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Azadirachta Indica Extract as corrosion Inhibitor for Copper in Nitric Acid Medium

Patel K.K. and Vashi R.T.*
Chemistry Department, Navyug Science College, Surat, Gujarat, 395009, INDIA

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Abstract

Azadirachta Indica (AI) leaves extract was investigated as a copper corrosion inhibitor in nitric acid. Corrosion rate and inhibition properties were studied by using weight-loss, effect of temperature, polarization and electrochemical impedance spectroscopic methods. Corrosion rate increases with the increase in acid concentration and temperature. The percentage of inhibition efficiency (I.E.) increases with increase AI concentration. The value of free energy of adsorption (ΔG_a) and heat of adsorption (Q_{ads}) obtained were negative. The inhibition effect is discussed in view of AI molecules adsorbed on the metal surface and it follows Langmuir adsorption isotherm. Polarization curve indicates that inhibitor act as mixed type (anodic and cathodic) and the inhibition efficiency was found up to 98%. It was found that AI extract is good inhibitor for the corrosion of copper in nitric acid medium.

Keywords: Copper, corrosion, inhibitor, azadirachta indica (*AI*) leaves extract.

Introduction

Corrosion of metal/alloy, which can be defined as the deterioration of materials due to their reaction with the environment. Copper is a metal having a wide range of applications due to its good properties. It is used in electronics, for production of wires, sheets, and tubes, and also to form different alloys; its corrosion resistance becomes less while the aggressive solution concentration increases[1,2]. Copper corrosion in nitric acid solution induced a great deal of research[3,4]. It is noticed that presence of heteroatom in organic compound such as nitrogen[5,6], oxygen[7,8], phosphorus[9] and sulphur[10,11] molecules are good corrosion inhibitors, but these compounds are highly toxic.

The purpose of the present work is to develop eco-friendly corrosion inhibitors having good inhibition efficiency at low risk of environmental pollution[12]. From many decades researchers attracted to study plant extracts as eco-friendly inhibitors for metal corrosion. Green inhibitors are environment friendly, source of non-toxic compounds, bio-degradable and of potentially low cost inhibitors for preventing metal corrosion[13]. Most of the naturally occurring substances are safe and can be extracted by simple procedure. As a natural product they are safe and can be extracted by simple procedure.

Azadirachta Indica (Neem) is more useful for its medicinal, chemical and biological activities. It is one of the richest sources of secondary metabolites in nature[14]. Natural products have been isolated from various parts of a tree[14-16], many of them acts as insect antifeedant, antifungal, insect growth regulatory, antiviral and antimalarial properties[17,18]. The *AI* extract has been reported to effectively inhibit the metal corrosion in acidic medium[19-28]. The present study investigated the inhibition effect of *AI* leaves for copper corrosion in nitric acid solution by using weight loss, effect of temperature, potentiodynamic polarization and electrochemical impedance spectroscopic methods.

Material and Methods

Sample and Solution preparation: The copper metal used in this study with a chemical composition 99.99 % Cu and 0.01 % S. The copper specimens of the size 4.5 × 2 × 0.178 cm were used. The specimens were cleaned by washing with distilled water, degreased by acetone, washed once more with bidistilled water and finally dried and weighted by using electronic balance. The corrosive solution was prepared by diluting analytical grade of HNO_3 purchased from Merck using double distilled water.

Preparation of extract: The *AI* leaves were dried, ground to powder form and boiling with double distilled water to making extract of different concentrations 0.6, 0.8, 1.0 and 1.2 g/l.

Mass loss measurements: For mass-loss experiment, the copper specimen having an area of 0.1988 dm^2 were each suspended and completely suspended in 230 ml of HNO_3 solution with and without different *AI* extract concentrations using glass hooks at 301± 1 K for 24h. The coupons were retrieved after 24h, washed by distilled water, dried well and reweighed. From the weight loss data, Corrosion rate (CR) was calculated.

Temperature effect: To study the temperature effect, the copper coupons were completely immersed in 230 ml of 1 M HNO_3 solution with and without different *AI* extract concentrations using glass hooks and corrosion rate were determined at 313, 323 and 333K for 2h to calculate inhibition efficiency, activation energy (Ea) and heat of adsorption (Q_{ads}).

