (green inhibitors). Green inhibitors are usually from plant materials which

are nontoxic, biodegradable and environmentally friendly; therefore, they are good substitutes for organic and inorganic corrosion inhibitors which are not biodegradable and are toxic to the environment. Medicinal herbs, plants, fruits and vegetable peel extracts are presently the major sources of green corrosion inhibitors (Emembolu et al., 2000; Aejitha et al., 2015). Phytochemicals, such as saponins, alkaloid, steroids, tannin, glycosides and amino acids which are found in some plant extracts have contributed to their inhibitive ability (Fouda et al., 2013; , Loto et al., 2016), because it gets adsorbed on the surface of the metal thereby protecting the metal from losing electrons (Ebenso et al., 2009; Maduelosi and Timothy, 2019). When metals lose electrons, they get oxidized and form oxides of the metal, which are undesirable.

The green inhibitor used in this research is *Urena lobata*, commonly referred to as Caesar weed or Congo jute. It is a weed commonly found on waterways, open woodlands, waste areas, roadsides and disturbed locations in tropical and sub-tropical regions. It is a tender perennial, erect, variable, ascendant shrub or subshrub with height of about 0.5 m to 2.5 m (Weeds of Australia, 2022). Evaluation of the corrosion inhibitory ability and thermodynamic properties of the leaves extract of this plant, using gravimetric method, adsorption isotherms, Scanning Electron Microscope (SEM), Potentiodynamic Polarization (PDP) and Electrochemical Impedance Spectroscopy (EIS) was carried out in this research.

MATERIALS AND METHODS

Collection and preparation of coupons

The copper sheets used for this research were gotten from mechanical engineering workshop, Rivers State University, Port Harcourt, Rivers State, Nigeria. The copper had a thickness of 1.5 mm. They were mechanically cut into coupons with measurements of 3 cm x 3 cm. The coupons were kept in moisture free desiccators after weighing for corrosion studies.

Collection and preparation of plant extracts

Urena lobata leaves were collected from Nwineenae's garden, Zaami Street, Bori, Rivers State, Nigeria. Ethanol was used to extract from the plant powder via the batch extraction method. Extract concentrations of 0.5, 1.0, 1.5, 2.0 and 2.5 g/L were prepared and used for the study. The phytochemical components of the leaves extract were determined by GC-MS analysis.

Gravimetric analysis (weight loss method)

Corrosion study was carried out by measuring weight

loss of the metals at different temperatures, extract concentrations and time intervals. The corrosion rates (CR), degree of surface coverage (θ), and inhibition efficiencies (% I.E) values were obtained from the weight loss data, applying the equations below:

$$CR \text{ (gcm}^{-2}\text{h}^{-1}\text{)} = \frac{WL}{At} \quad (1)$$

WL is weight loss in gramme, A is surface area of the metal and t is the immersion time in hours.

$$\theta = \left[\frac{CR_{Blank} - CR_{Inhibitor}}{CR_{Blank}} \right] \quad (2)$$

$$\% \text{ IE} = \left[\frac{CR_{Blank} - CR_{Inhibitor}}{CR_{Blank}} \right] \times 100 \quad (3)$$

Where CR_{Blank} is the corrosion rate in the medium without plant extract while $CR_{Inhibitor}$ is the corrosion rate in the medium with plant extracts.

Thermodynamic analysis

Arrhenius equation was used to study the temperature effect on the corrosion process;

$$K = Ae^{-E_a/RT} \quad (4)$$

Where the rate constant is K, E_a is the activation energy, A is the pre-exponential Factor, R is the universal gas constant and absolute temperature as T. Mathematically, Rate Law states that;

$$\text{rate} = \frac{-d(I)}{d(t)} = K(I)^n \quad (5)$$

The Inhibitor is given as I, the concentration of the inhibitor in mol per dm^3 as (I), k is the rate constant, t is the reaction time and the order of the reaction as n. Assumptions of the pseudo zero order condition was used in the corrosion inhibition studies. Hence,

$$\text{rate} = \frac{-d(I)}{d(t)} = K(I)^0 \quad (6)$$

equation 5 becomes

$$\text{rate} = \frac{-d(I)}{d(t)} = K \quad (7)$$

Therefore, using the condensed Arrhenius equation, 8, in calculating the activation energy (E_a), the rate constant, K

is replaced with the corrosion rate CR

$$E_a = 2.303R \left[\log \left(\frac{CR_2}{CR_1} \right) \times \left(\frac{T_1 T_2}{T_2 - T_1} \right) \right] \quad (8)$$

