RESOLUTION OF ADSORPTION CHARACTERISATION AMBIGUITY THROUGH THE ADEJO-EKWENCHI ADSORPTION ISOTHERM: A CASE STUDY OF LEAF EXTRACT OF *HYPTIS SUAVEOLEN* POIT AS GREEN CORROSION INHIBITOR OF CORROSION OF MILD STEEL IN 2 M HCI

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Abstract

The resolution of ambiguity usually associated with the characterisation of adsorption process of the plant extracts as corrosion inhibitors for metal corrosion was achieved through the use of Adejo-Ekwenchi adsorption isotherm using the case of corrosion inhibition of mild steel corrosion in 2 M HCl by ethanol leaf extract of *Hyptis suaveolens* poit. The study was carried out using the weight loss method at the temperature range of 301 K to 313 K. The inhibition efficiency, % IE, was found to decrease with increase in temperature, suggestive of physical adsorption (physisorption) mechanism. The observed decrease in the value of parameter b of the Adejo-Ekwenchi isotherm is a confirmation of a physisorption for adsorption of this extract onto the metal surface. By the use of R^2 and ΔG_{ads} this adsorption would be thought to well fit into Langmuir, Freundlich, Temkin and El-Awady. But the adsorption process best fit the Langmuir isotherm, as resolved through the Adejo-Ekwenchi isotherm. Implicitly, this is one clear case of emerging trends for sustainable development and human capacity building as focus is now on plant extracts, away from synthetic inhibitors, as eco-friendly inhibitors for metal corrosion prevention due reasons like cost, strict environmental regulations, renewability and biodegradability. The design and development of new and effective inhibitors will only be possible if adsorption of inhibitor onto the metal surface is clearly understood.

Keywords: adsorption; mild steel; hyptis suaveolens poit; adejo-ekwenchi isotherm; physisorption

INTRODUCTION

Adsorption is a process that occurs when gas or liquid solutes (adsorbate) accumulate on the surface of a solid or a liquid (adsorbent). Process of adsorption is present in many physical, biological and chemical systems and its applications are copiously found in industrial processes like: - activated charcoal, synthetic resins, water purification, drug action, corrosion inhibition, etc. (Al-Muhtaseb *et al.*, 2008; Graf *et al.*, 2012).

In the adsorption process, the relationship between the amount of the adsorbed substance to its bulk concentration at a particular temperature and pH is known as adsorption isotherm (Ho et al., 2002). Adsorption isotherm is usually described by an isotherm equation whose parameters express the surface properties and affinity of the adsorbate and from which the mechanism of adsorption process can be deduced (Radnia et al., 2011; Adejo et al., 2012). Adsorption isotherms are of extreme importance in understanding of many processes. For example, in corrosion inhibition process the mechanism of inhibition can better be understood through the use of such isotherms (Nnanna et al., 2012; Adejo et al., 2012). Details of mechanism of inhibition of metallic corrosion, design and even development of new and effective inhibitors will only be possible if adsorption of inhibitor onto the metal surface is clearly understood (Abiola & Otaigbe, 2008).

A number of authors have opined that discussion of the adsorption behaviour of plant extracts as inhibitors in terms of thermodynamic parameters is inadequate due to the simple fact that extracts are usually mixtures of many components of different molecular thermodynamic parameters are molar quantities (Lebrini et al., 2011). Therefore, adsorption isotherms may come to rescue in this regard. The fitness of adsorption data into an isotherm is usually ascertained through the value of regress coefficient, R^2 , while ΔG_{ads} values can used to determine whether the process is physisorption or chemisorption. But as has been argued by Ho et al., (2002), the transformation of non-linear isotherm equations to linear forms implicitly alters their error structure and may also violate the error variance and normality assumptions of standard least squares. From this point of view R^2 can be said to be not too suitable for such important judgement. Also, it has be argued that value of ΔG_{ads} below -20 kJ/mol is an indication of physisorption, and value above that is said to be due to be chemisorption. But cases exist where values of ΔG_{ads} are below -20 kJ/mol, but the adsorption process clearly shows features of chemisorption, and vice versa. In a case of this nature some authors have to tag such adsorption as mixture adsorption (physisorption and chemisorption) (Ating et al., 2010). Even then this assertion does not solve the ambiguity. As put by Popova et al., (2011) the use of ΔG_{ads} criterion only will be difficult to make a clear distinction, especially in the case where charged species are adsorbed due to possibility of coulombic interaction between charged species, which can increase the ΔG_{ads} value, even if no new chemical bonds are formed. It is, therefore, evident that there exist ambiguity in the use R²

and ΔG_{ads} as sole data in the characterisation of adsorption process.

