ERJ Equatorial Research Journal

Equatorial Journal of Chemical Sciences:
Volume 2; Issue 1: 2018

ADSORPTION KINETICS OF *PICRALIMA NITIDA* SEED EXTRACT AS A GREEN CORROSION INHIBITOR FOR ZINC IN 0.5 M H_2SO_4

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Accepted: 30 – 4 – 2018

Abstract

The potential properties of the ethanol extract of Picralima nitida seed, towards sulphuric acid corrosion of zinc is tested using weight loss methods. It was found that the extract acted as a good corrosion inhibitor for zinc corrosion in 0.5 M H_2SO_4 solution. The protective ability of the extract was discussed in view of Langmuir adsorption isotherm. It was revealed that the adsorption of the extract on zinc surface is governed by spontaneous process. The inhibition efficiency (IE) increases in line with corresponding increase in extract concentration. The temperature effect of the corrosion inhibition on the zinc was studied. Revealation from the studies indicated that the presence of extract increases the activation energy of the corrosion reaction. Furthermore, from the calculated thermodynamic parameters, it was observed that Picralima nitida extract provides a good protection to zinc against pitting corrosion in sulphate ion containing solutions.

Keywords: Zinc, corrosion, inhibition, Picralima nitida seed, inhibitor.

Citation: Ezeugo J.N.O., Onukwuli O.D. and Omotioma M. (2018). Adsorption Kinetics of Picralima Nitida Seed Extract as a Green Corrosion Inhibitor for Zinc in 0.5 M H2SO4. *Equatorial Journal of Chemical Sciences,* 2 (1): 1-13.

Introduction

Corrosion is an electrochemical process that gradually returns metals such as zinc to its natural state in the environment. In other words, corrosion can be said to be destruction of material resulting from exposure and interaction with the environment. It is a major problem that requires immediate confrontation for safety, environment, and economic reasons (Sanjay et al, 2009). Zinc consists of wide variety of alloys used since ancient times. Building industry frequently uses zinc alloys in roofing of house and other construction work because of its ductility and malleability. Therefore, zinc alloys are widely used in the production of many components and die-casting fittings in automobile and manufacturing and the mechanical industry, thanks to its super or super plasticity. Zinc, in spite of the so called super plasticity is not spared by corrosion, especially after prolonged period of exposure in corrosive environment, such as H2SO4. For this reasons a lot of efforts have been made using corrosion preventive practices and the use of green corrosion inhibitors is one of them (Zaabat et al, 2003). The use of green inhibitors for the control of corrosion of zinc (El-Etre et al, 2003; Umoren et al,2012; Nnabuko et al,2015) and alloys which are in contact with aggressive environment is an accepted and growing practice (Ihebrodike et al, 2012). Large numbers of organic compounds are being studied to investigate their corrosion inhibition potential. Revelation of these studies shows that organic compounds are not only expensive, but also toxic to living beings (Al-Otaibi et al, 2014).

Plant extracts and organic species have therefore become important as an environmentally acceptable, readily available, and renewable source for a wide range of inhibitors (Matjaz & Jennifer, 2014). They are the rich sources of ingredients which have very high inhibition efficiency (Alkalezi et al, 2015) and hence termed "Green Inhibitors (Oguzie et al, 2013). Green corrosion inhibitors are biodegradable and do not contain heavy metals or other toxic compounds (Kevitcheu Mbeku et al, 2008). The successful uses of naturally occurring substances to inhibit the corrosion of the metals in acidic and alkaline environment have been reported by some research groups (Chris O.Okunji et al,2005) to mention but a few. Research efforts to find naturally organic substances or biodegradable organic materials to be used as effective corrosion inhibitors of a wide number of metals has been one of the key areas in this research work. The seed of *Picralima nitida* (akuammidine) has chain preventive properties of flavonoids, phenolics, cardiac glycosides, steroids, tannins, saponins, terpenoids which attributed to their abilities to scavenge free radicals induce detoxification, inhibit stress response proteins and interfare with DNA binding activities of some transcription factors. akuammidine has hypotensive, skeletal muscle relaxant and local analgesic activities. Its local analgesic activity is about three times as potent as cocaine. The aim of this study is to investigate the inhibitive properties of *PNS* extract onto zinc in sulphuric acid media. Several studies have already been carried out and have remained focused on the *PNS* extract for their various pharmacological activities.

