DISSOLUTION BEHAVIOUR OF MILD STEEL USING *RAPHANUS SATIVUS* EXTRACT IN HYDROCHLORIC ACID MEDIUM

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ABSTRACT *The inhibitive nature of RaphanusSativusleaves extract on MS in 1.0 N HCl has been investigated by using mass loss technique with various periods of contact as well as temperature. The observed result reveals that the percentage of inhibition efficiency increased with increase of inhibitor concentration, but decreases with period of contact and temperature. Thermodynamic parameters (Viz; E_a, Q_{ads} and ΔG_{ads}) suggests that the adsorption of RSL extract was exothermic, spontaneous and physisorption. It obeys Langmuir and Temkin adsorption isotherm. The corrosion product formed on the metal surface may also be confirmed by the spectral studies such as FT-IR, XRD and SEM-EDX.*

Keywords: *MS-Mild Steel, IE- Inhibition Efficiency, RSLE-RaphanusSativusleaves extract.*

I. INTRODUCTION

MS is the primary element have huge applications and subsequently are used nearly all the arena in global. It has ideal properties, which includes greater electrical and thermal conductivity, ease of fabricating, interconnecting and wide variety of practicable mechanical properties. But it's easily reacted with the polluted surroundings. The rust preventive activity of many manmade synthetic compounds was superior however maximum of them are extraordinarily harmful to each people and the encircling. The regarded risky outcomes of maximum synthetic organic inhibitors and restrictive ecological regulations have now made many researchers to recognize the necessity to develop inexpensive, non-hazardous and ecologically benign natural products as corrosion inhibitors such as *Gossipiumhirsutum L*[1], Guar Gum[2], Beet Root[3], *Tridaxprocumbens L*[4],*Isertia coccinea*[5], *Ocimum tenuiflorum*[6], *Cassia alata*leaves[7], *Eugenia Jambolana*[8], *Pyrus pyrifolia*[9], *Brugmansia-suaveoles*and *Cassia-roxburghii*[10], *Musa-paradisica*[11], *Zenthoxylum-alatum*[12], *Spirulina-platensis*[13], *Lupinous-albus*[14], *Occimum-viridis*, *Telferia-occidentalis*, *Azadirachta-indica*and *Hibiscus-sabdariffa* [15],*Medicago-sative*[16], *Areca-catechu*[17],*canna indica* [18],*murrayakoenigii*[19], *piper nigrum*[20] , *strychnosnuxvomica*[21] , *Albizialebbeck*seed [22] and *Hibiscus Esculenta*leaves [23]. In this existing examination, an attempt has been made to observe the dissolution of MS in acid environment containing alcoholic extract of *RaphanusSativus*leaves. Electrochemical and non-electrochemical analysis have been used to evaluate the inhibition performance of *Raphanussativus*leaves extract to decrease the dissolution procedure of MS in acid environment. Surface morphology of the metal became tested beneath Scanning Electron Microscope (SEM). Chemical constituents of the corrosion products found on the MS surface have been characterized by means of the analytical strategies viz; FT-IR, XRD and EDX.

II. MATERIAL AND METHODS

2.1 *Raphanussativus* leaves extract:

Raphanussativus leaves (RSL) were gathered from the source and dried below shadow for approximately 7 days, grinded well, then immersed in ethanol for about forty-eight hours. Then it's filtered accompanied with the aid of evaporation and the natural extract has been gathered. From this extract, different concentration of ten to thousand ppm stock solution prepared and used throughout our present research.

2.2 Steel sample (MS) preparation:

Commercially accessible metal specimens (5x2x2cm) were polished with distinct grade of emery wheel eighty and 120 then degreased with trichloro ethylene, washed with distilled water, wiped clean and dried, then saved in desiccators for our present research.

2.3 Non-electrochemical approach:

From the non-electrochemical approach, the corrosion rate (CR) has been calculated usage of the subsequent relationship.

$CR\ (mmpy) = [87.6 \times W] / DAT$ (2.1)

The Percentage of IE and degree of surface coverage (Theta (θ)) were calculated usage of equation two and three respectively.

$Percentage\ of\ IE = [(W_1 - W_2) / W_1] \times 100$ (2.2)

$Theta(\Theta) = (W_1 - W_2) / W_1$ (2.3)

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

2.3.1 Activation energy (E_a)

Assumptions of Arrhenius concept is expressed with the aid of equation 4 and its derived form (2.5).

