Evaluation Of The Protective Nature Of *Schleichera oleosa* Bark Extract As Green Inhibitor Of Mild Steel Corrosion In Sulphuric Acid

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Abstract: The Adsorption and inhibition effect of Schleichera Oleosa Bark (SOB) on mild steel corrosion in 0.5 M H_2SO_4 at 303 K and 313 K were studied using weight loss method. The results obtained showed that SOB act as an effective corrosion inhibitor and inhibition efficiency was found to increase with increase in SOB concentration but decrease with temperature suggesting physical adsorption mechanism. Arrhenious low leads to estimate the activation energy of the corrosion process and the values of heat of adsorption indicating the adsorption of the inhibitor on surface of mild steel was exothermic. Langmuir, Temkin, Flory- Huggins and Frumkin adsorption isotherms were found to confirm the adsorption characteristics of the inhibitor on metal surface.

***Index Terms*— Adsorption isotherms, corrosion, inhibitor, mild steel, *Schleichera Oleosa* , sulphuric acid.**

I. INTRODUCTION

Corrosion is an unattractive phenomenon, it cause disastrous damage to metals and alloys structure causing economic consequences and environmental pollution. A number of organic compounds are known to be applicable as corrosion inhibitors for mild steel in acid environment. Despite of significant inhibition efficiency, most of these substances are not only expensive but also toxic non biodegradable thus causing pollution problems. The implementation of Green Chemistry should involve reconsideration of the economic viability of the process by taking into account the environmental hazards associated with the procedure. Hence, these deficits have encouraged for their substitution. Chemistry in recent millennium has accepted the concept of green chemistry and adopted it wide to satisfy the challenge of protecting the human health and surroundings. The utilization of plant extract as inhibitor for metal corrosion in aggressive environments is reported [1, 2]. Green corrosion inhibitors of plant extracts having complex molecular structures and different chemical, biological and physical properties. However, little has been accounted the use of bark extract of *Schleichera Oleosa* (SOB) belonging to the sapindaceae family as corrosion inhibitor for mild steel corrosion in H_2SO_4 medium.

The present investigations intended to study the potential of SOB extract as an inhibitor on the mild steel corrosion in 0.5 M H_2SO_4 using weight loss method. The effect of temperature on the dissolution of metal in acid, containing the inhibitor was also noticed. Some thermodynamic parameters were computed and tried to fit the adsorption data to Langmuir, Temkin, Frumkin and Flory-Huggins adsorption isotherms.

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II. EXPERIMENTAL METHODS

The fresh bark of *Schleichera Oleosa* were collected, washed, shade dried and powdered. 25 g of the powder was weighed and transferred to a 1000 ml round bottomed flask, heated with 500 ml of 0.5 M H_2SO_4. This mixture was refluxed for three hours and kept overnight and filtered. The following day it was filtered and the filtrate was made up to 500 ml using 0.5 M H_2SO_4. From this 5% stock solution, inhibitor test concentrations of 0.1, 0.2, 0.3, 0.4, 0.5 v/v % were prepared by diluting with 0.5 M H_2SO_4.

Mild steel sheets of composition in (wt %) C: 0.091, Mn: 0.195, Si: 0.016, P: 0.020, S: 0.013, Cr: 0.027, Mo: 0.020, Ni: 0.018, Fe: 99.6 cut into rectangular coupons of size 5 cm x1 cm provided with hole to enable suspension in test solutions were used for the study. These MS pieces were mechanically polished to remove any rest on it. The metal pieces were then degreased with acetone, washed in double distilled water and polished with emery paper, cleaned, dried and stored in desiccators.

