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Azadirachta Indica (Neem) Extract as Green Inhibitor for Corrosion of Brass in Nitric Acid Media

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ABSTRACT

The inhibitive action of leaves extract of Azadirachta Indica (AI) on brass in nitric acid was studied using weight loss, effect of temperature, Polarization, Electrochemical Impedance Spectroscopic (EIS) and Scanning Electron Microscope (SEM) methods. Corrosion rate increases with the increase in acid concentration and temperature. As inhibitor concentration increases percentage of inhibition efficiency (I.E.) increases. The value of free energy of adsorption (ΔG^0_{ads}), heat of adsorption (Q_{ads}), energy of activation (E_a), enthalpy of adsorption (ΔH^0_{ads}) and entropy of adsorption (ΔS^0_{ads}) were calculated. The inhibition effect is discussed in view of AI molecules adsorbed on the metal surface and it obeys Langmuir adsorption isotherm. Polarization curve indicates that inhibitor act as mixed type and the I.E. was found up to 97.26%. Present study indicates that AI extract is a good inhibitor for the corrosion of brass in nitric acid medium.

Keywords: Brass, Nitric acid, *Azadirachta Indica* (*AI*), Corrosion, Inhibitor.

INTRODUCTION

Brass is broadly used as tubing material for condensers and heat exchangers in various cooling water systems [1,2]. Copper and its alloys are widely used in industry especially in heating and cooling system because of their excellent electrical and thermal conductivity [3]. This family of copper alloys is susceptible to a corrosion process known as dezincification and it susceptibly increases as zinc content increases [4].

The purpose of the present work is to develop eco-friendly corrosion inhibitor having good inhibition efficiency at low risk of environmental pollution [5]. 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 [6]. A number of studies have recently appeared in the literature on topic of the corrosion inhibition of α-brass in acidic medium [7-8]. Vashi *et al*. have studied *Azadirachta Indica* extract as corrosion inhibitor for copper in nitric acid and ethylamines as corrosion inhibitors for zinc in nitric acid [9,10].

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 [11]. Natural products have been isolated from various parts of a tree [11-13], many of them acts as insect antifeedant, antifungal, insect growth regulatory, antiviral and antimalarial properties [14,15]. The *AI* extract has been reported to effectively inhibit the metal corrosion in acidic medium [16-22]. The aim of the present study is to investigate the inhibition effect of *Azadirachta Indica* as a cheap and environment friendly corrosion inhibitor for brass in various concentration of HNO_3 medium by weight loss, effect of temperature, polarization, EIS and SEM methods.

MATERIALS AND METHODS

Preparation of sample and solution: The brass metal used in this study with a chemical composition 60.73% Cu, 39.21% Zn, 0.01% Fe, 0.02% Sn and 0.04% Pb. The brass specimens of the size 4.4 x 2.0 x 0.181 cm were used. The specimens were cleaned by washing with distilled water, degreased by acetone, washed once more with doubled distilled 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: *Azadirachta indica* leaves were collected and oven dried at 50°C then dried leaves were ground to powder form. 4.0 g of the powder was digested in 1.0 litre of 0.1, 0.25, 0.5 and 1.0 M HNO_3. The resultant solution was kept for 24 h, filtered and stored. From the stock solution, leaves extracts test solutions were prepared at different concentrations [23].

Weight loss measurements: For weight-loss measurement, the copper specimen having an area of approximately 0.206 dm^2 were each completely suspended in 230 mL of HNO_3 solution with and without different *AI* extract concentrations using glass hooks at 301± 1 K for 24 h. The coupons were retrieved after 24 h, washed with distilled water, dried well and reweighed. From the weight loss data, corrosion rate (CR) was calculated.

Temperature effect: To study temperature effect, brass coupons were completely immersed in 230 ml of 0.1 M HNO_3 solution with and without different *AI* extract concentrations using glass hooks and corrosion rate were determined at 313, 323 and 333 K for 2 h to calculate inhibition efficiency (I.E.), activation energy (E_a) and heat of adsorption (Q_{ads}) and free energy of adsorption (ΔG^0_{ads}).

