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RESEARCH ARTICLE

CORROSION BEHAVIOR OF ZINC IN 1.0 N HYDROCHLORIC ACID WITH CNIDOSCOLUS CHAYAMANSA – A GREEN APPROACH

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ABSTRACT

Inhibition effect of *Cnidoscolus Chayamansa* leaves extract on corrosion of Zinc in 1.0 N HCl was investigated by mass loss measurement with various period, contact and temperature. The observed result indicates that the corrosion inhibition efficiency was increased with increase of inhibitor concentration and temperature. The thermodynamic parameters (viz; E_a, ΔH_{ads}, ΔG_{ads}, ΔS_{ads}) were evaluated for corrosion process, which suggest that the adsorption is endothermic, spontaneous and chemisorptions. The inhibitor follows Langmuir and Temkin adsorption isotherm. The protective film formed on the metal surface was analyzed by using various spectroscopic studies viz; UV, FT-IR , XRD and SEM-EDX techniques.

INTRODUCTION

Rich coatings of zinc have proven to be an excellent method of protecting steel from corrosion due to the familiar electro chemical process. Corrosion resistance property of zinc is more important that nearly half of the world's annual consumption of the metal is used to protect steel from rusting. Due to various industrial applications and economic importance of zinc, it's protection against corrosion attracted much attention. The corrosion behavior of zinc is affected by both, alkaline and acid medium. The corrosion being more severe at pH values below 6 and above 12.5 (Shreir L.L., 2000). Hydrochloric acid is a strong inorganic acid that can be used in many industrial processes. Aliphatic amine (Chatterjee P., *et al,* 1989; Vshi R. T., *et al,* 2010; Harshida G., *et al,* 2012*)*, aromatic amine (Mahida, M.B., *et al,* 2012), organic compounds (Shanthamma Kampalappa Rajappa., *et al,* 2003), and few Schiff bases (Desai M.N., *et al,* 2008), have been extensively used as corrosion inhibitor by most of the authors. The inhibitor must be eco-friendly to replace the used one, which is more toxic and harmful to the environment. Most of the recent published literature reveals that the study of corrosion inhibition of different metals with various green inhibitor have been reported. A few examples are Red Peanut Skin (James A.O., *et al,* 2011), Musa species peels (Eddy N.O., *et al,* 2009), Henna extract (Ostovari A., *et al,* 2009), *Delonix regia* extracts (Abiola O.K., *et al,* 2007), Rosemary leaves (Kliskic M., *et al,* 2000), *opuntia* extract (El-Etre A.Y., 2003), *khillah* (Ammi visnaga) seeds (El-Etre A.Y., 2006), *Carica Papaya* and *Camellia Sinensis* Leaves (Loto C.A., *et al,* 2011), *Ricinus communis* Leaves (Saratha R., *et al,* 2009), *Justicia gendarussa* (Satapathy A.K., *et al,* 2009*)*, *Vitis vinifera* (Deepa Rani P., *et al,* 2010), *Punica granatum* peel (Deepa Rani P., *et al,* 2010), *Cnidoscolus Aconitifolius* (Benedict U., *et al,* 2014), have been studied on various metals and alloys. However only a limited number of literature is available for the corrosion inhibition by green inhibitor with zinc metal surface. Some investigators have been reported with zinc metal is *Ocimum tenuiflorum* (Sharma Sanjay K., *et al,* 2009), Red onion skin (James A.O., *et al,* 2009)*, Nypa fruticans Wurmb* (Orubite Okorosaye K., *et al,* 2004), *Aloe vera* (Aboia O.K., *et al, 2010)*, henna (*lawsonia*) Leaves (El-Etre A.Y., *et al* 2005). In our present attention is to study the effect of adsorption and corrosion inhibition of *Cnidoscolus Chayamansa* leaves extract on zinc metal surface with 1.0 N Hydrochloric acid environment.

