



Original Research Article

Equilibrium and Thermodynamic Characteristics of the Corrosion Inhibition of Mild Steel Using Sweet Prayer Leaf Extract in Alkaline Medium

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ABSTRACT

The usage of synthetic-based inhibitors for protection of metals against corrosion is always accompanied by environmental and economic challenges, including a threat to human health. Therefore, the corrosion inhibition efficiency of sweet prayer (*Thaumatococcus danielli*) leaf extract on mild steel in alkaline medium (NaOH) was investigated. The effects of temperature and concentration of the inhibitor on corrosion inhibitive potential of the leaf extract were evaluated. The equilibrium data were subjected to isotherm analysis (Langmuir, Freundlich and Temkin models). Langmuir model was the best model that provided information on the adsorption of the corrosion systems. The values of Langmuir equilibrium constant (K_L) increased from 0.1581 g/L to 0.6523 g/L as the temperature increased from 298 K to 333 K. The findings revealed that sweet prayer leaf extract at different concentrations had inhibitive effects of mild steel on alkaline medium. The values of activation energy of the inhibited corrosion systems were greater than that of obtained for uninhibited system. Thermodynamic study revealed a feasible and spontaneous adsorption of sweet prayer leaf extract on mild steel. The trends of inhibition efficiencies and the results obtained from the thermodynamic parameters suggested a physical adsorption mechanism. The study proved that the sweet prayer leaf extract could serve as a low-cost corrosion inhibitor of mild steel in alkaline medium.

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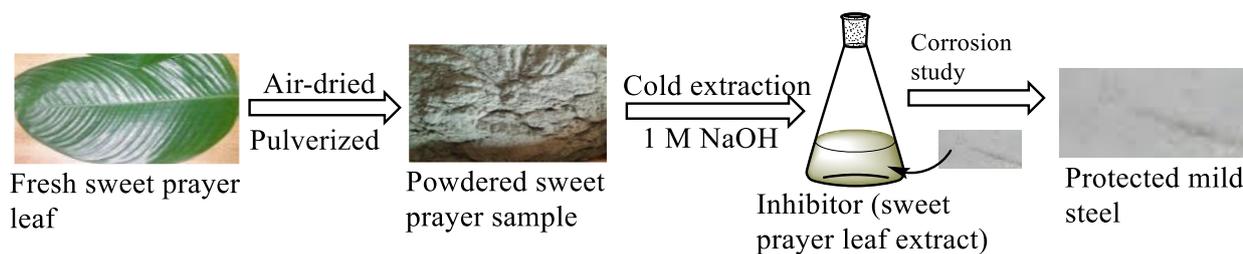
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GRAPHICAL ABSTRACT



Introduction

Mild steel is a structural material that is widely used for a number of industrial applications. Mild steel is popular and generally used as an engineering material for both construction and machinery because of its outstanding mechanical properties (ductility and malleability) coupled with low-cost and availability [1,2]. Unfortunately, the wide utility and acceptability of mild steel are impeded by loss of mechanical strength (efficiency) and aesthetical value [3] due to corrosion *via* rapid interaction with the environment such as solvents and medium (acidic or alkaline) [4,5]. The environmental and mechanical inflicative cost by corrosion is always high [6]. In industries, corrosion of mild steel has been reported to cause numerous accidents as a result of equipment wearing and leakages, industrial plant shut down [7], high production cost, inefficient loading capacity, and health issues as a result of consumption of pharmaceutical and food product manufactured with corroded equipment [8]. The associated challenges inspire the quest for suitable and effective strategies for controlling corrosion of mild steel in harsh media. The usage of inhibitors is an excellent approach [9] and many inhibitors such as organic compounds, rare earth metals and synthetic compounds are available [10,11]. By definition, a corrosion inhibitor is a substance that protects a metal surface from corroding when exposed to a harsh environment or solution. Inhibitors have various industrial

