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The Effect of *Solanam Xanthocarpum* Leaves Extract on Corrosion Inhibition of Carbon Steel in Acidic Medium

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Abstract: *Solanam xanthocarpum leaves (SXL) extract was used as green corrosion inhibitor for carbon steel in 1N HCl medium using weight loss, gasometric, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) studies. Surface morphology was tested using metallurgical microscope studies. Maximum inhibition efficiency (83.2%) was obtained at the best concentration of 800 ppm of SXL extract using weight loss studies. The effect of temperature and immersion time on the corrosion inhibition of carbon steel in 1N HCl was also studied. Polarization studies revealed that the inhibition action of SXL extract was under mixed control. Adsorption of SXL extract on the surface of carbon steel was found to obey the Langmuir adsorption isotherm. The mechanism of inhibition was confirmed by kinetic and thermodynamic parameters obtained from 303K to 363K temperatures. Surface morphology using metallurgical microscope confirmed the development of a protective film on the carbon steel surface.*

Keywords: *Corrosion inhibitors, Solanam xanthocarpum, Carbon steel corrosion, Langmuir adsorption isotherm, Mixed type inhibitor.*

1. INTRODUCTION

Carbon steel pipelines are used worldwide to transport liquids and gases [1]. This results in the formation of scale and rust. Conventional methods use hydrochloric and sulfuric acids to remove it but this leads to severe corrosion [2]. Therefore, the protection of metals in industries is highly significant [3] especially in acid media. Among the various methods, the use of corrosion inhibitors controls the corrosion of metals in acid medium [4]. Through the decades, organic compounds have proved themselves to be good corrosion inhibitors [5]. They block the active surface sites and reduce the corrosion rate [6]. However, they are not ecofriendly due to the presence of toxic metals [7]. The toxicity of these compounds has lined way to discover the use of non-toxic natural products as inhibitors that are ecofriendly [8]. Till date, many plant extracts have been used as effective corrosion inhibitors for steel in acid media such as *Fenugreek* [9], *Eclipta alba* [10], *Solanum tubersosum* [11], *Nauclea latifolia* [12], *Lawsonia* [13], *Sida rhombifolia* [14], *Datura metel* [15], *Carica papaya* [16], *Mentha pulegium* [17], *Phyllanthus amarus* [18], etc.,

Solanam xanthocarpum belongs to solanaceae family commonly known as a yellow-berried nightshade [19]. It is a prickly, bright green perennial herb that grows on all kinds of soil but does well on dry and hot temperate regions [20]. It is widely distributed in India, Ceylon, Asia, Malaya, Tropical, Auastrana and Polynessia [21]. Different parts of *Solanam xanthocarpum* plays a vital role in Ayurveda medicines as is used to relieve pains, cough, asthma, gonorrhea, fever, sore throat and heart diseases [22]. This paper reports the effect of *Solanam xanthocarpum* leaves (*SXL*) extract as green corrosion inhibitor for carbon steel in 1N HCl medium using weight loss, gasometric, potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) studies. Surface morphology of the carbon steel was tested using metallurgical microscope.

2. EXPERIMENTAL

2.1. Preparation of Materials and Reagent

Carbon steel strips containing 0.18C, 0.30Mn, 0.15Si, 0.02S, 0.02P, the rest Fe in weight percentage were used in this investigation. Coupons cut with 4.5cmx2cmx0.2cm containing a small hole of about 2mm diameter near the upper edge were used for weight loss and gasometric measurements whereas, the specimens were embedded in Teflon leaving a working area of 1.0 cm^2 was used for electrochemical studies. Different grades of emery papers were used to mechanically polish the strips and acetone was used for degreasing. The 1N HCl solution was prepared by the dilution of an analytical reagent grade (AR) 37% HCl with doubly distilled water.

2.2. Preparation of Plant Extracts

The dried *Solanam xanthocarpum* leaves were crushed to make fine powder. The powdered material (10g) was refluxed in 100ml of distilled water for an hour. The refluxed solution was filtered through Whatmann No.1 filter paper and the filtrates were heated on water bath by which the dried compound is obtained, which was free from moisture content [23]. The stock solution (1N HCl) containing various concentrations (200ppm to 800ppm) of *SXL* extract were prepared and used for corrosion studies.