The corrosion at temperature T_2 and T_1 are represented by CR_2 and CR_1 respectively. The Heat of Adsorptions (Q_{ads}) of the leaves extract were calculated using equation 9

$$E_a = 2.303R \left[\log \left(\frac{\theta_2}{1-\theta_2} \right) - \log \left(\frac{\theta_1}{1-\theta_1} \right) \times \left(\frac{T_1 T_2}{T_2 - T_1} \right) \right] \quad (9)$$

Given that θ_1 and θ_2 are the respective degree of surface coverage values at temperatures T_1 and T_2 . Since the reaction was done at constant pressure, the Heat of Adsorption (Q_{ads}) values were approximately equated to the change in enthalpy of adsorption (ΔH_{ads}).

Electrochemical impedance spectroscopic (EIS) analysis

EIS analysis was done in conventional three electrode cell with a Gamry Reference 3000 advanced electrochemical workstation. The reference electrode used was saturated calomel electrode (SCE). The counter electrode used was platinum electrode and the working electrode was metal (copper) with an exposed surface area of 1 cm². The electrolytes were the test solutions (1 M NaOH) in the presence and absence of different inhibitor concentration. Before the tests, to get a steady state for accurate reading, at open circuit potential (OCP), the copper electrode was immersed in the test solution for 1 hr. Measurements of Electrochemical Impedance Spectroscopy (EIS) were carried out over a broad range of frequency from 100 kHz to 10 mHz, with a 10 mV amplitude sinusoidal voltage. The Nyquist plots that provided charge transfer resistance values were gotten from the diameter of the semicircles and the corrosion inhibition efficiency (η_{EIS}) was calculated using equation 10:

$$\eta_{EIS}(\%) = \frac{R_{ct(inh)} - R_{ct}}{R_{ct(inh)}} \times 100 \quad (10)$$

where R_{ct} is the charge transfer resistance without inhibitor and $R_{ct(inh)}$ is the charge transfer resistance with inhibitor.

Potentiodynamic polarization (PDP)

The potentiodynamic polarization (PDP) measurements were done in the potential range of +250 to -250 mV in an open circuit potential (OCP) at 1 mVs⁻¹ scan rate.

To confirm validity of full potentiodynamic polarization scan, we conducted separate cathodic and anodic polarization experiments of all samples, starting from the OCP with a new solution and a new specimen. Electrochemical parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}), cathodic and anodic Tafel slope (β_c and β_a) were deduced by the Tafel extrapolation technique. The corrosion inhibition efficiency (η_{PDP}) was obtained using equation 11:

$$\eta_{PDP}(\%) = \frac{i_{corr}^0 - i_{corr}}{i_{corr}^0} \times 100 \quad (11)$$

Where i_{corr}^0 is the corrosion current density without inhibitor and i_{corr} is the corrosion current density with inhibitor. Gamry Echem Analyst software was used to analyze data obtained from EIS and PDP curves. Each electrochemical measurement was repeated at least three times using the same experimental conditions to guarantee a satisfactory reproducibility.

Surface analysis

Scanning Electron Microscopy (SEM) technique was done to investigate the adsorption of *Urena lobata* extract on the copper specimens. The specimens were introduced into 1 M NaOH without and with *Urena lobata* extract for six hours and SEM of copper specimens were taken.

