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Corrosion Inhibition of Carbon Steel in 1M HCl Solution Using Sesbania Sesban Extract

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Abstract

Corrosion inhibition effect of *Sesbania sesban* extract (SSE) on the carbon steel in 1M HCl solution was investigated by potentiodynamic polarization and weight loss techniques. The corrosion rate of carbon steel was decreased in the presence of SSE. The corrosion inhibition efficiency was found to increase with SSE concentration to attain 91.08% with 2.00 g/l at 25°C. The polarization studies showed that SSE acts as mixed type inhibitor. The effect of temperature, acid concentration and immersion time on the corrosion rate of carbon steel in HCl solution in the absences and presence of SSE was also studied. Values of corrosion inhibition efficiency calculated from weight loss technique were in good agreement with those obtained from potentiodynamic polarization test. The results showed that the adsorption of the extract on the carbon steel surface obeys Langmuir isotherm. The activation parameters, apparent activation energy (E_a), the enthalpy of activation (ΔH_a) and entropy of activation (ΔS_a) for the corrosion of carbon steel in 1M HCl in the absence and presence of SSE were calculated and discussed.

Keyword: Corrosion inhibition, Carbon steel, Hydrochloric acid

1. Introduction

Metallic materials are still the most widely used group of materials particularly in both mechanical engineering and the transportation industry. However the usefulness of metals and alloys is constrained by one common problem known as corrosion (Buchweishaija, 2009). Corrosion is the destructive attack of a metal by chemical or electrochemical reaction with the environment (Uhlig et al., 2008). The basic cause of the corrosion of the metals is their tendency to return to their stable state. Nearly all metals are inherently unstable and it is their natural tendency to seek self-destruction by reacting with their environment to attain a state of lower energy by forming a metal compound. This is the state in which the majority of metals are found in nature (Haward, 1968). The use of inhibitors is one of the best methods of preventing metals against corrosion. Most of the corrosion inhibitors are synthetic chemicals, expensive and very hazardous to environments. Therefore, it is desirable to source for environmentally safe inhibitors. Plants represent a class of interesting source of compounds currently being exported for use in metal corrosion protection in most systems. The use of phytochemicals as corrosion inhibitors can be traced back to 1960s when tannins and their derivatives were used to protect corrosion of steel, iron and other tools (Ashassi-Sorkhabi et al., 2006).

Up till now extracts of plants leaves such as *Hibiscus Subdariffa* which was used by El-Hosary in 1972 (Al-Malki, 2007), *Papaia* (Zucchi et al., 1985), *Eucalyptus* which was used by Pravinar in 1993 (Buchweishaija, 2009), *EmbilicaUflicianalis* (Sanghvi et al., 1997), *RosmarinusOfficinalis L.* (Kliskic et al., 2000), *LawsoniaIntermis* (Al-Sehaibani et al.,2010), *Tobacco* (Davis et al., 2001), *Opuntia* (El-Etre et al., 2003), *Mytruscommunis* (Al-Sultan,2005), *Artemisia* (Bouklah et al., 2006), *Eclipta alba* (Shyamala et al., 2009), *Fenugreek* (Ehteram, 2008), *Vernonia Amygdaline* (Odiongenyi et al., 2009), *Phaseolusaureus* (Rajalakshimi et al., 2008), *Piper guinensis* (Ebenso et al., 2008), *Combretumbracteosum* (Okafor et al., 2009), *CyamopsisTetragonaloba* (Subhashini et al., 2010), *Murrayakoenigii* (Sharmila et al., 2010), *Annona squamosal* (Lebrini et al., 2010), *Black Pepper* (Dahmani et al., 2010), *Moringaoleifera* (Singh et al., 2010), *AzadirachtaIndica* (Okafor et al., 2010), *Jatrophacurcas* (Kumar et al., 2010), *Citrulluscolocynthis* (Chauhan et al., 2010), Palicourea*Guianensis* (Lebrini et al., 2011), green tea (Loto, 2011), *Pterocarpussoyauxi* (Iloamaeke et al., 2012) (5, 6, 1) have been studied for corrosion inhibition of metals in acid and neutral medias. The present study aimed at investigating the inhibitive properties of ethanol extract of *Sesbaniasesban* plant leaves on the corrosion of carbon steel in 1M HCl solution.

2. Experimental Details

2.1 Specimen Preparation

Carbon steel specimens of size 5.5 cm x 2.5 cm x 0.3 cm containing a small hole of 4 mm diameter near the upper edge with composition (wt. %) Si (2), Mn (0.4), C (0.368), S (0.1), Cr (0.07), Mo (0.012) and Fe (97.05) were employed for the weight loss test. For electrochemical study, carbon steel circular strips of the same composition with an exposed area of 2.54 cm^2 were used. Before each test, each specimen was polished with emery papers of 60, 1000, 1200 grades, washed with tap water followed by distilled water, dried with tissues, degreased with acetone and dried with tissues again (Khaled, 2010&Hassan et al., 2011).

2.2 Preparation of Plant Extract

Fresh leaves of *Sesbania sesban* plant which was used in this study was collected from Al-Sarragi in Basrah city. The leaves were washed with tap water several times followed by distilled water and put on the tissue to dry. After one day the leaves were dried more in an electric furnace for 15 – 30 min and grounded to powder form. Limited weight (grams) of the powder of leaves was immersed in 60 ml of ethanol for three days. After three days the solution was filtered and used as natural corrosion inhibitor in 1M HCl solution (Nahle et al., 2010).

