

Corrosion Inhibition Of Mild Steel In Hydrochloric Acid Solution Using Hog Plum Seed Extract

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Abstract—Corrosion inhibition of mild steel in 2 M hydrochloric acid solution by Hog plum (*Spondias mombin*) seed extract (HPSE) was studied using gravimetric and thermometric measurements. The study was conducted using varied concentration of the inhibitor from 0.1 to 0.5 g/l in the acidic solution. It was found that the corrosion rate of mild steel reduced in presence of the inhibitor in the acidic medium and thus, the seed extract act as a good corrosion inhibitor for mild steel in all concentrations of the extract. However, the inhibition action depends on the concentration of the Hog plum seed extract in the acid solution. The results of weight loss and thermometric measurements indicated that inhibition efficiency on mild steel corrosion increase with increasing inhibitor concentration with inhibition efficiency of 86.16 % achieved by addition of 0.5 g/l concentration to the acidic medium. The adsorption of HPSE on the surface of the mild steel obeys Langmuir adsorption isotherm. The Gibb's free energy of adsorption showed that the adsorption process of HPSE inhibitor on mild steel in HCl solution is spontaneous which is consistent with physical adsorption process.

Keywords— *Mild steel, hydrochloric acid, Hog plum seed, corrosion inhibitor and adsorption isotherm*

I. INTRODUCTION

Mild steel is an alloy of iron and carbon with carbon content ranges between 0.05% - 0.3%. It is commonly used for general engineering purposes due to its availability at low cost and good mechanical properties, but it is susceptible to corrosion when in contact with corrosive environment. Industrial processes such as acid pickling normally rendered mild steel to corrosion induced failure which can be hazardous to human health, environment and economy [1, 2].

Corrosion is the deterioration of metal due to chemical attacks and/or reaction between it and its environments. It is a spontaneous occurrence due to inclination of metals to return to the forms in which they were naturally found (i.e. their thermodynamic stable state of low energy). Corrosion is an unceasing

problem and ever present, often difficult to eliminate completely. Prevention and reduction are more realistic and attainable rather than total elimination. It has been reported that material selection, design and coating can be used to control corrosion [3, 4]. Corrosion of metallic materials can also be regulated by altering the corrosive environments through the addition of small quantities of special chemicals (inhibitors) to large volumes of corrosive solutions [5].

High cost, toxicity and non-biodegradability have been problems associated with the use of synthetic inhibitors, and some organic corrosion inhibitors [6]. Consequently, there have been continuous search for green corrosion inhibitors that are effective and environmentally friendly when compared to organic and synthetic inhibitors employed in industries [7]. Studies have shown that green corrosion inhibitors are natural products of plants origin with organic compounds such as alkaloids, tannins, pigments and amino acids, which exhibit strong inhibitive ability, biodegradable and inexpensive [6, 8, 9].

Over the years, research studies have been focused on corrosion inhibition of extracts from plant leaves, stems, roots and seeds, and they were found to be good inhibitors for metals and their alloys in acidic solutions [9-16]. Studies have also shown that exudates (gums) of some trees can acted as green corrosion inhibitors for metals in acidic media [17-19]. Ability of green corrosion inhibitors to protect metals and alloys against the attack of corrosive media depends on the active phytochemical compounds with antioxidant activities

In quest to explore other plant extracts that are cheap and readily available as a green corrosion inhibitors, the presence work experimental studied the degree of corrosion inhibition of Hog plum seeds extract (HPSE) on mild steel in HCl solution using weight loss (gravimetric) and thermometric measurement methods. Hog plum plant (*Spondias mombin*) is a tree and flowering plant (Figure 1a) in the family Anacardiaceae and naturally distributed in tropical Americas, West Indies and western African countries. The matured fruits of the plant is yellowish with leathery skin and a thin layer pulp with a sharp, somewhat acid taste and are edible (Figure 1b). The inner part of the fruit contained hard non-edible seed

and are normally dropped as waste. Hog plum seed contains active phytochemical compounds with antioxidant activities (phenols, tannins, carotenoids, alkaloids and flavonoids) which can act as good corrosion inhibition for metal in acidic solution [20, 21].



