

Influence of *Cissus populnea* stem extract on kinetics and thermodynamics of mild steel corrosion in acidic medium

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Abstract. The inhibition of mild steel corrosion in 1.0 M HCl and 0.5 M H₂SO₄ by *Cissus populnea* stem extract was investigated using weight loss measurements in the temperature range of 303 – 333 K. Results from the study revealed that *Cissus populnea* stem extract inhibited the corrosion of mild steel in both acid media. Inhibition efficiency of the stem extract increased with increase in the extract concentration but decreased with immersion time. The kinetics of the reaction aligned with a first order type. Thermodynamics investigations showed that *Cissus populnea* extract is adsorbed on the steel surface through physical interactions, and the adsorption process was endothermic and spontaneous. Langmuir, Freundlich and Temkin isotherm models best described the adsorption mechanism of the stem extract on the steel surface.

Keywords: corrosion inhibition, *Cissus populnea* extract, mild steel, kinetics, temperature dependence, corrosion rate.

1. Introduction

Most metals are inherently unstable and have the natural tendency to react with their environments to obtain lower energy states by forming their oxides and sulfides. Corrosion is the deterioration of metals/alloys by chemical attack or reaction with the environment. It is a complex problem, which is often difficult to eliminate completely, but it can be controlled by appropriate methods [1]. Steel is particularly used in most structural shapes such as beams, plates, bars, pipes, reaction vessels, tanks etc. Steel materials are very susceptible to attack in aggressive media and happen to be the most commonly exposed alloy in industrial environments hence, posing a great challenge to engineers, scientists and other stakeholders [2].

The use of inhibitors is one of the best options for protecting steel against corrosion. Corrosion inhibitors are substances that, when added to a particular environment, decrease the rate of the environment's attack on metal/alloy. Corrosion inhibitors help to extend the life span of an equipment, prevent system shut downs and failures, avoid product contaminations, prevent loss of heat transfer and preserve an attractive appearance of structural materials.

Corrosion inhibitors decrease or prevent the corrosion of a metal/alloy in an aggressive media by:

- i) Adsorption of inhibitor ions/molecules onto the metal surface;
- ii) Increasing or decreasing the anodic and/or cathodic reaction on the metal surface;
- iii) Decreasing the diffusion rate for reactants to the surface of the metal;
- iv) Decreasing the electrical resistance of the metal surface.

Inorganic and organic compounds have been employed and are still being used in the control of corrosion of metals and alloys [3-5]. However, most of

these compounds are expensive and toxic. In an attempt to find corrosion inhibitors which are environmentally safe and readily available, attention has been drawn to the use of ecofriendly materials such as extracts from plant parts [6 - 8].

Plant materials are cheap, eco-friendly, readily available and renewable source of materials. Materials obtained from plants are a great source of heterocyclic compounds. The adsorption of these compounds on metal/alloy surfaces have been reported to slow down the corrosion process in metals/alloys [9], hence, recent developments in the area of corrosion mitigation has been on the screening of the extract of different plant parts for their inhibitive properties [8, 10-12]. Alkaloids have been identified as the major chemical constituent of *Cissus populnea* (CP) stem extract. Other phytochemical compounds are flavonoids, saponins, tannins, cyanogenic and cardiac glycosides, anthraquinones and terpenoids [13, 14].

In a previous study, we reported the inhibitive properties of CP stem extract against the dissolution of aluminum in 0.5 M HCl [9]. The study showed that CP stem extract inhibited the dissolution of aluminum in the acid medium. This present study reports on the kinetics and thermodynamics of the corrosion of mild steel in 1.0 M HCl and 0.5 H₂SO₄ in the presence of *Cissus populnea* stem extract using weight loss measurements.

2. Experimental

2.1. Materials preparation

Mild steel coupons of dimension 3x2x0.11 cm² and percentage composition: Si - 0.03 %, P - 0.04 %, S - 0.04 %, Mn - 0.60 % and the rest Fe, were used for this study. Analar grade reagents were purchased from Emole Chemicals in Makurdi, Nigeria.