2.3 Electrochemical Measurements

Potentiodynamic polarization measurements were carried out using computerized electrochemical analyzer model 35415. The polarization measurements were made to evaluate the corrosion current, corrosion potential and Tafel slopes. The experiments were performed by using of a three electrodes cell designed specifically for potentiodynamic polarization measurements. The carbon steel specimen of 2.54 cm^2 was the working electrode; platinum electrode was used as auxiliary electrode and standard calomel electrode (SCE) was used as reference electrode. A luggin capillary arrangement filled with the same tested solution was used to keep the reference electrode close to the working electrode. The sequences of the experimental work can be summarized as follow:

- A cleaned specimen was mounted on the electrode holder (working electrode).

- The auxiliary electrode (platinum electrode), reference electrode (SCE), luggin capillary and other components were placed in the test cell.

- 400 ml of the electrolyte was added to the test cell and the luggin capillary was filled with the same test solution.

- The temperature of the solution was brought to desire value by using water path.

- 10 min was given for each experiment to attain the steady state open circuit potential (Shyamala et al., 2008 & Ehteram, 2008).

- Then the carbon steel specimen was polarized to about ±200 mV anodically and cathodically from the open circuit potential to obtain the corrosion potential, corrosion current and Tafel slopes (Al-Sultan, 2005).

- At the end of the test, corrosion cell content evacuated and cleaned with distilled water then the experiment was repeated with another sample.

2.4 Weight Loss Tests

In the weight loss experiments, the clean coupons were weighed. After weighing the specimens accurately to the forth decimal, the coupons were immersed in 100 ml of the electrolyte (hydrochloric acid) in the absence and presence of the inhibitor. After specified period of time each specimens was taken out of the test solution, washed with distilled water, dried and weighed (Subhashini et al., 2010 &Kumar, 2010). The results of the experiments are the mean of two run. The experiments were performed for various parameters such as:

- Concentration variation (0.2, 0.4, …, 1.8, 2 g/l).
- Temperature variation (25, 35, 45, 55, 65°C).
- Different acid concentrations (0.4, 0.8, 1.2, 1.6, 2 M).
- Different immersion time (1, 6, 12, 24, …, 42, 48 hours).

3. Results and Discussion

3.1 Effect of Inhibitor Concentration

The electrochemical parameters, anodic Tafel constant (β_a), cathodic Tafel constant (β_c), corrosion potential (E_{corr}), corrosion current density (i_{corr}) and the corresponding inhibition efficiency which was calculated from equation 1 for the corrosion of carbon steel in 1M HCl solution without and with different concentrations (0.4 – 2 g/l) of SSEat 25°Care illustrated in table 1. Also figure 1 show the anodic and cathodic potentiodynamic polarization curves for the previous tests.

$$\mathrm{IE\%} = \frac{i_{corr(uninh)} - i_{corr(inh)}}{i_{corr(uninh)}} * 100 \qquad (1)$$

It is clearly observed from table 1 that SSE reduces the corrosion current density so increase the corrosion inhibition efficiency and the suppression in current increases as the extracts concentration increases. The corrosion current density of carbon steel in 1M HCl solution reduced from $231.98 \mu A/cm^2$ to 20.96 and the corrosion inhibition efficiency increased to 91.08% in the presence of 2 g/l of SSE at 25°C. In general, the increase in the inhibition efficiency could be attributed to the increased coverage of the surface area of the carbon steel by SSE molecules as it blocks the available sites for reaction on the surface (Sharmila et al., 2010 &Ibrahim et al., 2011).

An inhibitor can be classified as cathodic or anodic type if the displacement in corrosion potential is more than 85 mV with respect to the blank (Ibrahim et al., 2011). The addition of SSE in 1M HCl solution does not show any significant change in corrosion potential suggesting that the prepared extract control the corrosion by controlling both anodic and cathodic reactions. Although there was not a specific relation between corrosion potential and extract concentration (Lebrini et al., 2011). These results reveal that SSE in 1M HCl solution acts as mixed type inhibitor (Sharmila et al., 2010).

Also the change in anodic and cathodic Tafel constants, as shown in tables 1, supports the inhibitive characteristics of the SSE (Sharmila et al., 2010, Lebrini et al., 2011 & Ibrahim et al., 2011).

Table 1. Polarization parameters and the corresponding inhibition efficiency for the corrosion of carbon steel in 1M HCl solution in the absence and presence of different concentrations of SSE at 25℃

SSE Concentration (g/l)	β_a (mV/Dec)	β_c (mV/Dec)	E_{corr} (mV)	I_{corr} ($\mu A/cm^2$)	I.E %
0.0	79.4	-123.1	-403.8	231.98	--------
0.4	41.3	-106.1	-390.0	29.42	87.31
0.8	146.0	-272.5	-433.3	28.67	87.64
1.2	85.5	-124.7	-418.1	26.84	88.43
1.6	69.7	-140.9	-396.2	25.61	88.96
2.0	39.7	-101.0	-383.0	20.69	91.08