applications such as cleaning of rust, descaling and alkalisng of an oil well. An inclusion of inhibitor to any medium to which mild steel is subjected is a good practical approach. This is achieved by preventing the metal surface to interact with the external environment [12] and subsequently enhancing adsorption process, and increasing the lifespan of the steel. Notably, the choice of inhibitor is influenced by certain factors such as efficiency, environmental impacts and cost. A good inhibitor should prevent any metal from reactivity. Similarly, a good inhibitor should be cheap, readily available and non-toxic. Several studies have been carried out on the efficiencies and anti-corrosion effects of synthetic-based inhibitors; however, some environmental and economic concerns still remain puzzled [13]. The shortcomings that arose from the use of synthetic-based inhibitors have triggered the interest in the use of green inhibitors, which are non-toxic, eco-friendly and biodegradability [14], and are also characterised by simplicity in production as well as relatively low-cost [15]. Considering global clamouring for sustainable and greener environment and reduced production cost [16], green inhibitors remain a preferable and sustainable option and have therefore recently gained attention. Some studies have reported the use of green inhibitors especially extracts from *Jatropha curcas* seed husk [17], *Baphia nitida* [18], *Vernonia amygdalifolia* [19], *Rollinia occidentalis* [20], *Xanthium strumarium* [21], *Glycyrrhiza glabra* [22],

Moringa oleifera [23], among others, for corrosion inhibition of mild steel in different media. Nevertheless, some significant improvements need to be achieved. Hence, a low-cost, readily available and an underutilised sweet prayer plant is another plant that could be harnessed to impact anti-corrosion effect on mild steel.

Sweet prayer plant (*Thaumatococcus danielli* L.) also known as 'miracle fruit' is a wild shrub that grows widely in the rainforest of West Africa, majorly in Nigeria, Ghana and Ivory Coast. The plant can attain a height of 3–4 m with up to 46 cm long papery leaves [24], which are used for wrapping of food and making local mat in Nigeria.

This study aimed at investigating the inhibitive effect of sweet prayer leaf extract on corrosion of mild steel in alkaline medium (NaOH). The effects of the concentration of the inhibitor and temperature on the corrosion inhibitive potential of the extract were investigated. The isotherm and thermodynamic parameters of the tandem inhibitive process were also explored. To date, there has been a paucity of information on the corrosion inhibition potential of sweet prayer plant extract. The growing industrial interest in finding a green corrosion inhibitor for mild steel has necessitated this study, as it will provide useful scientific data on the inhibition level of sweet prayer leaf extract.

Experiments

Materials

Sodium hydroxide, acetone, ammonium chloride, and other reagents used in this study were of analytical grade. The mild steel of various coupons with size (14.25 x 11.42 x 4.35 mm) was used for this study. The mild steel was obtained from a metal dealer at Oja-Oba, Akure, Nigeria and cut into the required dimensions at the Department of Mechanical Engineering, The Federal University of Technology, Akure, Nigeria. The metal pieces were degreased by immersion and washed in acetone for 2 min. The mild steel samples were thereafter abraded by emery, washed with distilled water, dried with a clean cloth, immersed in acetone, re-dried, weighed and store in a desiccator prior usage.

Preparation of leaf extract

Matured sweet prayer leaves were obtained from Oja-Oba, Akure, Nigeria and authenticated at the Department of Crop, Soil, and Pest Management, The Federal University of Technology, Akure, Nigeria. The leaves (**Fig. 1A**) were cut into pieces, air-dried at room temperature for 2 weeks. Subsequently, the dried leaves (**Fig. 1B**) were ground and sieved to obtain a fine powder (**Fig. 1C**). A 70 g of the powdered leaf sample was extracted with 765 mL of 1.0 mol/L NaOH using a cold extraction method for 48 h. The extract was filtered and concentrated in an oven at 70 °C.



Fig. 1. Pictorial view of (A) sweet prayer fresh matured leaves, (B) air-dried leaves, and (C) powdered sample

Determination of weight loss

Weight loss measurements were carried out by weighing a known amount of degreased metal steel and immersing into 50 mL of 1.0 mol/L NaOH using different concentrations (0.2, 0.4, 0.6, 0.8, and 1.0 g/L) of the inhibitor at various temperatures (293, 303, 313, 323, and 333 K) for 3 h. Blank experiments were also conducted for comparison purposes. On completion of each experiment, the mild steel was removed from the inhibitor solution, rinsed, dried and weighed. The values obtained for the gravimetric study were an average of triplicate experiments. By definition, the weight loss (WL), which was used for estimation of the corrosion rate (CR), inhibitor efficiency (IE) and surface coverage was calculated using equation 1;

$$WL = W_o - W \quad (1)$$

where W_o = initial is the weight of the mild steel before immersion in inhibitor solution, and W = final is the weight of the mild steel after the experiment.

