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Inhibition of Carbon Steel Corrosion in 0.5 N Sulphuric Acid Using Ziziphus Spina-Christi Leaf Extract

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ABSTRACT

In the present work, we report the performance of an aqueous extract obtained from leaves of the *Ziziphus spina-christi* as a green and sustainable corrosion inhibitor for carbon steel in 0.5 N sulphuric acid solution. The experimental strategy was based on the influence of the corrosion rate due to extract concentration (v/v %) (0.4 % to 20 %) and temperature (20 to 60 °C), which was evaluated from the mass loss method. An increase in the concentration of the extract proportionally enhanced the inhibition efficiency up to 91.7% at 20% concentration. From kinetic and thermodynamic standpoints, the extract resulted in raised values of activation energy (ΔE_a^*) and activation enthalpy (ΔH_a^*), as well as the activation Gibbs free energy (ΔG_a^*), implying an energy barrier increases for the corrosion process and the formation of a higher energy and less favorable transition state. In addition to this, negative values of the activation entropy ΔS_a^* suggested the production of a very ordered, electrically insulating and protective layer of adsorbed extract molecules, which isolates the metal surface from the corrosive environment.

تثبيط تآكل الفولاذ الكربوني في حمض الكبريتيك باستخدام مستخلص أوراق نبات السدر (*Ziziphus spina-Christi*)

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الكلمات المفتاحية:

منحنيات الامتصاص.
مثبطات التآكل.
فقدان الكتلة.
حامض الكبريتيك.
نبات السدر.

الملخص

في هذه الدراسة، نقدم تقريراً عن أداء مركب طبيعي مستخلص من أوراق نبات السدر (*Ziziphus spina-christi*) باعتباره مانع تآكل صديق للبيئة ومستداماً للفولاذ الكربوني في محلول حمض الكبريتيك بتركيز 0.5 عيارى. استندت الاستراتيجية التجريبية إلى تقييم تأثير معدل التآكل الناتج عن تركيز المستخلص (حجم/حجم) (0.4 % إلى 20 %) ودرجة الحرارة (20 إلى 60 درجة مئوية) باستخدام طريقة فقدان الكتلة. أدت الزيادة في تركيز المستخلص إلى تحسين كفاءة التثبيط بشكل متناسب حتى 91.7 % عند تركيز 20 %. من وجهة نظر الحركية والديناميكا الحرارية، أدى المستخلص إلى ارتفاع قيم طاقة التنشيط (ΔE_a^*) والإنثالبي التنشيطي (ΔH_a^*)، وكذلك طاقة جيبس الحرة للتنشيط (ΔG_a^*)، مما يشير إلى زيادة حاجز الطاقة لعملية التآكل وتشكيل حالة انتقالية أقل توقعاً. بالإضافة إلى ذلك، تشير القيم السلبية لانتروبيا التنشيط ΔS_a^* إلى تكوين طبقة منظمة للغاية وعازلة للكهرباء ووقائية من جزيئات المستخلص الممتصة، والتي تعزل سطح المعدن بكفاءة عن البيئة المسببة للتآكل.

1. Introduction

Due to its good mechanical properties and low cost, carbon steel is

among the most used metallic materials in industry. But, in an acidic environment, it is very prone to rusting [1, 2]. A lot of focus has been

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on the formulation of high-performance, cost-effective and environmentally friendly steel corrosion inhibitors in recent years. Corrosion inhibitors have been accepted as a good and realistic approach to control corrosion of these [3]. Corrosion inhibitors are chemical compounds which have the ability to decrease the rate of corrosion of a metal surface when they are present in the environment, acting on the metal or when they adsorb onto the metal surface [4, 5]. Most corrosion inhibitors are either inorganic or organic compounds with heteroatoms that play a crucial role in their efficiency as corrosion protection agents [6-11].

However, most of the synthetic corrosion inhibitors are related to toxic nature and have potential risk to human being and the environment. Therefore, since the last decade, there has been a growing interest in the use of natural plant extracts as green corrosion inhibitors to protect metals and alloys from aqueous media-induced corrosion [10, 12-16]. Corrosion inhibition activity of these plant extracts was probably due to the presence of some bioactive compounds like tannins and saponins, etc., which act as inhibitors and are considered to adsorb physically on the metal surface, and this leads to a decrease in the rate of corrosion [15-19]. In this study, *Ziziphus spina-christi*, known as Sidr and Nabeq in common Arabic, was used for its ability as a green corrosion inhibitor. *Ziziphus spina-christi* is an extensive warm-temperate and subtropical deciduous tree species. Origin: It is native to North Africa, Southern Europe, the Mediterranean, the Middle East and to some extent in South and East Asia [20]. Traditionally, *Ziziphus spina-christi* was used as a medicinal plant to treat a few diseases like fever, pains, wounds, ulcers, inflammatory diseases, asthma, dandruff and eye diseases. Also, in recent research, it was found that it had a lot of pharmacological activities like antibacterial, antifungal, antioxidant, antihyperglycemic, and antinociceptive activities [21].