Firstly, *Picralima nitida* plant is a tree that can reach a height of 35 meters, but is usually less. It is a commonly used herbal remedy in West Africa. All parts of the plant are bitter throughout its distribution areas (Igboasoiyi et al, 2007). The seeds, barks, roots and leaves have a reputation as a febrifuge and remedy for malaria as well as also being extensively used for pain relief and treatment of chest and stomach problems, pneumonia and intestinal worms (Obasi et al,2012). The seed of *Picralima nitida* (akuammidine) has chain preventic properties of flavonoids, phenolics, cardiac glycosides, steroids, tannins, saponins, terpenoids which attributed to their abilities to scavenge free radicals induce detoxification, inhibit stress response, proteins and interfare with DNA binding activities of some transcription factor. Akuammidine has hypotensive, skeletal muscle relaxant and local analgesic activities. Its local analgesic activity is about three times as potent as cocaine (Aguwa et al, 2001). The powdered seed as a result of proven phytocompound are used in the treatment of pneumonia and other chest pains (Iwu et al, 1990). They are also stimulants and tonic made from the seed have intensive bitter flavor. A decoction of the seed is taken as a treatment for measles presently, to the best of our knowledge no reported work in area of environment has been carried out on the corrosion inhibitive properties of PNS extract. Therefore, the aim of this research is to undertake a thorough investigation towards that, in 0.5 M H_2SO_4 using the seed extracts of picralima nitida. The study was done using weight loss method. The effect of temperature and concentration on the rate of corrosion were also studied, and some thermodynamic and kinetic parameters were calculated, too.

EXPERIMENTAL METHODS

Materials

Gravimetric test were performed on 99.988% zn, other components (we %) are: Pb 0.003, Cd 0.003, Fe 0.002, Sn 0.001, Cu 0.00, Al 0.001. The sheet of zinc was cut into coupons (2.6cm x 2.6cm x 0.015cm), cleaned and polished with emery paper to expose shining polished surface. The coupons were degreased with acetone in order to remove any trace of oil and impurities and finally washed with double distilled water, dried in air and then stored in desiccators prior to use. The aggressive solution of 0.5 M H_2SO_4 was made from analytical grade, sulphuric acid and distilled water. *PNS* collected from Uke in Anambra state, Nigeria, was sun-dried for three days. The dried seeds were ground to increase the surface area and stored in a closed container. For every of the extraction process, 30 grams of each of the ground *PNS* were measured and soaked in 100ml of ethanol for 48 hours. At the end of the 48hrs, each plant mixture was filtered. The filtrate is the mixture of the plant extract and the ethanol. The extract of *PNS* obtained in ethanol solvent was concentrated, distilled off the solvent and evaporated to dryness. The plant extract was weighed and stored for the corrosion inhibition study.

Gravimetric Method

The gravimetric method was carried out applying one factor at a time. Considering the said method, the method was carried out at different temperatures and with various concentrations of the *Picralima nitida* extract. Weighed zinc coupons were separately immersed in 250 ml open beakers containing 200ml of 0.5 M H_2SO_4. More so, zinc coupons were separately immersed in 150ml open beakers containing 200ml of 0.5 M H_2SO_4 with various concentrations of the extract.

