

Corrosion inhibition of mild steel in acid medium using *Talinum Fruticosum* as an eco-friendly inhibitor

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Abstract: The inhibition effect of *Talinum fruticosum* leaves extract (TFL) on corrosion of mild steel in 1N HCl was investigated through mass loss measurements with various time and temperature and electrochemical techniques such as Potentiodynamic polarization, Electrochemical Impedance Spectroscopy (EIS) measurements. In this investigation the percentage of inhibition efficiency is increased with increase of inhibitor concentration. The maximum of 83.2% inhibition efficiency is achieved even after 360 hrs exposure time. In temperature studies, the observed results reflect that the percentage of inhibition efficiency is increased with increase of temperature and it suggests that the mechanism follows chemical adsorption. Both kinetic as well as thermodynamic parameters are calculated and discussed in detail. In electrochemical studies, Tafel plot shows, the corrosion current density (I_{corr}) values decreased with increase of inhibitor concentration. The inhibition efficiency of TFL extract attained a maximum value of 81.43% at 1000 ppm of inhibitor concentration. The value of charge transfer resistance (R_{ct}) increased with increase in the inhibitor concentration. The values of double layer capacitance (C_{dl}) tend to decrease and the inhibition efficiency attained maximum of 85% with rise in inhibitor concentration. These results also in good agreement with those observed from the mass loss data. The thin film formation on the metal surface may also be confirmed by FT-IR, EDX spectral studies and the morphology of the specimen was analysed by SEM image.

IndexTerms -TFL-*Talinum fruticosum* leaves, MS-Mild Steel, IE-Inhibition efficiency.

I. INTRODUCTION

MS is the first, and generally the most weldable class of steel. MS is tough, ductile and malleable. It is mainly used as an all-purpose engineering material. Its malleability makes it ideal for use in constructing pipelines, and it is also ideal as structural steel, such as in the automotive industry. MS has good tensile strength but poor corrosion resistance. Corrosion control of metal is of technical, economic, environmental and aesthetical importance. It is a constant and continuous problem, often cannot be eliminated completely. Prevention is more practical and achievable than complete elimination. Among the various methods to avoid or prevent destruction or degradation of metal surface, the corrosion inhibitor is one of the best-known methods of corrosion protection and one of the most useful on the industry. This method is following stand up due to low cost and practice method [1,2]. Large number of organic compounds have been studied and are still being studied to assess their corrosion inhibition potential. However, most of these substances are not only expensive but also possess health and environmental hazards prompting the search for their replacement. Plants represent a class of interesting source of compounds currently being explored for use in metal corrosion protection in most systems, as possible replacement of toxic synthetic inhibitors. The green corrosion inhibitors are bio degradable, non-toxic, environmentally benign, and low cost, are obtained from renewable resources with minimal health and safety concerns. Presence of hetero atom (S, N, O) with free electron pairs, aromatic rings with delocalized π -electrons, high molecular weight alkyl chains, substituent group in general improves inhibition efficiency. Examiners namely Sobhi [3], Rajam et al. [4], Saratha et al. [5], Sangeetha et al. [6], Okafor et al. [7], Abu-Dalo et al. [8], Cang Hui et al.[9], Vasudha et al.[10], Bright et al. [11-18], Deeparani et al. [19-22], Petchiammal et al. [23-25], A.Sharmila et al. [26], Deivanayagam et al. [28,29], Malarvizhi et al.[30], Patel et al.[31] and Muruganandam et al. [32], have been described the great inhibition effect of various green inhibitors on various metals. In continuity of our research work the present study explain the inhibiting properties of the *Talinum fruticosum* leaves (TFL)extract in 1 N HCl using weight loss and electrochemical methods.

II. MATERIAL AND METHODS

2.1. Stock solution of TFL extract

TFL were collected from the various source and dried under shadow for about 168 hours, grinded well, then soaking in ethyl alcohol for about 3 days. Then it is filtered followed by evaporation in order to eliminate the ethanol completely and the pure 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.

2.2. Specimen preparation

MS specimens were mechanically pressed cut to form different coupons, each of dimension exactly 20cm² (5x2x2cm) with emery wheel of 80, 120 and degreased with trichloroethylene, washed with distilled water, cleaned and dried, then stored in desiccators for our present study.

