Asian Journal of Applied Chemistry Research

8(4): 64-73, 2021; Article no.AJACR.69723 ISSN: 2582-0273

Thermodynamic and Adsorption Study of the Corrosion Inhibition of Mild Steel by *Aframomum chrysanthum* Extract in 0.1 M Hydrochloric Acid Solution

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Authors' contributions

This work was carried out in collaboration among all authors. Author IMP wrote the first draft of the manuscript. All other Authors read and made necessary correction. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/AJACR/2021/v8i430200 <u>Editor(s):</u> (1) Dr. Angélica Machi Lazarin, State University of Maringá, Brazil. <u>Reviewers:</u> (1) Suchetan P. A., Tumkur University, India. (2) P. Mahalingam, Arignar Anna Government Arts College, India. Complete Peer review History: <u>http://www.sdiarticle4.com/review-history/69723</u>

Original Research Article

Received 14 April 2021 Accepted 18 June 2021 Published 23 June 2021

ABSTRACT

The study reports the corrosion inhibition activity of methanol extract of *Aframomum chrysanthum* on mild steel in 0.1 M HCl, using gravimetry analysis. The weight loss of the mild steels was observed to increase with increasing immersion time. The inhibition efficiency (%IE) was also observed to have increased with increasing concentrations of the inhibitor but decreases with increasing immersion time. The effect of temperature change on the inhibition efficiency was also studied and it was observed that for every increase in temperature there was a corresponding increase in weight loss and decreased in the %IE. The highest values of %IE; 46.66, 56.66, 60.0, 80.0 & 93.33 was observed at temperature 303 K for 0.2, 0.4, 0.6, 0.8 & 1.0 g/L respectively. Activation energy (E_a) values and the enthalpy values reviews that the adsorption process followed

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a physisorption's mechanism. Change in enthalpy (Δ H) and entropy change (Δ S) of the reaction was positive indicating the endothermic nature and the spontaneity of the reaction. Three adsorption isotherms were tried on the inhibition process and only the Temkin isotherm gave the best fit with R² value of 0.903, describing the best adsorption mechanism. The adsorption equilibrium constants K_{ads} were positive, indicating the feasibility of the adsorption of the inhibitor to the metal surface. Gibb's Free Energy change of adsorption, Δ G_{ads} are negative indicating that the adsorption of the extract of *Aframomum chrysanthum* on mild steel surface is spontaneous. The values of Δ G_{ads} shows physisorption mechanism. All confirming that *Aframomum chrysanthum* extract is a good corrosion inhibitor on mild steel in 0.1 M HCI.

Keywords: Corrosion rate; mild steel; corrosion inhibition; physisorption; Aframomumm chrysanthum, temkin isotherm; Langmuir isotherm.

1. INTRODUCTION

Mild steel is the major item of construction of the cisterns in the petrochemical industrial processes involving storage of acids before use. It is one of the most commonly used ferrous metals in the industries for the fabrications and other engineering applications because of its relatively lower cost, availability of and efficiency. The commonest form of corrosion involves mild steel. especially in acid solution. This tends to limit its industrial applications on process safety and economics grounds coupled with environmental safety [1]. The consequence of the effect of safety corrosion including hazards and interruptions in plant operations have attracted the attention of researchers to the control and prevention of corrosion of mild steel.

The safest means of mitigating corrosion among other measures is corrosion inhibition, it is the best alternative as it allows for the use of cheap and readily available engineering materials and in industrial applications with minimal environmental effects [2].

Over the years, the inhibitive effects of many chemicals have been successfully exploited to mitigate corrosion. However, the use of chemicals as corrosion inhibitors has its limitations by its inherent toxicity, availability and cost, therefore the need to use naturally sourced inhibitors. Inhibitors of plant origin otherwise known as green inhibitors form an excellent kind of inhibitors because of their availability in nature, low toxicity, biodegradability and cheap cost. These substances generally contain a variety of natural products that are rich in conjugated aromatic structures and long aliphatic chains containing nitrogen, sulphur and oxygen heteroatoms with free electron pairs which readily bond with the metal surface. Extracts from plant leaves, stems and roots have shown to be effective inhibitors for metals and alloys in acidic medium [3-8]. The green inhibitors/organic materials inhibit activities of corrosion by adsorptions. Thus, inhibitors with adsorption capability will hinder the dissolution or corrosion reaction of such metal in the corrosive medium.