Estimation of corrosion rate, inhibition efficiency and degree of surface coverage

Corrosion rate (CR), inhibition efficiency (IE) and degree of surface coverage (θ) were calculated from the weight loss determination. The CR, IE and θ were evaluated using Equations 2 - 4, respectively.

$$CR = \frac{WL}{A \cdot t} \quad (2)$$

$$I.E. (\%) = \frac{WL}{W_o} \cdot 100 \quad (3)$$

$$\theta = \frac{I.E. (\%)}{100} \quad (4)$$

where t = exposure time (h), and A = surface area (cm^2).

Isotherm and thermodynamic evaluations

For evaluation of corrosion isotherm parameters, Langmuir, Freundlich and Temkin models, were used. The respective equations of these models are presented in Equations 5-7.

$$\frac{C}{\theta} = \frac{1}{K_L} + C \quad (5)$$

$$\log \theta = \log K_F + n_F \log C \quad (6)$$

$$\theta = \frac{-\ln K_T}{2a} - \frac{\ln C}{2a} \quad (7)$$

where C is the inhibitor concentration (g/L), a is the molecular interaction parameter, K is the equilibrium constant of the adsorption-desorption process in all cases. The values of K in each give information on how favourable the inhibition process is, with higher values indicating favourable adsorption.

To evaluate the effect of temperature on corrosion rate of mild steel immersed in various NaOH concentrations media in the presence of sweet prayer inhibitor, thermodynamic parameters were studied. The activation energy of the corrosion process was estimated using the Arrhenius equation (Equation 8).

$$\ln CR = \ln A - \frac{E_a}{RT} \quad (8)$$

where A is the frequency factor, E_a is the activation energy (kJ/mol), T is absolute temperature (Kelvin), with values changing from 293 K - 333 K, and R is the gas constant (8.314 J/mol K).

A plot of $\ln CR$ against $\frac{1}{T}$, gave the slope $\left(-\frac{E_a}{T}\right)$

and intersect ($\ln A$). The value of E_a was obtained from the slope while the value of A was obtained from the intercept of the plot. To estimate other thermodynamic parameters, Equations 9 and 10 were used.

$$\Delta G^\circ = -RT \ln(55.5K) \quad (9)$$

$$\ln K = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{R} \cdot \frac{1}{T} \quad (10)$$

where K is the equilibrium constant of the model, ΔG° is the standard Gibb's energy change (kJ/mol), ΔH° is the standard enthalpy change (kJ/mol), ΔS° is the standard entropy change (J/mol K) and the molar concentration of water in solution is 55.5 mol/L. Equation 10 is known as van't Hoff equation.

Fourier Transform Infrared (FTIR) analysis

The FTIR analyses of the samples were carried using infrared spectrophotometer (Varian 660 MidIR Dual MCT/DTGS) in the spectral region 4000 cm^{-1} – 400 cm^{-1} .

Results and discussion

The potential of the extract of sweet prayer leaf to inhibit corrosion of mild steel in 1.0 mol/L NaOH solution was investigated using different concentrations of the plant extract and at various temperature values. Cold extraction using NaOH solution was used to extract the corrosion inhibitor components from sweet prayer leaves. Gravimetric method was used to probe into the efficiency of the sweet prayer plant extract to inhibit corrosion in alkaline medium. Control experiments were performed in alkaline medium in the absence of an inhibitor.

Influence of temperature and concentration of inhibitor on inhibition efficiency and corrosion rate

The results on the inhibition efficiency and corrosion rate of mild steel in 1.0 mol/L NaOH are shown in **Figs 2A** and **B**, respectively. The inhibition efficiency (**Fig. 2A**) of the inhibitor increased significantly with a rise in the concentration of the inhibitor between 293 K and 313 K. However, at 323 K the value of IE slightly increased when the inhibitor concentration was increased from 0.2 g/L to 1.0 g/L. At 333 K, the IE was almost independent of the concentration of the inhibitor. The value of IE of the leaf extract decreased with an increase in temperature from

298 K to 333 K. The optimum IE (90 %) was obtained at an inhibitor concentration of 1.0 g/L at 298 K. Similar observations have been reported earlier [20,21]. Amoo *et al.* [25] also reported a similar report of methyl red inhibitor in alkaline medium.

