



Investigation on The Effect of *Zizyphus Spina Christi* on the Corrosion Inhibition of Medium Carbon Steel in 0.5M Hydrochloric Acid Solution

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ABSTRACT

This study aimed to examine the influence of an inhibitor, *Zizyphus spina Christi* (ZSC) leaves, on the corrosion of medium carbon steel in 0.5 M Hydrochloric acid (HCl) solution over the temperature at 30 and 60 °C by weight loss measurements. Chemical analysis has been used to determine the impact of adding hydrochloric acid on the corrosion and corrosion inhibition of mild steel in 0.5 M hydrochloric acid (HCl) at 30 and 60 °C in the absence and presence of aqueous and alcoholic extracts of (ZSC) plant. The efficiency of corrosion inhibition was assessed using a weight loss measurement method. The standard approach to measuring the weight loss of metal cylinders exposed to corrosive environments is called laboratory immersion corrosion testing. At both temperatures of 30 °C or 60 °C, it has been discovered that the effects of inhibitor concentration on the corrosion rate of low steel increase with inhibitor concentration. To find the most accurate isothermal equilibrium data set models for the employed inhibitor, experimental data was used. All of the employed adsorption isotherms, such as the Temkin and Langmuir isotherms, fit the results, although applying an alcohol extract inhibitor at 60 °C showed a slight improvement.

دراسة تأثير مستخلص الكحولي و المائي لإوراق السدر على تثبيط تآكل الحديد المتوسط الكربون في 0.5 مولارى من محلول حمض الهيدروكلوريك.

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الكلمات المفتاحية:

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عمليات الإمتزاز

الملخص

الهدف من هذه الدراسة هو دراسة تأثير مثبط اوراق السدر على تآكل الفولاذ المنخفض (الفولاذ المتوسط الكربون) في محلول 0.5 مولار من حمض الهيدروكلوريك (HCl) على مدى درجات حرارة 30 و 60 درجة مئوية. عن طريق قياسات فقدان الوزن. تم استخدام التحليل الكيميائي لتحديد تأثير إضافة حمض الهيدروكلوريك على تآكل وتثبيط تآكل الفولاذ الطري في 0.5 مولار من حمض الهيدروكلوريك (HCl) عند درجتى 30، 60 درجة مئوية في غياب وجود المستخلصات المائية والكحولية لنبات السدر، تم تقييم كفاءة تثبيط التآكل باستخدام طريقة قياس فقدان الوزن. يُطلق على الطريقة القياسية لقياس فقدان الوزن للأسطوانة المعدنية المعرضة للبيئات المسببة للتآكل اسم اختبار التآكل بالغمر في المختبر. عند درجتى الحرارة 30 درجة مئوية أو 60 درجة مئوية، تم اكتشاف أن تأثير تركيز المثبط على معدل تآكل الفولاذ المنخفض يزداد مع تركيز المثبط. للعثور على

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نماذج مجموعات بيانات التوازن متساوي الحرارة الأكثر دقة للمثبط المستخدم، تم استخدام البيانات التجريبية. جميع الايزو الامتزاز المستخدم، مثل الايزوتيميكيونولانجيمور، يناسب النتائج، على الرغم من أن تطبيق مثبط مستخلص الكحول عند 60 درجة مئوية أظهر تحسناً طفيفاً.