2.3 Mass loss technique

In mass loss technique, MS specimens in triplicate were completely immersed in 100ml of the test solution in the presence and absence of the TFL extract. The specimens were withdrawn from the test solutions after immersion of 24 to 360 hours at room temperature and different temperature ranges from 303K to 333K after an hour. The mass loss was measured 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.

$$\text{Corrosion Rate (mmpy)} = [87.6 \times W] / \text{DAT} \quad (2.1)$$

W = Mass loss (mg), D = Density (gm/cm³), A = Area of specimen (cm²), T = time in hours.

The % of inhibition efficiency (IE) and degree of surface coverage (θ) were calculated using equation (2.2) and equation (2.3) respectively.

$$\% \text{ IE} = [(W_1 - W_2) / W_1] \times 100 \quad (2.2)$$

$$\theta = (W_1 - W_2) / W_1 \quad (2.3)$$

Where, W_1 and W_2 are the corrosion rates in the absence and presence of the TFL extract respectively.

2.3.1 Activation energy (E_a)

Assumptions of Arrhenius concept is expressed with the aid of equation (2.4) and its derived form (2.5).

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

$$\log (\text{CR}_2/\text{CR}_1) = E_a / 2.303 R (1/T_1 - 1/T_2) \quad (2.5)$$

Where, 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})

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

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.

2.3.3 Free energy of adsorption (ΔG_{ads})

$$\Delta G_{\text{ads}} = -2.303 RT \log (55.5 K) \quad (2.7)$$

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

2.3.4. Langmuir adsorption isotherm

The Langmuir adsorption isotherm can be expressed by the following Equation

$$\log C/\theta = \log C - \log K \quad (2.8)$$

2.4. Electrochemical techniques:

2.4.1. Potentiodynamic polarization measurement

From Tafel plot, the percentage of inhibition efficiency (IE) was determined by the following relationship.

$$\% \text{ IE} = [(I_{\text{Corr}} - I_{\text{Corr}(1)}) / I_{\text{Corr}}] \times 100 \quad (2.9)$$

2.4.2. Impedance spectroscopy measurement

From Nyquist plot, the percentage of inhibition efficiency (IE) was determined by the following relationship.

$$\% \text{ IE} = [(R_{\text{ct}} - R_{\text{ct}(1)}) / R_{\text{ct}}] \times 100 \quad (3.0)$$

III RESULT AND DISCUSSION

3.1. Mass loss Technique:

In our present study, dissolution control of MS using TFL extract in 1N HCl were carried out by mass loss method at distinctive time and temperature. For this we dipped the polished MS pieces in various concentrations of inhibitor solutions for different contact period (24 to 360hrs) as shown in table 3.1. The IE values calculated using mass loss data, for different concentrations of TFL extract using MS in 1N HCl solutions are presented in table 3.1. It is seen that the IE increased with increase of inhibitor concentration. In the presence of TFL inhibitor, the corrosion rate reduced from 11.856 mmpy to 4.417 mmpy after 24 hours and 6.029 mmpy to 1.007 mmpy even after 360 hours respectively. The maximum of 83.2 % of inhibition efficiency was obtained at higher concentration of the extract. These achievements mainly due to the presence of hetero atoms such as nitrogen and oxygen in the TFL extract.

Table 3.1: The mass loss and IE of MS in 1 N HCl containing various concentration of TFL extract with different contact period

Con. of inhibitor (ppm)	Corrosion rate (mmpy)						Inhibition efficiency (%)					
	24 Hrs	72 hrs	120 hrs	168 hrs	240 hrs	360 hrs	24 hrs	72 hrs	120 hrs	168 hrs	240 hrs	360 hrs
0	15.111	9.376	6.463	5.479	4.324	6.029	-	-	-	-	-	-
10	11.856	7.284	4.928	4.051	3.045	4.200	21.5	21.3	23.7	26.1	29.6	30.3

50	9.764	5.889	4.091	3.354	2.534	3.270	35.4	37.2	36.7	38.8	41.4	45.8
100	8.601	4.882	3.347	2.823	2.115	2.526	43.1	47.9	48.2	48.5	51.1	58.1
500	5.579	3.332	2.231	1.826	1.394	1.875	63.1	64.46	65.5	66.7	67.7	68.9
1000	4.417	2.634	1.766	1.361	0.976	1.007	70.8	71.9	72.7	75.2	77.4	83.2

