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Corrosion Inhibition of Copper Metal by Prosopis Cineraria Leaves as Green Corrosion Inhibitor in Acidic Medium

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Abstract: *Ethanollic extract of Prosopis cineraria leaves (EEPcL) behaves as a green corrosion inhibitor to replace toxic chemicals. The corrosion inhibition of copper in 0.5 N HCl by Prosopis cineraria leaves has been studied by mass loss method and electrochemical techniques (potentiodynamic polarization and electrochemical impedance spectroscopy). The inhibition efficiency was found around 85.85 % with 1.064 g/L of inhibitor concentration at 48 hours of immersion period. The inhibition efficiency is found to increase with increasing concentration of extract. Electrochemical and EIS measurements shows that the presence of EEPcL has caused the adsorption of inhibitor on the metal surface. The adsorption behavior of the inhibitor follow Langmuir adsorption model where the value of free energy of adsorption, $-\Delta G$, is less than 40 kJ/mol indicates that it is a physical adsorption.*

Keywords: *Electrochemical impedance spectroscopy, Corrosion, Adsorption, Copper, Prosopis cineraria.*

I. INTRODUCTION

Copper is a very important metal because it has a wide range of applications due to its good properties like its excellent heat conductivity and good corrosion resistance in water. Copper is an industrially important metal because it is used in electronics, for production of wires, sheets, tubes, heating and cooling systems, and condensers and also to form various alloys. In heating and cooling systems, condensers there are deposition of carbonates and oxides, which decreases the heating transmission [1]. To overcome this problem these systems should be regularly cleaned to descale, Diluted hydrochloric acid is used to clean such surfaces. Copper is resistant toward the influence of atmosphere and many chemicals, but in aggressive media it is susceptible to corrosion.

In such conditions it is necessary to stop corrosion of copper. We have to use copper corrosion inhibitors because no protective passive layer is possible. To inhibit copper corrosion in such solutions small amounts of corrosion inhibitor can be used to reduce corrosion rates [2].

In the past various organic and inorganic corrosion inhibitors were used but these are toxic and costly. The presence of large molecules with functional groups containing of heteroatom (such as oxygen, nitrogen, sulphur, and phosphorus), triple bonds or aromatic rings in the inhibitor's chemical structure enhance the adsorption process[3]. So nowadays the research has been carried out for extraction of compounds which are non toxic, natural and environmentally friendly inhibitors such as Prosopis juliflora, Rosamarinus officinalis L., Hibiscus Rosa-sinensis, Nympa fructicans, red onion skin, Allium cepa and Allium sativum, Vernonia amygdalina, punica granatum, vitis vinifera, Piper nigrum capparidaceae, Sida acuta (Wire Weed), Carica Papaya and Camellia Sinensis, Cannabis Plant Extract, Calophyllum inophyllum seed, Jatropha Curcas leaves Acacia nilotica, Ephedra Sarcocarpa Plant Extract, Citrullus colocynthis Fruits, Thymus vulgarise, Azadirachta indica Seed, Aloe vera leaf etc. [4-24]. Prosopis cineraria is prickly tree or shrub and commonly found in dry and arid regions of north western India, Southern India, Pakistan, Afganistan and Arabia. Leaves are extensively used as fodder for cattle, goats and camel[25,26]. Leaves contain specigerine, steroids such as campesterol, cholesterol, sitosterol, stigmasterol, Tricosan-1-ol, Methyl docosanoate, Diisopropyl-10,11-dihydroxyicosane-1,20-dioate[26-28]. Amino acids isolated from leaves and pods are Aspartic acid, Glutamic acid, Serine, Glycine, Histidine, Threonine, Arginine, Alanine, Proline, Tyrosine, Valine, Methionine, Cysteine, Isoleucine, Leucine, Phenylalanine and Lysine.[29,30]

II. EXPERIMENTAL

A. Extraction of leaves extract

Leaves of *Prosopis cineraria* in natural condition were air dried for 8 to 10 days in shade. Then grinded and powdered, finely powdered dried material was taken in 500 ml round bottom flask and sufficient quantity of ethyl alcohol was added and left for

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desired period. Then the resulting solution is extracted by standard method as discussed literatures.