The knowledge of thermodynamic parameters associated with such inhibitors is fundamentals to an understanding of this efficiency of inhibition and corrosion rate, under corrosive medium at various temperature conditions. Adsorption and thermodynamic behaviour of organic inhibitors, especially extracts from plants on mild steel in the acidic medium have been widely reported [4-14]. The spontaneity of the adsorption process depends on the change in Gibbs free energy of adsorption (ΔG_{ads}). The magnitude and the sign of ΔG_{ads} also depend on the value of adsorption equilibrium constant, K_{ads}. When the rate of adsorption is higher than desorption, the ΔG_{ads} is indicates negative. which spontaneous adsorption. If ΔG_{ads} value less than – 20kJ/mol indicates physical adsorption while - 40kJ/mol and above indicates chemical adsorption [15-22]. The adsorption of Zenthoxylum alatum on mild steel in 5% and 15% HCl has been reported to have obeyed Langmuir adsorption isotherm [23]. Also, the adsorption of *Eucalyptus* leaf extract as eco-friendly corrosion inhibitor on mild steel in 0.5 M H₂SO₄ and 0.5 M H₃PO₄ solutions was reported to obeyed Temkin isotherm.[24] Corrosion inhibition of gum exudates of Azadiracta indica (GAI) on mild steel in 1.0 M HCI was investigated and the study revealed that the inhibitor effectively reduced the corrosion rate of mild steel according to Langmuir adsorption isotherm [25]. A similar report revealed spontaneous corrosion inhibition of some synthetic anti-biotic derivatives on mild steel in acidic medium, evaluated values for free energy change (-0.0, -39.60, -34.63 & 33.01 kJmol⁻¹) revealed a chemisorption process, and the adsorption data found well fitted in Langmuir isotherm [17].

The thermodynamics and kinetic inhibitions of aluminum in hydrochloric acid medium by date palm leaf extract have also studied, and the result showed that hot water extract of the date palm leaves has inhibition efficiency of 4 - 88% at the tested condition [21]. The reported work on the modelling and optimization of inhibition efficiency of Euphorbia heterophylla for mild steel in hydrochloric acid medium revealed the efficiency of Euphorbia heterophylla extract at the investigated conditions [22]. Aframomum chrysanthum plant has been used for other purposes like spicing of traditional dishes, flavouring of coffee, special kinds of bread and well as for medicine. There is no information on the use of this plant extract as a corrosion inhibitor. Hence this study focus was to investigate the corrosion inhibition efficiency of methanol extract of Aframomum chrysanthum on mild steel. It also aimed at evaluating the thermodynamics and adsorption parameters such as activation energy, enthalpy change, entropy change, adsorption equilibrium constant and free energy change of corrosion inhibition potentials of Aframomum chrysanthum plant extract on mild steel in a standard acidic medium.

2. METHODOLOGY

2.1 Extraction Process

The plant sample was airdried to a constant weight and later pulverized in to powder using a mortar and pestle. 500 g of pulverized plant sample was emptied into an air tight container and was extracted with 1000 ml of methanol in a batch extraction process. Methanol solvent was recovered using a rotary evaporator. The methanol extract was obtained at a yield of 90%.

2.2 Weight Loss Measurement

Determination of inhibition effect of *Aframomum chrysanthum* extract Corrosion inhibition effect of *Aframomum chrysanthum* on mild steel in 0.1 M HCl solution was investigated by determining the inhibition efficiency and corrosion rate. The inhibition efficiency and corrosion rate were obtained from the gravimetric method of analysis as reported by Malarvizhi and coworkers [25]. Corrosion rate (CR) inhibition efficiency (IE) and surface coverage (θ) were calculated using equations (1), (2), and (3) respectively

Corrosion rate
$$(g/h/cm^2) = \frac{\Delta W}{At}$$
 (1)

Where Δw is the weight difference of the coupons before and after in immersion(*g*), A is

the area of the metal coupon in (cm^2) , t is the period of immersion in hours.