3.2. Effect of temperature

MS pieces are dipped in acid with different temperatures (303 to 333 K) and the influence of temperature on MS in 1N HCl using TFL extract was examined and the results are presented in table 3.2. The inhibition efficacy increased with increase of extract concentration and reached a maximum of 81.43% at 333K. This is due to the formation of bond between the metal surface and the TFL extract molecule. This exhibits the dissolution of the MS increased at elevated temperatures. This reflection is attributed to the general rule that the rate of chemical reaction, which says that chemical reaction increases with increasing temperatures. Also, an increased temperature supports the formation of activated molecules, which may be doubled in number, with 10°C rise in temperature, thereby increasing the reaction rate. This is because the reactant molecules are able to overcome the energy barrier more quickly by attaining more energy. An increase in temperature may also increase the solubility of the protective layers on the metal, thus increasing the susceptibility of the metal to corrosion.

Table 3.2: The mass loss and IE of MS in 1 N HCl containing various concentration of TFL extract with different temperature

Con. of inhibitor (ppm)	Corrosion rate (mmpy)				Inhibition efficiency (%)			
	303 K	313 K	323 K	333 K	303 K	313 K	323 K	333 K
0	485.42	597.01	686.29	814.62	-	-	-	-
10	396.15	474.26	518.90	608.17	18.39	20.56	24.39	25.34
50	345.93	401.73	451.94	485.42	28.73	32.70	34.14	40.41
100	278.98	267.82	278.98	329.19	42.52	55.13	59.34	59.58
500	290.14	200.86	217.60	251.08	40.22	66.35	68.29	69.17
1000	145.07	167.38	172.96	156.22	70.11	71.96	75.39	81.43

3.2.1. Activation energy

Table 3.3: Calculated values of activation energy (E_a) and heat of adsorption (Q_{ads}) of TFL extract on MS in 1N HCl

Con. of inhibitor (ppm)	Activation energy (E_a) (kJ/mole)	Heat of adsorption (Q_{ads}) (kJ/mole)
0	71.83	-
10	71.09	11.86
50	70.34	6.360
100	68.90	0.169
500	66.32	3.364
1000	68.14	8.062

It was seen that the value of activation energy for the blank (71.83 kJ/mol) is higher than in the presence of inhibitors (Table 3.3), which clearly indicates that the adsorption process is chemisorption. Based on the experimentally determined activation energy values, the additive is chemically adsorbed on the MS coupons. Therefore, it is probable that a monolayer strong protective coverage on the entire MS surface was obtained. The calculated Q_{ads} values are ranged from 0.169 to 11.86 kJ/mol. The positive values of Q_{ads} indicate that the adsorption of TFL on the surface of MS is endothermic.

3.2.2. Langmuir adsorption isotherm

By plotting the values of $\text{Log } C/\theta$ versus $\text{Log } C$, linear plots were generated (Fig-3.1) and the investigated data fitted with the Langmuir adsorption isotherm for the adsorption of TFL extract on MS means that there is no interaction between the adsorbed species (i.e; adsorbate and adsorbent). The Langmuir adsorption isotherm is best fit at 333K for MS. The regression coefficients (R^2) were observed from the plots and the reported values is almost close to unity ($R^2 = 0.9906$).

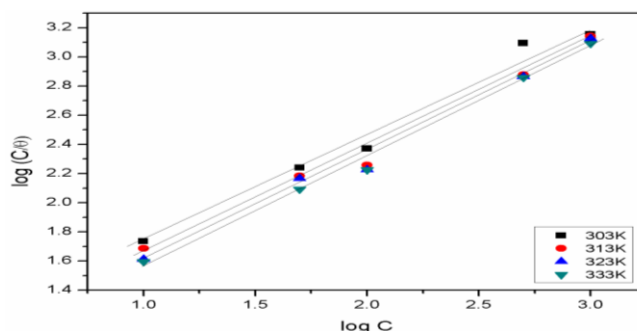


Fig 3.1: Langmuir isotherm for the adsorption of TFL on MS in 1N HCl.

3.2.3. Free energy of adsorption

Table 3.4: Langmuir parameters for the adsorption of TFL extract on MS in 1 N HCl

Adsorption isotherm	Temperature (K)	Slope	K	R ²	ΔG _{ads} (kJ mol ⁻¹)
Langmuir	303	0.7477	0.9643	0.9780	-10.028
	313	0.7232	0.9259	0.9827	-10.253
	323	0.7493	0.8407	0.9840	-10.321
	333	0.7538	0.8052	0.9906	-10.521

The calculated values of ΔG_{ads} are placed in Table 3.4. The negative values of ΔG_{ads} suggested that the adsorption of TFL extract onto metal surface is a spontaneous process and the adsorbed layer is more stable one.