Figure 1: Hog Plum (a) tree and (b) ripe fruits.

II. MATERIALS AND METHOD

A. Work materials and specimen preparation

The materials used in conducting the corrosion experiment include: mild steel strips, Hog plum (*Spondias mombin*) seeds, emery papers, acetone, distilled water, ethanol, containers and hydrochloric acid solution. The mild steel used was sourced from a local market in Ado-Ekiti, Nigeria. The elemental composition of the mild steel, as shown in Table I, was determined using a solid technique optical emissions spectrograph.

The mild steel was mechanically cut into coupon test specimens (Figure 2) of surface area of 11.8 cm^2 each ($2.5 \times 2.0 \times 0.2 \text{ cm}$ dimension). They were then descaled with wire brushing, polished using series of grits emery paper from grit 60 up to grit 120 in order to ensure a smooth surface and reduce corrosion attack on the samples.



Figure 2: Mild steel coupon test specimens.

The specimen samples were then thoroughly cleaned with distilled water, degreased with ethanol and then dipped into acetone, dried in air and kept in air tight containers until needed for the corrosion test so as to reduce risk of corrosion on them from the environment.

B. Preparation of Inhibitor (Hog Plum Seed Extract)

The Hog plum seeds were collected at Christ School environment, Ado-Ekiti, Nigeria. Flesh of the seeds was removed and then washed, and sun dried to constant weight. The seeds were grinded (Figure 3(a)) and kept in a nylon prior use. 100 g of the

grinded seed was later measured, soaked in ethanol in a glass bottle that was tightly covered, as shown in Figure 3(b), to prevent evaporation at room temperature for 48 hours. The mixture was then filtered to obtain the extract filtrate. As shown in Figure 3(c), a concentrated extract was then obtained from the filtrate through rotary evaporator that distilled off the ethanol at 76°C for 20 minutes.

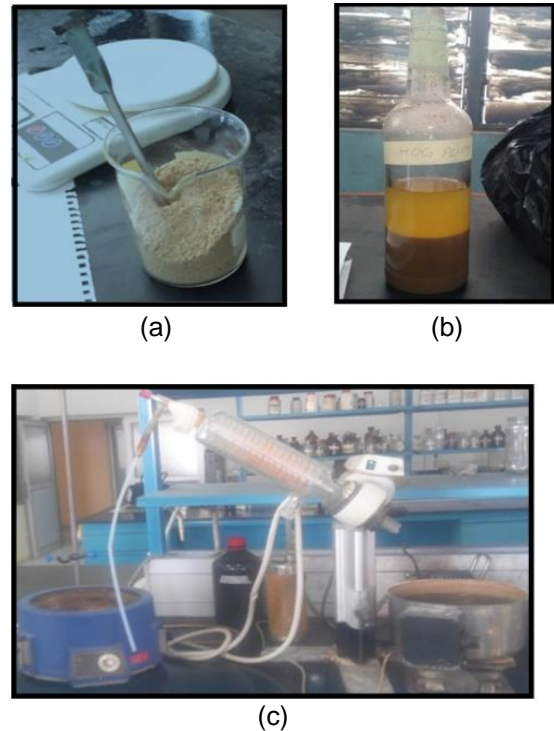


Figure 3: (a) Grinded Hog plum seed, (b) Grinded Hog plum seed soaked in ethanol and (c) Rotary evaporator for the Hog plum seed Extract.

C. Preparation of acidic solution

Standard concentrated HCl acid with density of 1.18 g/cm^3 , percentage purity of 36% and 11.97 molar was used. 2 molar solution of HCl was prepared by diluting 83.54 cm^3 of the concentrated HCl in 500 cm^3 of distilled water. Different concentrations of the inhibitor test solution were prepared by correctly weighed and dissolved 0.1 to 0.5 g/l of the HPSE inhibitor in the prepared 2 M hydrochloric acid solution in different beakers to carry out the corrosion experiment.