Inhibition efficiency (IE) =
$$\left(\frac{W_0 - W_1}{W_0}\right) \times 100$$
 (2)

Surface Coverage (
$$\theta$$
) = $\left(\frac{W_0 - W_1}{W_0}\right) \times 100$ (3)

Where W_1 and W_0 are the weight losses of mild steel in the presence and absence of the inhibitors respectively.

2.3 Determination of Activation Energy (E_a)

The plot of log CR against $I/_T$ in eq (4) gives a slope from which the activation energy E_a was estimated. The Arrhenuis equation described the relationship between the corrosion rate (CR) and temperature (*T*) [12,15]

$$logCR = \frac{-E_a}{2.303RT} + \log y \tag{4a}$$

$$Slope = \frac{-E_a}{2.303R}$$
(4b)

 E_a is the activation energy, R is the gas constant, T is the temperature in Kelvin and y is the exponential factor.

2.4 Determination of Enthalpy and Entropy Change

The enthalpy change (ΔH) and entropy change (ΔS) were calculated using the transition state theory. An alternative of the transition theory is the Arrhenius Equation.

$$CR = \frac{RT}{Nh} \exp\left(\frac{\Delta S}{R}\right) exp\left(\frac{\Delta H}{RT}\right)$$
(5)

The linear form of the above equation is

$$Log \frac{CR}{T} = log \frac{R}{Nh} + log \left\{ exp\left(\frac{\Delta S}{R}\right) \right\} + log \left[exp\left(-\frac{\Delta H}{RT}\right) \right]$$
(5b)

Since
$$Log e^x = \frac{x}{ln10}$$
, equation (5*b*) becomes

$$\log \frac{CR}{T} = \log \frac{R}{Nh} + \left(\frac{\Delta S}{ln10}\right) + \left(\frac{-\Delta H}{2.303R}\right)$$
(5c)

$$\log \frac{CR}{T} = \frac{-\Delta H}{2.303R} \left(\frac{1}{T} \right) + \log \frac{R}{Nh} + \left(\frac{\Delta S}{2.303} \right)$$
(6)

Where h, is the plank constant, N is the Avogadro's number ΔS is the entropy change and ΔH is the enthalpy change. The change in enthalpy ΔH and entropy change ΔS were evaluated from the plot of $Log \frac{CR}{T}$ against 1/T.

The change in enthalpy \Rightarrow slope = $\frac{-\Delta H}{2.303R}$ (6b)

The entropy change = intercept = $log \frac{R}{Nh} + \frac{-\Delta H}{(6c)}$

2.303R

2.4.1 Adsorption isotherm and adsorption constant

The nature of corrosion inhibition has been deduced in terms of the adsorption characteristics of the inhibitor [23] on how the organic inhibitors adsorb on the metal surface [26]. The adsorption isotherm model that best describes the adsorption of Aframomum chrysanthum extract on mild steel in 0.1 M HCI solution was obtained by fitting the concentration and the degree of surface coverage of the inhibitor θ into the various adsorption isotherm models. These isotherms include Langmuir & Temkin were expressed in their linear form as:

The Langmuir adsorption isotherm model;

$$\frac{c}{\theta} = \frac{1}{K_{ads}} + C \tag{7}$$

Temkin adsorption isotherm model; [13,19]

 $\theta = lnC + K_{ads}$

(8)

2.4.2Determination of adsorption thermodynamics parameters

The expression for Gibb's free energy change of adsorption ΔG_{ads} presented in Equation 9 was used to investigate the feasibility and the nature of the adsorption.

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

 K_{ads} is the adsorption equilibrium constant obtained from the isotherm and the number 55.5 is the molar concentration of water in solution.

