

Research Article

Experimental Study on Adsorption and Corrosion Inhibition Properties of *Urena lobata* Leaves Extract on Mild Steel in Acidic Medium

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Abstract

The corrosion inhibitive effect of *Urena lobata* leaves extract on mild steel in 1M HCl solution was investigated via gravimetric, Electrochemical and Surface analyses. Adsorption isotherms were investigated using the Langmuir and Freundlich plots. Phytochemicals of the extract were investigated using Gas Chromatography-Mass Spectrometric (GC-MS) technique. The effect of the leaves extract through immersion time, temperature and concentration on the corrosion inhibition were obtained. The results obtained showed that the increase in extract's concentration increased the inhibition efficiency and the protection was more on the surface of the metal at lower temperature. Maximum inhibition efficiency of 95.8% was obtained at 2.5g/L extract concentration. Corrosion rate of mild steel increased with temperature increase in the acidic 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. Hence indicating that the plant extract has mixed properties. The electrochemical results confirmed the inhibitory ability of the plant extract. The inhibition efficiencies (% IE) were attributed to the phytochemical components (alkaloids, saponins, flavonoids, tannins, cynagenic glycosides) present in the extract. The extracts of *Urena lobata* are effective corrosion inhibitors of mild steel.

Keywords

Urena Lobata, Inhibition Efficiency, Corrosion Inhibitors, Corrosion Rate, Mild Steel

1. Introduction

Globally, acid solutions are employed in industries to get rid of rust and scales from metal equipment surfaces like water tanks and heat exchangers. It is eminent that corrosion of metal-based materials easily occurs in acidic media. Corrosion is referred to as a process in which a refined metal is converted naturally to a form that is stable such as its oxide, sulphide or hydroxide state that enhances deterioration of the

material [8, 11].

Corroded products act as poisonous contaminants in food, drugs, paints, dyes and other chemical production industries [14]. Addition of inhibitors to the metal surface in solution is one of the methods of preventing/reducing corrosion of metals and these inhibitors possess suitable functional groups, long alkyl chain, aromatic rings, multiple bonds and presence of

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heteroatoms such as oxygen, sulphur, phosphorus and nitrogen which make up their inhibitive ability [10].

Owing to rising environmental rules and ecological alertness for an environmentally friendly and sustainable environments, more consideration is geared towards developing nontoxic, biodegradable, environmentally friendly replacements for organic and inorganic corrosion inhibitors, used in the past [3] in which medicinal herbs, plants, fruits and vegetable peel extracts are the major [1].

Recently, plant extracts are being used as metals/alloys corrosion inhibitors and the inhibitive effect of most plant extracts is traced to the presence of some organic compounds such as saponins, alkaloid, steroids, tannin, glycosides and amino acids [6, 9], which inhibit corrosion because it gets adsorbed on the surface of the metal thereby protecting the metal from losing electrons. This has lately become the focus of thorough examination in the field of corrosion research [2].

The Green inhibitor used is the *Urena lobata*, commonly referred to as Caesarweed or Congo jute, is a tender perennial, erect, variable, ascendant shrub or subshrub with height of about 0.5 m to 2.5 m. It is a weed commonly found on waterways, open woodlands, waste areas, roadsides and disturbed locations in tropical and sub-tropical regions [17]. This plant extract is being considered since it has phytochemicals with the potential of inhibiting corrosion like other green inhibitors.

The inhibitive effect of *Urena lobata* leaves extract as a corrosion inhibitor on mild steel in 1 M HCl solutions is being evaluated by determining the phytochemical components of the extract and the corrosion inhibitory ability and thermodynamic parameters of the extract by gravimetric analysis, Adsorption Isotherms, Scanning Electron Microscope (SEM), Potentiodynamic Polarization (PDP) and Electrochemical Impedance Spectroscopy (EIS).

2. Materials and Method

2.1. Collection and Preparation of Coupons

The mild steel sheet used for this research were gotten from mechanical engineering workshop, Rivers State University, Port Harcourt, Rivers State, Nigeria. The mild steel had a thickness of 0.9 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.

2.2. Collection and Preparation of Plant Extracts

The leaves of *Urena lobata* were collected from Nwinee-nae's garden, Zaami street, Bori-Ogoni, Rivers State, Nigeria. Ethanol was used to extract from the plant powder via the

batch extraction method. leaving the plant extract paste. 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.

2.3. Gravimetric Analysis (Weight Loss Method)

The corrosion study was carried out by determination of weight loss of the metals at different temperatures, acid concentrations and different time intervals.

The corrosion rates (CR), degree of surface coverage (θ), and inhibition efficiencies (% IE) 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 without plant extract while $CR_{inhibitor}$ is the corrosion rate with plant extracts.

2.4. Thermodynamic Analysis

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

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

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 the 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.

Though, Assumptions of the pseudo zero order condition corrosion was used in the inhibition studies. Hence,

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

equation (6) 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

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

The corrosion at temperature T_2 and T_1 are represented by CR_2 and CR_1 respectively and the universal gas constant as R.

The Heat of Adsorption (Q_{ads}) of the leaves extract were calculated using equation (9).

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

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}).