MATERIALS AND METHODS

Properties of Cnidoscolus Chayamansa Leaves

Cnidoscolus Chayamansa is commonly known as 'chaya' plant. Chaya leaves are an excellent source of a number of essential nutrients for a healthy, balanced diet. It's an outstanding green leaves has rich and nutritious twice than spinach, Chinese cabbage and amaranth. The leaves are very high in protein, calcium, iron, carotene, and vitamins A, B and

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C, Also contains Flavonoids, Terpenoids, Glycosides, Steroids, Alkaloids, Carbohydrates, Amino acid , Tannins, Chlorides, Copper, Nitrates, Potassium, Zinc, Carbonates and bicarbonates.Chaya traditionally has been recommended for a number of ailments including diabetes, obesity, kidney stones, hemorrhoids, acne, and eye problems.

Stock Solution of Cnidoscolus Chayamansa Extract

Cnidoscolus Chayamansa (CCL) 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.

Specimen Preparation

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

Mass Loss Method

In the mass loss measurements, Zinc specimens in Triplicate were completely immersed in 100ml 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 of the specimens is taken as the difference in weight 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)

RESULTS AND DISCUSSION

Table- 1 reflects that the variation of corrosion parameters with concentration of CCL extract on zinc at different period of contact in 1.0 N hydrochloric acid. It is revealed that the corrosion rate was decreased gradually with the increase of inhibitor concentration. The maximum of 73.5% inhibition efficiency was achieved at 1000 ppm of inhibitor concentration after 72 hrs exposure time. This is mainly due to the active phyto chemical constituent of CCL extract viz, π bonds, hetero atoms (O, N and S), cystine, 2-amino-4-(methylthio)butanoic acid etc .

Variation of corrosion parameters with various concentration of CCL extract on zinc at different temperature in 1.0 N Hydrochloric acid is shown in Table-2. As can be seen from Table-2 that the corrosion rate was decreased and the percentage of inhibition efficiency increased with increase of inhibitor concentration with rise in temperature from 303 to 333K. It suggested that the adsorbed layer retards the corrosion rate with rise in temperature from 303 to 333K. The maximum of 82.09% inhibition efficiency was achieved at 333K. The value of inhibition efficiency was increased with rise in temperature. Thus, the inhibition efficiencies were temperature dependent and it can be chemically adsorbed at all temperatures.

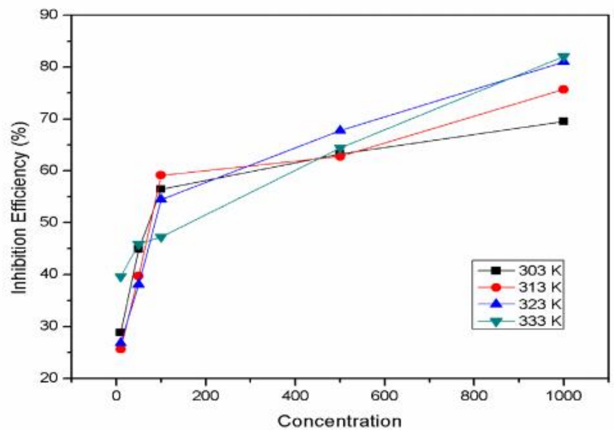


Figure 1 The corrosion parameters of zinc in 1.0 N Hydrochloric acid containing various concentration of CCL extract with different exposure temperature.

Table 1 The corrosion parameters of zinc in 1.0 N Hydrochloric acid containing various concentration of CCL 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	35.418	-	14.637	-	7.617	-	5.599	-	4.836	-	3.045	-
10	27.677	21.9	13.342	8.8	6.255	17.9	4.775	14.7	3.326	31.2	2.095	31.2
50	24.569	30.6	12.401	15.3	5.691	25.3	4.331	22.7	2.946	39.1	1.862	38.8
100	23.224	34.4	10.442	28.7	4.821	36.7	3.898	30.4	2.546	47.4	1.572	48.4
500	14.576	58.8	6.861	53.1	3.759	50.6	2.574	54.0	1.925	60.2	1.263	58.5
1000	9.605	72.9	3.874	73.5	2.749	63.9	1.709	69.5	1.623	66.5	1.055	65.4