D. Weight loss experiment

The weight loss (gravimetric) experiment was conducted according to the ASTM practice standard G-31 [22]. The prepared mild steel coupons were weighed and recorded, then immersed completely in the aggressive solutions in the beakers without and with added studied inhibitor in different concentration as shown in Figure 4(a). At every 3 hours of exposure in the acidic solutions, the mild steel coupons were retrieved with the aid of acid resistance plastic clip, cleaned thoroughly with emery paper to remove the corrosion product, rinsed with distilled water, dipped into acetone, air-dried, reweighed and recorded to

determine the weight loss, corrosion rate and inhibition efficiency. The experiment was made to run

continuously for 18 hours at a room temperature of 27°C.

TABLE I: CHEMICAL COMPOSITION OF MILD STEEL

Element	C	Si	Mn	Cu	P	Cr	Fe	others
Composition (%)	0.18	0.26	0.78	0.17	0.16	0.11	98.08	0.26

E. Thermometric experiment

Thermometric measurement has been used to evaluate corrosion of metals and alloys in corrosive media according to Solomon, et al. [23]. This method was carried out at room temperature to compare the behaviour of the acidic solution attack on mild steel. The measurement was conducted using digital thermometer with probe to measure temperature in the reaction system during the test as shown in Figure 4(b).

The beakers were filled with the prepared corrosive solution and mild steel coupons were carefully dropped in the test solution with the aid of acid resistance plastic clip and quickly close while the thermometer probe was pierce through the foil and cock that cover the test tube (Figure 4). The change in temperature was monitored and recorded every 15 minutes interval until maximum temperature was reached. The experiment was performed for test solutions with different concentrations of the HPSE inhibitor and without the inhibitor.

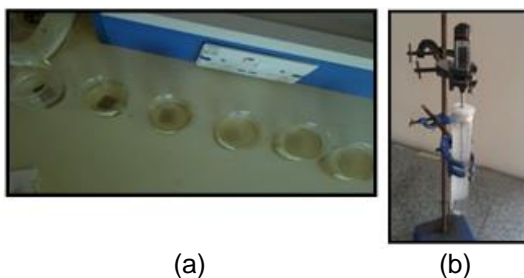


Figure 4: Experimental setup of (a) weight loss measurement and (b) thermometric measurement.

III. RESULTS AND DISCUSSION

A. Weight loss method

The weight loss was calculated by finding the differences between the initial weight, W_i (g) before immersion and final weight W_f (g) after immersion of each coupon in the prepared corrosive HCl solution with and without Hug plum seed extract, and recorded same as the weight loss W (g) as shown in Equation (1).

$$W = W_f - W_i \quad (1)$$

Table II and Figure 5 show the recorded weight loss of mild steel against time of exposure in absence (0 g/l concentration) and presence (0.1 - 0.5 g/l concentration) of inhibitor in the acidic solution.

From the results of the weight loss, it can be observed that as the exposure time increases the weight loss of mild steel increased in the acidic

solution both in absence and presence of the HPSE inhibitor. This implies that the corrosion of mild steel increases with time in acidic medium and that the corrosion cannot be entire eliminated but can only be reduced with addition of inhibitor into acidic solution. Also, it was found that the weight loss of the mild steel substantially reduced with addition of HPSE in the acidic solution and this becomes progressively obvious as the exposure time increases (Figure 5). Mild steel in acidic solution without HPSE recorded weight loss of 0.47 g more than mild steel in acidic solution with 1.0 g/l concentration of HPSE at the 3 hours of exposure time, whereas this difference increases to 1.87 g at the end of 18 hours of exposure time. Furthermore, there was notable reduction in weight loss of the mild steel as the concentration of HPSE in the acidic medium increases.