2.5. Electrochemical Impedance Spectroscopy (EIS)

The electrochemical 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 (mild steel) with an exposed surface area of 1 cm^2 . The electrolytes were the test solutions (1 M HCl) 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 mild steel 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

applying 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.

2.6. 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.

2.7. Surface Analysis

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

3. Results

Table 1. 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

Chemical Constituents	Composition mg/ml
Cynagenic Glycosides	0.33±0.01

Table 2. Thermodynamic Parameters for Mild Steel Coupons in 1 M HCl Solutions at 30 °C.

Plant sample	Extract concentration (g/L)	E_a (kJ/mol)	ΔH_{ads} (kJ/mol)
<i>Urena lobata</i>	Blank	80.9	
	0.5	64.2	45
	1.0	61	48.1
	1.5	54.7	56.8
	2.0	47.3	69.7
	2.5	37	68.8

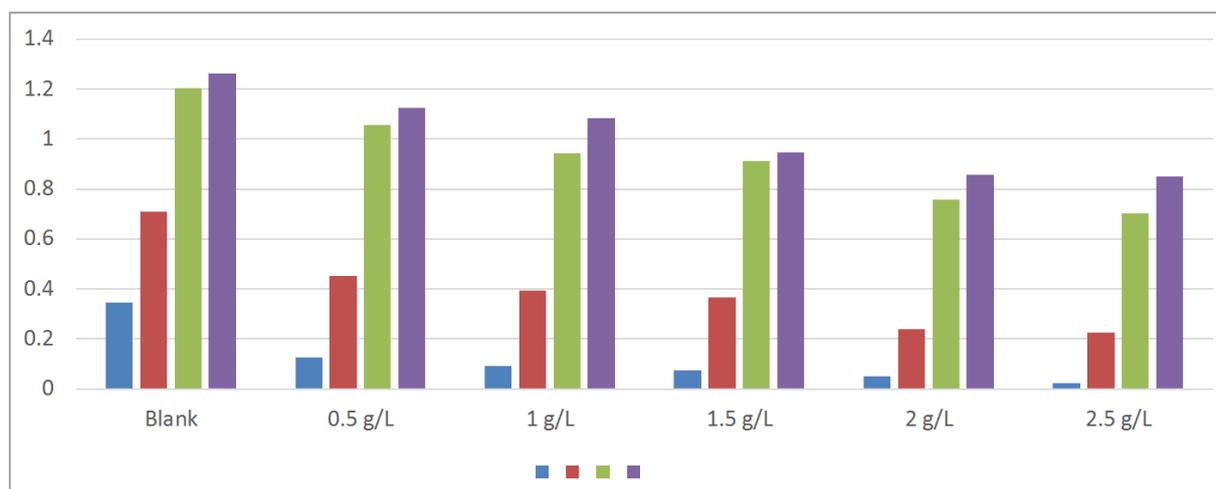


Figure 1. Mild Steel Weight Loss in Acidic Media at Ambient Temperature and Different Time Intervals.

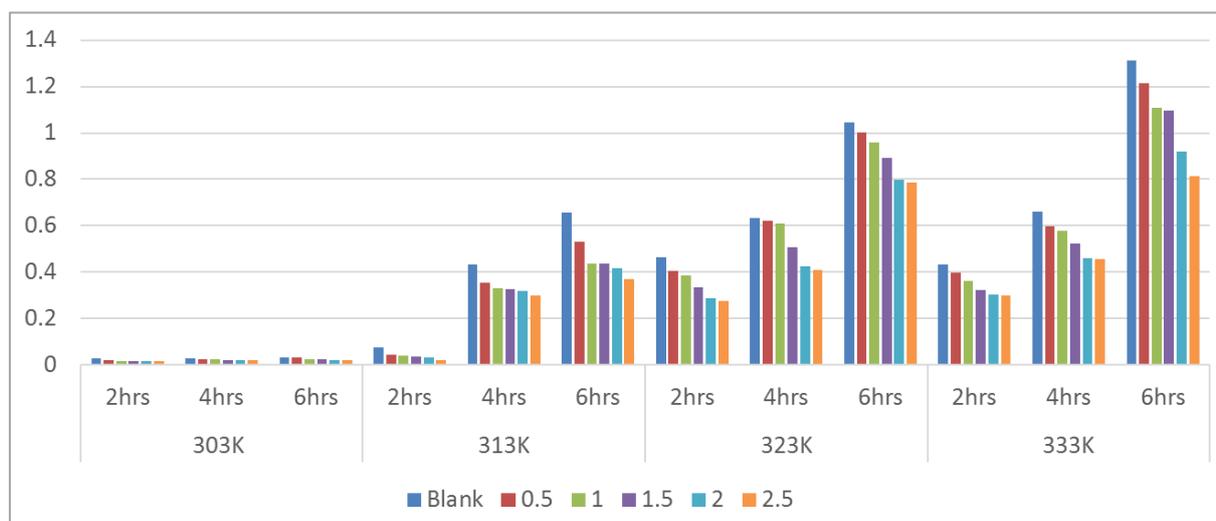


Figure 2. Mild Steel Weight Loss in Acidic Medium at Varying Temperatures and varying time.