The variation of weight loss was monitored periodically at various temperatures in the absence and presence of various concentrations of the extracts. At the appropriate time, the coupons were taken out, immersed in acetone, scrubbed with a bristle brush under running water, dried and reweighed. The weight loss was calculated as the difference between the initial weight and the weight after the removal of the corrosion product. The experimental readings were recorded. The weight loss (Δw), corrosion rate (CR) and inhibition efficiency (IE) were determined using the eqns. (3,4 and 5), respectively. The surface coverage was obtained using equation 6 [17,

$$w = W_i - W_f \qquad (3)$$

$$CR = \frac{w_i - w_f}{At} \qquad (4)$$

$$(IE\%) = \frac{w_0 - w_1}{w_0} \times 100 \qquad (5)$$

$$\theta = \frac{w_0 - w_1}{w_0} \qquad (6)$$

Where w_i and w_f are the initial and final weight of zinc samples respectively, W_1 and W_0 are the weight loss values in presence and absence of inhibitor, respectively. A is the total area of the zinc sample and t is the immersion time.

Effect of temperature on the corrosion rate

Effect of temperature on the corrosion rate was described using Arrhenius equation

$$CR = A\ e^{-Ea/RT} \qquad (7)$$

Where CR is the corrosion rate of the zinc, A is the pre-exponential factor, Ea is the activation energy, R is the universal gas constant. eq. (7) can be linearized to form eq. (8).

$$In\ (CR) = In\ A - \left({}^{Ea}/{}_{R}\right)\left(\frac{1}{T}\right) \qquad (8)$$

Considering the corrosion rate of the zinc at T_1 and T_2 as Cr_1 and CR_2, then eq. (8) can be expressed by eq. (9) [18, 20].

$$In\ \left(\frac{CR_2}{CR_1}\right) = \left(\frac{Ea}{2.303R}\right)\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \qquad (9)$$

Thermodynamic parameter for the adsorption process

The heat of adsorption Q_{ads} ($kjmol^{-1}$) was calculated using eq. (10) [21]

$$Qads = 2.303R\ \left[\log\left(\frac{\theta_2}{1-\ \theta_2}\right) - log\left(\frac{\theta_1}{1-\ \theta_1}\right) \times \frac{T_2\, T_1}{T_2 -\ T_1}\right] \qquad (10)$$

Where R is the gas constant, θ_1 and θ_2 are the degree of surface coverage at temperature T_1 and T_2 respectively.

Consideration of the Adsorption isotherm

The data obtained for the degree of surface coverage were used to test for the applicability of different adsorption isotherms (Langmuir, Frumkin, Temkin and Flory-Huggins isotherms).

1. Langmuir Isotherm

Langmuir isotherm can be expressed by eqn. (11, 12)

$$\frac{C}{\theta} = \frac{1}{K} + C \qquad (11)$$

Where C is the concentration of the inhibitor, K is the adsorption equilibrium constant and θ is the degree of surface coverage. In logarithmic form, eqn. (11) can be expressed in eqn. (12)

$$\log\frac{C}{\theta} = \log C - \log K \qquad (12)$$

2. Frumkin isotherm

Frumkin adsorption isotherm can be expressed according to eq. (13)

$$\log\left(Cc) * \left(\frac{\theta}{1-\ \theta}\right)\right) = 2.303logK + 2\ \alpha\theta \qquad (13)$$

Where K is the adsorption –desorption constant and α is the lateral interaction term describing the interaction in adsorbed layer.

3. Temkin isotherm

Temkin isotherm can be expressed by eq. (14) [19]

$$\theta = \frac{2.303\log K}{2a} - \frac{2.303\log C}{2a} \qquad (14)$$

Where k is the adsorption equilibrium constant, a is the attractive parameter, θ is the degree of surface coverage, C is the concentration of the inhibitor

4. Florry-Huggins Isotherm

The Flory-Huggins isotherm can be expressed by eqn. (15).

$$\log\left(\frac{\theta}{C}\right) = \log k + x\log(1-\ \theta) \qquad (16)$$

Where x is the size parameter and is a measure of the number of adsorbed water molecules. The free energy of adsorption (ΔG_{ads}) was calculated according to eqn. (16).

$$\Delta G_{ads} = -2.303RT \log (55.5K) \quad (17)$$

Where R is the gas constant. T is the temperature, K values obtain from the isotherms (Langmuir, Frumkin, Temkin and Flory-Huggins isotherm) were used to obtain the values of ΔGads according to eq. (17).

