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Corrosion inhibition efficacy of ethanolic extract of *mimusops elengi* leaves (MEL) on copper in natural sea water

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Abstract
The inhibitive nature of alcoholic extract of *Mimusops elengi* leaves (MEL) extract on Copper in Natural Sea Water is investigated by mass loss measurement with various contact period and temperature. The observed results suggest that the percentage of inhibition efficiency is increased with increase of inhibitor concentration. The maximum % of inhibition efficiency is attained at 86.84% after 120 hours exposure time. In temperature studies, the adsorption of inhibitor on the metal surface follows the mechanism of chemical adsorption. The calculated values of ΔH_{ads} and ΔG_{ads} suggest that the adsorption may be endothermic and spontaneous process. The inhibitor obeys Langmuir adsorption isotherm. The protective film formed on the metal surface in the presence and absence of inhibitor is analysed by UV, FT-IR, EDX and SEM spectral studies.

Keywords: Spontaneous, UV, FT-IR, SEM and EDX

1. Introduction

Copper is an important metal that has a wide range of applications due to its good properties. It is used in electronics, for production of wires, sheets, tubes, and also to form alloys. Copper is resistant toward the influence of atmosphere environments and using chemicals, however, it is known that in aggressive media it is susceptible to corrosion. The use of copper corrosion inhibitors in such conditions is necessary since no protective passive layer can be expected. The possibility of the copper corrosion prevention has attracted many researchers so until now numerous possible inhibitors have been investigated. But the use of chemical inhibitor has been found to be more expensive, toxic, non bio-degradable and harmful to living things[1]. Hence there is a big problem for researchers to search the non-toxic, eco-friendly corrosion inhibitors. Recent years, several green inhibitors have been used for the prevention of corrosion by most of the investigators. Few examples are *Allium Cepa* [2], *Eucalyptus globulus* [3], *Phyllanthus amarus* [4], *bitter* leaf powder [5], Carob seed oil [6], *Bacopa monnieri* [7], *Citrullus Vulgaris* peel [8], *Albizia lebbeck* seed [9], *Hibiscus Esculenta* leaves [10], *Jatropha curcas* [11], *Eugenia Jambolana* [12]. In continuous of our research work, our present investigation is the corrosion inhibitive efficacy of *Mimusops elengi leaves* on Copper in Natural Sea Water have been studied with various periods of contact and temperature using the mass loss measurements and the corrosion product on metal surface is

2. Materials and methods

2.1 Specimen preparation

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

2.2 Preparation of Mimusops elengi (MEL) Extract:

About 3 Kg of *Mimusops elengi,* leaves was collected from in and around Western Ghats and then dried under shadow up 5 to 10 days. Then it is grained well and finely powdered, exactly 150g of this fine powder was taken in a 500ml round bottom flask and a required quantity of ethyl alcohol was added to cover the fine powder completely, and left it for 48 hrs. Then the resulting paste was refluxed for about 48 hrs, the extract was collected and the excess of alcohol was removed by the distillation process. The obtained paste was boiled with little amount of activated charcoal to remove impurities, the pure plant extract was collected and stored.

2.3 Properties of *Mimusops elengi* leaf:

Mimusops elengi belongs to *sapotaceae* family and it is an annual herbaceous climbing plant with a long history of traditional medicinal uses in many countries, especially in tropical and subtropical regions. The peel extract of this plant is used to regulate thyroid function and glucose metabolism. The important phytochemicals compound present in this plant is flavonoids, alkaloids, saponins, triterpenes [13]

2.4 Mass loss measurement

In the mass loss measurements on Copper in triplicate were completely immersed in 50ml of the test solution in the presence and absence of the inhibitor. The metal specimens were withdrawn from the test solutions after an hour at 313K to 333K and also measured 24 to 360 hrs at room temperature. The Mass loss was taken as the difference in weight of the specimens before and after immersion using LP 120 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 relationship.

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

Where, mmpy = millimeter 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 the following equations.

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

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

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

2.5 Adsorption studies

2.5.1 Activation energy

The activation energy (E_a) for the corrosion of Copper in the presence and absence of inhibitors in natural sea water environment was calculated using Arrhenius theory. Assumptions of Arrhenius theory is expressed by equation (4).