From a phytochemical point of view, *Ziziphus spina-christi* leaves contain a plethora of multi-bioactive compounds such as alkaloids, sterols like β -sitosterol, flavonoids, triterpenoids, saponins and sapogenins. These components are known to have various biological activities and may be used as antibacterial, antioxidant, antimicrobial, and anti-inflammatory agents [22]. In addition, these active compounds contribute to the overall medicinal value of the plant and are responsible for its potential use as a source of green corrosion inhibitors [12]. Various solvents were employed by the investigators to extract from the *Ziziphus spina-christi* plant, and the resulting extracts were used as a corrosion inhibitor on two steels, carbon steel and mild steel [12, 23-26]. Inhibition of Carbon Steel Corrosion in 1.0 M HCl by the Ethanolic Extract of *Ziziphus spina-christi* Leaves was studied by Sara Lahmady et al. The ethanoic extract of leaves of *Ziziphus spina-christi* was used as a green inhibitor to prevent the corrosion of carbon steel in 1.0 M HCl [12].

Another research conducted by Sarah Z. Al-Ashoor et al. The methanolic extract of *Ziziphus spina-christi* leaves was prepared from the leaves of *Ziziphus spina-christi*, and it was applied as a green corrosion inhibitor for carbon steel in 0.1 M HCl [23]. The synergistic behavior of zinc ions with aqueous extract of *Ziziphus spina-christi* leaves was studied by Zahra Shahryari and Khalil Gheisari for the corrosion of mild steel in a saline medium of 3.5 wt % NaCl [24]. Natarajan Rajamohan et al studied the corrosion protection of carbon steel in HCl solution using the *Ziziphus spina-christi* extract with ethyl acetate solvent [25, 26].



Fig. 1: *Ziziphus spina-christi* plant

The selection of *Ziziphus spina-christi* is scientifically motivated by its rich content of alkaloids, flavonoids, and tannins. These molecules possess heteroatoms (oxygen and nitrogen) and conjugated double bonds, which act as active adsorption centers. These centers facilitate the formation of a stable coordinate bond with the metal surface, creating a protective barrier that inhibits the anodic and cathodic reactions in acidic media.

While previous research has extensively evaluated *Ziziphus* extracts in hydrochloric acid (HCl), there is a notable lack of information regarding their efficacy in sulfuric acid (H_2SO_4). Sulfuric acid presents a different corrosive mechanism and is widely used in industrial descaling. Therefore, this study uniquely addresses this gap by investigating, for the first time, the inhibitory performance of *Ziziphus spina-christi* aqueous extract in 0.5 N H_2SO_4 , providing new insights into its thermodynamic stability and adsorption behavior under these specific industrial conditions.

2. Experiments and Methods

2.1 Chemicals and Materials

Regarding the chemical reagents, all substances were used as received, without undergoing further purification processes. Acetone (CH_3COCH_3 , 99%) was used for cleaning the carbon steel samples. Sulfuric acid (H_2SO_4 , 98%) was the corrosive agent used. The accurate handling and preparation of these chemicals were crucial to ensure the consistency of the test environment and the validity of the corrosion rate measurements.

Carbon steel samples were obtained from the Iron Factory at Misurata. The samples consisted of carbon steel rods composed of iron with some additional elements (C, Si, Mn, P, S) in varying proportions, as shown in Table 1. The rods were cut into cylindrical specimens with a length of 4 cm and a diameter of 1 cm. The surfaces of the specimens were polished using emery paper of different grades (120–1200), then washed with distilled water followed by acetone.