1. Introduction

Corrosion is simply the degradation of metallic materials as a result of interaction with their environment, and it has a detrimental impact on the advancement of science and technology with a variety of records of process failure and losses in the oil and gas sector. In addition to causing material loss, corrosion compromises worker safety slows business operations and endangers the environment. Corrosion costs several countries throughout the world a significant portion of their GDP [1]. Acidic, alkaline, and saline operating conditions are all used in the metallurgical processing and usage of steel and its alloys. In the petrochemical industries, the interaction of steel with acid, which occurs during acid cleaning, and oil well maintenance, frequently causes surface degeneration and mechanical fractures that contribute to equipment failure and breakdown [2]. In many harsh conditions, synthetic organic compounds are utilized as corrosion inhibitors to stop the corrosion of various metals and alloys. Researchers concentrate their efforts on creating low-cost, non-toxic, biodegradable, and environmentally friendly natural compounds of plant origin as corrosion inhibitors [3–11]. Numerous substances, many of which are chemical in nature and act as corrosion inhibitors, such as aromatic heterocyclic compounds, aliphatic amines, lanthanides, Schiff's bases, imidazole, and its derivatives, are among them. Additionally, hazardous solvents, surfactants, and other property boosters are used in the production of corrosion inhibitors. Despite the fact that these inhibitors are effective, their usage is limited because of their toxicity, high cost, and issues with secondary contamination [12–13]. In order to produce safe inhibitors, research is currently focused on plant extracts since they are eco-friendly, easily accessible, affordable, and renewable sources of efficient corrosion inhibitors [14–20]. Plant extracts with nitrogen, oxygen, or sulphur atoms have been utilized to prevent the corrosion of zinc and other metals in solutions like HCl and NaCl [21–25]. The inhibition of these organic chemicals is ascribed to interactions between the inhibitor molecules and the carbon steel surface through their adsorption. These organic inhibitors create a hydrophobic film of adsorbed molecules that shields the metal surface and prevents the metal from dissolving in the electrolyte. The organic compounds are added to acid solutions for the corrosion investigations, and metal specimens are submerged in the acid solution for a set amount of time. Corrosion rate is defined as the decrease in metal weight per unit of time. The corrosion rate is decreased by adding a compound to the corrosive solution because it causes less metal to lose weight. There are several models in the literature to explain how inhibitors work. Organic molecules are adsorbed on the corroding metal surface, and this adsorption frequently results in a displacement reaction where the adsorbed water molecules are removed [26, 27, 28].

2. Materials and Methods

2.1. Steel Materials Preparation

Commercial steel rods were chosen as test items for the investigations on corrosion. Using a diamond wheel cutting machine, these medium steel rods were formed into cylinders of 40 mm in length and 10 mm in diameter. The cylinders were then polished to a mirror finish using emery paper of various grades (60, 100, 120, 180, 220, 320, 400, and 1200 grade). The manufacturer provided information on the chemical composition of the carbon steel rods shown in Table 1.

Table 1: Chemical compositions of Medium Steel Used in this Work.

Fe	C	Si	Mn	S	Al	Cu
95.0	0.45	330.1	0.91	0.25	0.64	0.49

2.2. Preparation of zizyphus spina Christi leaves

Aqueous and alcoholic extracts of the *Zizyphus Spina-Christi* leaves are used to prepare a stock solution of the plant's leaves: In a round bottom flask with an air condenser, 20 g of dried and ground *Zizyphus Spina-Christi* leaves powder was heated for 24 hours at 70 °C to 80 °C with 500 ml of distilled water and ethanol. After being allowed to stand overnight, the extract was filtered and made up to 500 ml with distilled water.

2.3. Corrosion Measurements

A weight loss approach was used to calculate the rates of corrosion for steel samples that were inhibited and uninhibited. At 30 and 60 °C, weight loss measures were taken. These cylinders were submerged in 0.5M HCl, and the loss weight of each successive weighting sample exposed to the test solution for rinsed, dried, and weighted w_2 was noted. According to weight loss in $\text{gm}/\text{cm}^2 \text{min}^{-1}$, the corrosion rate of weight loss (CR) was determined using Equ. (1):

$$CR = \frac{W_2 - W_1}{A \cdot t} (1) \quad [29]$$

Where W_1 is the original weight (gm) and W_2 are the weights after immersion in the test electrolyte, specimen in the absence and presence of an inhibitor, respectively, A is the surface area of the mild steel specimen and t is the time of each exposure. The percentage of inhibition efficiency (I. E) of a corrosion inhibitor is calculated from the corrosion rate values using Equ. (2).

$$I. E = \left[1 - \frac{CR_{Corr,add}}{CR_{Corr,free}} \right] \times 100 \quad (2) \quad [30]$$

Where, $CR_{Corr,free}$ and $CR_{Corr,add}$ are the corrosion rates in the absence and presence of a certain concentration of the inhibitor.

$$\text{Surface Coverage} = \text{Inhibition Efficiency} / 100 \quad (3) \quad [30].$$

3. Results and Discussion

3.1. Corrosion Rate and Protection Efficiency

The data for the area of specimens and inhibitor concentration of the current study are presented in Table (2) for alcoholic extracts, at temperatures 30 °C or 60 °C. Table (3) show the same data for aqueous extract, at temperatures of 30 °C and 60 °C.