$CR = A_{exp}\ (-E_a/RT)$ (2.4)

$\log (CR_2/CR_1) = E_a /2.303\ R\ (1/T_1-1/T_2)$ (2.5)

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

2.3.2.Heat of adsorption (Q_{ads})

$Q_{ads} = 2.303\ R\ [\log (\theta_2/1- \theta_2)-\log (\theta_1 /1- \theta_1)] \times (T_2T_1/T_2-T_1)$ (2.6)

In which R is the gas constant, $\theta_1$and θ_2 are the degree of surface coverage at temperatures T_1 and T_2 respectively.

2.3.3 Langmuir adsorption isotherm

$\log C/\theta = \log C - \log K$ (2.7)

Where in θis 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 steel surface.

2.3.4 Free energy of adsorption (δg_{ads})

$\Delta G_{ads} = -2.303\ RT \log (55.5\ K)$ (2.8)

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

2.4 Electrochemical approach:

2.4.1. Potentiodynamic polarization measurement

The corrosion rate in the presence [$I_{corr}(1)$] and in the absence [I_{corr}] of the inhibitors have been analyzed by Tafel extrapolation method. Percentage of IE has been derived by the following relationship.

$Percentage\ of\ IE = [(I_{Corr} - I_{Corr(1)}) / I_{Corr}] \times 100$ (2.9)

In which I_{Corr} is the corrosion current without inhibitor (blank) and $I_{corr(1)}$ is the corrosion current with inhibitor.

2.4.2. Impedance spectroscopy measurement (AC)

Inhibition efficiencies have been additionally derived from charge transfer resistance (R_{ct}) values with and without inhibitors through the usage of subsequent relationships.

$Percentage\ of\ IE = [(R_{ct} - R_{ct(1)}) / R_{ct}] \times 100$ (2.10)

III. RESULT AND DISCUSSION

In this examination, anti-corrosive performance of MS with the use of *Raphanussativus*Leaves (RSL) extract in 1.0N HCl environment had been completed by way of both electrochemical and non-electrochemical technique at variant of time and temperature.

3.1 Non-electrochemical approach:

3.1.1 Mass loss technique

Dissolution parameters of MS in 1.0 N HCl withdissimilar RSL concentration at various time of contact is obtained and placed in Table 3.1. Maximum of 80.77 percentage of IE is executed after 24 hrs exposure time. That is mainly due to the presence of hetero atoms such as sulphur, nitrogen and oxygen present in the inhibitor molecules can able to hold with the metal ions on the MS surface, very strongly and shield the metal ions from acidic environment.

Table 3.1: CR and IE Data of MS in 1.0N HCl comprising RSLE with various exposure time

Con. of Inhibitor (ppm)	CR (mmpy)						IE (%)					
	24 hrs	72 hrs	120 hrs	168 hrs	216 Hrs	360 hrs	24 hrs	72 hrs	120 hrs	168 hrs	216 hrs	360 hrs
0	95.53	52.31	36.55	30.26	31.46	22.94	-	-	-	-	-	-
10	66.73	39.21	28.41	23.71	24.82	19.05	30.14	25.04	22.27	21.65	21.11	16.96
50	53.70	31.85	23.48	20.69	21.72	15.85	43.79	39.11	35.76	31.63	30.96	30.91
100	38.36	23.48	17.85	15.94	18.24	13.49	59.85	55.11	51.16	47.32	42.02	41.19
500	22.08	13.25	9.58	9.00	11.42	8.73	76.89	74.67	73.79	70.25	63.69	61.94

1000	18.37	11.39	8.18	7.00	7.88	6.12	80.77	78.23	77.62	76.87	74.95	73.32

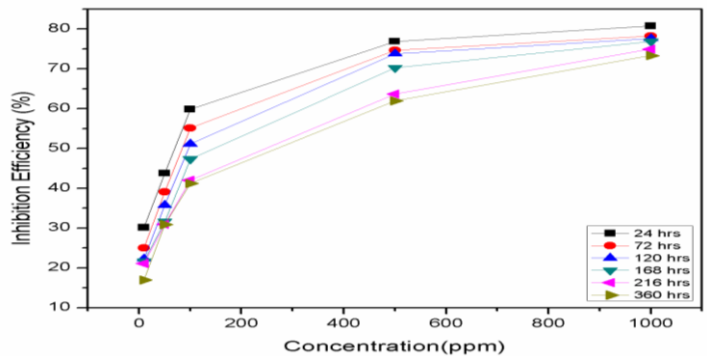

Fig 3.1: IE Diagram of MS in 1.0N HCl comprising RSLE with various exposure time.