B. Specimen preparation

The rectangular coupons of copper of dimensions (3cm× 2.4cm× 0.16cm) were used with a small hole of 2mm diameter near upper edge for hanging in test media. Each coupon was given surface treatment by standard method discussed in literature for each experiment.

C. Test solution

The 0.5 N HCl solutions was prepared by using doubly distilled water. HCl used was of analytical reagent quality.

D. (A) Chemical method-

In weight loss method the surface treated Cu coupons were dipped in beakers containing various concentrations of inhibitor in aggressive medium. Concentrations of inhibitor were in the range of 0.0532 to 1.064 g/L marked as (C1, C2, C3, C4, C5 and C6). From the weight loss method, the Inhibition efficiency and Corrosion rate of copper were calculated using equations (1 and 2).

$$\%I.E.= (W_1-W_2)/W_1 \times 100 \dots\dots\dots(1)$$

$$\text{Corrosion Rate (mmY-1)} = (87.6 \times DW) / DA T \dots\dots\dots (2)$$

where W1 and W2 are weight loss of copper in absence and presence of the inhibitor respectively, A is surface area in cm², D (8.933 gcm⁻³) is density of metal and T is time period in hours (h) and DW= W1- W2 (loss in weight).

These coupons were made to immerse for desired immersion period at room temperature (303±1°K). Before measurement, each coupon was surface treated dried in desiccators for 24 hours and then weight was taken. From the weight loss method, the Inhibition efficiency and corrosion rate of copper were calculated using standard equation.[31]

E. Electrochemical Measurements

All electrochemical measurements potentiodynamic polarization and impedance (EIS) measurements were made using CHI760D Electrochemical workstation Instrument, USA. The working electrode (WE) is made of copper with an exposing geometrical surface area of 1 cm²to the electrolyte, platinum as the auxiliary electrode (CE), and saturated calomel electrode (SCE) as the reference electrode (RE).

The electrochemical impedance spectroscopy, EIS, was carried out with the open circuit potential E_{ocp} for every sample. All of the samples were immersed for 30 min over a frequency range of 100 kHz to 10 m Hz with signal amplitude of 5 mV and a scan rate of 2 mVs⁻¹. Next, the value of R_{ct} was calculated from plot. The inhibition efficiency, IE, was calculated by the following equation (3).

$$\%I.E. = \frac{R_{ctinh} - R_{ct}}{R_{ctinh}} \times 100 \dots\dots\dots(3)$$

Where R_{ct} and R_{ctinh} refer to charge transfer resistance without and with the addition of an inhibitor[32]. All experiments were carried out solutions at 298±1 K. Before each measurement, the electrode surface treatment was given in the same manner as the weight loss experiments.

III. RESULT AND DISCUSSION

A. Weight loss measurements: corrosion rates and inhibition efficiency by weight loss measurements

Weight loss, corrosion rate, Fractional Surface coverage (θ), Adsorption Equilibrium Constant (K_{ad}) and inhibition efficiency of the extract obtained by weight loss method for different inhibitor concentrations at various periods of immersion in 0.5N HCl are presented in Table-1.

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Table-1 Corrosion Parameters of acid corrosion of Copper without and with different concentrations of EEPcL for various Immersion time(h)at 303±1 K