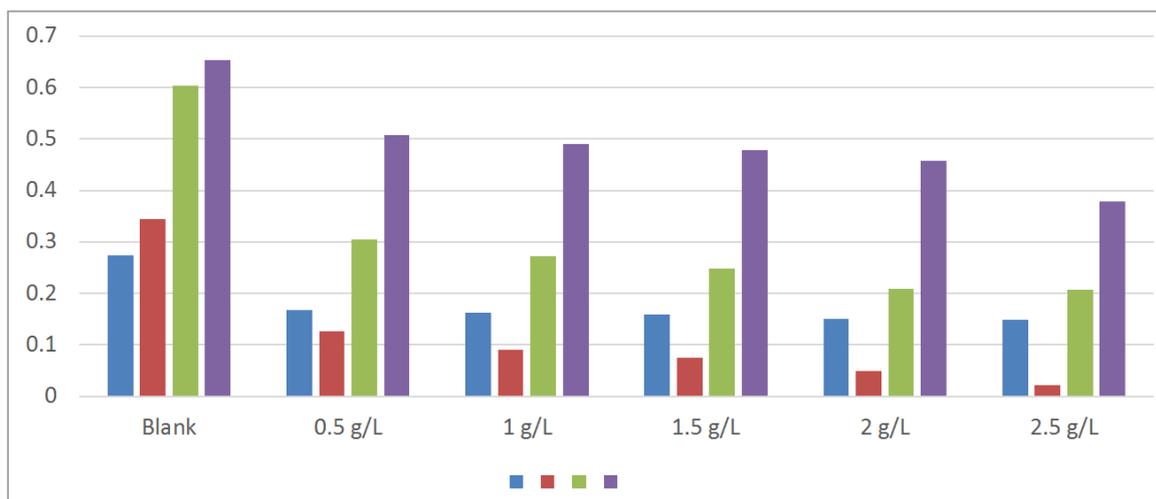


Figure 3. Mild Steel Weight Loss in Varying Concentrations of Acidic Medium at Ambient Temperature for 24 hrs.

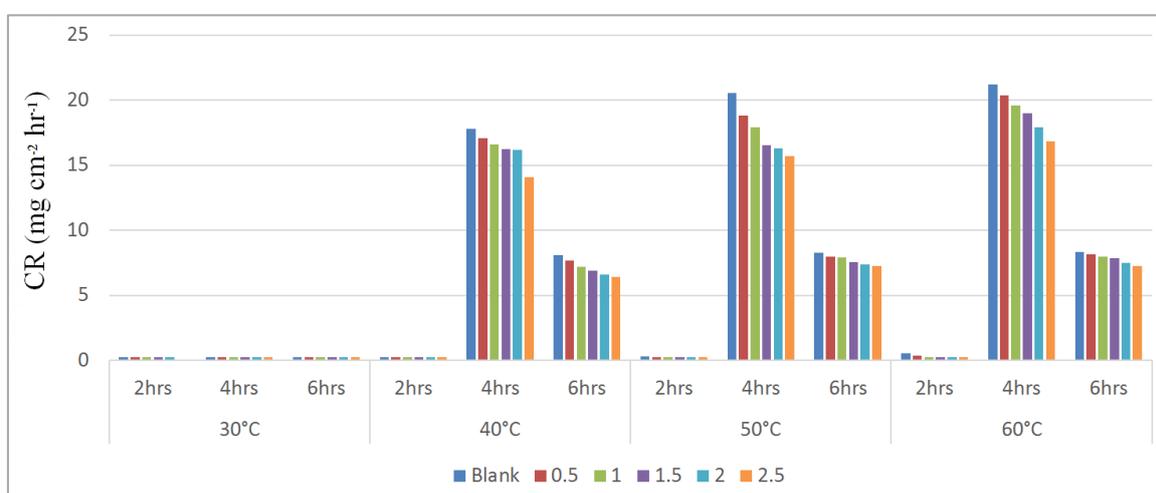


Figure 4. Corrosion Rate of Mild Steel in 1 M HCl Solution at Different Temperatures.

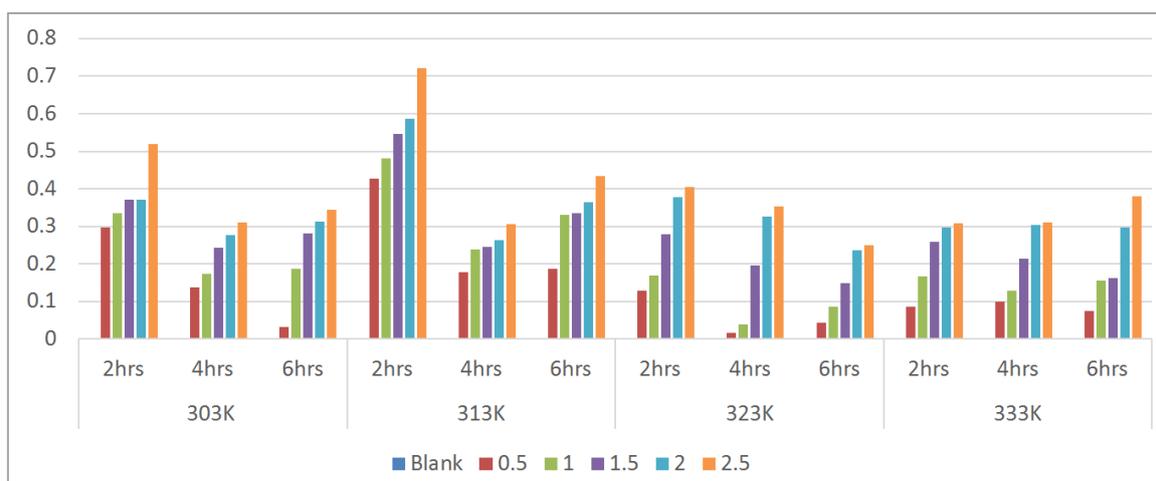


Figure 5. Surface Coverage of Mild Steel in 1 M HCl Solution at Different Temperatures.

