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RESEARCH ARTICLE

CORROSION INHIBITION OF PURE COPPER USING *PISONIA GRANDIS* LEAVES EXTRACT IN ACID ENVIRONMENT

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ABSTRACT

The inhibition effect of *Pisonia grandis* leaves extract on corrosion of copper in 1.0 N hydrochloric acid was investigated by mass loss measurements with various exposure time and temperature. The present investigation revealed that the percentage of inhibition efficiency is increased with increase of inhibitor concentration and decreased with rise in period of contact. In temperature studies, the observed results reflect that the percentage of inhibition efficiency is decreased with increase of temperature and it suggests that the mechanism follows physical adsorption. The thermodynamic parameters (viz; E_a, ΔH, ΔG_{ads}, ΔS) were evaluated for corrosion inhibition process which suggests that the adsorption is exothermic, spontaneous and physisorption. This inhibitor follows Langmuir adsorption isotherm. The protective layer formed on metal surface was analyzed by using spectroscopic studies viz; UV, FT-IR, XRD, SEM and EDX techniques.

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INTRODUCTION

Historically, copper was the first metal to be used by people. Traditionally it has been one of the important metals used to make coins, along with silver and gold. Copper is easily stretched, moulded, shaped and conducts heat and electricity more efficiently. As a result, copper was important to early humans and continues to be a material of choice for a variety of domestic, industrial, and high-technology applications today. Mostly copper is used to make electrical equipment, heat exchanger tubes and pipes etc. Copper is resistant towards the influence of atmospheric environments however, when contact with aggressive media it is susceptible to corrosion. The prevention of copper corrosion has more attracted many researchers so until now numerous possible inhibitors have been investigated (Khaled, 2009; Faiza *et al.,* 2010; Rahmouni *et al.,* 2007; Amel SEDIK., *et al.,* 2014). The inhibiting action of these inhibitors is attributed to their adsorption to the metal/solution interface. It has been observed that adsorption depends mainly on certain physico-chemical properties of the inhibitor group, like presence of hetero-atom such as N, O and S, as well as multiple bonds in their molecular structure, and also functional groups, aromaticity, electron density at the donor atoms and π-orbital character of donating electrons are assumed to be active centers of adsorption. Most of the corrosion inhibitors are toxic for the environment inorder to overcome these difficulties many scientists used eco-friendly inhibitors.

Thus recent years, several green inhibitors have been used for the prevention of corrosion by most of the investigators. Few examples are *cnidoscolus chayamansa* leaves (Bright *et al.,* 2015), *allium cepa* (Sulaiman *et al.,* 2012), *eucalyptus globules* (Rekkab *et al.,* 2012), *phyllanthus amarus* (Sribharathya *et al.,* 2013), *bitter* leaf powder (Ayeni *et al.,* 2012), carob seed oil (Dris Ben Hmamou., *et al.*, 2012), *bacopa monnieri* (Ambrish Singh., *et al.*, 2012), *citrullus vulgaris* peel (Petchiammal *et al.,* 2012), *albizia lebbeck* seed (Petchiammal *et al.,* 2013), *hibiscus esculenta* leaves (Petchiammal *et al.,* 2013), cannabis (Abd-El-Nabey *et al.,* 2013), *jatropha curcas* (Deepa Rani *et al.,* 2011), *azadirachta indica* seed (Sangeetha *et al.,* 2011), and *eugenia jambolana* (Deepa Rani *et al.,* 2013). Our present investigation is an anti-corrosive effect of *Pisonia grandis* leaves on copper in 1.0 N hydrochloric acid have been investigated with various period of contact and temperature using the mass loss measurements and the Corrosion product on metal surface is analysed by UV, FT-IR, SEM-EDX and XRD spectral studies.

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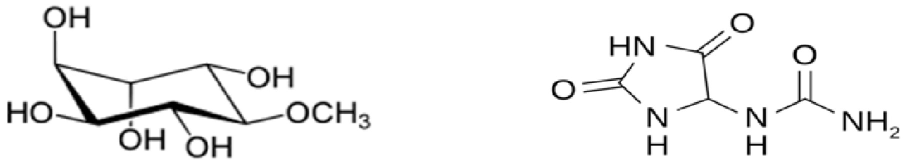
MATERIALS AND METHODS

Properties of *Pisonia grandis* leaves

Pisonia grandis (*Nyctaginaceae*) commonly known as 'Leechai kottai keerai' is distributed throughout India from the Himalayas down to Ceylon. The plant is used traditionally as an antifungal and anti-rheumatic. Leaves are useful in chronic rheumatism, wound healing, analgesic, anti inflammatory, diuretic, hypoglycaemic agent and also used as vegetable. The important phytochemical constituents like flavonoid, glycoside, Pinitol, Allantoin, 4-0-methyl-5'-0-actylmyricelin 3-o-gluco (2 to 1) rhamnoside have been isolated from methanolic extract of the plant leaves. Steroids viz octocosanol, betasitosterol, alphaspinosterol and dulcitol have also been reported from the plant.