Time (h)	Prosopis cineraria Leaves extract Concentration (g/L)	Corrosion parameters				
		Weight loss (g)	Corrosion rate (ρ_{corr}) (mmpy)	Inhibition Efficiency (IE%)	Fractional Surface coverage (θ)	Adsorption Equilibrium Constant (K_{ad})
3	C0= Blank	0.0035	0.7094	NIL	NIL	NIL
	C1= 0.0532	0.0029	0.5878	17.14	0.1714	3.8890
	C2 =0.1064	0.0027	0.5472	22.86	0.2286	2.7847
	C3= 0.2128	0.0024	0.4864	31.43	0.3143	2.1538
	C4= 0.4256	0.0021	0.4256	40.00	0.4000	1.5664
	C5= 0.6384	0.002	0.4054	42.86	0.4286	1.1748
	C6 =1.064	0.0012	0.2432	65.71	0.6571	1.8014
6	C0= Blank	0.0046	0.4662	NIL	NIL	NIL
	C1= 0.0532	0.0034	0.3446	26.09	0.2609	6.6342
	C2 =0.1064	0.0033	0.3344	28.26	0.2826	3.7024
	C3= 0.2128	0.0031	0.3141	32.61	0.3261	2.2738
	C4= 0.4256	0.0031	0.3141	32.61	0.3261	1.1369
	C5= 0.6384	0.0024	0.2432	47.83	0.4783	1.4359
	C6 =1.064	0.0011	0.1115	76.09	0.7609	2.9904
12	C0= Blank	0.0094	0.4763	NIL	NIL	NIL
	C1= 0.0532	0.0065	0.3294	30.85	0.3085	8.3864
	C2 =0.1064	0.0062	0.3141	34.04	0.3404	4.8508
	C3= 0.2128	0.0044	0.2229	53.19	0.5319	5.3401
	C4= 0.4256	0.0042	0.2128	55.32	0.5532	2.9091
	C5= 0.6384	0.0037	0.1875	60.64	0.6064	2.4131
	C6 =1.064	0.0031	0.1571	67.02	0.6702	1.9100
18	C0= Blank	0.0158	0.5337	NIL	NIL	NIL
	C1= 0.0532	0.0117	0.3952	25.95	0.2595	6.5870
	C2 =0.1064	0.0125	0.4222	20.89	0.2089	2.4812
	C3= 0.2128	0.0103	0.3479	34.81	0.3481	2.5093
	C4= 0.4256	0.0084	0.2837	46.84	0.4684	2.0699
	C5= 0.6384	0.0052	0.1757	67.09	0.6709	3.1931
	C6 =1.064	0.0048	0.1621	69.62	0.6962	2.1538
24	C0= Blank	0.0278	0.7043	NIL	NIL	NIL
	C1= 0.0532	0.0214	0.5422	23.02	0.2302	5.6215
	C2 =0.1064	0.0221	0.5599	20.50	0.2050	2.4240
	C3= 0.2128	0.0174	0.4408	37.41	0.3741	2.8087
	C4= 0.4256	0.0123	0.3116	55.76	0.5576	2.9609
	C5= 0.6384	0.0095	0.2407	65.83	0.6583	3.0174
	C6 =1.064	0.0084	0.2128	69.78	0.6978	2.1706
48	C0= Blank	0.1324	1.6772	NIL	NIL	NIL
	C1= 0.0532	0.1134	1.4365	14.35	0.1435	3.1494
	C2 =0.1064	0.0862	1.0919	34.89	0.3489	5.0372
	C3= 0.2128	0.0824	1.0438	37.76	0.3776	2.8515
	C4= 0.4256	0.0412	0.5219	68.88	0.6888	5.2011

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	C5= 0.6384	0.0256	0.3243	80.66	0.8066	6.5349
	C6 =1.064	0.0186	0.2356	85.95	0.8595	5.7503
72	C0= Blank	0.2842	2.4000	NIL	NIL	NIL
	C1= 0.0532	0.2341	1.9769	17.63	0.1763	4.0228
	C2 =0.1064	0.2013	1.7000	29.17	0.2917	3.8705
	C3= 0.2128	0.2173	1.8351	23.54	0.2354	1.4468
	C4= 0.4256	0.1642	1.3867	42.22	0.4222	1.7171
	C5= 0.6384	0.0736	0.6215	74.10	0.7410	4.4822
	C6 =1.064	0.0714	0.6030	74.88	0.7488	2.8011

Table-1 shows the calculated values of corrosion rate (mm^{-1}), inhibition efficiency (IE%) and the degree of surface coverage for copper corrosion in 0.5 N HCl in the absence and presence of EEPcL. In fig-1 & 2, it is clear that the weight loss and corrosion rate of copper decreases in the presence of EEPcL as compared to the blank (i.e. without inhibitor). The corrosion rate also decreases with increasing concentration of the EEPcL.

The corrosion rate was increasing from 0.7094 mm^{-1} at 3 hours to 2.4000 mm^{-1} at 72 hours in blank solution but corrosion rate was decreasing up to 0.6030 mm^{-1} at 72 hours at 1.064 g/L of EEPcL.