RESULTS AND DISCUSSION

Adsorption kinetic studies and corrosion inhibition mechanism.

The values of IEs and θs of different *PNS* extract concentrations are given in Table 1. The tabulated data revealed that, the *PNS* extract acted as a good corrosion inhibitor for the acid corrosion of zinc. The corrosion inhibition increases with increasing extract concentration. The results of the ethanol extract of PNS as revealed by Ezeugo (2017), revealed that the extract contains toluene, formular C_7H_8, molecular weight 92, cyclohexane having formula C_8H_{16}, molecular weight 112, hexane, 1,3-cyclopenta deine, molecular weight 156. It also contains at least ten non-volatile acids including eicosane and citric acids. The adsorption of the compounds on the surface of the electrode created some barriers for mass and charge transfers (Oguzie et al,2008).

The outcome of this situation leads to the protection of the zinc surface from the attack of the aggressive anions of the acid. The extent of protection increases with increasing of the surface fraction occupied by the adsorbed molecules. As the extract concentration is increased, the number of the adsorbed molecules on the surface increases. Table 2 represents also the values of adsorption isotherm parameters. From the table, a parameter (θ), which was estimated from the IE values, could be used to represent the fraction of the surface occupied by the adsorbed molecules. In-depth examination of Table 1 revealed that the values of θ increases with increasing inhibitor concentration. The dependence of the fraction of the surface occupied by the adsorbed molecules on the inhibitor Concentration (C) is shown in Fig. 1 a plot of C/θ versus C gives a straight line with unit slope. The results indicated that the adsorption of inhibitor molecules on the zinc surface followed Langmuir isotherm. In order words, the result suggested that there are no interactions or repulsion forces between the adsorbed molecules. It is of interest to note here that, the θ values obtained from the other used techniques also obey the Langmuir adsorption isotherm.

. **Table 1: Corrosion inhibition of zinc in 0.5 M H_2SO_4 with *picralima nitida* seed extracts.**

Time (hr)	Temperature (K)	Inhibitor conc. (gL^{-1})	Weight loss (g)	Corrosion rate (Mg/cm^2 hr)	Inhibition efficiency (%)	Degree of surf.cov.
12	303	0	0.671	6.213	-	-
		0.2	0.327	30.28	51.27	0.5127
		0.45	0.302	2.796	54.99	0.5499
		0.7	0.223	2.065	66.77	0.6677
		0.95	0.163	1.509	75.71	0.7571
		1.2	0.101	0.935	84.95	0.8495
12	313	0	0.68	6.296	-	-
		0.2	0.34	3.148	49.7	0.497
		0.45	0.277	2.565	59.02	0.5902
		0.7	0.199	1.843	70.56	0.7056
		0.95	0.142	1.615	78.99	0.7899
		1.2	0.141	1.306	79.14	0.7914
12	323	0	0.645	5.972	-	-
		0.2	0.411	3.806	39.56	0.3956
		0.45	0.319	2.954	53.09	0.5309
		0.7	0.233	2.157	65.74	0.6574
		0.95	0.183	1.694	73.09	0.7309
		1.2	0.175	1.62	74.26	0.7426