Potentiodynamic polarization measurements: For polarization study, metal specimens were immersed with and without *AI* extract in 0.5 M HNO_3 solution. In the electrochemical cell copper specimens as a working electrode, saturated calomel electrode as a reference electrode and platinum electrode as an auxiliary electrode and allowed to establish a steady-state open circuit potential (OCP) for approximately 15 min. The polarization curves were plotted with current-potential. An anodic and cathodic polarization curve gives correspondently anodic and cathodic Tafel lines. The intersect point of Tafel lines gives the corrosion potential (E_{corr}) and corrosion current (i_{corr})[29].

Electrochemical impedance spectroscopy measurements: EIS measurements were made at corrosion potentials over a frequency range of 0.1 Hz to 10^5 Hz by a sine wave with potential perturbation amplitude of 5 mV. The real Z' and imaginary Z'' parts were measured at various frequencies. From the plot of Z' Vs. Z'', the charge transfer resistance (R_{ct}), and double layer capacitance (C_{dl}) were calculated. An experiment was carried out both with and without inhibitor.

Results and Discussion

Weight loss experiments: The weight loss experiments for copper corrosion in different concentration (0.5, 0.75 and 1 M) of HNO_3 solution containing different *AI* concentration (0.6, 0.8, 1.0, 1.2 g/l) at 301±1 K for exposure period of 24h was investigated.

Inhibition efficiency (I.E.) was calculated by following equation,

$$\text{I.E.}(\%) = \frac{W_u - W_i}{W_u} \times 100 \quad (1)$$

Where: W_u - Weight loss without inhibitor, W_i - Weight loss with inhibitor. The surface coverage (θ) of the copper specimen for different inhibitor concentration in HNO_3 solution have been evaluated by weight loss experiments using following equation,

$$\theta = \frac{W_u - W_i}{W_u} \quad (2)$$

From the results obtain from table-1 shows that when acid concentration increase, corrosion rate increase and inhibition efficiency decrease.

The results obtained were presented in table-2 and in figure-1, the corrosion rate of 0.5 M HNO_3 was decreased from 90.52 to 5.03 mg/dm^2 while inhibition efficiency increases from 69.45 to 94.45 % with increase in inhibitor concentration from 0.6-1.2 g/l. It can be concluded that inhibition efficiency is directly proportional to the inhibitor concentration.

Table-1
Corrosion rate (CR) and inhibition efficiency (I.E.) for copper corrosion in different concentration of HNO_3 solution in the presence and absence of *AI* extract for 24h at 301 ± 1 K.

Inhibitor Concentration g/l	Acid Concentration					
	0.5 M		0.75 M		1.0 M	
	CR mg/dm^2	I. E. %	CR mg/dm^2	I. E. %	CR mg/dm^2	I. E. %
Blank	90.52	-	281.55	-	1030.67	-
0.6	27.65	69.45	90.50	67.86	356.98	65.36
0.8	25.14	72.23	80.45	71.43	311.73	69.75
1.0	19.11	78.89	65.36	76.78	276.53	73.17
1.2	5.03	94.45	35.20	87.50	196.09	80.97

Table-2
Inhibition efficiency, corrosion rate and surface coverage of *AI* extract on Copper corrosion in 0.5 M Nitric acid for an immersion period of 24h at 301 ± 1 K

Inhibitor concentration g/l	CR (ρ) mg/dm^2	log ρ	I.E. %	surface coverage θ	1-θ	log(θ/1-θ)
Blank	90.52	1.9567	-	-	-	-
0.6	27.65	1.4417	69.45	0.6945	0.3055	0.3567
0.8	25.14	1.4004	72.23	0.7223	0.2777	0.4151
1.0	19.11	1.2813	78.89	0.7889	0.2111	0.5725
1.2	5.03	0.7016	94.45	0.9445	0.0555	1.2309