(i) Pinitol **(ii) Allantoin**

Fig. 1. Chemical structure of the main active compounds present in PGL extract

Stock solution of *Pisonia grandis* extract

Pisonia grandis (PGL) leaves were collected from the source and dried under shadow for about 7 days, grinded well, then soaked in a solution of ethyl alcohol for about 48 hours. Then it is filtered followed by evaporation in order to remove the alcohol solvent completely and the pure plant leaves extract was collected. From this extract, different concentration of 10 to 5000 ppm stock solution was prepared using double distilled water and used throughout our present investigation.

Specimen preparation

Rectangular specimen of Copper was mechanically pressed cut to form different coupons, each of dimension exactly 20 cm^2 (5x2x2cm) with emery wheel of 80 and 120 and degreased with trichloroethylene, washed with distilled water, cleaned and dried, then stored in desiccators for our present study.

Mass Loss method

In the mass loss measurements, copper specimens in triplicate were completely immersed in 100 ml of the test solution in the presence and absence of the inhibitor. The specimens were withdrawn from the test solutions after immersion of 24 to 360 hours at room temperature and also with different temperature ranges from 303 K to 333 K after an hour. The Mass loss was taken as the difference in weight of the specimens before and after immersion using digital balance with sensitivity of ±1 mg. The tests were performed in triplicate to guarantee the reliability of the results and the mean value of the mass loss is reported. From the mass loss measurements, the corrosion rate was calculated using the following relationship.

$$\text{Corrosion Rate}\left(\text{mmpy}\right) = \frac{87.6 \times W}{DAT} \qquad \ldots\ldots\ldots\ldots\ldots(1)$$

(Where, mmpy = millimetre per year, W = Mass loss (mg), D = Density (gm/cm^3), A = Area of specimen (cm^2), T = time in hours). The inhibition efficiency (%IE) and degree of surface coverage (θ) were calculated using equation (2) and equation (3) respectively.

$$\%\ IE = \frac{W_1 - W_2}{W_1} \times 100 \qquad \ldots\ldots\ldots\ldots\ldots(2)$$

$$\theta = \frac{W_1 - W_2}{W_1} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

(Where W_1 and W_2 are the corrosion rates in the absence and presence of the inhibitor respectively)

RESULTS AND DISCUSSION

Effect of time variation

Corrosion parameters of copper with different concentration of PGL extract at various period of contact in 1.0 N hydrochloric acid are shown in Table 1. It revealed that the corrosion rate was decreased gradually with the addition of inhibitor. The maximum of 81.6% inhibition efficiency was achieved at 5000 ppm of inhibitor concentration after 24 hrs exposure time. This was achieved due to the co-ordination between the copper metal ion and the hetero atom (oxygen, nitrogen) mainly present in the phyto constituent of the inhibitor.

Effect of Temperature

Corrosion parameters of copper in 1.0 N hydrochloric acid containing various concentration of PGL extract with different temperature ranges from 303 to 333 K and the observed values are listed out in the Table 2. The observed result indicates that the corrosion rate decreased with increase of inhibitor concentration with rise in temperature. The maximum of 90.07% inhibition efficiency is achieved at 303K. The value of inhibition efficiency is decreased with rise in temperature is suggested that the physical adsorption mechanism. This result clearly suggests that the adsorption of main active components present in the inhibitor may shield the metal surface at room temperature. However it may be deshielded from the surface with rise in temperature.

Effect of Temperature

Activation energy

The value of activation energy (E_a) for the corrosion of copper in the presence and absence of PGL extract in 1.0 N hydrochloric acid is calculated using the following Arrhenius equation (4) and it's derived form equation (5)

$$CR = A_{exp}\,(-E_a/RT) \qquad \ldots\ldots\ldots\ldots\ldots(4)$$

$$\log\,(CR_2/CR_1) = E_a\,/2.303\,R\,(1/T_1-1/T_2) \qquad \ldots\ldots\ldots\ldots\ldots(5)$$

Where CR_1 and CR_2 are the corrosion rates of Copper at temperatures, T_1 and T_2 respectively.

