Effect of Cansjera Rheedii leaves extract on mild steel in Natural Sea Water

[1]V.Rama, [2]I. Malarvizhi , [3]S. Selvaraj
[1]Research Scholar, Reg.No : 11244 , Post Graduate&Research Department of Chemistry
Sri Paramakalyani college,Alwarkurichi -627412
Affiliated to Manonmaniam Sundranar University Abishekapatti, Tirunelveli -627012, Tamilnadu, India
[2]Research Scholar, Sri Paramakalyani college,Alwarkurichi -627412
[3]Associate Professor & Head, Deparment of Chemistry, Sri Paramakalyani college,
Alwarkurichi -627412

Abstract

Cansjera Rheedii leaves has shown excellent inhibition effect on Mild Steel in Natural Sea Water environment. The inhibition efficiency increased with the increase of inhibitor concentration and attain, maximum of 96%. This is due to the adsorption of active inhibitor molecules such as oxygen, hetero atom present in the phytochemical constituent may bind with the metal atom, present on the surface. However, the inhibition efficiency gradually decreased with the rise in temperature and suggests the physisorption process. The various thermodynamic parameters viz activation energy (E_a), enthalpy of adsorption (ΔH_{ads}) and free energy changes (ΔG_{ads}) indicates that the adsorption of inhibitor on metal surface follows physisorption, endothermic and spontaneous process respectively. The inhibitor is found to obey Langmuir adsorption isotherms. The morphological studies may confirmed by SEM images. The corrosion products formed on the metal surface also characterised by FT-IR, and EDX spectroscopy.

Key Words: Mild Steel, Green inhibitor, Natural Sea Water, Mass Loss, Adsorption, Spectral studies.

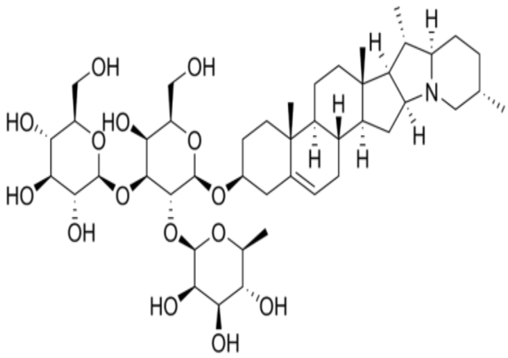
1.0. Introduction:

Mild steel is most familiar material widely employed in a variety of industries in world wide. But the main problem of using this material undergoes dissolution in acidic solutions. In various industrial processes, acid solutions are commonly used for removal of rust and scale. Use of inhibitor is one of the best method to prevent metal dissolution is very common [1, 3]. Most of the well-known acid inhibitors are organic compounds containing hetero atoms viz; nitrogen, sulfur, oxygen, heterocyclic compounds with a polar functional group and conjugated double bond [4, 5]. These kinds of compounds are adsorbed on the metallic surface and block the active corrosion sites. Most of the synthetic chemicals are costly, toxic to both human being and the environment. In order to overcomes, these difficulties choosing the inhibition are plenty, cheap, nontoxic and environmentally friendly natural products as corrosion inhibitors. These natural organic compounds are either synthesized or extracted from aromatic herbs, spices and medicinal plants. Plant extracts are an incredibly rich source of naturally synthesized chemical compounds that can be extracted by simple procedures with low cost and are biodegradable in nature. The plant extract are rich sources of molecules which have appreciably high inhibition efficiency and hence termed as "Green Inhibitors". These inhibitors do not contain heavy metals or other toxic compounds. Recent studies using plants containing heteroatom such as oxygen, nitrogen and sulphur *like Ocimum viridis, Phyllanthus amarus, Annona squamosal, Argan, Psidium guajava, black pepper, Punica granatum, Mentha pulegium, Cnidoscolus chayamans, Solanum Torvum, Pisonia Grandis, mimusops elengi, Sauropus Androgynus, Kingiodendron pinnatum, Wrightia Tinctoria [6-20]* have also been used for inhibition of corrosion. In continuous of our research work, the present investigation is the *Cansjera Rheedii* leaves extract used as corrosion inhibitor on mild steel in Natural sea water have been investigated with various periods of contact and temperature using the mass loss measurements. The corrosion product formed on the metal surface is analysed by FT-IR EDX and the morphological studies by SEM-image.