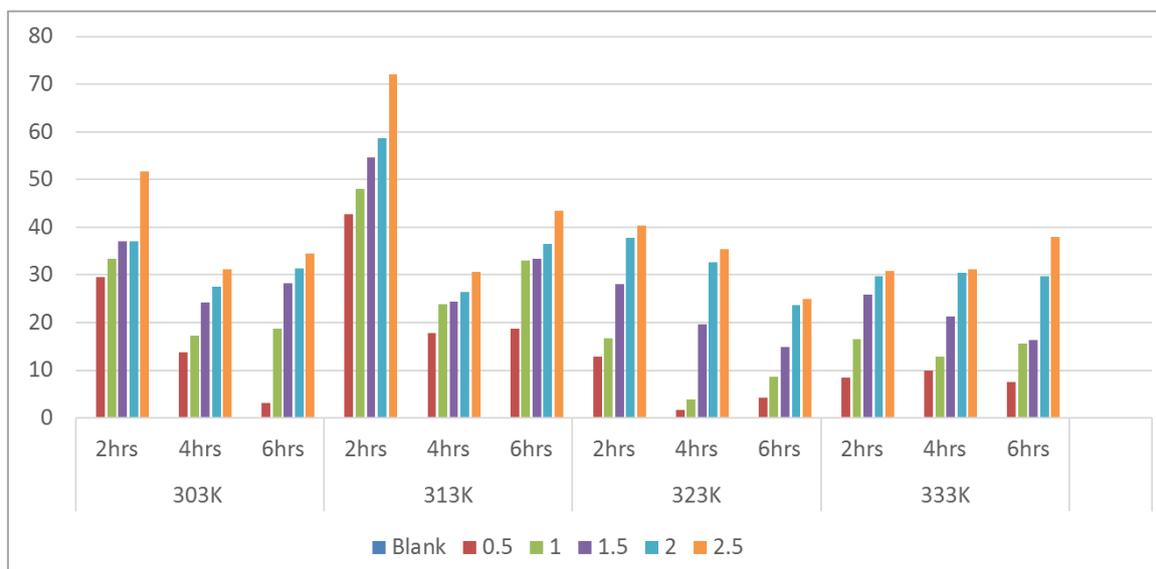


Figure 6. Extract's Inhibition Efficiency on Mild Steel in Acidic Medium at Varying Temperatures.

Table 3. Mild Steel 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
-0.301	-0.529	-0.370	-0.889	-1.071	0.228	0.069	0.588	0.770
0	-0.477	-0.319	-0.775	-0.780	0.476	0.319	0.775	0.780
0.176	-0.432	-0.262	-0.553	-0.587	0.608	0.438	0.729	0.763
0.301	-0.432	-0.231	-0.423	-0.526	0.733	0.532	0.724	0.827
0.398	-0.286	-0.143	-0.394	-0.510	0.684	0.541	0.792	0.908