3. 1.2. Effect of Temperature

Corrosion Rate (CR) and IE for MS in acid media with the temperature range from 303K to 333K in presence and absence of RSL extract are given in Table 3.2. Interpretation of those effects obviously shows that the corrosion rate increases with temperature readily in the absence of inhibitor. Also, the same trend has been noticed with the rise in temperature. The efficiency reached a high value of 79.09 % in 1.0N HCl solution at 303 K, which represents wonderful inhibitive capability of *Raphanussativus*leaves extract. However, the outcomes indicate the process is physisorption.

Table 3.2: CR and IE Data of MS in 1.0N HCl comprising RSLE with various temperature

Con. of inhibitor (ppm)	CR (mmpy)				IE (%)			
	303 K	313 K	323 K	333 K	303 K	313 K	323 K	333K
0	1467.4	1740.8	2170.5	2432.7	-	-	-	-
10	1182.8	1456.3	1791.1	2070.0	19.39	16.35	17.48	14.90
50	870.4	1171.7	1500.9	1746.4	40.65	32.69	30.85	28.21
100	675.1	931.8	1177.3	1439.5	53.99	46.47	45.76	40.83
500	491.0	675.1	909.5	1054.6	66.54	61.22	58.09	56.65
1000	306.8	424.1	585.9	675.1	79.09	75.64	73.01	72.25

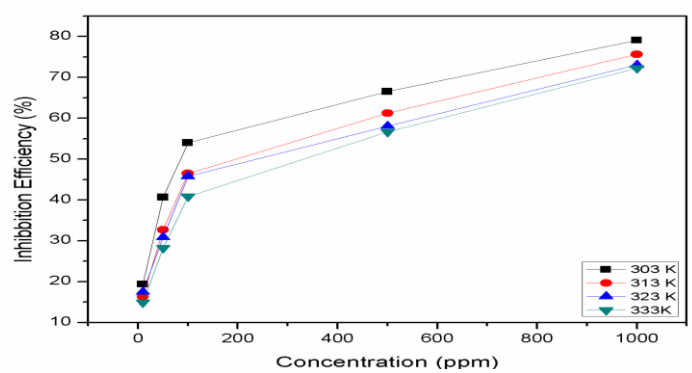

Fig 3.2: IE Diagram of MS in 1.0N HCl comprising RSLE with various temperature

3.1.3. Thermodynamic and Adsorption Parameters:

3.1.3.1. Activation Energy:

Table 3.3: Data of E_a and Q_{ads} of RSLE on MS in 1.0N HCl

Con. of inhibitor (ppm)	Activation energy(E_a) (kJ/mole)	Heat of adsorption (Q_{ads}) (kJ/mole)
0	71.7309	-
10	72.1809	-8.88
50	73.3179	-15.532
100	73.8238	-5.901
500	73.8838	-11.739
1000	74.0838	-10.425

Table3.3 reflects that the higher values for E_a were found in the presence of inhibitors.E_a increased with increase of the inhibitor concentration, and all the observed values of E_a were higher than that in the absence of *Raphanussativus*leaves extract. The negative values of Q_{ads}suggest that the adsorption of inhibitors on mild steel surface is an exothermic and spontaneous process.

3.1.3.2. Langmuir Adsorption Isotherm (LAI):

Table 3.4: LAI for the Adsorption of RSLE on MS in 1.0N HCl

Log C	Log(C/θ)			
	303K	313K	323K	333K
1	1.7124	1.7865	1.7575	1.8268
1.6989	2.0899	2.1845	2.2097	2.2486
2	2.2677	2.3328	2.3395	2.3890
2.6989	2.8759	2.9121	2.9349	2.9458
3	3.1018	3.1212	3.1366	3.1412

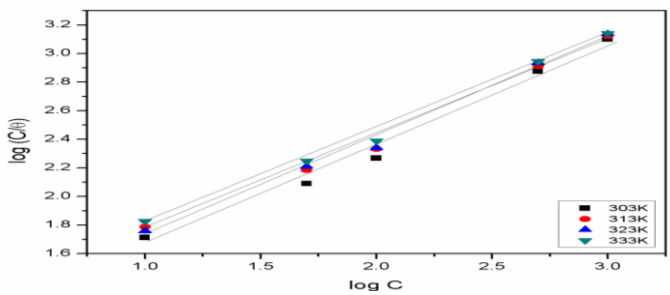

Fig 3.3: Diagram of LAI for the adsorption of RSLE on MS in 1.0N HCl

Adsorption of RSL inhibitor on the surface of mild steel is best described with the aid of Langmuir isotherm. By plotting the values of log (C/θ) Vs log C, linear plots were created (Fig 3.3). It is seen that Langmuir adsorption isotherm is relevant at 333 K than at 303K. The regression co-efficient (R^2) of the adsorption isotherm data show that Langmuir adsorption is best fitted into the study with R^2 ranges from 0.9829 to 0.9919.