RESULTS AND DISCUSSION

The phytochemical screening results of the leaves extract show the presence of alkaloids, saponins, flavonoids, tannins, and cynagenic glycosides in the plant sample as seen in (Table 1). These phytochemicals found possess lone pair of electrons so they can easily donate electrons to the metal surface thereby reducing the rate at which the metal losses electrons. The results in Table 2 show that the weight loss increased with increase in time of immersion for different extract concentrations. This is attributed to the protective ability of the extract on the metal surface. The active molecules in the media interact with the Meta, get adsorbed on it and shield it from corrosive attack. Similar observation has been made by earlier researchers (Enyinnaya et al., 2021; Shukla and Ebenso, 2011). From the results presented in (Table 3), the weight loss increased with increase in temperature and time for different extract concentrations. There was an increase in protection of the coupons as the concentration of extract increased, indicating that the extract's active molecules shielded the metal from corrosive attack. This is similar to the observations earlier

Table 1: Some phytochemical components of *Urena lobata* leaves.

Chemical Constituents	Composition mg/ml
Alkaloids	0.11±0.01
Flavonoids	1.30±0.03
Tannins	7.93±0.01
Saponins	0.29±0.01
Cynagenic Glycosides	0.33±0.01

Table 2: Copper weight loss in basic medium at ambient temperature and different time intervals.

Extract concentration (g/L)	Weight loss (g)			
	24hr	72hr	120hr	168hr
Blank	0.010	0.048	0.293	0.303
0.5	0.009	0.019	0.281	0.293
1.0	0.008	0.017	0.272	0.287
1.5	0.006	0.015	0.268	0.281
2.0	0.005	0.014	0.257	0.280
2.5	0.004	0.012	0.143	0.277

Table 3: Copper weight loss in basic medium at varying temperatures and time.

Extract conc. (g/L)	Weight loss (g)											
	303K			313K			323K			333K		
	2hrs	4hrs	6hrs	2hrs	4hrs	6hrs	2hrs	4hrs	6hrs	2hrs	4hrs	6hrs
Blank	0.005	0.009	0.010	0.010	0.647	0.882	0.012	0.747	0.899	0.019	0.771	0.906
0.5	0.004	0.005	0.009	0.008	0.621	0.836	0.010	0.683	0.870	0.014	0.741	0.891
1.0	0.003	0.004	0.007	0.005	0.604	0.784	0.007	0.651	0.864	0.009	0.712	0.870
1.5	0.002	0.003	0.006	0.003	0.590	0.749	0.006	0.602	0.823	0.008	0.691	0.857
2.0	0.001	0.002	0.005	0.002	0.589	0.717	0.004	0.592	0.801	0.006	0.651	0.818
2.5	0.000	0.001	0.003	0.001	0.511	0.702	0.003	0.571	0.791	0.004	0.612	0.791

Table 4: Copper weight loss in varying concentrations of basic medium at ambient temperature for 24 hrs.

Extract concentration (g/L)	Weight loss (g)			
	0.5M	1M	2M	3M
Blank	0.236	0.010	0.011	0.008
0.5	0.233	0.009	0.009	0.005
1.0	0.223	0.008	0.004	0.004
1.5	0.217	0.006	0.003	0.002
2.0	0.216	0.005	0.001	0.000
2.5	0.175	0.004	0.000	0.000

reported (Odusote and Ajayi, 2013). Results presented in (Table 4) shows the weight loss of copper immersed in 0.5M, 1M, 2M, and 3M solutions of NaOH for 24hrs. The results reveal that weight loss decreased with increase in extract concentration and concentration of the media. This may be due to increase in phytochemical components with concentration. Similar observation has been reported (Odidika, 2020). The chart in (Figure 1) shows that there was little or no difference between the corrosion rate of the samples with and without extract concentration at 303K. The chart also showed that corrosion rate increased significantly at 313 K, 323 K and 333 K. The maximum was observed at 4hrs duration. The