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2.2. Preparation of *Cissus populnea* stem extract

Fresh stems of *Cissus populnea* plant were procured at North Bank market, Makurdi Local Government Area of Benue State, Nigeria. The stem of the plant was first of all scrapped with the aid of a knife to remove the outer covering. The stem was later shredded using a knife. The shredded plant stem was soaked in hot water for about 15 minutes to allow for easy extraction and then squeezed with hands to remove the extract which was then filtered using a sieve to obtain a chaff free extract. The air-dried extract was blended with an electric blender into fine powder and kept dry in an air-tight container prior to use. Inhibitor test solutions of 0.2 g/L - 1.0 g/L concentrations were prepared by dissolving 0.05 g - 0.25 g CP extract powder in 250 mL 1.0 M HCl and 0.5 M H₂SO₄ respectively [9].

2.3. Weight loss measurements

Weight loss measurements were carried out as reported in previous studies [9]. Pre-weighed mild steel coupons were immersed with the aid of glass hooks in duplicates in 250 mL 0.5 M H₂SO₄ and 1.0 M HCl solutions in the absence and presence 0.2 g/L - 1.0 g/L CP stem extract. They were removed at specified time intervals, washed in water, cleaned with ethanol, dried with acetone and reweighed. The average loss in weight was taken as the difference between the initial and final weights of the mild steel. The corrosion rate of the mild steel as a function of time was investigated by carrying out the experiment for 24 - 168 h. The effect of temperature on the corrosion process of the steel in both the free acid and inhibited solutions was investigated in a similar manner for three hours with the water bath maintained at 303, 313, 323 and 333 K respectively.

Eqs. 1 to 4 were used to calculate the weight loss (g), corrosion rate (g·h⁻¹·cm⁻²), inhibition efficiency (IE, %) and the degree of surface coverage (θ) respectively.

$$\Delta w = w_2 - w_1 \quad (1)$$

$$CR(g \cdot h^{-1} \cdot cm^{-2}) = \frac{\Delta w}{At} \quad (2)$$

$$IE_{exp} = \left(1 - \frac{w_1}{w_2}\right) \times 100 \quad (3)$$

$$\theta = 1 - \frac{w_1}{w_2} \quad (4)$$

where w_1 and w_2 are the weight (g) of mild steel with and without the inhibitor, Δw is the weight loss of mild steel in time, θ is the extent of surface coverage of the inhibitor, A is the area of the metal coupon (cm²) and t is the immersion period (h) [9].

3. Results and discussion

3.1. Variation of weight loss of mild steel with CP stem extract

The corrosion rates of mild steel in 0.5 M H₂SO₄ and 1.0 M HCl solutions were investigated at 0.2 g/L, 0.4 g/L, 0.6 g/L, 0.8 g/L and 1.0 g/L CP stem extract concentrations for 24 h, at room temperature. The results obtained for the variation of the loss in weight of the mild steel with CP concentration is presented in Figure 1.

It is evident from Figure 1 that the loss in weight of the steel, hence, its corrosion rates in 0.5 M H₂SO₄ and 1.0 M HCl decreased with increase in the concentration of the stem extract. This suggests that increase in the concentration of CP stem extract led to a corresponding increase in surface coverage of the adsorbed inhibitor on the steel surface. Increase in surface coverage of an inhibitor on the surface of a metal/alloy has been reported to provide a barrier for the surface of the metal/alloy hence preventing further corrosion in aqueous media [8, 15]. Figure 2 illustrates the trend of IE of CP stem extract as a function of concentration. Inhibition efficiency of the stem extract was observed to increase with the inhibitor concentration signifying the concentration dependent nature of the corrosion inhibition process. This observation is corroborated by other researchers [9, 16]. Figure 2 also reveals that CP stem extract gave the optimum IE of 84.62% in 1.0 M HCl as compared to 72% in 0.5 M H₂SO₄. The higher value of IE in 1.0 M HCl maybe attributable to the presence of the chloride ions in HCl which could have improved the adsorption of the organic cations of the inhibitor by forming intermediate bridges between the positively charged metal surface and the positive end of the organic inhibitor [9, 17].

