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Dissolution behaviour of Cu-Zn alloy in acid containing *Pandanus amaryllifolius* leaves extract

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Abstract
Dissolution behaviour of Cu-Zn alloy in 1.0 N hydrochloric acid containing ethanolic extract of *Pandanus amaryllifolius* leaves has been investigated by mass loss measurements at various exposure time and temperature. The inhibition efficiency increased with increase of inhibitor concentration but decreased with rise in temperature. The values of activation energy (E_a), heat of adsorption (Q_{ads}) and free energy of adsorption (ΔG_{ads}) is support that the adsorption is spontaneous, exothermic and physisorption. Inhibitor obeys both Langmuir and Temkin adsorption isotherms. The corrosion product has been analysed by using UV, FT-IR, XRD, and SEM-EDX spectroscopic techniques.

Keywords: Mass loss, *Pandanus amaryllifolius*, Adsorption isotherm, Cu-Zn alloy

1. Introduction
Cu-Zn alloy are of considerable importance as they form the backbone of modern industries. Cu-Zn alloy has been widely used in various industries for shipboard, power plant condensers and petrochemical, marine applications and in heat exchanger tubes, for example, in desalination, cooling water systems and power generation. Cu-Zn alloy materials are relatively noble and used for many applications have superior physical and mechanical properties, although it could present particular corrosion problems such as dezincification and pitting corrosion in chloride-containing solutions leading to structural failure. The dissolution behaviour of Cu-Zn alloy was studied in different environments such as sea water (Ravichandran R., *et al*., 2005 [12, 13]; Hostis V.L., *et al*., 2003 [9]; Joseph Raj X., *et al*., 2011[10]), NaCl (EL-Mahdy G.A., *et al*., 2013[8]; Ravichandran R., *et al*., 2005[12,13]; Ravichandran R., *et al*., 2004[14]; TulinKıyak., 2007[15]), HNO_3 (Abed Y., *et al*., 2004 [1]; Mihit M., *et al*., 2006[11]) by many investigators. The existing data shows that most of the organic inhibitors act by adsorption on the metal surface. The adsorption of inhibitors occurs through a compound containing heteroatom such as nitrogen, oxygen and sulphur. These compounds which are adsorbed on the metallic surface may block the active corrosion sites. Even though many synthetic organic compounds showed good anticorrosive activity, most of them are highly toxic to both human beings and the environment. Now a days the safety and environmental issues of corrosion inhibitors arisen in industries has always been a global concern. Most of the investigators have more interest only to chosen various part of green plant extract as corrosion inhibitors for metal in various environment such as *Vitisvinifera* (Deepa Rani P., *et al*., 2010)[5], *Ocimum tenuiflorum* (Deepa Rani P., *et al*., 2011)[6,7], *Punica Granatum* (Deepa Rani P., *et al.*, 2011)[6,7]*, Emblica* officinalis (Deepa Rani P., *et al*., 2010) [5], *Jatropha curcas* (Deepa Rani P., *et al.*, 2011)[6,7] and *Vinca rosea* (Deepa Rani P., *et al.*, 2011)[6, 7]. In our present investigation, we have chosen eco-friendly inhibitor, a green approach to prevent environmental pollution by harmful organic chemicals. The influence of *Pandanus amaryllifolius* leaves extract on Cu-Zn alloy in 1.0 N hydrochloric acid using mass loss measurements with different time and temperature has been studied. The characterization of corrosion product on Cu-Zn alloy in the presence of green inhibitor is also reported by UV, FT-IR, SEM-EDX and XRD spectral studies.

2. Materials and Methods

2.1. Properties *of Pandanus amaryllifolius* Leaves

The leaf of *Pandanus amaryllifolius* (PAL) commonly known as pandan, is often used to give a refreshing, fragrant flavour to South-East Asian dishes. Besides its culinary value, pandan leaves are used in the perfume industry and also medicinally as a diuretic, cardio-tonic and anti-diabetic. The leaves contain various alkaloids, quercetin, carotenoids, tocopherols, tocotrienols and essential oils. The major compound responsible for the unique pleasant aroma of *P. Amaryllifolius* is 2-acetyl-1-pyrroline. Additional 30 aroma components have been found of which the main ones are hexanal, 2-hexenal, 3-methyl pyridine, 2-penten-1-ol, nonanal, benzaldehyde and linalool. It has been further reported that the ethanol extract of the leaves exhibited excellent heat-stable antioxidant property. However, it might be more practical to optimize the repellent components of pandan leaves for use in some instances. As the repellent is non-toxic, there are also good prospects to develop cockroach-repellent spray cans for domestic use.