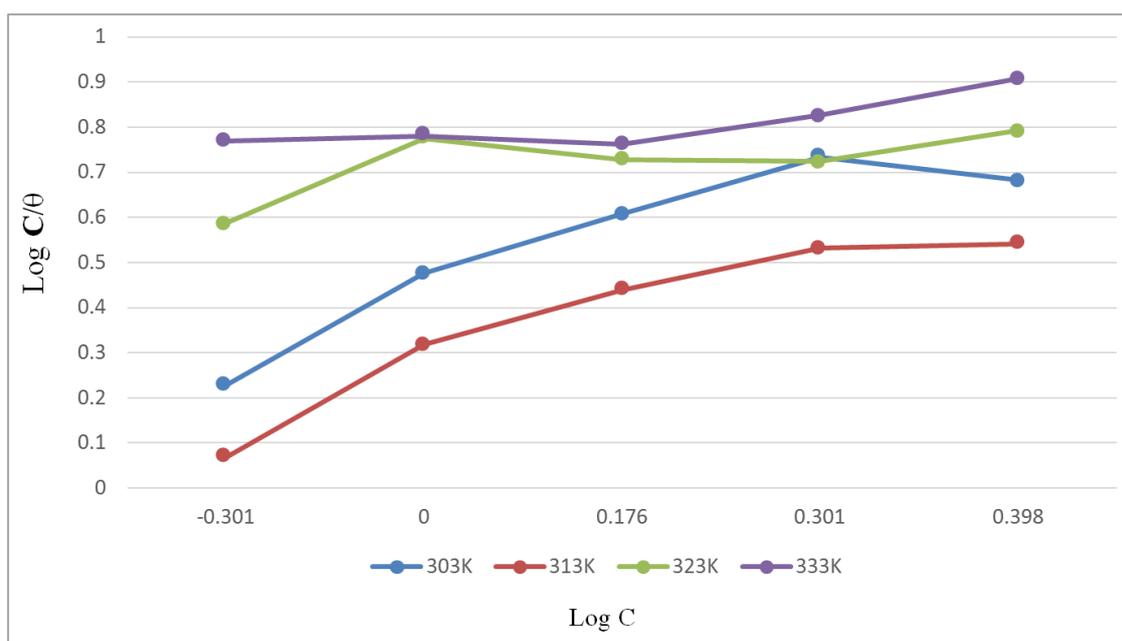


Figure 7. Langmuir plots for Urena lobata at Different Temperatures in 1 M HCl on Surface of Mild Steel.

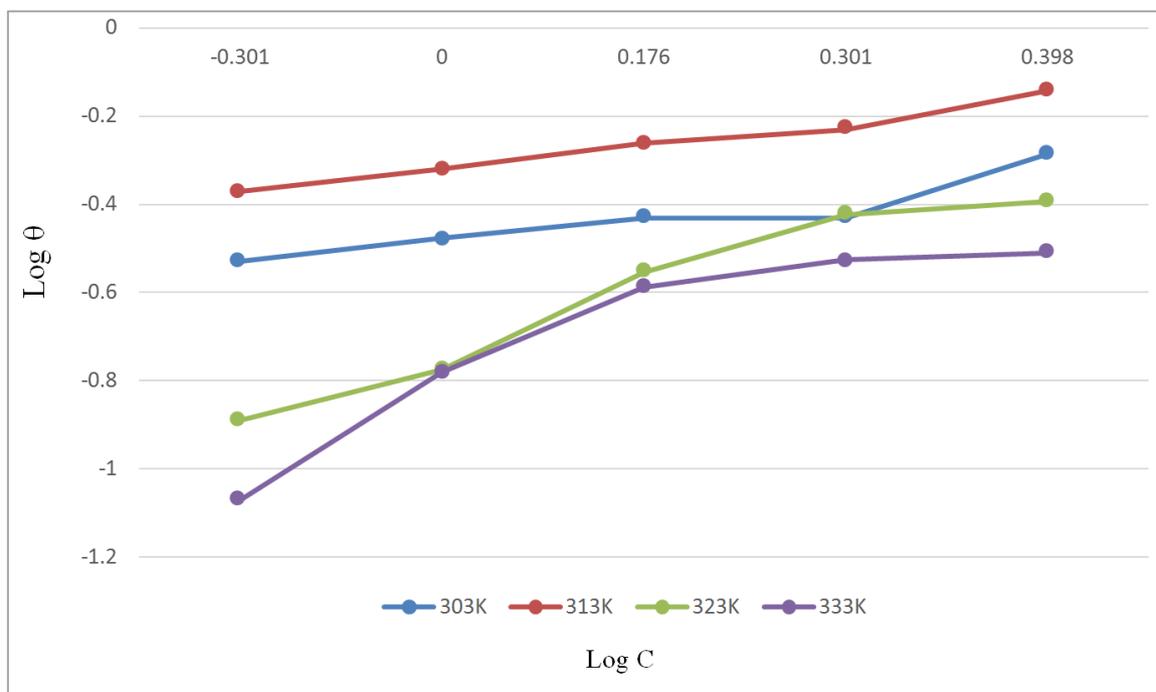


Figure 8. Freundlich Plots for *Urena lobata* at Different Temperatures in 1 M HCl on Surface of Mild Steel.

