

Alcoholic extract of “*Gymnema Sylvestre*” leaves on mild steel in acid medium *Quinquefasciatus* say

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Abstract

Effect of “*Gymnema Sylvestre*” leaves extract on corrosion of mild steel in 1.0 N hydrochloric acid was investigated by mass loss measurement with various period of contact and temperature. The observed results indicate that the corrosion inhibition efficiency was increased with increase of inhibitor concentration and decreased gradually with rise in temperature. The maximum percentage of inhibition efficiency attained 96.92%. The thermodynamic parameters (viz; E_a , Q_{ads} , ΔH_{ads} , ΔG_{ads} , ΔS_{ads}) were evaluated for corrosion process, which suggest that the adsorption is exothermic, spontaneous and Physisorptions. The inhibitor follows Langmuir adsorption isotherm. The corrosion products formed on the metal surface was analyzed by using various spectroscopic studies UV, FT-IR and EDX techniques and the film formation also confirmed by SEM image.

Keywords: Mild Steel, corrosion inhibition, mass loss, adsorption, spectral studies

1. 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 [6]. Most of the synthetic chemicals are costly, toxic to both human being and the environment. In order to overcome, these difficulties choosing the inhibition are plenty, cheap, non-toxic 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* [6], *Phyllanthus amarus* [7], *Annona squamosa* [8], *Argan* [9], *Psidium guajava* [10], *black pepper* [11], *Punica granatum* [12], *Mentha pulegium* [13], *Cnidioscolus chayamansa* [14], *Solanum Torvum* [15], *Pisonia Grandis* [16], *mimusops elengi* [17], *Sauropus Androgynus* [18], *Kingiodendron pinnatum* [19], *Wrightia Tinctoria* [20] have also been used for inhibition of corrosion. In continuous of our research work, the present investigation is the *Gymnema Sylvestre* leaves extract used as corrosion inhibitor on mild steel in 1.0N HCl 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 UV, FT-IR EDX and the morphological studies by SEM-image.

2. Materials and methods

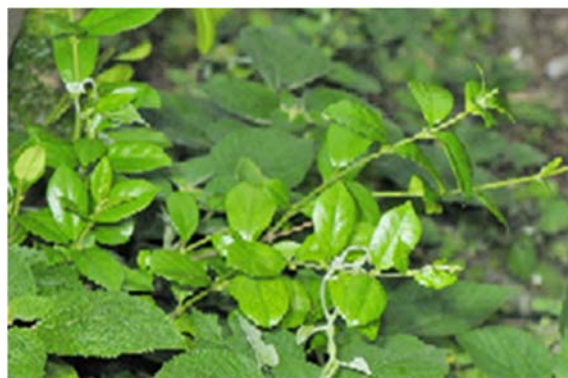
2.1 Specimen preparation

Mild steel specimen were mechanically pressed cut to form different coupons, each of dimension exactly 20cm² (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 investigation.

2.2 Preparation of *Gymnema Sylvestre* Leaves (GSL) Extract

About 3 Kg of “*Gymnema Sylvestre*” leaves was collected from in and around Western Ghats and then dried under shadow for 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 about 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.

Picture of *Gymnema Sylvestre* Leaves





2.3 Properties of *Gymnema Sylvestre* leaf

Gymnema Sylvestre is a plant used in India and parts of Asia as a natural treatment for diabetes or "sweet urine." The herb's active ingredient, gymnemic acid, is extracted from leaves and roots, and helps to lower and balance blood sugar levels. The main phytochemical constituents present in this plant are gymnemic acid, Stigmasterol, lupeol, hentriacontane and pentriacontane. A few structures are shown below