The corrosion rate was more in blank solution as compared to solutions containing different concentration of inhibitor. The decrease in corrosion rate of Copper on introduction of the EEPcL to the corrosive medium 0.5 N HCl is indicating that the extract is showing inhibitive effect and retarding the acid corrosion of copper coupons.

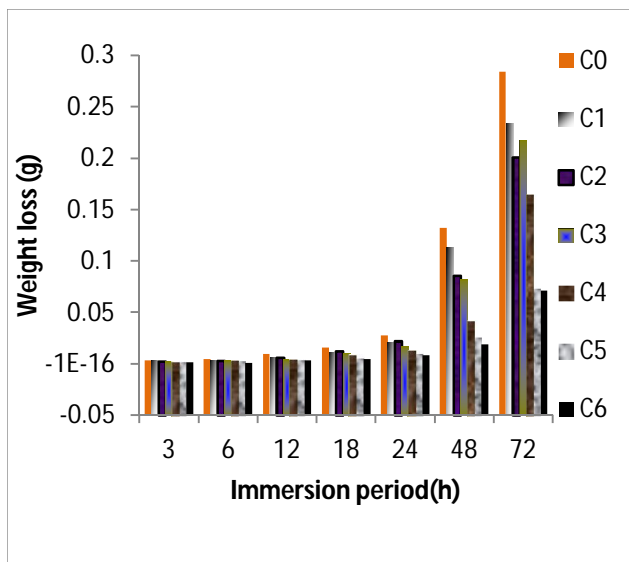


Fig-1 Weight loss v/s Immersion period

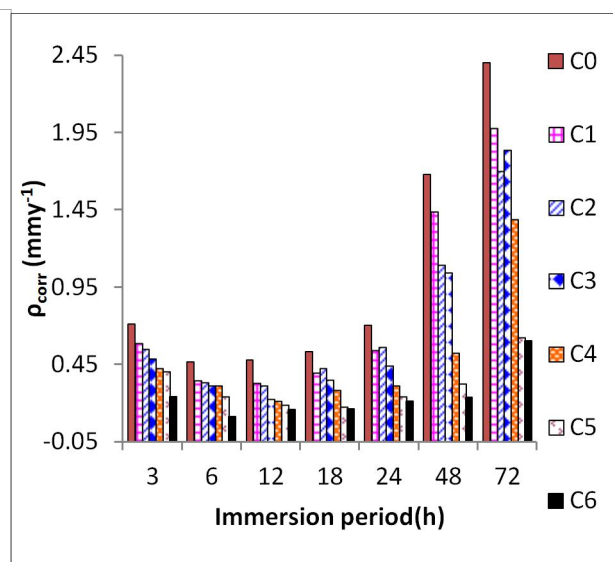


Fig-2 Corrosion rate(ρ_{corr}) v/s Immersion period(h)

It was observed in fig-3 that the inhibition efficiency increases with increasing concentration of the EEPcL. The maximum inhibition efficiency observed was 85.95 % at 1.064 g/L for 48 hours. It was also observed that inhibition efficiency was maximum at highest concentration of the EEPcL at all immersion periods from fig-2 and fig-3 it is seen that at lower immersion period and lower concentration of inhibitor irregular trend is observed in inhibition efficiency but on increasing immersion period and concentration of inhibitor, the inhibition efficiency showed a linear relation with increase in the extract concentration.

This is explained on the basis of formation of monolayer on surface of metal and increased adsorption of inhibitor on the metal surface.