2.0 Materials and methods

2.1 Properties of *Cansjera Rheedii leaves*:

Leaf of *Cansjera Rheedii* (CRL) (Family: Opiliaceae) is a climbing shrub commonly known as kalimanakeerai (in Tamil) is found mostly in India, China, Honkong and Australia. *Cansjera Rheedii* has a medicinal value of anthelmintic, hepatoprotective, antipyretic, diuretic activities. It has also been reported that the main phytoconstituent of saponins, flavonoids, glycosides, phenolic compounds, alkaloids and tannins.



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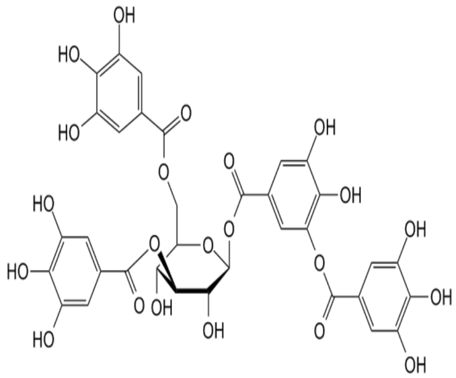


Tannins

Figure - 1: Chemical structure of the main active compounds present in *Cansjera Rheedii leaves* extract.

2.2 Stock solution of *Cansjera Rheedii Leaves* Extract:

Cansjera Rheedii leaves (CRL) was collected from the source and dried under shadow for about 10 days, grained well, then soaked in a solution of ethyl alcohol for about 48 hrs, Then it is filtered followed by evaporation in order to remove the alcohol solvent completely and the pure plant extract was collected. From this extract, different concentration of 10 to 1000ppm stock solution was prepared using double distilled water and used throughout our present investigation.

2.3 Specimen preparation

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

2.4 Mass loss method

In the mass loss measurements on mild steel 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 24 to 360 hrs at room temperature and also measured 313K to 333K. 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 = 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)

3.0 Results and Discussion

3.1 Effect of time variation

The dissolution behavior of mild steel in Natural Sea Water containing the presence and absence of CRL extract with various exposure times (24hrs to 360 hrs) are shown in Table-1**.** The observed values are clearly indicates that the presence of CRL extract, the corrosion rate moderately decreased from 0.1394 to 0.044 mmpy for 24 hrs and 0.0619 to 0.0123 mmpy after 360 hrs with increase of inhibitor concentration (0 to 1000 ppm). The maximum of 96.15 % of inhibition efficiency is achieved after 72 hrs exposure time, suggests that the adsorption process occurs mainly due to the presence of active phytochemical constituents present in the inhibitor molecule especially oxygen containing species and the metal ion from the surface of the metal.

Table-1: The corrosion parameters of mild steel in Natural Sea Water containing different concentration(0 to 1000ppm) of CRL extract after 24 to 360 hours exposure time

Conc. of inhibitors (ppm)	24 hrs		72 hrs		120 hrs		240 hrs		360 hrs	
	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E
0	0.1394	-	0.4029	-	0.1580	-	0.0743	-	0.0619	-
10	0.1162	16.66	0.3487	13.46	0.1394	11.75	0.0720	31.18	0.0557	10.00
50	0.0929	33.33	0.2634	34.61	0.1022	35.29	0.0604	18.75	0.0495	20.00
100	0.0929	33.33	0.2169	46.15	0.0511	67.64	0.0604	18.75	0.0433	30.00
500	0.0697	50.00	0.1317	67.30	0.0139	91.98	0.0255	65.62	0.0216	65.01
1000	0.0464	66.67	0.0154	96.15	0.0092	94.12	0.0139	81.26	0.0123	80.01

3.2. Effect of Temperature

Dissolution behavior of Mild Steel in Natural Sea Water containing various concentration of CRL extract at 303to 333K and the observed values are listed in Table-2. The observed results reveals that the corrosion rate decreased with increase of inhibitor concentrations and increased with rise in Temperature from 303 to 333K. The maximum of 81.81% inhibition efficiency is achieved at 303K. However the value of inhibition efficiency is decreased with rise in Temperature may suggests and support the facts that the process of adsorption follows **physisorption**.