Table 2 The corrosion parameters of zinc in 1.0 N Hydrochloric acid containing various concentration of CCL 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	742.53	-	774.22	-	868.05	-	950.07	-
10	528.78	28.79	575.38	25.68	635.03	26.84	574.14	39.57
50	409.48	44.86	466.64	39.73	537.48	38.08	514.49	45.85
100	323.73	56.40	316.27	59.15	395.19	54.48	501.44	47.22
500	272.78	63.27	288.31	62.76	280.24	67.72	338.02	64.42
1000	226.18	69.54	188.27	75.68	164.66	81.03	170.25	82.09

Table 3 Calculated values of activation energy (E_a) and heat of adsorption (Q_{ads}) of CCL extract on zinc in 1.0 N Hydrochloric acid environment.

Concentration of inhibitor (ppm)	% of I.E		E_a (KJ mol^{-1})	Q_{ads} (KJ mol^{-1})
	30°C	60°C		
0	-	-	69.577	-
10	28.79	39.57	68.212	13.485
50	44.86	45.85	69.426	1.1141
100	56.40	47.22	71.166	4.0866
500	63.27	64.42	69.311	1.391
1000	69.54	82.09	65.165	19.493

Effect of Temperature

The values of E_a for the corrosion of Zinc in the presence and absence of CCL extract is calculated using the following Arrhenius equations (4) and its derived from equation (5).

$CR = A_{exp} (-E_a/RT)$ -------- (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 rates of zinc 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 blank (69.577 kJ/mol) is lower than in the presence of inhibitor on 100 ppm is given in Table 3. But this value is higher than the presence of inhibitor up to 1000 ppm which is clearly indicates that the adsorption process is physisorption followed by chemisorptions.

Adsorption Consideration

The heat of adsorption on Zinc Copper in the presence of inhibitor 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)

Where R is the gas constant, $\theta_1$and θ_2 is the degree of surface coverage at temperatures and T_1 and T_2 respectively. The Q_{ads} values are ranged from 1.1141 to 19.493 kJ/mol (Table 3). This value clearly revealed that the adsorption of CCL extract on the surface of zinc metal is endothermic.

Table 4 Langmuir adsorption parameters of CCL extract on zinc in 1.0 N Hydrochloric acid environment.

	Log C/ θ			
Log C	30°C	40°C	50°C	60°C
1	1.541	1.5904	1.5712	1.4026
1.6989	2.047	2.099	2.1183	2.0376
2	2.2487	2.2280	2.2638	2.3258
2.6989	2.8978	2.9013	2.8683	2.8899
3	3.1578	3.1210	3.0914	3.0857

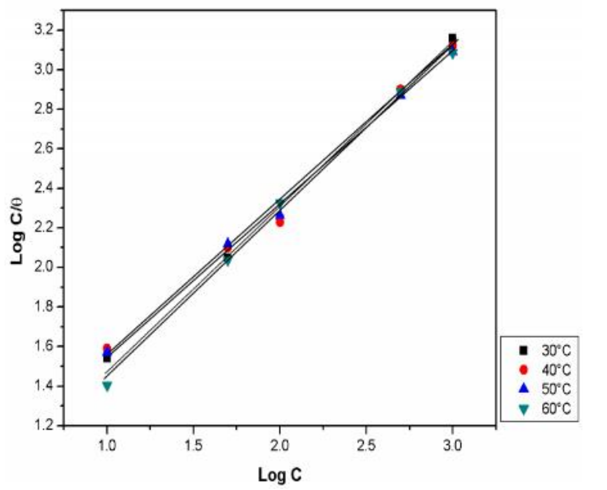


Figure 2 Langmuir adsorption parameters of CCL extract on zinc in 1.0 N Hydrochloric acid environment.

Table 5 Temkin adsorption parameters of CCL extract on zinc in 1.0 N Hydrochloric acid environment.