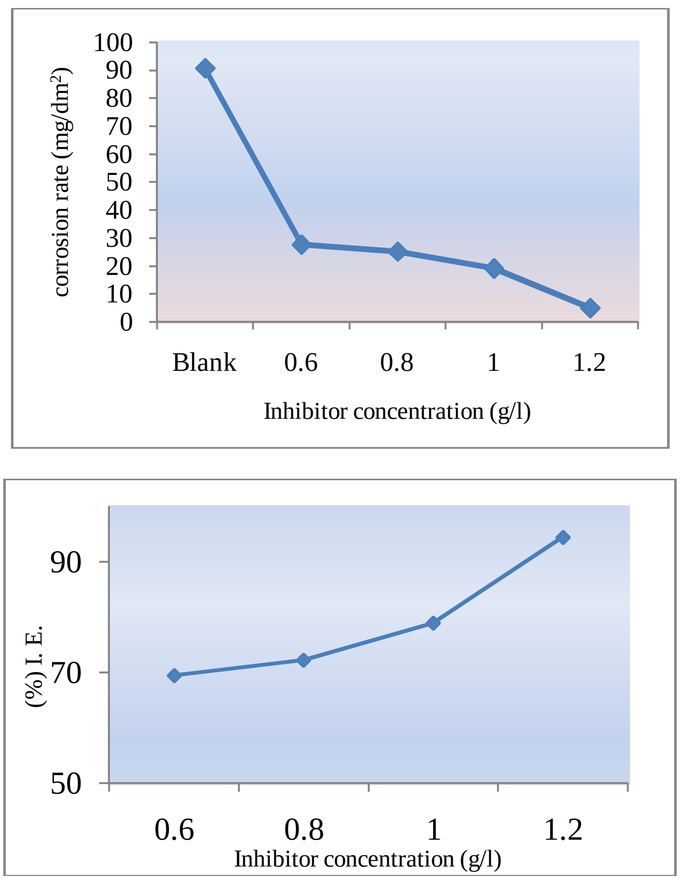

Figure-1
Corrosion rate and Inhibition efficiency of copper corrosion in 0.5 M HNO_3 solution in presence of different concentration *AI* extract for an immersion period of 24h

The surface coverage θ value was calculated by using equation-2. The plot of inhibitor concentration C_{inh} versus C_{inh}/θ is presented in figure-2 gives straight line with almost unit slope indicate that the system follows Langmuir Adsorption Isotherm[30]. This isotherm can be represented as,

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \tag{3}$$

Where, K_{ads} is the equilibrium constant and C_{inh} is the inhibitor concentration.

Linear plot obtained from figure-2 shows that constituents of *AI* extract on a copper surface making a barrier for charge and mass transfer between the metal and environment follows Langmuir adsorption isotherm which shows inhibition efficiency.

Temperature effect: The mass loss experiments were also carried out at different temperature 313, 323 and 333K in 1M HNO_3 to investigate the influence of temperature on the rate of corrosion for immersion period of 2h.

The value of energy of activation (E_a) has been calculated with the help of Arrhenius equation[30].

$$\log\frac{\rho_2}{\rho_1} = \frac{E_a}{2.303R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \tag{4}$$

Where: ρ_1 and ρ_2 are the corrosion rate at temperature T_1 and T_2 respectively.

As data given in table-3, the values of E_a are higher in inhibited acid than uninhibited acid ranging from 65.15 – 78.94 kJmol^{-1}, which indicates that the inhibitors are strongly adsorbed on metal surface.

From the data of table-3 and figure-4, as temperature increases, rate of corrosion increase while percentage of inhibition efficiency decreases.

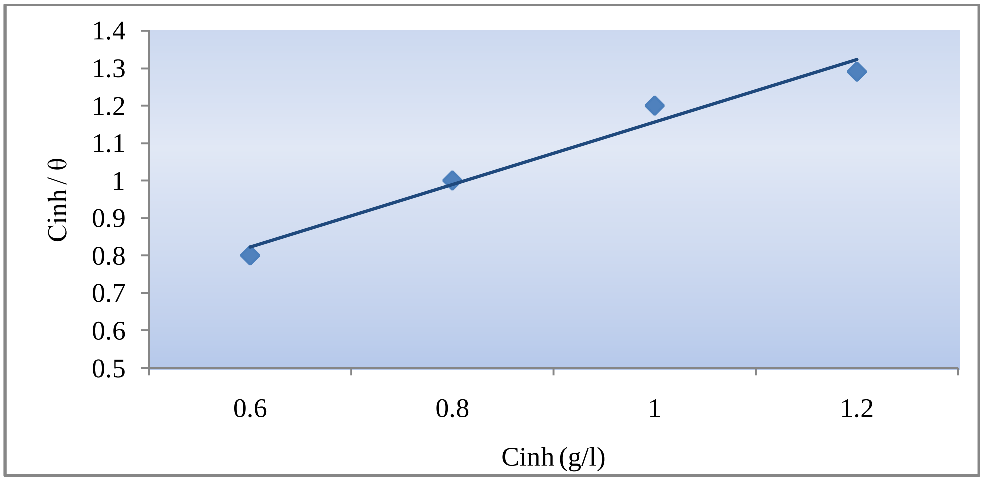

Figure-2

Langmuir adsorption isotherm for copper corrosion in 0.5 M HNO_3 solution at different concentration of *AI* extract