A known weight of mild steel coupon was immersed in 100 ml of the test solution in an open beaker. The beaker was transferred into a water bath maintained at 303K. The gravimetric analysis was carried out at different immersion periods (0.5 h, 1h, 3h, 6h, 12h, and 24h), each sample was withdrawn from the test solution washed, rinsed in acetone, dried in air and reweighed. The experiment was repeated at 313 K in 0.5 h immersion period. After measuring the weight loss, surface coverage (θ), percentage inhibition efficiency

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(IE %) and corrosion rate (CR) were calculating using the following formula

$$\theta = \left({IE\%}/{100} \right) \quad (1)$$

$$IE(\%) = \left(\frac{W_0 - W}{W_0} \right) x\, 100 \quad (2)$$

$$CR(mpy) = 534\, W/DAT \quad (3)$$

where, w is the weight loss in g, D is the density of mild steel in gm / cm^2 (7.9 gm / cm^2), A is the area of the specimen in cm^2 , T is the exposure time in hours , W_0 is the weight loss without inhibitor and W is the weight loss with inhibitor respectively.

III RESULT AND DISCUSSION

A. *Weight loss measurements*

Because of the simplicity and high reliability, weight loss methods were used for monitoring corrosion rate of metal and inhibition efficiency of extract. The immersion time is an important parameter, which can be used to evaluate the stability of inhibitor behavior [3].The variation of weight loss of metal with time for the corrosion of mild steel in 0.5 M H_2SO_4 in the absence and presence of various concentration of SOB extract at 303 K was shown in Fig 1 .

The slope of each line represented the corrosion rate of mild steel at the specified conditions. The linear increase in the weight loss with time in the absence and presence of SOB indicated that insoluble surface films do not form on the electrode surface during metal corrosion in H_2SO_4 solution. This means that SOB was first adsorbed on the electrode surface impeding corrosion either by merely blocking the reaction sites or by altering the mechanism of anodic and cathodic processes [4]. It followed from the data of Fig 1 that the weight loss decreased, and therefore the corrosion inhibition strengthened, with increase in inhibitor concentration. This trend may result from the fact that adsorption and surface coverage increased with SOB concentration and thus the surface was efficiently separated from the medium [5].

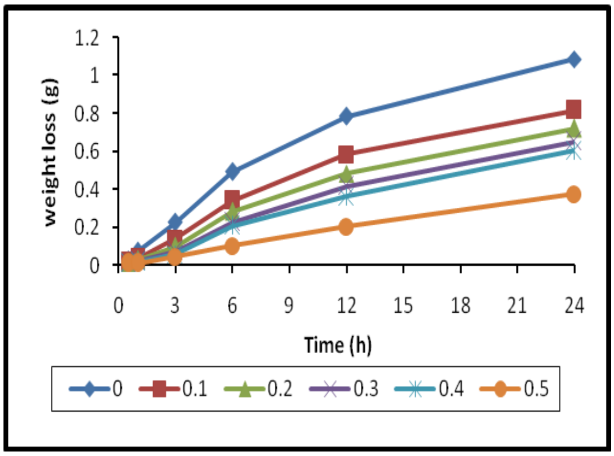

Fig 1: Variation of weight loss with different concentrations of SOB extract for MS corrosion in 0.5 M H_2SO_4 at 303 K

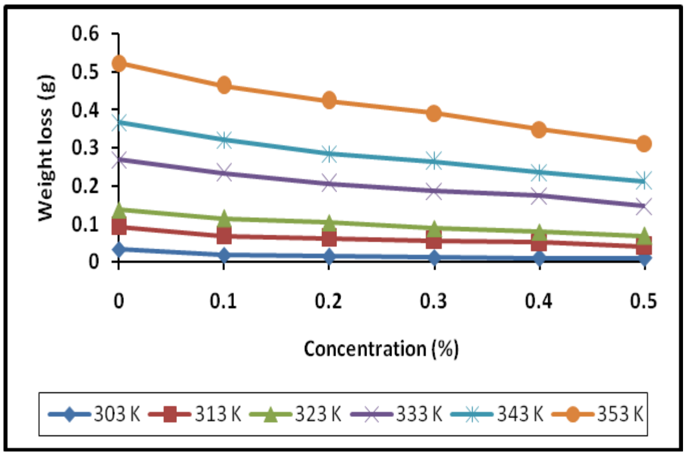

Fig 2: Deviation of weight loss with different concentrations of SOB extract at varying temperature

Fig 2 revealed that the weight loss decreased as the concentration of SOB increases and also that it increased with the temperature. At higher temperatures of (303 to 353 K), the weight loss of mild steel was found to increase with increasing temperature, indicating the desorption of adsorbed protective film at higher temperature.