Log C	θ			
	30°C	40°C	50°C	60°C
1	0.2879	0.2568	0.2684	0.3957
1.6989	0.4486	0.3973	0.3808	0.4585
2	0.5640	0.5915	0.5448	0.4722
2.6989	0.6327	0.6276	0.6772	0.6442
3	0.6954	0.7568	0.8103	0.8209

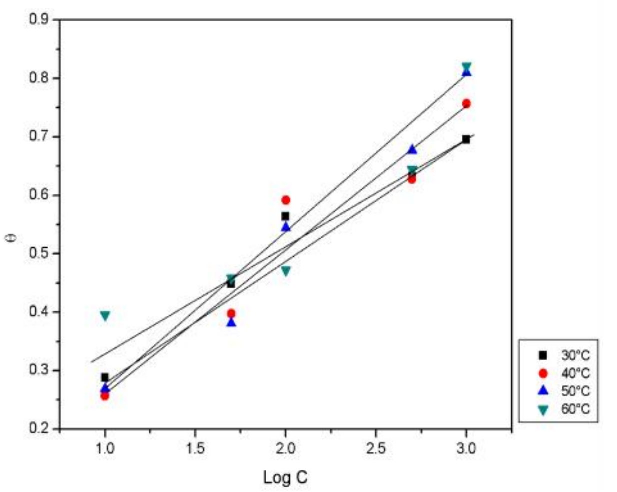


Figure 3 Temkin adsorption parameters of CCL extract on zinc in 1.0 N Hydrochloric acid environment.

The adsorption isotherms are used to investigate the mode of adsorption and the characteristic of adsorption of inhibitor on the metal surface. In our present study the Langmuir, Temkin isotherm are investigated. The Langmuir and Temkin adsorption isotherm can be expressed by the equation (7) and equation (8) given below.

$\log C/\theta = \log C - \log K$ ---------- (7)
$\theta = K \ln C$ --------- (8)

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

By plotting values of log C/θ versus log C, linear plots were generated (Fig. 2) and conforming that the experimental data fitted with the Langmuir adsorption isotherm for the adsorption of CCL extract on metal surface. It means that there is no interaction between the adsorbed species (i.e; adsorbate and adsorbent). The Langmuir adsorption isotherm is better fit at 333K for Copper (R^2 =0.9962).

A plot of θ versus log C gives almost a straight line for metal in CCL extract in acid medium. (Fig 3)**.** The straight line indicated that the inhibitor obeyed Temkin adsorption isotherm.

The adsorption of CCL extract on the metal surface is related to the free energy of adsorption (ΔG_{ads}) by the following equation (9)

$\Delta G_{ads} = -2.303\ RT \log (55.5\ K)$ ---------(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 and Temkin adsorption isotherm is substituted in equation (9) and the calculated values of ΔG_{ads} are placed in Table 6. The negative values of ΔG_{ads} suggested that the adsorption of CCL extract onto metal surface is a spontaneous process and the adsorbed layer is more stable one.

(ΔS/R)], from which the values of ΔS and ΔH were calculated and listed in Table 8.

The positive value of enthalpy of activation reflects the endothermic nature of metal dissolution process meaning that dissolution of metal is difficult.

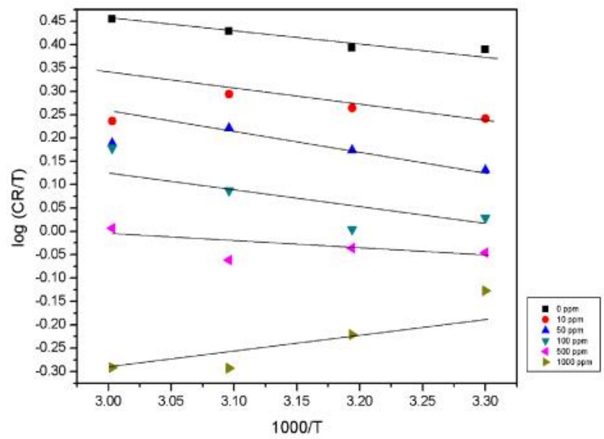


Figure 4 The relation between log (CR/T) and 1000/T for different concentration of CCL extract on zinc in 1.0 N Hydrochloric acid environment.

Table 8 Thermodynamic parameters of Zinc in 1.0 N Hydrochloric acid obtained from weight loss measurement.