Table-2: The corrosion parameters of Mild Steel in Natural Sea Water containing different concentration of CRL extract at 313 to 333 K after one hour exposure time.

Conc. of inhibitor (ppm)	303 K		313 K		333 K	
	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E	C.R (mmpy)	% I.E
0	6.1375	-	23.9923	-	45.1949	-
10	4.4636	27.27	18.9707	20.93	40.7312	9.87
50	2.7898	54.54	17.2968	27.90	38.4993	14.81
100	2.2318	63.63	13.9490	41.86	35.7095	20.98

500	1.6738	72.72	10.6012	55.81	32.3617	28.39
1000	1.1159	81.81	6.6955	72.09	26.2242	41.97

3.3 Activation energy:

The activation energy (E_a) for the corrosion of Mild steel in the presence and absence of CRL extract is calculated using the following Arrhenius equation (4) and its derived form equation (5).

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

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

(Where CR_1 and CR_2 are the corrosion rate at the temperature T_1 and T_2 respectively, E_a is the activation energy and R is the universal gas constant).

The value of activation energy (E_a) for the blank (55.8386 kJ/mol) is lower than in the presence of inhibitors (Table-3). This observation clearly indicates that the adsorption process also is **physisorption.**

3.4 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 equation (6).

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

(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 -34.4054 to -51.1208 kJ/mol. This negative value indicate and suggests that the adsorption of CRL extract on the Mild steel surface is **exothermic.**

Table -3: Calculated values of Activation energy (E_a) and heat of adsorption (Q_{ads}) of CRL extract on Mild Steel in Natural Sea Water environment.

S.No	Conc. of inhibitor (ppm)	% of I.E		E_a ($KJmol^{-1}$)	Q_{ads} ($KJmol^{-1}$)
		30°	60°		
1.	0	-	-	55.8386	--
2.	10	27.27	9.87	61.8372	-34.4054
3.	50	54.54	14.81	73.4054	-54.0188
4.	100	63.63	20.98	77.5424	-52.7263
5.	500	72.72	28.39	82.8351	-53.2994
6.	1000	81.81	41.97	88.2935	-51.1208

3.5 Adsorption studies:

Process of adsorption are very important phenomenon to determine the corrosion rate of reaction mechanism. The most frequently use of isotherms are viz: Langmuir, Temkin, Frumkin, Flory- Huggins, Freund lich, Bockris-Swinkles, Hill-de Boer, Parson's and the El-Amady, thermodynamic-kinetic model.

3.5.1. Langmuir, El-Awady and Frumkin Adsorption Isotherm

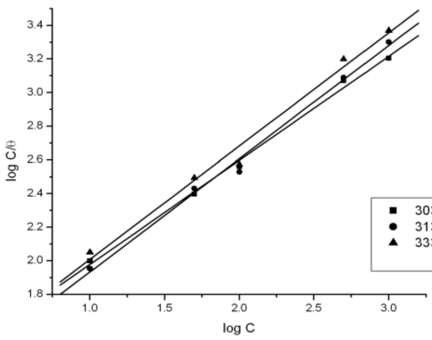
The Langmuir adsorption isotherm of CRL extract on Mild steel surface proceeded according to the following equation (7).

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

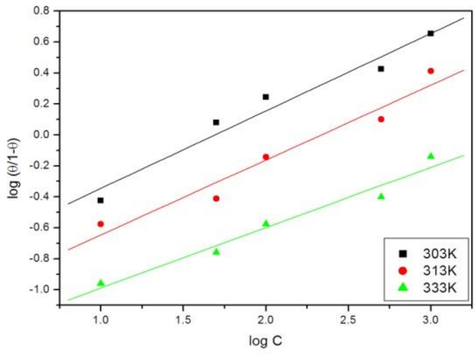
(Where θ is the degree of surface coverage, C is the concentration of the inhibitor solution and K is the equilibrium constant of adsorption of inhibitor on the metal surface).