2.3. Weight Loss Measurements

The weight loss method is most widely used simple and more reliable method to measure the corrosion rate [24]. Weight loss measurements were conducted under total immersion of already weighed carbon steel coupons in 100ml of the test solution containing 200, 400, 600 and 800ppm of *Solanam xanthocarpum* leaves (*SXL*) extract and in blank solution for 3 hours. After the specified time, the specimens were washed immediately with double distilled water, dried and weighed. From the measured weight loss data, the corrosion rate (mmpy), inhibition efficiency (IE) and surface coverage (θ) were calculated using the formula;

$$\text{Corrosion rate (mmpy)} = kW/ATD$$

Where k = 8.76×10^4 (constant), W = weight loss in g, A= area in square cm, T= time in hours and D= density in gm / cu.cm

$$\text{Inhibition Efficiency (\%)} = (W_B - W_I / W_B) \times 100$$

Where W_B and W_I are weight loss per unit time in the absence and presence of the extract.

The degree of surface coverage (θ) was calculated from the weight loss measurement results using the following formula;

$$\text{Surface coverage } (\theta) = W_B - W_I / W_B$$

Where W_B is the weight loss in the absence of the *SXL* extract, W_I is the weight loss in the presence of the *SXL* extract. A suitable isotherm was fitted graphically from these data.

2.4. Effect of Immersion Time and Temperature

To examine the efficiency of inhibitor at various immersion time from 3 hours to 24 hours at 30°C and at four different temperatures (30, 50, 70 and 90°C), the weight loss study was carried out in the blank solution as well as in the finest concentration (800ppm) of *SXL* extract. From the weight loss, the corrosion rate and inhibition efficiency were calculated.

2.5. Gasometric Method

This method was carried out as described earlier [25]. The result obtained in this method is more accurate than conventional weight loss method, provided the evolved hydrogen gas does not react with inhibitor. Moreover, the hydrogen penetration into the metal is small compared to the total volume of hydrogen gas. Volume measurements were made for a period of two hours in all the cases. From the volume of hydrogen gas liberated, the inhibition efficiency was calculated using the formula;

$$\text{Inhibition Efficiency (\%)} = (V_0 - V_1 / V_1) \times 100$$

Where, V_0 and V_1 is the volume of hydrogen gas evolved in the absence and presence of *SXL* extract.

2.6. Potentiodynamic Polarization Studies

Potentiodynamic polarization studies were carried out using three electrode electrochemical cell in which carbon steel with the surface area of 1.0cm^2 has been used working electrode (WE), a platinum foil with the surface area of 1.0cm^2 as counter electrode and saturated calomel electrode (SCE) as reference electrode, provided with a Luggin capillary connected to a electrochemical analyzer (BioLogic, VSP, France). The WE was placed vertically facing the counter electrode, and a reference electrode was placed near to the WE. The WE was immersed in 1N HCl solution for 30 min until a steady-state open circuit potential (OCP) was obtained. The HCl solution was degassed with ultrapure nitrogen bubbling for 30 min to avoid any reactions with dissolved oxygen. Tafel polarization scans were carried out by changing the electrode potential automatically from -700mV to -200 mV versus SCE at the scan rate of 1 mV/s.

From the Tafel polarization curves, corrosion potential and corrosion current were calculated. The inhibition efficiency was calculated using the formula [26];

$$IE\ (\%) = (I_{Corr} - I^*_{Corr} / I_{Corr} \times 100$$

Where I_{corr} and I^*_{corr} are corrosion current in the absence and presence of *SXL* extract.

2.7. AC-Impedance Measurements

Electrochemical impedance studies (EIS) provides more information on both the resistive and capacitive behavior at metal/solution interface. Therefore, this method has been widely used in investigating corrosion inhibition processes. This technique was carried out to evaluate the corrosion behavior of carbon steel in 1N HCl solution in the absence and presence of the best concentration (800ppm) of the extract by using Electro-chemical analyzer (BioLogic, VSP, France) as described earlier. Experiments were carried out at the open circuit potential for the frequency range of 100kHz to 1mHz. A plot of Z' Vs Z" were made. From the plots, the charge transfer resistance (R_t) and the double layer capacitance (C_{dl}) were calculated using the following equation [27];

$$C_{dl} = 1 / 2\pi f_{max} R_t$$

Where R_t is charge transfer resistance and C_{dl} is double layer capacitance. The experiments were carried out in the absence and presence of optimum concentration (800ppm) of the *SXL* extract. The percentage of inhibition efficiency was calculated using the equation [28];

$$IE\ (\%) = (R^*_t - R_t / R^*_t) \times 100$$

Where R_t* and R_t are the charge transfer resistance in the presence and absence of *SXL* extract.