Potentiodynamic polarization measurements: Both the potentiodynamic and EIS measurement are carried out using CHI608C –series, U.S. Model with CH- instrument. For polarization study, metal specimens were immersed with and without *AI* extract in 0.1 M HNO_3 solution. In the electrochemical cell brass specimens having an area of 1 cm^2 was used as a working electrode, Ag/AgCl 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 Vs potential. An anodic and cathodic polarization curve gives corresponding anodic and cathodic Tafel lines. The intersect point of Tafel lines gives the corrosion potential (E_{corr}) and corrosion current (i_{corr}) [24].

Electrochemical Impedance Spectroscopy (EIS) measurements: EIS measurements are made at corrosion potentials over a frequency range of 0.1 to 105 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 in absence and presence of inhibitor.

Scanning Electron Microscope (SEM) study: The surface morphology measurement of brass specimens were examined using FEI, Model- Inspect computer controlled SEM.

RESULTS AND DISCUSSION

Weight loss experiments: The weight loss experiments was carried out in 0.1, 0.25, 0.5 and 1.0 M HNO_3 solution containing 600, 800, 1000, 1200 ppm concentration of *Al* at 301±1 K for an exposure period of 24 h was investigated.

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

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

Where: Wu - Weight loss in absence of inhibitor, Wi - Weight loss in presence of inhibitor.

The surface coverage (θ) of the brass specimen for different inhibitor concentration in HNO_3 solution have been evaluated by weight loss experiments using the following equation,

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

As the acid concentration increases corrosion rate increase while I.E. decreases. At constant acid concentration, as the inhibitor concentration increases corrosion rate decreases while I.E. increases (Table-1 and Figure-1, 2).

Table 1: Effect of HNO_3 concentration on corrosion rate (CR) and inhibition efficiency (IE) of brass having different concentration of *Al* extract.

Inhibitior concen tration (ppm)	**Acid concentration**							
	0.1 M		**0.25 M**		**0.5 M**		**1.0 M**	
	CR (mg /dm^2d)	**I.E. (%)**	**CR (mg/dm^2d)**	**I.E. (%)**	**CR (mg/dm^2d)**	**I.E. (%)**	**CR (mg/dm^2d)**	**I.E. (%)**
Blank	1820.39	-	6577.67	-	9393.20	-	15902.91	-
600	119.42	93.44	548.54	91.66	2567.96	72.66	7572.82	52.38
800	79.42	95.64	203.58	94.98	2291.26	75.61	6368.93	59.95
1000	54.56	97.00	184.47	95.68	1169.90	85.55	4611.65	71.00
1200	29.71	98.37	111.65	97.69	825.24	91.21	4330.09	72.77

Table 2: Inhibition efficiency (I.E.), corrosion rate and surface coverage (θ) of *AI* extract on brass in 0.1 M HNO_3 for an immersion period of 24h at 301 ± 1 K

Inhibitor	**Inhibitor Concentration**	**C.R.(ρ) (mg/dm^2d)**	**log ρ**	**I.E. (%)**	**Surface coverage θ**	**1-θ**	**log(θ/1-θ)**
Blank	0	1820.39	3.26	-	-	-	
Azadirachta Indica (Neem)	600	119.42	2.08	93.44	0.93	0.07	1.15
	800	79.42	1.90	95.64	0.96	0.04	1.34
	1000	54.56	1.74	97.00	0.97	0.03	1.51
	1200	29.71	1.47	98.37	0.98	0.02	1.78

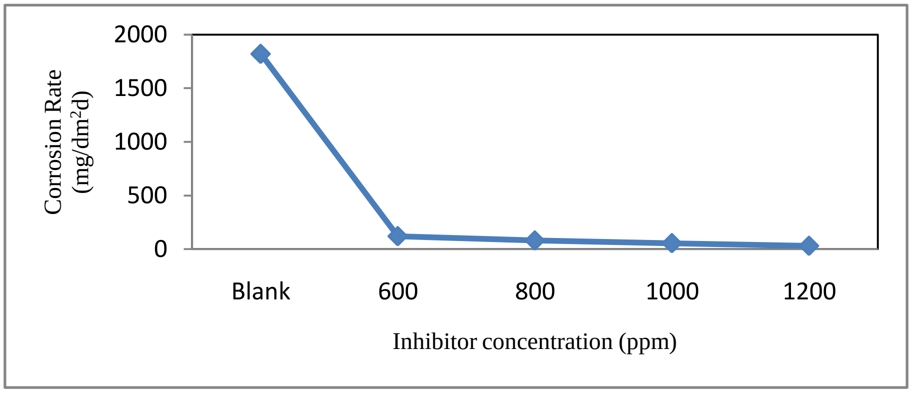

Fig.1: Corrosion rate of brass in 0.1 M HNO_3 solution in presence of different concentration *AI* extract for an immersion period of 24h