AIM OF THE STUDY

In this paper we have unambiguously shown that the information obtained through the newly proposed twoparameter Adejo-Ekwenchi isotherm (Adejo & Ekwenchi, 2014), in conjunction with that of other isotherms, can be used to eliminate the observed inadequacies in adsorption characterisation process. We consider this submission as part of our contributions towards the emerging trend of resolving of an otherwise ambiguous issue, a very welcome development in the field of science.

RESEARCH METHODS Materials

Leaves of Hyptis suaveolens poit were collected from the bush, identified in the Biological Science Department, Benue State University, Makurdi-Nigeria, then shadedried and pounded into powder. 20 g of the powder was soaked in 200 mL of 98% ethanol (BDH Chemicals Ltd, England) and left standing for two days, with occasional shaking. The extract was filtered using glass wool and ethanol was slowly evaporated using thermostated water bath (Clifton: Nickel-Electro Ltd, England) at 321 K to avoid any possible structural changes that might occur to any of the components (Adejo et al., 2013a). The residue obtained therefrom was preserved in a desiccator. Stock solutions containing 0.1, 0.2, 0.3, 0.4 and 0.5 g/dm³ were prepared in 2 M HCl (M & B).

Coupons (2 cm x 2 cm x 0.13 cm) were prepared as reported in Adejo et al., (2013) from a sample of mild steel of composition (%W): Fe(98.84), Mn(0.56), P(0.04), C(0.27), Si(0.25) and S(0.04).

Methods

Coupons, degreased in acetone (99% BDH Chemicals Ltd, England), were carefully weighed using ae Adam AFP (d ±0.0001g) electronic weighing balance and immersed in 50 mL of the acid solutions, without and with various concentrations of the inhibitor at the temperature range of 301 K to 313 K in the water bath for 8 h. At the retrieval of each coupon, the reaction was terminated in saturated solution of ammonium acetate (97% Labtech Chemicals Limited) as outlined by Orubite-Okorosaye, K. and Oforka, N. C. (2004), and then stored in the desiccator until ambient temperature was attained and reweighed. The %IE and surface coverage (6) were calculated through equations (1) and (2), respectively (Maayta, 2002; Rani & Selvaraj, 2010a; Siaka et al., 2012; Adejo et al., 2013b).

$$1E\% = 1 - \frac{W_{\text{inh}}}{W_{\text{uninh}}} \times 100$$
 (1)
$$\theta = (1 - \frac{W_{\text{inh}}}{W_{\text{uninh}}})$$
 (2)

$$\theta = \left(1 - \frac{W_{\text{inh}}}{W_{\text{uninh}}}\right) \tag{2}$$

where W_{unink} and W_{ink} are weight losses in absence and present of inhibitor, respectively.

RESULTS AND DISCUSSION

Table 1 shows the variation of %IE of the ethanol leaf extract of Hyptis suaveolens poit as eco-friendly corrosion inhibitor of the corrosion of mild steel sample in 2 M HCl with the extract concentration and temperature. The Table clearly shows that the %IE increases with increase in concentration of the extract and decreases with rise in temperature. The decrease in the %IE with rise in temperature is suggestive of physisorption (Rani & Selvaraj, 2010b; Singh *et al.*, 2010).

Table 1. Variation of Inhibition Efficiency (%IE) with Concentration and Temperature of Leaf Extract of Hyptis

suaveoien	poit						
Conc. (g	g/dm3)	%IE					
	301	K 30	05 K 30	9 K 313			
K							
0.1	40.21	38.25	31.21	27.02			
0.2	52.28	50.57	37.27	29.86			
0.3	64.96	54.63	42.39	33.58			
0.4	69.95	58.33	48.41	35.51			
0.5	72.06	63.79	55.91	40.04			

The inhibitive action of organic compounds has been ascribed to the formation of surface layers and films on the metal surface, thereby reducing the accessibility of the corrodant to the metal surface. Adsorption isotherms have been used to characterise this inhibitive action. The most frequently used of such isotherms are the Langmuir, Freundlich, Frumkin, Temkin, Flory-Huggins, Bockris-Swinkels and the El-Awady thermodynamic-kinetic model and the recent proposed Adejo-Ekwenchi isotherm (Khadom et al., 202010; Adejo et al., 2014). The linearised forms of the various equations are given below.