The corrosion rate of mild steel in the absence and presence of SOB extract and inhibition efficiencies of various concentrations of the extract were shown in Fig 3 and 4 respectively. The result obtained showed that the rate of corrosion of mild steel decreased with increase in the concentration of SOB extract but increased with increase in temperature.

The inhibition efficiency of the extract decreased with increase in temperature, indicating that the adsorption of SOB extract on mild steel surface is physical adsorption as reported by other researchers [6-8]. In this work, the maximum inhibition efficiency of SOB extract was noticed at 303 K, and the increase in temperature resulted in a drastic decrease in surface coverage [9]. Literature survey of the phytochemical analysis of barks of *Schleichera oleosa* opened the presence of various constituents as lupeol, lupeol acetate, betulin, betulinic acid, beta-sitosterol, scopoletin, taraxerone, tricadenic acid A and tannin [10-12].

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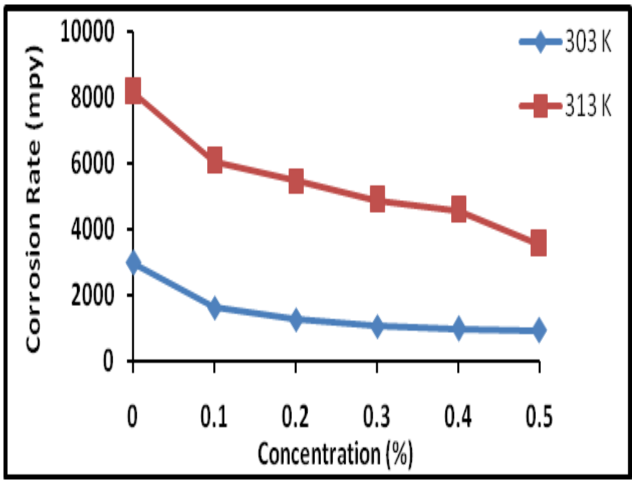

Fig 3: Variation of corrosion rate with different concentrations of SOB extract at 303 K and 313 K.

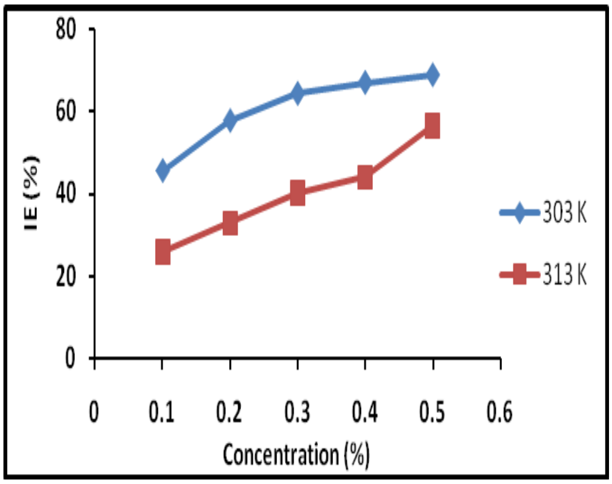

Figure 4: Variation of Inhibition efficiency with different concentration of SOB extract at 303 K and 313 K

The inhibiting effect of the extract can be attributed to the presence of phytochemical constituents present in this extract. This extract contains oxygen atom and aromatic ring that are the center of adsorption.

Effect of temperature.

