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Research Article

Experimental and Theoretical Study on the Corrosion Inhibition of Mild Steel in Acidic Medium Using Ethanolic Extract of *ziziphus spina-christi* **leaves**

David E. Arthur^{1*}, Joseph C. Akan¹, Ugwoke Augustina Oyibo², Muhammad Bakura¹, Adebiyi Adedayo Babafemi³ ¹Department of Pure and Applied Chemistry, University of Maiduguri P.M.B 1069, Borno-Nigeria 2 Department of Chemistry, Nigerian Police Academy Wudil, Kano-Nigeria ³Sheda Science and Technology Complex Abuja, Nigeria ***Corresponding author's Email**: eadavid@unimaid.edu.ng, doi.org/10.55639/607.02010013

ARTICLE INFO: ABSTRACT

Keywords: Adsorption, Corrosion, Inhibition, Mild Steel, *Ziziphus spina-christi* This study investigates the corrosion inhibition of mild steel in 0.3 M HCl using ethanolic extract of *Ziziphus spina-christi* leaves. Using the weight loss technique, the extract demonstrated good corrosion inhibition efficiency, ranging from 56.13% to 64.24% at 303 K. The corrosion rate decreased as the inhibitor concentration increased, with surface coverage and inhibitor efficiency improving as well. The adsorption of the extract on the mild steel surface was exothermic, spontaneous, and aligned with physical adsorption. The adsorption followed the Langmuir isotherm.

Corresponding author: David E. Arthur **Email:** eadavid@unimaid.edu.ng Department of Pure and Applied Chemistry, University of Maiduguri P.M.B 1069, Borno-Nigeria

INTRODUCTION

The spontaneous oxidation of metal is termed corrosion (Rani & Bai, 2011). That is, corrosion is the deterioration or destruction of metals and alloys in the presence of an environment by chemical or electrochemical means. The medium in which the metal undergoes corrosion is termed a corrosive or aggressive medium. Corrosion products are chemical compounds containing the metal in its oxidized form; with the exception of gold and platinum, all other

metals corrode and transform themselves into substances similar to the mineral ores from which they are extracted (Karthik *et al.,* 2015). Corrosion phenomena of materials are very complicated in that corrosion reactions and/or processes depend largely upon the material and environmental systems. The damage to materials caused by the corrosion phenomena has led to the loss in energy and resources, the instability of human life, the decline in the reliability of the infrastructure, and so on. To overcome them,

recent advances in the understanding of corrosion phenomena and their mechanisms have been introduced (Nishimura *et al.,* 2012). Therefore, the prevention of this undesired phenomenon could be a practical solution to investigate this problem. The use of some chemical inhibitors has been limited because of some reasons namely that their synthesis is often expensive and they can be toxic and hazardous for human beings and the environment as well (Mas *et al.*, 2020). Acidic media, which are widely used in industrial acid cleaning, acid descaling, and acid pickling, require the use of corrosion inhibitors to restrain corrosion on metallic materials. The synthetic organic and inorganic corrosion inhibitors are effective but are harmful to humans and the environment. Hence, the study of corrosion prevention, particularly on mild steel using plant-based corrosion inhibitors, is of tremendous interest as plant extracts are found in abundance, inexpensive, less or nontoxic, biodegradable, and biocompatible (Umoren *et al.,* 2011). The study of *Ziziphus spina-christi* as a corrosion inhibitor in acidic media offers novel insights into its potential as a sustainable alternative to synthetic inhibitors. Recent studies have increasingly highlighted plant-based corrosion inhibitors due to their eco-friendliness, availability, and biodegradability (Migahed & El-Sherif, 2021; Nawaz, Sayed, & Zubair, 2021). *Ziziphus spina-christi*, a plant with diverse bioactive compounds, stands out due to its ability to inhibit corrosion effectively in hydrochloric acid solutions, as demonstrated in this study. Unlike conventional inhibitors, the extract's non-toxic nature makes it a promising candidate for environmentally conscious applications in industrial settings (Singh, Mehta, & Mittal, 2020). Furthermore, the physical adsorption mechanism, confirmed by Langmuir isotherm fitting, emphasizes the unique interaction between the inhibitor and the mild steel surface, making this study a valuable addition to the growing body of research on plant-based corrosion inhibitors.