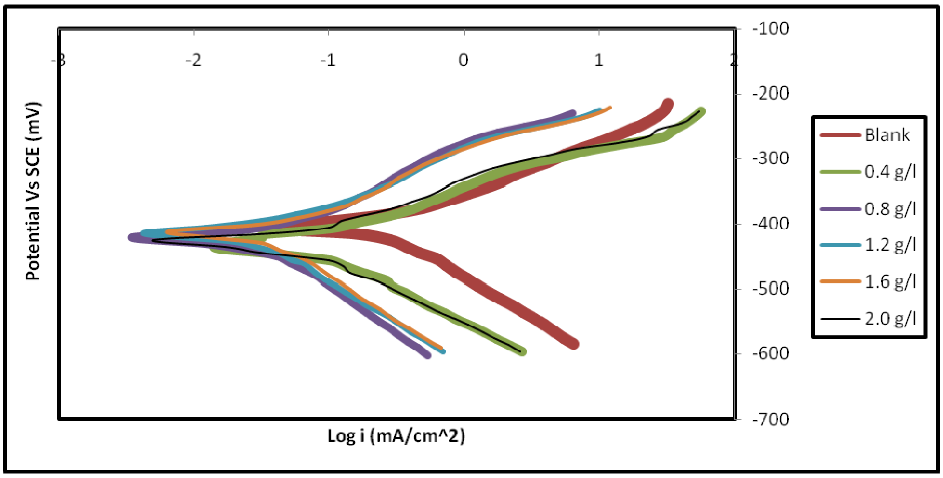


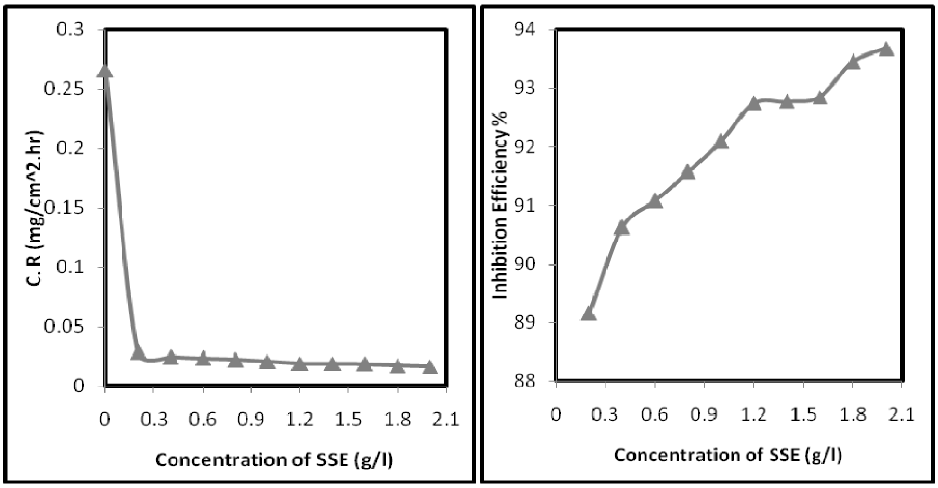
Figure 1. Polarization curves of carbon steel in 1M HCl solution without and with different concentrations of SSE at 25℃

Values of corrosion inhibition efficiency calculated from potentiodynamic polarization test were in good agreement with those obtained from weight loss technique. Various corrosion parameters such as weight loss, corrosion rate, inhibition efficiency which was calculated from equation 2 and surface coverage in the presence of different concentrations (0.2, 0.4, …, 1.8, 2 g/l) of SSE for the corrosion inhibition of carbon steel in 1M HCl solution duration 24 hours at 25℃ are illustrated in table 2. Figure 2 shows the variation of corrosion rate ($mg\,cm^{-2}hr^{-1}$) and inhibition efficiency with concentration of SSE in 1M HCl solution. It can be seen that corrosion rate of carbon steel was significantly lowered down in presence of the extract and with the increase in concentration of the extract the corrosion rate decreased gradually. Corrosion rate of carbon steel in 1M HCl solution decreased from 0.2659 $mg\,cm^{-2}hr^{-1}$ to0.0168$mg\,cm^{-2}hr^{-1}$.On the other hand, corrosion inhibition efficiency increased with increasing the extract concentration. The corrosion inhibition efficiency of the SSE in1M HCl solution reached to 93.68% with 2 g/l concentration. Also as explained previous this behavior can be attributed to the increase of surface covered and adsorption of natural compounds on the surface of the carbon steel as the inhibitor concentration increases. Due to adsorption the corrosion sites of carbon steel get blocked and adsorbed film acts as barrier between carbon steel surface and corrosion medium (Al-Malki, 2007; Iloamaeke et al., 2012; Hassan et al., 2011; Nahle et al., 2010).

$$IE\% = \frac{C.R_{uninh} - C.R_{inh}}{C.R_{uninh}} \qquad (2)$$

Table 2. Corrosion parameters obtained from weight loss measurement for carbon steel in 1M HCl solution for 24hours immersion time at 25°C in the absence and presence of different concentrations of SSE

concentration (g/l)	weight loss (mg)	CR ($mg/cm^2.hr$)	I.E%	θ
0.0	199.5	0.2659	---------	--------
0.2	23.0	0.0288	89.16	0.8916
0.4	18.8	0.0249	90.63	0.9063
0.6	18.0	0.0237	91.08	0.9108
0.8	17	0.0224	91.57	0.9157
1.0	16.2	0.0210	92.10	0.9210
1.2	15.05	0.0193	92.74	0.9274
1.4	14.8	0.0192	92.77	0.9277
1.6	14.6	0.0190	92.85	0.9285
1.8	13.4	0.0174	93.45	0.9345
2	13.0	0.0168	93.68	0.9368



(A) (B)

Figure 2. (A) Variation of corrosion rate of carbon steel with concentration of SSE at 25°C, (B)Variation of percentage corrosion inhibition efficiency with the concentration of SSE at 25°C

3.2 Effect of Temperature

The effect of temperature on the various corrosion parameters, corrosion potential, corrosion current density and inhibition efficiency, was studied in 1M HCl solution at temperature range 25 - 65°C in the absence and presence of 2g/l of SSE. The results are listed in table 3. Also, the anodic and cathodic polarization curves of the tests are showed in figure 3.