Heat of adsorption

The heat of adsorption (Q_{ads}) on copper in the presence of inhibitor in acid medium is calculated by the following equation (6).

$$Q_{ads} = 2.303\,R\,[\log\,(\theta_2/1-\theta_2)-\log\,(\theta_1\,/1-\theta_1)]\times(T_2T_1/T_2-T_1)\ ..(6)$$

Table 1. The corrosion parameters of Cu in 1.0 N hydrochloric acid containing various concentration of PGL extract with different exposure time

Con. of inhibitor (ppm)	24 hrs		72 hrs		120 hrs		168 hrs		216 hrs		360 hrs	
	C.R	I.E (%)	C.R	I.E (%)	C.R	I.E (%)	C.R	I.E (%)	C.R	I.E (%)	C.R	I.E (%)
0	9.736	-	3.795	-	2.839	-	2.450	-	2.161	-	1.350	-
10	8.371	14.0	3.368	11.3	2.529	10.9	2.235	8.8	2.012	6.9	1.225	9.3
50	7.434	23.6	3.001	20.9	2.167	23.7	2.075	15.3	1.817	15.9	1.134	16.0
100	6.212	36.2	2.532	33.3	1.911	32.7	1.856	25.2	1.609	25.5	1.008	25.3
500	5.357	44.9	2.112	44.4	1.234	36.5	1.656	32.4	1.410	34.8	0.864	36.0
1000	3.646	62.6	1.473	61.2	1.104	61.1	1.035	57.8	0.962	55.5	0.626	53.6
2000	3.076	68.4	1.338	64.7	0.908	68.0	0.928	62.1	0.876	59.5	0.602	55.4
3000	2.628	73.0	1.107	70.8	0.831	70.7	0.774	68.4	0.772	64.3	0.554	58.9
5000	1.792	81.6	0.789	79.2	0.680	76.1	0.684	72.1	0.672	68.9	0.496	63.3

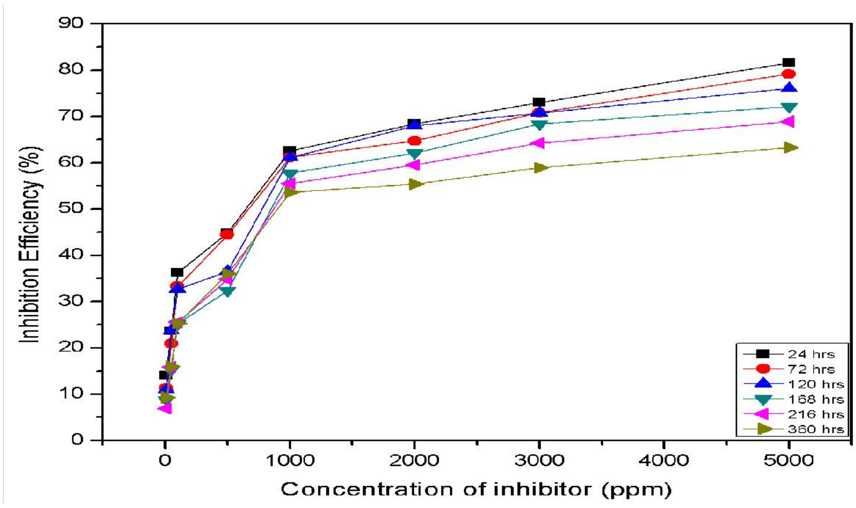


Fig. 1. The corrosion parameters of Cu in 1.0 N hydrochloric acid containing various concentration of PGL extract with different exposure time

Table 2. The corrosion parameters of Cu in 1.0 N hydrochloric acid containing various concentration of PGL extract with different exposure temperature

Con. of inhibitor (ppm)	303 K		313 K		323 K		333 K	
	C.R	% I.E	C.R	% I.E	C.R	% I.E	C.R	% I.E
0	133.26	-	171.97	-	223.41	-	274.85	-
10	115.62	13.24	127.87	25.64	207.24	7.24	250.85	8.73
50	91.62	31.25	90.64	47.29	191.56	14.26	227.33	17.29
100	71.04	46.69	66.63	61.25	143.55	35.7	196.46	28.52
500	51.93	61.03	54.87	68.09	96.03	57.02	152.86	44.38
1000	23.03	82.72	39.68	76.93	54.38	75.66	88.68	67.74
2000	20.09	84.92	32.83	80.90	47.52	78.73	74.47	72.90
3000	17.15	87.13	26.46	84.61	41.64	81.36	64.18	76.65
5000	13.23	90.07	22.54	86.89	33.81	84.87	58.30	78.79

E_a is the activation energy and R is the universal gas constant. The value of activation energy in the ranges (73.547 – 79.860 kJ/mol) for copper in 1.0 N hydrochloric acid containing various concentration of inhibitor. The value of activation energy for blank (73.547 kJ/mol) is lower than in the presence of inhibitors (Table 3), which is clearly indicates that adsorption process is physisorption.