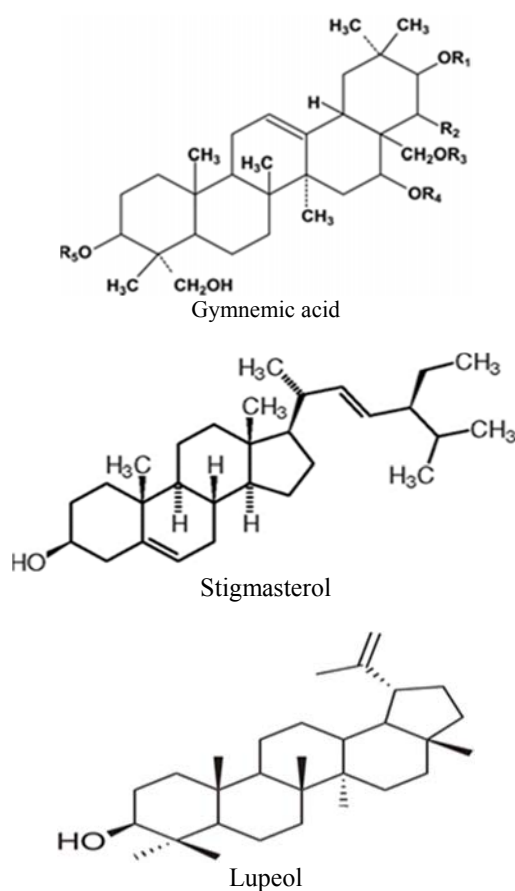


Fig 1: Chemical structure of the main active compounds Present in *Gymnema Sylvestre* leaves

2.4 Mass loss measurement

In the mass loss measurements the specimen of mild steel 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 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 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 following relationship.

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

Where

mmpy = millimeters 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.

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

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

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 metals in the presence and absence of inhibitors in 1.0N hydrochloric acid, natural sea water environment was calculated using Arrhenius theory. Assumptions of Arrhenius theory is expressed by equation (4).

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

$$\log(\text{CR}_2/\text{CR}_1) = E_a/2.303 R (1/T_1 - 1/T_2) \quad \text{---- (5)}$$

Where CR_1 and CR_2 are the corrosion rate at the Temperature T_1 (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 1.0N Hydrochloric acid, Natural sea water environment is calculated by the following equation (6).

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

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-7 is given below.

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

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.

2.5.4. Free energy of adsorption

The equilibrium constant of adsorption of various plant extract

on the surface of Mild steel is related to the free energy of adsorption ΔG_{ads} by equation (8).

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

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

3. Result and Discussion

3.1 Mass loss measurements

Dissolution behavior of Mild steel in 1.0N hydrochloric acid containing the absence and presence of GSL extract with various exposure times (24hrs to 360 hrs) are shown in Table-1. The observed values are clearly indicates that in the presence of GSL extract the value of corrosion rate decreased from

1.511 to 0.0464 mmpy for 24 hrs and 0.0542 to 0.0077mmpy for 360 hrs with increase of inhibitor concentration from 0 to 1000 ppm. The maximum percentage of inhibition efficiency attained 96.92 for 24 hrs and 85.73 for 360 hrs respectively. However %IE gradually decreased from 96.92 to 32.39 when the exposure time increase from 24 hrs to 120 hrs, clearly reveals that the initial formation of film may breakdown and leads to occurs further dissolution. But beyond this exposure time (120 hrs) the %IE increased to 85.73 even after 360hrs. 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⁻).

Table 1: The corrosion parameters of mild steel in 1.0N Hydrochloric acid containing different concentration of GSL extract after 24to 360 hours exposure time

Conc (ppm)	Corrosion rate (mmpy)					Inhibition efficiency (%)				
	24 hrs	72 hrs	120 hrs	240 hrs	360 hrs	24 hrs	72 hrs	120 hrs	240 hrs	360 hrs
0	1.5111	0.2092	0.3301	0.1139	0.0542	-	-	-	-	-
10	1.2786	0.1704	0.3161	0.1046	0.0433	15.38	18.51	4.22	8.16	19.99
50	0.5114	0.1394	0.2836	0.0953	0.0309	66.15	33.33	14.08	16.33	42.86
100	0.3952	0.1007	0.2696	0.0813	0.0232	73.84	51.85	18.31	28.62	57.15
500	0.2092	0.0464	0.2464	0.0674	0.0154	86.15	77.78	25.35	40.82	71.44
1000	0.0464	0.0387	0.2231	0.0418	0.0077	96.92	88.89	32.39	63.30	85.73

3.2 Temperature Studies

Dissolution behavior Mild Steel containing various concentration of GSL extracts in 1.0N hydrochloric acid with various Temperature ranges from 313K to 333K is investigated by mass loss measurements and the values are listed out in Table-2. The observed values of corrosion rate decreased from 19.5286 to 2.7898 mmpy with increase of inhibitor

concentrations. The maximum percentage of inhibition efficiency attained 85.71 % with increase of inhibitor concentration at 313 K. However the %IE gradually decreased due de-stability of film formation, when the temperature increased beyond 313K. This is suggest that the process is Physisorptions.