Figure 2B reveals the dependence of corrosion rate of mild steel on inhibitor concentration at different temperature values within the exposure time of 3 h. From this figure, it is evident that the corrosion rate of the mild steel in the absence of the leaf extract was higher than those of inhibited systems. It is clear that sweet prayer leaf extract acted as an efficient inhibitor that effectively reduced the attack of the corrosive NaOH solution. Corrosion rate expresses the rate of redox (reduction–oxidation) reaction, that is, the flow of electrons takes place within the anodic and cathodic sites of the mild steel [26]. In the present report, corrosion rate increased with a rise in temperature because of system agitation but decreased drastically as the concentration of the inhibitor increased. This phenomenon could be as a result of a decrease in contact between the mild steel and corrosion environment. As the inhibitor concentration increased, the contact area was reduced by resisting the OH^- (alkaline) activity. This observation was similar to the previous report on *Psidium guajava* leaf extract as an inhibitor of mild steel corrosion in alkaline medium [25].

Increase in IE as concentration increases can be attributed to adsorption of inhibitor [27]. As the temperature of the system increased, there was a possibility that the protective films broke down, which resulted in reduction in IE of the extract leading to high corrosion rate [28]. In other word, the decrease in IE as the temperature of the corrosion system increases could be linked to the enhancement of the metal dissolution process. The desorption rate of inhibitor molecules will also be large at high temperature [21].

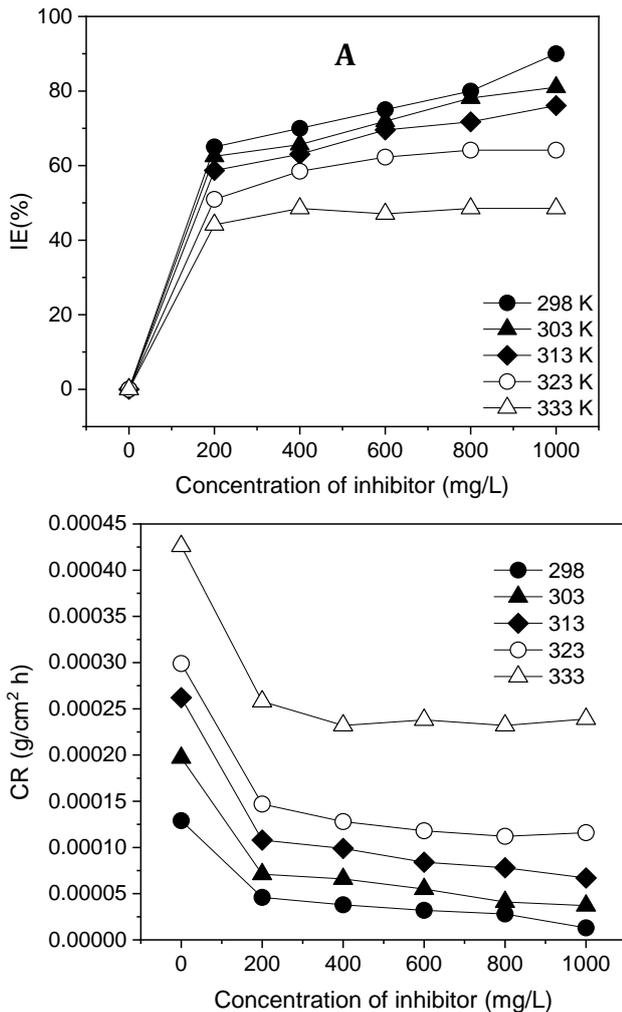


Fig. 2. Dependence of (A) the percentage of inhibition efficiency and (B) corrosion rate on the initial concentrations of sweet prayer leaf extract