Where, R is the gas constant, $\theta_1$and θ_2 is the degree of surface coverage at temperature and T_1 and T_2 respectively. The Q_{ads} values are ranged from -24.967 to -13.066 kJ/mol (Table 3). These negative values are reflected that the adsorption of PGL extract on copper follows exothermic process. In our present study the Langmuir isotherm is investigated. The Langmuir adsorption isotherm can be expressed by the equation (7) and equation (8) given below

$$\log C/\theta = \log C - \log K \qquad \text{..............(7)}$$

$$\theta = K \ln C \qquad \text{...............(8)}$$

Where, θ is the surface coverage, C is the concentration of the inhibitor solution and K is an adsorption coefficient.

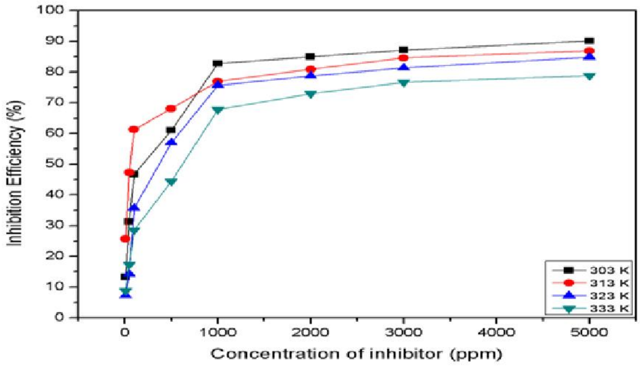


Fig. 2. The corrosion parameters of Cu in 1.0 N hydrochloric acid containing various concentration of PGL extract with different exposure temperature

Table 3. Calculated values of activation energy (E_a) and heat of adsorption (Q_{ads}) of PGL extract on Cu in 1.0 N hydrochloric acid

Concentration of inhibitor (ppm)	% of I.E		E_a (KJ mol^{-1})	Q_{ads} (KJ mol^{-1})
	30°C	60°C		
0	-	-	73.547	-
10	13.24	8.73	73.968	-13.066
50	31.25	17.29	75.084	-21.728
100	46.69	28.52	75.986	-21.992
500	61.03	44.38	76.505	-18.856
1000	82.72	67.74	78.738	-23.041
2000	84.92	72.90	78.422	-20.659
3000	87.13	76.65	78.501	-20.247
5000	90.07	78.79	79.860	-24.967

Table 4. Langmuir adsorption parameters for the adsorption of PGL extract on Cu in 1.0 N hydrochloric acid

Log C	Log C/ θ			
	30°C	40°C	50°C	60°C
1	1.8781	1.5911	2.1403	2.0589
1.6989	2.2041	2.0242	2.5448	2.4612
2	2.3308	2.2129	2.4467	2.5448
2.6989	2.9134	2.8658	2.9429	3.0518
3	3.0823	3.1139	3.1211	3.1692
3.301	3.3720	3.3930	3.4049	3.4383
3.477	3.5369	3.5497	3.5667	3.5926
3.699	3.7443	3.7600	3.7702	3.8025

By plotting the values of log(C/ θ) Vs log C, linear plots are generated (Fig. 3). Inspection of this Fig. 3 reveals that the experimental data fitted with the Langmuir adsorption isotherm, means that there is no interaction between the adsorbed species (i.e., adsorbate and adsorbent). The Langmuir adsorption isotherm is better fit at 313K for Copper (R^2 =0.9931). The equilibrium constant of adsorption of PGL extract on the surface of the metal is related to the free energy of adsorption (ΔG_{ads}) by the following equation (9)

$$\Delta G_{ads} = -2.303\, RT \log (55.5\, K) \qquad \text{................(9)}$$

Where, R is the gas constant, T is the temperature and K is the equilibrium constant of adsorption. The values of intercept (K) obtained from Langmuir adsorption isotherm is substituted in equation (9) and the calculated values of ΔG_{ads} are placed in Table 5.

The negative values of ΔG_{ads} suggested that the adsorption of PGL extract onto metal surface is a spontaneous process and the adsorbed layer is more stable one. Usually the adsorption of free energy involved in a physisorption process ($\Delta G_{ads} < 40$ KJ/mol).