2.2. Stock solution of *Pandanus amaryllifolius* extract

Pandanus amaryllifolius (PAL) leaves were collected from the source and dried under shadow for about 48 hours, 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 1000 ppm stock solution was prepared using double distilled water and used throughout our present investigation.

2.3. Specimen preparation

Rectangular specimen of Cu-Zn alloy 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.

2.4. Mass Loss method

In the mass loss measurements, Cu-Zn alloy 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 different with 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.

$$\text{Corrosion Rate}(\text{mmpy}) = \frac{87.6 \times W}{DAT} \quad \text{-------------------- (1)}$$

[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.

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

$$\theta = \frac{W_1 - W_2}{W_1} \quad \text{-------------------- (3)}$$

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

3. Results and Discussion

3.1. Effect of Time Variation

The dissolution behaviour of Cu-Zn alloy in 1.0 N hydrochloric acid environment containing various concentration of ethanolic extract of *Pandanus amaryllifolius* (PAL) leaves at different time (24 hrs to 360 hrs) and temperature (303 K to 333 K) are investigated by mass loss method. The observed corrosion parameters are placed in Table-1 and 2 (Fig-1 and 2). It is clearly indicates that the percentage of inhibition efficiency increased with increase of PAL concentration and exposure time. The maximum of 74.2% inhibition efficiency is achieved even after 360 hrs exposure time. The maximum inhibition efficiency may be due to the adsorption of the active plant constituent (mainly 2-acetyl-1-pyrroline, hexanal, 2-hexenal, 3-methyl pyridine, 2-penten-1-ol, nonanal benzaldehyde and linalool) on the Cu-Zn alloy metal surface.

Table1: The corrosion parameters of Cu-Zn alloy in 1.0 N hydrochloric acid containing various concentration of PAL extract with different exposure time

Concentration 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	0.9815	-	0.4409	-	0.6359	-	1.0425	-	1.3372	-	1.0683	-
10	0.8962	8.69	0.3912	11.3	0.5590	12.1	0.8878	14.9	1.1830	11.5	0.9232	13.6
50	0.7468	23.9	0.3414	22.6	0.5121	19.5	0.7956	23.7	1.0218	23.6	0.8037	24.8
100	0.6615	32.6	0.2845	35.5	0.3883	38.9	0.6949	33.3	0.8654	35.3	0.5989	43.9
500	0.5121	47.8	0.2063	53.2	0.2774	56.4	0.4237	59.4	0.5903	55.9	0.3812	64.3
1000	0.3841	60.8	0.1565	64.5	0.2134	66.4	0.2987	71.4	0.3699	72.3	0.2760	74.2

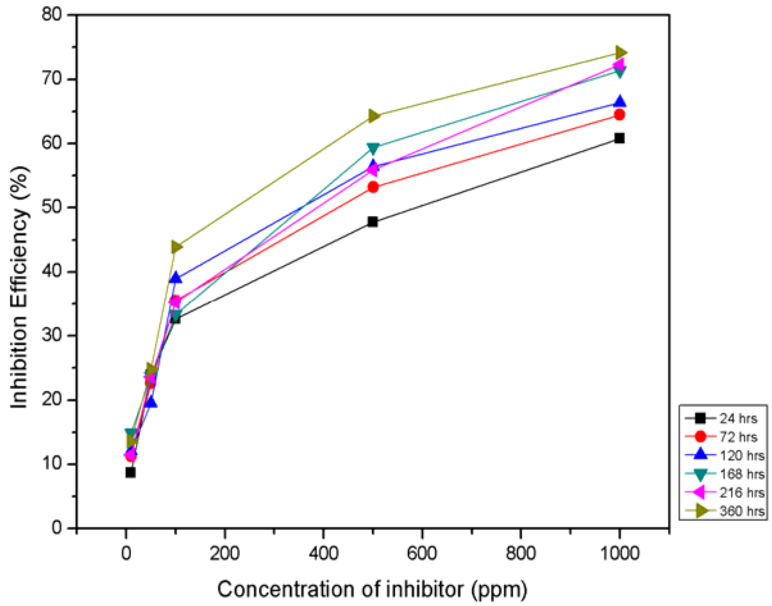


Fig1: Inhibition efficiency of Cu-Zn alloy containing various concentration of PAL extract at different exposure time in 1.0 N hydrochloric acid.

