Thermodynamic and Kinetic Parameters of Methanolic Extract of Leaves of *Cochlospermum Tinctorium a*s Eco-friendly Inhibitor for Corrosion of Mild Steel in HCL Medium

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Abstract

Thermodynamic, kinetic and adsorptive parameters of methanolic extract of leaves of *Cochlospermum tinctorium* as eco-friendly inhibitor for the corrosion of mild steel in HCl medium were evaluated through the weight loss method at 303 K and 311 K. The inhibition efficiency, %IE, increased with increase in extract concentration, but found to be better at 303 K than 311 K. The decrease in %IE with increase in temperature with the average value of 81.88 kJ/mol for activation energy, E_a, and the values of free energy, ΔG, for the adsorption process are all supportive of physical adsorption mechanism. The values of heat of adsorption, $Q_{ads,}$, are all negative, an indication that the adsorption process was exothermal. The half-life, $t_{1/2}$, value increased with increase in inhibitor concentration and low at high higher, implying the inhibitor performance increased with increase in the extract concentration and decreased with rise in temperature. The data best fitted the Langmuir adsorption isotherm.

Key Words: *Cochlospermum tinctorium*, eco-friendly inhibitor, thermodynamic parameter, half-life, adsorption isotherm.

Introduction

Humanity has now come to terms with the repercussions and the environmental accidents that our behaviour has impacted on the earth, putting it in a compromising future. Chemical weapons, pesticides, fertilizers, industrial effluents, paints and organic solvents have changed the health of plants and world inhabitants to the point of contaminating even the most remote places on the planet (Hodges*et al.,* 2010). As a result, man at the moment is environmentally conscious, trying to be prudent and transparent in the use of chemicals. This has resulted in the search for eco-friendly chemicals within the scientific and engineering circles. The focus on plant extracts, away from synthetic inhibitors, as eco-friendly inhibitors for metal corrosion prevention is due mainly to economic reasons, strict environmental regulations, renewability and biodegradability (Arora *et al*., 2007; Selvi*et al*., 2009, Alaneme and Olusegun, 2012). The extracts such natural substances contain mixtures of compounds having oxygen, nitrogen, sulphur and phosphorus which help in corrosion inhibition process (Jai and Ali, 2008). The inhibition efficiency of the compounds containing these elements in their structures as corrosion inhibitors can be attributed to the number of mobile electron pairs present, the orbital character of free

electrons and electron around the atoms (Arora *et al*., 2007). Quite a number of extracts from plants have been investigated (Avwiri and Igho, 2003; El-Etre, 2003; Orubite and Oforka, 2003; Benabdellah *et al*, 2006; Oguzie, 2006; Eddy *et al*., 2008; Eddy and Ebenso, 2008; Adejo *et al*., 2010, Nnanna *et al.*, 2012, Adejo *et al*., 2013).

Cochlospermeae is a well-known plant family in herbal medicine. The species *Cochlospermum tinctorium*, the plant of interest in this work, is a widely distributed plant within the savanna areas of West and Central Africa (Etuk*et al*., 2009). This plant is called balagarde in Hausa, abanzi in Igbo, cewatu in Yoruba, ukpatafulo in Idoma and kpavande in Tiv, all these being the tribes in Nigeria, signifying the importance of the plant in the country. In Mali and Ivory Coast the plant is widely used for the treatment of jaundice, malaria, abdominal pain, wound healing, haemorrhoids, intestinal worms, bilharizias and hepatitis (Togola*et al*., 2005). Nergard *et al.,* (2005) has shown that pectic polysaccharides obtained from the plant are responsible for its bioactivities.

In this study, methanol extract of leaves of the plant was put into a different use; as inhibitor of corrosion of mild steel in HCl solution. The thermodynamic, kinetic and adsorptive parameters of the inhibition process were evaluated at two temperatures of 303 Kand311 K, a narrow temperature range employed to maintain the molecular structure(s) of the component(s) responsible for the inhibition process in the course of the experiment.

Materials and Method
Material preparations

A sample of mild steel of composition (%W); Mn(0.56), P (0.04), C(0.27), Si(0.25), S(0.04) with rest being Fe, was obtained from Department of Mechanical Engineering, University of Agriculture, Makurdi-Nigeria. Coupons of the dimension of 2 cm x 2 cm x 0.13 cm (with a 2.5 mm hole drilled through an edge of each) were prepared as described in Eddy and Ebenso (2008).