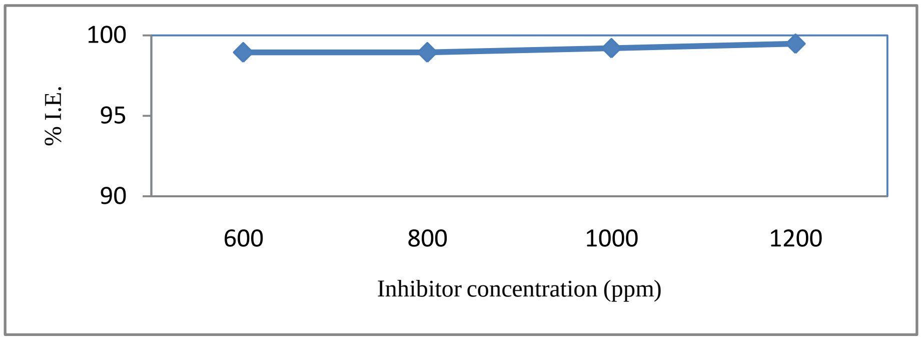

Fig.2: Inhibition efficiency of brass corrosion in 0.1 M HNO_3 solution in presence of different concentration *AI* extract for an immersion period of 24h

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-3 which gives straight line with almost unit slope indicates that the system follows Langmuir Adsorption isotherm [25]. This isotherm can be represented as,

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad (3)$$

Where, K_{ads} is the equilibrium constant and C_{inh} is the inhibitor concentration. Linear plot obtained from Figure-3 shows that constituents of *AI* extract on a brass surface making a barrier for charge and mass transfer between the metal and environment follows Langmuir adsorption isotherm which shows inhibition efficiency.

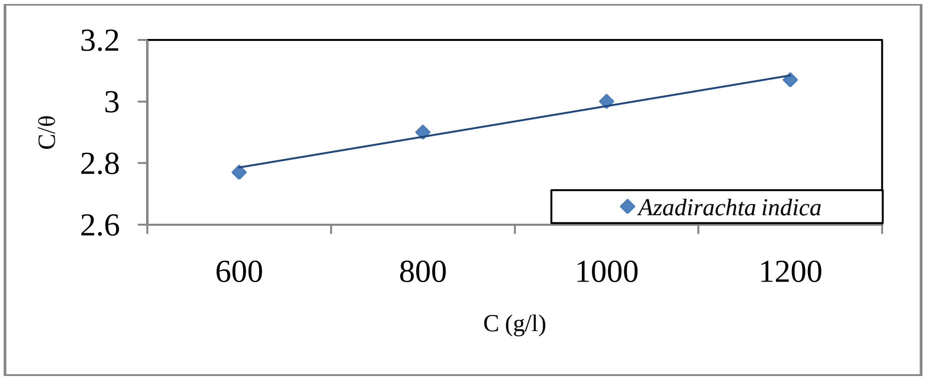

Fig.3: Langmuir adsorption isotherm plot of C/θ versus 'C' for brass in 0.1 M HNO_3 containing *AI* as green inhibitor at 301 K

Temperature effect: The mass loss experiments were also carried out at different temperature 313, 323 and 333 K in 0.1M 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 [22].

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

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 209.02 to 221.98 kJ/ mol 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 I.E. decreases. The values of heat of adsorption (Q_{ads}) were calculated by following equation [26].