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 Figs 2(a-c) shows Langmuir, El-Awady, and Frumkin isotherm model respectively. The values of K, R^2 and ΔG_{ads} are derived from these adsorption isotherm at different temperature ranges from 303K to 333K are given in Table -4. Langmuir adsorption isotherm model fit the corrosion rate data of CRL inhibitor on mild steel strongly and clearly indicates that the average value of Regression co-efficients R^2=0.9935 is almost unity. The other adsorption isotherm values viz, El-Awady (R^2=0.9796) and Frumkin (R^2=0.9796) are also move to near unity. we also attempt the Freundlich (R^2=0.9696), Temkin (R^2=0.9661), and Florry-Huggins (R^2=0.9402) adsorption isotherm models of CRL extract. This observed values relatively far from unity as compared with the values obtained with Langmuir adsorption isotherm which is clearly indicate that the weak correlation between the two variables (i.e surface coverage, inhibitor concentration). It is concluded that the inhibitor obeys Langmuir adsorption isotherm.



(a)



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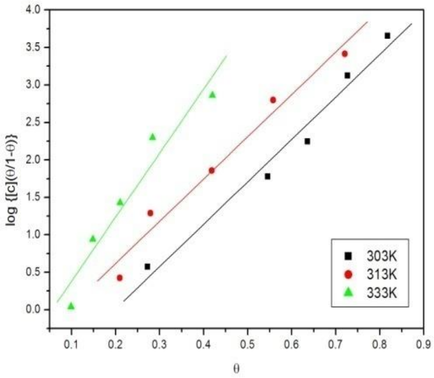


(c)

Fig. 2 (a) Langmuir, (b) El-Awady, (c) Frumkin isothserm for the adsorption of CRL inhibitor on mild steel in Natural sea water.

3.5.2. Free energy of adsorption:

The equilibrium constant of adsorption for various plant extract on the surface of Mild steel is related to the free energy of adsorption ΔG_{ads} by equation (8).

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

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

The values of intercept (K) obtained from Langmuir, El-Awady, Frumkin adsorption isotherm is substituted in equation (8) and the calculated values of ΔG_{ads} are placed in Table-4. In El-Awady adsorption isotherm, the decrease of equilibrium constant (K_{ads}) values suggest that the process is physisorption phenomenon, which attributed to electrostatic interaction between the charged metal and active inhibitor molecules. Also the values of 1/y are less than unity, showing that there is a multilayer adsorption of the inhibitor molecule on the metal surface.[21-22]. In Langmuir adsorption, the negative values of ΔG_{ads} suggested that the adsorption of CRL extract onto Mild steel surface is a spontaneous process and the adsorbed layer is more stable one.

Table- 4 Langmuir, El-Awady and Frumkin adsorption parameters for the adsorption of CRL inhibitor on mild steel in Natural sea water.

Temperature (Kelvin)	Adsorption Isotherms									
	Langmuir			El-Awady				Frumkin		
	K	R^2	$-\Delta G_{ads}$ kJ/mol	K	1/y	R^2	$-\Delta G_{ads}$ kJ/mol	K	R^2	$-\Delta G_{ads}$ kJ/mol
303K	5.0842	0.9884	14.2168	2.1×10^{-2}	0.5005	0.9802	5.2120	0.1415	0.9867	5.1927
313K	9.1622	0.9962	16.2189	4.6×10^{-3}	0.4841	0.9759	3.6778	0.2608	0.9858	6.9555
333K	20.668	0.9959	19.5079	2.9×10^{-4}	0.3899	0.9829	2.3322	0.2714	0.9665	7.5103

3.5.3 Thermodynamics parameters

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

$$CR=RT/Nh \exp(\Delta S/R) \exp(-\Delta H/RT) \text{ ----- } (9)$$

(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. 3) 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 out in Table-5. The positive value of enthalpy of activation clear that the endothermic nature of dissolution process is very difficult. The positive value of entropy (ΔS) is gradually increasing at the solid/liquid interface during the adsorption of the metal ions onto inhibitor molecules present in the extract.