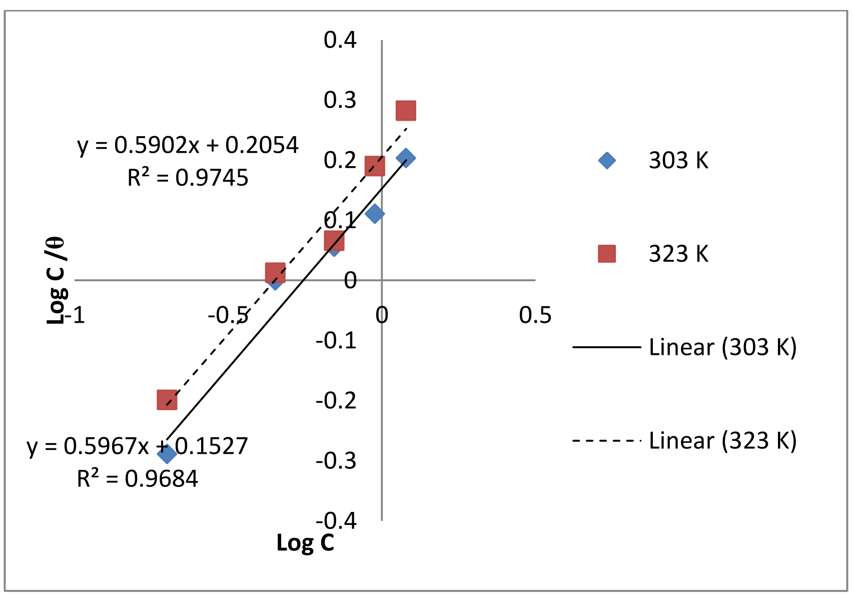

Figure 1 Plot of Langmuir isotherm for Zinc in H_2SO_4 with PNS extract at 303k and 323k