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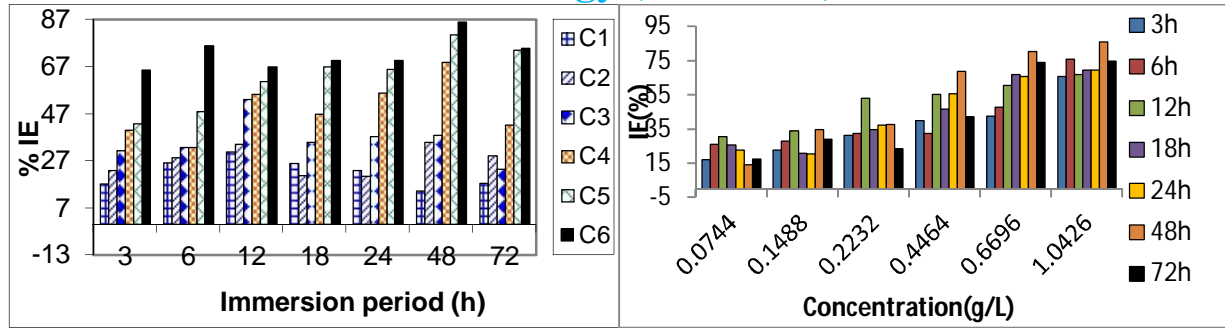


Fig-3 IE (%) v/s Immersion periods of EEPcL at various concentrations Fig-6.21 IE (%) v/s various concentrations of EEPcL at various Immersion periods

B. Effect of immersion time

Inhibition efficiencies were plotted against immersion time as seen from Fig-4. this figure shows that inhibition efficiency increases first up to 18 h for lower concentration (up to 0.2232 g/L). Fig-4 also shows that there is increase in inhibition efficiency at higher inhibitor concentration it increases up to 85.95% at 48 h for 1.064 g/L inhibitor concentration.

This figure shows that over all inhibition efficiency of the extract was increased with increasing immersion time from 3 to 48 h. The increase in inhibition efficiency up to 48 h reflects the strong adsorption of constituents present in the extract on the metal surface, resulting in a more protective layer formed at copper/hydrochloric acid solution interface.

This is an indication that the mass loss is sensitive to the concentration of the lignin extract. This trend is most likely due to the fact that adsorption and surface coverage of the Metal increases with concentration of the inhibitor. Thus the surface of the copper is more effectively separated from the medium. Thus, EEPcL extract effectively inhibit the copper corrosion in 0.5 M hydrochloric acid solutions

C. Adsorption Considerations

Adsorption isotherms are usually used to describe the adsorption process. In order to obtain the adsorption isotherm, the degree of surface coverage (θ) for various concentrations of the inhibitor has been calculated applying the following equation. Assuming that the adsorption of EEPcL extract molecules was mainly due to a monolayer adsorption and ignoring the interaction between the adsorbed molecules, then the Langmuir adsorption isotherm can be employed. Langmuir isotherm was tested for examining fitness to the experimental data. Langmuir isotherm is given by equation

$$C/\theta = 1/K_{ads} + C \quad \dots\dots\dots(4)$$

where θ is the degree of surface coverage, C the molar inhibitor concentration in the bulk solution and K_{ads} is the equilibrium constant of the process of adsorption. The plot of C/θ versus C was linear. Plot of C/θ against C as shown in Fig.-5 gave straight lines which clearly show that copper corrosion inhibition in HCl by EEPcL at room temperature studied obeys Langmuir adsorption isotherm. The correlation coefficient, slopes, and adsorption coefficients obtained from Langmuir isotherm plots are shown in Table-2. The free energy of adsorption, ΔG_{ads} , is associated with water adsorption equilibrium. ΔG_{ads} was calculated from the relation in equation (5).

$$K = 1/(55.5) \exp(-\Delta G_{ads}/RT) \quad \dots\dots(5)$$

The negative value of ΔG_{ads} , here indicate that the adsorption process on copper surface is spontaneous.

The correlation coefficient (R^2) and slope obtained was nearly equal to unity best fit experimental data. The ΔG_{ads} value indicates that the adsorption is physical. If the values are in the order of -20 kJ/mole or less this would indicate a physical adsorption, while those values of -40 kJ/mole or higher imply chemical adsorption which involve charge sharing or a transfer from the inhibitor molecules to the metal surface to form a physical bond.[33-35]

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Table-2 Correlation coefficient, slopes and ΔG_{ads} from langmuir adsorption

Time(h)	Slope	R ²	K _{ad}	ΔG_{ads} (KJmol ⁻¹)
3	1.116	0.943	3.79	-13.4753
6	1.239	0.988	10.99	-16.1589
12	1.221	0.979	10.64	-16.0771
18	1.192	0.989	10.31	-15.998
24	1.086	0.997	12.99	-16.5798
48	1.027	0.999	29.41	-18.6394
72	1.002	0.999	18.52	-17.4738