Table 5: Thermodynamic parameters of Mild Steel in Natural Sea Water obtained from weight loss measurements.

S.No	Concentration of CRL (ppm)	ΔH (kJ mol^{-1})	ΔS (J $k^{-1}mol^{-1}$)
1	0	21.6022	13.8692
2	10	24.1887	14.6093
3	50	28.6857	15.9588
4	100	30.5838	16.4977
5	500	33.0049	17.1848
6	1000	35.6579	17.8955

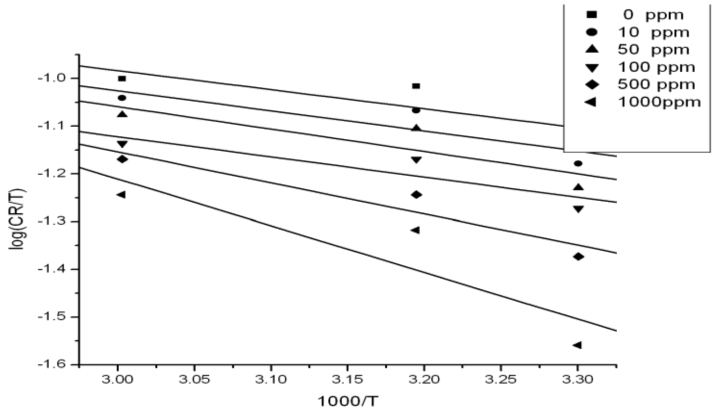


Figure- 3 The relation between log (CR/T) and 1000/T for different concentrations of CRL extract.

4. SPECTRAL STUDIES

4.1. FT-IR Spectroscopy:

4.1.1. FT-IR studies of CRL extract on Mild Steel surface in Natural Sea Water:

The figures-4 and 5 reflect that the FTIR spectrum of the ethanolic extract of inhibitor and the corrosion product on Mild Steel in the presence of CRL extract in Natural Sea Water . On comparing both of these spectra the prominent peak such as, the –OH stretching frequency for alcohol is shifted from 3379.29 to 3417.86 cm^{-1}, the –C=C stretching in alkene is shifted from to 1654.92 cm^{-1}to1641.42 cm^{-1}.The -C-C stretching frequency for aromatic ring is shifted from 1454.33 to 1514.12 cm^{-1},the –C-N stretching frequency for aliphatic amines is shifted from 1112.93 to1124.50. These results also confirm that the FTIR spectra support the fact that the corrosion inhibition of CRL extract on Mild Steel in Natural Sea Water by the adsorption of active phytochemical molecule in the inhibitor and the metal ion from the surface.