Generally, corrosion current density was found to increase with temperature rise. This is in accordance with the general rule guiding the rate of chemical reaction which says that chemical reaction increases with increasing temperature (Shyamala et al., 2008). On the other hand, it was seen that corrosion inhibition efficiency of SSE increased as the temperature increased which indicates that adsorption of the extract on the carbon steel surface is chemical in nature (Okafor et al., 2010). These results are in good agreement with those obtained from weight loss test (figure 4).

Table 3. Corrosion parameters obtained from potentiodynamic polarization curves for carbon steel immersed in 1M HCl solution in the presence of 2 g/l SSE at different temperatures

Tem. °C	β_a (mV/Dec)	β_c (mV/Dec)	E_{cor} (mV)	I_{corr} (µA/cm^2)	I.E %
25	39.7	-101.0	-383.0	20.69	91.08
35	44.6	-97.8	-394.0	36.68	91.15
45	94.4	-127.0	-417.9	60.68	91.60
55	96.4	-127.9	-411.9	73.72	92.91
65	84.6	-123.8	-397.3	115.48	93.92

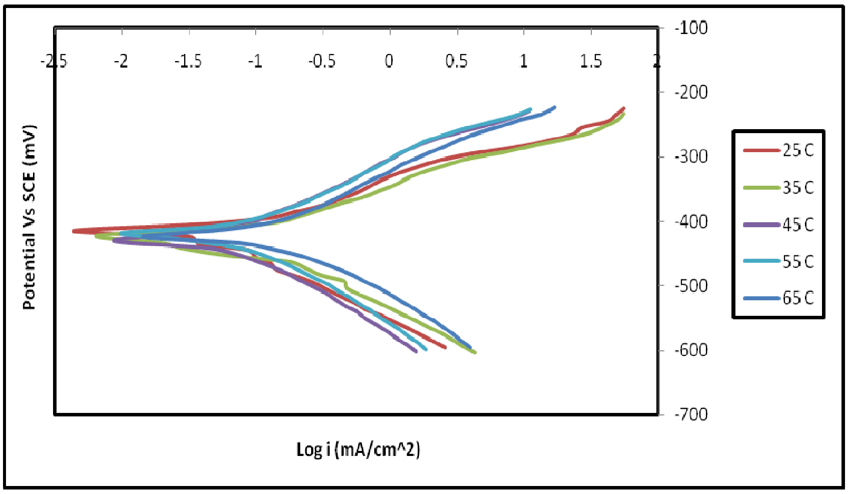
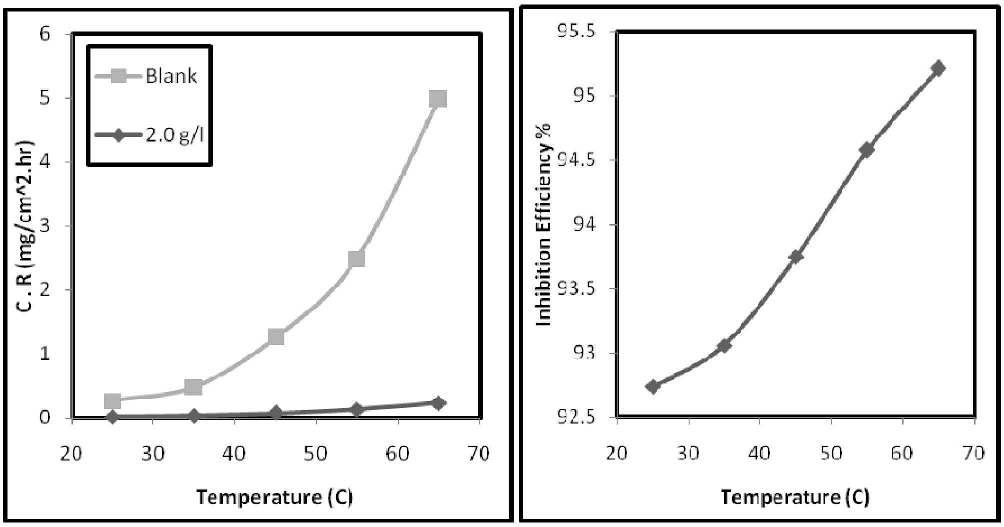


Figure 3. Polarization curves of carbon steel in 1M HCl containing 2.0 g/l of SSE at different temperatures



(A) (B)

Figure 4. (A) Plot of corrosion rate of carbon steel in 1M HCl without and with 2 g/l of SSE against temperature obtained from weight loss measurement, (B) Plot of inhibition efficiency of SSE in 1M HCl solution against temperature obtained from weight loss measurement