Leaves of the plant were shade-dried, ground and 20 g soaked in 200 ml of absolute methanol in a 250 ml volumetric flask, which was properly corked and kept, with occasional shaking, for 48 hours. The extract obtained, therefrom, was filtered and the methanol was carefully evaporated at 321 K, to available any possible structural changes to any component of the extract, using Clifton Model thermostated water bath. Stock solutions of the extract of the concentrations of 0.1, 0.2, 0.3, 0.4 and 0.5 g/dm^3 were prepared in 2 M HCl. Analytical grade reagents were all used, with double distilled used for all solution preparations.

Method

The Weight loss measurement was carried out as reported in Orubite-Okorosaye and Oforka (2004) at 303 Kand 311 K in the thermostated water bath. The first part of the experiment involved weighing the coupon using ae Adam AFP weighing balance (4100 Model) and suspending it in a beaker containing the corrodant, which was a 100 ml of the acid in 250 ml beaker, using a thread passed through the hole to tie it to a retort stand. The coupon was retrieved from the solution at an interval of 2 hours for 12 hours. The procedure was repeated using solution of different concentrations of the extract introduced into the corrodant. At retrieval, each coupon was dipped immediately into a saturated ammonium acetate solution at room temperature to terminate the reaction, washed with distilled water and then dried in acetone, kept in a desiccator to reach ambient temperature and carefully re-weighed. The weight loss was calculated using the expression,

$$W = (W_i - W_f) \qquad (1)$$

where W is weight loss of coupon, W_i is the weight before immersion and W_f the final weight of the coupon after retrieval.

The inhibition efficiency, *%IE,* and degree of surface coverage, *θ*, were calculated

by the use of equations (2) and (3), respectively.

$$\% IE = \left(1 - \frac{W_1}{W_2}\right) x \ 100 \qquad (2)$$

$$\theta = 1 - \frac{W_1}{W_2} \qquad (3)$$

where W_1 and W_2 are weight losses of the coupon in the presence and absence of inhibitor, respectively, at the same temperature.

The corrosion rate, CR, of the mild steel was obtained using the NACE (1984) formula (equation 4) (Orubite-Okorosaye and Oforka, 2004).

$$CR_{(mdd)} = \frac{534W}{DAT} \qquad (4)$$

where W is weight loss (mg), D the density of the mild steel (g/cm^3), A the surface area of the coupon in cm^2 and T the exposure time in hours and mdd is mg/dm/day.

Results and Discussion
Effect of the concentration and temperature

The extract has inhibitive effect on the corrosion of mild steel in HCl medium as it can clearly be seen from Table 1 that the corrosion rate decreases and IE% increases with increase in the extract concentration at the two temperatures. However, the inhibitor performance is higher at the lower temperature, suggestive of physical adsorption mechanism for adsorption of the

inhibitor onto the surface of the metal (Siaka*et al.,* 2012).

Thermodynamic parameters

The activation energy, E_a, of the corrosion process was evaluated by the used of Arrhenius equation (equation 5) (Atkins, 1982; Eddy *et al.*, 2008).

$$CR = A exp^{\left(\frac{-E_a}{RT}\right)} (5) \qquad (5)$$

where CR is corrosion rate, A the Arrhenius parameter, E_a the activation energy of the corrosion reaction, R the gas constant and T the temperature in Kelvin. A linearised form of equation (5) is given thus;

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

For corrosion rates at two temperatures (T_1 and T_2) the expression below is obtained from equation (6).

$$\frac{logCR_2}{CR_1} = \frac{E_a}{2.303R\left[\frac{1}{T_1} - \frac{1}{T_2}\right]} \qquad (7)$$

Corrosion rates were evaluated at 303 K and 311 K. In spite of the closeness of the temperatures it was found there exist a significant difference in the corrosion rate between the two temperatures. Calculated corrosion rate and inhibition efficiency at the two temperatures are shown in Table 1, while Table 2 shows the values of activation, heat of adsorption, free energy and half-life for the corrosion process.