2.8. Surface Morphological Studies

This study was carried out by immersing the carbon steel in 1N HCl in the absence and presence of the finest concentration of *SXL* extract for 2 hours at 30°C. After 2 hours, the specimens were taken out, dried and kept in desiccator. The protective film formed on the surface of carbon steel was confirmed by metallurgical microscope with the magnification of 1000x.

3. RESULTS AND DISCUSSION

3.1. Weight Loss Method

The corrosion inhibition of carbon steel in 1N HCl in the absence and presence of various concentrations of *SXL* extract ranging from 200 to 800ppm was determined after 3 hours of immersion period at 303K. Corrosion rates, inhibition efficiencies and surface coverage (θ) are calculated from loss in weight. The results are given in Table 1. The variation of inhibition efficiency with various concentrations of *SXL* extract is shown in Fig. 1.

Table1. *Corrosion parameters obtained from weight loss measurements for carbon steel in 1N HCl containing various concentrations of SXL extract*

Concentration of *SXL* extract (ppm)	Initial weight (g)	Final weight (g)	Weight Loss (gm)	Corrosion Rate (mmpy)	Inhibition Efficiency (%)	Surface Coverage (θ)
Blank	3.7096	3.6601	0.0495	20.4581	----	----
200	3.8311	3.8184	0.0127	5.2489	74.3	0.743
400	3.7613	3.7517	0.0096	3.9676	80.6	0.806
600	3.7914	3.7826	0.0088	3.6370	82.2	0.822
800	3.7835	3.7752	0.0083	3.4304	83.2	0.832

The analysis of these results show clearly that the corrosion rate decreases with increasing *SXL* extract concentration and reaching a maximum inhibition of 83.2% at a concentration of 800ppm. This behavior is due to the adsorption of natural compounds on the surface of the metal, as the inhibitor concentration increases [29]. It concludes that *SXL* extract is a good corrosion inhibitor for carbon steel in 1N HCl solution.

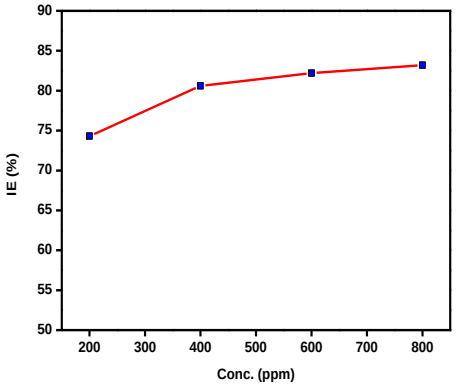

Fig1. *Influence of inhibition efficiency with various concentrations of SXL extract on carbon steel in 1N HCl solution*

3.2. Effect of Immersion Time

The effect of immersion time from 3 to 24 hours on the corrosion behavior of carbon steel in 1N HCl solution containing inhibitor at the best concentration of 800ppm was studied using weight loss measurement at 30°C and the calculated inhibition efficiency percentage were given in the Table 2. The plot of inhibition efficiency percentage against immersion time has been shown in Fig. 2. From the figure we infer that inhibition efficiency decreases with increasing immersion time. Higher inhibition efficiency of 83.2% at 3 h is due to the strong adsorption of constituents present in the *SXL* extract on the carbon steel surface, which forms a more protective layer on the carbon steel and hydrochloric acid solution interface. On the other hand the decrease in inhibition efficiency is due to the slow desorption of the adsorbed constituents with increasing immersion time.