Table 2: Surface area of steel specimens against alcoholic extracts concentration at 30 °C and 60 °C.

Inhibitor Concentration 0.5M HCl %	Area (cm ²) at 30 °C	Area (cm ²) at 60 °C
0.0	33.14	33.08
0.01	37.07	31.53
0.02	37.17	30.63
0.03	37.87	32.57
0.04	37.12	36.03
0.05	38.25	36.68

Table 3: Surface area of steel specimens against the aqueous extract concentration at 30 °C and 60 °C.

Inhibitor concentration in 0.5M HCl %	Area (cm ²) at 30 °C	Area (cm ²) at 60 °C
0.0	30.69	30.29
0.01	32.01	31.40
0.02	31.86	35.65
0.03	30.61	31.83
0.04	30.66	33.03
0.05	32.21	35.45

Table 4 and 5 provide the data corrosion rate estimations for steel samples using alcoholic and aqueous extracts, respectively. According to Figure 1, the corrosion rate in alcoholic extract is inversely correlated with the inhibitor concentration, which means that when the inhibitor concentration increases at both 30 °C and 60 °C, the corrosion rate decreases. This correlation is nearly linear. Despite the differences in inhibitor concentration, it should be noted that the temperature change has very little impact on the rate of corrosion for the steel samples.

Table 4: Corrosion rate for the steel samples in the alcoholic extract.

Inhibitor Concentration 0.5M HCl %	CR (mg)/cm ² .min*10 ⁻³ , 30 °C	CR (mg)/cm ² .min*10 ⁻³ , 60 °C
0.0	7.622	8.699
0.01	7.194	7.758
0.02	6.734	7.227
0.03	5.401	6.012
0.04	4.155	5.314
0.05	3.008	4.034

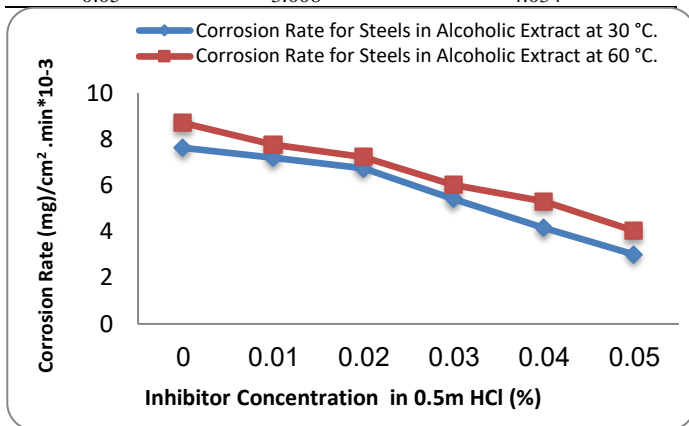


Fig (1): The corrosion rate in the alcoholic extract at both temperatures 30 and 60 °C.

The corrosion rate when employing aqueous extract is once more inversely proportional to the inhibitor concentration, as shown in Figure 2 below. At both 30 °C and 60 °C, the corrosion rate is reduced as inhibitor concentrations are increased. This correlation is nearly linear. Here, the temperature change significantly affects the rate of corrosion for the steel samples, unlike when employing alcoholic extract. The corrosion rate for the steel samples is lower at the higher temperature (60 °C) compared to the corrosion rate at 30 °C. According to some research, a metal's rate of corrosion doubles for every 10 °C increase in temperature [31]. However, this behavior conflicts with those findings. It was discovered that the dissolution of the iron-carbon alloy increases by 2 times when the temperature rises by 10 °C. The types of aqueous extract employed in this study may have an adverse impact on temperature increase.