Table 1. Values of Corrosion Rate (CR) and Inhibition Efficiency *(%IE)*

Conc. (g/L)	CR (mdd)		%IE	
	303 K	313 K	303 K	311 K
Blank	0.3872	0.7010	–	–
0.1	0.1561	0.4798	59.68	31.55
0.2	0.1499	0.3966	61.29	43.42
0.3	0.1374	0.3256	64.51	53.55
0.4	0.1124	0.3053	70.97	56.44
0.5	0.1062	0.2789	72.57	60.21

Table 2. Evaluated Values of Activation Energy (E_a), Heat of Adsorption (Q_{ads}), Free Energy (ΔG_{ads}) and Half-life ($t_{1/2}$) for the Inhibition Process.

Conc (g/dm³)	E_a (kJ/mol)	$-Q_{ads}$ (kJ/mol)	$-\Delta G_{ads}$ (kJ/mol)		$t_{1/2}$ (h)	
			303 K	311 K	303 K	311 K
Blank	49.36	-	-	-	-	-
0.1	93.37	114.28	16.91	14.02	35.02	7.39
0.2	80.91	70.95	15.33	13.86	34.67	9.63
0.3	71.74	44.59	14.66	13.87	40.08	11.73
0.4	83.09	62.19	14.68	13.43	44.73	11.91
0.5	80.29	54.72	14.32	13.25	49.17	14.88

The value of Ea was found to be higher in the presence of the extract than pure corrodant. E_a values in the presence of the inhibitor ranged from 71.74 to 93.37 kJ/mol. The values of E_a are close to 80 kJ/mol, the threshold value for physical adsorption mechanism, which is clearly supportive of physical adsorption mechanism (Eddy *et al.*, 2008).

$$Q_{ad} = \frac{2.303R\left[\log\left(\frac{Q_2}{1-Q_2}\right) - \log\left(\frac{Q_1}{1-Q_1}\right)\right]T_2T_1}{T_2 - T_1} \quad (8)$$

Free energy values for the adsorption process were evaluated by the use of expression;

$$\Delta\overline{G_{ads}} = -2.303T\log(55.5K) \quad (9)$$

where $\quad K = \dfrac{\theta}{(1-\theta)[c]}$

C is the concentration of the extract and 55.5 is concentration of water expressed in moles (i.e. ~ 1000 g/dm³) (Bastidas *et al.*, 2005). Calculated values of ΔG_{ads} are all negative, an indication of spontaneity of adsorption process. However, ΔG_{ads} values are more negative at 303 K than 311 K, implying that the spontaneity of the adsorption process and hence the stability of the adsorbed layer of the inhibitor is higher at lower temperature (Atkins, 1982).

Kinetic Consideration

Studies have shown that the corrosion reaction of metals is of first-order kinetics (Okafor *et al.*, 2010; Nair *et al.*, 2010) and therefore first-order equations were employed. Values of the half-life, $t_{1/2}$ in hours for the corrosion process in presence of the inhibitor were evaluated by the use of equation (10) (Atkins, 1982; Onen and

Q_{ads} values evaluated through equation (8) are all negative (average = -87.01kJ/mol), an evidence of exothermic process for the adsorption of this extract onto the mild steel surface. These values are large suggesting that the heat of adsorption for the process was quite large. The negative values also show that the adsorption and hence the efficiency of the inhibitor will decrease with increase in temperature (Eddy and Ebenso, 2008).

Nwufo, 2010

$$t_{\frac{1}{2}} = \frac{0.693}{k} \quad (10)$$

where k is the rate constant for the corrosion reaction, which was obtained by the use of the integrated rate equation for first-order reaction (equation 11).

$$lnA_t = lnA_o - kt \quad (11)$$

A_o is the initial weight of the steel sample and A_t is the weight left after the corrosion reaction. The $t_{1/2}$ was found to increase with increase in the inhibitor concentration (Table 2), implying a decrease in k with increase in the concentration. This means that the inhibition process increased with the concentration of the inhibitor. Values of $t_{1/2}$ were found to be higher at 303 K than at 311 K, indicative of the fact that the corrosion reaction increased at the higher temperature,

another evidence in support of physical adsorption mechanism as earlier stated.