Table 2: The corrosion parameters of mild steel in 1.0N Hydrochloric acid containing different concentration of GSL extract at 313 to 333 K

Conc. (ppm)	Corrosion rate (mmpy)			Inhibition efficiency (%)		
	313K	323K	333K	313K	323K	333K
0	19.5286	29.0140	32.9197	-	-	-
10	15.6220	25.1082	23.9923	20.00	13.46	27.11
50	11.1592	21.7605	22.3184	42.85	25.00	32.20
100	8.3694	18.9707	18.9707	57.14	34.62	42.37
500	5.5796	16.1800	17.2968	71.42	44.23	47.45
1000	2.7898	13.3910	13.39	85.71	53.85	59.32

3.3 Effect of Temperature

3.3.1 Activation energy

The values of corrosion rate obtained from the mass loss measurement are substituted in equation (4) and the values of activation energy (E_a) are presented in Table-3. The observed values are ranged from 14.6044 to 43.8702 kJ/mol for mild

steel in 1.0N HCl containing various concentration of inhibitor. The average value of E_a obtained from the blank (14.6044) is lower than that in the presence of inhibitor and indicated that there is a weak forces between the GSL inhibitor molecules and the mild steel surface.

Table 3: Calculated values of Activation energy (E_a) and heat of adsorption (Q_{ads}) of GSL extract on Mild steel in 1.0N HCL environment.

S. No.	Conc. of inhibitor (ppm)	% of I.E		E _a (KJmol ⁻¹)	Q _{ads} (KJmol ⁻¹)
		30°	60°		
1.	0	-	-	14.6044	--
2.	10	20.00	27.11	11.9995	11.1054
3.	50	42.85	32.20	19.3856	-12.7745
4.	100	57.14	42.37	22.8862	-16.6450
5.	500	71.42	47.45	31.6426	-28.4721
6.	1000	85.71	59.32	43.8702	-39.5588

3.3.2. Heat of adsorption

The value of heat of adsorption (Q_{ads}) on mild steel in 1.0N HCL containing various concentration of GSL extract is calculated using Equation (6) and the values of Q_{ads} are ranged from -11.1054 to -39.5588 kJ/mol (Table-3). These negative values are reflected that the adsorption of GSL extract on Mild steel is follows exothermic process.

3.3.3. 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 suggest that the inhibitor follows Langmuir adsorption isotherm.

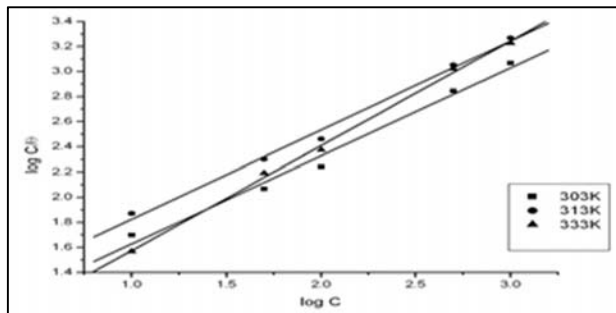


Fig 2: Langmuir isotherm for the adsorption of GSL inhibitor on Mild Steel in 1.0N HCL environment.

3.3.4. Free 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.

Table 4: Langmuir adsorption parameters for the adsorption of GSL inhibitor on Mild steel in 1.0 N HCL environment

Adsorption isotherms	Temperature (Kelvin)	Slope	K	R2	ΔG_{ads} (KJ/mol)
Langmuir	303	0.7009	8.4430	0.9865	-15.4948
	313	0.7098	13.0217	0.9929	-17.1339
	333	0.8352	5.4825	0.9979	-15.8333

3.3.5. Thermodynamics parameters

The another form of transition state equation which is derived from Arrhenius equation (4) is shown below (9)

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

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. 2)

with a slope of $(-\Delta H/R)$ and an intercept of $[\log(R/Nh) + (\Delta S/R)]$, 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 entropy (ΔS) is generally interpreted with disorder which may take place on going from reactants to the activated complex.