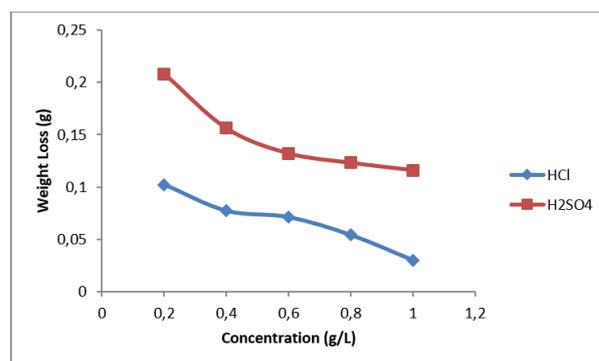


Figure 1. Weight loss of mild steel as a function of various concentrations of CP in 0.5 M H₂SO₄ and 1.0 M HCl.

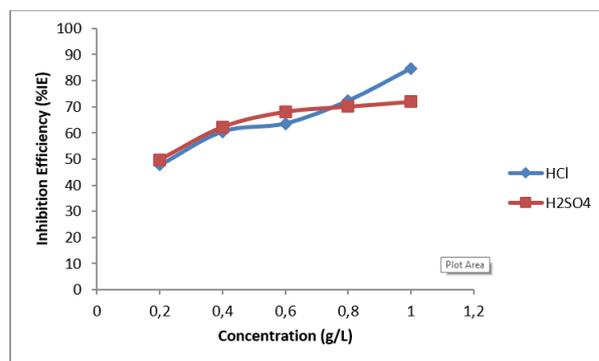


Figure 2. Inhibition efficiency as a function of various concentrations of CP.

3.2. Variation of weight loss of mild steel with time

In order to evaluate the effect of time on the corrosion rate of the steel coupons in the selected acid media, the corrosion rate of mild steel in 0.5 M H₂SO₄ and 1.0 M HCl solutions was separately investigated at 24 h, 48 h, 72 h, 96 h and 120 h using CP stem extract concentrations of 0.2 g/L, 0.6 g/L, and 1.0 g/L at room temperature. The results showed that corrosion rates of mild steel in both acid media increased with time, hence increasing the

magnitude of weight loss of the steel coupons in 0.5 M H₂SO₄ and 1.0 M HCl as illustrated in Figures 3 and 4 respectively. However, the magnitude of the weight loss of the coupons in the inhibited systems in both acid media were less compared to the ones in the free acid solutions (blanks). This shows that CP stem extract inhibited the corrosion of the steel coupons in both 0.5 M H₂SO₄ and 1.0 M HCl solutions.

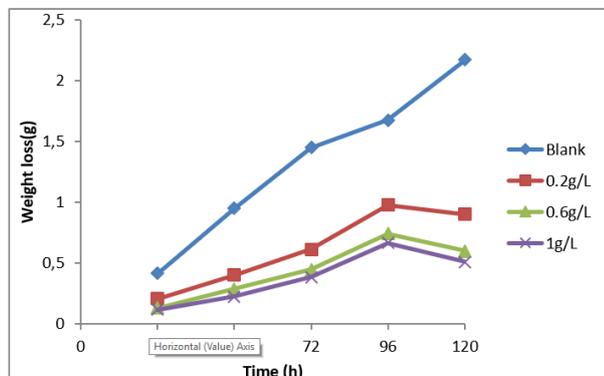


Figure 3. Weight loss of mild steel as a function of immersion time in 0.5 M H₂SO₄ in the absence and presence CP stem extract.

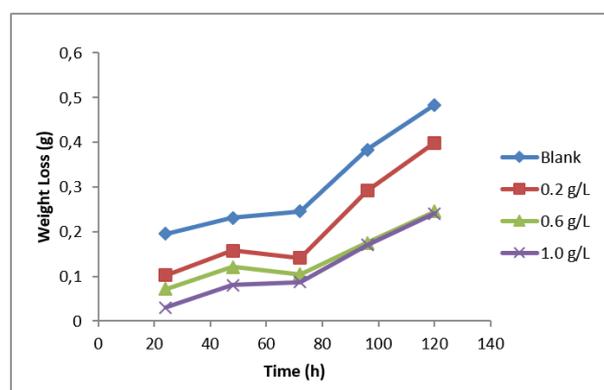


Figure 4. Weight loss of mild steel as a function of immersion time in 1.0 M HCl in the absence and presence CP stem extract.