Concentration of CCL extract (ppm)	ΔH (KJ mol^{-1})	ΔS (KJ mol^{-1})
0	1.9522	9.010
10	0.1558	8.313
50	1.9054	8.797
100	4.3867	9.492
500	1.0655	8.356
1000	-4.7993	6.344

Table 6 Langmuir and Temkin parameters of CCL extract on zinc in 1.0 N Hydrochloric acid environment.

Adsorption isotherm	Temperature	Slope	K	R^2	ΔG_{ads} (KJ mol^{-1})
Langmuir	303	0.8163	4.796	0.9941	-14.069
	313	0.7758	5.951	0.9890	-15.096
	323	0.7612	6.303	0.9960	-15.732
	333	0.846	3.882	0.9962	-14.877
Temkin	303	0.1984	1.2974	0.9501	-10.776
	313	0.2394	1.0672	0.9069	-10.623
	323	0.2698	0.9448	0.9611	-10.635
	333	0.2022	1.3729	0.8233	-11.999

Table 7 The relation between log (CR/T) and 1000/T for different concentration of CCL extract on zinc in 1.0 N Hydrochloric acid environment.

	Log (CR / T)					
1000 / T	0 ppm	10 ppm	50 ppm	100 ppm	500 ppm	1000 ppm
3.3	0.3893	0.2418	0.1308	0.0287	-0.0456	-0.1269
3.194	0.3933	0.2644	0.1734	0.0045	-0.0357	-0.2208
3.096	0.4293	0.2936	0.2213	0.0876	-0.0617	-0.2926
3.003	0.4553	0.2366	0.1889	0.1777	0.0065	-0.2914

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

$CR = RT/Nh \exp (\Delta S/R) \exp (-\Delta H/RT)$ ------------------------(10)
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 should give a straight line (Fig 4) with a slope of (−ΔH/R) and an intercept of [log(R/Nh)) +

Morphology Studies

UV Spectrum

Fig - 5 (a) & (b) shows that the UV visible spectrum of ethanolic extract of CCL and the corrosion product on the surface of zinc in the presence of CCL extract in 1.0 N hydrochloric acid respectively.

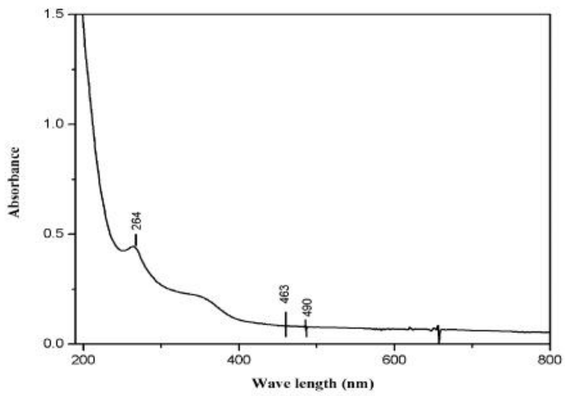


Figure 5A

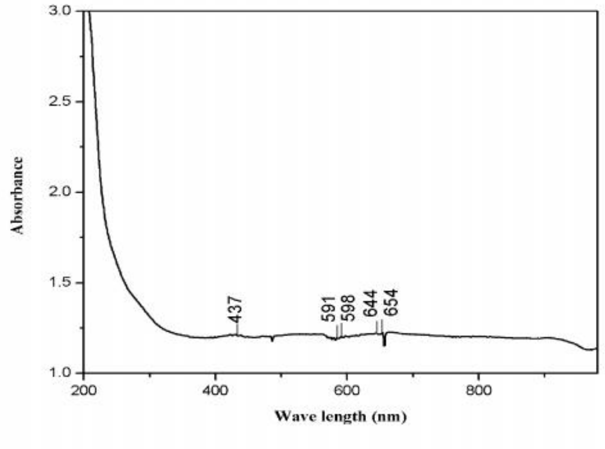


Figure 5B

Figure 5 UV spectrum of ethanolic extract of CCL (A), the corrosion product on zinc in 1.0 N HCl in the presence of CCL extract (B).