Table-3

Temperature effect on corrosion rate (CR), activation energy (Ea) and heat of adsorption (Q_{ads}) for copper in 1 M HNO_3 in absence ant presence of *AI* extract for an immersion period of 2h

***AI* Concentration g/l**	**Temperature**						**Mean (Ea) from Eq. (1) kJ mol^{-1}**	**Q_{ads} kJ mol^{-1}**	
	313 K		**323 K**		**333 K**				
	CR mg/dm^2	**I. E. %**	**CR mg/dm^2**	**I. E. %**	**CR mg/dm^2**	**I. E. %**		**313-323 K**	**323-333K**
Blank	442.46	-	754.19	-	1960.88	-	65.15	-	-
0.6	110.61	75.00	191.06	74.67	593.29	69.74	73.65	-1.47	-22.02
0.8	95.53	78.41	170.95	77.33	492.73	74.87	71.80	-5.27	-12.11
1.0	75.42	82.95	140.78	81.33	432.40	77.95	76.42	-9.29	-18.68
1.2	55.31	87.50	110.61	85.33	336.87	82.82	78.94	-15.57	-16.80

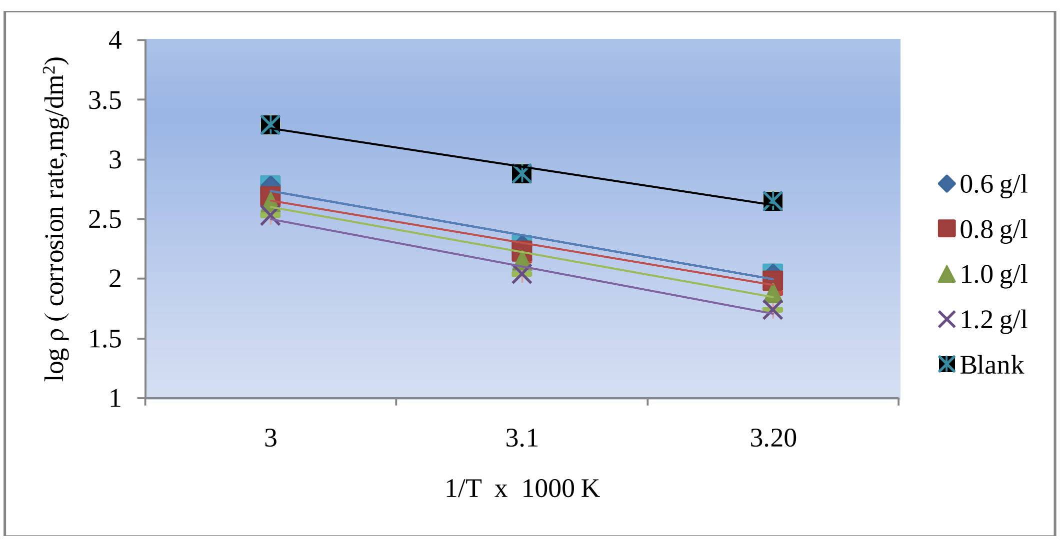

Figure-3

Arrhenius plot for copper corrosion in 1 M HNO_3 in absence and presence of the different concentration of *AI* extract

The values of heat of adsorption (Q_{ads}) were calculated by following equation[31].

$$Q_{ads} = 2.303R\left[\log\left(\frac{\theta_2}{1-\theta_2}\right) - \log\left(\frac{\theta_1}{1-\theta_1}\right)\right] \times \left[\frac{T_1T_2}{T_2-T_1}\right] \quad (5)$$

Where, θ_1 and θ_2 ($\theta = W_u - W_i / W_u$) are the fraction of the metal surface covered by the inhibitors at temperature T_1 and T_2 respectively. The negative and lower values of Q_{ads} support higher Inhibition efficiency achieved by spontaneous adsorption of the inhibitor.

Potentiodynamic polarization study: Figure-5 represents the Potentiodynamic polarization curves of copper in 0.5 M HNO_3 in the presence and absence of *AI* extract. Associated electrochemical parameters such as corrosion potential (E_{corr}), corrosion current density (i_{corr}), anodic Tafel slope(β_a), cathodic Tafel slope (β_c) and percentage inhibition efficiency (I.E.) are given in table-4.

From figure-5 and table-4, it was observed that the addition of *AI* extract in acid solution indicates the significant decrease in the corrosion current density (i_{corr}) and decrease in the corrosion rate with respect to the blank. There is significant change in the anodic and cathodic slopes after the addition of the inhibitor. This Tafel curves indicate that *AI* function as a mixed-type inhibitor.

