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Corrosion Inhibition Efficiency of *Chromolaena odorata* Leaves Extract on Mild Steel in Sulphuric Acid Medium

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ABSTRACT

The weight loss technique was utilized to explore how siam leaf extract affects the corrosion of mild steel in a 1.5 M H_2SO_4 solution. The investigation took place under varying conditions: without the extract and with different concentrations (2.0 – 10.0 g/L) at temperatures ranging from 298 to 328 K, with an exposure time of 6 hours. The findings indicated that higher amounts of the extract led to better inhibition of corrosion, but increased temperatures had an adverse effect. The collected data underwent analysis employing Langmuir, Temkin, and Freundlich isotherm models, with Langmuir exhibiting the most substantial correlation among them. Moreover, the thermodynamic analysis revealed that the adsorption of the extract onto the mild steel surface was endothermic, spontaneous, and aligned with a physical adsorption mechanism. Consequently, siam leaf extract proves to be a promising green corrosion inhibitor in preventing corrosion.

1. Introduction

Multiple industries rely on metal-based equipment exposed to diverse corrosive environments, rendering them susceptible to corrosion [1]. Protecting these materials from deterioration has become a focal point, aiming to mitigate potential hazards in industrial settings. While synthetic

inhibitors, both inorganic and organic compounds, have displayed high efficiency in commercial use for deterring metal corrosion, their costliness and toxicity pose significant environmental and health concerns [2, 3].
Recent research has explored extracts from various plants, such as avocado leaves [4], *Acalypha godseffiana* [5], bitter melon [6], watermelon [7], and *Moringa oleifera* [8]. There remains a keen interest in further investigation in this field, particularly in assessing plant sources that have fewer competing applications, potentially utilized for nutrition or pharmaceutical purposes [9].
Siam weed, prevalent in different parts of Nigeria, especially the Southern region, offers readily available leaves at minimal or no cost. This study aims to assess the efficacy of siam leaf extract in inhibiting mild steel corrosion in a sulphuric solution.

2. Materials and Method

2.1 Metal Preparation

A mild steel sheet was purchased from the local steel market in Warri, Delta State. The composition of the mild steel used in this investigation includes the following elements and their respective weight percentages: C (0.215), Si (0.258), Cu (0.017), S (0.009), Mn (0.467), P (0.007), Cr (0.021), Ni (0.001), and Fe (99.005). Upon acquisition, the mild steel sheet underwent cleaning and was subsequently cut into coupons measuring 40mm in length, 20mm in width, and 2mm in thickness. Each coupon was drilled with small holes and attached with a thread to facilitate easy suspension into the corrosive test media. Mechanical polishing using emery papers was carried out to remove scale, followed by cleaning with propanone and ethanol. The prepared coupons were then stored in a desiccator to ensure they remained free from moisture.

2.2 Siam Leaf Extract (SLE) Preparation

Siam leaves were gathered from an uncultivated farmland in Kwale, located in the Ndokwa-West Local Government Area of Delta State. These leaves were sun-dried and finely crushed. A quantity of 380 grams of the powdered leaves was placed into 500 ml of ethyl alcohol and subjected to extraction using a Soxhlet extractor for a period of 48 hours. The resulting mixture was filtered to isolate the extract, which was then concentrated by evaporating the ethanol at 80^{o}C utilizing a rotary evaporator. The concentrated extract was carefully stored in bottles and placed in a desiccator for preservation before its utilization.

Figure 1. Siam Weed (*Chromolaena odorata*)

2.3 Preparation of Test Solution

Six different test solutions were prepared by measuring specific amounts of the stock extract (2.0 g, 4.0 g, 6.0 g, 8.0 g, and 10.0 g) and combining them with 1000cm^3 of 1.5 M H_2SO_4. This produced inhibitor solutions of concentrations 2.0 g/L, 4.0 g/L, 6.0 g/L, 8.0 g/L, and 10.0 g/L. The sixth solution served as a control, containing only 1.5 M tetraoxosulphate (IV) solution without the extract.