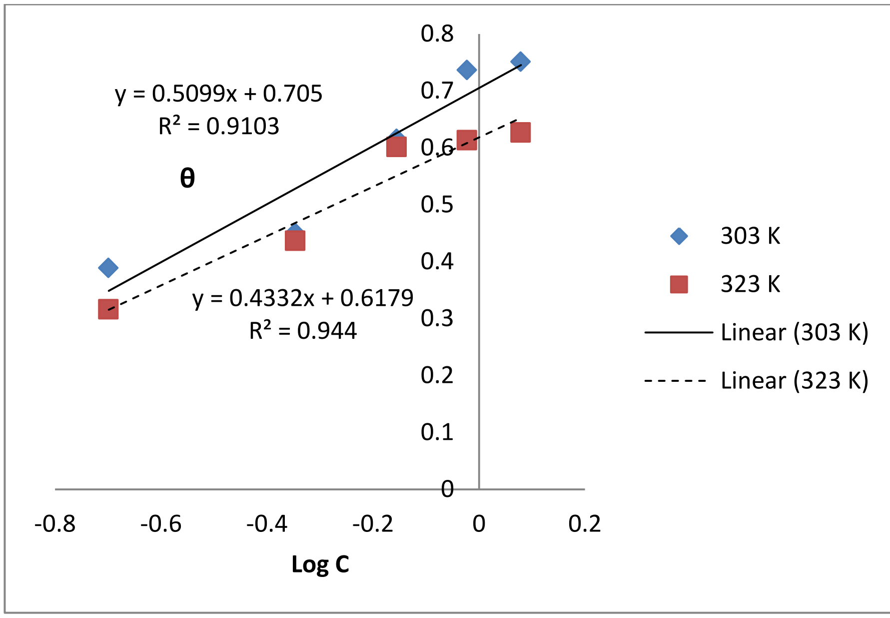

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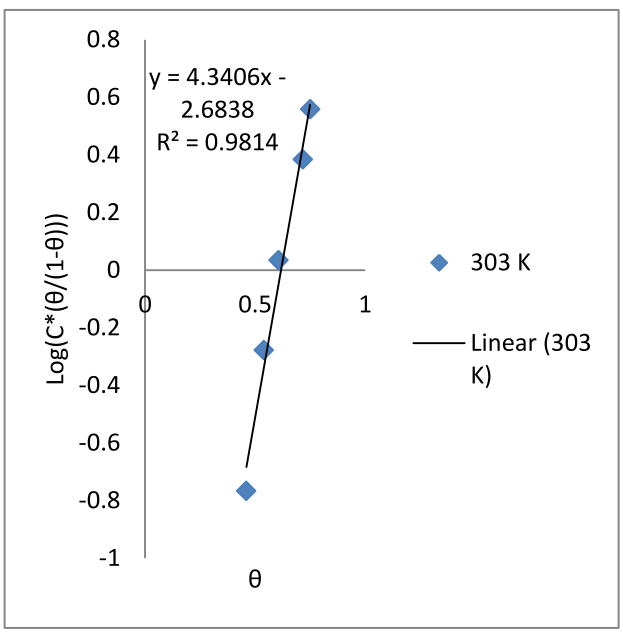
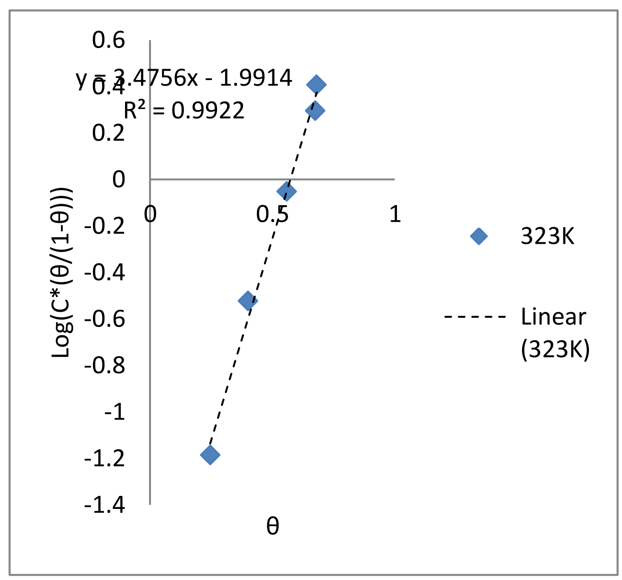
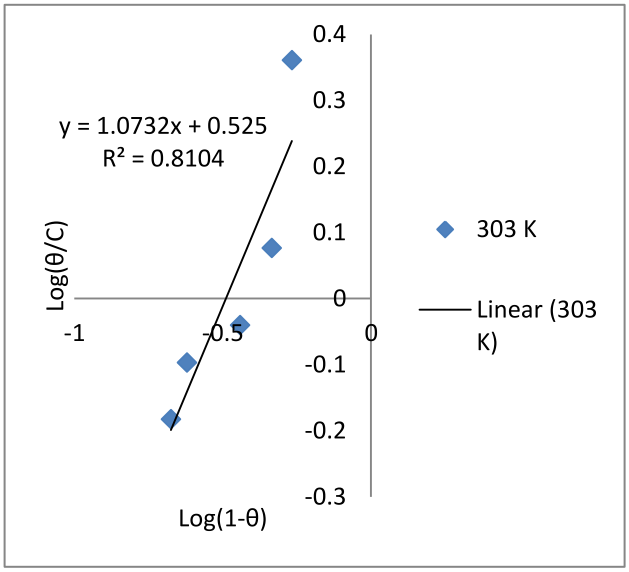
Figure 2 Plot of Temkin isotherms for Zinc in H_2SO_4 with PNS extract at 303k and 323k