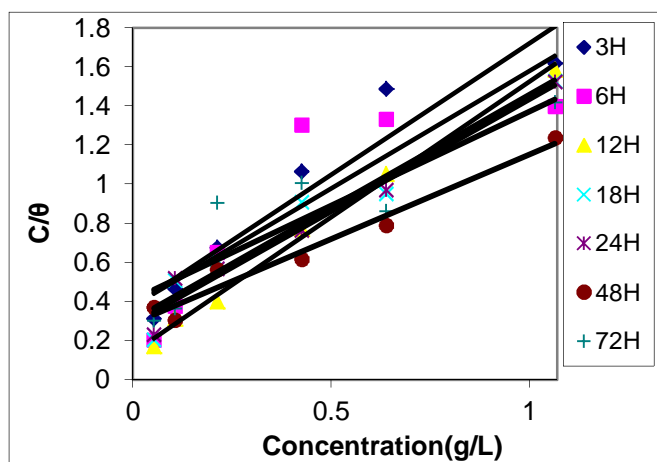


Fig -5 Langmuir isotherm for the adsorption of EEPcL inhibitor on Copper

D. Electrochemical measurements

1) *Potentiodynamic polarization study:* The potentiodynamic polarization curves of Copper in 0.5N HCl solution in the presence and absence of different EEPcL concentration are shown in Fig.-6 electrochemical corrosion kinetic parameters, corrosion potential E_{corr} , cathodic and anodic Tafel slopes (β_c and β_a) and corrosion current density I_{corr} obtained from the tafel polarization curves are listed in Table-3. The %IE is defined as:

$$\%IE = \frac{[I^0_{corr} - (I_{corr})]}{I^0_{corr}} \times 100 \dots\dots\dots(6)$$

Where I^0_{corr} and I_{corr} are the corrosion current density values without and with inhibitor, respectively [36,37]. The polarization resistance (R_p) was calculated from the linear I-E plots. It can be seen from polarization results that the I_{corr} values decreases R_p increases considerably in the presence of inhibitor. The corrosion current density was calculated from Stern-Geary[38-40,] equation in (7)

$$I_{corr} = \frac{\beta_a \beta_c}{2.3R_p (\beta_a + \beta_c)} \dots\dots\dots(7)$$

Table-3 shows that Corrosion current rate decreases with increasing concentration of inhibitor EEPcL. Maximum Inhibition efficiency (%IE) 94.11 was observed at C6. Fig-6 and Table-3 show that both the rate of cathodic and anodic reactions are reduced

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as the concentrations of inhibitor increased by reducing the current densities on both sides of the polarization curves. From electrochemical polarization measurements, it is clear from the results that the addition of inhibitor causes a decrease of the current density. The Fig-6 and table-3 show that there is shift in polarization from negative to more positive potential with increasing concentration of inhibitor indicate that inhibitor acts as the anodic inhibitor in Fig-6 shows the shift in the anodic Tafel slopes of the tests with different concentrations of extract is higher than the shift in the cathodic Tafel slopes; which indicates that the decrease in the oxidation rate of the metal with increasing extract concentrations corresponds to the relative drop in the corrosion rate.