Table 5: the Corrosion Rate for steel samples in the aqueous extract.

Inhibitor concentration in 0.5M HCl %	CR (mg)/cm ² .min*10 ⁻³ , 30 °C	CR (mg)/cm ² .min*10 ⁻³ , 60 °C
0.0	5.990	3.078
0.01	4.893	2.366
0.02	4.687	1.917
0.03	4.186	1.722
0.04	3.802	1.211
0.05	1.690	1.093

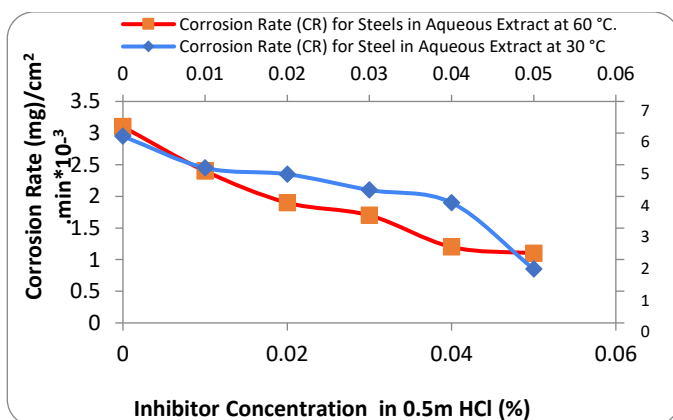


Fig (2): weight loss and corrosion rate calculations for the aqueous extract at both temperatures 30 and 60 °C.

Table 6 and 7 show the Inhibition Efficiency (IE %) at temperatures of 30 and 60 °C for both the alcoholic and aqueous

extracts respectively. As the concentration of the extract in solution is raised, the results demonstrate a continuous increase in inhibitory efficiency [32]. At the maximum concentration for (ZSC), the inhibitory efficiency was recorded as 96.1 % at 60 °C, while at the optimal concentration, which is recorded as 5% concentration for (ZSC), the efficiency appeared to peak at roughly 94 % at 30 °C. These outcomes demonstrate that both extracts have a respectable level of corrosion-inhibiting potential. These values exceed those that were previously reported [33]. In that study, the highest values for 30 and 50 °C were 81 % and 73 %, respectively. The findings demonstrated that the damping efficiency rises with temperature and heat, indicating that the inhibitor has heat stability. The maximum inhibitory efficiency was calculated at a concentration of 5 %, reaching a value of 96 % at a temperature of 60 °C. The rate of wear and efficiency must decrease as the temperature rises in order for this to happen [34]. The efficiency of the inhibitor's adsorption action increases with temperature. This is so that corrosion on the metal surface is prevented by the inhibitor's adsorption, which serves as a protective layer. It is highlighted in the current experiment that the inhibitor's efficiency is stronger at 60 °C when employing both alcoholic and aqueous extracts.

Table 6: inhibitory efficiency of the alcoholic extract at temperatures of 30 and 60 °C.

Inhibitor concentration in 0.5M HCl %	IE %, at 30 °C	IE % at 60 °C
0.0	0.00	0.00
0.01	84.17	88.79
0.02	85.27	91.59
0.03	88.38	92.26
0.04	91.95	94.51
0.05	94.04	96.09

Table 7: Inhibitor efficiency calculations for the aqueous extract at temperatures 30 and 60 °C.