2.4 Phytochemical Analysis
The siam leaf extract (SLE) underwent a phytochemical analysis to explore its bioactive components, employing established procedures documented in prior studies [10 - 11].

2.5 Measurements of Weight Loss
The initially weighed coupons were placed into 100 millilitres of a 1.5 M sulpuric acid solution. These coupons were submerged in solutions containing different concentrations (2.0 g/L, 4.0 g/L, 6.0 g/L, 8.0 g/L, and 10.0 g/L) of the test solution. The entire setup was maintained at temperatures of 25ºC, 35ºC, 45ºC, and 55ºC within a controlled water bath for duration of 6 hours. Following this period, the coupons were removed, cleaned, dried, and then reweighed.

Figure 2. Experimental set-up of the measurements of weight loss in water bath

The variance between the initial and concluding weights of the coupons denotes the weight loss. Equations 1-3 were employed to evaluate the extent of surface coverage (θ), inhibition efficiency (IE), and corrosion rate (CR) [12].

$$\mathrm{CR} = \frac{87600\mathrm{W}}{\mathrm{A}\rho \mathrm{t}} \tag{1}$$

$$\mathrm{IE}(\%) = \left(1 - \frac{\mathrm{CR_i}}{\mathrm{CR_o}}\right) \times \frac{100}{1} \tag{2}$$

$$\theta = \left(1 - \frac{\mathrm{CR_i}}{\mathrm{CR_o}}\right) \tag{3}$$

Here, A signifies area of coupon surface exposed, measured in square centimetres (cm^2).t denotes the duration of exposure in hours (h), ρ represents the coupon's density in grams per cubic centimetre (g/cm^3), CR_o signifies the corrosion rate without the inhibitor (in absence of extract), and CR_i represents the corrosion rate when the inhibitor is present.

2.6 Determination of Activation Energy (E_a)
The activation energy is determined by analysing the slope of the graph depicting ln (CR) against the reciprocal of temperature (1/T) following Equation 4 [13].

$$\ln \mathrm{CR} = \frac{-\mathrm{E_a}}{\mathrm{R}}\left(\frac{1}{\mathrm{T}}\right) + \ln \mathrm{A} \tag{4}$$

In this context, E_a stands for the activation energy, while R represents the universal gas constant (8.314 J/molK), CR signifies the corrosion rate, T denotes the absolute temperature measured in Kelvin (K), and A refers to the exponential factor.

2.7 Computation of Changes in Entropy and Enthalpy

Equation 5, describing the transition state, was employed for the computation of alterations in entropy and enthalpy [14].

$$\log\left(\frac{CR}{T}\right) = -\frac{\Delta H_{ads}}{2.303R}\left(\frac{1}{T}\right) + \left[\log\left(\frac{R}{Nh}\right) + \left(\frac{\Delta S_{ads}}{2.303R}\right)\right] \quad (5)$$

Here, N represents Avogadro's constant, CR stands for the corrosion rate at temperature T, and h denotes Planck's constant. A plot of $\log\left(\frac{CR}{T}\right)$ against $\frac{1}{T}$ was created, and the alterations in entropy (ΔS_{ads}) and enthalpy (ΔH_{ads}) were computed from the intercept $\left[\log\left(\frac{R}{Nh}\right) + \left(\frac{\Delta S_{ads}}{2.303R}\right)\right]$ and the slope($-\frac{\Delta H_{ads}}{2.303R}$) respectively.

2.8 Adsorption Constant and Adsorption Isotherm

In order to identify the most suitable model describing the adsorption of siam leaf extract on mild steel surfaces, the values of surface coverage (θ) and extract concentration (C) were applied to the Langmuir, Temkin, and Freundlich isotherm models. Equations 6-8 outline the formulas for the Freundlich, Temkin, and Langmuir models, respectively.