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

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

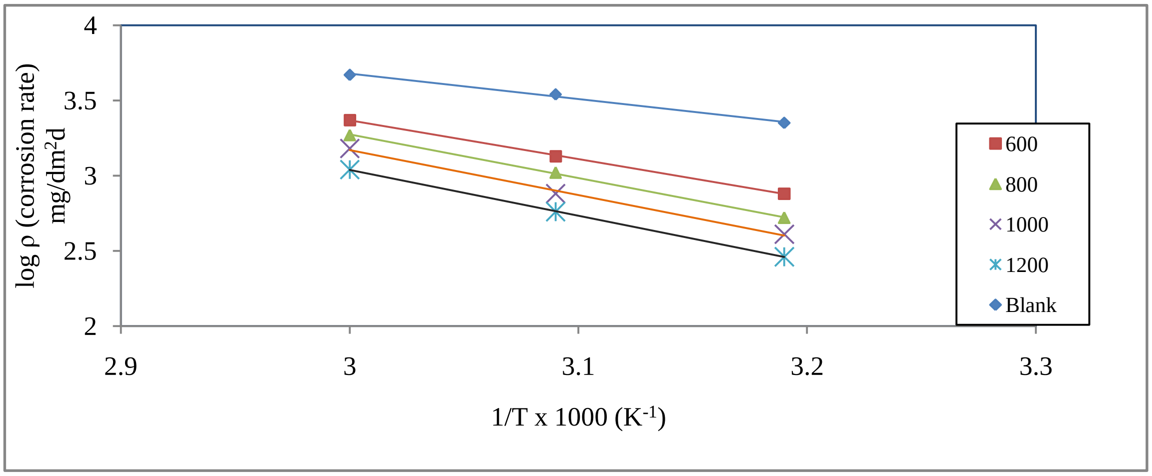

Fig 4: Arrhenius plots for brass in 0.1 M HNO_3 in absence and presence of the different concentration of *AI* extract

Table 3: Temperature effect on corrosion rate (CR), activation energy (E_a) and heat of adsorption (Q_{ads}) for brass in 0.1M HNO_3in absence and presence of *AI* extract for an immersion period of 2h.

***AI* concen tration (ppm)**	**Temperature**						**Mean (E_a) From Equation (4) (kJ/mol)**	**Qads (kJ /mol)**	
	313 K		**323 K**		**333 K**				
	CR mg /dm²d	**I. E. (%)**	**CR mg /dm²d**	**I. E. (%)**	**CR mg /dm²d**	**I. E. (%)**		**313-323 K**	**323-333 K**
Blank	2213.52	-	3436.92	-	4660.2	-	32.11	-	-
600	757.2	65.79	1339.8	61.01	2330.04	50.00	48.72	-18.98	-40.01
800	524.16	76.32	1048.44	69.49	1863.96	60.00	54.86	-29.15	-35.30
1000	407.76	81.57	757.32	77.96	1514.52	67.56	57.02	-21.01	-45.78
1200	291.24	86.84	582.48	83.05	1106.76	76.25	57.84	-26.24	-38.72

Potentiodynamic polarization study: Figure-5 represents the potentiodynamic polarization curves of brass in 0.1 M HNO_3 in absence and presence of *AI* extract. Electrochemical parameters such as corrosion potential (E_{corr}),corrosion current density (i_{corr}), anodic Tafel slope (βa), cathodic Tafel slope (βc) 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 corrosion current density (i_{corr}) and decrease in the corrosion potential with respect to 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 and predominant cathodic inhibitor.
Inhibition efficiency (I.E.) from polarization study was calculated using following equation.

$$\mathrm{I.E.}\ (\%) = \frac{i_{corr\ (unin\ h)} - i_{corr\ (in\,h)}}{i_{corr\ (unin\ h)}} \qquad (6)$$

Electrochemical impedance spectroscopy (EIS) measurements: Nyquist plots for the corrosion of brass in 0.1 M HNO_3 solution in absence and presence of *AI* extract was examined by EIS method at room temperature was shown in Table-5 and in Figure 6.

It is observed from Figure 6 that the impedance diagram is almost semi circular in appearance, but not perfect semicircle. The difference has been attributed to frequency dispersion. The semi circular nature of the plots indicates that the corrosion of brass 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 [27].

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

Where ‘f’ is the frequency at the maximum height of the semicircle on the imaginary axis and R_{ct} is the charge transfer resistance [28].

Inhibition efficiency (I.E.) from EIS method was calculated using following equation:

$$\mathrm{I.E.}\ (\%) = \frac{\mathrm{Cdl\ (uninhi\ .) - Cdl\ (inhi\ .)}}{\mathrm{Cdl\ \ (uninhi\ .)}} \times 100 \qquad (8)$$

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 [29] 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 brass in nitric acid solution.

Scanning Electron Microscopy (SEM) study: The surface morphology of brass electrodes were evaluated by the SEM technique. Fig. 7 shows the morphology of bare, uninhibited, and inhibited brass surfaces and the micrograph reveals that the surface was strongly damaged in the absence of inhibitor. According to the micrographs, the inhibited surface is free from pits and is smooth, indicating that a good protective film is present on the brass surface and also confirming the highest I.E.