Inhibitor concentration in 0.5M HCl %	IE %, at 30 °C	IE % at 60 °C
0.0	0.00	0.00
0.01	64.56	71.84
0.02	71.01	78.36
0.03	72.27	82.47
0.04	75.23	84.25
0.05	77.51	88.93

3.2. Adsorption Isotherms:

The forces that control adsorption in general include covalent bonding, electrostatic attraction, hydrogen bonding, non-polar interactions between the adsorbed species, lateral associative interaction, solvation, and desolvation [35]. Adsorption of the inhibitor molecules is primarily influenced by the type, charge, and electronic properties of the metal surface as well as the electrochemical potential at the solution interface and temperature-dependent ionic species adsorption from the solvent. Since organic inhibitors are employed to prevent corrosion, extract molecules that have been adsorbed onto metal surfaces produce protective films as a result. To establish that adsorption is in fact the inhibitory mechanism, isotherm equations were applied [36, 37, and 38]. In order to comprehend the interface between the inhibitor and carbon steel surface, two distinct adsorption isotherms were examined. Tested isotherms included Temkin and Freundlich. In addition to being straightforward, these two adsorption isotherms make it simple to use their parameters to characterize a corrosion inhibition system. The model that best fits the experimental findings was identified using the linear regression coefficient of determination (R^2).

3.2.1. Langmuir Adsorption Isotherm

The adsorptive capacity of various adsorbents is measured and compared using the Langmuir isotherm, which characterises gas-solid phase adsorption [39]. The link between a material's surface covering and concentration of inhibitors is described by the Langmuir isotherm and it is expressed as: [40]

$$K_{ads} = \frac{\theta}{1-\theta} \cdot I_{inh} \quad (3) [40]$$

The values of the adsorption constant "K", at each inhibitor concentration, were calculated for both temperatures. The obtained values of the adsorption constant were used to determine Gibb's free adsorption energy (Δ) using the following equation [32]:

$$\Delta G^{\circ}_{ads} = - R T \ln (55.5 \times K_{ads}) \quad (4) [41]$$

where R is the universal constant "8.314 J mol⁻¹ K⁻¹" and "T" is the absolute temperature of "30 and 60 °C" and the constant value of "55.5" is the concentration of water in solution in mole.

The negative ΔG°_{ads} comply adsorption process on a surface in general with the spontaneity of the adsorption system and the stability of the adsorbed layer on the metal surface. ΔG°_{ads} values of around -20 kJ mol⁻¹ or lower are well known to be associated with the physisorption phenomenon, where the electrostatic interaction between the charged molecule and the charged metal is assembled [33, 42]. However, when these values are around -40 kJ mol⁻¹ or higher, they are associated with the phenomenon of chemisorption [43].

This explains that the degree of surface coverage plays a crucial role in improving the adsorption process in corrosion media. As shown in Figures 3 and 4, a perfectly linear plot was obtained with regression constant (R^2) = (0.97754) at an inhibitor temperature of 30 °C and (0.9653) at a temperature of 60 °C.

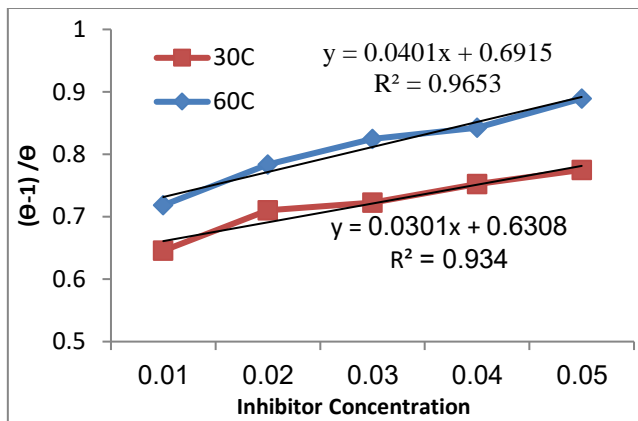


Fig 3: Langmuir Adsorption Isotherm of the alcoholic extract at both temperatures 30 and 60 °C.

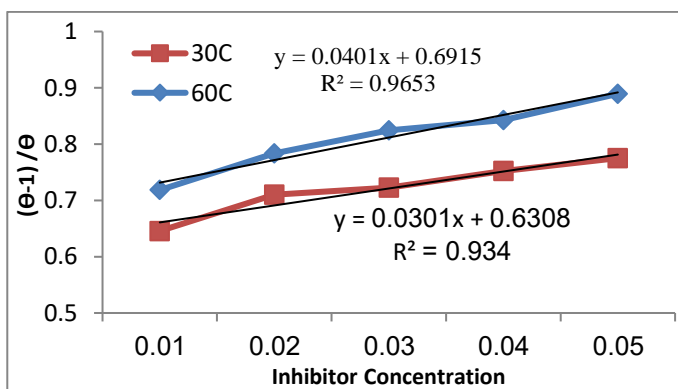


Fig 4: Langmuir Adsorption Isotherm of the aqueous extract at both temperatures 30 and 60 °C.