TABLE II: WEIGHT LOSS (G) VALUES OBTAINED AT ROOM TEMPERATURE

Inhibitor Conc. (g/l)	Exposure time (hour)					
	3	6	9	12	15	18
0	0.79	1.23	1.72	2.07	2.31	2.50
0.1	0.32	0.52	0.54	0.57	0.60	0.63
0.2	0.30	0.38	0.38	0.39	0.41	0.44
0.3	0.15	0.34	0.35	0.36	0.38	0.41
0.4	0.15	0.20	0.20	0.22	0.23	0.32
0.5	0.14	0.21	0.22	0.23	0.24	0.26

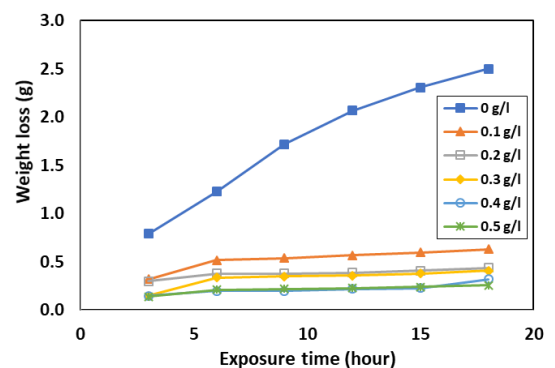


Figure 5: Graph of weight loss against exposure time.

After the weight loss has been established from the experiment, the corrosion rate, CR ($\text{g}/\text{cm}^2\text{h}$) was calculated according to Equation (2):

ed with addition of coconut pod ashes in the cast mould.

Furthermore, the cooling rate was calculated from the cooling data as follows [19]:

$$CR = \frac{W}{At} \quad (2)$$

where A is the area of the sample (cm^2) and t is the immersed time in hours. Thereafter, the inhibition efficiency, IE (%), and surface coverage, θ , were computed using Equations (3) and (4), respectively:

$$IE = \left(\frac{CR_a - CR_p}{CR_a} \right) \times 100 \quad (3)$$

$$\theta = \left(\frac{CR_a - CR_p}{CR_a} \right) \quad (4)$$

TABLE III: CORROSION RATE, INHIBITION EFFICIENCY AND SURFACE COVERAGE

Inhibitor Conc. (g/l)	Corrosion rate ($\text{g}/\text{cm}^2\text{h}$)						CR	IE %	θ
	Exposure time (hour)								
	3	6	9	12	15	18			
0	0.0223	0.0174	0.0162	0.0146	0.0131	0.0118	0.0159		
0.1	0.0090	0.0073	0.0051	0.0040	0.0034	0.0030	0.0053	66.59	0.666
0.2	0.0085	0.0054	0.0036	0.0028	0.0023	0.0021	0.0041	74.23	0.742
0.3	0.0042	0.0048	0.0033	0.0025	0.0021	0.0019	0.0032	80.12	0.801
0.4	0.0042	0.0028	0.0019	0.0016	0.0013	0.0015	0.0022	86.04	0.860
0.5	0.0040	0.0030	0.0021	0.0016	0.0014	0.0012	0.0022	86.16	0.862

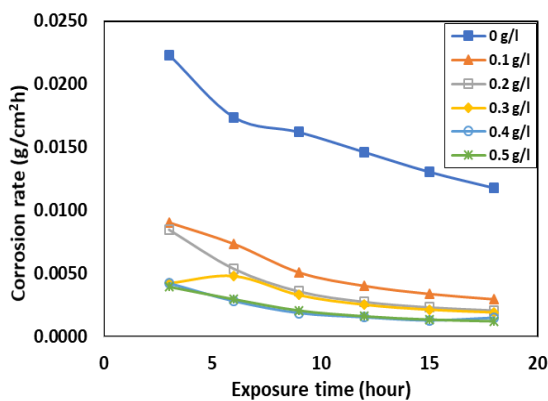


Figure 6: Graph of corrosion rate against concentration of HPSE in HCl Medium.

Figure 6. It can be seen in the Figure 6 that the corrosion rates substantial decreased with addition of the studied inhibitor, and also decreased with the increase in the concentrations of the inhibitor. It was also observed that corrosion rate of mild steel in the absence and presence of inhibitor reduces with increase in exposure time. This could be attributed to increase in concentration of stagnant ions blocking the creation of more ions which consequently reducing the activities of Fe^{2+} in the medium, and thus causing a decrease in corrosion reaction of the mild steel in its stationary acidic environment.