In this spectrum, the three absorption bands were noticed (264,463,490 nm) (Fig 5(a)) and in the presence of inhibitor five bands was appeared (437,591,598,644,654 nm).It is clearly indicates that the band is shifted to longer wavelength region (Bathochromic shift (or) Red shift) or disappearance of absorption bands, may confirmed that the strong co-ordination bond between the active group present in the inhibitor molecules and the ions from the metal surface.

FT-IR Analysis

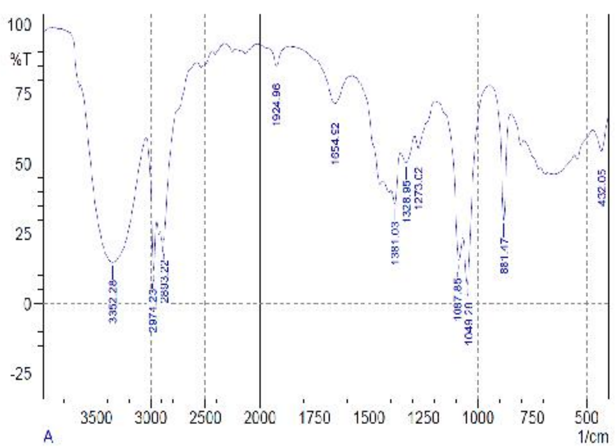


Figure 6 FT-IR spectrum of ethanolic extact of *Cnidoscolus chayamansa* leaves (CCL).

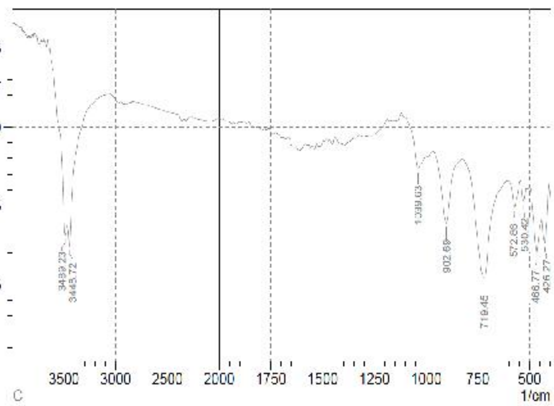


Figure 7 FT-IR spectrum for the corrosion product on zinc in the presence of CCL extract with 1.0 N Hydrochloric acid.

The Fig - 6 and 7 reflect that the FTIR spectrum of the ethanolic extract of inhibitor and the XRD analysis of Corrosion product on zinc in the presence of CCL extract in 1.0 N HCl. On comparing both of the spectra the prominent peak such as, the C-N stretching in amine is shifted from 1049.28 to 1039.63 cm^{-1}, 881.47cm^{-1} corresponds to S-OR stretching frequency is shifted to 719.45 cm^{-1}. These results also confirm that the FTIR spectra support the fact that the corrosion inhibition of CCL extract on Zinc in 1.0 N hydrochloric acid may be the adsorption of active molecule in the inhibitor and the surface of metal.

XRD Analysis

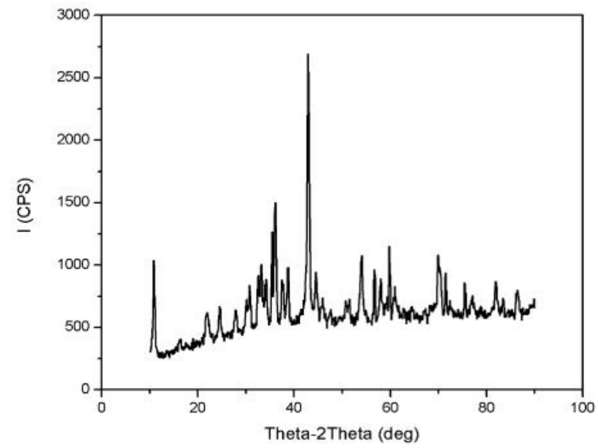


Figure 8 XRD- Analysis of Corrosion Productof the corrosion product on Zinc in the presence of CCL extract in 1.0 N HCl.

The Corrosion product released from the Zinc metal formed on their layer over the metal surface examined by XRD studies in the presence of inhibitor as shown in Fig. 8. It reveals that the film may be mainly combine with a rich amount of $Zn(N_3)_2$ and ZnS, $ZnCl_2$, $ZnCO_3$,ZnP_4 etc with inhibitor.