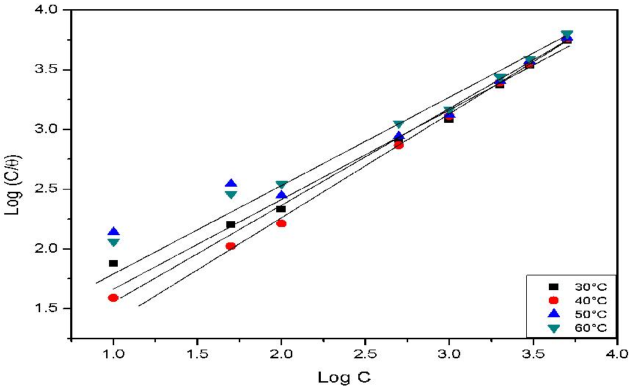


Fig. 3. Langmuir adsorption parameters for the adsorption of PGL extract on Cu in 1.0 N hydrochloric acid

An alternative formula of the Arrhenius equation is the transition state equation

$$CR = RT/Nh \exp(\Delta S/R) \exp(-\Delta H/RT) \qquad \text{..............(10)}$$

Where, h is the Planck's constant, N is the Avogadro's number, ΔS is the entropy of activation, and ΔH is the enthalpy of activation. A plot of log (CR/T) vs. 1000/T should give a straight line (Fig-4) with a slope of (−ΔH/R) and an intercept of [log(R/Nh)) + (ΔS/R)], from which the values of ΔS and ΔH were calculated and listed in the Table-8. The data shows that the thermodynamic parameter (ΔH) of the corrosion of copper in 1.0 N hydrochloric acid solution in the presence of the inhibitors are higher than those in the free acid solution indicating more energy barrier for the reaction in the presence of the inhibitor is attained. The positive value of enthalpy of activation clearly indicates that the endothermic nature of dissolution process is very difficult. The increase of ΔS is generally interpreted with disorder which may take place on going from reactants to the activated complex.

Morphology examination of copper

UV Spectrum

Fig-5(a) & (b) shows that the UV spectrum of the corrosion product on copper in 1.0N hydrochloric acid in the absence of PGL extract and the corrosion product on the surface of copper in the presence of PGL extract in 1.0N HCl respectively. In this spectrum, the one absorption band around 366nm were noticed (Fig-5(a)) but in the presence of inhibitor two bands was appeared (363 and 658 nm). When compared both of these spectrum, one new additional band (ie., 558nm) clearly indicate that the main active compounds coordinate with the metal ion.

Table 5. Langmuir parameters for the adsorption of PGL extract on Cu in1.0 N hydrochloric acid

Adsorption isotherm	Temperature	Slope	Log K	R^2	ΔG_{ads} (KJ mol^{-1})
Langmuir	303	0.7059	1.0407	0.9817	-16.157
	313	0.8234	0.6652	0.9931	-14.440
	323	0.5948	1.4402	0.9528	-19.695
	333	0.6351	1.3578	0.9860	-19.779

Table 6. The relation between log (CR/T) and 1000/T for different concentration of PGL extract on Cu in 1.0 N hydrochloric acid

1000 / T	Log (CR / T)								
	0 ppm	10 ppm	50 ppm	100 ppm	500 ppm	1000ppm	2000ppm	3000ppm	5000ppm
3.300	-0.3567	-0.4184	-0.5195	-0.6299	-0.7660	-1.1191	-1.1785	-1.2472	-1.3599
3.194	-0.2601	-0.3888	-0.5382	-0.6719	-0.7562	-0.8969	-0.979	-1.0729	-1.1426
3.096	-0.1601	-0.1927	-0.2269	-0.3958	-0.5268	-0.7738	-0.8323	-0.8897	-0.9802
3.003	-0.083	-0.1230	-0.1658	-0.2292	-0.3382	-0.5746	-0.6501	-0.7150	-0.7568

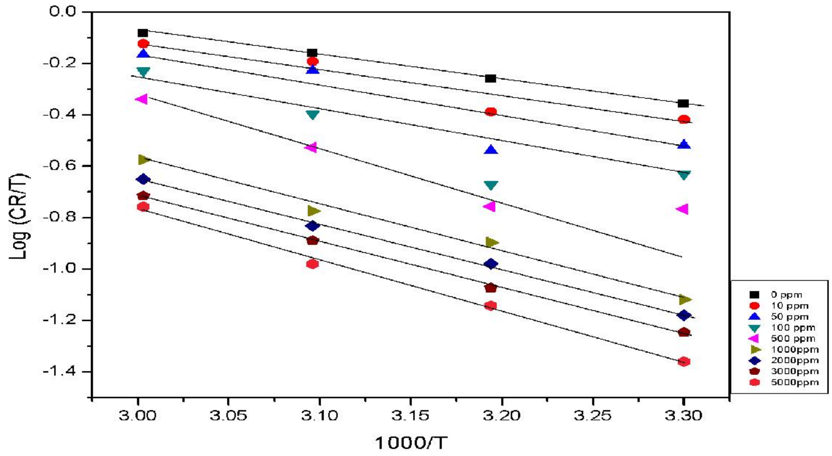


Fig. 4. The relation between log (CR/T) and 1000/T for different concentration of PGL extract on Cu in 1.0 N hydrochloric acid