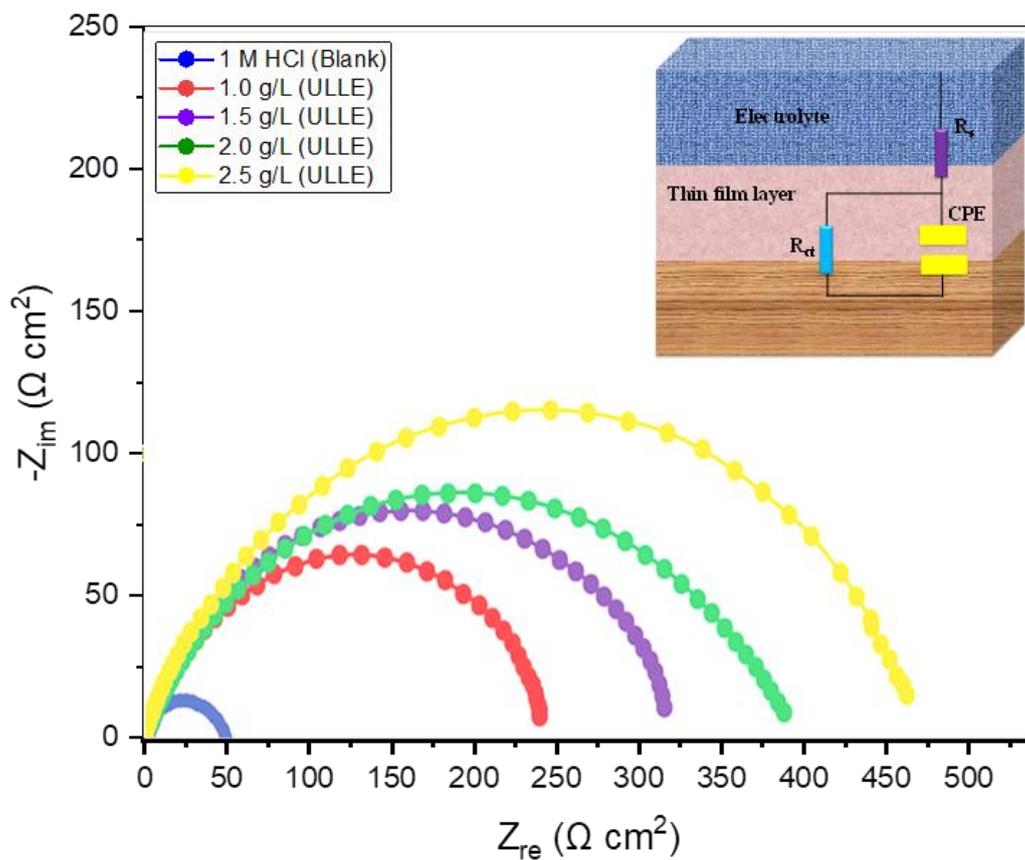
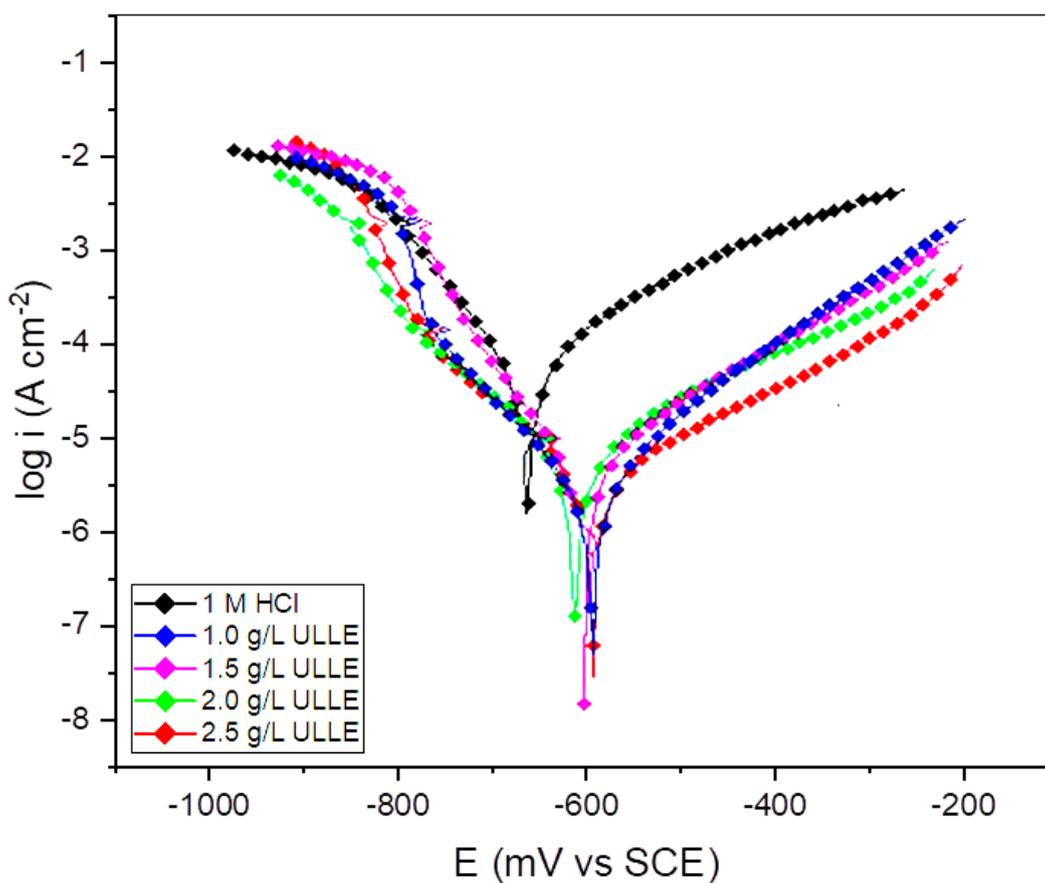


Figure 9. Nyquist Plot of Mild Steel in 1 M HCl without and with Different Concentrations of ULLE (insert: equivalent circuit).

Table 4. Mild Steel EIS Values in the Presence and Absence of Varying *Urena lobata* Leaves Extract (ULLE) Concentrations in Acidic Medium.