3.2.2. Temkin Adsorption Isotherm:

The Temkin model of isotherm adsorption contains a factor which explicitly takes into account the interactions of ions of the aqueous solution and the membrane (adsorbent - adsorbate). The surface coverage values for "ZSC" were fitted into the Temkin adsorption isotherm model, which has the form [44]:

$$\text{Exp} (-2a \Theta) = K \cdot I \quad (5)[44]$$

Temkin isotherm was represented by a linear plot "Θ" versus the logarithm of inhibitor concentration "log I". According to Temkin equation (4) This indicates that it has the strongest adherence to Temkin adsorption isotherm compared with the same inhibitor applied at a temperature of 30 °C. At 60 °C it has a relatively stronger adherence to Langmuir adsorption isotherm. The plot of (log θ) vs. (log I) is typical of Freundlich adsorption isotherm at 30 and 60 °C.

As shown in Figure (5), it was found to be the best fit among these four adsorption isotherms, a straight line with ($R^2 = 0.9769$) at temperatures 30°C, ($R^2=0.981$) for inhibitor temperature at 60°C where the value is closer to unity of the alcoholic extract.

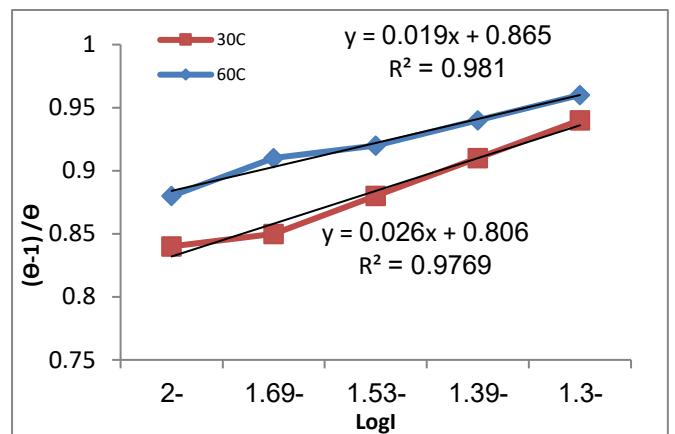


Fig 5: Temkin Adsorption Isotherm of the alcoholic extract at temperatures of 30 and 60 °C.

As shown in Figure (6) a straight line with ($R^2 = 0.9659$) at temperatures 30°C of aqueous extract, as (R^2) for inhibitor temperature where the value is more close to unity.

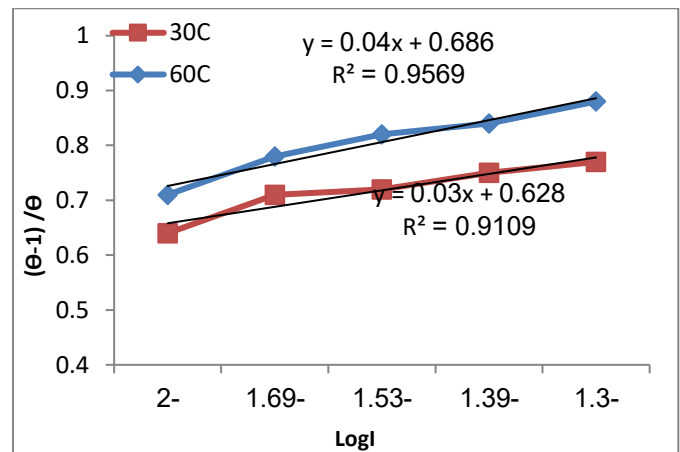


Fig 6: Temkin Adsorption Isotherm of the aqueous extract at temperatures of 30 and 60 °C.