Table 1: Chemical composition of iron samples

Element	Fe	C	Si	Mn	P	S
wt%	98.606	0.30	0.23	0.57	0.011	0.0004

2.2 Instruments and Chemicals Used

A range of laboratory instruments and analytical tools were employed throughout the experimental procedures to ensure accuracy and reliability of the results. These included a micrometre, which was used to measure the diameter of the cylindrical iron samples with high precision, and a vernier calliper, which was essential for accurately measuring the lengths of the specimens. A highly sensitive analytical balance was utilized to determine the initial and final weights of the metal samples before and after immersion in the test solutions, allowing for the precise calculation of weight loss due to corrosion. A thermostatically controlled water bath (Clifton Bath, England) was used to maintain consistent temperatures during the immersion period, ensuring controlled thermal conditions across different experimental runs.

2.3 Solution Preparation

2.3.1 Plants Aqueous Extracts

In this article, the leaves of the *Ziziphus spina-christi* were collected from the city of Ghat in the southwestern part of Libya. They were washed several times with water to separate the dust and other impurities. Later, the leaves were dried at room temperature and finally powdered. The preparation of the plant extracts was carried out by macerating 5 g of plant powder in 200 mL of boiling distilled water; the mixture was left at room temperature for one full day with constant stirring to promote the extraction of active compounds into the aqueous solution. Then the extract was filtered through filter paper, and a filtrate was obtained and used as a stock solution for preparing different concentrations by diluting with water to obtain a series of (v/v) % of 0.4 %, 1%, 2 %, 3 %, 4 %, 5 %, 10 % and 20%, and the respective concentrations utilized in the corrosion studies were derived therefrom.



Fig. 2: Preparation of the Aqueous Extracts of *Ziziphus spina-christi* Leaf

2.3.2 Corrosive Media

In order to perform a corrosion inhibition test, a number of solutions were prepared with known concentrations. A stock solution of sulfuric acid (H_2SO_4) was initially prepared at 5 N concentration and then diluted to a working concentration of 0.5 N, used as the corrosive medium for the entire study. A range of concentrations of plant extract, from 0.4% to 20 (v/v%), was obtained. The corrosion studies solution was prepared. The test solutions for the corrosion studies were prepared in 50 mL volumetric flasks by dissolving a known volume of the prepared plant extract to the desired concentration range in 5 mL of the 5 N sulfuric acid solution. Finally, distilled water was added to each flask to make up the volume. The identical methodology was utilized for the blank solution that served as the control.

2.4 Weight Loss Method

The weight loss method is one of the most straightforward and widely used techniques for evaluating corrosion behavior. It involves determining the corrosion rate based on the mass loss of a metal specimen over a specific period. In this study, the metal samples were first accurately weighed before immersion in the corrosive medium, and this initial mass was recorded as W_1 . The specimens were then immersed in the test solution for a fixed duration of 180 minutes. After exposure, the samples were carefully removed, rinsed thoroughly with distilled water to eliminate residual acid or inhibitor, dried at room temperature without applying heat, and then reweighed to determine the final mass, W_2 . The weight loss due to corrosion was calculated as the difference between the initial and final masses ($W = W_1 - W_2$).

The corrosion rate (R) was determined using the equation:

$$R = W / (A \cdot t) \quad (1)$$

where W is the weight loss in grams, A is the surface area of the specimen in cm^2 , and t is the exposure time in hours. The efficiency of corrosion inhibition ($\text{Inh} \%$) was evaluated using the formula:

$$\text{Inh} \% = [1 - (R_{\text{inh}} / R_0)] \times 100 \quad (2)$$

where R_{inh} is the corrosion rate in the presence of the inhibitor, and R_0 is the rate in its absence. Furthermore, surface coverage (θ), which provides insight into the extent of inhibitor adsorption on the metal surface, was calculated using the relation:

$$\theta = 1 - (R_{\text{inh}} / R_0) \quad (3)$$

These parameters collectively provide a comprehensive assessment of the corrosion process and the effectiveness of the plant-based inhibitors studied. In this work, the weight loss method at different temperatures and concentrations was employed to calculate the efficiency and surface coverage, and Langmuir adsorption isotherms were employed to investigate the adsorption behavior onto the surface of carbon steel.

2.5 Experimental Procedure

The experimental process was performed according to the typical method of weight loss to examine the corrosion rate of carbon steel under the influence of and free of plant-based inhibitors. The metal samples were first polished mechanically with various grades of emery papers from 120 to 1200 grit to achieve a homogeneous and flat

surface. The dimensional length and diameter of the samples were measured, and then, the samples were weighed with an analytical balance to note down the initial mass (W_1). Afterwards, the samples were rinsed with distilled water, soaked in acetone to eliminate organic impurities, and to make sure the surface is well prepared. The test solution (50 mL) was pre-equilibrated at the desired temperature by placing it in a water bath for 10 min before immersion. Once prepared, each metal sample was subsequently dipped in the solution for 180 min. After exposure, the samples were gently taken out, rinsed with distilled water, dried in air and reweighed to obtain the final weight (W_2).