3.3 Activation Parameters of Inhibition Process

The apparent activation energy (E_a), the enthalpy of activation (ΔH_a) and entropy of activation (ΔS_a) for the corrosion of carbon steel in 1M HCl solution in the absence and presence of SSE were calculated from Arrhenius equation and Arrhenius transition state equation (Dahmani et al., 2010&Singh et al., 2010):

$$\log C.R = -\frac{E_a}{2.303RT} + \log \text{constant} \tag{3}$$

$$\log \frac{\mathrm{C.R}}{\mathrm{T}} = \left[\log \frac{\mathrm{R}}{N_A \mathrm{h}} + \frac{\Delta \mathrm{S_a}}{2.303\mathrm{R}}\right] - \frac{\Delta \mathrm{H_a}}{2.303\mathrm{RT}} \tag{4}$$

A plot of logarithm corrosion rate of carbon steel obtained from weight loss measurements versus the reciprocal of absolute temperature ranges (25 – 65°C), gives a straight line as shown in figure (5) with slope $-E_a/2.303R$. On the other hand, a plot of $\log C.R/T$ versus $1/T$ gives a straight line (figure (6)) with a slope equal to $-\Delta H_a/2.303R$ and an intercept of $\log R/Nh + \Delta S_a/2.303R$, from which the values of ΔH_a and ΔS_a were calculated. The values of E_a, ΔH_a and ΔS_a are listed in table 4.

The results showed that the activation energy decreased in the presence of different concentrations of SSE in 1M HCl. The decrease in corrosion activation energy in the presence of the inhibitor compared to its absence and the increase in inhibition efficiency with rise in temperature is explained as chemisorption of inhibitor molecule on the carbon steel surface (Okafor et al., 2010 & Znini et al., 2011). So as explained previous, SSE in 1M

HCl solution is a chemisorption inhibitor. The positive signs of enthalpies reflect the endothermic nature of dissolution process. Moreover, the average difference value of the $E_a - \Delta H_a$ is 2.6365 which is approximately equal to the average value of RT (2.6438). Therefore, it is indicated that the corrosion process is a unimolecular reaction as it is characterized by the following equation (Dahmani et al., 2010 & Lebrini et al., 2011):

$$E_a - \Delta H_a = RT \quad (5)$$

The entropy of activation in the absence and presence of SSE has negative values. This indicates that the activated complex in the rate determining step represents an association rather than dissociation, meaning that, a decrease in disordering takes place on going from reactants to the activated complex (Lebrini et al., 2010).

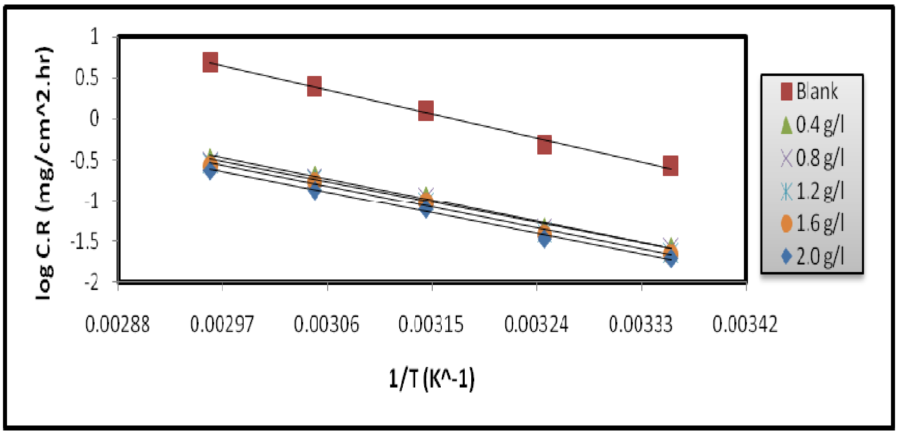


Figure 5. Plot of log C.R against 1/T for carbon steel in 1M HCl in the absence and presence of different concentration of SSE

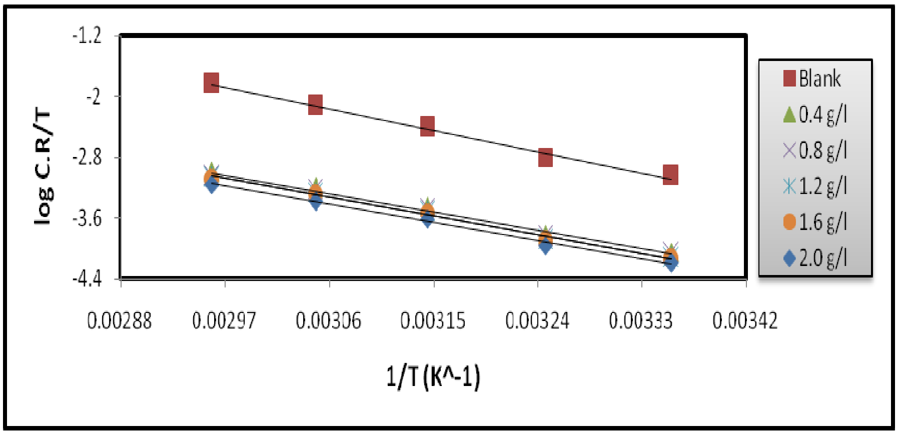


Figure 6. Plot of log C.R/T against 1/T for carbon steel in 1M HCl solution in the absence and presence of different concentration of SSE