The effect of temperature on the rate of corrosion of mild steel in H_2SO_4 containing various concentrations of SOB extract was investigated using Arrhenius equation as given in (4) [13,14].

$$CR = A\ exp(-E_a | RT) \quad (4)$$

Where A is the frequency factor, E_a is the activation energy of corrosion process, R is molar gas constant and T is the temperature. Let the corrosion rates of mild steel at 303 K (T_1) and 313 K (T_2) denoted as CR_1 and CR_2. Equation (4) becomes

$$log(CR_2 | CR_1) = \frac{Ea}{2.303R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \quad (5)$$

The values of activation energy were listed in Table I. The data showed that the activation energy (E_a) of the corrosion in mild steel in acid solution in the presence of extract are higher than those in free acid solution indicating that extract exhibit low inhibition efficiency at elevated temperatures. The E_a values ranged from 47.12 to 70.01 $KJmol^{-1}$ and were lower than the threshold value of 80 $KJmol^{-1}$ required for chemical adsorption. This is an indication of spontaneous adsorption of the inhibitor molecules on mild steel surface and is attributed to physical adsorption [8, 13, 15].

Thermodynamic/Adsorption Parameters

The heat of adsorption Q_{ads} of SOB extract on the surface of mild steel was calculated using (6) [13,14,16,17].

$$Q_{ads} = 2.303R\{log(\theta_2 | 1-\theta_2) - log(\theta_1 | 1-\theta_1)\} \times \frac{T_1 \times T_2}{T_2 - T_1} \quad (6)$$

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. Calculated values of activation energy and are shown in Table I. The values of heat of adsorption ranged from –42.10 to –80.89 $KJmol^{-1}$(Table I), indicating that the adsorption of SOB extract on mild steel surface is exothermic [6,17].

Table I: Thermodynamic parameters for adsorption of *Schleichera Oleosa* extract on mild steel surface

Concentration (v/v %)	Activation energy ($KJmol^{-1}$)	Heat of adsorption ($KJmol^{-1}$)
0	47.12	
0.1	70.01	-68.82
0.2	68.53	-80.89
0.3	66.64	-78.02
0.4	64.5	-74.78
0.5	65.78	-42.10

B. Evaluation of different adsorption Isotherms

Data obtained for the degree of surface coverage were used for the evaluation of various adsorption isotherms such as Langmuir, Frumkin, Temkin and Flory-Huggins isotherms.

Langmuir Adsorption Isotherm

Langmuir adsorption isotherm is expressed according to (7) [18, 19].

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad (7)$$

where C is the concentration of the inhibitor, K is the adsorption equilibrium constant and θ is degree of surface coverage of the inhibitor. Taking logarithm of both sides of (7) yields (8).

$$log\left[\frac{c}{\theta}\right] = \log c - \log K_{ads} \quad (8)$$

Plotting log C/ θ against log C gave a linear relationship as shown in Fig 5. The parameters of Langmuir isotherm are presented in Table 2. The R^2 values of 0.98 and 0.97 indicate strong adherence to Langmuir adsorption isotherm [20]. The application of Langmuir isotherm to the adsorption of extract of SOB on surface of mild steel indicated that there is no interaction between the adsorbate and adsorbent [21].

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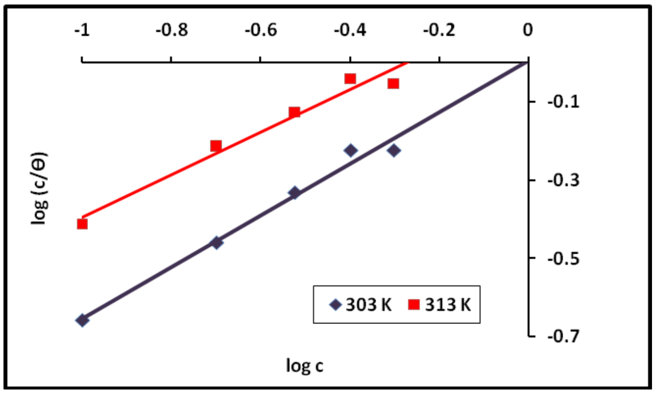

Fig 5: Langmuir adsorption isotherm of SOB extract on mild steel surface

Temkin Adsorption Isotherm

For Temkin adsorption isotherm, the degree of surface coverage (θ) is related to inhibitor concentration (C) according to (9) [22, 23].

$$\exp(-2a\theta) = K_{ads}C \qquad (9)$$

where K_{ads} is the adsorption equilibrium constant and a, is the attractive parameter.