3. RESULTS AND DISCUSSION

3.1 Inhibition Efficiency, Corrosion Rates and Weight Loss with Effect of Time

The variation of weight loss (WL), inhibition efficiency (IE) and corrosion rate (CR) obtained from weight loss method of mild steel in 0.1 M HCI at ambient temperature as a function of concentration and time in the presence and absence of *Aframomum chrysanthum* is shown in Tables 1 to 3 respectively. The weight loss increased with increase in immersion time as shown in Table 1. For each immersion, there is decrease in weight loss as concentration of

inhibitor increases with the blank having the highest weight loss for each immersion time and for the entire of process. The inhibition efficiency of Aframomum chrysanthum on the mild steel increased with increasing concentration of inhibitors decreased with but increasing immersion time. The behaviour could be because of the increase in adsorption of inhibitor on the metal surface or the solution interface as the concentration increases. Highest efficiency of 85.71% was observed after 6 hours of immersion which suggests that Aframomum chrysanthum adsorption on the mild steel surface was completed within 6 hours, afterwards the aggressive action of the acid medium was increasingly felt than the absorbed inhibitor, leading to reduced inhibition efficiency with increased exposure time. The corrosion rate increased initially and subsequently decreased steadily as time increases. Weight loss, inhibition efficiency and corrosion rates of mild steel in blank and various concentrations of Aframomum chrysanthum are summarized in Tables 1 to 3.

3.2 Weight Loss, Inhibition Efficiency and Corrosion on Rate with Effect of Temperature

Gravimetric measurements under these conditions were undertaken for 3 hours immersion periods at 303K, 313K, 323K and 333K, to evaluate the effect of temperature change on the inhibitive effect of Aframomum chrysanthum. The inhibitor concentration for this study were 0.2g/L, 0.4g/L, 0.6g/L, 0.8g/L and 1.0g/L. The results obtained are presented in Tables 4 to 6. It was observed in the result shown that weight loss proportionally increases with temperature. For each temperature, the weight loss decreased as the concentration of the inhibitor increased. Inhibition efficiency increased with increase in concentration of the inhibitor. This evidently showed the effectiveness of Aframomum chrysanthum as a corrosion inhibitor for mild steel in the HCI, acidic medium. The reduction in inhibition efficiency with increasing temperature has also been attributed to nature of adsorption, in which the inhibitor is physically adsorbed at lower temperature while chemisorptions favoured is at higher temperature. In this case, physisorption was favoured. The corrosion rate increased with increase in temperature of immersion. The weight loss, inhibition efficiency and corrosion rate at different temperatures are summarized in Tables 4 to 6.

Extract conc. (g/L)			Weight Los	ss (g)	
	6 h	24 h	48 h	72 h	96 h
Blank	0.021	0.206	0.418	0.550	0.569
0.2	0.013	0.201	0.240	0.537	0.557
0.4	0.008	0.165	0.219	0.427	0.510
0.6	0.005	0.121	0.206	0.400	0.472
0.8	0.004	0.080	0.199	0.363	0.466
1.0	0.003	0.078	0.160	0.304	0.443

Table 1. Weight Loss (g) of mild steel at carious concentration of *Aframomum chrysanthum* at different time intervals

Table 2. Inhibition efficiency of various concentration of Aframomum chrysanthum at different time intervals

Extract conc. (g/L)	Inhibition Efficiency (%)						
	6 h	24 h	48 h	72 h	96 h		
0.2	38.100	2.4300	42.580	20.550	2.110		
0.4	61.900	19.900	47.600	22.360	10.370		
0.6	76.200	41.260	50.480	27.270	17.050		
0.8	80.950	61.170	52.390	34.000	18.100		
1.0	85.710	62.130	61.720	44.730	22.150		

Table 3. Corrosion rates of mild steel at various concentration of Aframomum chrysanthum and at different time intervals