Table2. *Effect of immersion time on percentage inhibition efficiency of carbon steel in1N HCl at 30℃ in the presence of the best concentration (800ppm) of SXL extract*

System	Inhibition Efficiency (%)							
	Time (h)							
	3	**6**	**9**	**12**	**15**	**18**	**21**	24
800 ppm of SXL extract	83.2	83.0	82.7	82.4	82.1	81.9	81.7	81.4

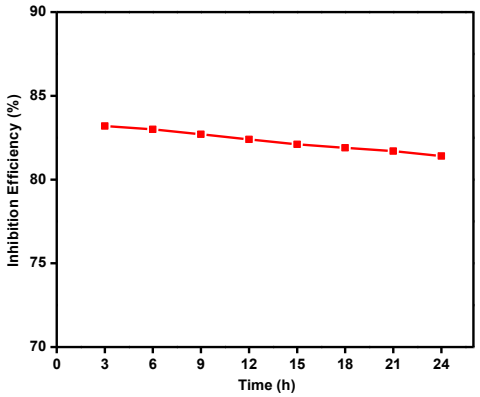

Fig2. *Effect of immersion time on percentage inhibition efficiency of carbon steel in 1N HCl at 30°C in presence of the best concentration (800ppm) of SXL extract*

3.3. Effect of Temperature

The influence of temperature on the corrosion inhibition action of *SXL* extract was determined by weight loss method at the best concentration of 800ppm at different temperatures (30, 50, 70 and 90°C) for a fixed immersion time of 3 hours is shown in Fig. 3. The data of corrosion rates and their corresponding efficiency are given in Table 3. From the Table 3, it is clear that the corrosion rate of

carbon steel increased with temperature both in the absence and presence of *SXL* extract and the inhibition efficiency was found to decreased from 83.2% - 75.6%. This shows that the adsorption of the extract on the carbon steel may be due to physical adsorption. However, the inhibitor could be effectively used at 30°C and the maximum efficiency being 83.2%.

Table3. *Corrosion of carbon steel in the absence and presence of the best concentration of SXL extract in 1N HCl at different temperatures obtained by weight loss method*

System	Temperature (°C)	Corrosion Rate (mmpy)	Inhibition Efficiency (%)
Blank	30	122.46	---
	50	152.92	---
	70	189.46	---
	90	241.60	---
800 ppm of SXL extract	30	20.54	83.2
	50	27.27	82.2
	70	39.11	79.4
	90	59.10	75.6

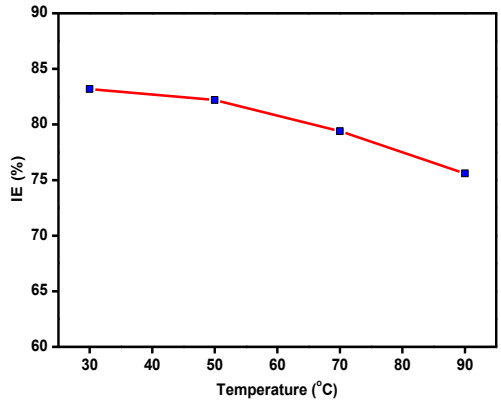

Fig3. *Effect of temperature on the corrosion inhibition efficiency of carbon steel in1N HCl in presence of the best concentration (800ppm) of SXL extract*

3.4. Gasometric Method

The results obtained in the gasometric method are shown in Table. 4. From the table it is observed that the hydrogen penetration into the metal decreased as the concentration of the inhibitor increased from 200 to 800ppm and showed maximum inhibition efficiency of 83.7% at 800ppm concentration of the extract. These values support the *SXL* extract had the tendency to inhibit corrosion on carbon steel in acid medium.

Table4. *Inhibition efficiency obtained from gasometric measurements for carbon steel in 1N HCl containing various concentrations of SXL extract at 30°C*

Conc. of *SXL* Extract (% in v/v)	Volume of Hydrogen Gas evolved (mL)	Inhibition Efficiency (%)
Blank	7.4	--
200	1.8	74.6
400	1.4	80.8
600	1.3	82.6
800	1.2	**83.7**

3.5. Potentiodynamic Polarization Studies

Fig. 4 shows the anodic and cathodic polarization curves of carbon steel in 1N HCl solution without & with best concentration (800ppm) of *SXL* extract. And the electrochemical parameters including corrosion potential (E_{corr}), corrosion current dencity (I_{corr}), cathodic and anodic Tafel slopes (b_a & b_c) and inhibition efficiency are listed in the Table 5. It can be seen from the Fig. 4 and Table 5 that in the presence of *SXL* extract, Tafel slope decreased to more negative potential and increased to more positive potentials related to the blank curve. Both the anodic and cathodic curves show lower current density (I_{corr}) and the corrosion potential (E_{corr}) was not shifted significantly in the presence of best concentration (800ppm) of *SXL* extract. Therefore, it could be concluded that the anodic iron dissolution and cathodic hydrogen evolution reaction were inhibited by best concentration (800ppm)

of *SXL* extract through merely blocking the reaction sites of carbon steel surface without affecting the reaction mechanism [30]. This suggests that the inhibitor is mixed type inhibitor [31].