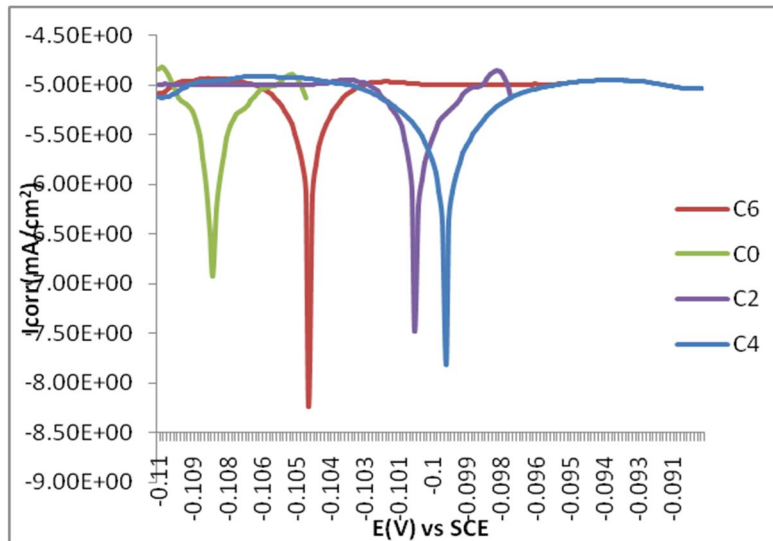


Fig-6 Polarisation curves for copper in absence and presence of EEPcL

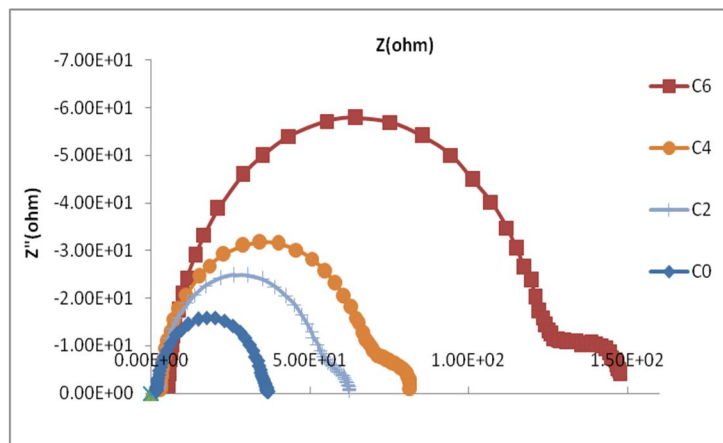
Table-3 Electrochemical and corrosion parameters for Copper in the presence of 0.5N HCl and various concentrations EEPcL

Concentration (g/L)	E_{corr} (-mV)	β_c (-mV/dec)	β_a (mV/dec)	R_p (Ωcm^2)	I_{corr} (mAcm^{-2})	%IE
C0 Blank	134	333.87	166.58	376.25	0.128253	
C2 0.1064	100	25	142.11	784.06	0.011773	90.81985
C4 0.4256	101	52.91	87.78	580.55	0.024690	80.74837
C6 1.064	109	82.25	17.99	848.28	0.007556	94.10853

2) *Electrochemical impedance measurements:* The corrosion study of copper in 0.5 N HCl solutions in the presence of various concentration of EEPcL was investigated by the EIS at 25°C. Nyquist plots are shown in Fig.-7. The impedance response of copper is significantly changed after the addition of EEPcL. The charge transfer resistance (R_{ct}) and the double layer capacitance (C_{dl}) values were derived using the Nyquist plots shown in Fig-7. Impedance parameters are given in Table-4. The inhibition efficiency (IE) is calculated from the equation (3). The R_{ct} values were calculated from the difference in impedance at lower and higher frequencies. The Nyquist plot Fig-7 shows semicircles with single capacitive loop and increasing diameter as the concentration of the plant extract increases. From table we can see that as the concentration of EEPcL is increased there is increase in the value of R_{ct} in 0.5 N HCl solutions indicates that a charge-transfer process is controlling the corrosion of copper. The C_{dl} values shown in the Table-4 are found to decrease with increase in the inhibitor concentration. This shows that the plant constituents are adsorbed on the

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metal surface resulting in a decrease in double layer capacitance [37,41]. The increasing charge transfer resistance R_{ct} values indicate reduced corrosion rate in the presence of the plant extract, confirming that the plant EEPcL shows good corrosion inhibition efficiency.