3.1.3.3. Temkin Adsorption Isotherm (TAI):

Table 3.5: TAI for the adsorption of RSLE on MS in 1.0N HCl

Log C	θ			
	303K	313K	323K	333K
1	0.1939	0.1635	0.1748	0.1490
1.6989	0.4065	0.3269	0.3085	0.2821
2	0.5399	0.4647	0.4576	0.4083
2.6989	0.6654	0.6122	0.5809	0.5665
3	0.7909	0.7564	0.7301	0.7225

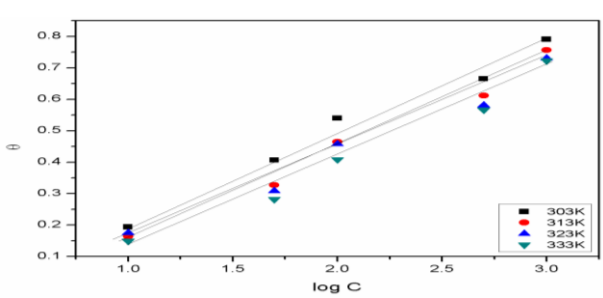

Fig 3.4: Diagram of TAI for the adsorption of RSLE on MS in 1.0N HCl

A plot of θ verses logC gives almost a straight line for mild steel in RSLE in acid medium (Fig 3.4 & Table 3.5). The straight line point out that the inhibitor obeyed Temkin adsorption isotherm.

The positive value of K_{ads} for Langmuir and Temkin adsorption isotherms are indicative of favourable adsorption.

3.1.3.4. Free Energy of Adsorption (FEA):

Table 3.6: FEA of RSLE on MS in 1.0N HCL

Adsorption isotherm	Temperature (K)	Slope	K	R^2	ΔG_{ads} (kJ mol^{-1})
Langmuir	303	0.7111	8.5251	0.9829	-15.519
	313	0.6794	11.3404	0.9874	-16.774
	323	0.6988	10.5307	0.9906	-17.111
	333	0.6664	13.3201	0.9919	-18.291
Temkin	303	0.2877	0.8334	0.9801	-9.660
	313	0.2895	0.7290	0.9811	-9.631
	323	0.2706	0.7722	0.9657	-10.093
	333	0.2807	0.6948	0.9699	-10.113

Table 3.7: The relation between log (CR/T) and 1000/T for RSLE on MS in 1.0N HCL

1000 / T	Log (CR / T)					
	0 ppm	10 ppm	50 ppm	100 ppm	500 ppm	1000 ppm
3.300	0.6851	0.5915	0.4583	0.3479	0.2096	0.0055
3.194	0.7452	0.6677	0.5734	0.4738	0.3338	0.1319
3.096	0.8274	0.7439	0.6672	0.5617	0.4496	0.2586
3.003	0.8636	0.7935	0.7197	0.6358	0.5006	0.3069

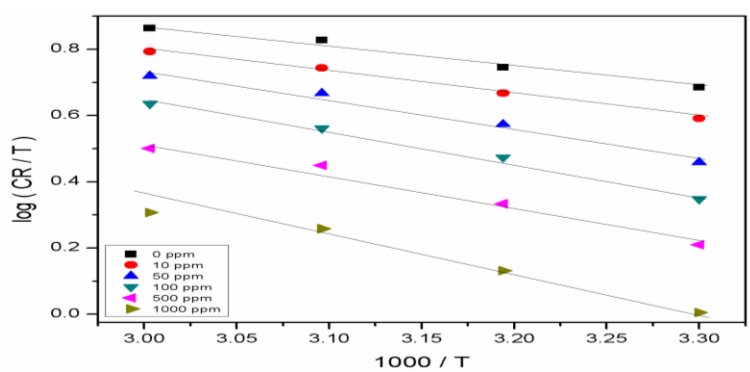

Fig 3.5: Diagram of the relation between log (CR/T) and 1000/T for RSLE on MS in 1.0N HCL

A plot of log (CR/T) versus 1000/T was shown in Fig 3.5. Straight lines were acquired with slope (−ΔH/2.303 R) and intercept of [log (R/Nh)+(ΔS/2.303 R)] from which ΔH and ΔS were calculated and are listed in Table 3.8.