Figures 5 and 6 show the trend in inhibition efficiency of CP stem extract with immersion time of the steel coupons in 0.5 M H₂SO₄ and 1.0 M HCl solutions respectively. Inhibition efficiency of the CP was observed to decrease with immersion time in both acid media. Though an optimum inhibition efficiency of 84% at an inhibitor concentration of 1.0 g/L was obtained in 1.0 M HCl compared to 76.29% in 0.5 M H₂SO₄, IE > 70% up to the immersion time of 72 h before decreasing to 61% at the end of the investigation in 0.5 M H₂SO₄.

The decrease in IE with immersion time has been attributed in similar studies to the desorption of the inhibitor species from the surface of the steel [8, 18, 19]. This phenomenon is characteristic of the physisorptive mechanism of adsorption of the inhibitor to the surface of the steel [18 - 21].

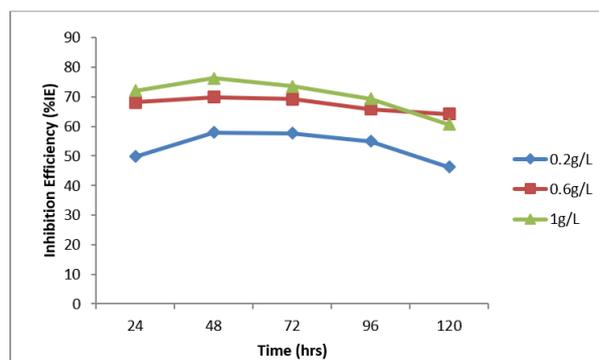


Figure 5. Inhibition efficiency as a function of time for the corrosion of mild steel in 0.5 M H₂SO₄.

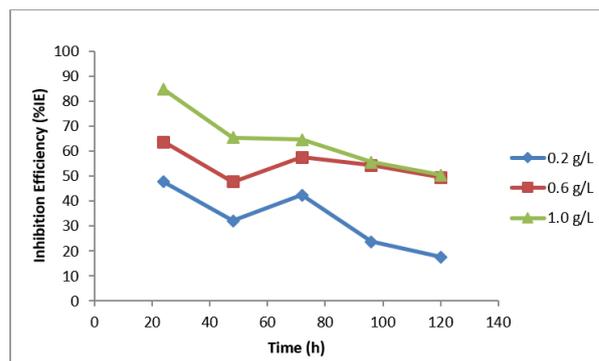


Figure 6. Inhibition efficiency as a function of time for the corrosion of mild steel in 1.0 M HCl.

3.3. Kinetics: rate constants (k) and half-lives ($t_{1/2}$)

The kinetics of the dissolution of the steel coupons in 0.5 M H₂SO₄ and 1.0 M HCl solutions in the absence and presence of 0.2 g/L, 0.6 g/L and 1.0 g/L CP stem extract were investigated at 303 K by plotting $-\log(\text{weight loss})$ versus time as presented in Figures 7 and 8 respectively. Linear plots with regression values that were close to unity were obtained indicating that the kinetics of the corrosion process aligned with the first order kinetics according to Eq. 5 [15, 22, 23]:

$$-\log(w) = \frac{k_1 t}{2.303} \quad (5)$$

where w is the weight loss of mild steel, k_1 is the rate constant for a first order reaction and t is time in hours.

The half-life ($t_{1/2}$) for the first order reaction is related to the rate constant as shown in Eq. 6 [15, 22, 23]:

$$t_{1/2} = \frac{0.693}{k_1} \quad (6)$$

Values of k_1 for the corrosion process and $t_{1/2}$ of mild steel in the absence and presence of the plant extract are presented on Tables 1 and 2. The rate constant is the tendency of the mild steel to corrode in the various test solutions [22]. It can be observed from Tables 1 and 2 that the values of k_1 for uninhibited test solutions were higher. The values of k_1 in the uninhibited systems however, decreased with increasing concentrations of CP stem extract revealing the inhibitive nature of the stem extract. This is corroborated by the increasing values of $t_{1/2}$ of mild steel in the presence of the stem extract. This implies that as the half-life increased, the corrosion rate

of the steel decreased thereby extending the life span of the mild steel in both acid media.