3.2. Effect of Temperature

Table 2: The corrosion parameters of Cu-Zn alloy in 1.0 N hydrochloric acid containing various concentration of PAL extract with different exposure temperature

Concentration 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	25.093	-	32.262	-	38.408	-	41.480	-
10	20.996	16.33	29.189	9.24	34.310	10.67	38.919	6.17
50	18.948	24.49	26.117	19.05	32.775	14.67	36.871	11.11
100	15.875	36.73	22.020	31.75	29.189	23.99	34.311	17.28
500	8.194	67.35	12.290	61.91	15.363	60.00	24.069	41.98
1000	5.121	79.59	8.194	74.60	10.242	73.33	14.339	65.43

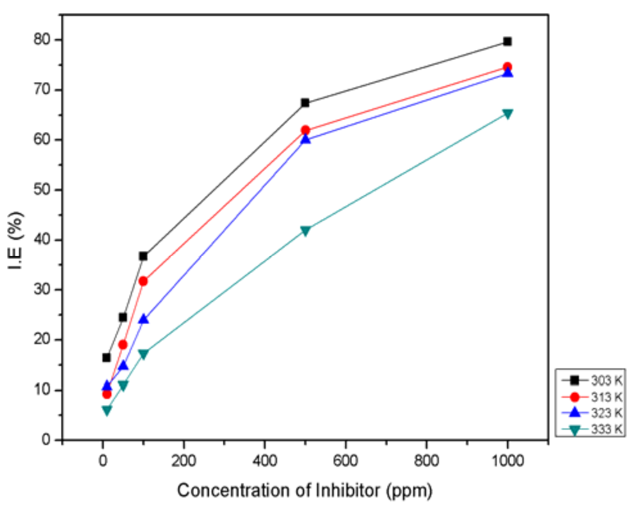


Fig 2: The corrosion parameters of Cu-Zn alloy in 1.0 N hydrochloric acid containing various concentration of PAL extract with different exposure temperature

Corrosion parameters of Cu-Zn alloy in 1.0 N hydrochloric acid containing various concentration of PAL extract with different ranges of temperature from 303 to 333 K and the observed values are listed in Table-2. The observed result indicates that the corrosion rate decreased with increase of inhibitor concentration and increases with rise in temperature from 303 to 333K. The maximum of 79.59% inhibition efficiency is achieved at 303K. However value of inhibition efficiency is decreased with rise in temperature beyond this suggested that the physical adsorption mechanism.

3.3. Activation Energy

The values of activation energy (E_a) for the corrosion of Cu-Zn alloy in the presence and absence of PAL extract is calculated using the following Arrhenius equations (4) and its derived form equation (5).

$$CR = A_{exp} (-E_a/RT) \text{ -------------------- } (4)$$

$$\text{Log} (CR_2/CR_1) = E_a / 2.303\, R\, (1/T_1 - 1/T_2) \text{ ----------------- } (5)$$

Where, CR_1 and CR_2 are the corrosion rates of Cu-Zn alloy at temperatures T_1 and T_2 respectively, E_a is the activation energy and R is the universal gas constant. The value of activation energy for the blank (71.707 kJ/mol) is lower than in the presence of inhibitors (Table-3), which is clearly indicates that the adsorption process is physisorption.

3.4. Adsorption Studies

The values of heat of adsorption (Q_{ads}) on Cu-Zn alloy specimen in the presence of inhibitor is arrived by the following equation (6)

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

Where, R is the gas constant, $\theta_1$and θ_2 are the degree of surface coverage at temperatures T_1 and T_2 respectively. The calculated Q_{ads} values (Table-3) are ranged from -30.428 to -20.221 kJ/mol. This negative value indicates that the adsorption of PAL extract on the surface of Cu-Zn alloy is exothermic process.

Table3: Calculated values of activation energy (E_a) and heat of adsorption (Q_{ads}) of PAL extract on Cu-Zn alloy 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	-	-	71.707	-
10	16.33	6.17	72.660	-30.428
50	24.49	11.11	73.064	-26.661
100	36.73	17.28	73.936	-28.586
500	67.35	41.98	76.488	-29.301
1000	79.59	65.43	76.089	-20.221

This adsorption isotherm of PAL extract on Cu-Zn alloy surface proceeded according to the following equation (7)

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

By plotting the values of log(C/ θ) Vs log C, linear plots are generated (fig-3). Inspection of this figure reveals that the experimental data fitted with the Langmuir adsorption isotherm, means that there is no interaction between the adsorbed species. The inhibitor also obeys Temkin adsorption isotherm which is represented in fig-4. The equilibrium constant of adsorption of PAL extract on the surface of the Cu-Zn alloy is related to the free energy of adsorption (ΔG_{ads}) by the following equation (8).

$$\Delta G_{ads} = -2.303\, RT \log (55.5\, K) \text{ -------------------- (8)}$$

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 and Temkin adsorption isotherm is substituted in equation (8) and the calculated values of ΔG_{ads} are placed in Table-7. The negative values of ΔG_{ads} suggested that the adsorption of PAL extract onto Cu-Zn alloy surface is a spontaneous process and the adsorbed layer is more stable one.