Extract conc. (g/L)	Corrosion rate (g/h/cm ²)					
	6 h	24 h	48 h	72 h	96 h	
Blank	5.8×10⁻⁴	1.43×10 ⁻³	1.45×10⁻³	1.27×10⁻³	9.8×10 ⁻⁴	
0.2	3.6×10⁻⁴	1.40×10⁻³	8.3×10⁻⁴	1.01×10 ⁻⁴	9.7×10 ⁻⁴	
0.4	2.2×10⁻⁴	1.15×10⁻³	7.6×10⁻⁴	9.9×10 ⁻⁴	8.9×10⁻⁴	
0.6	1.4×10 ⁻⁴	8.4×10 ⁻⁴	7.2×10 ⁻⁴	9.3×10 ⁻⁴	8.2×10⁻⁴	
0.8	1.1×10⁻⁴	5.6×10⁻⁴	6.9×10 ⁻⁴	8.4×10 ⁻⁴	8.1×10⁻⁴	
1.0	8.3×10⁻⁴	5.4×10 ⁻⁴	5.5×10 ⁻⁴	7.0×10 ⁻⁴	7.6×10⁻⁴	

Table 4. Weight Loss (g) of mild steel at carious concentration of Aframomum chrysanthum at different temperature

Extract conc. (g/L)			Weight Loss (g)		
	303 K	313 K	323 K	333 K	
Blank	0.030	0.041	0.082	0.210	
0.2	0.016	0.036	0.062	0.165	
0.4	0.013	0.034	0.054	0.083	
0.6	0.012	0.028	0.053	0.080	
0.8	0.006	0.024	0.045	0.072	
1.0	0.002	0.020	0.033	0.068	

Table 5. Inhibition efficiency of various concentration of Aframomum chrysanthum at different temperature

Extract conc. (g/L)	Inhibition efficiency (%)						
	303 K	313 K	323 K	333 K			
0.2	46.66	12.20	24.39	21.42			
0.4	56.66	17.07	34.15	60.48			
0.6	60.00	31.71	35.37	61.90			
0.8	80.00	41.46	45.12	65.71			
1.0	93.33	51.22	57.31	67.62			

Extract conc. (g/L)		Co	rrosion rate (g/h/cn	n ²)	
	303 K	313 K	323 K	333 K	
Blank	1.70×10⁻³	2.20×10⁻³	4.60×10⁻³	1.160×10 ⁻²	
0.2	8.9×10⁻⁴	2.00×10⁻³	3.40×10⁻³	9.20×10 ⁻³	
0.4	7.2×10⁻⁴	1.90×10⁻³	3.00×10⁻³	4.60×10 ⁻³	
0.6	6.6×10⁻⁴	1.60×10 ⁻³	2.90×10⁻³	4.40×10 ⁻³	
0.8	3.3×10⁻⁴	1.30×10⁻³	2.50×10⁻³	4.00×10 ⁻³	
1.0	1.1×10⁻⁴	1.10×10 ⁻³	1.90×10⁻³	3.80×10 ⁻³	

 Table 6. Corrosion rates of mild steel at various concentration of Aframomum chrysanthum

 and at different temperatures

3.3 Thermodynamic Parameters: Activation Energy (Ea), Enthalpy Change (∆H) and Entropy Change

As shown in Fig. 1 and Table 7, the activation energy, E_a , was evaluated by plotting a graph of logCR against 1/T at various concentrations of the inhibitor. There was an increase in the activation from uninhibited (blank) solution to the solution with the concentration of 0.2 g/L, it then decreased from 0.4 to 0.6 g/L and finally increased and attained the highest value of the activation energy of about 93.86 kJ/mol. In general, the higher value of E_a in the presence of inhibitor as compared to blank indicates chemisorption mechanism [25]. The higher value of E_a in the presence of an inhibitor was due to the increased energy barrier. This further confirms the formation of a complex compound between the inhibitor and the mild steel [27]. The result explains the inhibitive tendency of Aframomum chrysanthum because of the increased barrier for the metal dissolution.

The values of enthalpy ΔH and entropy change, ΔS were obtained by plotting a graph of logCR/T against 1/T as shown in Fig. 2 and the values obtained presented in Table 7. The values for the energy changes were calculated using the slope and intercept of the graph. The values of enthalpy change were positive indicating the endothermic nature of the corrosion process on the mild steel. The highest value (89 kJ/mol) of enthalpy change was noticed at the 1.0 g/L inhibitor process whereas the least value was 47.73 KJ/mol for 0.4 g/L. However, the value of entropy change on the other hand was negative indicating that a decrease in disorderliness took place on going from reactants to the activated complex.