Conc (g/l)	R_{ct} ($\Omega \cdot \text{cm}^2$)	C_{dl} ($\mu\text{F}/\text{cm}^2$)	$IE_{R_{ct}}(\%)$
C0 Blank	35.217	5.47935×10^{-06}	
C2 0.1064	60.087	3.21145×10^{-06}	41.38998
C4 0.4256	78.54	2.45692×10^{-06}	55.16043
C6 1.064	142.361	1.35547×10^{-06}	75.26219

Fig-7 Nyquist plot of copper immersed in 0.5 N HCl with and without EEPcL

Table-4 Impedance parameters for copper in 0.5N HCl in the presence of EEPcL

D. Surface analysis: SEM studies

Scanning electron micrographs of corroded surface of copper after immersion in 0.5 N HCl solution are shown in Fig.-8 at the magnification of 5000. A large number of grooves and cracks on corroding the surface of the copper coupons are easily visible. Fig.-9 show the scanning electron micrographs of copper coupons exposed to 0.5 N HCl for 72 hours in the presence of C6 1.064g/L EEPcL at magnifications of 5000. The inhibitor molecule covers the metal surface, providing protection against corrosion, as proved by the absence of pits grooves and cracks on the surface. Scanning electron microscopic studies shows that the extent of corrosion of copper decreases with the increase of concentration of the inhibitors.

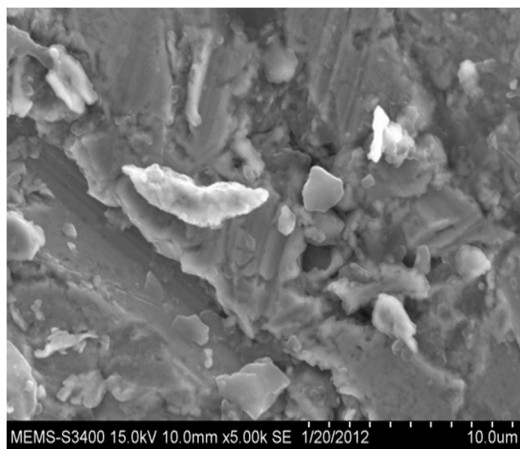


Fig-8 SEM micrograph of polished surface for copper

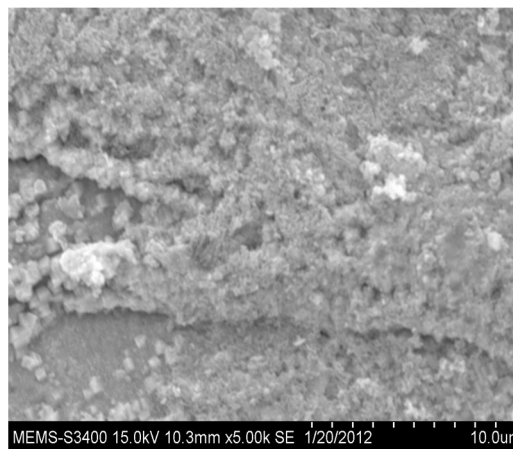


Fig:9 SEM micrograph of copper in presence of inhibitor before its exposure to electrolyte

IV. CONCLUSION

The following conclusions may be drawn from our studies

- A. Ethanolic Extract of Prosopis cineraria Leaves (EEPcL) is found to be an effective inhibitor for copper in acidic medium giving up to 85.85% at 1.064 g/L for 48 hours of immersion period, corrosion rate decreases with increase in inhibitor concentration.
- B. It was concluded that EEPcL is good adsorption inhibitors for the corrosion of copper. The inhibition efficiency increases on

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- increasing concentration of extract.
- C. The values of ΔG_{ads} is less than -20 KJmol^{-1} this concludes that adsorption of EEPcL is spontaneous and physical in nature.. The inhibitor used in the present study followed Langmuir isotherm.
 - D. Maximum IE% from Tafel plot (94.10%), and from EIS studies maximum IE% (75.26%) was obtained .The Inhibition efficiency calculated from impedance and tafel polarization measurements show that on increasing inhibitor concentration %IE also increases.
 - E. Electrochemical potentiodynamic study and EIS results also show that increase in and R_{ct} values and decrease in current density I_{corr} and C_{dl} values confirm that the EEPcL is adsorbed on the copper .
 - F. SEM studies shows that the extent of corrosion of copper decreases with the increase of concentration of the inhibitors.
 - G. The good inhibition efficiency of EEPcL is due to the phytochemical constituents present in it. These phytochemical constituents increase the adsorption behaviour of the inhibitors as these contain multiple bonds and hetero atoms like nitrogen and oxygen From these observations it has been proved that inhibitors EEPcL show an eco-friendly effective corrosion inhibitor for copper corrosion in HCl medium.

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