Table 5: Thermodynamic parameters of Mild Steel in 1.0N HCL obtained from weight loss measurements.

S. No.	Concentration of GSL (ppm)	ΔH (kJ mol ⁻¹)	ΔS (J k ⁻¹ mol ⁻¹)
1	0	4.7115	8.6462
2	10	3.3270	8.1252
3	50	6.3067	9.0022
4	100	7.5829	9.3355
5	500	11.0769	10.3627
6	1000	15.5875	11.6537

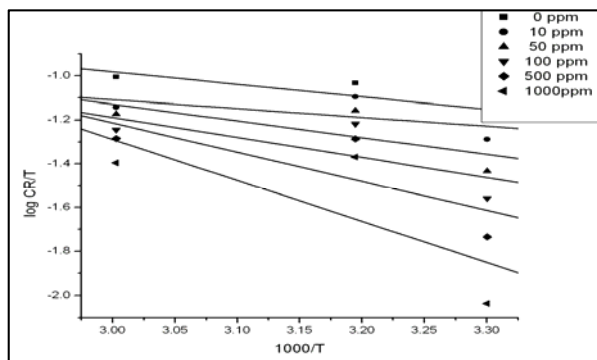


Fig 3: The relation between $\log(CR/T)$ and $1000/T$ For different concentrations of GSL extract

4. Spectral Studies

4.1 UV Analysis

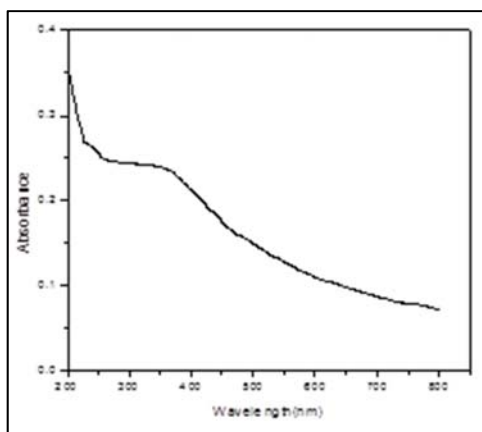


Fig 4.

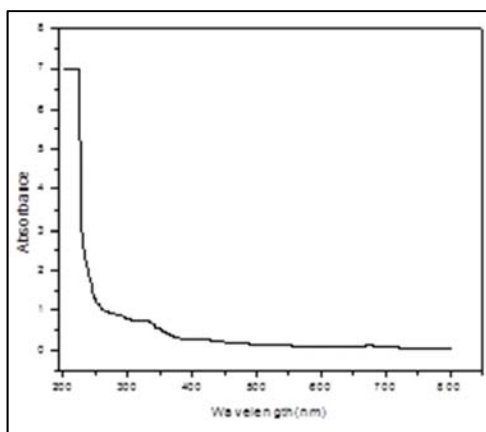


Fig 5.

Fig 4-5: UV spectrum of ethanolic extract of GSL and the corrosion product on Mild Steel in 1.0N HCL in the presence of GSL extract.

Fig 4 and 5 shows that the UV visible spectrum of ethanolic extract of GSL and the corrosion product on the surface of Mild Steel in the presence of GSL extract in 1.0N HCL respectively. In the absence of inhibitor, one absorption band around 360 nm were noticed and in the presence of inhibitor one broad band's was appeared (320 nm) which indicates the band is shifted to shorter wavelength region (i.e; Hypsochromic (or) Blue shift). It is clearly indicates that the extended conjugation of π -electron can able to co-ordinate with the metal atoms to prevent further dissolution.