3.3. Electrochemical techniques:

3.3.1. Potentiodynamic polarization measurements

Electrochemical corrosion kinetic parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), anodic and cathodic Tafel slopes (b_a and b_c) and % of IE for the corrosion of MS in 1 N HCl in the absence and presence of distinctive concentrations of the TFL extract are given in table 3.5 and its corresponding polarization curves are shown in Fig 3.2. This investigation revealed that the corrosion current density (I_{corr}) decreased with the addition of the TFL extract and corrosion potential moves to fewer negative values upon addition of the inhibitor. It is found out that I_{corr} value (615 to 114 $\mu A/cm^2$) decreased with increase of TFL extract concentration. It is indicating that the shielding action can ascribe by the N atom by donate the lone pair of electrons to the MS surface and hence reduce the rate of corrosion of the metal. The values of E_{corr} was moved to negative direction, which revealed that the TFL extract has stronger impact on cathodic reaction than on the anodic reaction. The observed result exhibit that the maximum IE was found to be 81.46% in 1 N HCl. The percentage of IE obtained from the polarization method was in good agreement with those obtained from the mass loss testing.

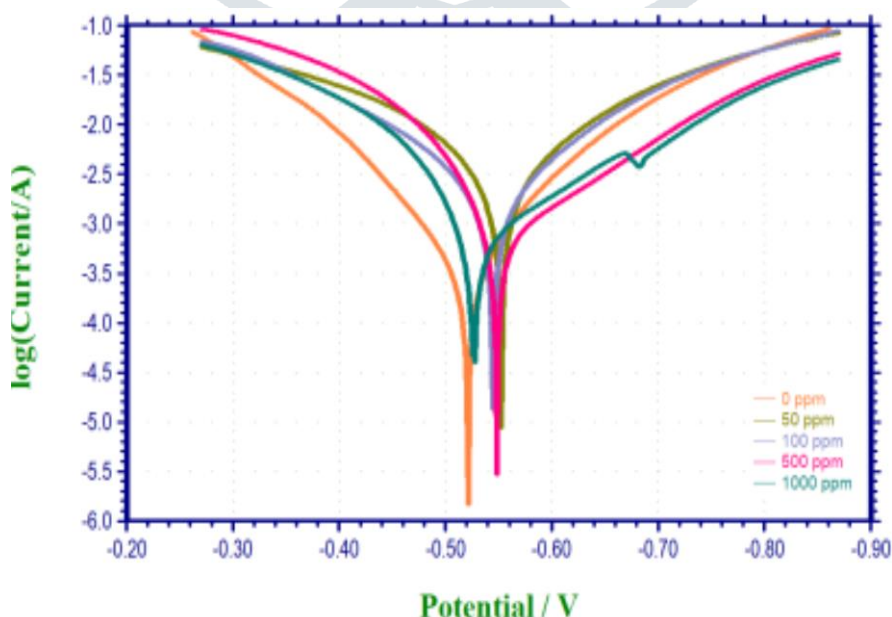


Fig 3.2: Tafel plot of MS in 1 N HCl containing various concentration of TFL extract

Table 3.5: Parameters derived from polarization measurements of MS in 1N HCl containing various concentration of TFL extract

Con. Of inhibitor (ppm)	-E _{corr} (mV)	b _a (mV/decade)	b _c (mV/decade)	I _{corr} (μA/cm ²)	I.E (%)
0	523	86	103	615	-
50	549	160	142	352	42.76
100	544	147	129	234	61.95
500	549	113	146	135	78.04
1000	525	128	148	114	81.46

3.3.2 Electrochemical impedance spectroscopy measurements

The observed value of R_{ct} increased from 0.986 to 6.779 Ω cm² with increase in the TFL extract concentration. The values of C_{dl} tend to be decreased and the percentage of inhibition efficiency achieved maximum of 85.45 % with rise in inhibitor concentration. The values of C_{dl} reduced with a rise in the TFL concentration. These results may have confirmed that the binding between the TFL and the metal ion can prevent the additional dissolution. Also, these results are in good agreement with the earlier results.