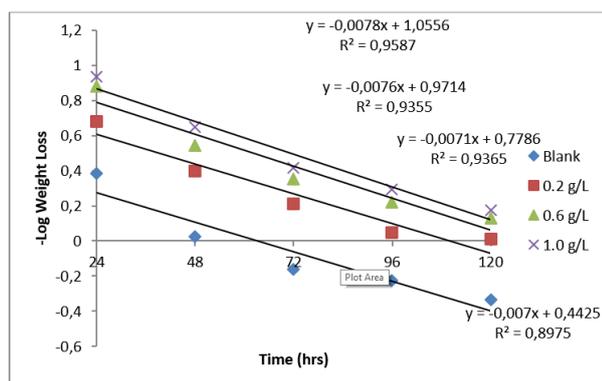


Figure 7. Plots of $-\log(\text{weight loss})$ versus time for the corrosion of mild steel in the absence and presence of CP stem extract in 0.5 M H_2SO_4 .

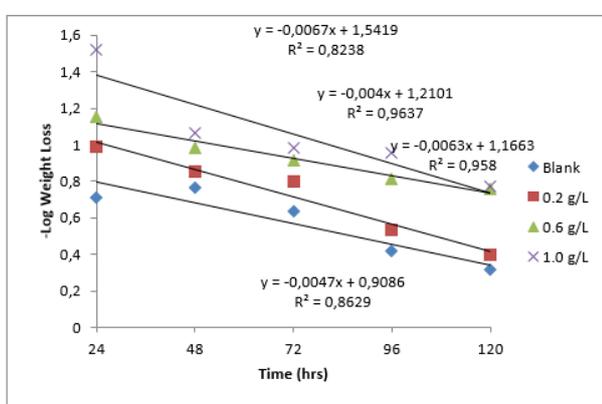


Figure 8. Plots of $-\log(\text{weight loss})$ versus time for the corrosion of mild steel in the absence and presence of CP stem extract in 1.0 M HCl .

Table 1. Kinetic parameters for the corrosion of mild steel in 0.5 M H_2SO_4 in the absence and presence of CP stem extract.

System	$k_1(10^{-3})$ [h^{-1}]	$t_{1/2}$ [h]	R^2
Blank	5.4644	126.82	0.897
0.2g/L	2.8970	239.21	0.936
0.6g/L	1.6934	409.23	0.935
1.0 g/L	1.5041	460.74	0.958

Table 2. Kinetic parameters for the corrosion of mild steel in 1.0 M HCl in the absence and presence of CP stem extract.

System	$k_1(10^{-3})$ [h^{-1}]	$t_{1/2}$ [h]	R^2
Blank	2.4337	284.75	0.862
0.2g/L	1.662	416.96	0.958
0.6g/L	1.151	601.87	0.963
1.0 g/L	0.875	770.42	0.823

3.4. Temperature considerations and thermodynamics

Thermodynamically, the average kinetic energy of reacting species in a system is a function of the temperature. The effect of temperature on the rate of corrosion of mild steel in uninhibited and inhibited solutions of 0.5 M H_2SO_4 and 1.0 M HCl at the temperature range of 303 - 333 K are presented in Figures

9 and 10 respectively. The plots revealed that the rates of corrosion of mild steel generally increased at higher temperature values both in the free acid solutions and in the presence of CP stem extract.

Reacting species become more energetic with a rise in temperature, this explains the increased rates of corrosion of the mild steel in both the uninhibited and inhibited test solutions at higher temperatures. This observation is corroborated by other researchers [16, 20, 24, 25]. However, the rate of corrosion of mild steel was relatively lower in the inhibited test solutions compared with the free acid solutions. Again, this signifies the inhibitive action of CP stem extract on the corrosion of mild steel in both acid media.