Isotherm study

The adsorption of inhibitor's molecules on the surface of metal can lead to reaction at the surface and this reaction can generate secondary compound for formation of protective film. The adsorption of the inhibitor's species on the surface of the metal is generally described by isothermal models. Therefore, adsorption isotherms are useful for proper description of adsorption process. Vital pieces of information on the nature of the inhibitor-mild steel interaction can be obtained from the isotherm study [29]. The characteristics of the metal surface and that of inhibitor, temperature of

corrosion reaction, χ_{charge} , type of solvent, and the electrochemical potential at solution interface dictate the overall adsorption characteristics. The concentration dependent data, of the corrosion inhibition of the sweet prayer inhibitor of mild steel in alkaline medium, were subjected to three isothermal models, namely, Langmuir, Freundlich and Temkin for understanding of the adsorption process. The isothermal curves and parameters are presented in **Figure 3** and **Table 1**, respectively. All the models were adjudged using the values of the adjusted determination coefficient (R_{adj}^2). The

closer the value of R_{adj}^2 to unity, the better the fit. Hence, among the three isothermal models, Langmuir model is the best model that described the adsorption process. This signifies that Langmuir model exhibited the highest R_{adj}^2 values in the temperature ranges (298 K - 333 K) than the corresponding R_{adj}^2 values of the Freundlich and Temkin models. Langmuir plots were made using **Equation 5** and the reciprocal of the intercept of the plots at different temperature values gave the values of Langmuir constant (K_L). The value of the slope of each plot was set at 1.0. As the Langmuir isotherm was adopted, the monolayer coverage was formed and the adsorbed inhibitor molecules did not interact. The adsorption process was influenced by the nature of the sweet prayer inhibitor and the metal surface [30]. The values of Langmuir adsorption equilibrium constant (K_L) increased from 0.1581 g/L to 0.6523 g/L as the temperature increased from 298 K to 333 K. This phenomenon implies that the magnitude of the energetic interaction between the inhibitor and meta steel increases as temperature value increases. The inhibition efficiency of any corrosion inhibitor depends on the values of the binding/equilibrium constant (K) of the inhibitor. The value of K gives the strength between the inhibitor's surface and the adsorbed

species (21). The lower the values of K, the weaker the interaction, and *vice versa*. The values of K obtained in this study shows that the

interaction of the Sweet prayer leaf extract with adsorbed species increased as the temperature increased.