Table5. *Potentiodynamic polarization parameters for carbon steel in 1N HCl in the absence and presence of the best concentration of SXL extract*

Conc. of *SXL* Extract (ppm)	E_{corr} (V vs SCE)	I_{corr} (mA/cm^2)	Tafel Slope(mV/decade)		Inhibition Efficiency (%)
			b_a	b_c	
Blank	- 0.442	3.785	204.6	224.8	__
800 ppm	- 0.453	0.592	200.6	222.4	84.4

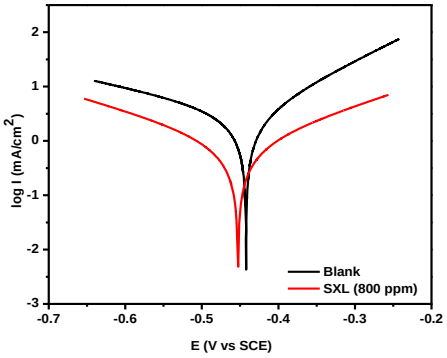

Fig4. *Potentiodynamic polarization curves for carbon steel in 1N HCl solution in the absence and presence of the best concentration of SXL extract*

3.6. AC- Impedance Measurement

Fig. 5 shows the impedance diagram for carbon steel in 1N HCl in the absence and presence of best concentration (800ppm) of *SXL* extract. As noted from Fig. 5, the obtained impedance diagrams are perfect semicircles and this was attributed to charge transfer reaction mainly control the corrosion of carbon steel in 1N HCl. The impedance parameters such as charge transfer resistance (R_t) and double layer capacitance (C_{dl}) were calculated from Nyquist plot and the calculated inhibition efficiency are listed in Table 6.

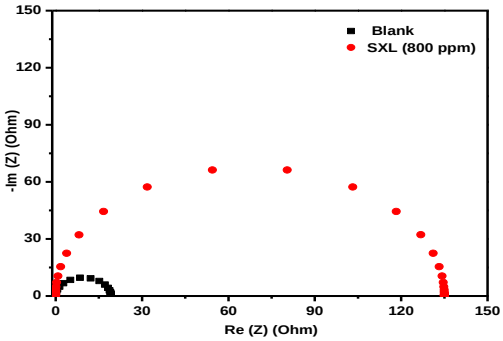

Fig5. *Impedance diagrams for carbon steel in 1N HCl solution in the absence and presence of the best concentration of SXL extract*

Table6. *Impedance parameters for the corrosion of carbon steel in 1N HCl in the absence and presence of the best concentration of SXL extract at 30^{0}C*

Conc. Of *SXL* Extract (ppm)	R_t (Ω cm^2)	C_{dl} (µF/cm^2)	Inhibition Efficiency (%)
Blank	20.65	116.9	-
800 ppm	136.04	11.70	84.9

It can be seen that the increase in charge transfer resistance (R_t) indicating that the adsorbed extract forms a protective film which become barrier to hinder the mass and charge transfer processes. Meanwhile, the decrease in C_{dl} values due to increase in thickness of electrical double layer, suggests that the adsorption of inhibitor molecules take place at metal-solution interface [32]. Therefore, the protection efficiency increases. The excellent concentration of 800ppm of the *SXL* extract shows the maximum R_t value of 136.04 Ω cm^2 and the minimum C_{dl} value of 11.70µF/cm^2 compared to blank solution was obtained which gave the maximum inhibition efficiency of 84.9%. This result has good concurrence with the results obtained from non-electrochemical methods.