As shown in Table III and Figure 7, the values of inhibition efficiency IE (%) of HPSE as corrosion inhibitor of mild steel in HCl acidic medium increase with increase in inhibitors concentrations. The corrosion inhibition of HPSE could be attributed to the displacement of adsorbed water molecules by the inhibitor and thus resulting to definite adsorption of the inhibitor on the mild steel surface as indicated in Solomon et al. [23] and Stephen and Adebayo [14].

where CR_a and CR_p are the corrosion rates of sample in absence and presence of the inhibitor respectively.

The obtained corrosion rates of mild steel in the HCl acidic solution for the different concentrations (0.0 - 0.5 g/l) of added HPSE inhibitor are depicted in Table III and

It was observed that there was a diminishing effect in the inhibition efficiency of HPSE as the concentration of the inhibitor increases from 0.1 to 0.5. Difference of 10.02%, 7.10%, 3.87% and 0.065% were recorded as the concentration of inhibitor increases from 0.1 to 0.5 at 0.1 increment. This could be due to saturation effect leading to gradual reduction in the formation of adsorbed diametric film on the surface of the mild steel as concentration of inhibitor increases.

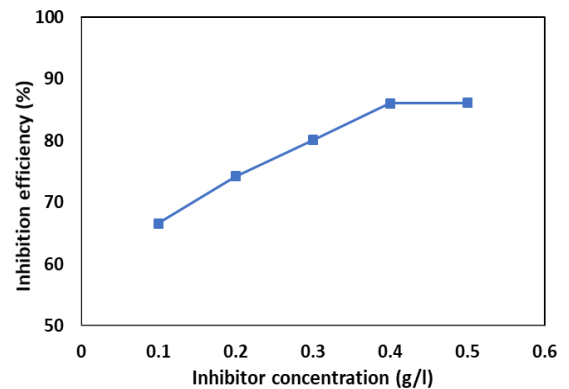


Figure 7: Graph of inhibition efficiency versus concentration of HPSE in HCl Medium.

B. Thermometric Measurement

As previously explained, corrosion reaction was studied using digital thermometric readings obtained by inserted the thermometer in the test solution in absence and presence of different concentrations of the HPSE inhibitor. The recorded temperatures obtained during the thermometric experiment were used to determine the reaction number, R ($^{\circ}\text{C}/\text{min}$), according to Equation (5), and as established in literatures [23, 24] this was taken as corrosion rate:

$$R = \frac{T_m - T_i}{t} \quad (5)$$

where T_i and T_m ($^{\circ}\text{C}$) are the initial temperature and maximum temperature of the system, respectively, and t (min) is time taken to reach the maximum temperature. The inhibition efficiency, IE (%), was calculated from the values of the computed reaction number according to Equation (6):

$$IE = \frac{R_a - R_p}{R_a} \times 100 \quad (6)$$

where R_a and R_p are the reaction numbers in absence and presence of the inhibitor, respectively.

Table IV and Figure 8 show the corrosion reaction on mild steel in absence and presence of the inhibitor during the application of thermometric measurement method. It could be observed in the Figure 8 that as exposure time increases the temperature also increases until it reach maximum at around 200 minutes, and thereafter decreases.