Table 7. Thermodynamic parameters of Cu in 1.0 N hydrochloric acid obtained from weight loss measurement

S. No.	Concentration of PGL extract (ppm)	ΔH (KJ mol^{-1})	ΔS (KJ mol^{-1})
1	0	7.746	10.309
2	10	9.061	10.667
3	50	11.468	11.358
4	100	12.289	11.518
5	500	12.601	11.519
6	1000	14.771	12.000
7	2000	14.558	11.876
8	3000	14.948	11.939
9	5000	16.560	12.388

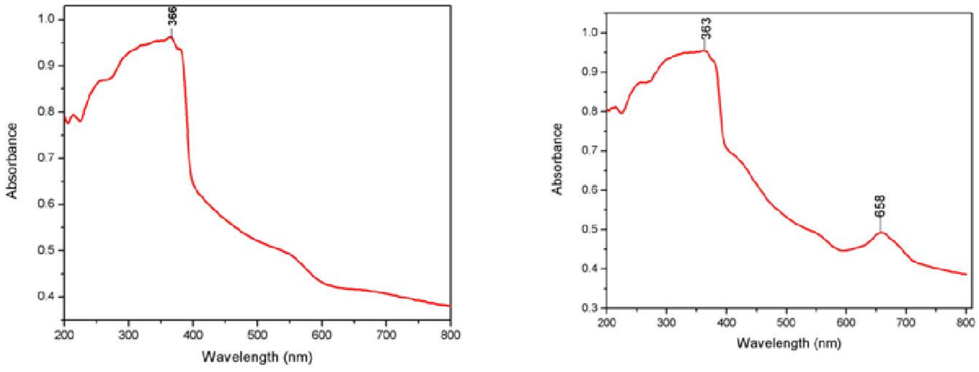


Fig-5(a) **5(b)**

Fig. 5. UV spectrum of (a) Ethanolic extract of PGL, (b) Corrosion product on Cu in the presence of PGL extract in 1.0 N hydrochloric acid

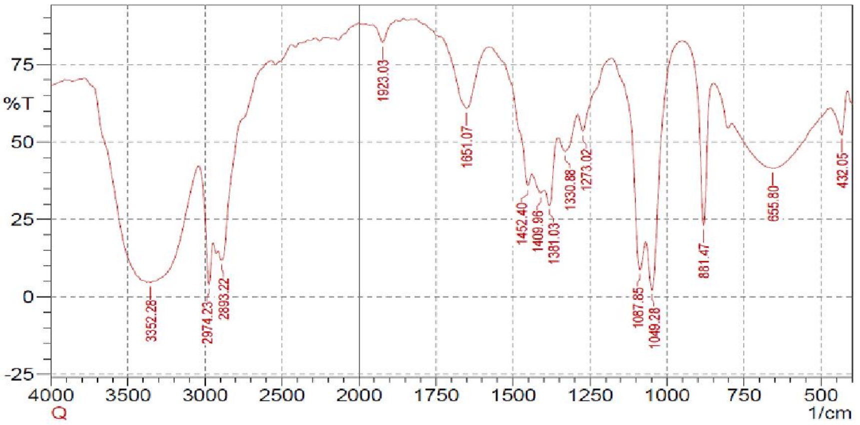


Fig. 6. FT-IR spectrum of ethanolic extract of *Pisonia grandis* (PGL) Leaves

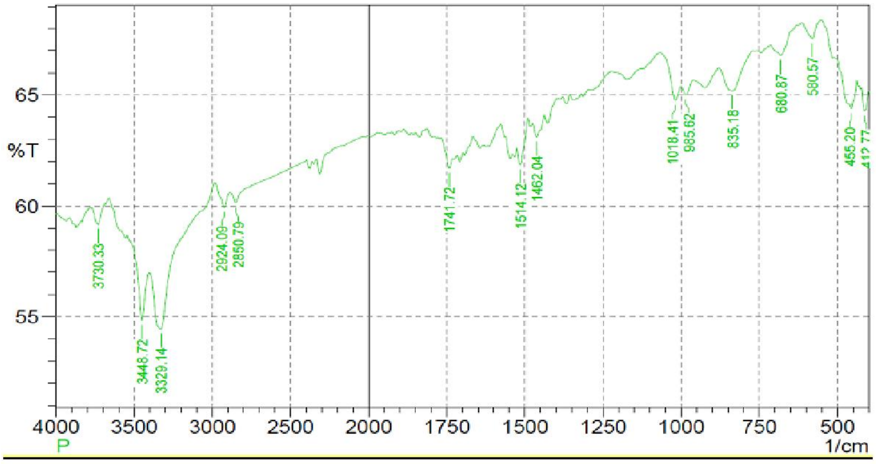


Fig. 7. FT-IR spectrum for the corrosion product on Cu in the presence of PGL extract with 1.0N hydrochloric acid