Langmuir
$$\frac{C}{\theta} = \frac{1}{K} + C$$
 (3)

Freundlich $\log \theta = \log K_F + n_F \log C$ (4)

Frumkin $\log \frac{\theta}{(1 \oplus 0)C} = \log K + \frac{2a\theta}{2.500}$ (5)

Temkin $-2\alpha\theta = \ln KC$ (6)

El-Awady $\log \frac{\theta}{1-\theta} = \log K' + y \log [C]$ (7)

Adejo - Ekwenchi $\log \frac{1}{(1-\theta)} = \log K_{AE} + b \log C$ (8)

The fitness adsorption data to an adsorption isotherm has been determined by many authors through the use of the value of regression coefficient, R^2 , from the plot of the surface coverage against the bulk concentration of the adsorbate (extract). Going by this it can be asserted that the data for this extract fitted into the Langmuir, Freundlich, Temkin, El-Awady and Adejo-Ekwenchi isotherms (Table 2).

The equilibrium constant, Kads, values of the isotherms (Table 2) are all positive, implicitly, indicative of favourable adsorption in all the isotherms (Adeio et al., 2012). The S- shaped curves of the plot of %IE against the logarithm of concentration in Figures 1 and 2, at 301 K and 313, respectively, further attest to the formation of adsorbed layer by the extract on the metal surface (Abiola, 2005; Adejo et al., 2012).

Table 2. Various Adsorption Isotherm Parameters for Adsorption of Leaf Extract of *Hyptis suaveolens* poit onto the mild steel surface

Isotherm T	otherm Temp R^2 Slope Intercept K_{ads} - ΔG_{ads}						
	(K)						
(kJ/mol)	` /						
Langmuir							
301	0.9948	1.0796	0.1479	6.7613		14.84	
305	0.9949	1.3351	0.1346	7.4294		15.27	
309	0.9393	1.5791	0.1552	6.4432		15.11	
313	0.9815	2.2140	0.1975	5.0633		14.68	
Freundlich					n		
301	0.9805	0.3814	-0.0097	0.9779	0.3814	10.00	
305	0.9796	0.3039	-0.1035	0.7880	0.3039	9.58	
309	0.9652	0.3495	-0.1708	0.6748	0.3495	9.31	
313	0.9503	0.2339	-0.3462	0.4506	0.2339	8.38	
Temkin					α		
301	0.9853	0.4820	0.8805	1.3406	-0.5550	10.79	
305	0.9867	0.3465	0.7336	2.0408	-1.1363	12.00	
309	0.9250	0.3343	0.6257	2.9437	-0.3851	13.09	
313	0.9222	0.1754	0.3445	1.2782	-0.2020	11.09	
El-Awady					у		
301	0.9322	1.5305	-0.2835	0.6527	1.5305	8.99	
305	0.9347	1.0440	-0.2583	0.5657	1.0440	8.74	
309	0.9969	1.0913	-0.4528	0.3846	1.0913	7.86	
313	0.9904	0.6246	-0.4941	0.1618	0.6246	5.71	
Adejo-							
Ekwenchi					b		
301	0.9758	0.4994	0.7069	5.0921	0.4994	14.13	
305	0.9779	0.3103	0.5173	3.2908	0.3103	13.21	
309	0.9593	0.2107	0.3620	2.3014	0.2107	12.46	
313	0.9524	0.1027	0.2338	1.7132	0.1027	11.85	

Conventionally, the free energy (ΔG_{ads}) for an adsorption process can be obtained through the use of equations (9) and (10), and its value can be used to provide further evidence of suitability of an isotherm.

$$\Delta G_{ads} = -2.303RTlog(55.5K)$$
 (9)

where

$$K - \frac{\theta}{(1-\theta)C}$$
 (10)

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; Adejo *et al.*, 2014). Calculated values of ΔG_{ads} through the equation (9) are presented in Table 3.