Table 3.8: Thermodynamic parameters of MS in 1.0N HCl of RSLE

Con. of inhibitor (ppm)	ΔH (kJ/mole)	ΔS (kJ/mole)
0	71.7309	-
10	72.1809	-8.88
50	73.3179	-15.532
100	73.8238	-5.901
500	73.8838	-11.739
1000	74.0838	-10.425

Inspection of these data revealed that the enthalpy (ΔH) for dissolution reaction of MS in 1.0N HCl in the presence of RSL inhibitor concentration was better (72.1809 to 74.0838 kJ mol−1) than that in the absence of inhibitor (71.7309 kJ mol−1). The negative entropy in the existence of the inhibitor signifies the generation of more ordered firm layer of active molecules of the extracts on the mild steel surface as extract concentration increases.

3.2. Electrochemical approach:

3.2.1. Potentiodynamic Polarization Measurements:

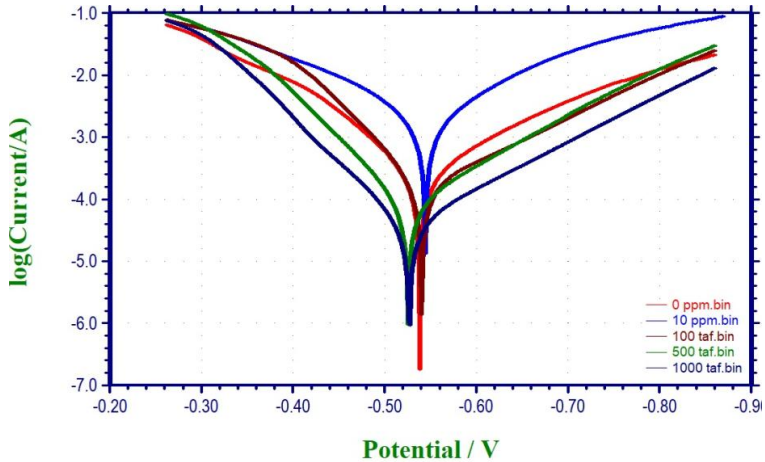

Fig 3.6: Tafel plot of MS in 1.0 N HCl containing various conc. (0 to 1000 ppm) of RSLE

Potentiodynamic polarization computation of MS in 1.0N HCl having distinctive concentration of RSLE was studied and the observed statistics are placed in Table-9 (Fig-6). It was found out that the corrosion current density (I_{corr}) decreased from 263.9 to 44.7 µA/ cm^2 with increase of inhibitor concentration. It is indicate that the inhibition action can attributed by the sulphur and nitrogen atoms can donate its lone pair of electrons to the metal surface to absorb more easily and hence reduce the corrosion rate of the metal. The values of corrosion potential (E_{corr}) was shifted to positive direction (ie. From -540.1 to -526.3 mv) with increase of inhibitor concentration indicates that the inhibitor has stronger influence on anodic reaction. The maximum percentage of IE was attained 83.07 % with increase of inhibitor concentration (1000 ppm). These results are in good agreement with previous data.

Table 3.9: Parameters derived from polarization measurements of MS in 1.0 N HCl containing various concentration of RSLE

Con. Of inhibitor (ppm)	$-E_{corr}$ (mV)	b_a (mV/decade)	b_c (mV/decade)	I_{corr} (µA/ cm^2)	I.E (%)
0	540.1	88.75	129.09	263.9	-
10	539.2	149.3	129.01	226.3	14.28
100	537.3	68.88	146.09	192.9	26.90
500	524.4	68.18	119.82	87.2	66.96
1000	526.3	73.00	130.87	44.7	83.07

3.2.2. Electrochemical Impedance Spectroscopy Measurements:

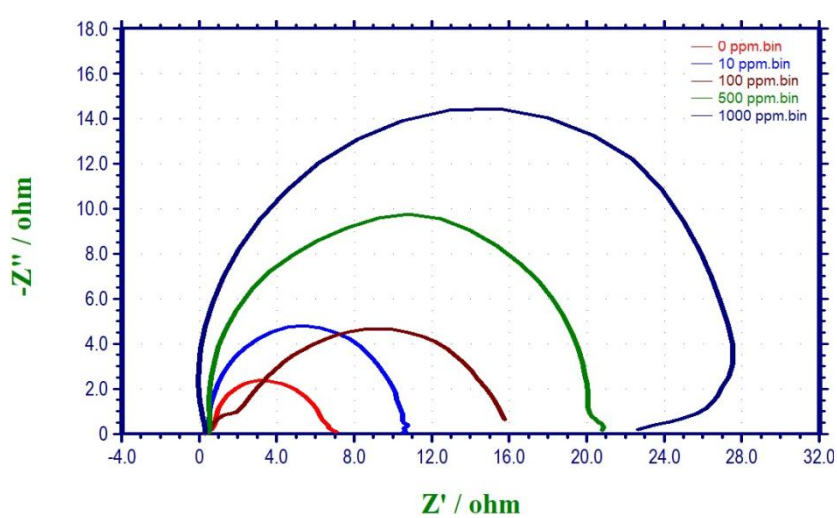

Fig 3.7: Nyquist plot of MS in 1.0 N HCl containing various concentration of RSLE