The isotherm of the Langmuir adsorption model [15]:

$$\frac{c}{\theta} = \frac{1}{K_{ads}} + C \quad (6)$$

Model for Temkin adsorption isotherm [16]:

$$\theta = \ln C + K_{ads} \quad (7)$$

Model for the Freundlich adsorption isotherm [2]:

$$\log\theta = \log K_{ads} + n\log C \quad (8)$$

2.9 Determination of Parameters for Thermodynamic Adsorption

To investigate the characteristics and viability of adsorption, the alteration in Gibb’s free energy of adsorption (ΔG) was calculated utilizing Equation 9 [17].

$$\Delta G_{ads} = -RT\ln(55.5K_{ads}) \quad (9)$$

The number 55.5 denotes the molar concentration of water within the solution, while R represents the universal gas constant, and K_{ads} signifies the adsorption equilibrium constant derived from the isotherm at the absolute temperature (T).

3. Results and Discussion

3.1Result for Phytochemical Screening of Siam Leaf Extract

Table 1indicates the result of phytochemical analysis of siam leaf extract (SLE) and it was discovered that the Siam Leaf Extract (SLE) contains active elements that likely played a role in its inhibitory effect. These bioactive elements might have the ability to create a shielding layer on the surface of mild steel and be absorbed onto it [4]. This observation aligns with earlier research outcomes [18, 15].

Table 1. Result of phytochemical screening

Phytochemical constituents	Presence
Tannins	++
Saponins	+
Alkaloids	+
Flavonoids	++
Phenols	+
Steroids	++
Anthraquinones	+

Where + = Present, ++ greatly present

3.2 Temperature and Concentration Effects on mild steel corrosion rates

Figure 3 illustrates a decrease in corrosion rates with higher extract concentrations, yet rates escalated with rising temperatures. Across all temperatures analysed, corrosion rates were consistently higher without the inhibitor compared to varied inhibitor concentrations. The elevated corrosion rates with increased temperature were attributed to higher average kinetic energy and steel dissolution at higher temperatures [19]. These findings align with previous research [20, 14, 21].

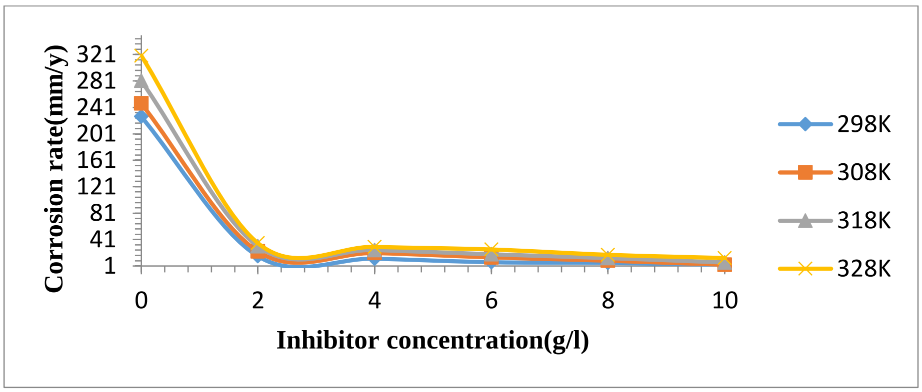

Figure 3. Effect of Temperature and Concentration on Mild Steel Corrosion Rate in 1.5 M H_2SO_4.

3.3 The Impact of Temperature and Concentration on the Efficiency of Siam Leaf Extract

Figure 4 demonstrates that the inhibition rate decreases with an increase in corrosion temperature but rises with the amount of the extract. It reaches its maximum inhibition efficiency at 98.82% with a 10.0 g/L inhibitor concentration at a corrosion temperature of 298 K after a 6-hour exposure. The enhancement in the extract's inhibitory effectiveness with concentration may be attributed to a higher concentration of adsorbed extract particles at the interface between the mild steel surface and the exposed environment, leading to improved coverage of the mild steel surface. Additionally, the decline in the corrosion inhibitory efficacy of siam leaf extract with a temperature increase can be linked to the detachment of adsorbed extract particles and increased reactivity at higher temperatures. These observations correspond with findings from prior studies [22 - 24].