The ideal Langmuir isotherm plot should have a slope of unity and intercept of zero (Mejeha *et al.*, 2010; Akalezi *et al.*, 2012). The slopes of the Langmuir plots here are close to unity and intercepts not too far from zero, with good R^2 values, indicative of suitability of the Langmuir isotherm to the adsorption behaviour of this extract. Moreso, the values of ΔG_{ads} obtained through this isotherm are in good agreement with those obtained through the conventional method as show in Table 3. Langmuir isotherm is applicable to both physisorption and chemisorption.

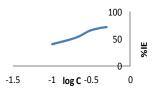


Figure 1. Relationship between Inhibition Efficiency and

logarithm of Concentration at 301K for the Extract of *Hyptis suaveolens* poit

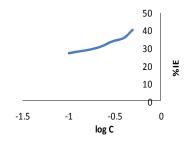


Figure 1. Relationship between Inhibition Efficiency and logarithm of Concentration at 313K for the Extract of *Hyptis suaveolens* poit

. Table 3. Values of ΔG_{ads} obtained through equilibrium constant

•	Jonistant					
	Conc.(g/dm ³)		- ∆G _{ads} (kJ/mol)			
		301 K	305 K	309 K	313 K	
	0.1	14.82	14.81	14.21	13.86	
	0.2	14.31	14.33	13.12	12.42	
	0.3	14.61	13.71	12.63	11.81	
	0.4	14.46	13.36	12.51	11.25	
	0.5	14.16	13.38	12.71	11.21	

The parameter n of the Freundlich isotherm relates to the adsorption intensity, and it varies with the heterogeneity of the material (Chatterjee *et al.*, 2009). It has a typical value is 0.6 (Khadom *et al.*, 2010), of which no value in Table 2 is close and ΔG_{ads} values for this isotherm are also not comparable to those obtained through the conventional method. Therefore, the adsorption process of leaf extract of *Hyptis suaveolens* poit onto the metal sample cannot be modeled through the Freundlich isotherm, in spite of the seemly good values of R^2 .

The Temkin isotherm mostly favours chemisorption and an indication of interaction of uncharged molecules on a heterogeneous surface. The negative values of parameter α show clearly the participation of molecular species in the adsorption. The seemly adherence of this adsorption process to Temkin adsorption isotherm (through R^2 values), is suggestive of participation of molecular species in the adsorption process (Umoren *et al.*, 2009). But, this adsorption has the features of physisorption as %IE was found to decrease with increase in temperature. Moreso, values of ΔG_{ads} obtained in the case of this isotherm are not comparable to those obtained through the conventional method, and so the adsorption process here cannot be reasonably modeled by Temkin isotherm.

The values of parameter, y of the El-Awady kinetic-thermodynamic model are all greater than unity, except at 313 K, it implying formation of multilayers of inhibitor molecules on the metal surface (Al-Lohedan *et al.*, 1996; Saratha & Meenaskshi, 2010; Khadom *et al.*, 2010). But, this is in contrast to Langmuir isotherm which is solely monolayer. In addition, values of ΔG_{ads} through the isotherm are not comparable to those obtained through the

conventional method, and therefore this cannot be a good model for the adsorption process.

The Adejo-Ekwenchi isotherm is centred on the fact that for an adsorption process, the amount of adsorbate uptake from bulk concentration is has inverse relationship with the difference between the total available surface on the adsorbent surface and the fraction that is covered by the adsorbate at a given temperature, prior to the attainment of maximum value of surface coverage (Adejo Ekwenchi, 2014). In fact, this isotherm is termed a fundamental isotherm for adsorption process as it seems to be obeyed virtually by any adsorption process.

The value of parameter b of the Adejo-Ekwenchi isotherm clearly resolves the controversy observed above. Decrease in b value with rise in temperature signifies physisorption, while increase or fairly constant value indicates chemisorption. From Table 2 it is obvious that the adsorption of the plant extract onto the metal surface is physisorption as b decreases with rise temperature. Supportively, the values of ΔG_{ads} obtained through this isotherm are close to those through Langmuir isotherm and also very reasonably compare with those obtained through the conventional method.

CONCLUSION

Going by R^2 and ΔG_{ads} values, the ethanol extract of *Hyptis suaveolens* poit as an inhibitor for corrosion of mild steel 2 M HCl can be said to follow Langmuir, Freundlich, Temkin and El-Awady isotherms. But from the data obtained through Adejo-Ekwenchi isotherm, the adsorption process of this extract has unambiguously be resolved to best fit into Langmuir isotherm and the mechanism is physisorption.

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