Table 3.10: Parameters derived from impedance measurements of MS in 1.0 N HCl containing various concentration of RSLE

Con. of inhibitor (ppm)	R_{ct} (Ω cm^{-2})	C_{dl} (μF cm^{-2})	I.E (%)
0	6.379	0.0073	-
10	10.202	0.0031	37.47
100	15.262	0.0011	58.20
500	20.460	0.0007	68.82
1000	27.260	0.0004	76.59

It is clear from Table-10, that the Nyquist plot shows there was a regular increase in the diameter of each semicircle which is due to the increase in the number of inhibitive molecules present in the extract when the concentration was raised from 0 to 1000 ppm. This increase of the diameters has remarkably confirmed that the corresponding R_{ct} values increased with increase in the inhibitor concentration. It was found out that the addition of inhibitor extract increase the values of R_{ct} from 6.379 to 27.260 Ω cm^{-2}. The value of the double layer capacitance (C_{dl}) decreased with increase in the *Raphanussativus*Leaves (RSL) extract concentration. This observed result suggests that the RSLE inhibited the corrosion of MS in 1.0N HCl environment.

3.2.3. Cyclic Voltammetry:

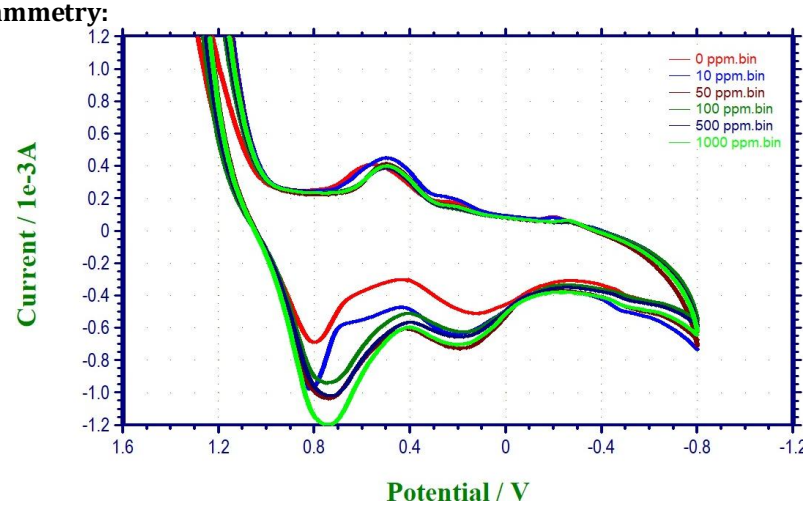

Fig 3.8: Cyclic voltammograms in the absence and presence of RSLE in MS immersed in 1.0N HCl

Cyclic voltammetry was used to continuously screen the changes of current passed through the electrode during the immersion time. These changes are associated to the adsorption behaviour of the inhibitors on the electrode surface. In this technique, the potential waveform was constantly implemented during the experiment and variation of the charge under peak was monitored. As a result, a massive wide variety of CV curves were recorded and displayed in actual time, which might be used to observe the changes in MS surface and the adsorption process.