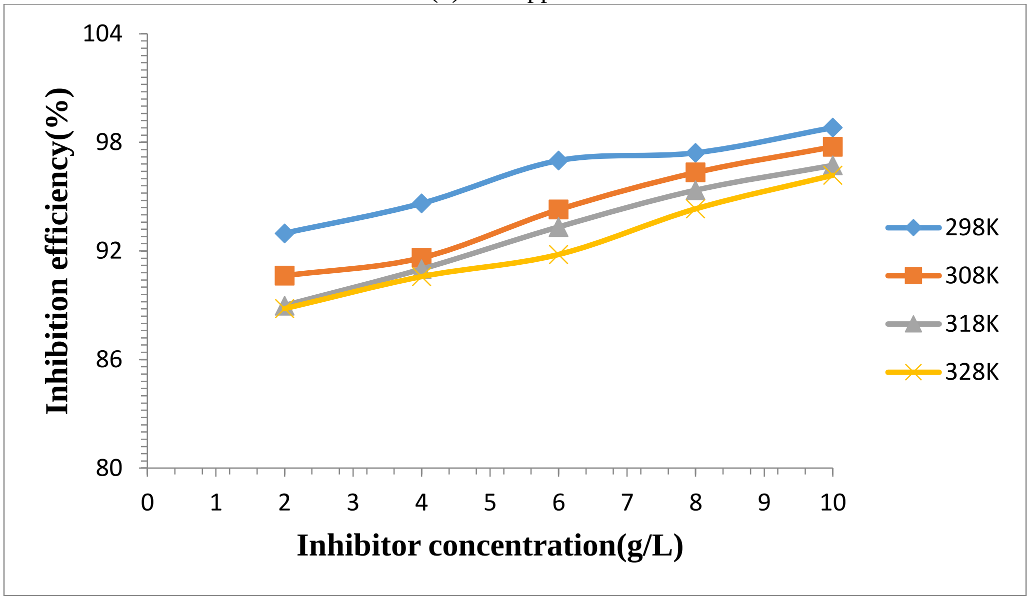

Figure 4. Changes in inhibition effectiveness with different concentrations of the extract across varying temperatures

3.4 Thermodynamic Parameters

3.4.1 Activation energy (E_a)

Figure 5 exhibits the Log CR versus 1/T graph for various extract concentrations. By examining the slope of the plot, the activation energies were calculated both with and without the extract, as shown in Table 2. It's noted that in the presence of the extract, the resulting activation energy surpasses that observed without the inhibitor. This finding hints at the physical mechanism underlying the adsorption process [25]. Moreover, the decrease in corrosion intensity might stem from heightened energy barriers within the inhibited process, potentially elucidating the escalation in activation energy values with increasing concentration [26–27].

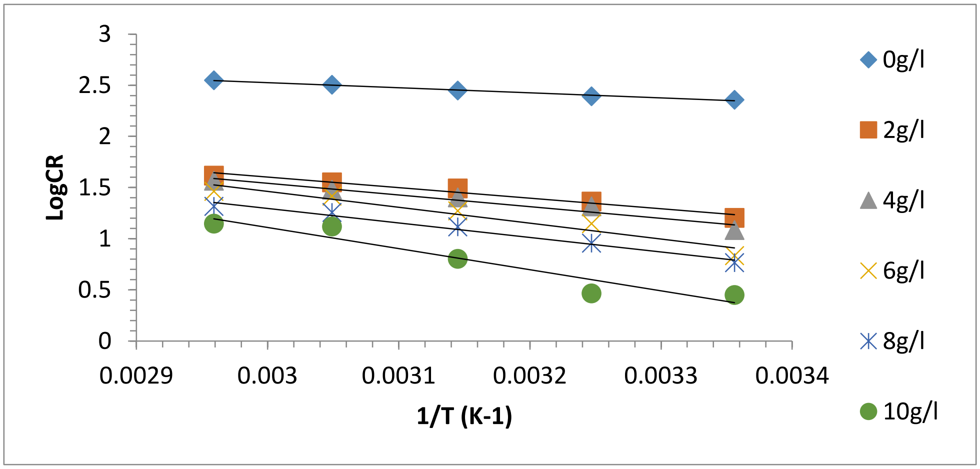

Figure 5. A graph depicting Log CR against 1/T across various concentrations of siam leaf extract