Table 4. Activation parameters for the dissolution of carbon steel in 1M HCl in the absence and presence of different concentration of SSE

Concentration (g/l)	E_a (KJ/mol)	ΔH_a (KJ/mol)	ΔS_a (J/mol.K)	$E_a - \Delta H_a$ (KJ/mol)
0.0	62.7375	60.1009	-54.9641	2.6365
0.4	55.1610	52.5244	-99.1538	2.6365
0.8	53.4396	50.8031	-105.0013	2.6365
1.2	55.7469	53.1103	-98.5564	2.6365
1.6	55.1571	52.5206	-100.7641	2.6365
2.0	53.7307	51.0941	-106.6595	2.6365

3.4 Effect of Acid Concentration

The effect of concentration of HCl solution (0.4, 0.8, 1.2, 1.6 and 2M) on the corrosion rate of carbon steel and corrosion inhibition efficiency of SSE duration 24 hours at 25℃ was studied. As shown in figure (7), it is clearly seen that the corrosion rate of carbon steel in HCl solution without and with SSE increases with increasing the acidity. However, the presence of SSE caused a sharp decrease in corrosion rate of carbon steel. In acidic solution hydrogen evolution increases (i.e. increase the cathodic reaction) and this leads to increase in the corrosion rate. But in alkaline and neutral solutions the corrosion rate was less than in acid solution because the cathodic reaction for alkaline and neutral solutions is slower; also iron becomes increasingly passive in alkaline and neutral solutions (Al-Rikabi, 2003).

From figure (8) it can be noted that the corrosion inhibition efficiency of SSE increases with increasing the acidity of the solution. This behavior can be attributed to more easily ionization of the inhibitors under more acidic solution and so more easily adsorption on the metal surface (Chauhan et al., 2010). The maximum inhibition efficiency of 94.34%was obtained in the presence of 2 g/l of SSE in 2M HCl solution duration 24 hours at 25℃, respectively.

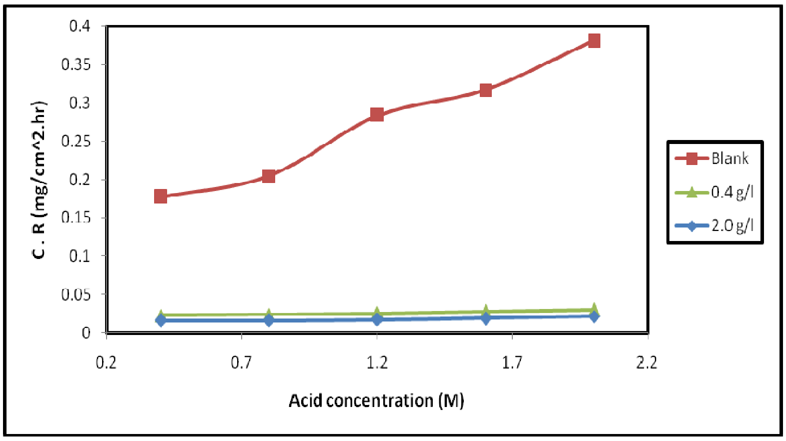


Figure 7. Variance of corrosion rate of carbon steel against concentration of HCl solution without and with 0.4 and 2.0 g/l of SSE duration 24 hours at 25℃

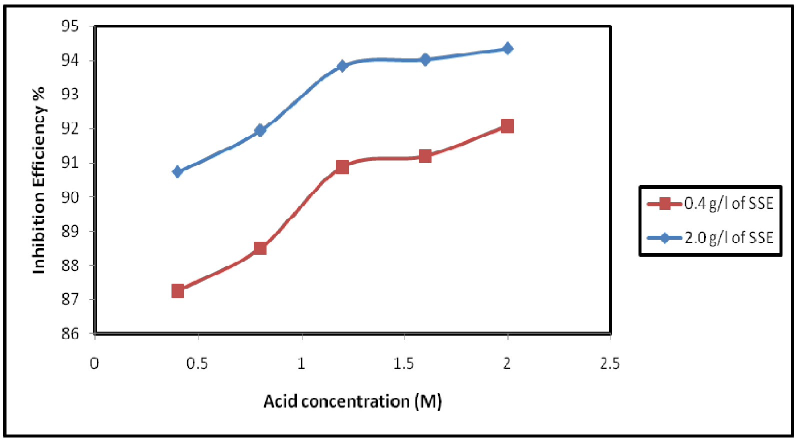


Figure 8. Variance of corrosion inhibition efficiency of SSE against concentration of HCl solution duration 24 hoursat 25℃

3.5 Effect of Immersion Time

To investigate the effect of exposure time on the weight loss and inhibition efficiency of SSE, experiments were carried out at various time intervals. Figure (9) represents the relation between time and weight loss of carbon steel in 1M HCl solution without and with different concentrations of the extract at 25℃. Inspection of the figure reveals that weight loss increases with increasing time from 1 to 48 hours in 1M HCl solution in all tested solutions. However, the presence of the extracts caused a decrease in the rate of weight loss (Okafor et al., 2010).