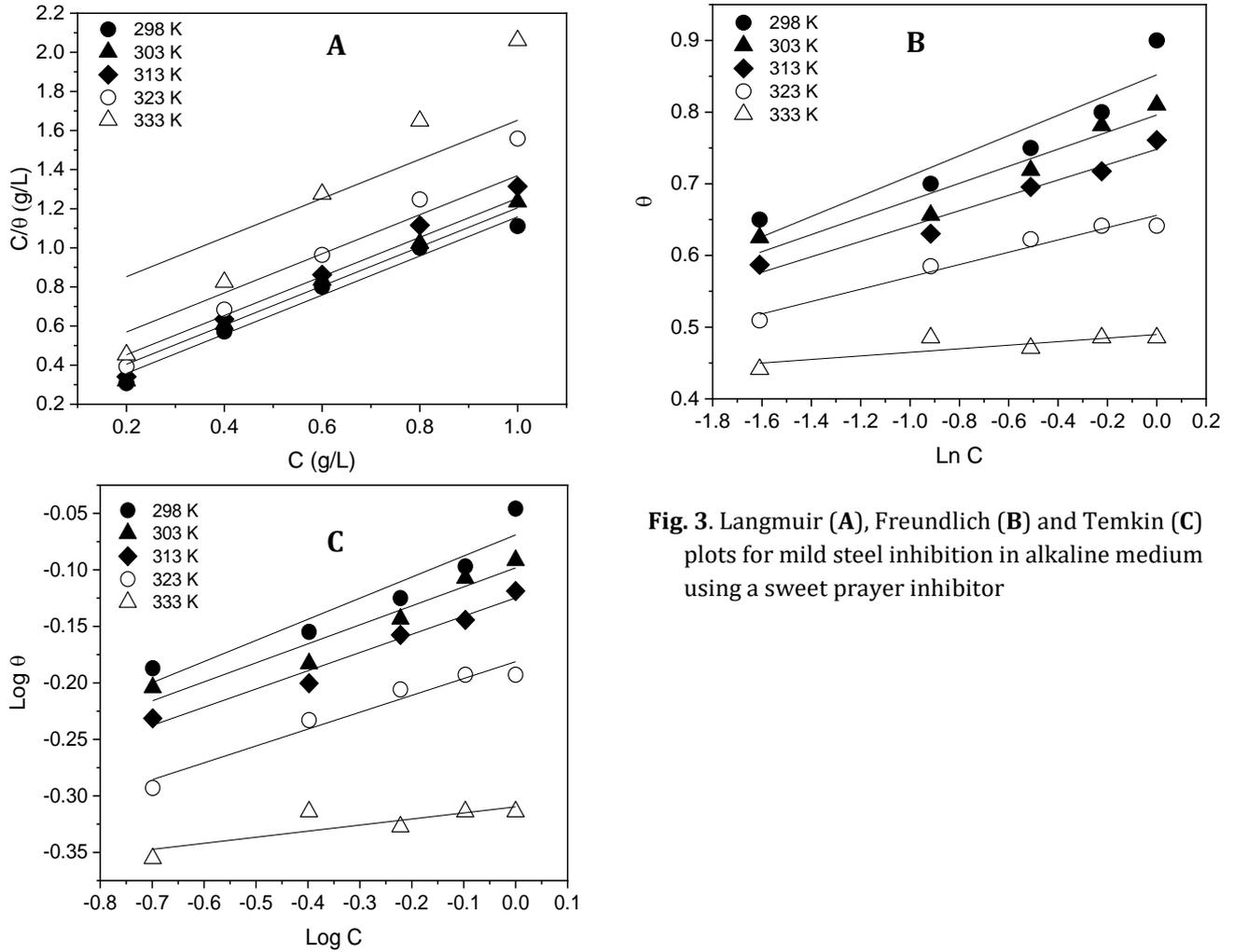


Fig. 3. Langmuir (A), Freundlich (B) and Temkin (C) plots for mild steel inhibition in alkaline medium using a sweet prayer inhibitor

Table 1. Isotherm parameters of the corrosion inhibition of sweet prayer leaf extract for mild steel in alkaline medium

Temperature (K)	Langmuir		Freundlich			Temkin		
	K_L	R_{adj}^2	K_F	$1/n_F$	R_{adj}^2	K_T	a	R_{adj}^2
298	0.1581	0.9800	1.537	-14.49	0.8713	0.8499	0.9445	0.8283
303	0.2045	0.9815	1.473	-10.18	0.9116	0.9218	0.5756	0.8948
313	0.2534	0.9651	1.449	-8.011	0.9625	0.9643	0.3414	0.9499
323	0.3692	0.9040	1.410	-5.519	0.9382	1.000	0.1701	0.9495
333	0.6523	0.7446	1.133	-3.229	0.5767	1.000	0.0000	0.5722

Thermodynamic study

Understanding thermodynamic parameters plays an essential role in understanding the inhibitive mechanism of corrosion process. **Figure 4a** shows the Arrhenius plots ($\ln CR$ versus $1/T$) for uninhibited and inhibited systems. The plots enabled the calculations of activation energy (E_a) and the Arrhenius constant or frequency factor (A). The values of E_a and A at different inhibition concentrations are presented in **Table 2**. The values of E_a increased with increasing inhibitor concentration. This indicates that the presence of inhibitor induced corrosion as the surface area decreased due to increase in energy barrier. Films are formed on the metal surface leading to lower corrosion rate as a result of high activation energy [31,32]. The results revealed that the lowest value (25.12 kJ/mol) of E_a was obtained in the absence of an inhibitor. However, the E_a values (37.81–62.42 kJ/mol) for inhibitive systems were higher than that of uninhibited system. The observation could be due to formation of an adsorptive film as earlier reported [33]. In addition, the value of E_a for physical adsorption is always < 80 kJ/mol, hence the mechanism of physical adsorption is supported in this research [34].

The values of standard Gibbs' free energy (ΔG°) of adsorption (293–333K) were estimated from the Langmuir plot because the Langmuir model was the best model that described the equilibrium isothermal data. The values of ΔG° were negative (**Table 2**), which is an indication that the adsorption process was spontaneous at all temperature values. This may also indicate the stability of the adsorbed film on the surface of the metal. The spontaneity of the corrosion system decreased slightly as the temperature increased. The values of ΔG° reported in this study were less than 20 kJ/mol, confirming the

occurrence of physical adsorption in which electrostatic interaction between the charged metal and charged molecule of inhibitor dominates. The values of standard enthalpy change (ΔH°) and entropy change (ΔS°) were evaluated from Fig. 4A (van't Hoff plot). As shown in **Table 2**, the value of ΔH° is negative, which indicates the adsorption process is an exothermic [35]. Similarly, the disorderliness between the metal surface and the inhibitor solution decreased because the value of ΔS° is negative.