3.4.2 Changes in Entropy and Enthalpy

Figure 6 displays the Log CR/T plotted against 1/T for various corrosion processes. The intercept and slope of the graph were utilized to calculate the values of ΔS and ΔH, as outlined in Table 2. The table clearly indicates that all the enthalpy change values are positive, signifying an endothermic process of mild steel corrosion in the corrosive environment. Furthermore, all entropy change values are negative and decrease with an increase in inhibitor concentration. This indicates a reduction in the randomness of the reacting system at the boundary layer between the molecules of the extract and the surface of mild steel [2].

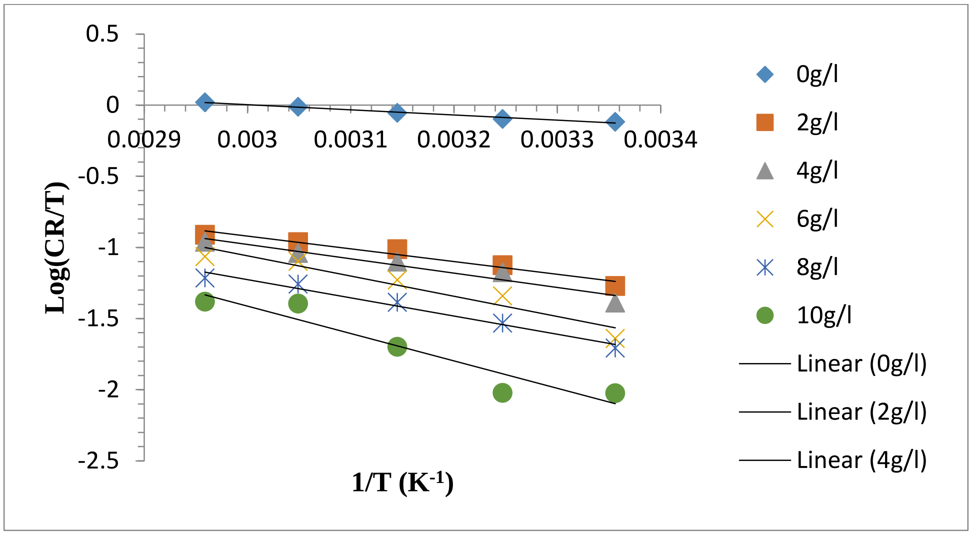

Figure 6. A plot to compute entropy and enthalpy

Table 2: Thermodynamic parameters of siam leaf extract

Inhibitor concentrations(g/L)	E_a (kJ/mol)	R^2	ΔH_{ads} (kJ/mol)	ΔS_{ads} ($Jmol^{-1}K^{-1}$)	R^2
0	9.56591	0.9919	6.92878	-176.722	0.9859
2.0	19.75602	0.9676	17.11869	-163.849	0.9562
4.0	21.87944	0.9509	19.24288	-158.599	0.9365
6.0	29.79678	0.9340	27.16022	-136.383	0.9208
8.0	27.12001	0.9807	24.48345	-147.595	0.9759
10.0	39.56182	0.9164	36.92526	-113.837	0.9053

3.5 Adsorption Isotherm Study

Figures 7-9 illustrate the different adsorption isotherm models applied in this study. Gravimetric data were fitted into Langmuir, Temkin, and Freundlich isotherms. Table 3 presents the R^2 values for these models, aiding in determining the best-fitted model. The Langmuir model exhibited coefficients of regression (R^2) closest to unity, suggesting its suitability for the adsorption of siam leaf extract on the mild steel surface within a 1.5 M H_2SO4 environment. Consequently, the Langmuir model was employed to calculate the adsorption equilibrium constant, K_{ads}, using Equation 6.