$CR = A\exp(-E_a/RT)$ -------------- (4)

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

Where CR_1 and CR_2 are the corrosion rate at the temperature T_1 (313K) and T_2 (333K) respectively.

2.5.2 Heat of adsorption

The heat of adsorption on the surface of various metals in the presence of plant extract in natural sea water environment is calculated by the following equation.

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

Where R is the gas constant, θ_1 and θ_2 are the degree of surface coverage at temperatures T_1 and T_2 respectively.

2.5.3 Langmuir adsorption isotherm

The Langmuir adsorption isotherm can be expressed by the following Equation-4.10 is given below [38-40].

$\log C/\theta = \log C - \log K$ -----------------(7)

Where θ is the degree of surface coverage, C is the concentration of the inhibitor solution and K is the equilibrium constant of adsorption of inhibitor on the metal surface.

2.5.4 Free energy of adsorption

The equilibrium constant of adsorption of various plant extract on the surface of copper, mild steel and zinc is related to the free energy of adsorption ΔG_{ads} by equation (8).

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

Where R is the gas constant, T is the temperature, K is the equilibrium constant of adsorption.

3. Result and Discussion

The dissolution behavior of Copper in Natural sea water environment containing in the absence and presence of MEL extract with various exposure times (120to 480 hrs) are shown in Table-1. The observed values are clearly indicates that in the presence of MEL extract the value of corrosion rate decreased from 0.1547to 0.0203mmpy (120 hrs) and 0.0691 to 0.0162 mmpy (480 hrs) with increase of inhibitor concentration from 0 to 500 ppm. The maximum of 86.84% of inhibition efficiency is achieved at 500ppm inhibitor concentration even after 120 hrs exposure time. This achievement is mainly due to the presence of active phytochemical constituents present in the inhibitor molecule which is adsorbed on the metal surface and shield completely to prevent further dissolution from the aggressive media of chloride ion(cl^-). The observation of maximum surface coverage clearly suggests that the hetero atoms (such as nitrogen and oxygen) present in the inhibitor molecules can able to bind with the metal ions from the surface, very strongly and protect the metal ions from corrosive environment.

Table 1: The corrosion parameters of Copper in Natural Sea Water containing different concentration of MEL extract after 120 to 480 hours exposure time

Exposure time (hrs)	Conc. of Inhibitor (ppm)	Mass loss (mg)	Corrosion rate (mmpy)	Surface coverage (θ)	Inhibition Efficiency (%)
120	0	38	0.1547	--	--
	10	30	0.1222	0.2105	21.05
	50	16	0.0651	0.5783	57.83
	100	12	0.0488	0.6842	68.42
	250	08	0.0325	0.7895	78.95
	500	05	0.0203	0.8684	86.84
240	0	46	0.0936	--	--
	10	17	0.0346	0.6304	63.04
	50	14	0.0285	0.6956	69.56
	100	15	0.0305	0.67.39	67.39

	250	12	0.0244	0.73.91	73 91
	500	08	0.0162	0.8261	82.61
360	0	50	0.0678	--	--
	10	23	0.0312	0.5399	53.99
	50	16	0.0217	0.6800	68.00
	100	16	0.0217	0.6800	68.00
	250	12	0.0162	0.7600	76.00
	500	08	0.0108	0.8400	84.00
480	0	68	0.0691	--	--
	10	39	0.0397	0.4265	42.65
	50	29	0.0295	0.5735	57.35
	100	25	0.0254	0.6323	63.23
	250	18	0.0183	0.7353	73.53
	500	16	0.0162	0.7647	76.47

3.1 Temperature Studies

The inhibition efficiency of MEL extract on copper in Natural Sea Water at 313 to 333K is shown in Table-2. In the absence of inhibitor, the corrosion rate increased from 7.3325 to 30.3080 at 313 to 333K, but in the presence of inhibitor, the value of corrosion rate decreased from 7.3325to 2.9330 mmpy and 30.3080 to 3.9107mmpy with increase of inhibitor concentration at 313and 333K. The maximum 87.09% inhibition efficiency is achieved even at the temperature range of 333K respectively. The value of inhibition efficiency is increased with rise in temperature (313-333K) is due to the adsorption of active inhibitor molecules on the metal surface is higher than the desorption process. It is clear shows that the inhibitor follows chemisorptions process.