Figures 11 and 12 illustrate the trend of the inhibition of CP stem extract with a rise in temperature. The plots reflect decrease in inhibition efficiency of CP with temperature rise in both acid media. This lowering of inhibition efficiency values with increasing temperature has been reported by other scholars to be associated with physisorption [8, 19, 20].

Physisorption involves weak metal-inhibitor interactions which can be disrupted by any slight perturbation to the system. Such perturbations include the enhanced rates of hydrogen gas evolution at higher temperatures, which increasingly agitates the interface and also promotes dispersal of adsorbed inhibitor. The high temperature disruption of the system is sufficiently minimized at high extract concentration and the inhibiting effect is more or less stable [8].

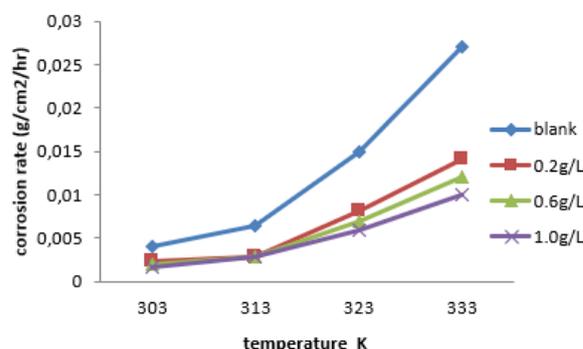


Figure 9. Corrosion rate of mild steel as a function of temperature in 0.5 M H_2SO_4 in the absence and presence of CP extract.

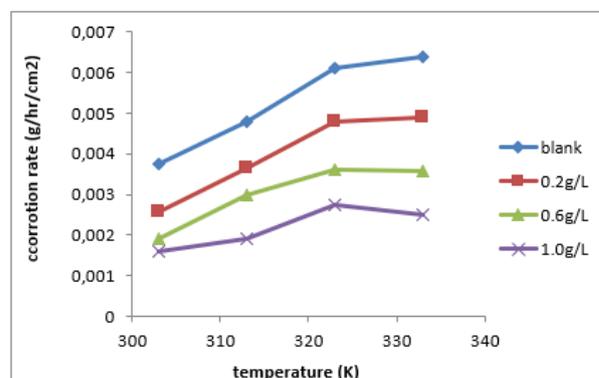


Figure 10. Corrosion rate of mild steel as a function of temperature in 1.0 M HCl in the absence and presence of CP extract.

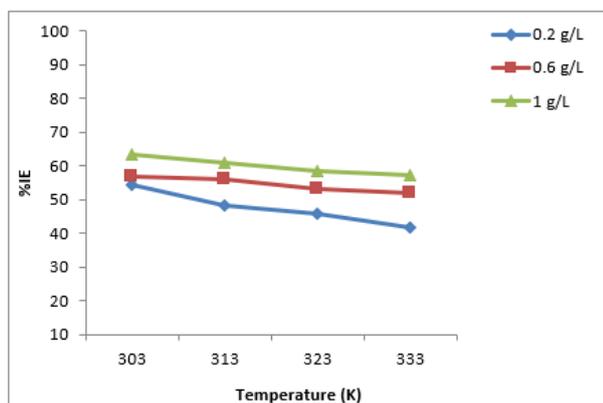


Figure 11. Inhibition efficiency of CP extract as a function of temperature in 0.5 M H₂SO₄.

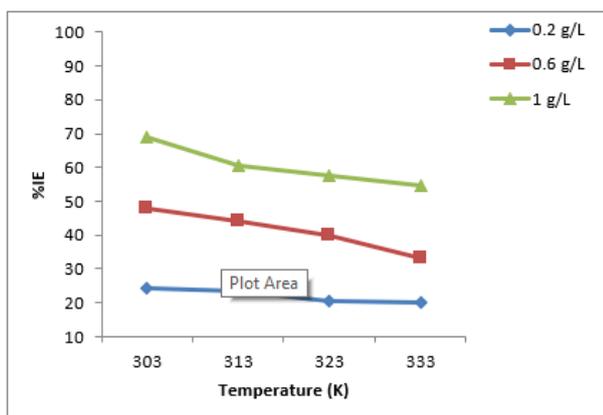


Figure 12. Inhibition efficiency of CP extract as a function of temperature in 1.0 M HCl.