Inhibition efficiency (I.E.) was calculated using following equation.

$$I.E.(\%) = \frac{i_{corr} - i_{corr(inh)}}{i_{corr}} \times 100 \quad (6)$$

Electrochemical impedance spectroscopy (EIS) measurements: Nyquist plots for the corrosion of copper in 0.5 M HNO_3 solution in the presence and absence of *AI* extract was examined by EIS method at room temperature was shown in figure-6 and table-5.
It is observed from figure-6 that the impedance diagram is almost semicircular in appearance, but not perfect semicircle. The difference has been attributed to frequency dispersion. The semicircular nature of the plots indicates that the corrosion of copper is mainly controlled by charge transfer process.

The diameter of capacitive loop in the presence of inhibitor is bigger than that in the absence of inhibitor.

The high frequency capacitive loop is related to the charge transfer resistance (R_{ct}). To calculate the double layer capacitance (C_{dl}), the frequency at which the imaginary component of the impedance is maximum was found as presented in the following equation[32].

$$C_{dl} = \frac{1}{2\,\pi F_{max}\,R_{ct}} \quad (7)$$

Where f is the frequency at the maximum height of the semicircle on the imaginary axis and R_{ct} is the charge transfer resistance[33].

Inhibition efficiency was calculated using following equation.

$$I.E.(\%) = \frac{C_{dl} - C_{dl(inh)}}{C_{dl}} \times 100 \quad (8)$$

The addition of inhibitor increase R_{ct} value while decreases in C_{dl} values which is due to the adsorption of inhibitor on the metal surface. The results suggest that the inhibitor acts by the formation of a protective layer on the surface, which modifies the metal/solution interface.

The result indicates that *AI* leaves extract performs as good inhibitor for the corrosion of copper in nitric acid media. Eddy and Ebenso[34] noted that *AI* leaves extract contains saponin, tannin, alkaloid, glycoside, anthraquinone and flavanoid as major phytochemical constituents.

The results indicate that *AI* leaves extract performs as good inhibitor for the corrosion of copper in nitric acid solution.

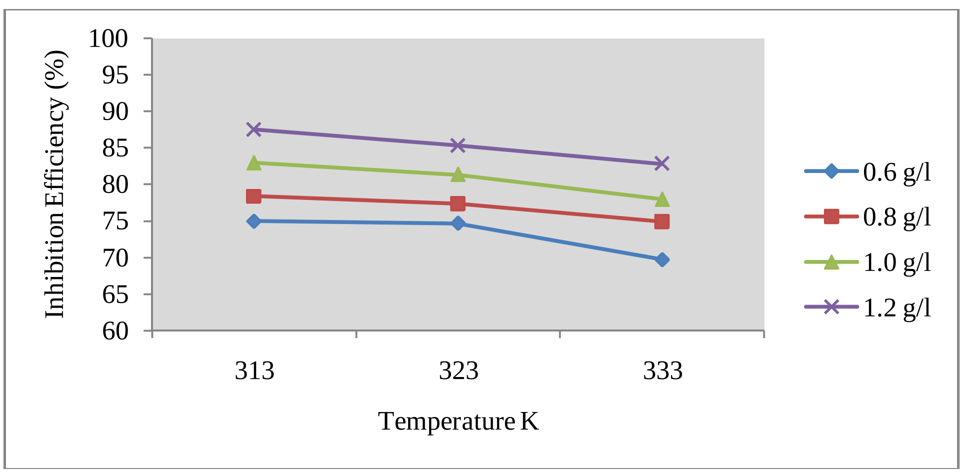

Figure-4
Effect of temperature on inhibition efficiency for copper corrosion in 1 M HNO_3 at different concentration of *AI* extract for immersion period of 2h