FT-IR Spectrum

The Fig-6 and 7 reflects that the FT-IR spectrum of the ethanolic extract of PGL inhibitor and the corrosion product on copper in the presence of inhibitor in 1.0 N hydrochloric acid respectively. On comparing both these spectra, the prominent peak is shifted from 3352.28 to 3329.14 cm^{-1} for =N-H group, the frequency at 1087.85cm^{-1} is attributed to –C-N stretching is shifted to 1018.41 cm^{-1} and the -O-H frequency is shifted from 2974.23 to 2924.09 cm^{-1} and the frequency is attributed to -C-H bending is shifted from 1452.40 to 1462.04 cm^{-1}. Thus the FT-IR spectra support the fact that the corrosion inhibition of PGL inhibitor on copper in 1.0 N hydrochloric acid may be the adsorption of active molecule in the inhibitor and surface of the metal.

EDX Spectrum

EDX spectroscopy was used to determine the elements present on the copper surface in the absence and presence of inhibitor. Fig- 8 and 9 represents the EDX spectra for the corrosion product on copper metal surface in the absence and presence of optimum concentrations of PGL extract in 1.0 N hydrochloric acid. In the absence of inhibitor, the spectrum may concluded that the existence of chlorine due to the formation of metal chloride and in addition to this, spectrum consists of oxygen atom is present in the form metal oxide.

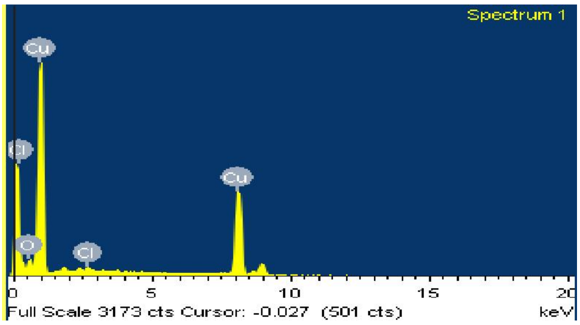


Fig.8. EDX spectrum of the corrosion product on Cu surface in 1.0 N hydrochloric acid

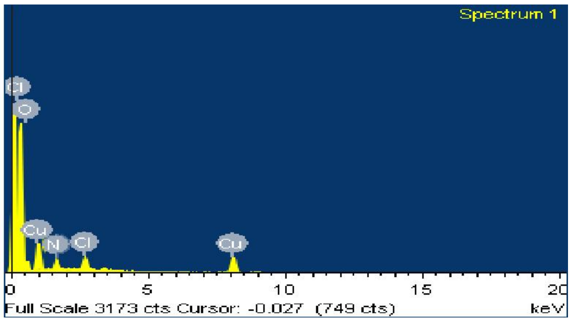


Fig.9. EDX spectrum of the corrosion product on Cu in the presence of PGL extract in 1.0 N hydrochloric acid

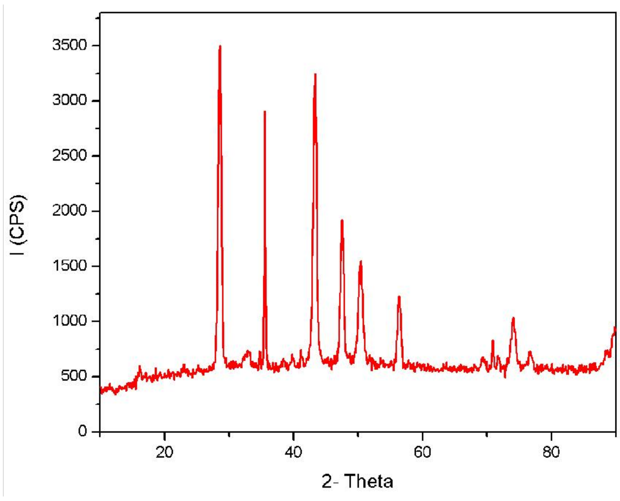
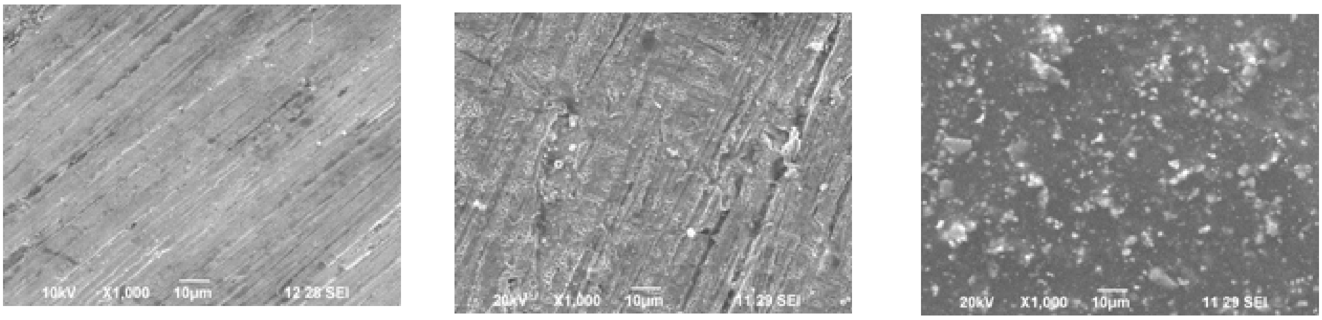