Mechanism of brass: The behavior of anodic dissolution of brass corrosion in nitric acid is very complex and consists of dissolution of both zinc and copper. Generally, the anodic reaction for Cu is considered as follows [30–32]:

$Cu \rightarrow Cu\ (I)_{ads} + e^{-}$ (fast step) (9)

$Cu\ (I)_{ads} \rightarrow Cu\ (II) + e^{-}$ (slow step) (10)

where, Cu (I) ads is an adsorbed species on to copper surface and does not diffuse into the bulk solution. Meanwhile, the adsorption of copper ions, Cu (II), on the surface due to its reduction in presence of Zn must be taken into consideration. Also, the anodic dissolution of Zn is considered according to following equation:

$Zn \rightarrow Zn^{2+} + 2e^{-}$ (11)

It should be noticed that the cathodic reaction is not hydrogen evolution reaction at all, because the corrosion potential in all experiments is well above the redox potential of hydrogen evolution in this media. In this way, the predominant mechanism of cathodic reaction has been reported according to the following equation [31]:

$NO_3^{-} + 4H^{+} + 3e^{-} \rightarrow NO + 2H_2O$ (12)

Also, oxygen reduction in acidic media can be considered to be as another cathodic reaction based on Eq.(12).

Table 4: Potentiodynamic polarization data and Inhibition efficiency (I.E.) of *AI* extract as green inhibitor for brass in 0.1 M HNO_3

System	**E_{corr} (mV)**	**I_{corr} (µA / cm^2)**	**Tafel slope (mV / decade)**			**IE (%) Calculeted from**	
			Anodic (+βa)	**Cathodic (-βc)**	**β (mV)**	**Polarization method**	**Weight loss method**
Blank	-0.03	1408	4.72	5.47	1.10	-	-
Azadirachta Indica (Neem)	-0.012	58.80	13.45	3.06	1.08	97.26	95.64

Table 5: EIS parameters for corrosion of Brass in 0.1 M HNO_3 containing *AI* extract

System	**R_{ct} (Ω cm^2)**	**C_{dl} (µF / cm^2)**	**IE (%) Calculated from**	
			EIS method	**Weight loss method**
Blank	212.82	6.43	-	-
Azadirachta Indica (Neem)	1301.00	0.26	97.26	95.64

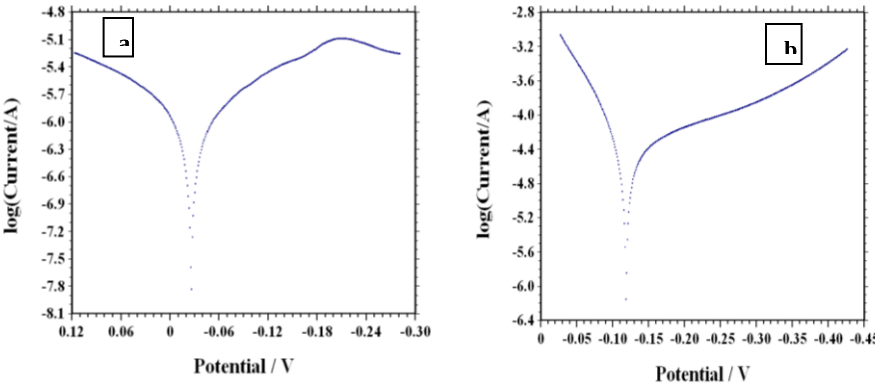

Fig.5: Potentiodynamic polarization curves for brass (a) in 0.1 M HNO_3 and (b) in 0.1 M HNO_3 in presence of 800 ppm *AI* extract.