3.7. Interpretation of Thermodynamic Data and Mechanism of Corrosion Inhibition

The corrosion inhibition mechanism of *SXL* extract (at 800ppm) has been interpreted from the kinetic and thermodynamic data given in Table 7. The activation energy at different temperatures was calculated by plotting $\log CR$ Vs $1/T$ in the Arrhenius type plot which shows a straight line shown in Fig.6 reveals the effect of temperature on carbon steel immersed in 1N HCl solution at the optimum concentration (800ppm) as well as in the absence of the *SXL* extract. The activation energy, E_a was found to be 9.04kJ mol^{-1} for blank and increased to 11.53 kJ mol^{-1} in presence of *SXL* extract in 1N HCl solution suggesting that the adsorbed extract form a physical barrier to charge and mass transfer leading to reduction in corrosion rate. The higher value of E_a in presence of the *SXL* extract is due to physical adsorption [33].

Table7. *Calculated values of activation energy (E_a), free energy of adsorption ($\Delta G°$), enthalpy of adsorption (ΔH), and entropy of adsorption (ΔS) for carbon steel in the absence and presence of the best concentration of SXL extract in 1N HCl solution*

System	Temp. (K)	E_a (kJ mol^{-1})	$\Delta G°$ (kJmol^{-1})	ΔH (kJmol^{-1})	ΔS (kJmol^{-1})
Blank	303	---	---	---	---
	323	9.04	---	6.35	----
	343	9.87	---	7.02	---
	363	12.58	---	9.56	---
800 ppm of SXL extract	303	---	-8.91	---	---
	323	11.53	-9.45	8.84	0.056
	343	16.61	-10.09	13.76	0.069
	363	21.38	-10.67	18.36	0.081

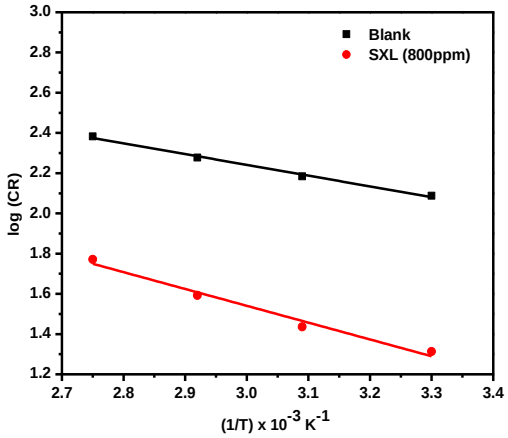

Fig6. *Arrhenius plots for carbon steel immersed in 1N HCl solution in the absence and presence of the best concentration (800ppm) of SXL extract*

The negative sign of change in free energy of adsorption ($\Delta G°$) suggests the spontaneous adsorption of *SXL* extract on carbon steel surface. In this study, the $\Delta G°$ values are in the range of -8.9kJ mol^{-1} to -10.67kJ mol^{-1}. It is clear that the mode of inhibition is due to physisorption as the values of free energy of adsorption are less than -20 kJ mol^{-1} [34]. The positive values of ΔH indicate that the dissolution of carbon steel is an endothermic process and the values of ΔS indicate that an increase in disordering from reactant to the activated complex [22] change in entropy was found to be greater than 0.056 indicates the reaction is irreversible [32].

From the literature survey, it was found that caffeic acid (Fig. 7a) and oleanolic acid (Fig. 7b) are the principal alkaloids present in the leaves extract of *Solanam xanthocarpum* [35]. These alkaloids contain hydroxyl groups adsorb over carbon steel surface and forms a compact protective thin layer reduces the corrosion rate. The degree of protection depends on the surface coverage (θ) by the adsorbed molecules. The values of surface coverage for various concentration of *SXL* extract are given in Table 1. The use of adsorption isotherm provides useful insight into the corrosion inhibition mechanism. A plot of C/ θ Vs C provide a straight line with unit slope suggests that the adsorption of various concentration of *SXL* extract on the surface of carbon steel in 1N HCl follows Langmuir adsorption isotherm (Fig. 8).