3.2.3. Freundlich Adsorption Isotherm:

The Freundlich model of isotherm adsorption has been chosen fourthly to evaluate the adsorption potential of the adsorbent and adsorbed solution. The Freundlich isotherm is given by [45]:

$$\text{Log } \theta = \text{log } K_{ads} + n \text{ log } I \quad (6)[45]$$

Where K_{ads} is the adsorption equilibrium constant, n is the interaction parameter and I is the inhibitor concentration.

As shown in Figures (7) and (8) a perfectly linear plot was obtained with regression constant (R^2) (0.9781) at an inhibitor temperature of 30 °C and (0.9558) at a temperature of 60 °C.

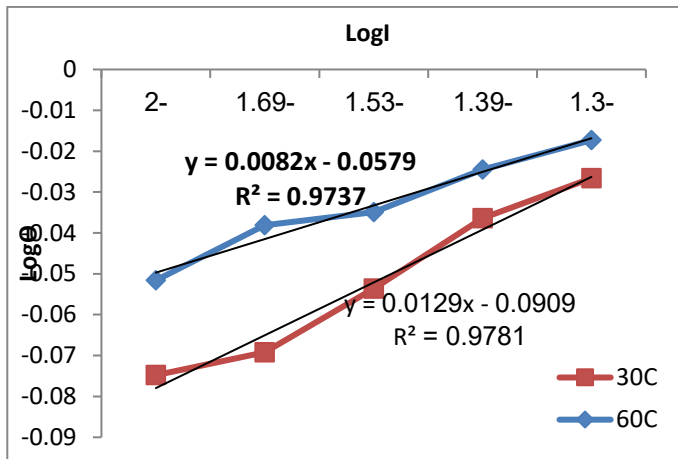


Fig 7: Freundlich Adsorption Isotherm of the alcoholic extract at temperatures of 30, 60 °C.

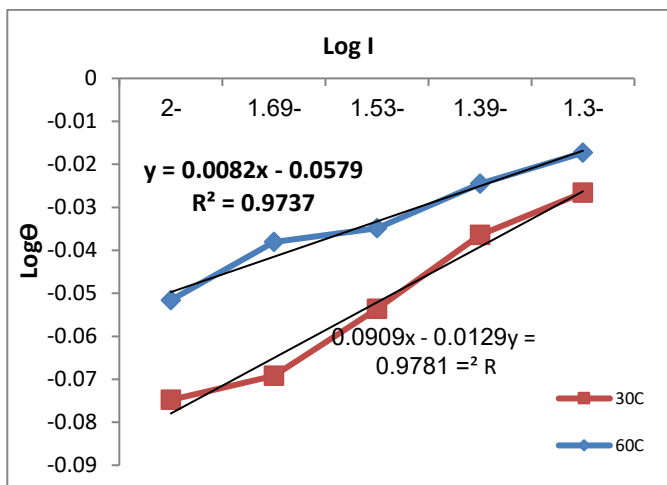


Fig 8: Freundlich Adsorption Isotherm of the aqueous extract at temperatures of 30 and 60 °C

4. Conclusions

Based on these results, it is possible to draw the following conclusions:

1. ZSC Christi leaves are an ecologically sound, reasonably priced, easily accessible, renewable source of materials and inhibitors.
2. A medium steel bar could experience less corrosion when exposed to aqueous and alcoholic extract. The solution with the highest concentration of ZSC Christi produced the greatest results.
3. Medium steel bar weight reduction was observed, measured, and properly recorded. With increasing concentrations of the leaf extract from ZSC, it was discovered that the rate of steel bar corrosion in the HCl solution decreased.
4. The concentration of the extract determines its inhibitory efficiency, which rises with increasing extract concentration in the acidic medium.
5. Almost all of the adsorption isotherms models that are currently in use were followed by the adsorption of the inhibitor of alcoholic and aqueous extracts.
6. The general finding is that "Alcoholic extract" will inhibit corrosion more successfully at 60°C than it will at 30°C.

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