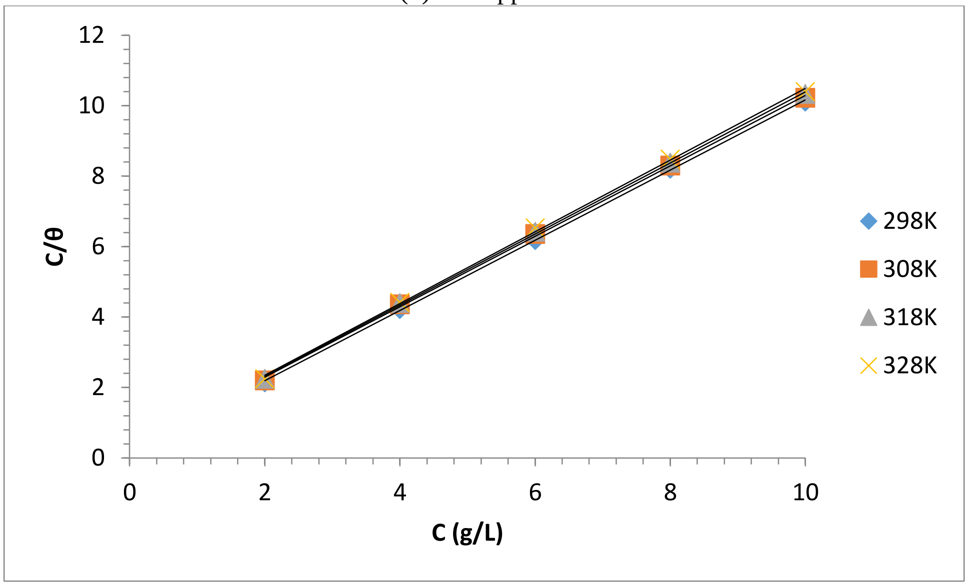

Figure 7. Langmuir isotherm plot

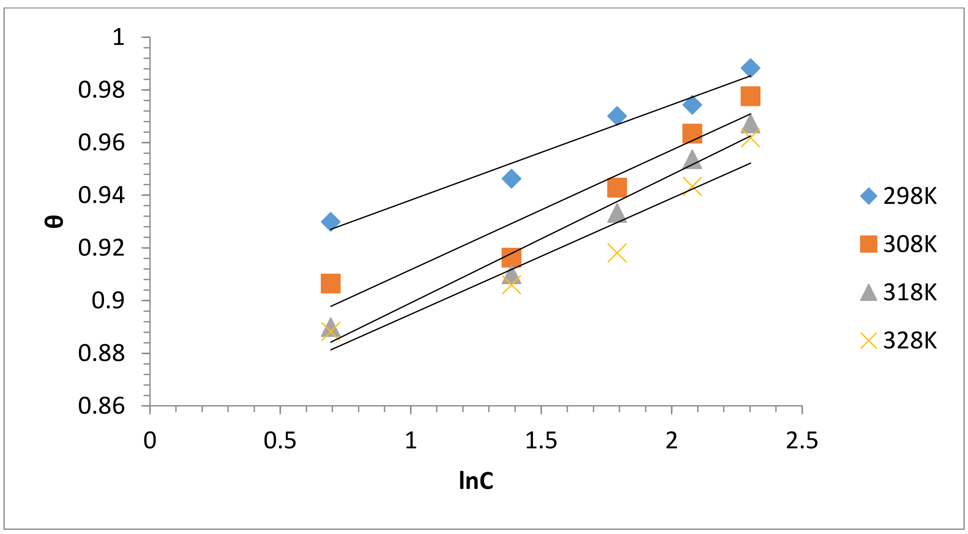

Figure 8. Temkin isotherm plot

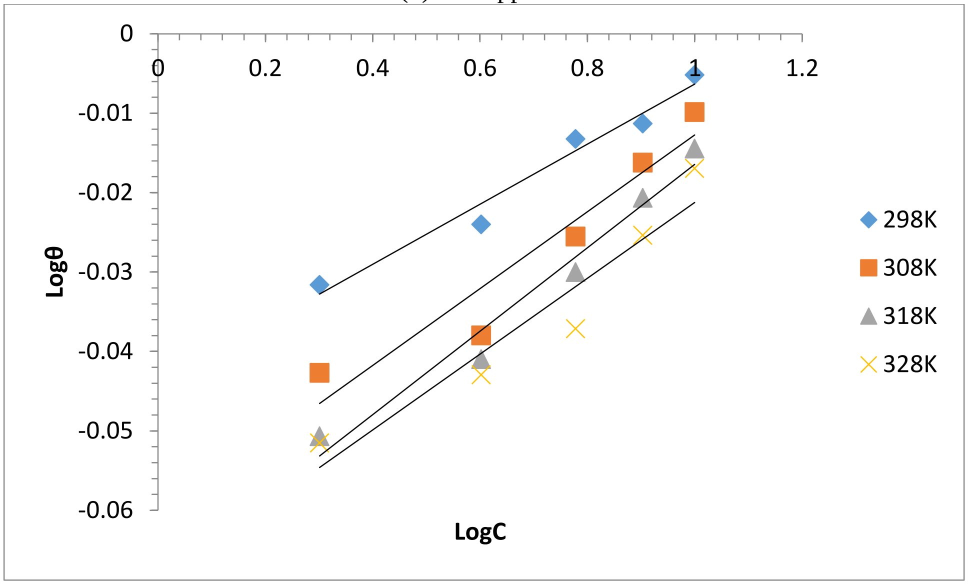

Figure 9. Freundlich isotherm plot

Table 3. The R^2 values of the different adsorption isotherm models.

T(K)		R^2	
	Langmuir	**Temkin**	**Freundlich**
298	0.9998	0.9685	0.9702
308	0.9995	0.9131	0.9166
318	0.9996	0.9644	0.9681
328	0.9992	0.9099	0.9160

3.6 Adsorption Equilibrium Constant (K_{ads})

Table 4 showcases the diverse values of the adsorption equilibrium constants derived from the Langmuir plot intercept across different corrosion temperatures. All these values are positive, indicating the viability of the adsorbed extract particles on the steel surface. Moreover, the table distinctly shows a decrease in the magnitude of K_{ads} as the system's temperature increases, suggesting reduced intermolecular interactions between the extract molecules and the mild steel surface. This observation might stem from the dissolution of the adhered extract particles at higher temperatures [28 - 29].

3.7 Change in Adsorption's Gibb's Free Energy (ΔG_{ads})