Observation have shown that the *Ea* values are more significant than the values of Δ H. This result indicates that the corrosion process must have involved a gaseous reaction.

3.4 Adsorption Isotherm

Different adsorption isotherms were tested in order to obtain more information about the interaction between the inhibitors and the mild steel surface. The various isotherms tested includes Langmuir and Temkin adsorption isotherms and linear regression coefficients (R^2) were used to determine the best fit. The values for each isotherm model are presented in Table 8 which was used to determine the most suitable model. The data fit into most into Temkin and Langmuir isotherms model but Temkin gives the best fit. Temkin isotherm with R^2 values of 0.903, best describes the adsorption mechanism of *Aframomum chrysanthum* extract on mild steel in 0.1M hydrochloric acid.

3.5 Adsorption Equilibrium Constant

Table 9 shows the adsorption equilibrium constants obtained at various temperatures from the intercept of the Temkin plot. The adsorption equilibrium constant K_{ads} are positive, indicating the feasibility of the adsorption of the inhibitor to the metal surface. The adsorption equilibrium constant increases with temperature up till 323 K. However, at 333 K, K_{ads} declines [28], this indicates that at higher temperature, the absorbed inhibitor tends to desorbs to denote back the mild steel surface.

3.6 Gibbs Free Energy Change of Adsorption

Also in Table 9, Gibb's Free Energy change of adsorption ΔG_{ads} gotten at different temperatures. As shown in the Tables 8 and 9. The ΔG_{ads} are negative indicating that the adsorption of the extract of *Aframonum chrysanthum* on mild steel surface is spontaneous and feasible. The values of ΔG_{ads} have shown that the mechanism of adsorption was physisorption for both considered isotherms.

System	Ea (KJ/mol)	ΔH (KJ/mol)	ΔS (J/mol/K)
Blank	53.54	50.91	-131.61
0.2 g/L	62.48	50.85	-106.08
0.4 g/L	52.58	47.73	-146.50
0.6 g/L	50.28	49.90	-140.39
0.8 g/L	68.18	65.56	-93.42
1.0 g/L	93.86	89.38	-22.18

Table 7. Result of thermodynamic parameters

1/T (K⁻¹)







Fig. 2. Plot of Log CR/T versus 1/T to determine ΔH and ΔS

Temperature (K)	Intercept	Slope	R^2	K _{ads} (M ⁻¹)	∆G _{ads} (kJ/mol)
303	0.367	0.789	0.854	2.725	-12.643
313	0.219	1.811	0.432	4.566	-14.404
323	0.717	1.276	0.231	1.394	-11.678
333	0.577	0.823	0.708	1.733	-12.642

 Table 8. Langmuir Adsorption Isotherm for Corrosion Inhibition of Mild Steel by Aframomum corroima in 0.1 M HCI

 Table 9. Temkin Adsorption Isotherm for Corrosion Inhibition of Mild Steel by Aframomum

 corroima in 0.1 M HCI

Temperature (K)	Intercept	Slope	\mathbf{R}^2	K _{ads} (M ⁻¹)	ΔG _{ads} (kJ/mol)
303	0.850	0.272	0.835	3.676	-13.193
313	0.466	0.244	0.903	4.098	-14.122
323	0.511	0.181	0.854	5.525	-15.376
333	0.733	0.275	0.829	3.636	-14.693



Fig. 3. Temkin isotherm for the adsorption of *Aframomum chrysanthum* onto mild steel surface in 0.1 M HCl at various temperatures

4. CONCLUSION

The study concluded that he adsorption of *Aframomum chrysanthum* extract on mild steel in 0.1M HCI is feasible spontaneous and by physical adsorption according to Temkin Isotherm model.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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Peer-review history: The peer review history for this paper can be accessed here: http://www.sdiarticle4.com/review-history/69723