Table 2: The corrosion parameters of Copper in Natural Sea Water containing different concentration of MEL extract at 313 to 333 K

Temperature (K)	Conc. of Inhibitor (ppm)	Mass loss (mg)	Corrosion rate (mmpy)	Surface coverage (θ)	Inhibition Efficiency (%)
313	0	15	7.3325	--	--
	10	13	6.3549	0.1333	13.33
	50	10	4.8883	0.3333	33.33
	100	11	5.3772	0.2666	26.66
	250	7	3.4218	0.5333	53.33
	500	6	2.9330	0.6000	60.00
323	0	34	16.6205	--	--
	10	26	12.7092	0.2352	23.52
	50	25	12.2209	0.2647	26.47
	100	15	7.3325	0.5588	55.88
	250	14	6.8437	0.5882	58.82
	500	7	3.4218	0.79411	79.41
333	0	62	30.3080	--	--
	10	32	15.6428	0.4837	48.37
	50	29	14.1763	0.5325	53.22
	100	19	9.2879	0.6935	69.35
	250	18	8.7991	0.7096	70.96
	500	8	3.9107	0.8709	87.09

3.2 Activation energy

The values of Corrosion rate obtained from the mass loss measurements are substituted in equation (4) and the values of activation energy (E_a) are presented in Table-3 The observed values are ranged from 61.4971 to 12.4667 kJ/mol for copper in Natural Sea Water containing various concentration of inhibitor. The average value of E_a obtained from the blank (61.4971kJ/mol) is greater than that in the presence of inhibitor and indicated that there is a strong chemical co-ordination bond between the MEL inhibitor molecules and the copper ions from the surface.

Table 3: Calculated values of Activation energy (E_a) and heat of adsorption (Q_{ads}) of MEL extract on Copper in Natural Sea Water environment

S.No	Conc. of inhibitor(ppm)	% of I.E		E_a (KJmol^{-1})	Q $_{ads}$ (KJmol^{-1})
		30^o	60^o		
1.	0	--	--	61.4931	--
2.	10	13.3386	48.3714	39.0326	59.4512
3.	50	26.6619	53.2218	46.1452	18.9723
4.	100	33.3372	69.3517	23.6818	37.1517
5.	250	53.3314	70.9629	40.9276	8.39719
6.	500	60.0000	87.0913	12.4632	8.08728

3.3 Heat of adsorption:

The value of heat of adsorption (Q_{ads}) on copper in Natural Sea Water containing various concentration of MEL extract is calculated using Equation (6) and the values of Q_{ads} are ranged from 59.4512 to 8.08728 kJ/mol (Table-3). These positive values are reflected that the adsorption of MEL extract on copper follows endothermic process.

3.4 Adsorption studies
The adsorption isotherm is a process, which are used to investigate the mode of adsorption and it characteristic of inhibitor on the metal surface. In our present study the Langmuir adsorption isotherm is investigated. The straight line observed in Fig- 1 suggests that the inhibitor follows Langmuir adsorption isotherm.