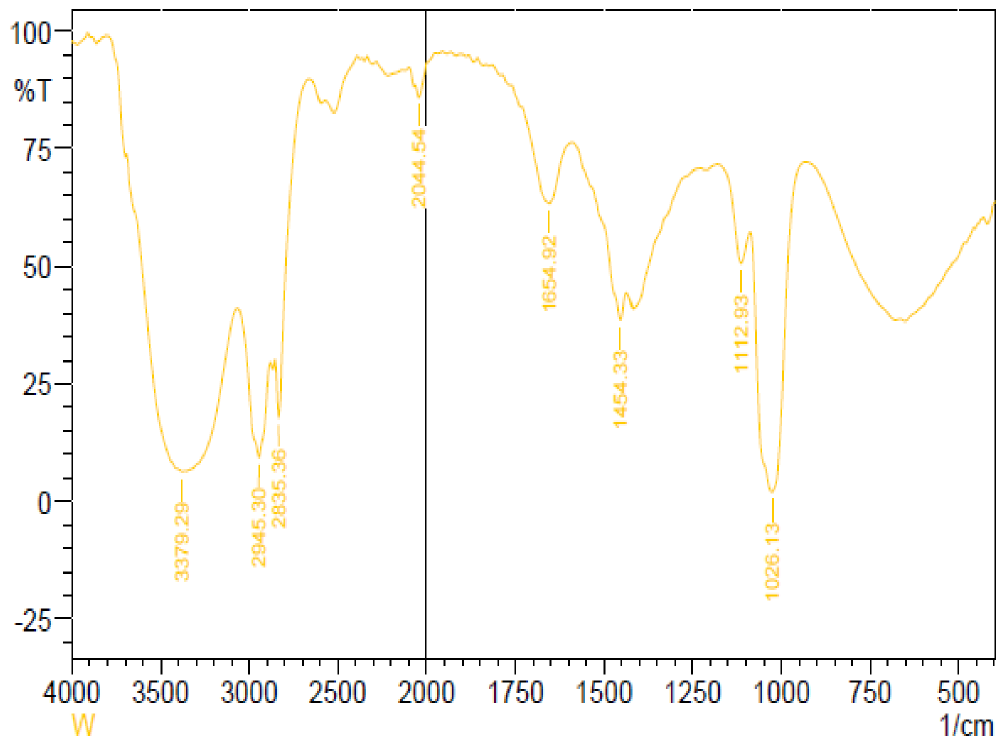


Figure 4: FT-IR spectrum of ethanolic extract of *Cansjera Rheedii leaves* (CRL)