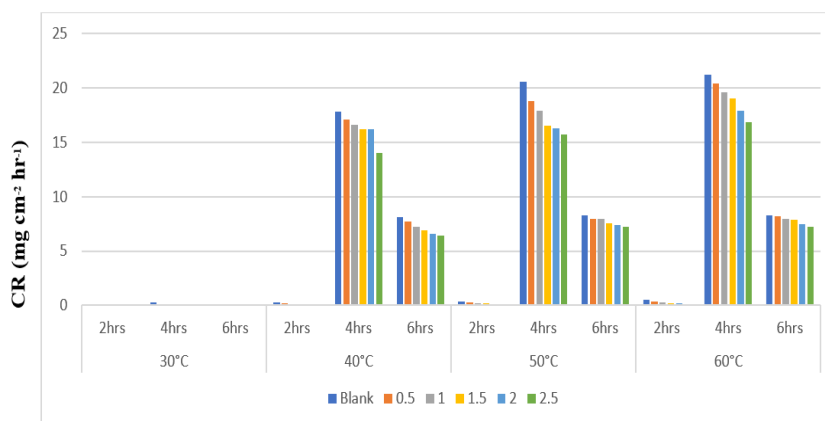
chart also revealed that there was a decrease in the corrosion rate with increase in the extract concentration which may be attributed to the protective ability of the leave extract. Similar findings have been reported (Odidika et al., 2020). The results in (Tables 5) shows that the surface coverages increased with increase in extract concentrations at the different temperatures. This implies that the metal surface gets more protected as the concentration of extract increases. Similar report has earlier been made (Loto et al., 2016). The result in (Table 6) reveals that the inhibition efficiency increased with increasing extract concentration at different temperatures.

Table 5: Surface Coverage of Cu in 1M NaOH solution at different temperatures.

Extract conc. (g/L)	Surface Coverage (θ)											
	303K			313K			323K			333K		
	2hrs	4hrs	6hrs	2hrs	4hrs	6hrs	2hrs	4hrs	6hrs	2hrs	4hrs	6hrs
Blank												
0.5	0.203	0.444	0.098	0.2	0.040	0.052	0.167	0.086	0.032	0.264	0.039	0.017
1.0	0.399	0.556	0.304	0.498	0.066	0.111	0.415	0.129	0.039	0.526	0.077	0.040
1.5	0.601	0.665	0.402	0.698	0.088	0.151	0.5	0.194	0.085	0.580	0.104	0.054
2.0	0.797	0.778	0.5	0.8	0.090	0.187	0.667	0.208	0.109	0.685	0.156	0.097
2.5	1	0.887	0.696	0.898	0.210	0.204	0.748	0.236	0.120	0.790	0.206	0.127

Table 6: Inhibition efficiency of the extract on copper in basic medium at varying temperatures.

Extract conc. (g/L)	Inhibition Efficiency (% IE)											
	303K			313K			323K			333K		
	2hrs	4hrs	6hrs	2hrs	4hrs	6hrs	2hrs	4hrs	6hrs	2hrs	4hrs	6hrs
Blank												
0.5	20.3	44.4	9.8	20	4	5.2	16.7	8.6	3.2	26.4	3.9	1.7
1.0	39.9	55.6	30.4	49.8	6.6	11.1	41.5	12.9	3.9	52.6	7.7	4
1.5	60.1	66.5	40.2	69.8	8.8	15.1	50	19.4	8.5	58	10.4	5.4
2.0	79.7	77.8	50	80	9	18.7	66.7	20.8	10.9	68.5	15.6	9.7
2.5	100	88.7	69.6	89.8	21	20.4	74.8	23.6	12	79	20.6	12.7

**Figure 1:** Corrosion Rate of Cu in 1 M NaOH solution at different temperatures**Table 7:** Thermodynamic Parameters for Cu Coupons in 1M NaOH Solutions at 30°C.