Table4: Langmuir adsorption parameters of PAL extract on Cu-Zn alloy in 1.0 N hydrochloric acid

Log C	Log C/ θ			
	30°C	40°C	50°C	60°C
0	1.787	2.0343	1.9718	2.2097
1.6989	2.3099	2.4191	2.5325	2.6533
2	2.4349	2.4983	2.6199	2.7625
2.6989	2.8706	3.007	2.9208	3.076
3	3.0990	3.1659	3.1347	3.1842

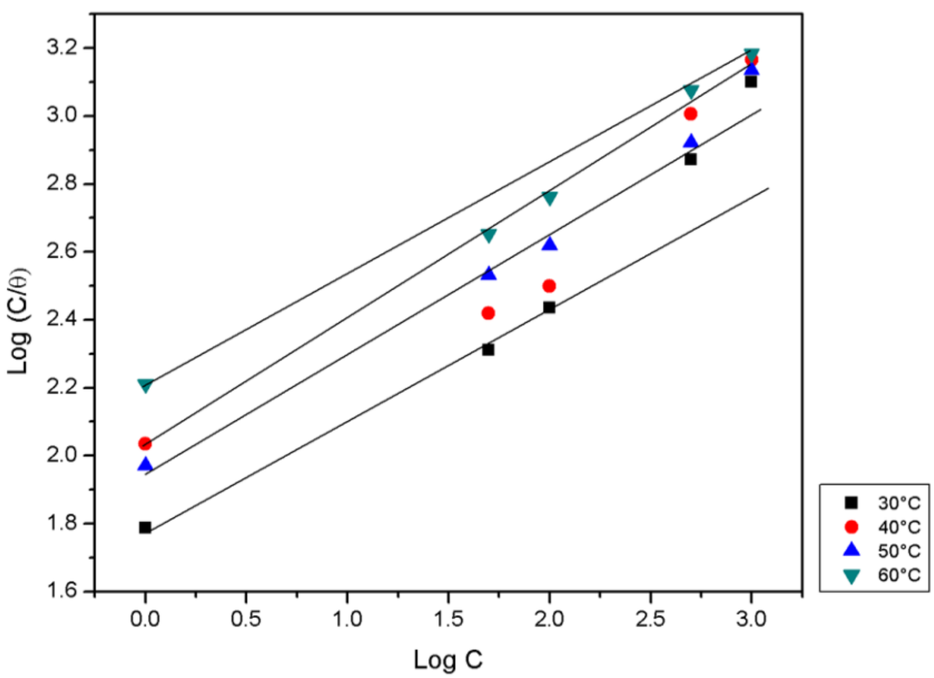


Fig 3: Langmuir adsorption parameters of PAL extract on Cu-Zn alloy in 1.0 N hydrochloric acid

Table5: Temkin adsorption parameters of PAL extract on Cu-Zn alloy in 1.0 N hydrochloric acid

Log C	θ			
	30°C	40°C	50°C	60°C
1	0.1633	0.0924	0.1067	0.0617
1.6989	0.2449	0.1905	0.1467	0.1111
2	0.3673	0.3175	0.2399	0.1728
2.6989	0.6735	0.6190	0.6000	0.4198
3	0.7959	0.7460	0.7333	0.6543

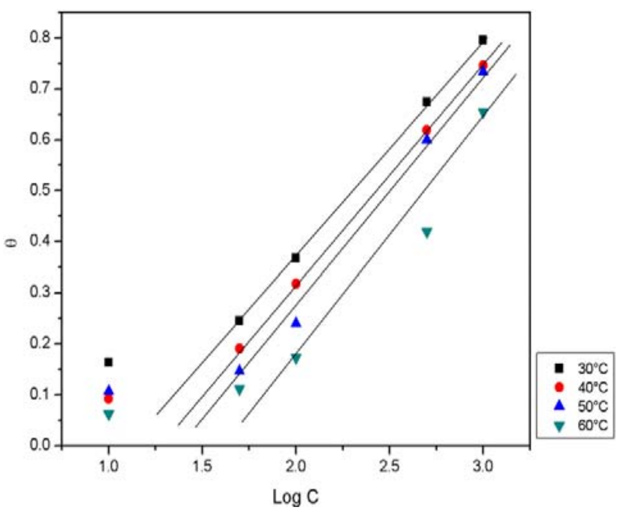


Fig4: Temkin adsorption parameters of PAL extract on Cu-Zn alloy in 1.0 N hydrochloric acid