EDX Spectrum

EDX spectroscopy was used to determine the elements present on the Zinc surface in the absence and presence of inhibitor. Fig.9 and 10 represents the EDX spectra for the corrosion product on metal surface in the absence and presence of optimum concentrations of CCL extract in 1.0 N hydrochloric acid. In the absence of inhibitor molecules, the spectrum may concluded that the existence of chlorine due to the formation of metal chloride.

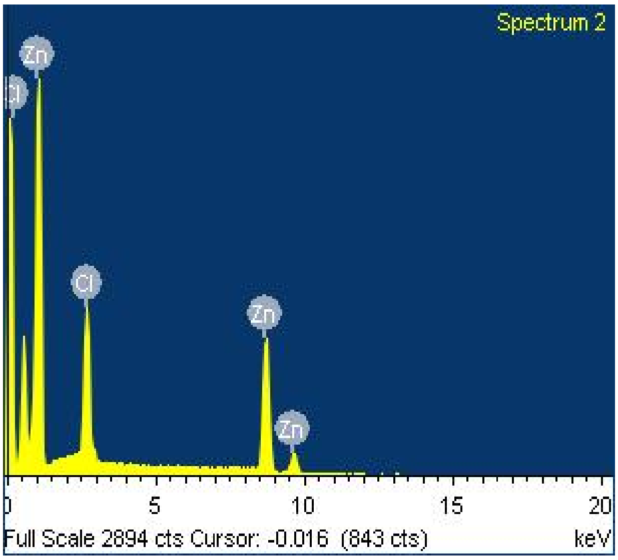


Figure 9 EDX spectrum of the corrosion product on Zinc surface in 1.0 N Hcl.

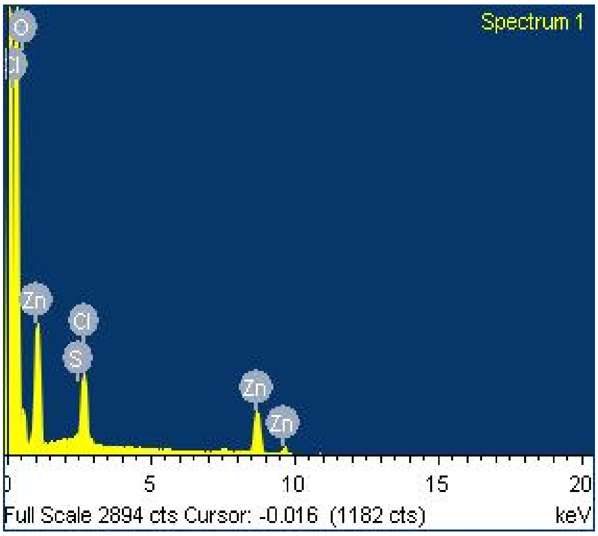
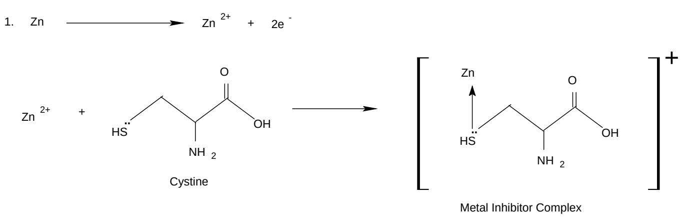
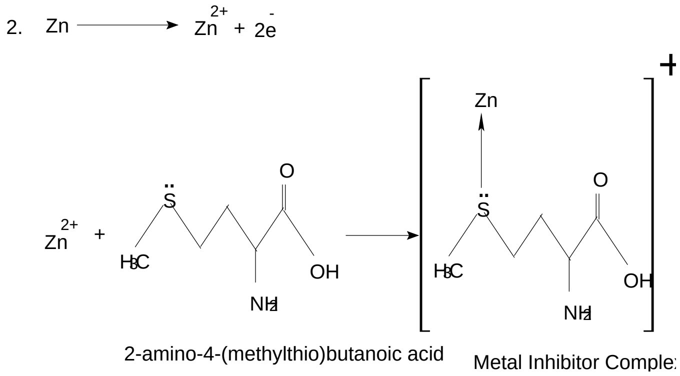


Figure 10 EDX spectrum of the corrosion product on Zinc in the presence of CCL extract in 1.0 N HCl

However, in the presence of the optimum concentrations of the inhibitors, sulphur 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 between metal atom and the active species such as cystine, 2-amino-4-(methylthio)butanoic acid during the adsorption process and prevent the further dissolution of metal against corrosion.