3.3. Morphological Studies:

3.3.1. FT-IR Analysis:

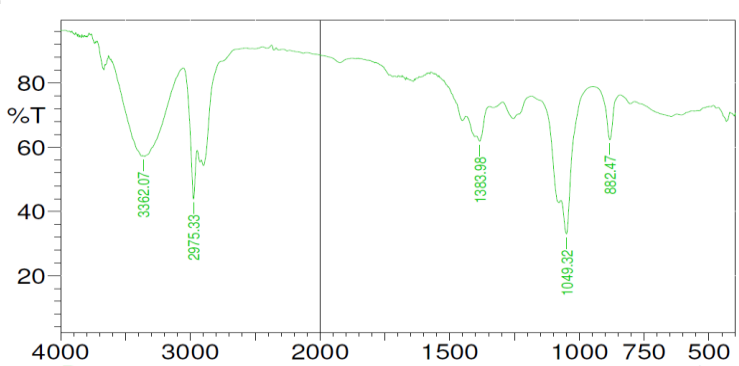

Fig 3.9: FT-IR spectrum of ethanolic extract of RSLE

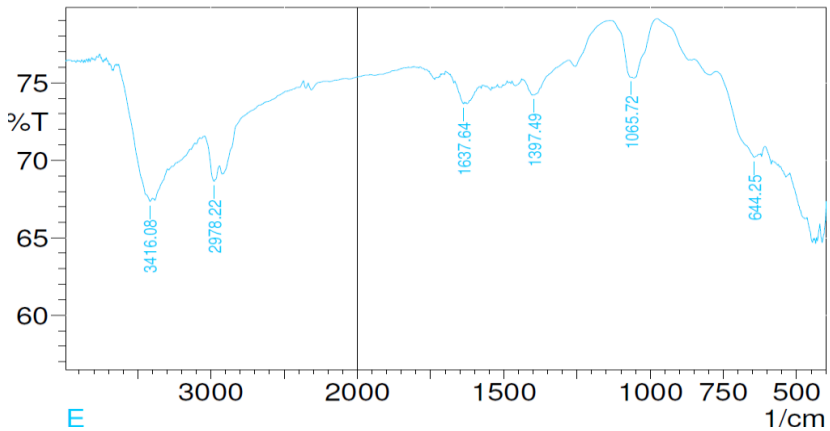

Fig 3.10: FT-IR spectrum for the corrosion product on MS in the presence of RSLEwith 1.0 N HCl

Fig 3.9 and 3.10 represents that the ethanolic crystal of RSLE and the corrosion product on the MS surface in the presence of RSL inhibitor. On comparing both the spectra, the –NH stretching frequency at 3362.07 cm^{-1} is shifted to 3416.08 cm^{-1}, the –CH stretching frequency at 2975.33 cm^{-1} I shifted to 2978.22 cm^{-1} and S-O stretching frequency at 1049.32 cm^{-1} is shifted to 1065.72 cm^{-1} clearly indicating that there is an interaction between the inhibitor and the MS surface.

3.3.2. EDX Analysis:

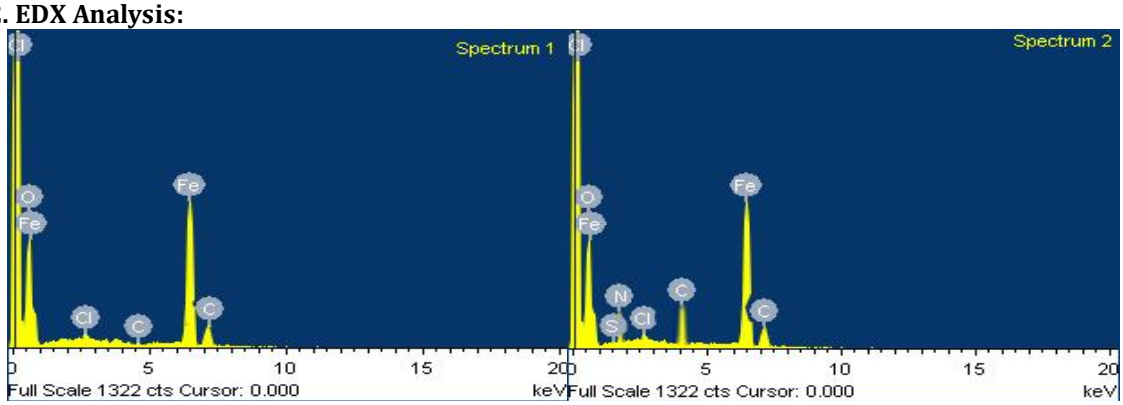

Fig 3.11: EDX spectrum of the corrosion product on MS in the absence and presence of RSLE in 1.0 N HCl

Fig 3.11 represents the EDX spectra for the corrosion product on MS surface in the absence and presence of optimum concentrations of RSLE in 1.0N HCl. In the absence of inhibitor spectrum consists of oxygen atom is present in the form of metal oxide. However, in the presence of the optimum concentration of the inhibitors, nitrogen and sulphur atoms are found to be present in the corrosion product on the MS surface. It clearly indicates that these hetero atoms present in the inhibitor may involve the complex formation with metal ion during the adsorption process and prevent the further dissolution.