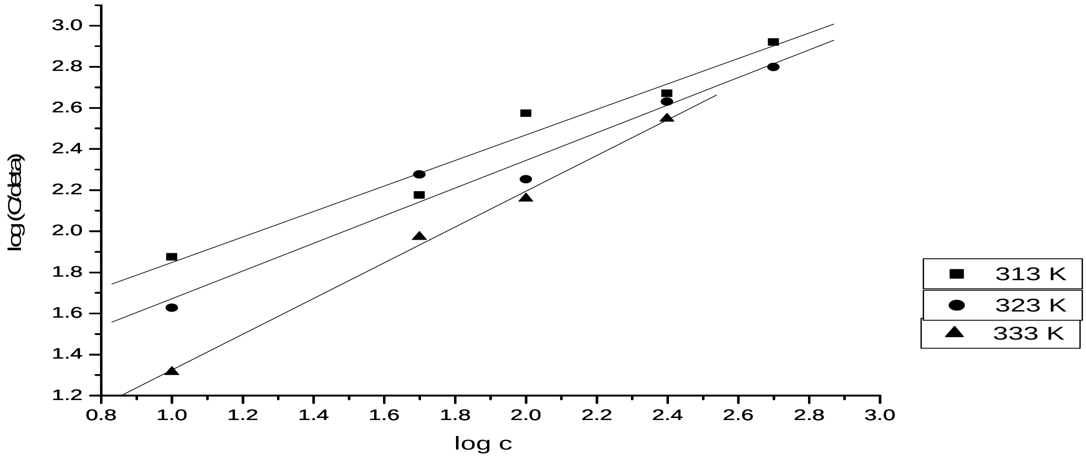


Fig 1: Langmuir isotherm for the adsorption of MEL inhibitor on Copper in Natural Sea Water Environment

3.4.1Free energy of adsorption:
The standard free energy of adsorption (ΔG_{ads}) can be calculated using the Equation- (8) and the observed negative values are (Table-4) ensure that the spontaneity of the adsorption process and the stability of the adsorbed layer is enhanced[21-23].

Table 4: Langmuir adsorption parameters for the adsorption of MEL inhibitor on Copper in Natural Sea Water

Adsorption isotherms	Temperature (Kelvin)	Slope	K	R^2	ΔG_{ads} kJ/mol
Langmuir	313	0.6205	1.2275	0.9631	-10.987
	323	0.6724	0.9998	0.9643	-10.787
	333	0.8698	0.9814	0.9962	-11.069

3.5 Thermodynamics parameters
The another form of transition state equation which is derived from Arrhenius equation is shown below

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

where h is the Planck's constant, N the Avogadro's number, ΔS the entropy of activation, and ΔH the enthalpy of activation. A plot of log (CR/T) Vs. 1000/T gives a straight line (Fig. 2) 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 Table-5. The positive value of enthalpy of activation clear 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.

Table 5: Thermodynamic parameters of copper in Natural Sea Water obtained from weight loss measurements

S.No	Concentration of MEL (ppm)	ΔH (kJ mol−1)	ΔS (J k−1 mol−1)
1	0	9.8027	-67.4090
2	10	16.1425	-48.2976
3	50	19.0216	-39.6012
4	100	9.1096	-71.3158
5	250	16.6911	-48.5221
6	500	4.2548	-82.5214

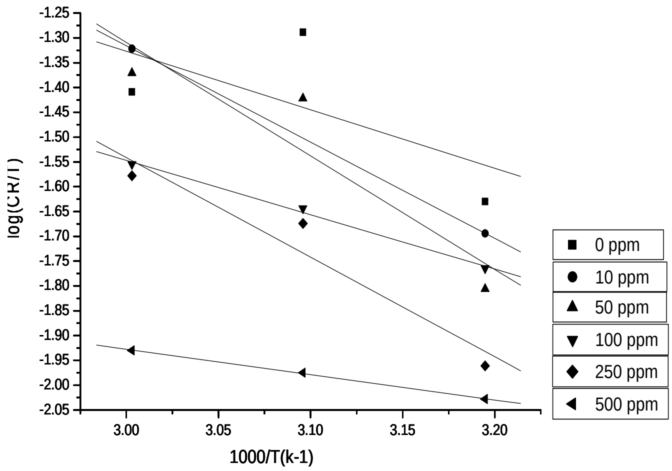
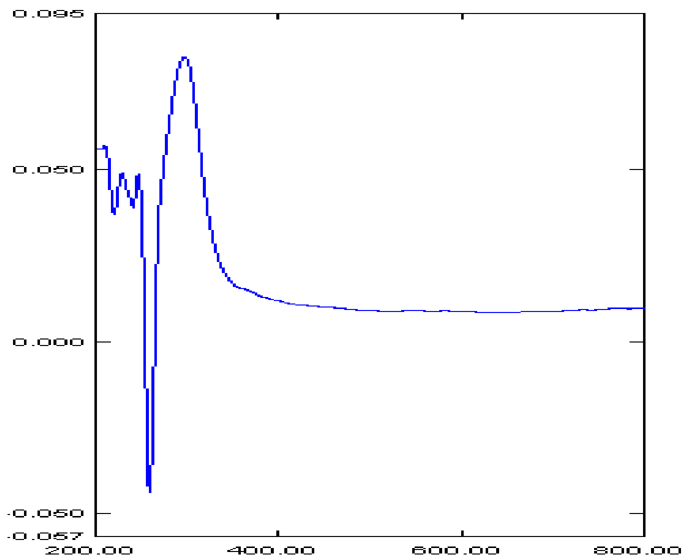
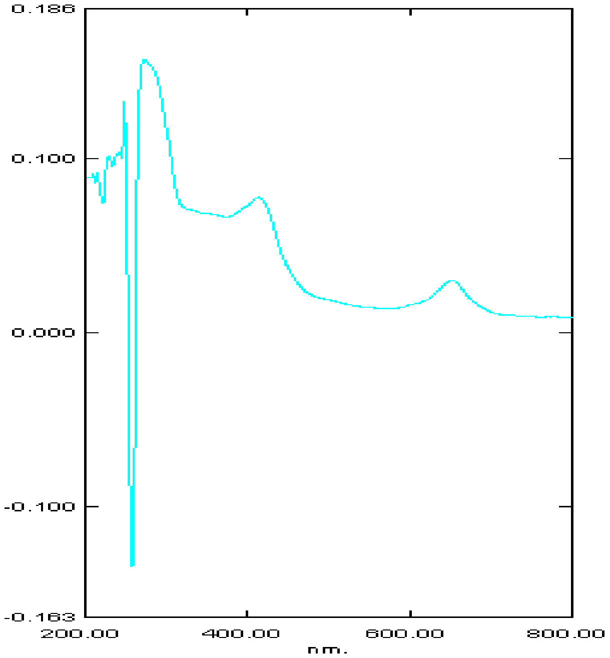