It was found that the inhibition efficiency of SSE in 1M HCl solution increased slightly with rise in time which may attributed to the formation of strong inhibitor layer which prevents the attack of acid on metal surface (Singh et al., 2010). Figure (10) shows this result which is in agreement with that reported in previous study (Rajalakshimi et al., 2008 & Singh et al., 2010).

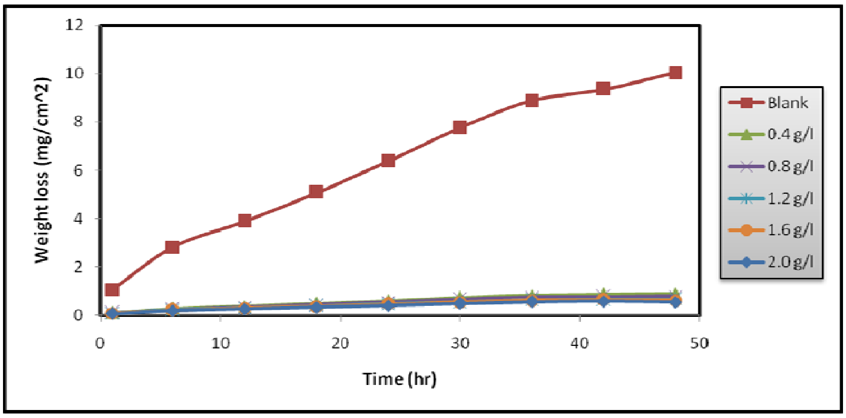


Figure 9. Variation of weight loss with time for carbon steel in 1M HCl solution without and with different concentrations of SSE at 25℃

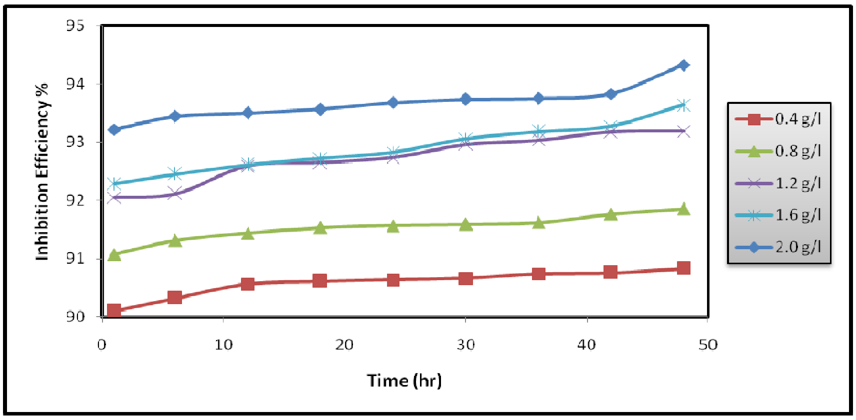


Figure 10. Variation of inhibition efficiency of SSE with time for carbon steel in 1M HCl solution at 25°C

3.6 Adsorption Isotherm

Addition of SSE molecules which adsorbed on the carbon steel surface and the interaction between them can be described by adsorption isotherms. The data were tested graphically by fitting to various isotherms including Langmuir, Frumkin and Temkin models. These models are given respectively by (Lebrini et al., 2011):

$$\frac{C}{\theta} = \frac{1}{K} + C \tag{6}$$

$$\left(\frac{\theta}{1-\theta}\right) \exp(-2j\theta) = KC \tag{7}$$

$$\exp(-2j\theta) = KC \tag{8}$$

Figures (11-13) show the linear fitting of SSE in 1M HCl solution according to the previous mentioned isotherm models at temperature range (25 – 65°C) and the values of R^2 for each line are listed in table (5). The best fit was obtained with the Langmuir isotherm.

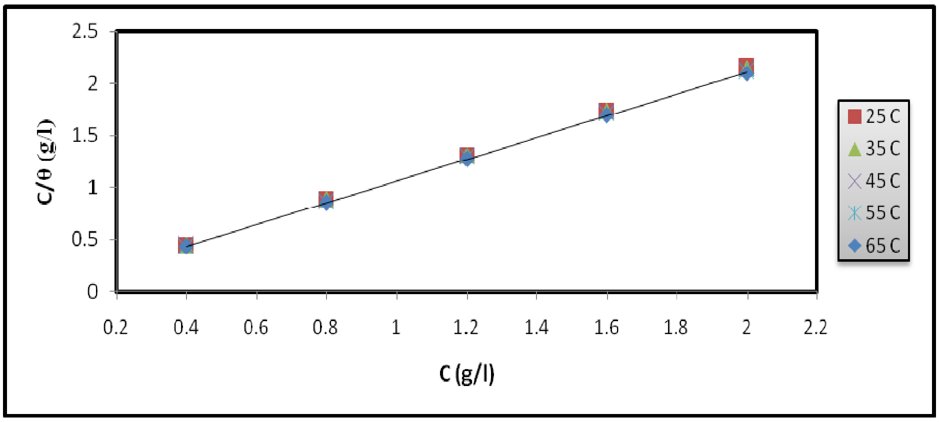


Figure 11. Langmuir isotherm for the adsorption of SSE on the surface of the carbon steel in 1M HCl solution at different temperatures