3.3.3. XRD Analysis:

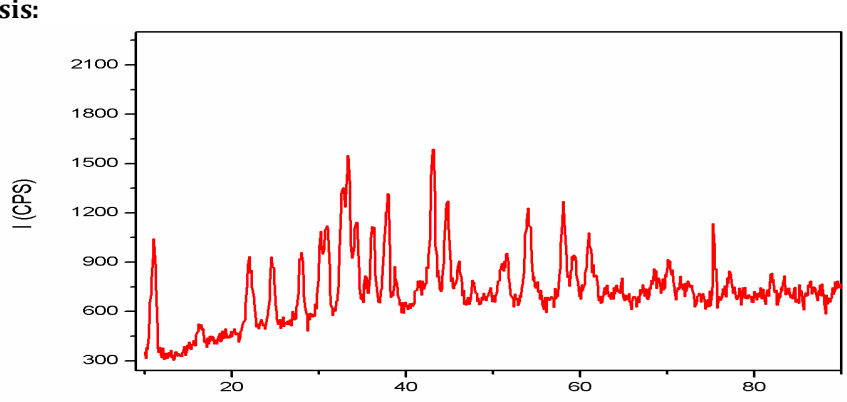

Fig 3.12: XRD spectrum of the corrosion product on MS in the presence of RSLE in 1.0 N HCl

The Corrosion product released from MS formed on their layer over the metal surface examined by XRD studies in the presence of inhibitor as shown in Fig 3.12. It reveals that the film may be mainly combining with a rich amount of FeS, $FeCl_2$, FeOetc with inhibitor.

3.3.4. SEM Analysis:

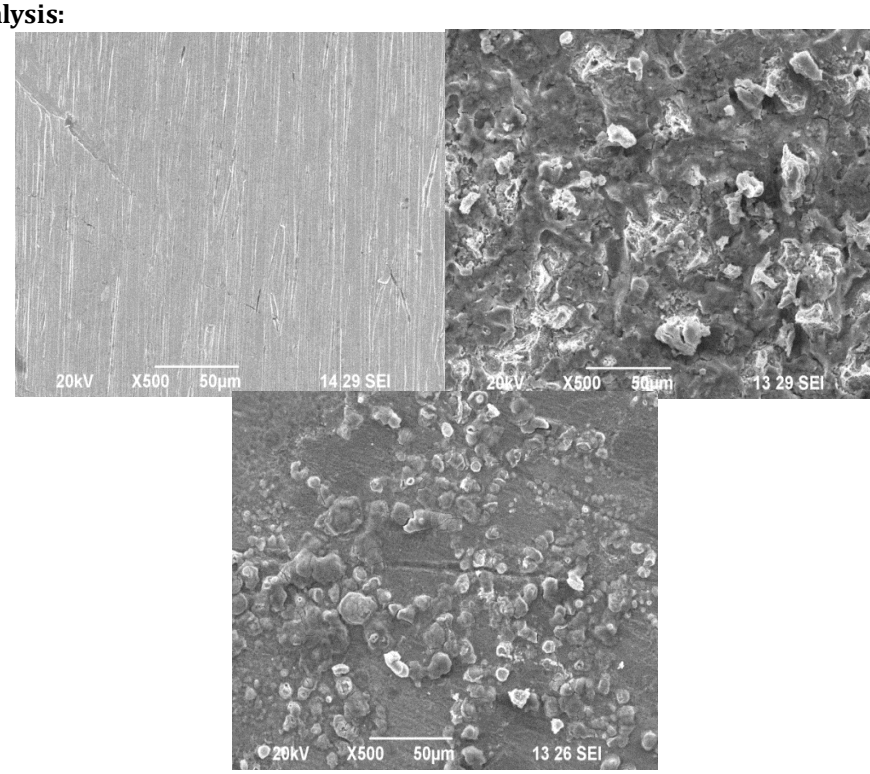

Fig 3.13(c):SEM micrograph of polished MS surface, MS in 1.0 N HCl in the absence and presence of RSLE

A SEM micrograph of the polished MS surface before immersion in 1.0N HCl is shown in Fig 3.13. This micrograph shows that surface is smooth and without any pits. The SEM image of the corroded MS in 1.0N HCl solution is shown in Fig 3.13 showed that the metal surface has number of pits. However, in the presence of the inhibitor Fig 3.13, the entire surface is almost covered with spongy film formation might be concluded that the adsorption can more efficiency inhibit the corrosion of MS surface.

IV CONCLUSION

This investigation proves that *Raphanussativus*leaves extract have great ability to inhibit the dissolution of MS in 1.0 N HCl. Results obtained from electrochemical and non-electrochemical methods are considerably in good agreement and confirmed that the inhibitor efficiency increased with increase of inhibitor concentration. It has been proved that the *Raphanussativus*leavesextract acts as anodic type inhibitor and obey the results of EIS revealed that the decrease of corrosion current I_{corr} by Polarization studies and increase in the charge transfer resistance and a decrease in double layer capacitances by EIS when the inhibitor is added and hence an increase in percentage of IE.

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