Fig 2: The relation between log (CR/T) and 1/T for different concentrations of MEL extract

3.6 Morphology studies
3.6.1 UV spectrum



3(a)



3(b)

Fig 3: UV spectrum of ethanolic extract of MEL (a), the corrosion product on copper in Natural Sea Water in the presence of MEL extracts (b)+

Fig - 3 (a) & (b) shows that the UV visible spectrum of ethanolic extract of MEL and the corrosion product on the surface of copper in the presence of MEL extract in natural sea water respectively. In this spectrum, the one absorption band around 302nm were noticed (Fig3(a)) and in the presence of inhibitor three bands was appeared (302,450, 690nm) which indicates the band is shifted to longer wavelength region (Bathochromic shift (or) Red shift). The change of absorption band may confirmed that the strong co-ordination bond between the active group present in the inhibitor molecules and the ions from the metal surface [25].

3.6.2 FT-IR studies of MEL extract on Copper surface in Natural Sea Water:

The Fig - 4 and 5 reflect that the FTIR spectrum of the ethanolic extract of inhibitor and the corrosion product on copper in the presence of MEL extract in Natural Sea Water. On comparing both of the spectra the prominent peak such as, the -O-H stretching frequency for alcohol is shifted from 3386.39 to 3413.39 cm^{-1}, the C-N stretching in amine is shifted from 1107 to 1123.23 cm^{-1}. 1401.03cm^{-1} corresponds to C-H stretching frequency is shifted to 1388.78 cm^{-1}. These results also confirm that the FTIR spectra support the fact that the corrosion inhibition of MEL extract on Copper in Natural Sea Water may be the adsorption of active molecule in the inhibitor and the surface of metal[24].