4.2. FT-IR Analysis

FT-IR studies of GSL extract on Mild Steel surface in 1.0N HCL

The figures-6 and 7 reflect that the FTIR spectrum of the

ethanolic extract of inhibitor and the corrosion product on Mild Steel in the presence of GSL extract in 1.0N HCL. On comparing both of these spectra the prominent peak such as, the $-OH$ stretching frequency for alcohol is shifted from 3610.20 to 3734.19 cm^{-1} , the $-C-H$ stretching in aromatic alkane is shifted from 2920.23 cm^{-1} to 2889.37 cm^{-1} . The $-C-H$ stretching frequency for aromatic is shifted from 775.38 to 690.52 cm^{-1} . These observed results also strongly support the fact that the corrosion inhibition of GSL extract on Mild Steel in 1.0N HCL may prevent further dissolution of the metal ion from the surface.

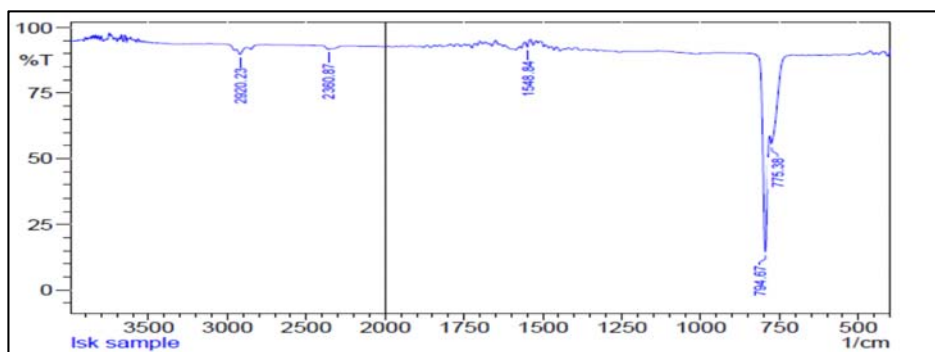


Fig 6: FT-IR spectrum of ethanolic extract of *Gymnema Sylvestre leaves* (GSL)

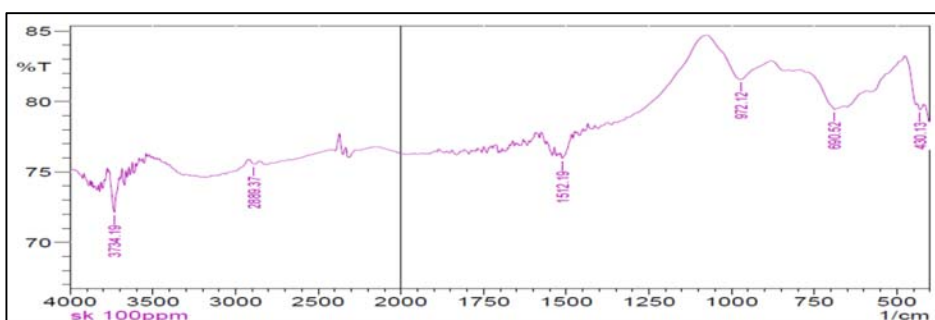


Fig 7: FT-IR spectrum for the corrosion product on Mild Steel in the

Presence of GSL extract with 1.0N HCL.

EDX Spectrum

EDX spectroscopy was generally used to determine the elements present on the Mild Steel surface in the absence and presence of inhibitor. Figures 8 and 9 represents the EDX spectra for the corrosion product on metal surface in the absence and presence of optimum concentrations of GSL extract in 1.0N HCL. In the absence of inhibitor molecules, the spectrum shows that the existence of oxygen present in the

metal. However, in the presence of the optimum concentrations of the inhibitors, chlorine, oxygen atoms are found to be present in the corrosion product on the metal surface. It clearly indicates that these hetero atoms such as oxygen can donate an unshared pair of electron to the olefins present in the inhibitor molecules may involve the complex formation with metal atom during the adsorption process and prevent the further dissolution of metal against corrosion.