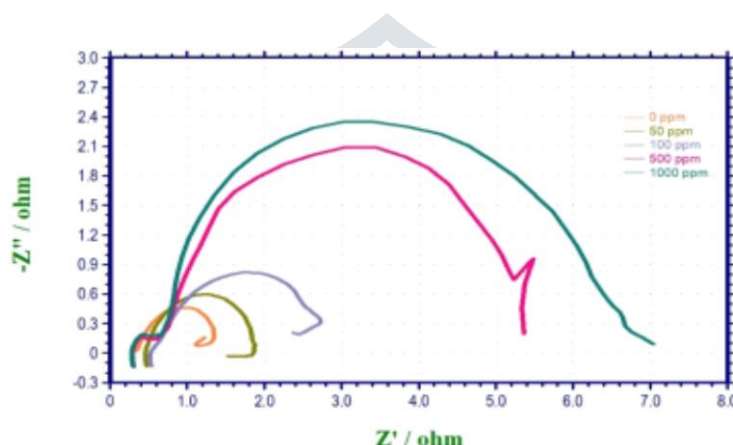


Fig 3.3: Nyquist plot of MS in 1 N HCl containing various concentration of TFL extract

Table 3.6: Parameters derived from impedance measurements of MS in 1 N HCl containing various concentration of TFL extract

Con. of inhibitor (ppm)	R _{ct} (Ω cm ²)	C _{dl} (μF cm ²)	I.E (%)
0	0.986	1.817	-
50	1.393	0.952	29.22
100	2.229	0.391	55.76
500	5.073	0.089	80.56
1000	6.779	0.071	85.45

3.4. Morphological Studies

3.4.1. FT-IR Analysis

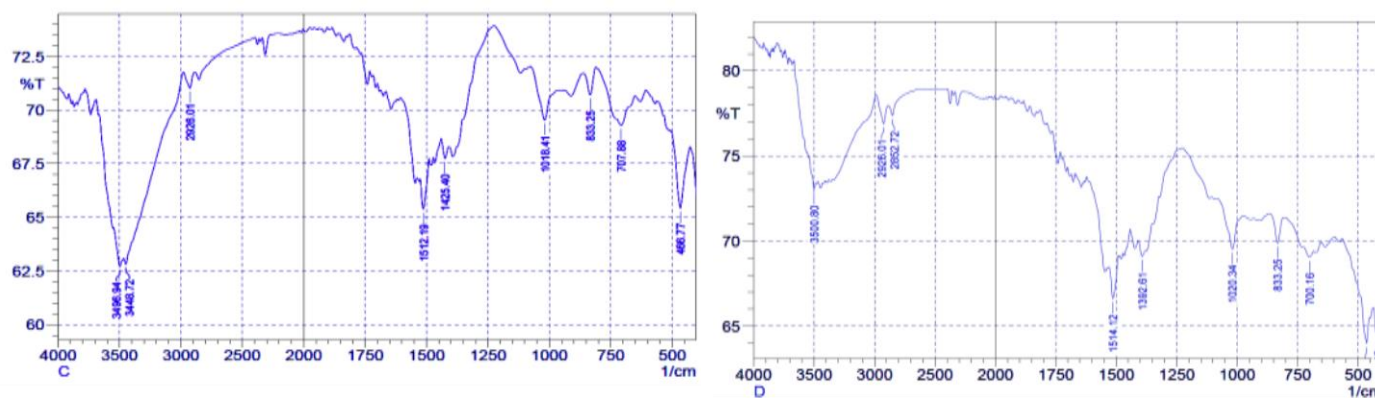


Fig 3.4: FT-IR spectrum of corrosion product in the absence and presence of TFL extract in 1N HCl

Fig-3.4 exhibit the FT-IR spectrum of corrosion product of MS in the absence and presence of TFL inhibitor in 1N HCl. On comparing both these spectra, the prominent peak is shifted from 3496.94 to 3500.80 cm^{-1} for N-H stretching, the frequency at 1512.19 cm^{-1} is attributed to N-O stretching is shifted to 1514.13 cm^{-1} and the C=C bending frequency is shifted from 707.88 cm^{-1} to 700.16 cm^{-1} . Thus, the FT-IR spectra support the fact that the dissolution control of TFL inhibitor on MS in 1N HCl may be due to the adsorption of active molecule in the inhibitor to the metal surface.