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

CR=RT/Nhexp (ΔS/R) exp (-ΔH/RT) -------------------- (9)

Where, h is the Planck's constant, N 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-5) 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 observed data shows that the thermodynamic parameters (ΔS and ΔH) for the corrosion of Cu-Zn alloy in 1.0 N hydrochloric acid solution in the presence of the inhibitors are higher than those in the free acid solution indicating that the more energy barrier for the reaction in the presence of the inhibitor is attained.

Table6: The relation between log (CR/T) and 1000/T for different concentration of PAL extract on Cu-Zn alloy in 1.0 N hydrochloric acid

1000 / T	Log (CR / T)					
	0 ppm	10 ppm	50 ppm	100 ppm	500 ppm	1000 ppm
3.300	-1.0819	-1.1593	-1.2039	-1.2807	-1.5680	-1.7721
3.194	-0.9868	-1.0303	-1.0786	-1.1527	-1.4060	-1.5821
3.096	-0.9248	-0.9738	-0.9937	-1.0440	-1.3227	-1.4988
3.003	-0.9046	-0.9323	-0.9558	-0.9870	-1.1409	-1.3659

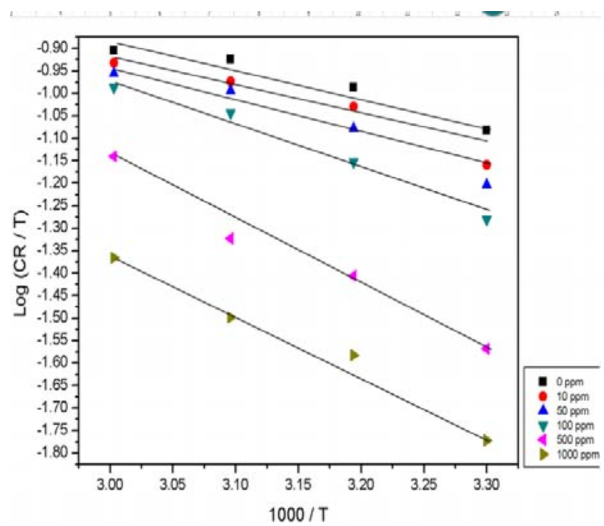


Fig 5: The relation between log (CR/T) and 1000/T for different concentration of PAL extract on Cu-Zn alloy in 1.0 N hydrochloric acid

Table7: Langmuir and Temkin parameters of PAL extract on Cu-Zn alloy in 1.0 N hydrochloric acid

Adsorption isotherm	Temperature(K)	Slope	Log K	R^2	ΔG_{ads}(KJ mol^{-1})
Langmuir	303	0.4252	1.7011	0.9371	-19.999
	313	0.3725	1.9248	0.8711	-21.988
	323	0.3736	1.9337	0.9773	-22.747
	333	0.3247	2.1668	0.9732	-24.937
Temkin	303	0.3340	-0.2457	0.9271	-8.694
	313	0.3425	-0.3192	0.9406	-8.541
	323	0.3362	-0.3338	0.8630	-8.723
	333	0.2903	-0.3198	0.8246	-9.082

Table 8: Thermodynamic parameters of Cu-Zn alloy in 1.0 N hydrochloric acid obtained from weight loss measurement

S. No.	Concentration of PAL extract (ppm)	ΔH (KJ mol^{-1})	ΔS (KJ mol^{-1})
1	0	5.0294	8.142
2	10	6.2441	8.183
3	50	7.0126	8.209
4	100	8.3520	8.254
5	500	11.4550	8.347
6	1000	10.9703	8.309