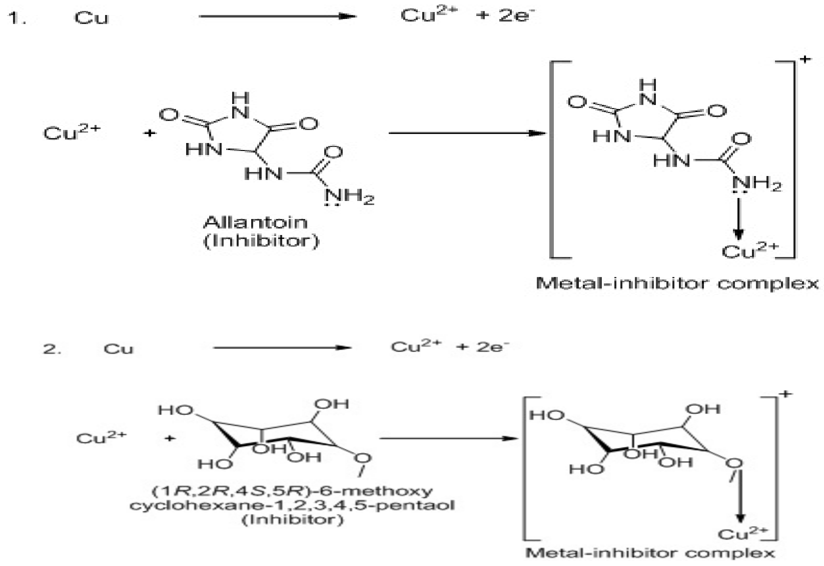
Fig.10. XRD- Analysis of corrosion product on Cu in the presence of PGL extract in 1.0 N hydrochloric acid



11(a) 11(b) 11(c)

Fig. 11. SEM micrographs of copper in (a) fresh copper, (b) without inhibitor, 0 ppm (c) with inhibitor, 1000 ppm

Proposed Mechanism



XRD-Analysis

However, in the presence of the optimum concentrations of the inhibitors, nitrogen and oxygen atoms are found to be present in the corrosion product on the metal surface. It clearly indicates that these hetero atoms present in the inhibitor may involve the complex formation with metal ion during the adsorption process and prevent the further dissolution of metal against corrosion.

The corrosion products are scrapped from the copper surface in the presence of PGL extract inhibitor is examined by XRD studies are shown in Fig-10. It reveals that the composite may be mainly combine with a rich amount of Copper azide $Cu(N_3)_2$ and $Cu(ClO_4)_2$, CuCl and CuO with inhibitor.

SEM- Analysis

The Surface morphology of Copper was examined by scanning electron microscopy and the images are represented in Fig 11(a-c). Fig. 11(a) reflects that the copper sample before immersion shows smooth surface, but after careful inspection of Fig. 11(b) indicates that the surface of metal has number of pits and cracks are visible in the surface. Fig. 11(c) is clearly indicate that, the maximum surface area and metal may protect with the film formation, can prevent further dissolution.

Conclusion

From our present investigation the following conclusions can be drawn. The *Pisonia grandis* (PGL) leaves extract is suitable corrosion inhibitor for copper in acid environment. The adsorption of the inhibitor on the surface of copper is exothermic, spontaneous process and its follows physisorption. Langmuir isotherm are best described the adsorption characteristics of the inhibitor. The value of E_a, Q_{ads} and ΔG_{ads} also confirm the spontaneous physical adsorption of inhibitor on the metal surface. The UV, FT-IR, XRD and SEM-EDX spectral studies also clearly supports that the co-ordination between the active molecules present in the inhibitor and the metal ion from the surface.

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