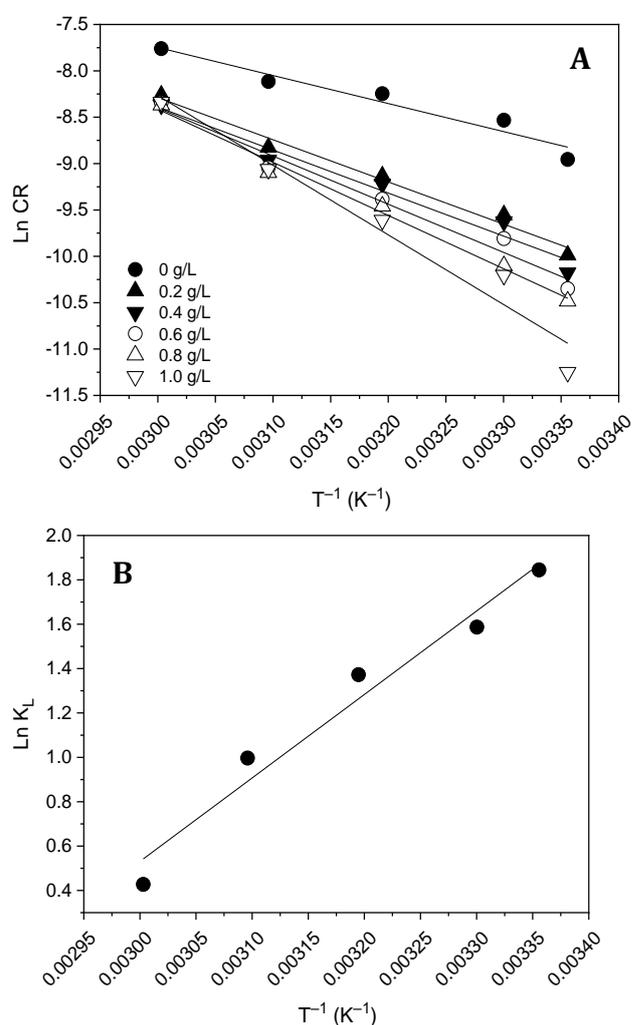


Fig. 4. Arrhenius (A) and van't Hoff (B) plots of the corrosion inhibition of sweet prayer leaf extract for mild steel in alkaline medium

Table 2. Thermodynamic parameters of the corrosion inhibition of sweet prayer leaf extract for mild steel in alkaline medium

Concentration (g/L)	E_a (kJ/mol)	A	R_{adj}^2	Temperature (K)	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/mol K)	R_{adj}^2
0.0	25.12	20.73	0.9228	298	-14.52	-31.28	-89.44	0.9543
0.2	37.81	2.259×10^5	0.9771	303	-14.12			
0.4	38.53	3.256×10^5	0.9521	313	-14.02			
0.6	43.17	1.502×10^7	0.9649	323	-13.46			
0.8	47.67	6.150×10^8	0.9861	333	-12.30			
1.0	62.42	1.810×10^{14}	0.9402					

Fourier transform infrared analysis

Fourier transform infrared (FTIR) spectra were obtained for the sweet prayer extract and the surface product of the metal steel inhibited in 1.0 mol/L NaOH solution after 24 h. The respective spectra are presented in **Figures 5A** and **B**. The spectra provided insights into the presence of functional groups present on the surface of the extract as well as the possible interactions between the inhibitor and the mild steel. For the sweet prayer extract, the peaks and assignments are as follows: 3790.81 cm^{-1} (free -OH), 3498.70 cm^{-1} (-OH stretching), 2908.22 cm^{-1} and 2894.49 cm^{-1} (CH_2 stretching mode), 1750.05 cm^{-1} and 1602.49 cm^{-1} (C=O stretching frequency), 1406.11 cm^{-1} (stretching mode of CH_2 bending vibration in CH_3), 1038.63 cm^{-1} (C-O stretching vibrations), and 688.12 cm^{-1} (C-C, C-O, and C-N). The peaks at 2325.90 cm^{-1} and 2392.70 cm^{-1} could be as a result of absorptions from triple and cumulated double bond systems [36].