4. Morphology Examination

4.1. UV Spectrum

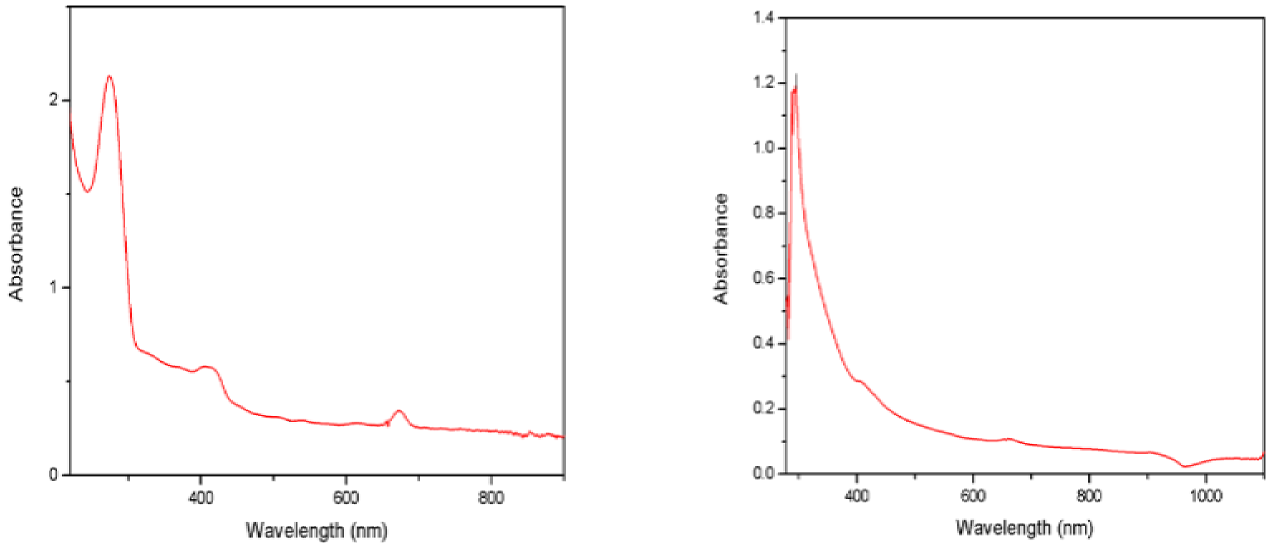


Fig 6(a) 6(b)

Fig6: UV spectrum of (a) Ethanolic extract of PAL, (b) Corrosion product on Cu-Zn alloy in the presence of PAL extract in 1.0 N hydrochloric acid

Fig-6(a) and 6(b) shows that the UV visible spectrum of the ethanolic extract of PAL and corrosion product on Cu-Zn alloy in the presence of PAL extract in 1.0 N hydrochloric acid environment respectively. On comparing both these spectra, a band at 274 and 538 nm is shifted to higher wavelength region (294,655 nm) i.e., bathochromic shift or red shift. However one band at 672 nm shifted to shorter wavelength region (660 nm) i.e., hypsochromic shift or blue shift in the presence of inhibitor. The observed results concluded that the co-ordination between the hetero atoms (oxygen, nitrogen) present in the inhibitor and surface of the metal ion.