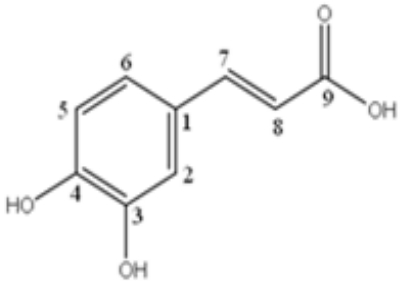

Fig7a. *Structure of caffeic acid (3,4-Dihydroxy cinnamic acid)*

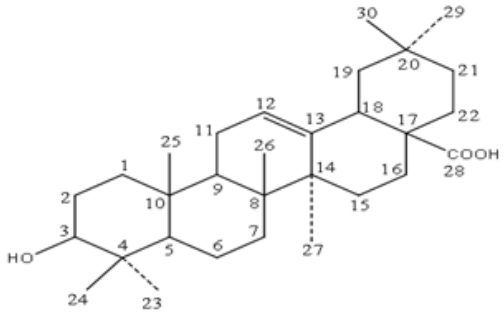

Fig7b. *Structure of oleanolic acid (3β-Hydroxyolean-12-en-28-oic acid)*

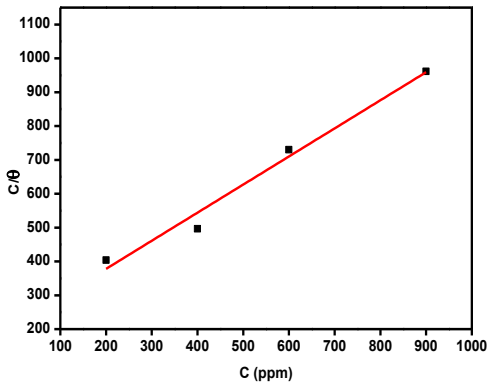

Fig8. *Langmuir adsorption isotherm plot for the adsorption of various concentrations of SXL extract on the surface of carbon steel in 1N HCl solution*

3.8. Surface Morphological Studies

Surface morphological study was carried out by immersing the carbon steel specimen in 1N HCl solution for 2 hours at 30°C in the absence and presence of the optimum concentration (800ppm) of *SXL* extract. After 2 hours, specimens were taken out dried and kept in a desiccator. Their surface was examined by metallurgical microscope with the magnification of 1000x are shown in Fig. 9(a&b). By comparing, the surface of carbon steel in the presence of inhibitor appears smoother than the absence of inhibitor due to the formation of protective film.

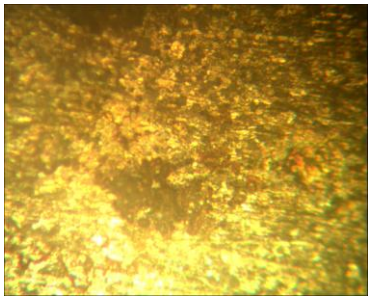

Fig9(a). *Metallurgical microscope image of carbon steel immersed in 1N HCl solution (blank)*

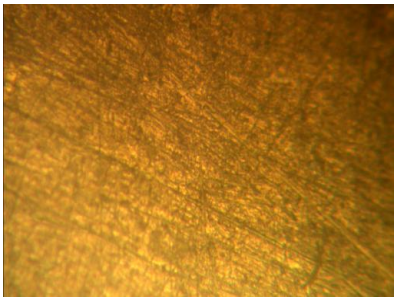

Fig9(b). *Metallurgical microscope image of carbonsteel immersed in 1N HCl solution containing 800ppm of SXL extract*

4. CONCLUSIONS

From the above studies, it can be concluded that the *SXL* extract shows good performance as corrosion inhibitor in 1N HCl solution and inhibit the corrosion of carbon steel in 1N HCl at the best concentration of 800ppm. The *SXL* extract showed the maximum efficiency of 83.2 % at 3 hours of immersion time and found to slightly decrease from 83.2% to 81.4% as the immersion time increased from 3 to 24 hours. Potentiodynamic polarization measurements showed that *SXL* extract acts as mixed type inhibitor. EIS studies also indicate that the *SXL* extract increases the charge transfer resistance and showed the inhibitive action of the inhibitor depends on adsorption of the molecules on the metal surface. The *SXL* extract inhibit the corrosion of carbon steel by getting adsorbed on the metal surface obeying Langmuir adsorption isotherm. The metallurgical microscope studies showed that the surface of carbon steel in 1N HCl is smoother in the presence of *SXL* extract than the uninhibited surface due to the formation of protective film on the inhibitor surface. The results obtained by weight loss, potentiodynamic polarization and electrochemical impedance spectroscopy studies are in reasonably good agreement.

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