The peaks and assignments of the FTIR spectrum of the mild steel surface film after immersion in the sweet prayer leaf extract in alkaline medium are as follows: 3400.53 cm^{-1} (-OH stretching), 2915.23 cm^{-1} and 2822.71 cm^{-1} (CH_2 stretching mode), 1668.30 cm^{-1} (N-H deformation mode), 1610.42 cm^{-1} (C=O stretching vibration), 1496.22 cm^{-1} (- CH_2 - bend), 1389.75 cm^{-1} (- CH_3 bend),

1098.43 cm^{-1} (C-C stretching and bending mode), 788.32 cm^{-1} (C-H of aliphatic and aromatic carbon), and 610.05 cm^{-1} (Fe-O band). The FTIR spectra of the leaf extract and that of mild steel after immersion in inhibitor's solution are slightly different. The absorption bands of the mild steel after immersion in inhibitor's solution shifted, indicating the interaction between mild steel and Sweet prayer leaf extract (37-38). The appearance of Fe-O band signified the interaction of the mild steel with the extract. The atoms (N, O, C, etc.), multiple bonds and aromatic ring are expected to increase the interaction of mild steel with the inhibitor (37).

Conclusion

In the present study, the corrosion inhibition potential of sweet prayer leaf extract for mild steel in alkaline medium was evaluated as a way of obtaining an enhanced anti-corrosion treatment. The leaf extract exhibited substantial inhibitory property. The adsorption of leaf extract onto the mild steel follows the mechanism of physical adsorption and Langmuir adsorption model. Inhibitor concentrations and temperature have profound effect on the corrosion efficiency of the leaf extract. The interaction of the Sweet prayer leaf extract with adsorbed species increased as the temperature increased.

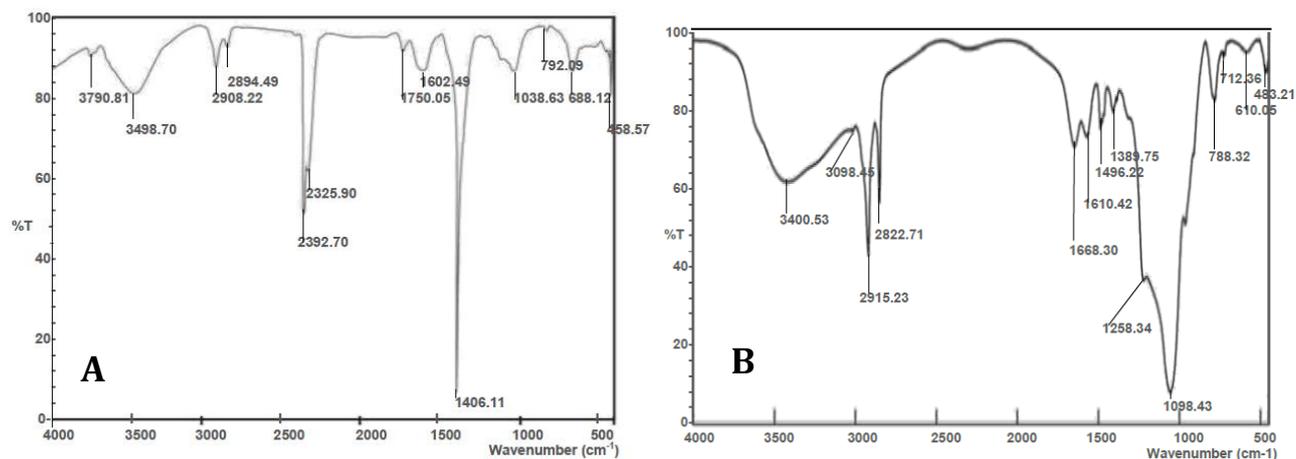


Fig. 5. Spectra sweet prayer leaf extract (A) and mild steel surface film after 24 h immersion in the sweet prayer leaf extract in alkaline medium.

The activation energies of the inhibited corrosion systems were greater than that of the uninhibited system (without inhibitor). The corrosion process was a feasible and spontaneous process. The disorderliness between the metal surface and the inhibitor solution decreased as a result of negative value of ΔS° . The atoms, such as N, O, and C, multiple bonds and aromatic rings present in the leaf extract are suggestive factors that might increase the interaction of mild steel with the inhibitor. The sweet prayer leaf extract could serve as a low-cost corrosion inhibitor of mild steel in alkaline medium.

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