The process was conducted three times in the following results: at first, inhibitor concentration-dependent corrosion rate and inhibition efficiency were investigated in the range of 0.4% to 20 (v/v%) at a constant temperature of 30 °C; second, temperature-dependent (20, 30, 40, 50, and 60 °C) corrosion rate was studied in the absence and presence of inhibitors to investigate the effect of temperature on the rate of corrosion. and third, Langmuir adsorption isotherms was employed to investigate the adsorption behaviour.

3. Results and Discussion

3.1 Corrosion rates and inhibition efficiencies

Corrosion rates and inhibition efficiencies were determined at various inhibitor concentrations for the metal under study to investigate the influence of inhibitor concentration on the rate of corrosion. The calculated values were computed and tabulated in Tab. 2, and the dependence of corrosion rate (CR) and inhibition performance ($\text{Inh} \%$) on the inhibitor concentration was presented in Figure 3.

Table 2: corrosion characteristics of *Ziziphus spina-christi* extract

C (v/v%)	CR*10 ⁻⁴	Inh%
0	19.179	-
0.4	7.167	47.256
1	6.08	55.255
2	4.949	63.577
3	4.529	66.669
4	3.359	75.283
5	2.277	83.244
10	1.895	86.054
15	1.675	87.670
20	1.130	91.683

From the data in Table 2, it can be seen that the corrosion rate decreased greatly with inhibitor concentration while $\text{Inh} \%$ kept increasing. A distinct slowing in the growth rate of inhibition efficiency was observed at concentrations exceeding ca. 5% (v/v). Before this point, the inhibition efficiency increased sharply, whereas beyond this level, the efficiency continued to rise but at a more gradual until it reached its maximum of 91.7% at 20% concentration. This implies a strong adsorption of inhibitor molecules on the metal surface, which contributes to inhibiting the metal dissolution process and/or the formation of a protective film on the metal surface [27, 28]. It can be observed in Figure 3 that the corrosion rate rapidly reduces with increasing extract concentration. This behaviour indicates that the active metal surface was largely covered and that led to the stabilization of the protective film [29].

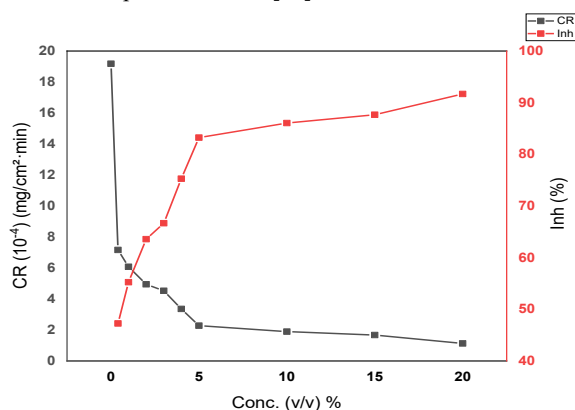


Fig. 3: Effect of inhibitor concentration on corrosion rate and efficiency of inhibition

3.2 Effect of temperature

The influence of temperature on the corrosion of iron in acidic medium with and without the inhibitor was studied by the weight loss method. The activation energy (ΔE_a^*), activation enthalpy (ΔH_a^*) and the activation entropy (ΔS_a^*) were calculated by using the Arrhenius and the transition state equations [30]. The Arrhenius equation is given by:

$$\ln R = \frac{-\Delta E_a^*}{R} + \ln A \quad (4)$$

where R is the corrosion rate, T is the absolute temperature (K), R is the universal gas constant, and A is the Arrhenius pre-exponential factor. The graph of $\ln R$ against $1/T$ (Figure 4) was linear, revealing that the corrosion process also follows the Arrhenius equation. The inhibited solution exhibited a larger slope than that of the blank, resulting in the enhancement of activation energy, which was attributed to the adsorption of inhibitor molecules on the iron surface [30].

The activation enthalpy and entropy were calculated from the transition state relation using the transition state equation:

$$\ln\left(\frac{R}{T}\right) = \left[\ln\left(\frac{R}{N^0 h}\right) + \frac{\Delta S_a^*}{R}\right] - \frac{\Delta H_a^*}{R} \quad (5)$$

where: h is Planck's constant, N^0 is Avogadro's number.