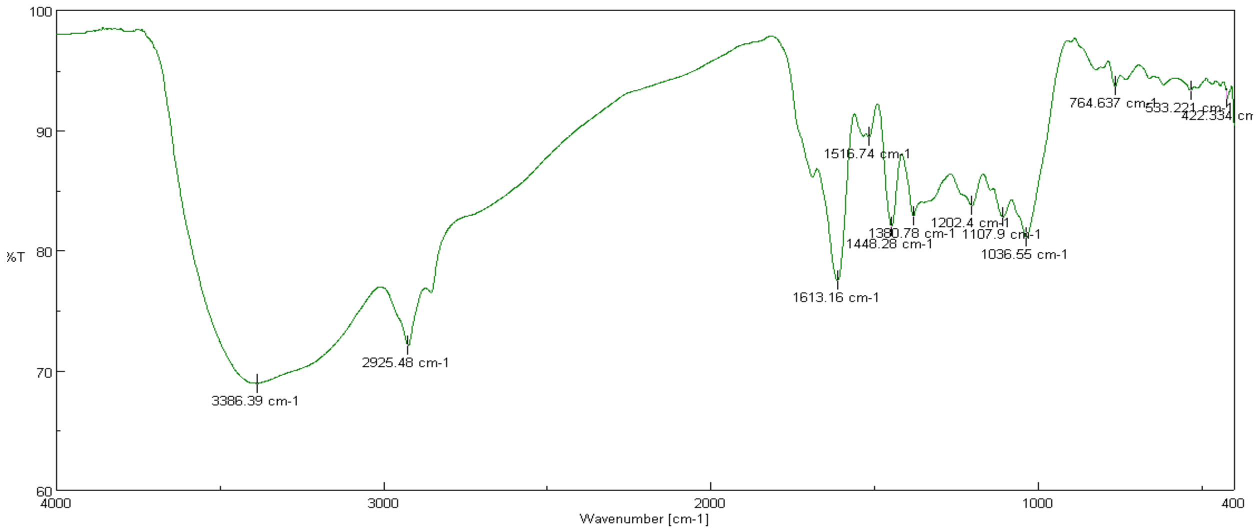


Fig 4: FT-IR spectrum of ethanolic extact of *Minuso pieiengi* laeves (MEL)

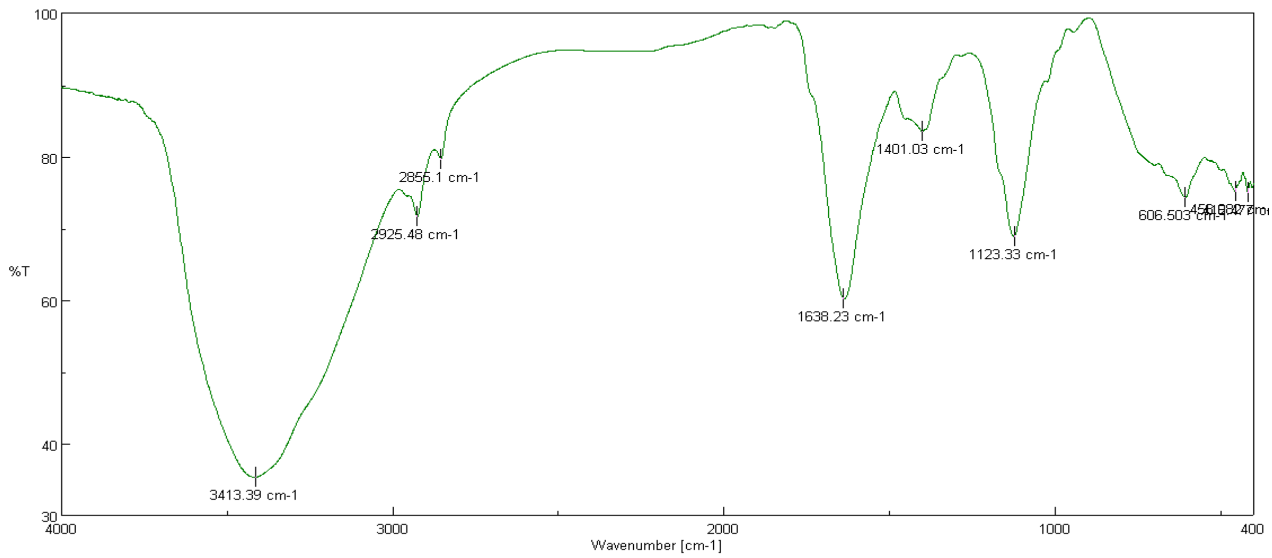


Fig - 5: FT-IR spectrum for the corrosion product on Copper in the presence of MEL extract with Natural Sea Water

3.7 EDX Spectra

EDX spectroscopy was used to determine the elements present on the copper surface before and after exposure to the inhibitor solution. Figure 6 and 7 represents the EDX spectra for the corrosion product on metal surface in the absence and presence of optimum concentrations of MEL extract with Natural sea water environment. In the absence of inhibitor molecules, the spectrum may confirms the existence of copper, iron, magnesium, carbon, stannum. However, in the presence of the optimum concentrations of the inhibitor, oxygen and nitrogen atoms are found to be present on the metal surface. It is clear that the hetero atoms (O, N) present in the inhibitor molecules may involve the adsorption process with metal atom to form a complex and hence it may protect the metal surface against the corrosion.