3.4.2. EDX Analysis:

EDX technique is used to detect what kind of element present in the corrosion products. Fig 3.5 represent an EDX spectra recorded for MS samples exposed in 1N HCl solution in the presence of optimum concentration of TFL extract. The EDX spectra of inhibited MS surface clearly indicate the presence of nitrogen and oxygen element. This observation may confirm that these hetero atoms present in the inhibitor may be involved in the co-ordination process which suppressed the corrosion process.

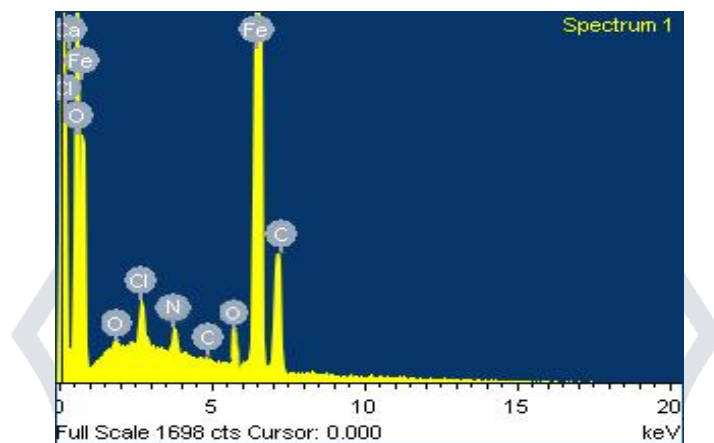


Fig 3.5: EDX spectrum of the corrosion product on MS in the presence of TFL extract in 1 N HCl

3.4.3 Scanning electron microscope (SEM) analysis:

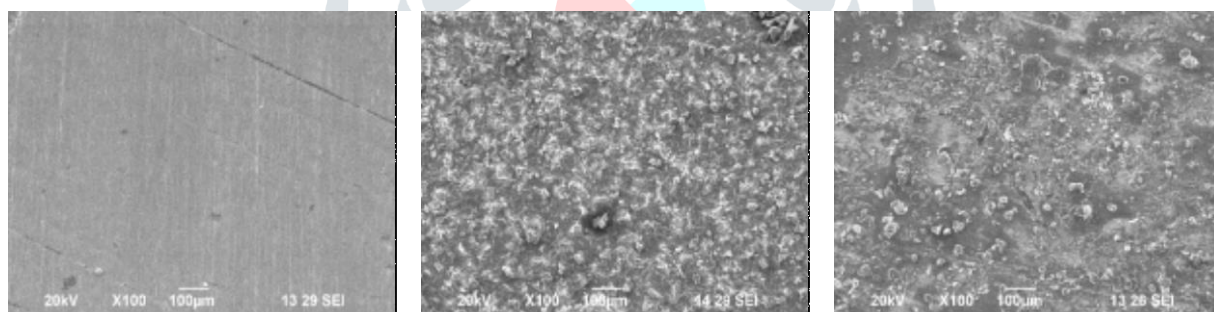


Fig 3.6: SEM micrograph of polished MS, MS in the absence and presence of TFL extract in 1N HCl

Fig 3.6 shows that the SEM micrograph of polished MS, MS in the absence and presence of TFL extract in 1N HCl. Surface morphology of MS were examined using scanning electron microscope, after the corrosion tests. It revealed that, MS corrosion decreased remarkably in the presence of the inhibitor. Inspections of the above images clearly indicate that there is severe damage, clear pits and voids on the surface of MS in the absence of TFL inhibitor. But in the inhibited MS surface, that there is the formation of spongy film and fewer pits and cracks are observed. It confirms that, protective inhibitor film was formed on the MS surface.

IV CONCLUSION

From our present investigation the following conclusions can be drawn.

- The ethanolic extract of *Talinum Fruticosum* leaves extract (TFL) acts as an efficient inhibitor for the corrosion of MS in acid environment.
- Inhibition efficiency increases with increase of inhibitor concentration and also increased with elevated temperature and suggests chemical adsorption.
- The adsorption of different concentrations of the TFL extract on the surface of the MS in 1N HCl follows Langmuir adsorption isotherm.
- The corrosion product over the surface of MS is characterized by FT-IR, SEM and EDX techniques also confirm protective film completely shielded the entire metal surface and reduce the further dissolution of metal against corrosion.

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