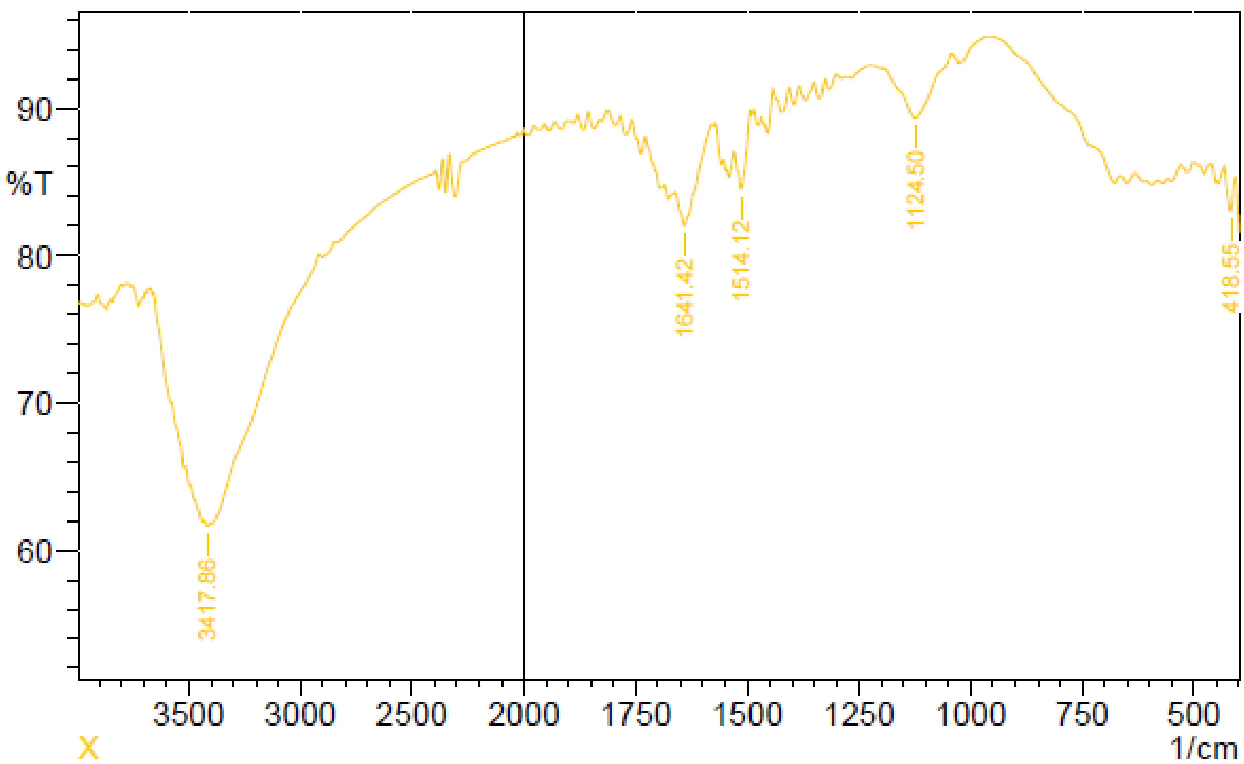


Figure 5 : FT-IR spectrum for the corrosion product on Mild Steel in the presence of CRL extract with Natural Sea Water.

5. EDX Spectrum:

EDX spectroscopy was used to determine the elements present on the Mild Steel surface in the absence and presence of inhibitor. Figs. 6 and 7 represents the EDX spectra for the corrosion products on metal surface in the absence and presence of optimum concentrations of CRL extract in Natural Sea Water. In the absence of inhibitor molecules, the spectrum may concluded that the existence of carbon present in the metal. However, in the presence of the optimum concentrations of the inhibitors, the hetero oxygen atom is found to be present in the corrosion product on the metal surface. It clearly indicates that this hetero atoms present in the inhibitor molecules may involve the complex formation with the metal ion during the adsorption process and prevent the further dissolution of metal against corrosion.

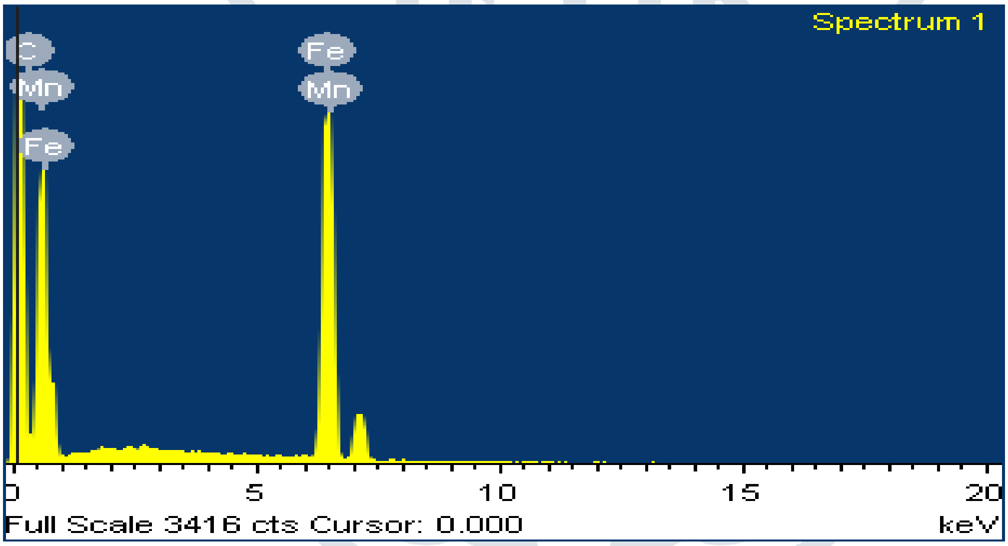


Fig 6: EDX spectrum of the corrosion product on Mild Steel surface in Natural Sea Water