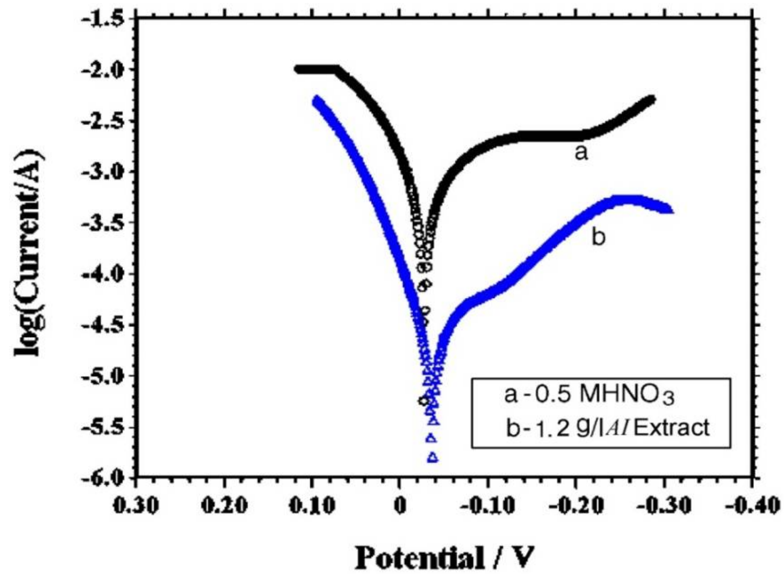

Figure-5
Potentiodynamic polarization plots for copper in (a) 0.5 M HNO_3 and (b) 0.5 M HNO_3 in the presence of 1.2 g/l *AI* extract

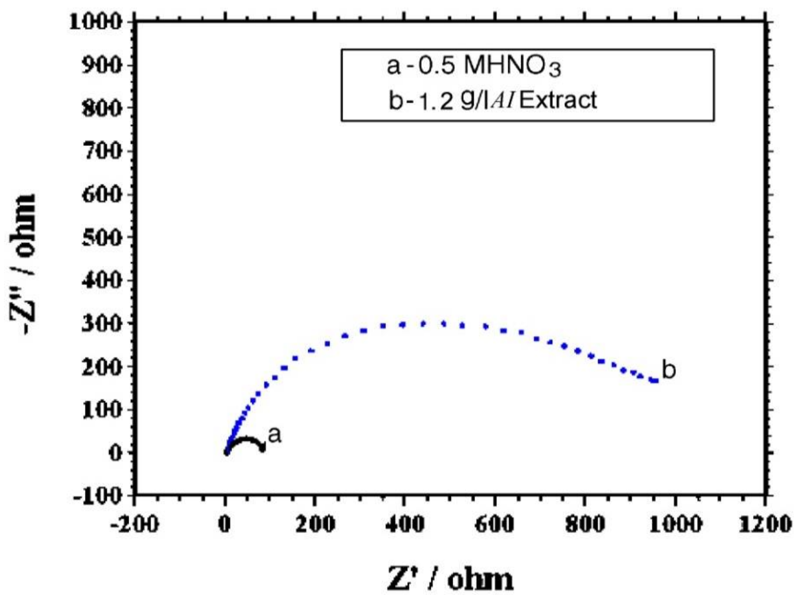

Figure-6
Nyquist plot for copper in (a) 0.5 M HNO_3 and (b) 0.5 M HNO_3 in the presence of 1.2 g/l *AI* extract

Table-4
Potentiodynamic polarization parameters of copper in 0.5 M HNO_3 and in presence of 1.2 g/l *AI* extract

System	E_{corr} V	I_{corr} ($\mu A/cm^2$)	Tafel Slope			Inhibition efficiency (I.E. %)	
			Anodic $+\beta_a$	Cathodic $-\beta_c$	β (mV)	By Polarization Method	By Weight loss Method
A	-0.028	2.062	5.887	2.327	0.7251	-	-
B	-0.035	0.040	15.011	6.783	1.16636	98.06	94.45

A= 0.5 M HNO_3 B= 0.5 M HNO_3 + 1.2 g/l *AI* extract

Table-5
EIS parameters for the corrosion of copper in 0.5 M HNO_3 containing *AI* extract

System	R_{ct} (Ω cm^2)	C_{dl} (μFcm^2)	I.E. (%)	
			By EIS method	By Weight loss method
A	85	53.52	-	-
B	970	0.53	99.00	94.45

Table-6
The phytochemical constituents of leaves extract of Azadirachta indica[34]

Phytochemicals	Leaves
Flavanoid	+
Anthraquinone	+
Phlobatanin	++
Cardiac glycosides	+++
Tannin	+++
Terpene	+++
Alkaloid	+++
Saponin	+++

+++ = present in large quantity
++ = moderately present
\+ = present in trace quantity

Conclusion

AI was found to be a good eco-friendly inhibitor for the corrosion control of copper in HNO_3 solution. The inhibition efficiency increase with increase in *AI* concentration. *AI* adsorbed on metal surface follows Langmuir adsorption isotherm. Tafel plot indicates *AI* acts as a mixed type inhibitor. AC impedance spectra reveal that a protective film is formed on the metal surface. All three techniques give almost identical values of Inhibition efficiency for copper in HNO_3.

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