4.2. FT-IR Spectrum

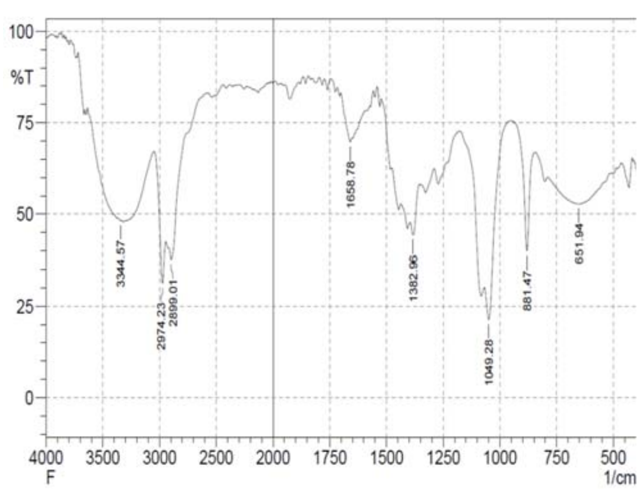


Fig7: FT-IR spectrum of ethanolic extact of PAL extract

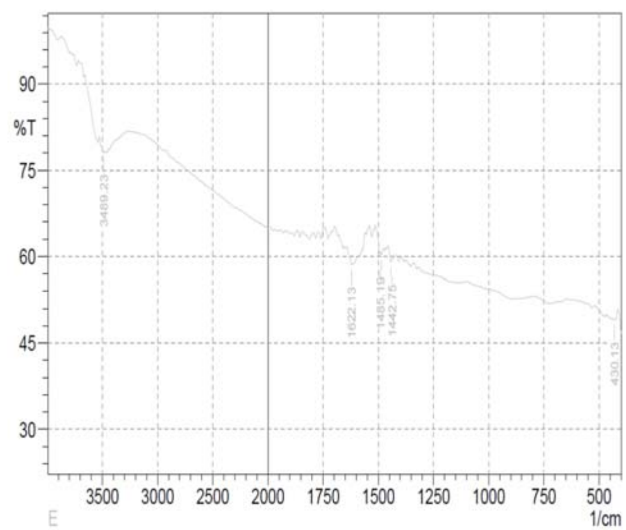


Fig 8: FT-IR spectrum for the corrosion product on Cu-Zn alloy in the presence of PAL extract with 1.0 N hydrochloric acid

The Fig-7 and 8 reflects that the FT-IR spectrum of the ethanolic extract of PAL inhibitor and the corrosion product on Cu-Zn alloy in the presence of inhibitor in 1.0 N hydrochloric acid respectively. On comparing these spectra, the prominent peak is shifted from 3344.57 to 3489.23 cm-1 for –NH stretching in secondary amine group and the frequency at 1658.78 cm^{-1} is attributed to C=C stretching is shifted to 1622.13 cm^{-1} indicates that the binding between metal ion and the inhibitor takes place through amino group. Thus the FT-IR spectra support the fact that the corrosion inhibition of PAL inhibitor on Cu-Zn alloy in 1.0 N hydrochloric acid may be the adsorption of active molecule in the inhibitor and surface of the metal ion.

4.3. EDX Spectrum

EDX spectroscopy was used to determine the elements present on the Cu-Zn alloy surface in the absence and presence of inhibitor. Fig-9 and 10 represents the EDX spectra for the corrosion products on metal surface in the absence and presence of optimum concentrations (1000 ppm) of PAL extract in 1.0 N hydrochloric acid. In the absence of inhibitor molecules, the spectrum may concluded that the existence of chlorine in the environment due to the formation of metal chloride. 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 molecules may involve the complex formation with metal ion during the adsorption process and prevent the further dissolution of metal against corrosion.

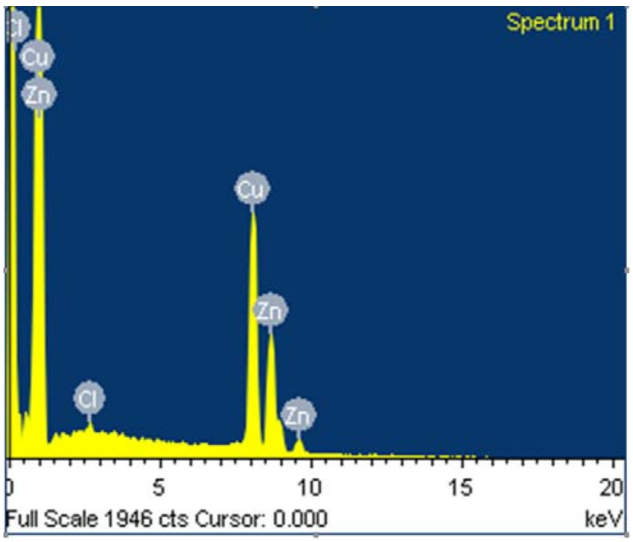


Fig 9: EDX spectrum of the corrosion product on Cu-Zn alloy surface in 1.0 N hydrochloric acid

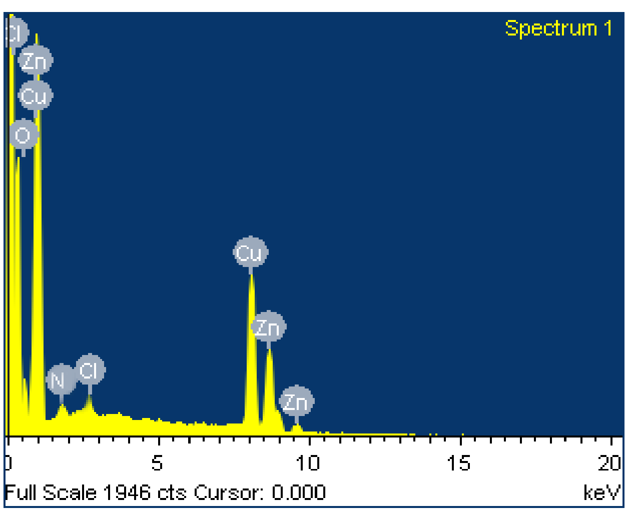


Fig 10: EDX spectrum of the corrosion product on Cu-Zn alloy in the presence of PAL extract in 1.0 N hydrochloric acid