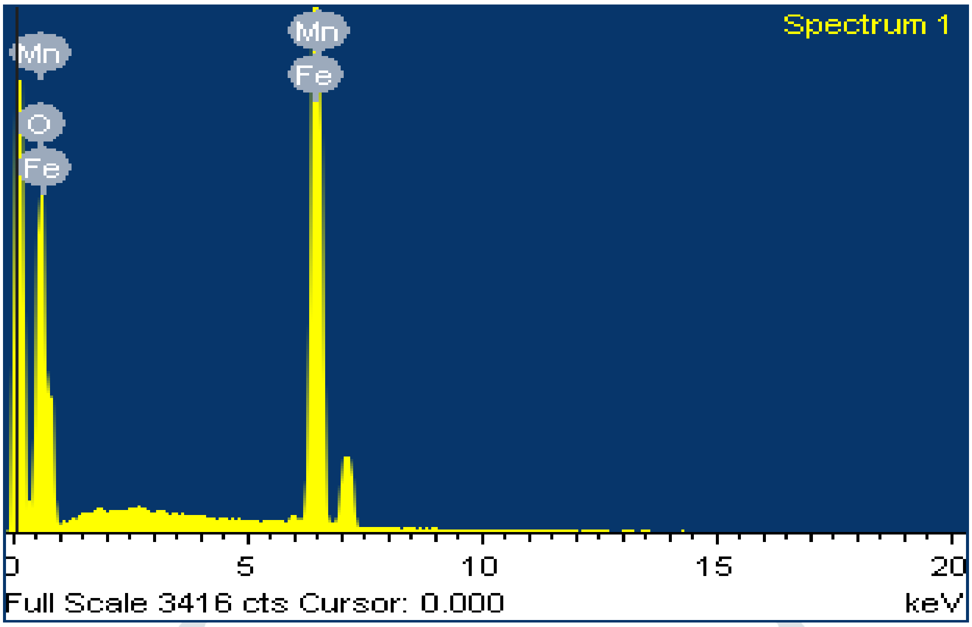
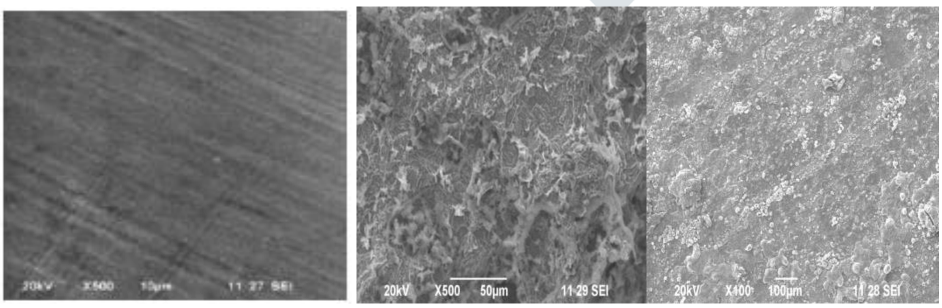


Fig 7 : EDX spectrum of the corrosion product on Mild Steel in the presence of CRL extract in Natural Sea Water

6. SEM Analysis

The Surface morphology of the Mild Steel surface was studied by scanning electron microscopy (SEM). The Figures 8(a-c) shows that the SEM micrographs of Mild Steel surface before and after immersion in Natural Sea Water respectively. The SEM photographs (fig-6a) showed that the surface of metal has number of pits and cracks are visible in the surface may be belong to the plug type of corrosion but in presence of inhibitor fig(6-c) the dissolution process significantly reduced by the formation of thin film covered the entire surface of the metal almost completely.



8 (a) 6(b) 6(c)

Figures 8 (a) to (c) SEM image of the Polished Mild Steel surface, SEM image of immersed in natural sea water and SEM image of immersed in Natural Sea Water with CRL extract respectively.

7.CONCLUSIONS

Cansjera Rheedii leaves has shown excellent inhibition performance for Mild Steel in Natural Sea Water environment. The inhibition efficiency increased with the increase of inhibitor concentration. The maximum inhibition efficiency was achieved 96.15% even 1000ppm concentration. However, the inhibition efficiency gradually decreased with rise in temperature i.e.81.81 %. This is due to the adsorption of active inhibitor molecules on the metal surface is follows physisorption process. The value of activation energy (E_a), enthalpy of adsorption (ΔH_{ads}), change in entropy (Δs) and free energy changes (ΔG_{ads}) indicates that the adsorption of inhibitor on metal surface follows chemical, endothermic and spontaneous process respectively. The inhibitor is found to obey Langmuir adsorption isotherms. The corrosion products characterised by FT-IR, and EDX spectroscopy. The thin film formation on the metal surface may also be confirmed SEM images.

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