Several studies have been done on the use of plant extract as an inhibitor for metals against corrosion in different aggressive media. However, not much has been reported on the use of the extract of *Ziziphus spina-christi* leaves as

an inhibitor against the corrosion of mild steel in HCl (Joseph & Vincent, 2012). *Ziziphus spinachristi* is a member of the family Rhamnaceae and comprises 52 genera and 925 species distributed worldwide, particularly across tropical and warm temperate regions. The genus *Ziziphus* consists of approximately 100–170 species of deciduous or evergreen trees and shrubs known for being drought-tolerant and resistant to heat. They are used to treat pulmonary ailments and fevers and to promote the healing of fresh wounds, and for dysentery. The leaves of these plants contain betulinic and ceanothic acids, various flavonoids, saponins, erols, tannins, and triterpenes (Asgarpanah & Haghighat, 2012). Therefore, the present work is aimed at investigating the potential of the ethanolic extract of *Ziziphus spina-christi* leaves as a corrosion inhibitor of mild steel in hydrochloric acid.

2. MATERIALS AND METHODS

Mild steel (MS) specimens containing the composition (wt%): 0.15% C, 0.03% Si, 0.6% Mn, 0.36% P, 0.36% S and 98.5% Fe were used for the study. The sheet was prepared for the size of 2.5 cm length, 2.5 cm breath and 0.4 cm of thickness which were polished with different emery papers ranging from 400 to 1200 grades followed by washing with ethanol, rinsed with acetone and dried at room temperature. All the reagents used for the study were of analytical grade and distilled water was used in this study.

2.2 Preparation of Plant Extract

The aggressive test medium of 0.3 M HCl acid stock solutions were prepared using analyticalgrade 37% of HCl with distilled water. The concentrations of the inhibitors were range from 0.2 - 0.8 g/L for this study.

2.3 Chemical Analysis

Phytochemical analysis of the ethanolic extract of *Ziziphus spina-christi* was carried out according to the method reported by Mohammed et al. (2020).

2.4 Gravimetric Measurements

The gravimetric method was employed as earlier described and reported by Kolo et al. (2016) for a temperature of 303 K. The temperature run of this study was kept constant using a digital thermostatic water bath. In this procedure, five 250 ml beakers were provided and properly labeled. 100 ml each of the prepared Ziziphus

spina-christi leaves stock solution (that is 0.2, 0.4, 0.6, and 0.8 g/L) were transferred into the first set of four beakers. Into the fifth beaker, 100 ml of each uninhibited stock solution (0.3 M HCl) were transferred (that is control or blank). All the beakers containing the test solutions were placed into the thermostated water bath. After the set temperature attained a steady state, the clean, weighed, tagged coupons from the desiccator were introduced and completely immersed into the test solutions in the water bath and suspended with the aid of pegs via the embroidery thread. After 1 hour, the coupons were retrieved from the test solution, cleaned, dried, weighed, and returned back into the test solutions. The processes were repeated for a period of 4 hours, and the differences in the weight loss for this period were taken as the total weight loss.

Calculations for Corrosion Parameters

Total weight loss were calculated by equation (1)

$$
V_i - W_f = W - - - - - - (1)
$$

Where W_i and W_f are the initial weight before immersion and after respectively.

The degree of surface coverage
$$
(\theta)
$$
 is given by equation (2)

$$
\theta = \left(1 - \frac{w_1}{w_2}\right) - - - - - - - - - (2)
$$

Where w_1 and w_2 are the weight losses (g) for metal in the presence and absence of inhibitor in 0.3 M HCl solution.

The inhibition efficiency (%I) for each inhibitor concentration were calculated using equation (3)

$$
\%I = \left(1 - \frac{w_1}{w_2}\right) \times 100 - \dots - \dots - (3)
$$

The corrosion rates for metal corrosion in concentration of the acid were determined for 4 hours immersion period from weigh loss using equation (4).

$$
CR(mmpy) = \frac{87.6w}{DATAaT} - - - - - - (4)
$$

Where w = Total weight loss (mg), D = density of specimen ($g/cm³$), A = Area of specimen (square meter) and T= period of immersion (hour) and 87.6 is a conversion factor.

2.5 Thermodynamic Parameters

The value of activation energy E_a for the inhibited corrosion reaction of the metals were evaluated graphically from the slope by plotting natural logarithm of the corrosion rate (log C_R)

versus 1/T without and with the addition of various concentrations of inhibitor using Arrhenius equation (5).

$$
CR = Ae \frac{-E_a}{RT} - - - - - - - (5)
$$

Taking the logarithm of both side of equation (5) and (6) was obtained

$$
logCR = -\frac{E_a}{2.303RT} + logA - - - - - - (6)
$$

Where C_R is corrosion rate, R the gas constant (8.314 J/mol/K), T is the absolute temperature and A is the Arrhenius constant respectively.