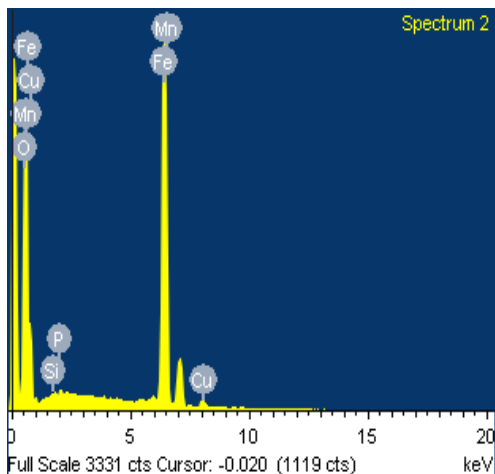


Fig 8.

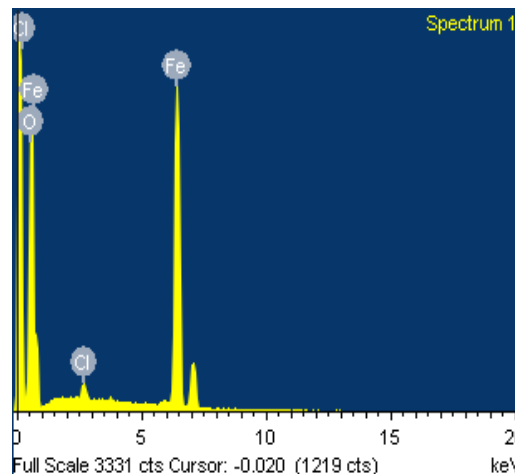


Fig 9.

Fig 8, 9: EDX spectrum of the corrosion product on Mild Steel surface in 1. 0N HCL and EDX spectrum of the corrosion product on Mild Steel in the presence of GSL extract in 1. 0N HCL.

SEM Analysis

The surface morphology of Mild Steel surface was studied by scanning electron microscopy (SEM). The Figures 10 and 11 shows that the SEM micrographs of Mild Steel surface before and after immersion in 1.0N HCL respectively. The SEM image (Fig-10) 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 with the film formation on the metal surface. It is clearly indicates that the formation of thin film on the metal surface almost complete area to protect further corrosion from the corrosive environments.

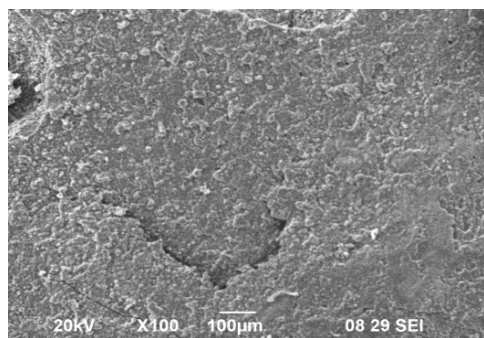


Fig 10.

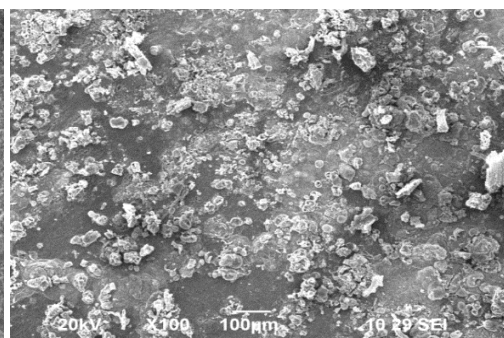


Fig 11.

Fig 10, 11: SEM image of the Mild Steel surfaces immersed in 1.0N HCL and SEM image of the Mild steel immersed in 1.0N HCL with GSL extract

Conclusions

Gymnema Sylvestre leaves has shown excellent inhibition performance on Mild Steel in acid environment. The inhibition efficiency increased with the increase of inhibitor concentration. The maximum inhibition efficiency was achieved 96.92%. Also, the inhibition efficiency gradually decreased with the rise in Temperature i.e. 85.71% and follows physical adsorption mechanism. The value of activation energy (E_a), enthalpy of adsorption (ΔH_{ads}) and changes of free energy

(ΔG_{ads}) indicates that the adsorption of inhibitor on metal surface follows physical, endothermic and spontaneous process respectively. The inhibitor is found to obey Langmuir adsorption isotherms. The thin film formation on the metal surface may also confirmed by SEM images and the corrosion products characterized by UV, FT-IR and EDX spectroscopy.

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