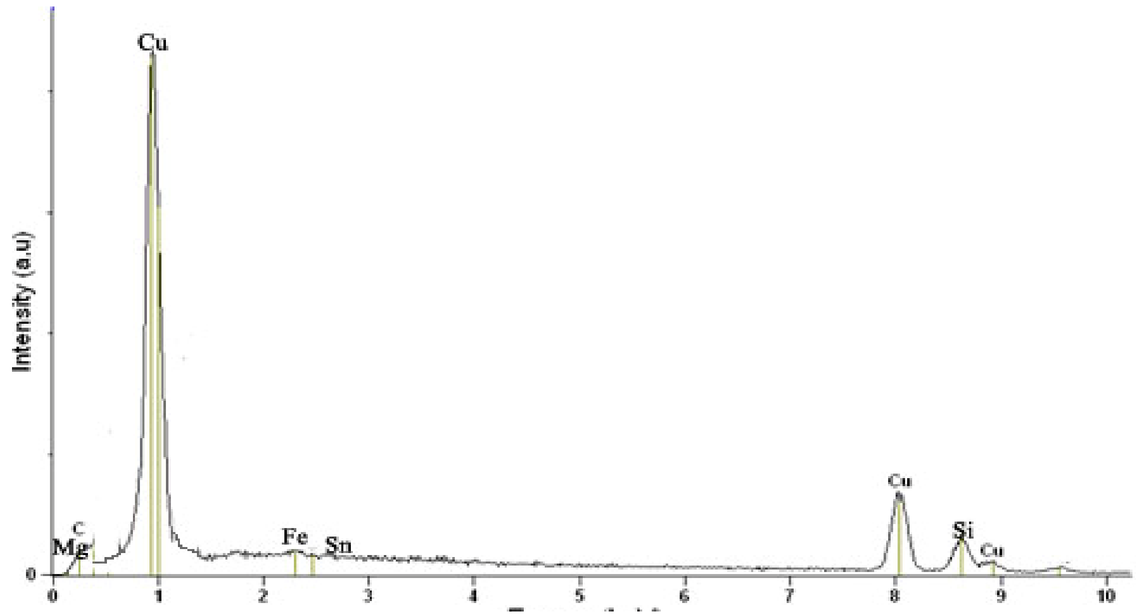


Fig 6: EDX spectrum of the corrosion product on copper surface in Natural seawater

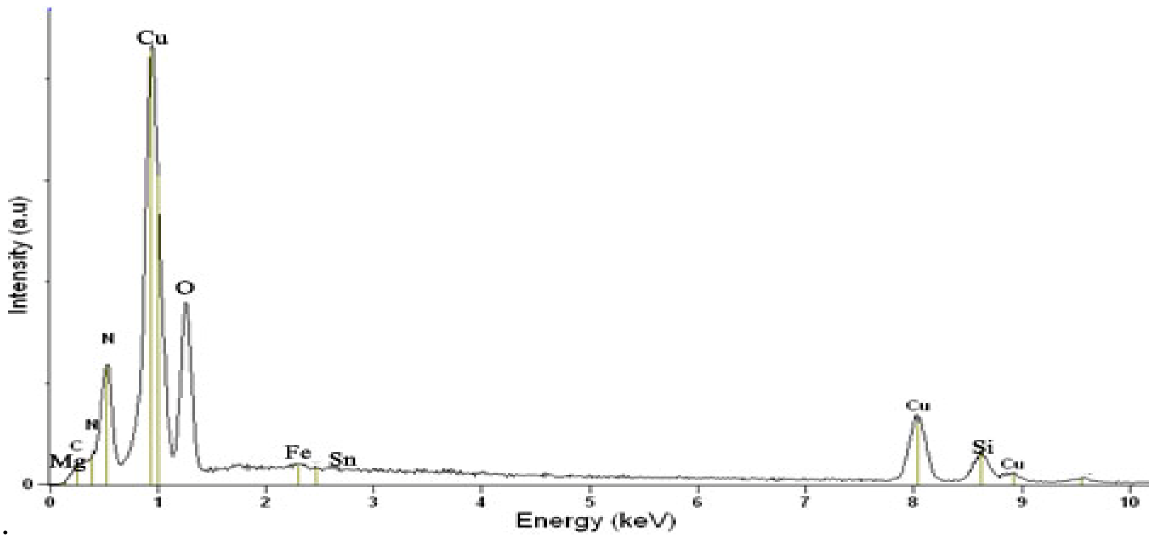
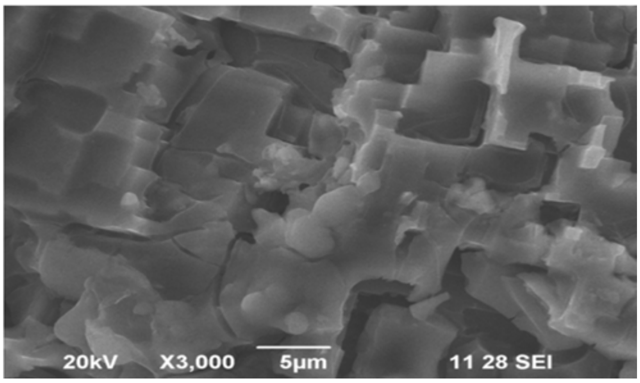