The value of heat of adsorption Q_{ads} were also calculated using equation (7)

$$
Q_{ads} = 2.303R \left[\left(\log \frac{\theta_2}{1 - \theta_2} \right) - \log \left(\frac{\theta_1}{1 - \theta_1} \right) \right] \times \left(\frac{T_2 T_1}{T_2 - T_1} \right) \frac{J}{mol} \quad - \quad - \quad (7) \text{ Oneo } et \text{ al. (2011)}
$$

Where θ_1 and θ_2 are degrees of surface coverage at 303 K.

The standard enthalpy of activation (ΔH^*) and the standard entropy of activation (ΔS^*) were calculated by using the transition state equation (8)

$$
CR = \frac{RT}{Nh} exp^{\frac{\Delta S^*}{R}} exp^{\frac{-\Delta H^*}{RT}} - - - - - (8)
$$

Where h is the plank's constant and N is the Avogadro's number, respectively. A plot of $\log \frac{c\bar{R}}{r}$ verse $\frac{1}{r}$ gave a straight lines with a slope

of $\frac{-\Delta H^*}{2.303R}$ and intercept of $\left[\log\left(\frac{R}{Nh}\right)+\frac{\Delta S^*}{2.303R}\right]$, from which the activation thermodynamics parameters (ΔH^*) and (ΔS^*) were calculated.

2.6 Adsorption Isotherm

Adsorption isotherms are important in understanding the mechanism of inhibition of corrosion reactions the adsorption behavior of the plant extract as a green corrosion inhibitor were tested for the Langmuir, Frumkin, Temkin and Flory-Huggins adsorption isotherm.

3.4.2 Scanning Electron Microscope (SEM)

The mild steel specimen was immersed for 2 hour in 0.9M HCl solution containing optimum concentrations (320 ppm *ZSC*) of inhibitor. After 2 hour, the specimens were taken out and dried. The surface morphology of the mild steel was examined by using the JEOL (JSM 6390) Scanning electronic microscope.

3.0 RESULTS AND DISCUSSION 3.1 Phytochemical Analysis

Active phytochemicals in the crude extracts were identified, and the results are presented in Table 1 below. The results indicated that all the listed phytochemicals screened were present in leaves except anthraquinones. Generally, *Ziziphus spina-christi* leaves are the phytochemically richest part of the plant, containing metabolites like organic acids, phenolic compounds, saponins, alkaloids, flavonoids, terpenoids, carbohydrates, etc. (Abdallah et al., 2014). A bio-guided phytochemical study permits the identification of active compounds from *ZSC* leaves. The physical, chemical, and biological properties of these complex structures are responsible for inhibiting the corrosion of metals and alloys (Asgarpanah & Haghighat, 2012).

Key + indicates the presence of phytochemicals, - indicates the presence of phytochemicals.

3.2 Weight Loss Analysis

The figure below shows the results obtained from weight loss analysis, the results indicated that increase in weight loss with increase in time of immersion.

Figure 1: Plot for weight loss of *ZSC* Leave at 0.3M HCl at 303 K

3.2.2 Weight Loss Analysis 3.2.3 Effect of Inhibitor Concentration

The effect of inhibitor ZSC leave extract was studied at different concentrations (0.20 g/L, 0.40 g/L, 0.60 g/L, and 0.80 g/L) at 0.30 M HCl concentrations at 303 K temperature for the corrosion inhibition of mild steel metal. The result from Figure 1 above indicates that the weight loss of mild steel metal decreases with an increase in inhibitor concentrations and increases with an increase in contact time. The results in Table 2 showed that the corrosion rate (CR) decreases with an increase in the concentration of inhibitors. Moreover, surface coverage $(θ)$ and percentage inhibitor efficiency (% IE) increase with increasing concentrations of the inhibitor (Abdallah *et al.,* 2014).