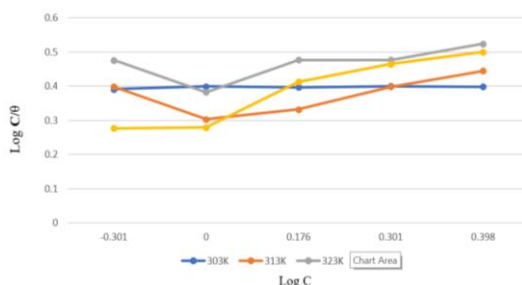
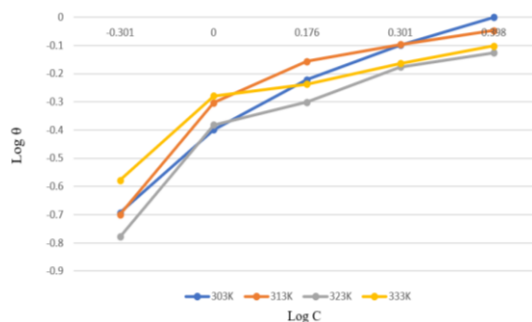
Extract concentration (g/L)	E _a (kJ/mol) ΔH _{ads} (kJ/mol)	
	E _a (kJ/mol)	ΔH _{ads} (kJ/mol)
Blank	54.3	-
0.5	54.6	-1.5
1.0	40.1	31.8
1.5	32.4	33.8
2.0	53.2	1.5
2.5	0	0

This is attributed to the increase in protection of the metal as the extract concentration increases, which may be due to the increase in organic molecules in the extract. Such observation has earlier been made (Loto et al., 2016). The maximum inhibition efficiency was 100% at 303K in

2.5g/L extract concentration. From (Table 7) the enthalpy of adsorption (ΔH_{ads}) for the leaves extract suggested physical adsorption of the inhibitor on the metal surface since most of the values are less than 80 kJmol⁻¹. Table 8 shows the results of the parameters used in plotting the

Table 8: Extract's Surface Adsorption Values for Langmuir and Freundlich Adsorption Isotherm.

	Log C	Log (θ)				Log C/ θ			
		303K	313K	323K	333K	303K	313K	323K	333K
1 M NaOH	Blank								
	-0.301	-0.693	-0.699	-0.778	-0.578	0.391	0.398	0.476	0.277
	0	-0.399	-0.303	-0.382	-0.279	0.399	0.303	0.382	0.279
	0.176	-0.221	-0.156	-0.301	-0.237	0.397	0.332	0.477	0.413
	0.301	-0.099	-0.097	-0.176	-0.164	0.400	0.398	0.477	0.465
	0.398	0	-0.047	-0.126	-0.102	0.398	0.445	0.524	0.500

**Figure 2:** Langmuir Plots for *Urena lobata* at Different Temperatures in 1 M NaOH on Surface of Copper**Figure 3:** Freundlich Plots for *Urena lobata* at different temperatures in 1 M NaOH on Surface of Copper

Langmuir and Freundlich Charts. The Langmuir plots in (Figure 2) were done using $\text{Log } C/\theta$ against $\text{Log } C$, while the Freundlich plots in Figure 3 was done using $\text{Log } \theta$ against $\text{Log } C$. It was observed that the Langmuir plots fitted better when compared to that of Freundlich and there was a monolayer adsorption on the surface of the metal.

Electrochemical impedance spectroscopy

Figure 4 presents the result of the electrochemical analysis of the leaves extracts of *Urena lobata* on copper in 1 M NaOH solution. From the plots of the imaginary impedance ($-Z_{\text{img}}$) against the real impedance (Z_{real}); it is observed that there is a formation of a semicircle at the real impedance, beginning from zero (0) (the origin of the

plots; region of high impedance to about 450 Ω (region of low impedance). The beginning of the semicircle measures the solution resistance while the end of the semicircle measures the charge transfer resistance as shown in Table 9. From the chart, the diameter of the semicircle increased as the concentration of the extract increased. This may be due to the adsorption of the active components present in the extract on the surface of the copper, thereby causing a reduction in the rate at which electrons move from the metal; thereby increasing the impedance. The blank shows the least diameter of the semicircle which is attributed to the absence of inhibitor in it, which would have reduced the flow of electrons from the metal. Table 9 shows 91.7% maximum inhibition efficiency in the 2.5g/L extract concentration. Similar observation has been reported (Lebrini et al., 2010).