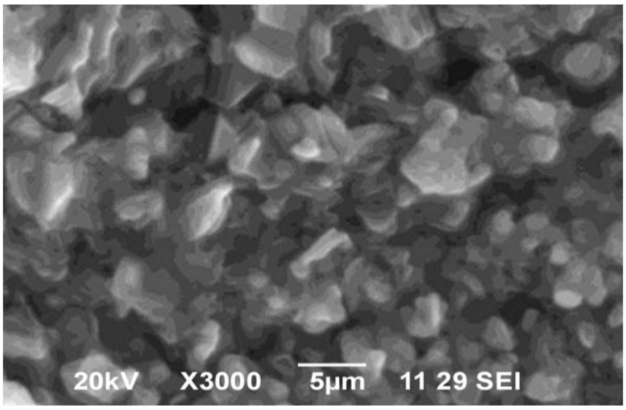
Fig 7: EDX spectrum of the corrosion product on copper surface with the presence of MEL extract in Natural seawater.

3.8 SEM Analysis

The surface morphology of copper surface was studied by scanning electron microscopy (SEM). The Fig-8(a) and (b) shows the SEM micrographs of copper surface before and after immersion in Natural seawater respectively. The SEM photographs (a) showed that the surface of metal has number of pits and cracks are visible in the surface, but in presence of inhibitor they are minimized on the metal surface. It is clearly indicates that the formation of spongy mass covered on the entire metal surface to reduce further dissolution of the metal.



(a)



(b)

Fig 8: SEM images of the copper surfaces: (a) immersed in Natural seawater; (b) immersed in natural sea water with MEL extract

4. Conclusion

From the above observations and findings, the following conclusions may be drawn.

I. Using *Mimusops elengi* leaves (MEL) extract copper in natural sea water

❖ Corrosion of copper in natural sea water is increased with increase of exposure period from 120 to 480 hours. Using MEL extract on copper, the corrosion rate markedly reduced with increase of concentrations from 0 to 500ppm. The maximum 86.84 % of inhibition efficiency is achieved even after 120 hours exposure time. This is due to strong bindings between the inhibitor molecule and ions from the metal surface. In temperature studies, the percentage of inhibition efficiency increased with rise of temperature from 313 to 333K is due to the adsorption of active inhibitor molecules on the metal surface is higher than desorption process. The maximum 92% inhibition efficiency is attained and follows chemisorptions. The activation energy (E_a), heat of adsorption (Q_{ads}), Standard free energy adsorption (ΔG_{ads}), enthalphy (ΔH), entrophy (ΔS), suggests that, strong chemical bond, endothermic, spontaneous process respectively. The MEL inhibitor obeys Langmuir adsorption isotherm. The film formation may confirm UV, FT-IR, SEM, EDX, spectral studies.

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