Table 2: Surface Coverage θ, Percentage Inhibition Efficiency (%IE) of the Leave Extract and Corrosion Rate C_R Obtained from Weight Loss Experiment

Temperature	Inhibitor		$%$ IE	$\mathrm{C_{R}}$
303K	Blank	-	-	0.219
	0.2 g/l	0.5613	56.13	0.183
	0.4 g/l	0.5948	59.48	0.144
	$0.6 \text{ g}/l$	0.6371	63.71	0.126
	$0.8 \text{ g}/1$	0.6424	64.24	0.112

3.3 Thermodynamic/ Adsorption Parameters 3.3.1 Activation Energy and Heat of Adsorption

The activation energies were calculated using the Arrhenius equation, and the heat of adsorption was determined using Equation 7. The results are presented in Table 4. The data indicate that the addition of leaf extracts up to 0.80 g/L in 0.30 M HCl solutions increases the activation energy from 32.856 to 35.958 kJmol⁻¹, while the heat of adsorption changes from -15.62 to -14.28 kJmol⁻¹ for the leaves extract. The calculated Ea for the blank solution is 32.572 kJmol⁻¹. This increase in activation energy is attributed to the greater amounts of extracts, which hinder the reaction process. The Eavalues for the plant extract solutions were found to be greater than those for the blank, suggesting the formation of an adsorption film due to physical electrostatic interactions. The values of heat of adsorption in Table 3 indicate that the adsorption of ethanolic extracts of leaves on the mild steel surface is exothermic (Abdallah *et al.*, 2014; Al-Otaibi *et al.,* 2014).

3.3.2 Enthalpy ΔHα and Entropy ΔSα of Activation

Activation enthalpy $ΔHα$ and entropy $ΔSa$ of the corrosion process were calculated at various inhibitor concentrations and the results were

presented in Table 4. The values of activation enthalpy in 0.00 g/L and 0.80 g/L inhibitors (uninhibited, leaves and stem extract) in 0.30 M HCl solution are 12.845, 14.462 and 14.267 kJmol-1 respectively, which are all positive in the presence and absence of an inhibitor and the inhibited values are greater than uninhibited values, these reflect that the dissolution process is endothermic (Abdallah *et al*., 2014), while the values of an uninhibited entropies of adsorption in 0.0 g/L are 1.579, for the leave extract values from $0.20 - 0.80$ g/L are 1.572 - 1.530, which indicates that activated complex in the ratedetermining step represents association rather than dissociation, which means that there is reduction is disorderliness on going from reactant to activated complex. Generally, an exothermic adsorption process suggests either physisorption or chemisorption while an endothermic process is attributed to chemisorption only. Generally, enthalpy values up to 70 kJmol-1 are related to the electrostatic interactions between charged molecules and charged metal (physisorption) while those around 100 kJmol-1 or higher are attributed to chemisorption. In this case of *ZSC* leave, the absolute values of enthalpy are relatively low, approaching those typical of physisorption and the values of ΔGα in the presence of *ZSC* extracts are large and negative and are accompanied by exothermic adsorption process (Umoren et al., 2011; Abdallah *et al.,* 2014; Al-Otaibi *et al.,* 2014; Ghalib *et al.,* 2020).

3.3.3 Free Energy of Adsorption ΔGads

The free energy of adsorption was calculated and the values of ΔGads for the tested isotherm in Table 4 revealed that for the leave extract, - 18.364 is the lowest and -11.129 is the highest in magnitude which are negatively less than -40 kJmol-1 as required for the mechanism of chemical adsorption take place. Consequently, the adsorption process of *ZSC* leave extract on the MS surface is spontaneous and the mechanism of physical adsorption is considered to be the nature of metal-inhibitor interaction. Any ΔGads which is negatively less than the threshold value -40 kJmol-1 required for the mechanism of chemical adsorption to take place, the adsorption process is physisorption (Abdallah *et al.,* 2014).

3.3.4 Adsorption Isotherms

The four selected models of adsorption isotherms: Langmuir, Temkin, Frumikn and Flory-Huggins isotherms were used for this study to describe the model of adsorption and to establish the best adsorption isotherm(s). The results of this investigation are presented in Table 4. Their correlation coefficient (R^2) values were used to determine the best-fit isotherm among them. The parameter was obtained from the plot of each isotherm at temperatures 303 K and the best fit was considered by taking the values of \mathbb{R}^2 which will give exactly or nearly equal unity at temperature.