The plots of $\ln(R/T)$ against $1/T$ for Figure 5 also yielded straight lines from which ΔH_a^* and ΔS_a^* were calculated. From the regression analysis of these plots, the activation enthalpy (ΔH_a^*) was determined from the slope, which corresponds to $(-\frac{\Delta H_a^*}{R})$. Meanwhile, the activation entropy was extracted from the y-intercept of the lines, based on the term $\left[\ln\left(\frac{R}{N^0 h}\right) + \frac{\Delta S_a^*}{R}\right]$ in the transition state equation. The positive values of ΔH_a^* imply that the dissolution process is endothermic, and the negative values of ΔS_a^* indicate a reduction in disorder in the formation of the activated complex [31, 32].

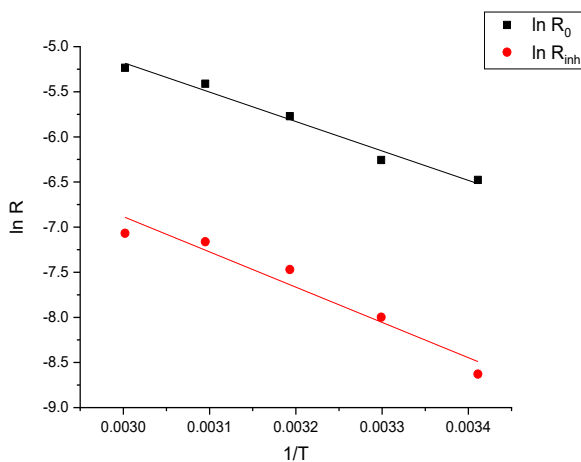


Fig. 4: Arrhenius Plots of $\ln R$ versus $1/T$ for Iron Corrosion in the Absence and Presence of the Inhibitor

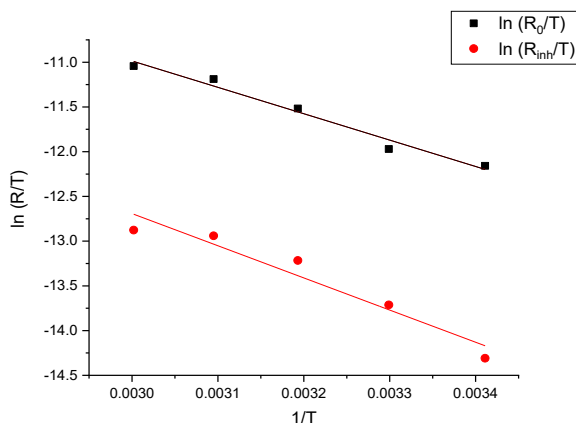


Fig. 5: Transition State Plots of $\ln(R/T)$ versus $1/T$ for Iron Corrosion in the Absence and Presence of the Inhibitor

The activation Gibbs free energy (ΔG_a^*) was subsequently calculated at each temperature using the fundamental thermodynamic relation:

$$\Delta G_a^* = \Delta H_a^* - T \Delta S_a^* \quad (6)$$

Where T is the absolute temperature.

Thermodynamic activation parameters for the corrosion of iron in acidic medium without and with *Ziziphus spina-christi* extract are given in Table 3.

Table 3: Thermodynamic Activation Parameters for Iron Corrosion in Acidic Medium in the Absence and Presence of *Ziziphus spina-christi* Extract

Thermodynamic parameters	Blank	<i>Ziziphus spina-christi</i>
ΔE_a^* KJ/mol	+27.12	+32.49
ΔH_a^* KJ/mol	+24.52	+29.89
ΔS_a^* J/mol . K	-215.32	-213.39
ΔG_a^* KJ/mol at 30 °C	+89.79	+94.56

Thermodynamic and kinetic analyses of carbon steel corrosion in sulfuric acid showed that *Ziziphus spina-christi* extract introduced considerable alterations to the activation parameters. The ΔE_a^* and ΔH_a^* values of the activation energy and enthalpy of activation increased positively according to the Arrhenius and transition-state equations, respectively, where the ΔE_a^* value increased from 27.12 kJ mol⁻¹ for the blank solution to 32.49 kJ mol⁻¹ with the addition of the extract, and the ΔH_a^* value increased from 24.52 to 29.89 kJ mol⁻¹. These results verify that the endothermic dissolution of the metal is faced with a higher energy barrier when more layers of inhibitor are adsorbed on the metal surface [33]. Correspondingly, at 30 °C, the Gibbs free energy of activation ΔG_a^* increased from 89.79 to 94.56 kJ mol⁻¹, which means that the formation of the activated complex for corrosion was further disfavored with the adsorption of extract molecules and the increasing of a stable protective layer on the metal surface [34]. Moreover, the negative values of the activation entropy (ΔS_a^*) are also in accord with this interpretation, which indicates that the activated complex at the transition state is more ordered and less random, and it is probably made reflecting the condensed and organized structure of the inhibitor molecules at the metal/solution interface [35].