Conc. (g/L)	R_s ($\Omega \text{ cm}^2$)	R_{ct} ($\Omega \text{ cm}^2$)	C_{dl} ($\mu\text{F cm}^{-2}$)	N	η_{EIS} (%)
Blank	0.875	147.0	59.46	0.890	-
1.0	0.891	517.4	31.7	0.892	71.6
1.5	0.936	726.9	19.5	0.897	79.8
2.0	0.912	1317.3	10.4	0.901	88.8
2.5	0.947	2568.6	2.03	0.907	94.3

**Figure 10.** Polarization Curves of Mild Steel in 1 M HCl in the Absence and in the Presence of Different Concentrations of ULLE.**Table 5.** Polarization Curve Values for Mild Steel in Acidic 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	874.0	-687.4	148.2	196.1	-
1.0	258.3	-590.3	127.5	184.0	70.4
1.5	131.4	-600.1	121.7	183.6	85.0
2.0	92.1	-618.4	119.3	165.4	89.5
2.5	36.8	-591.5	115.3	181.6	95.8

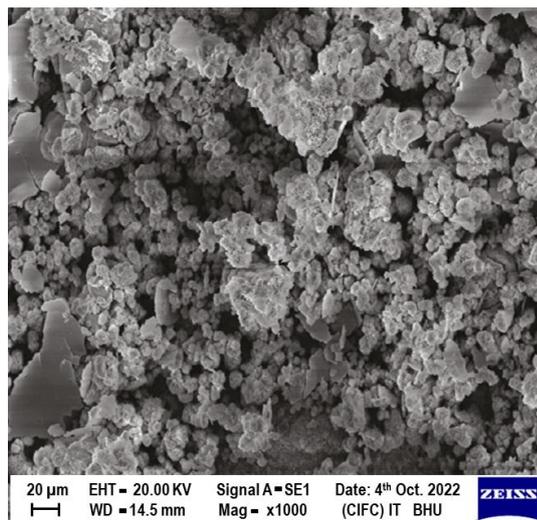


Figure 11. Scanning Electron Micrographs of Mild Steel Sample after 6 h Immersion in 1 M HCl (blank).

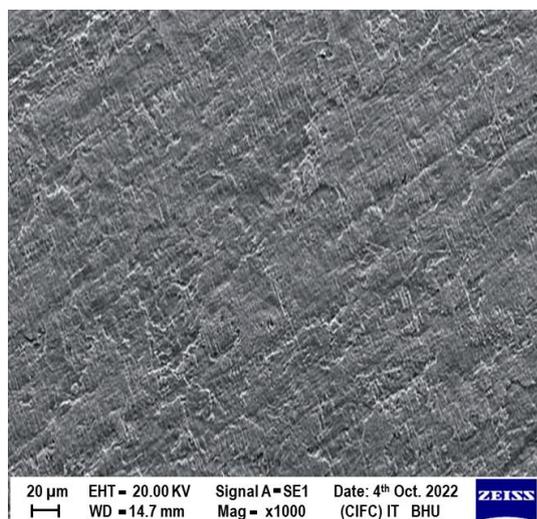


Figure 12. Scanning Electron Micrographs of Mild Steel Sample after 6 h Immersion in 1 M HCl in the presence of 2.5 g/L ULLE.

4. Discussions

The phytochemical results presented in Table 1 shows the presence of alkaloids, saponins, flavonoids, tannins, and cyanogenic glycosides in the plant sample. These phytochemicals may be responsible for the corrosion inhibitory ability of the *Urena lobata* plant extract as seen from the results obtained from this study since the phytochemicals found has can easily donate electrons (i.e they possess lone pair of electrons) to the metal surface thereby forming a protective covering that reduces the rate at which the metal losses electrons. Similar report was made by Enyinnaya *et al* [4].

Figure 1 show the weight loss of mild steel immersed in 1 M HCl solution for 24 hrs, 72 hrs, 120 hrs and 168 hrs. The results show that the weight loss increased with increase in

time of immersion for different extract concentrations (blank, 0.5 g/L, 1.0 g/L, 1.5 g/L, 2.0 g/L and 2.5 g/L). This is attributed to the protective ability of the extract on the metal surface. This may be because there was more interaction between active molecules in the medium and the metal thereby increasing the adsorption level by forming a thin coating that shields it from corrosive attack and hence increasing its inhibitory ability compared to that of the acidic medium. Similar findings were made by Shukla & Ebenso [16].

The results in Figure 2 show the weight loss of mild steel immersed in 1 M HCl solutions at 303, 313, 323 and 333 K at different time intervals (2hrs, 4hrs, and 6hrs). From the results, the weight loss increased with increasing temperature and time for different extract concentrations (blank, 0.5 g/L, 1.0 g/L, 1.5 g/L, 2.0 g/L and 2.5 g/L). It was observed that there was an increase in protection of the coupons as the concentration of extract increased indicating that the extract's active molecules may have formed a thin coating that shielded it from corrosive attack which was similar with observations made by Odusote & Ajayi [13]. The results also reveal that adsorption reduce as temperature increases thereby indicating physisorption similar with findings by Odusote & Ajayi [13].

Figure 3 shows the weight loss of mild steel immersed in 0.5M, 1M, 2M, and 3M solutions of HCl for 24hrs. The results show that the weight loss increased with increase in extract concentration. The more the interaction between the compounds present in the extract the more the protection of the coupons, Odidika *et al* [12] reported similar observation.