The relationship between the corrosion rate of mild steel in the various test solutions and temperature was evaluated using the Arrhenius equation (Eq. 7).

Taking the logarithm of both sides of Eq. 7, Eq. 8 is obtained.

$$CR = A \exp^{-E_a/RT} \quad (7)$$

$$\log CR = \log A - \frac{E_a}{2.303 RT} \quad (8)$$

where CR is the rate of corrosion of the metal, A is the Arrhenius or pre-exponential factor, E_a is the apparent effective activation energy, R is the universal gas constant, and T is the absolute temperature of the system (K). If the corrosion rates of the metal at two temperatures T_1 (303 K) and T_2 (333 K) are known, then Eq. 8 can be transformed into Eq. 9:

$$\log(CR_2/CR_1) = \frac{E_a}{2.303R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right) \quad (9)$$

E_a values obtained in the presence of CP stem extract were observed to be lower than that in the uninhibited solutions (Table 3) which support the mechanism of physical adsorption. For physical adsorption, it is expected that E_a values should be less than 80.000 kJ·mol⁻¹ [9, 26].

Enthalpy (ΔH^*) and entropy (ΔS^*) of activation of the corrosion inhibition were evaluated using the Eyring Eq.:

$$\log CR/T = \left[\log \left(\frac{R}{nh} \right) + \frac{\Delta S^*}{2.303 R} \right] - \frac{\Delta H^*}{2.303 RT} \quad (10)$$

where CR is the corrosion rate at temperature T , R is the molar gas constant, n is Avogadro's constant 6.0225×10^{23} and h is the Planck's constant (6.6261×10^{-34} Js). A plot of $\log CR/T$ versus $1/T$ is a straight line graph with a slope of $(-\Delta H^*/2.303 R)$ and an intercept of $\left[\log \left(\frac{R}{nh} \right) + \frac{\Delta S^*}{2.303 R} \right]$ [9, 16, 20, 21].

Table 3 shows that the enthalpies of activation were all positive which reveals the endothermic nature of the dissolution process of the mild steel. Entropies of activation were all negative. Negative values of ΔS^* have been reported by Refat and Ishaq to represent the association mechanism of the corrosion process whereby a decrease in disorder takes place on going from the reactants to the activated complex [21, 26].

Table 3. Thermodynamic parameters for the adsorption CP stem extract on mild steel surface.

System	E_a [kJ mol ⁻¹]	ΔH_{ads}° [kJ mol ⁻¹]	ΔS_{ads}° [kJ mol ⁻¹]
0.5 M H₂SO₄			
Blank	49.46	48.44	-16.68
0.2 g/L	50.49	50.28	-15.84
0.6 g/L	50.36	50.3	-15.43
1.0 g/L	53.23	51.03	-15.13
1.0 M HCl			
Blank	12.72	50.68	-212.09
0.2 g/L	14.81	51.66	-203.32
0.6 g/L	17.41	61.02	-198.24
1.0 g/L	17.77	224.6	-154.67

3.5. Adsorption

The adsorption mechanisms of CP stem extract on the steel surface in the different acid media were investigated by fitting surface coverage (θ) values derived from weight loss data into various adsorption isotherms. Langmuir, Freundlich and Temkin isotherms (Figures 13, 14 and 15 respectively) were found to best describe the adsorption of CP stem extract on the steel surface with all the plots having linear slopes and regression coefficients (R^2) values close to unity [8, 9, 26].

The adsorption equilibrium constant, K_{ads} is related to the standard free energy of adsorption, ΔG_{ads}° by Eq. 11:

$$\Delta G_{ads} = -2.303 \times RT \log(55.5 K_{ads}) \quad (11)$$

Calculated values of K_{ads} and ΔG_{ads}° are presented on Table 4. The values of ΔG_{ads}° were negative and less than -40 kJ mol⁻¹ for all the three adsorption models. This implies that the adsorption of CP stem extract on the steel surface was spontaneous and physisorptive [20, 21, 24, 26].