4.4. XRD – Analysis

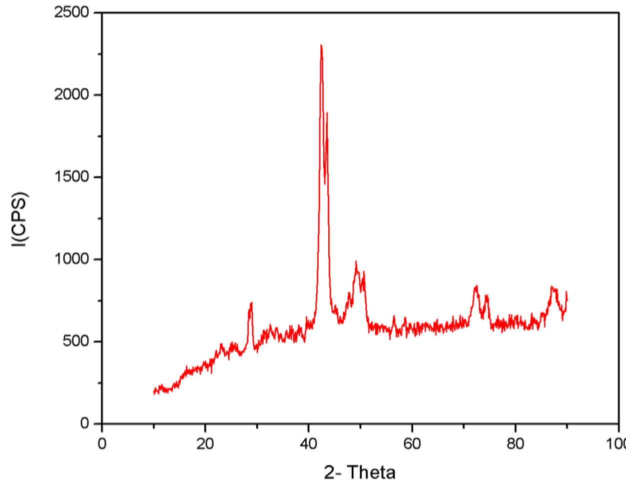


Fig 11: XRD- spectrum of corrosion product on Cu-Zn alloy in the presence of PAL extract in 1.0 N hydrochloric acid

The Corrosion products scraped from the Cu-Zn alloy formed on the metal surface examined by XRD studies in the presence of inhibitor as shown in Fig-11. It is reveal that the film may be mainly combine with a rich amount of [$CuCl_2(H_2O)_2$], [$CuCl_2$], [$Cu_2Cl(OH)_3$] and Zinc salt such as [$ZnCl_2$], [Zn_3N_2] etc with inhibitor.

4.5 SEM– Analysis

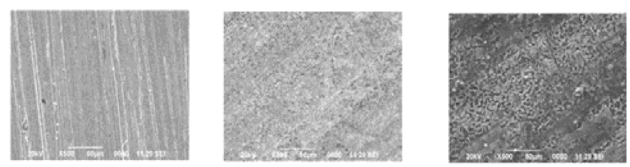
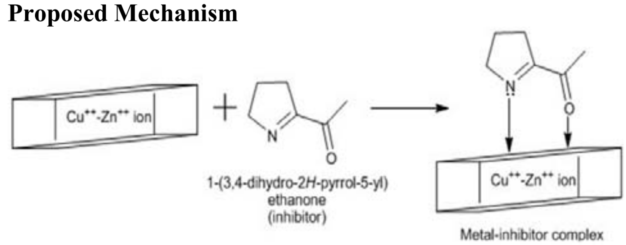


Fig-12(a) **12(b)** **12(c)**

Fig12: SEM micrographs of (a) Polished Cu-Zn alloy surface, (b) without inhibitor, 0 ppm (c) with inhibitor, 500 ppm

A SEM micrograph of the polished Cu-Zn alloy surface before immersion in 1.0 N hydrochloric acid solution is shown in Fig-12(a). This micrograph shows that the surface is smooth and without pits. The SEM micrographs of the corroded Cu-Zn alloy in 1.0 N hydrochloric acid solution are shown in Fig-12(b), showed that the metal surface has number of pits are visible by noticed. However in the presence of the inhibitor Fig-12(c), the entire surface of the metal covered with the film formation might be concluded that the adsorption can efficiently inhibit the corrosion of Cu-Zn metal alloy.

Proposed Mechanism



5. Conclusion

From our present investigation the following conclusions can be drawn.

The *Pandanus amaryllifolius* (PAL) leaves extract is used as a suitable corrosion inhibitor for Cu-Zn alloy in 1.0 N hydrochloric acid environment. The dissolution behaviour of Cu-Zn alloy in 1.0 N hydrochloric acid environment is depending upon the exposure time, temperature and zinc content present in the alloy. Corrosion inhibition may be due to the adsorption of the plant constituents on the metal surface of Cu-Zn alloy and achieve maximum of 74.2% I.E. for time variant and 79.59% I.E. for temperature variant. The adsorption of the inhibitor on the surface of Cu-Zn alloy is exothermic, spontaneous process and is consistent with the mechanism of physical adsorption. Langmuir and Temkin isotherms are best described the adsorption characteristics of the inhibitor. The corrosion product over the surface of Cu-Zn alloy in the presence of PAL extract is characterized by UV, FT-IR, XRD and SEM-EDX spectral studies and may conform that the complex film formed between the metal ion and the active groups present in the inhibitor molecule.

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