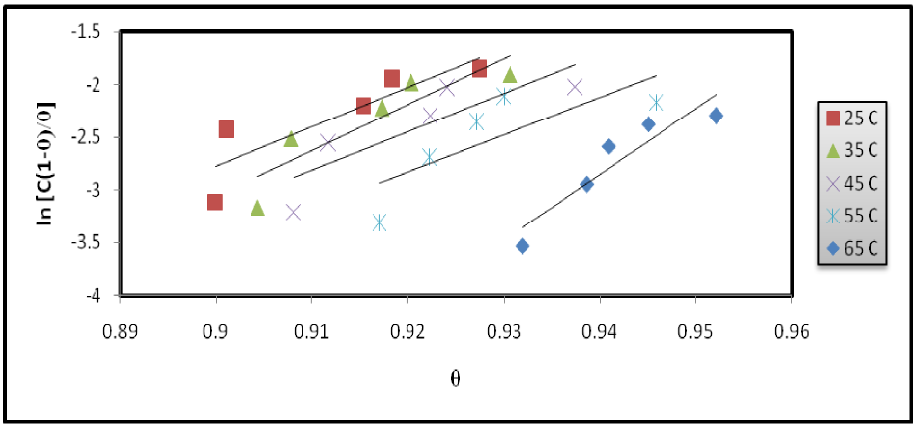


Figure 12. Frumkin isotherm for the adsorption of SSE on the surface of the carbon steel in 1M HCl solution at different temperatures

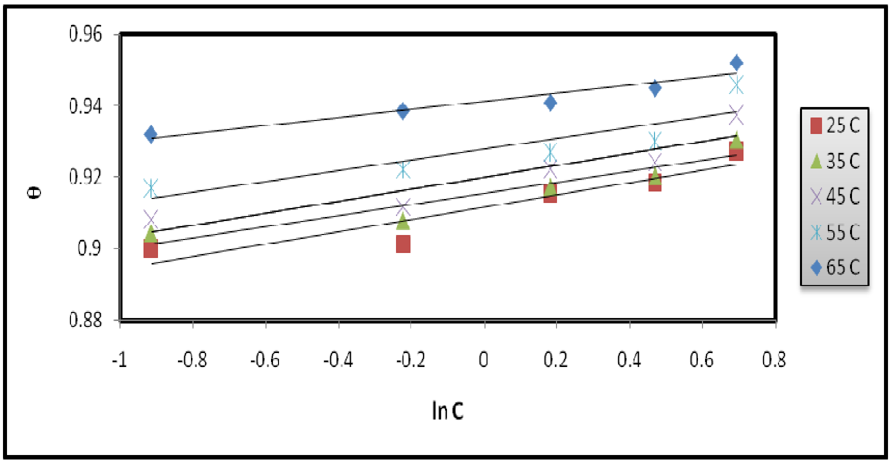


Figure13. Temkin isotherm for the adsorption of SSE on the surface of the carbon steel in 1M HCl solution at different temperatures

Table 5. Values of R^2 for the linear fitting of adsorption of SSE on the carbon steel surface in 1M HCl solution at temperature range (25 - 65°C) according to Langmiur, Frumkin and Temkin isotherms

Tem. (°C)	Langmuir model	Frumkin model	Temkin model
25	0.999	0.7705	0.9455
35	0.9999	0.8047	0.9854
45	0.9998	0.7297	0.8594
55	0.9997	0.6043	0.8979
65	0.9999	0.8589	0.9807

4. Conclusion

The following conclusions can be drawn from the present investigation:

- SSE was found to be an efficient natural corrosion inhibitor for carbon steel in 1M HCl solution by using potentiodynamic polarization and weight loss techniques.
- The results obtained from potentiodynamic polarization and weight loss measurements are in good agreement.
- The corrosion inhibition efficiency of the extract increases with increasing of its concentration.
- The adsorption of SSE on the carbon steel surface in 1M HCl solution obeys Langmuir isotherm.
- Corrosion inhibition efficiency of SSE in 1M HCl solution increases as the temperature increases which indicates that the adsorption process of SSE on the surface of the carbon steel in 1M HCl solution is chemical in nature.
- The presence of SSE in 1M hydrochloric acid solution decreases the activation energy of corrosion process which proved the previous point.
- The corrosion inhibition efficiency of SSE increases with the rise in acidity of the solution.
- With the rise in immersion time of the carbon steel coupons in 1M HCl solution, the corrosion inhibition efficiency of SSE in 1M HCl solution slightly increases.

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Symbols

β_a, β_c Anodic and Cathodic Tafel constants

C. R Corrosion rate, (mg $cm^{-2}hr^{-1}$)

$C.R_{uninh}$ Corrosion rate of uninhibited media, (mg $cm^{-2}hr^{-1}$)

$C.R_{inh}$ Corrosion rate of inhibited media, (mg $cm^{-2}hr^{-1}$)

C Concentration of the inhibitor (g l^{-1})

E_{corr} Corrosion potential, (mV)

h Planks constant ($6.626*10^{-34}$), (J.s)

IE% Inhibition Efficiency

$i_{corr(uninh)}$ Corrosion current density for uninhibited solution, (μA cm^{-2})

$i_{corr(inh)}$ Corrosion current density for inhibited solution, (μA cm^{-2})

i_{corr} Corrosion current density, (μA cm^{-2})

j Molecular interaction constant

K Binding constant of the adsorption reaction

N_A Avogadro's number ($6.022*10^{23}$), (mol^{-1})

R Gas constant (8.314), (J $mol^{-1}K^{-1}$)

T Temperature, (°C)

θ Surface coverage

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