Table 9: Copper EIS Values in the Presence and Absence of Varying *Urena lobata* Leaves Extract (ULLE) Concentrations in NaOH Solution.

Conc. (g/L)	R_s (Ω cm ²)	R_{ct} (Ω cm ²)	C_{dl} (μ F cm ⁻²)	N	η_{EIS} (%)
Blank	0.928	140.2	68.08	0.864	-
1.0	0.973	466.5	35.25	0.875	69.9
1.5	0.966	549.9	21.90	0.879	74.5
2.0	1.101	918.6	14.83	0.884	84.7
2.5	1.114	1697.1	6.77	0.887	91.7

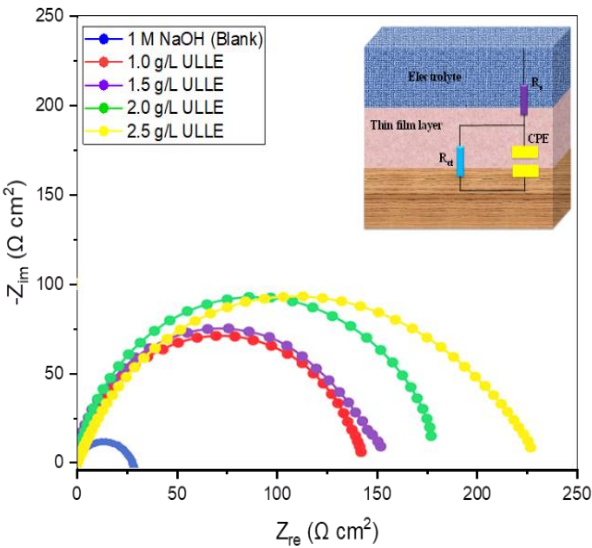


Figure 4: Nyquist Plot of Copper in 1 M NaOH with and without different concentrations of ULLE (insert: equivalent circuit).

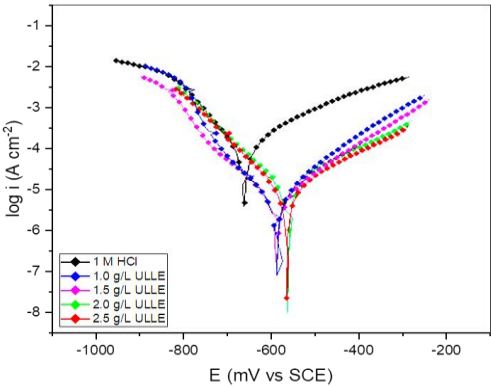


Figure 5: Polarization Curves of Copper in 1 M NaOH Solution with and without Various Concentrations of ULLE

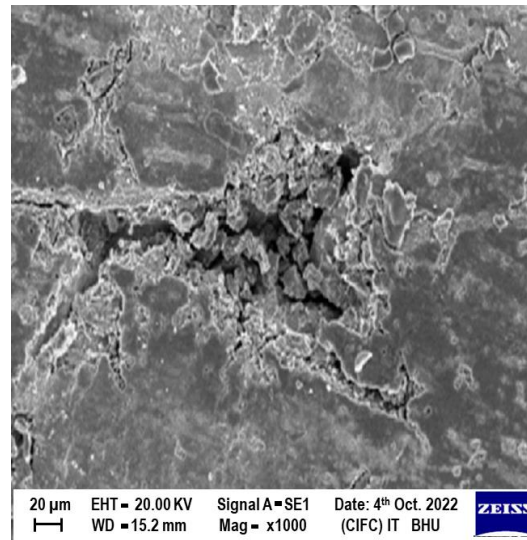
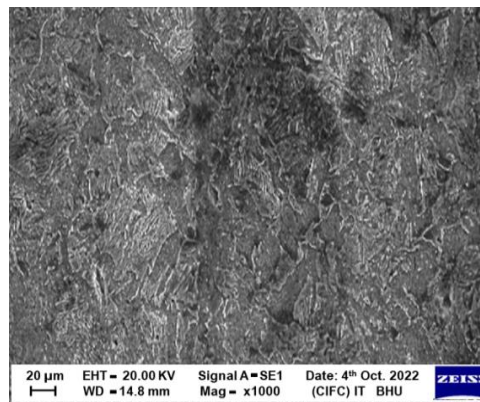
Potentiodynamic polarization