TABLE IV: THERMOMETRIC VALUES OBTAINED IN ABSENCE AND PRESENCE OF HPSE

Time (min)	Conc. of inhibitor (g/l)					
	0	0.1	0.2	0.3	0.4	0.5
Temperature ($^{\circ}\text{C}$)						
0	30.1	30.1	29.7	29.4	29.7	29.9
15	30.4	30.2	29.8	29.8	30.1	30.0
30	30.9	30.2	29.8	30.0	30.3	30.1
45	31.4	30.2	29.9	30.1	30.5	30.1
60	31.6	30.4	30.1	30.3	30.6	30.2
75	32.0	30.4	30.4	30.4	30.5	30.3
90	32.3	30.6	30.6	30.5	30.6	30.4
105	32.8	30.8	30.8	30.6	30.8	30.4
120	33.2	31.1	31.1	31.2	31.0	30.7
135	33.5	31.4	31.3	31.3	31.1	31.0
150	33.9	31.8	31.6	31.6	31.1	31.2
165	34.4	32.2	31.8	31.7	31.5	31.4
180	34.8	32.6	32.1	31.7	31.8	31.7
195	34.9	32.8	32.3	31.9	32.0	32.0
210	34.7	32.7	32.0	31.8	31.8	31.7
225	34.4	32.3	31.7	31.5	31.7	31.6
240	34.0	32.1	31.0	31.4	31.6	31.4
255	33.7	31.8	30.9	31.0	31.4	31.2
270	33.5	31.3	30.7	30.5	30.7	31.0
285	33.3	30.8	30.4	30.4	30.3	30.4
300	33.0	30.7	30.3	30.2	30.3	30.2
315	32.6	30.5	30.2	30.1	30.1	30.1
330	32.3	30.5	30.0	30.0	29.9	30.0
345	31.8	30.1	29.9	29.9	29.8	29.8
360	31.0	29.8	29.8	29.7	29.7	29.7
375	30.5	29.8	29.8	29.6	29.8	29.6
390	30.3	29.7	29.6	29.6	29.8	29.6
405	29.9	29.7	29.5	29.5	29.6	29.5
420	29.6	29.6	29.7	29.4	29.5	29.4

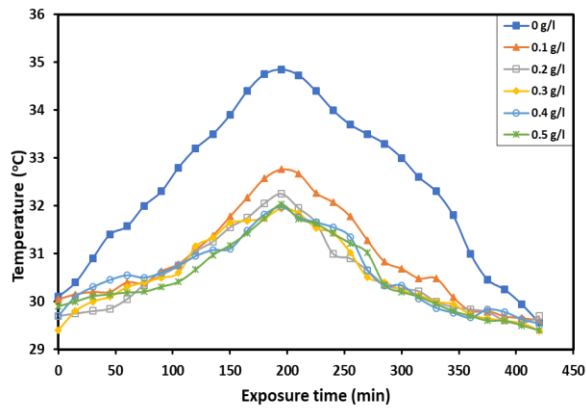


Figure 8: Graph of temperature against exposure time in HPSE concentrations.

As noted by other literatures on corrosion, the increase in temperature indicates progress in the corrosion reaction whereas the reduction could be attributed to absorption of the HPSE inhibitor on the surface of the mild steel which hindered the creation and free movement of the Fe^{2+} ions and consequently reduced the corrosion reaction of the steel and amount of heat generated in the acidic medium during the test [12, 14].

Similar to the weight loss method, the reaction number (corrosion rate) as shown in Figure V was found to reduce considerably with the addition of HPSE inhibitor, and further decreases as the concentrations of the inhibitor increases. Inhibition efficiency also increases as the concentration of inhibitor increases which could be due to higher formation of diametric film on the surface of the mild steel with more presence of HPS extraction in the acidic medium.

C. Adsorption considerations

Adsorption considerations using adsorption isotherms are very good tools in determining the

mechanism of the electromechanical interaction between organic inhibitor and metal surface. They help to illustrate the adsorption behaviour and also evaluates thermodynamic of inhibitors-metal interface corrosion [23, 25]. The inhibition of corrosion of the mild steel in the acidic medium with the addition of different concentration of HPS extract can be ascribed to the absorption of the molecules of the extract on the surface of the steel. Thus, the adsorption isotherms shows the relationship between inhibitor concentration in the corrosive medium and the surface coverage. Langmuir adsorption isotherm model was used in this study to examine thermodynamic adsorption parameter. The Langmuir adsorption equation is as stated in Equation (7):

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh}(7)$$

where C_{inh} is concentration of inhibitors, K_{ads} is the adsorption equilibrium constant, and θ is the degree of surface coverage of different concentration of the inhibitor. The degree of surface coverage as depicted in Table III was calculated from weight loss measurement for various concentrations of the studied HPS extract by dividing the IE (%) with 100.