3.3.4.1 Langmuir isotherm

Langmuir adsorption isotherm is expressed according to Equation (9)

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

Where C is the concentration of inhibitor, K is the adsorption equilibrium constant and θ is the surface coverage of the inhibitor. Taking logarithm of both side of equation (9)

$$
\log\frac{\zeta}{\theta} = \log C - \log K\tag{10}
$$

(9)

Plotting $\log \frac{c}{a}$ against logC gave a linear relationship as showed in figure 3.

The application of Langmuir isotherm to the adsorption of extracts of ZSC leaves on the surface of MS indicated that there is no interaction between the adsorbate and adsorbent (Joseph & Vincent, 2012; Ghalib, Hadeel, & Alaa, 2020).

3.3.4.2 Temkin isotherm

For the temkin isotherm, the degree of surface coverage (θ) is related to an inhibitor concentration (C) is given in the equation (11) below;

Figure 2: Langmuir isotherms for adsorption of ethanolic extract of *ziziphus spina-christi* leave on mild steel surface

Where K is the adsorption equilibrium constant and a, is the attractive parameter. Rearranging and taking logarithm of both side of equation (11)

$$
\theta = \frac{-2.303 \log K}{2a} - \frac{2.303 \log C}{2a} \tag{12}
$$

Plot of θ against logC, as presented in figure 6.

The adsorption parameters obtained from the Temkin adsorption isotherm are recorded in the table above and the values of the attractive parameter (a) are all negative in all cases which indicates that repulsion exists in the adsorption layer.

Figure 3: Temkin isotherms for adsorption of ethanolic extract of *ziziphus spina-christi* leave on mild steel surface

3.3.4.3 The Frumkin adsorption isotherm

The Frumkin adsorption isotherm is given by equation 13 (Eddy, Ekwumemgbo, & Odoemelam, 2018). (13)

Where K is the adsorption-desorption constant and α is the lateral interaction term describing the interaction in adsorbed layer. Plot of log $\left[\frac{\theta}{1-\theta}\right] \times \left(\frac{\theta}{1-\theta}\right)$ versus θ as presented in figure 4.

Figure 4: Frumkin isotherms for adsorption of ethanolic extract of *ziziphus spina-christi* leave on mild steel surface.

The Frumkin adsorption isotherms parameter (α) exhibits a negative value, indicating that the inhibitor exhibits an attractive behavior on the mild steel surface.

3.3.4.4 Flory-Huggins adsorption isotherm

The Flory-Huggins adsorption isotherm is given by equation 14 (Migahed & El-Sherif, 2021).

$$
\log\left(\frac{e}{c}\right) = \log K + x\log\left(1 - \theta\right) \tag{14}
$$

where x is the size parameter and is a measure of the number of adsorbed water molecules substituted by a given inhibitor molecule. Plot of $\log(\frac{\theta}{c})$ against $\log(1-\theta)$ are shown in figure 8.

Figure 5: Flory-Huggins isotherms for adsorption of ethanolic extract of *ziziphus spina-christi* leave on mild steel surface

The values of the size parameter of Flory-Huggins (x) are all negative which indicates that the absorbed species of ethanolic extracts are compact since they could displace more than one water molecule from the mild steel surface.

Deave Daniell Inhibitor Conc	Activation Energy Ea	Heat of Adsorption Q_{Ads}	
0.00	32.572	$\overline{}$	
0.20	32.856	-15.62	
0.40	34.599	-15.44	
0.60	35.786	-14.67	
0.80	35.958	-14.28	

Table 3: Activation Energy Ea ($kJmol^{-1}$) and Heat of Adsorption Q_{Ads} ($kJmol^{-1}$) at Various Inhibitors of Leave Extracts

Table 5: Adsorption parameters for adsorption of ethanolic extract of *ziziphus spina-christi* leave on mild steel surface at 303 K

3.4 Surface Morphology Studies 3.4.1 Fourier Transform Infrared Spectroscopic Analysis (FT-IR)

The FT-IR spectra of the *ZSC* leave and corroded MS sample (corrosion product), inhibited sample were carried out and results are

in Figure 8-10 below. Comparing the spectrum of leave extracts and corrosion product with inhibitors as shown below, the results indicates various chemical shifts of as presented in these figures.

Figure 6: FT-IR Spectrum of *ZSC* Leave

 Figure 7: FT-IR Spectrum of Inhibited Product

 Figure 8: Overlap spectra of *ZSC* of Leave and Inhibited Product

The table below gives the frequency value obtained from the plant crude extracts which compared with the frequency values of corrosion product, the differences in frequency values showed how the functional group changed position upon adsorption of extracts on the MS metal surface.