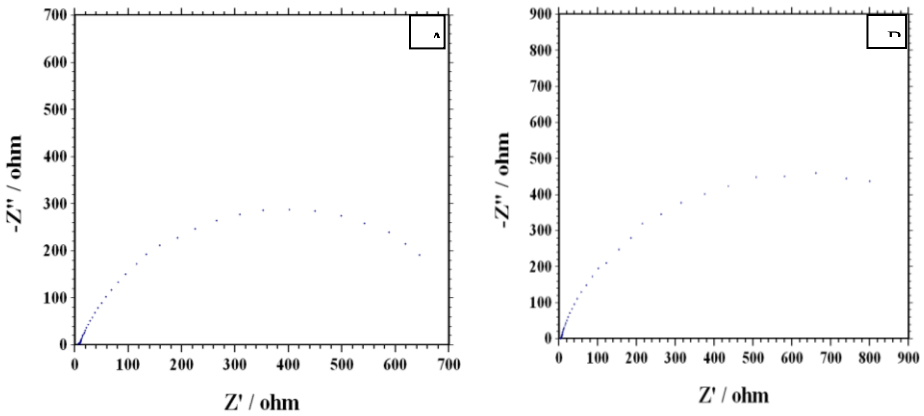

Fig 6: Nyquist plot for brass in (A) in 0.1 M HNO_3 (Blank) (B) in 0.1 M HNO_3 in presence of 800 ppm *AI* extract.

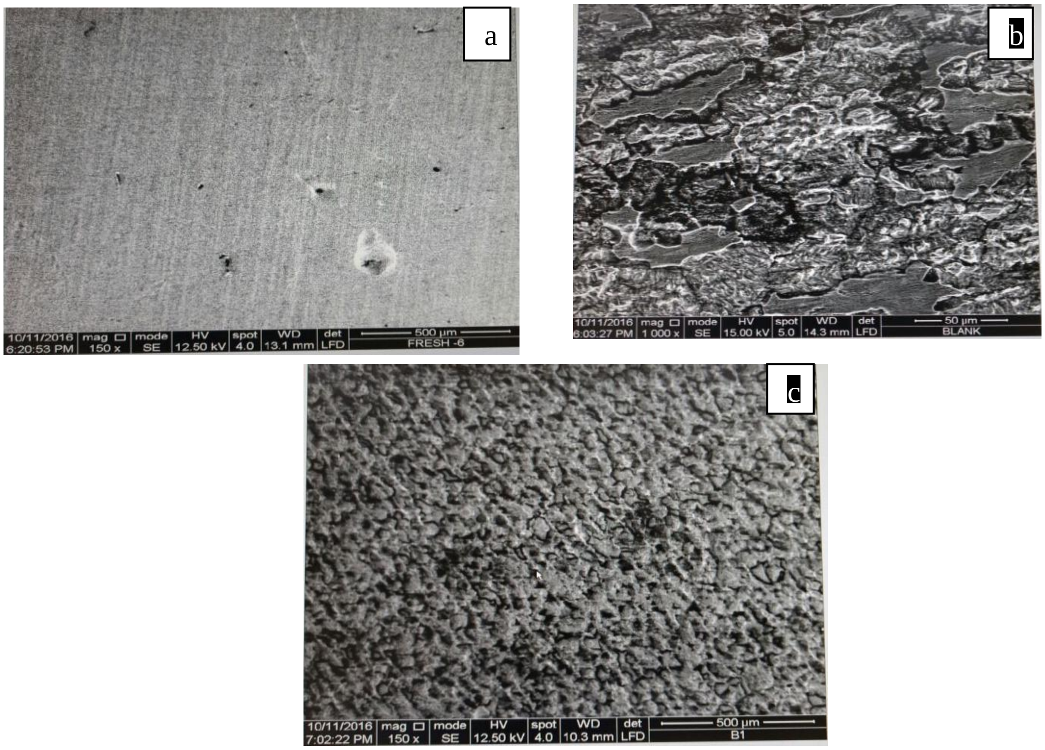

Fig.-7: SEM micrographs of brass (a) Polished brass plate (150 X), (b) immersion in 0.1 M HNO_3 in absence of inhibitor (1000 X), (c) immersion in 0.1 M HNO_3 in presence of 800 ppm *AI* inhibitor after 24 h (150 X)

APPLICATIONS

This study is useful for prevention of corrosion of brass by *Azadirachta Indica* (Neem) extract as green inhibitor in nitric acid media. The corrosion of brass is mainly controlled by charge transfer process and due to the formation of protective layer on the metal surface.

CONCLUSIONS

From the study the following conclusions can be drawn:

1. As the acid concentration increases the corrosion rate increases.
2. As the inhibitor concentration increase I.E. increases and corrosion rate decreases.
3. *AI extract* adsorbed on metal surface following Langmuir adsorption isotherm.
4. Tafel plots of polarization measurements indicates that the inhibitor behave as a mix type inhibitor.
5. EIS spectra exhibit semi circular in appearance which indicates that the corrosion of brass is mainly controlled by charge transfer process.
6. The SEM images confirm the formation of protective layer on the metal surface.

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