3.3. Adsorption Parameters

To better understand how the *Ziziphus spina-christi* extract interacts with the metal surface, we investigated its adsorption behavior using the Langmuir isotherm model at 30 °C. This model is widely recognized for describing the formation of a protective monolayer where inhibitor molecules occupy specific active sites on a homogeneous surface. The Langmuir relationship is expressed as follows:

$$\frac{\theta}{1-\theta} = KC \quad (7)$$

where C is the concentration of the extract and θ is the surface coverage derived from our weight loss measurements [31].

As shown in Figure 6, the experimental data at 303 K yielded a strong linear fit with a correlation coefficient (R^2) of 0.9523. This high linearity confirms that the adsorption of the plant's phytochemicals on the carbon steel surface follows the Langmuir model. From the intercept of this plot, we determined the adsorption equilibrium constant (K_{ads}) to be 0.4819, which reflects the binding strength between the extract molecules and the metal. Furthermore, we calculated the standard Gibbs free energy of adsorption (ΔG_{ads}^0) to evaluate the spontaneity and nature of this process:

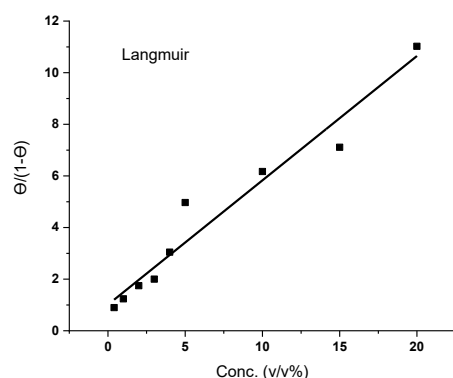


Fig. 6: Langmuir adsorption isotherm plot

$$(\Delta G_{\text{ads}}^0 = -RT \ln(55.5 K_{\text{ads}})) \quad (8)$$

The resulting value of -8.28 kJ/mol provides two key insights into the mechanism:

1. Spontaneity: The negative sign confirms that the adsorption of the extract molecules onto the steel surface occurs spontaneously.
2. Adsorption Type: The magnitude of $(\Delta G_{\text{ads}}^0)$ being less negative than the -20 kJ/mol threshold indicates that the process is predominantly governed by physical adsorption (physisorption).

This suggests that the inhibitor molecules are held to the surface through relatively weak electrostatic interactions, forming a stable protective film that effectively shields the metal from the aggressive sulfuric acid environment. These findings are consistent with the increase in activation energy discussed in the kinetic section, confirming the formation of a physical barrier that hinders the corrosion reaction.

4. Conclusion

The present work reveals that jujube extract (*Ziziphus spina-christi*) is a very good green and sustainable corrosion inhibitor for carbon steel in strong acid media. From these experimental results, the following important conclusions can be drawn:

1. The inhibitor exhibited the highest efficiency of 91.7%, which makes it a potential candidate for replacing toxic chemical inhibitors in industry applications.
2. Adsorption studies confirmed that the *Ziziphus spina-christi* extract follows the Langmuir isotherm model, forming a protective monolayer on the carbon steel surface. The calculated standard Gibbs free energy ($\Delta G_{\text{ads}}^0 = -8.28$ kJ/mol) indicates that the inhibition is predominantly governed by spontaneous physical adsorption (physisorption). This process involves the formation of a stable protective film through electrostatic interactions, which effectively increases the activation energy barrier of the corrosion reaction.
3. Thermodynamic analysis confirmed that the dissolution process is endothermic, as indicated by the positive values of activation enthalpy. The negative values of activation entropy reflect the formation of a stable, highly ordered barrier layer at the metal–solution interface.
4. This study suggests that this extract can be utilized as a cost-effective and environmentally friendly material for chemical cleaning and acid scale removal in industrial plants. However, for practical industrial applications, it is recommended to further investigate the long-term thermal stability and degradation kinetics of the extract's bioactive constituents at elevated temperatures to ensure sustained performance during extended cleaning cycles.

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