Proposed Mechanism





SEM Analysis

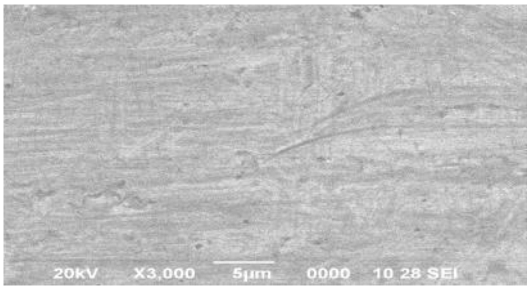
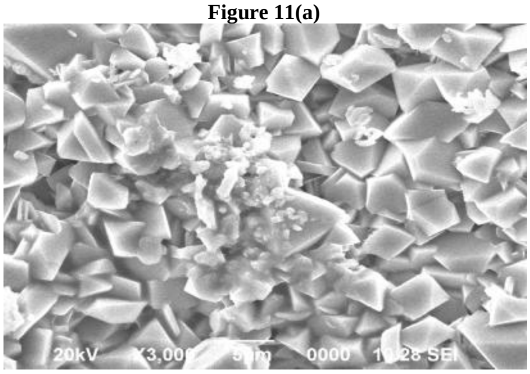
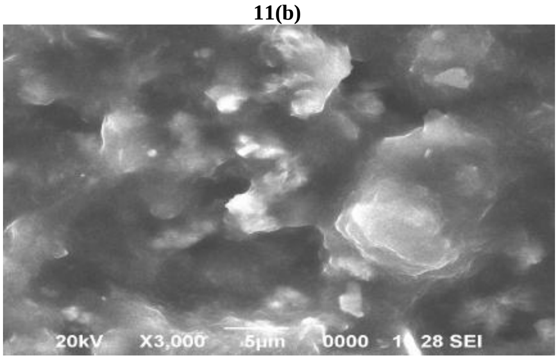


Figure 11(a)



11(b)



11(c)

Figure 11 SEM micrographs of Zinc (a) fresh Zinc (b) without inhibitor, 0 ppm (c) with inhibitor, 100 ppm.

The Surface morphology of Zinc was examined by scanning electron microscopy and the images are represented in Fig 11(a-c). Fig-11 (a) shows that the zinc sample before immersion seems smooth surface, but Fig-11 (b) showed that the surface of metal has number of pits and cracks in the surface, but in presence of inhibitor (Fig-11(c)) they are minimized on the metal surface.

It is clearly indicates that the formation of spongy mass covered the entire metal surface to reduce further dissolution of the metal.

CONCLUSIONS

Cnidoscolus Chayamansa leaves has shown excellent inhibition performance for zinc in 1.0 N HCl solution. The inhibition efficiency increased with the increase of inhibitor concentration. The maximum inhibition efficiency was achieved 73.5%. Also, the inhibition efficiency gradually increased with the rise in temperature ie, 69.54% to 82.09% for 303K and 333K respectively. This is due to the adsorption of active inhibitor molecules (cystine, 2-amino-4-(methylthio)butanoic acid) on the metal surface is higher than desorption process. It follows chemical adsorption mechanism . The value of activation energy (Ea), enthalpy of adsorption (ΔHads) and free energy changes (ΔGads) indicates that the adsorption of inhibitor on metal surface follows chemical, endothermic and spontaneous process respectively. The inhibitor is found to obey Langmuir, Temkin adsorption isotherms. The thin film formation on the metal surface may also be confirmed by SEM, EDX, FT-IR and UV spectral studies.

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