Adsorption Consideration

Common adsorption isotherms frequently used in trying to understand the mechanism of inhibition of corrosion reactions are the Langmuir, Frumkin, Freundlich, Temkin, Flory-Huggins, El-Awady and Bockris-Swinkels. A general equation to represent these isotherms is given by equation (12) (Eddy *et al.*, 2008).

$$f(\theta, x) exp^{-2a\theta} = KC \quad (12)$$

where $f(\theta, x)$ is configuration factor that is dependent upon the physical model and the assumption underlying the derivation of the isotherm, θ the degree of surface coverage, C the concentration of inhibitor in the electrolyte, x is the size ratio,

$$\frac{C}{\theta} = \frac{1}{K} + C \quad (13)$$

$$log\theta = logK_F + \frac{1}{n_F}logC \quad (14)$$

$$log\theta\frac{\theta}{(1-\theta)C} = logK + \frac{2a\theta}{2.303} \quad (15)$$

$$log\theta\frac{(\theta)}{1-\theta} = logK' + ylogc \quad (16)$$

The adsorptive parameters obtained from the isotherm plots given in Table 3. From the coefficient of determination, R^2 values it is evidently clear that the Langmuir isotherm is most applicable model for the adsorption of this extract onto the mild steel surface, while Freundlich and El-Awady are applicable at 311 K.

The adherence of the adsorption to the Langmuir isotherm has been argued by Orubite-Okorosaye and Oforka (2004) to signify that the extract covered both the anodic and cathodic sites on the metal surface. The argument is quite plausible considering the fact that Langmuir isotherm involves monolayer over the entire surface. The positive value of K signifies favourable adsorption, and judging from the values at the two temperatures, the adsorption process was more favourable at the lower temperature.

The parameter n_F in the Freundlich isotherm is related to intensity of adsorption and it varies with heterogeneity of the material (Chatterjee *et al*, 2009). The values of Freundlich isotherm constants n_F and K_F obtained are 0.7853 and 2.4444 and 0.0819 and 0.8287 at 303 K and 311 K, respectively. n_F values are within the normal beneficial adsorption of between 1 and 10 (Adejo *et al.,* 2008), but higher at higher temperature with even a better R^2. This is, however, in invariance with the observation in the case of Langmuir. According to Khadom *et al* (2010), typical value of n_F is 0.6, which implies that this adsorption process cannot be reasonably

Table 3: Values parameters for adsorption isotherms

Isotherm		303K	311 K
Langmuir	R^2	0.9931	0.9973
	K	17.1527	5.1840
Freundlich	R^2	0.8667	0.9811
	n_F	0.7853	2.4444
	K_F	0.0819	0.8287
Frumkin	R^2	0.6403	0.8150
	α	-2.8589	-2.5385
	K	3.2×10^5	55.6929
El-Awady	R^2	0.8441	0.9904
	y	0.3744	0.7507
	K	22.5595	3.6164

The negative sign of αin Frumkin isotherm signifies existence of molecular interaction within the adsorbed layer. The R^2 are relatively low, and therefore, this adsorption cannot also be reasonably modeled by the isotherm. The fact that R^2 values are better in case of Langmuir isotherm than Freundlich and Temkin isotherms implies that the adsorption is monolayer and close to ideal.

K corresponding to equilibrium constant for the adsorption isotherm in the case of El-Awady and is given by,

$$K = \left[K^{\left('(\square)\right]^{\frac{1}{y}}}\right) \tag{17}$$

where y is the number of inhibitor molecules occupying one active site of the adsorbent. Value of *y > 1* implies the formation of multilayers of inhibitor on the surface of metal; value of *y < 1* indicates the formation of monolayer on the metal surface (Niamien*et al*., 2012; Umasankareswari and Jeyaraj, 2012). y values are less than unity at the two temperatures, an indication of monolayer adsorption.   1/y is the number of surface active sites occupied by one inhibitor molecules (Ayobe *et al*., 2012), and its values are 2.6709 and 1.3321 at 303 K and 311 K, respectively.

Conclusion

This study has demonstrated that *Cochlospermum tinctorium* methanolic leaves extract has good inhibitive action against the corrosion of mild in HCl medium. The inhibition action, which was found to be spontaneous and exothermic, was better at lower temperature and can be better modeled by Langmuir isotherm.

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