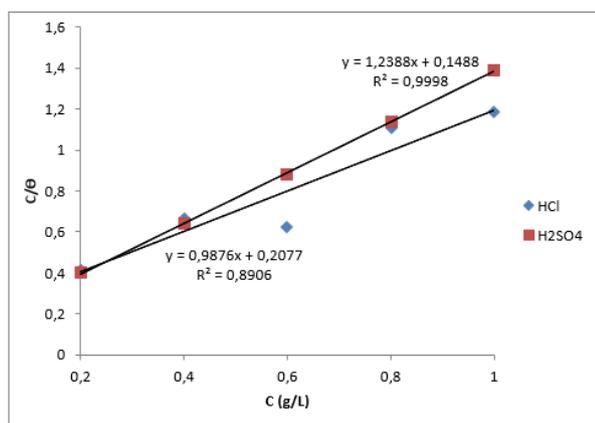


Figure 13. Langmuir isotherm for the adsorption of CP stem extract on the steel surface.

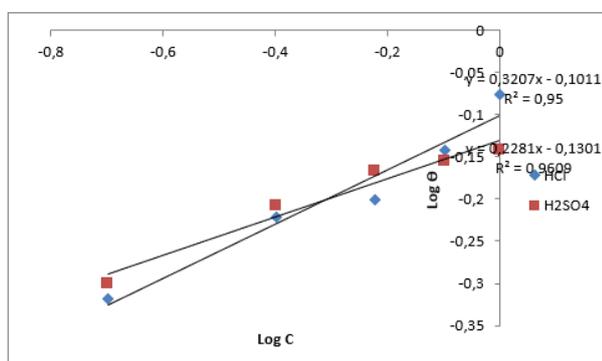


Figure 14. Freundlich isotherm for the adsorption of CP stem extract on the steel surface.

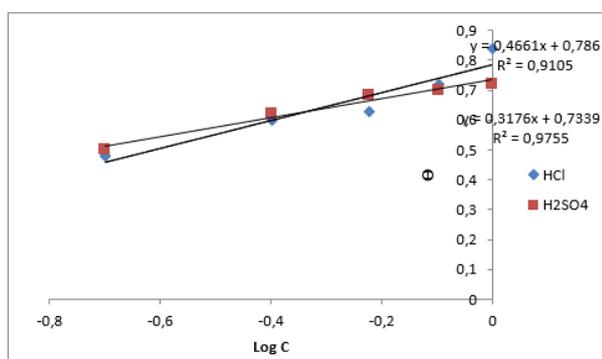


Figure 15. Temkin isotherm for the adsorption of CP stem extract on the steel surface.

Table 4. Adsorption parameters of CP stem extract on mild steel surface in 0.5 M H₂SO₄ and 1.0 M HCl respectively.

Isotherm	k_{ads}	R^2	ΔG_{ads} [kJ mol ⁻¹]
Langmuir			
HCl	4.831	0.890	-14.09
H ₂ SO ₄	6.757	0.999	-14.93
Freundlich			
HCl	1.262	0.950	-10.70
H ₂ SO ₄	1.690	0.960	-11.44
Temkin			
HCl	48.61	0.910	-19.91
H ₂ SO ₄	205.12	0.986	-23.53

4. Conclusion

Findings from this study revealed that *Cissus populnea* stem extract inhibited the corrosion of mild steel in

H₂SO₄ 0.5 M and 1.0 M HCl respectively. The inhibition process was concentration dependent. The kinetics of the reaction aligned with a first order type. The values of k_1 decreased with increasing CP concentrations while $t_{1/2}$ increased signifying the inhibitive effect of CP on the corrosion process in both acid media. Activation and standard free energy values of adsorption obtained showed that *Cissus populnea* extract adsorbed on the steel surface through physical interactions, and that the adsorption process was spontaneous. The positive values of enthalpy obtained in the study showed the endothermic nature of the adsorption process while the negative values of entropy reveal the association mechanism of the corrosion inhibition process whereby a decrease in disorder takes place on going from the reactants to the activated complex. Langmuir, Freundlich and Temkin isotherm models best described the adsorption mechanism of the stem extract on the steel surface.

Conflict of interest

The authors declare no conflict of interest.

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