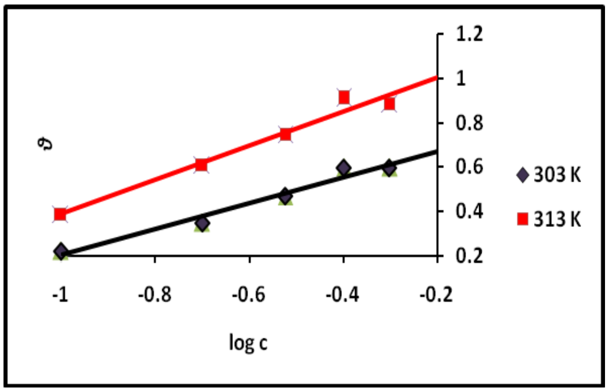

Fig 6: Temkin isotherm for adsorption of SOB on the surface of mild steel at different temperatures.

Rearranging and taking logarithm of both sides of Equation (9) gives (10).

$$\theta = \frac{-2.303 \log R}{2a} - \frac{2.303 \log C}{2a} \qquad \textbf{(10)}$$

Plots of θ against log C, as presented in Fig 6, gave linear relationship, which showed that adsorption data fitted with Temkin adsorption isotherm. Adsorption parameters obtained from the isotherms were recorded in Table II. The values of attractive parameter (a) were negative in all cases, indicating that repulsion exists in the adsorption layer [24].

Flory-Huggins Adsorption Isotherm

Flory-Huggins adsorption isotherm can be expressed according to (11) [25].

$$\log\left(\frac{\theta}{c}\right) = \log K_{ads} + x \log(1-\theta) \qquad \textbf{(11)}$$

where x is the size parameter and is a measure of the number of adsorbed water molecules substituted by a given inhibitor molecule. The plots of $\log\left(\frac{\theta}{c}\right)$ against log (1- θ) were shown in Fig 7 which gave a linear relationship showing that was fitted to Flory-Huggins isotherm. The values of the size parameter x were positive (Table II), point out that the adsorbed species of SOB extract was bulky since it could displace more than one water molecule from the mild steel surface.

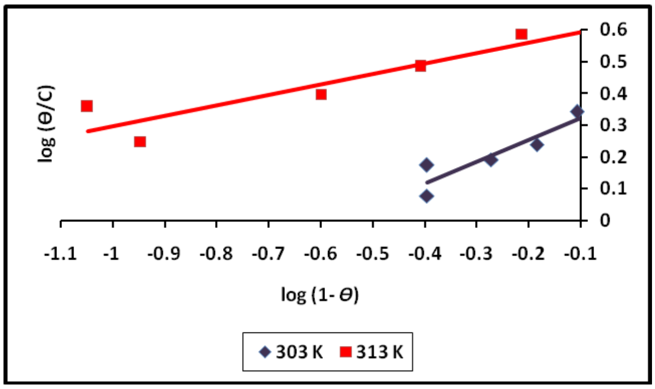

Fig 7: Florry Huggins isotherm for adsorption of SOB extract on mild steel surface.

Frumkin Adsorption Isotherm

Frumkin adsorption isotherm is given by (12) [13, 23].

$$\log\left\{[C] \times \left(\frac{\theta}{1-\theta}\right)\right\} = 2.303 \log K_{ads} + 2\alpha\theta \qquad (12)$$

where K_{ads} is the adsorption-desorption constant and α is the lateral interaction term describing the interaction in adsorbed layer, taking into account of the attraction ($\alpha > 0$) or repulsion ($\alpha < 0$) between the adsorbed species.