The values of various concentrations of the HPSE inhibitor upon the surface coverage (C/θ) produced a straight line graph as depicted in Figure 9. The equilibrium constant of adsorption, K_{ads} , was calculated from Figure 9 as the inverse of the intercept of the graph. The value of the slope of the graph was found to be greater than 1. The deviation of value of slope from unity implies a heterogeneous adsorbing species inhabiting more or less a distinct adsorption site at the metal-solution interface [26], and this also has been attributed to the molecular interaction among the adsorbed inhibitor species [17].

TABLE V: REACTION NUMBER AND INHIBITION EFFICIENCY IN ABSENCE AND PRESENCE OF HPSE

	Conc. of inhibitor (g/l)					
	0	0.1	0.2	0.3	0.4	0.5
Reaction Number	0.0244	0.0139	0.0131	0.0131	0.0117	0.0109
Inhibition Efficiency		42.95	46.32	46.38	52.02	55.15

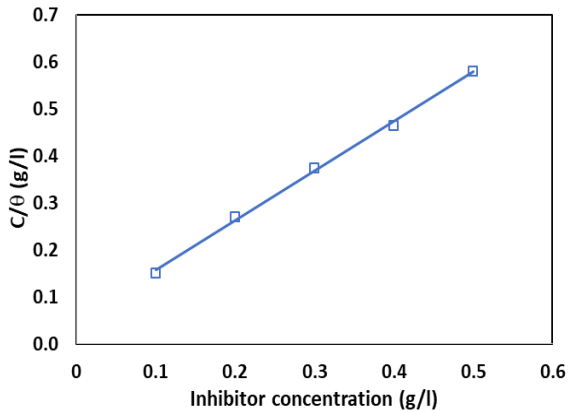


Figure 9: Langmuir isotherm model for mild steel in 2M HCl medium containing different concentration of HPS extract

The free energy of adsorption (ΔG_{ads}) and the equilibrium constant of adsorption (K_{ads}) is related as stated in Equation (8) [27]:

$$K_{ads} = (1/55.5)Exp(-\Delta G_{ads}/RT)(8)$$

where R and T are the universal gas constant and absolute temperature, respectively. The value 55.5 stands for the molar concentration of water at the electrode and electrolyte interface during the adsorption process. The Langmuir adsorption parameters calculated using Equations (6) and (7), and from the Langmuir isotherm model are shown in Table VI.

TABLE VI: ADSORPTION PARAMETERS FROM LANGMUIR ISOTHERM FOR WEIGHT LOSS METHOD

Temp.	Slope	K_{ads}	ΔG_{ads} (KJ/mol)	R^2
27°C	1.014	19.49	-17.42	0.99

The adsorption parameters indicate the binding power of the inhibitor to the steel surface [27, 28]. It has been reported that the value of free energy of adsorption (ΔG_{ads}) approximately -20 KJ/mol or lower is consistent with physisorption adsorption mechanism which involves presence of electrically charged metal surface and species in the bulk solution whereas value of around -40 KJ/mol or higher shows chemical adsorption mechanism that involves charge transfer between the inhibitor and molecules of the metal surface [17, 23]. As shown in Table VI, the calculated value of free energy of adsorption of (ΔG_{ads}) is negative and not up to -20 kJ/mol which indicates that the adsorption of HPSE on the mild steel surface is spontaneous through physical adsorption process.

IV. CONCLUSIONS

Gravimetric (weight loss) and thermometric measurements were employed to study the corrosion inhibition strength of Hug plum seed extract on mild steel in 2M HCl medium. The results of experimental studies showed that:

□HPSE exhibited good inhibition efficiency on mild steel corrosion in HCl solution.

□the inhibition efficiency increased with increase in concentration of HPSE inhibitor.

□Langmuir adsorption isotherm established the adsorption of the inhibitor molecules on mild steel surface in the HCl solution.

□the calculated value of Gibb's free energy of adsorption (ΔG_{ads}) revealed that the adsorption process of HPSE inhibitor on mild steel in HCl solution is spontaneous and also consistent with physical adsorption process.

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