Table 4 presents the determined values of the change in Gibbs' free energy (ΔG) calculated from the adsorption equilibrium constants at different temperatures using Equation 7. The negative ΔG_{ads} values suggest the viability of the adsorption process and the stability of the adsorbed layer. Furthermore, it's noteworthy that the obtained ΔG_{ads} values were below 20 KJ/mol, indicating a physical nature for the adsorption mechanism [30].

Table 4. Variations in Gibbs' free energy related to adsorption and the equilibrium constant for adsorption

T (K)	K_{ads}(mol^{-1})	ΔG (KJ/mol)
298	4.9383	-13.9076
308	3.3501	-13.3807
318	3.2531	-13.7374
328	3.1342	-14.0679

4. Conclusion

The research examined the effectiveness of siam leaf extract (SLE) in mitigating mild steel corrosion within a sulphuric acid environment. The inhibitory efficiency improved with higher extract amounts but declined as temperatures rose. The maximum efficiency, reaching 98.82%, occurred at a 10 g/L extract concentration and a corrosion temperature of 298 K. The activation energy increased alongside the extract concentration and was lower for the uninhibited process. Siam leaf extract adsorption aligned with the Langmuir isotherm model, and the values of Gibbs' free energy supported a physical adsorption mechanism. Consequently, the study concluded that siam leaf extract efficiently hinders the corrosion of mild steel in a sulphuric acid environment.

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