S/N	Extract $(cm-1)$	Corrosion product $(cm-1)$	Difference (cm
1.	3279	3231	0048
2.	2921	2113	0103
3.	1704	1864	0049
4.	1607	1640	-0033
5.	1514	1477	0037
6.	1365	1350	0015
7.	1037	1019	0018
8.	817	795	0022

Table 7: Values of Chemical Shift between the Leave Extract and Corrosion Product

The FT-IR spectrum of *ziziphus spina-christi* leave is given in Figure 6. A broad band at 3279 cm-1 is observed in the spectrum of *ziziphus spina-christi* leave extract indicating -OH or N-H stretching vibration. A peak observed at 2921- 2854 cm-1 is attributable to the –CH aliphatic compounds and a peak observed at 1607 -1514 cm-1 is attributable to the aromatic -C=Ostretching frequency. The absorption band at 1443cm-1 is due to the C- H bending mode of $CH₂$. The peak at 1365 cm-1 is due to $-OH$ of phenol. The peak at 1037 cm-1 is due to the C-

O-C stretching of ether compounds. The adsorption bands below 1000cm-1 correspond to the aliphatic and aromatic C-H groups. The FTIR spectrum (corrosion product) of the film formed on the surface of the mild steel after immersion in an acid solution of *ziziphus spinachristi* leave is shown in Figure 7. On comparing 6 and 7, there is a downshift from 3279 to 3231 cm-1, which is attributed to the change in the frequencies of hydroxyl and amino groups. The - C=O stretching frequency has increased from 1607 to 1640cm-1. This indicates that *ziziphus spina-christi* leave has co-ordinated with Fe2+

through O atoms in -C=O stretching frequency. The result shows that *ziziphus spina-christi* leave contains oxygen and nitrogen atoms in functional groups (O-H, N-H, C-O-C, C=O, C-H) and aromatic ring, which mainly bond with metal and protect mild steel from corrosion (Karthik *et al*., 2015).

The results from this study on *Ziziphus spinachristi* as a corrosion inhibitor provide significant insight into the potential of plantbased inhibitors in comparison to synthetic alternatives. This research highlights the effectiveness of ZSC leaves, which contain various phytochemicals such as alkaloids, flavonoids, and saponins, known for their corrosion-inhibiting properties. Studies such as those by Singh *et al.* (2020) and Migahed & El-Sherif (2021) have similarly demonstrated the success of plant extracts in reducing corrosion rates, and our findings corroborate these trends, revealing up to 64% inhibition efficiency with increasing inhibitor concentration.

In comparison to other green inhibitors, such as those derived from neem and moringa plants, ZSC leaves show comparable, if not superior, efficiency in mild steel corrosion protection in acidic environments (Nawaz et al., 2021). The mechanism of inhibition is primarily physisorption, which is corroborated by the thermodynamic parameters calculated from the weight loss and adsorption studies. The Langmuir adsorption model best fits the data, suggesting no interaction between the inhibitor molecules and the metal surface, further solidifying the non-toxic nature of this inhibitor. The environmental implications of this study are profound. *Ziziphus spina-christi* is abundant and

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easy to cultivate, making it a cost-effective, renewable resource. The use of this plant for corrosion inhibition offers a sustainable alternative to synthetic chemicals that can be harmful to both the environment and human health (Raja *et al.,* 2020). Additionally, its biodegradable nature makes it a suitable candidate for industrial applications in sectors such as manufacturing, construction, and transportation, where corrosion is a persistent challenge.

The potential industrial applications of this research are vast, particularly in industries that rely on steel components exposed to harsh environments, such as the oil and gas sector, marine industries, and automotive manufacturing. The findings also contribute to the growing body of knowledge on green chemistry, which aims to reduce the ecological footprint of industrial processes.

CONCLUSION

The study confirms that *Ziziphus spina-christi* leaf extract effectively inhibits corrosion on mild steel in hydrochloric acid. Phytochemical analysis revealed the presence of key compounds such as alkaloids, flavonoids, and tannins, which are responsible for the inhibition. The inhibition efficiency increased with extract concentration, and the adsorption mechanism was determined to be physical, with a preference for physisorption. Thermodynamic data suggests that the adsorption process is spontaneous and exothermic. FT-IR analysis further confirmed the interaction between the inhibitor and the steel surface, highlighting its potential as a green corrosion inhibitor

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