Figure 5 displays the graph of the potentiodynamic polarization of *Urena lobata* leaves extract on copper (Corrosion potential, E_{corr} (V/SCE) plotted against $\log i_{corr}$ (μ A cm⁻²)). During the potentiodynamic polarization

measurements, the potential of the working electrode was varied while observing the corrosion current (i_{corr}). The plots show the reactions at the anode and the cathode. The right hand side is the anodic branch while the left hand side is the cathodic branch. The topmost branch shows the blank (without inhibitor).

Table 10: Polarization Curve Values for Copper in Basic Medium with and without Different Concentrations of ULLE.

Conc. (g/L)	i_{corr} ($\mu\text{A cm}^{-2}$)	E_{corr} (mV/SCE)	β_a (mV dec ⁻¹)	β_c (mV dec ⁻¹)	η_{PDP} (%)
Blank	1052.8	-681.5	151.4	188.2	-
1.0	397.4	-599.7	135.8	182.0	61.9
1.5	200.7	-601.2	131.6	175.6	80.9
2.0	135.8	-574.9	128.3	181.7	87.1
2.5	85.2	-575.1	125.2	178.8	91.9

**Figure 5:** Scanning Electron Micrographs of Copper Sample after 6 h Immersion in 1 M NaOH (blank).**Figure 6:** Scanning Electron Micrographs of Copper Sample after 6 h Immersion in 1 M NaOH in 2.5 g/L ULLE.

From the plots, it was observed that as the concentration of the plant extract increased, the anodic and cathodic branch decreased gradually to a lower current density. The effect was more on the anodic branch. The inhibitor is a mixed type inhibitor since its effect was felt on both the cathodic and anodic branch. Such has also been observed earlier (Saxena et al., 2017).

Table 10 presents the corrosion current (i_{corr}), corrosion potential (E_{corr}), cathodic slope β_c (mV dec⁻¹), anodic slope β_a (mV dec⁻¹), and η_{PDP} (%). The anodic slope (β_a) values decreased more than the cathodic slope (β_c) values, suggesting that the inhibition affected the anodic branch more, thereby preventing it from corrosive attack.

The presence of the *Urena lobata* leaves extract decreased the corrosion current (I_{corr}) from 902.5 to 46.8 ($\mu\text{A cm}^{-2}$). This observation suggests that the presence of the extract in the test solution retarded the anodic reaction (loss of electrons) of the copper. The percentage inhibition efficiency obtained is directly proportional to the extract concentration.

Scanning electron microscopy

The copper surface morphology in 1 M NaOH solution without plant extract presented in (Figures 5 and 6) shows that the surface was badly damaged. This may be due to attack of the corrosion media on the metal. However, the micrograph of copper sample in 1 M NaOH with 2.5g/L of the extract (Figure 6) shows an improvement in the roughness of the surface, suggesting possible adsorption of the plant extract on the surface of the copper; thereby reducing loss of electrons (corrosion attack) which causes roughness metal surfaces.

Conclusion

Urena lobata leaves extract inhibited corrosion of copper in 1 M NaOH solution. The inhibition efficiency increased with increase in concentration of extract. The protection was more at lower temperature. Data from the gravimetric analysis gave best fit in the Langmuir adsorption isotherm. The enthalpy of adsorption (ΔH_{ads}) for the leaves extract was less than 80 kJmol⁻¹, suggesting physical adsorption of the extract on the metal surface. Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic Polarization (PDP) results confirmed the corrosion inhibitory potential of the plant extract with maximum inhibition efficiency of 91.9% in 2.5 g/L extract solution. Scanning Electron Microscopy (SEM) also confirmed the inhibitory action of the leaves extract.

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