Plots of $\log\left\{[C] \times \left(\frac{\theta}{1-\theta}\right)\right\}$ versus Θ as presented in Fig 8 were linear which shows the applicability of Frumkin isotherm. The positive sign of the constant α indicated highly attractive lateral interactions in the adsorbed layer [26]. With increasing corrosion inhibitor, concentration inhibitor molecules probably start to desorbs due to interaction between the inhibiting molecules already adsorbed at the surface and those present in the solution. With increasing concentration of the inhibitor, the interactions become stronger, leading to secondary desorption.

C. Free Energy of Adsorption

The equilibrium constant of adsorption of SOB extract on the surface of mild steel is related to the free energy of adsorption (ΔG_{ads}) according to (13) [17,27,28].

$$\Delta G_{ads} = -2.303RT\log(55.5.K_{ads}) \qquad (13)$$

where R is the gas constant and T is the temperature. The free energy of adsorption was calculated from values of K_{ads} obtained from Langmuir, Temkin, Flory-Huggins and Frumkin according to Equation (13) and was recorded in Table 2. The results showed that free energy of adsorption ΔG_{ads} were negative and less than the threshold value of –40 $KJmol^{-1}$ required for chemical adsorption, indicating that adsorption of SOB extract on mild steel surface was spontaneous and occurred according to the mechanism of physical adsorption [17, 29,30].

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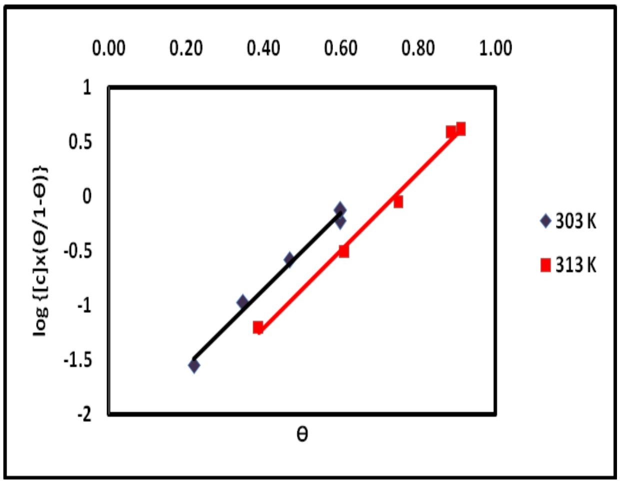

Fig 8: Frumkin isotherm for adsorption of SOB extract on the mild steel surface.

Table II: Adsorption parameters for adsorption of *Schleichera Oleosa* extract on mild steel surface.

Isotherms	Temperature K	R^2	log K_{ads}	ΔG_{ads} $KJmol^{-1}$	
Langmuir	303	0.98	-0.007	-10.08	
	313	0.97	-0.149	-9.56	
Temkin					**a**
	303	0.97	1.350	-17.94	-1.97
	313	0.97	1.500	-8.99	-1.49
Flory - Huggins					**x**
	303	0.84	0.393	-12.40	0.69
	313	0.82	0.626	-3.76	0.33
Frumkin					***α***
	303	0.99	-0.986	-4.40	1.77
	313	0.99	-1.136	-3.65	1.77

CONCLUSION

- SOB extract was found to be an inhibitor for mild steel corrosion in 0.5 M H_2SO_4
- Inhibition efficiency of the extract increased with an increase in concentration of SOB extract and which decreased with increase in temperature.
- Activation energies were higher in the presence of SOB extract showing physisorption. This trend in the mechanism of inhibition was further corroborated by the value of heat of adsorption obtained.
- The adsorption of SOB extract on the surface of mild steel is spontaneous, exothermic and was consistent with the models of Langmuir, Temkin, Flory-Huggins and Frumkin.

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