Figure 4 show the corrosion rate of mild steel immersed in 1 M HCl solution at 303 K, 313 K, 323 K and 333 K at different time intervals (2 hrs, 4 hrs, and 6 hrs). The charts show that there was a slight difference between the corrosion rate of the samples with and without extract concentration. The chart also showed that the corrosion rate at 303K was very low and increased significantly at 313 K, 323 K, 333 K showing maximum corrosion rate at 4hrs duration. The chart also showed that there was a decrease in the corrosion rate with increase in the extract concentration. This may be attributed to the protective ability of the leave extract. Similar findings have been reported by Odidika *et al* [12].

Figure 5 present the results of the surface coverage of mild steel immersed in 1 M HCl at 303 K, 313 K, 323 K and 333 K at different time intervals (2 hrs, 4 hrs, and 6 hrs). The results show that the surface coverages increased with increase in extract concentrations at the different temperatures as also reported by Loto *et al* [9]. This means that the surface of the coupons gets more protected as the concentration of extract increases.

Figure 6 shows the inhibition efficiency of the extract on mild steel in 1M HCl solution at different temperatures. The result reveals that the inhibition efficiency increased with increasing extract concentration at different temperatures as also reported by Loto *et al* [9]. The maximum inhibition effi-

ciency was 72% at 313K in 2.5g/L extract concentration.

Generally, physisorption enthalpy is less than 80 kJmol^{-1} while that of chemisorption approaches 100 kJmol^{-1} . From Table 3 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} .

Figures 7 show the Langmuir plots of the plant extract at different temperatures in 1M HCl for mild steel. The Langmuir plots were drawn using $\text{Log } C/\theta$ against $\text{Log } C$. Figures 8 show the Freundlich plots of the plant extract at different temperature in 1M HCl for mild steel. The Freundlich plots were drawn using $\text{Log } \theta$ against $\text{Log } C$. After comparing the data obtained from the Langmuir and Freundlich plots for the plant extract, it was observed that the Langmuir plots fitted better. This suggests Physisorption and there was a monolayer adsorption on the surface of the metal. Similar report was made by Enyinnaya *et al* [5].

Figure 9 presents the results of the electrochemical analysis of the corrosion inhibitory ability of the leaf extracts of *Urena lobata* on mild steel in 1 M HCl 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 450Ω (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 4. From the chart, the diameter of the semicircle increased as the concentration of the extract increased. This may be because of the adsorption of the active components present in the extract on the surface of the mild steel thereby causing a reduction in the rate at which electrons move from the metal, hence increasing the impedance. The blank shows the least diameter of the semicircle which maybe because there is no inhibitor in it to reduce the flow of electrons from the metal. The Table 4 shows a 94.3% maximum inhibition efficiency in the 2.5g/L extract concentration. Similar observation was made by Lebrini *et al.* [7].

Figure 10 displays the graph of the potentiodynamic polarization of *Urena lobata* leaves extract on mild steel (Corrosion potential, E_{corr} (V/SCE) plotted against $\log i_{\text{corr}}$ ($\mu\text{A cm}^{-2}$)). 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 righthand side is the anodic branch while the lefthand side is the cathodic branch. The topmost branch shows the blank (without inhibitor). 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. Saxena *et al.* [15] made similar observation.

Table 5 presents the corrosion current (I_{corr}), corrosion po-

tential (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 is affecting the anodic branch more than the metal dissolution branch, thereby preventing it from corrosive attack. The presence of the *Urena lobata* leaves extract decreased the corrosion current (I_{corr}) from 874 to $36.8 (\mu\text{A cm}^{-2})$. This observation suggests that the presence of the extract in the acid test solution retarded the anodic reaction (loss of electrons) of the mild steel. The percentage inhibition efficiency obtained is directly proportional to the extract concentration.

The mild steel surface morphology in 1 M HCl solution without plant extract in Figures 11 show that the surface was badly damaged. This may be due to attack of the corrosion media on the metals. Figures 12 is the Scanning electron micrograph of mild steel sample in 1 M HCl with 2.5g/L of the extract. From the figure, there is an improvement in the roughness of the surface compared to that of the blank, suggesting possible adsorption of the plant extract on the surface of the mild steel; thereby reducing loss of electrons (corrosion attack) that causes the roughness of the surface.

5. Conclusion

From the results obtained from this study, *Urena lobata* leaves extract inhibited corrosion of mild steel in 1 M HCl 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} suggested physical adsorption.

Electrochemical Impedance Spectroscopy (EIS) and Potentiodynamic Polarization (PDP) results confirmed the corrosion inhibitory potential of the plant extract with maximum inhibition efficiencies obtained were 94.3% and 95.8% in 2.5 g/L extract solutions. Another confirmation for the inhibitory action of the leaves extract was obtained using the Scanning Electron Microscopy (SEM).

6. Recommendation

Urena lobata leaves extract should be used as a corrosion inhibitor as an alternative to the synthetic corrosion inhibitors.

Further research on isolation and investigation of the corrosion inhibitory ability of the predominant phytochemicals should be carried out.

Abbreviations

CR: corrosion rates

EIS: Electrochemical Impedance Spectroscopy

IE: inhibition Efficiency
OCP: open circuit potential
PDP: Potentiodynamic Polarization
SCE: Saturated calomel electrode
SEM: Scanning Electron Microscopy
ULLE: Urena lobata leaves extract

Conflicts of Interest

The authors declare no conflicts of interest.

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