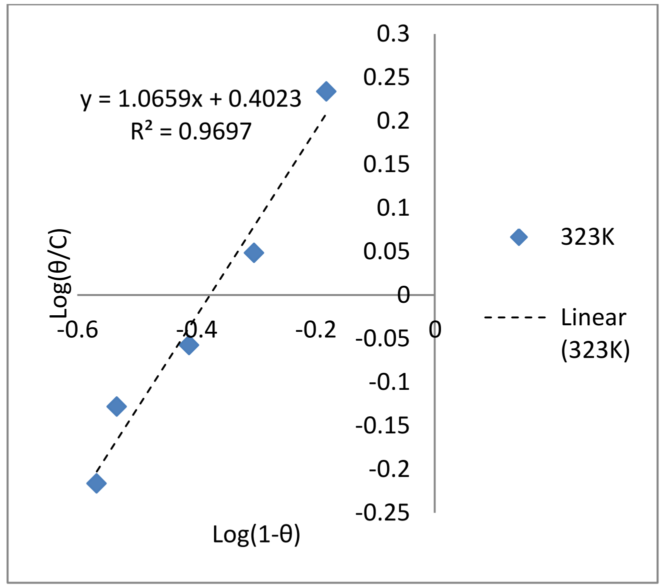


(a) (b)

Figure 3 (a,b) Frumkin isotherm for zinc in H_2SO_4 with PNS extract at 303k and 323k



(a)



(b)

Figure 4 (a,b) flory-huggins isotherm for Zinc in H_2SO_4 with PNS extract at 303k and 323k.

The standard adsorption free energy (ΔG_{ads}) was calculated using the following equation : [17].

$$K = \frac{1}{999}\exp\left(-\frac{\Delta G_{ads}}{RT}\right) \quad (17)$$

Where 999 is the concentration of water in solution expressed in gl^{-1}. R is gas constant, and T absolute temperature. The mean value of standard adsorption free energy (ΔG_{ads}) was $-9.39 kjmol^{-1}$. The negative value of ΔG_{ads} guarantees the spontaneity of the adsorption process and stability of the adsorbed layer on the metal surface. It is generally known that, values of ΔG_{ads} up to $-20\ KJmol^{-1}$ is consistent with electrostatic interaction between the charged molecules and the charged metal (physisorption). While those within the threshold of – 40 $kjmol^{-1}$ or higher are associated with chemisorptions as a result of sharing or transfer of electrons from the molecules to the zinc surface to form a coordinate type of bond (Alkalezi et al, 2015; Oguziel et al,2013; Ating et al,2010). Other researchers (Fouda et al,2015;Khaled et al,2008;Megahed et al,2010 &Ezeugo et al,2017) however suggested that the range of ΔG_{ads} of chemical adsorption processes for inhibitor in aqueous media lies between -21 and -42 $KJmol^{-1}$. From table 3 the values of ΔG_{ads} as recorded in the present work has been considered within the range of physical adsorption. Limited increase in the absolute value of ΔG_{ads} at 303 K temperature, then, heat of adsorption decreases again at 313 k initiating that the adsorption was somewhat favorable at the experimental temperature and *PNS* extract adsorbed according to physical mechanisms, i.e. desorption of inhibitor molecules when temperature increased. Moreover, the major characteristic of Langmuir isotherm can be expressed in terms of linear regression coefficient. The value of the linear regression coefficient is close to unity, hence adsorption of the *PNS* extract follows Langmuir isotherm and R^2 value is $0.974 \geq 0.968$. It is very important to note that the smaller values of R^2 indicated a highly favourable adsorption. $R^2 > 1$ unfavorable, $R^2 = 1$ linear, $0 < R^2 < 1$ favourable and if $R^2 = 0$ irreversible. The table 3 shows that various values of R^2 for the entire tested isotherms model. The values of k_{ads} are relatively small indicating that the interaction between the adsorbed extract molecules and metal surface is physically adsorbed.

A close look at Table 3 shows various inhibition concentrations (gL^{-1}) and their respective activation energy (kj mol^{-1}). From the table, calculated Ea value for the inhibited solution witT PNS extract is 37.09 and 39.31 $kjmol^{-1}$ in the presence of the inhibitor of 0.95 and 1.2 gL^{-1} extract concentrations, while with 0.45 and 0.70 gL^{-1} concentrations, activation energies are 3.47and 4.59$kjmol^{-1}$. The higher values of Ea suggest that dissolution of zinc in the presence of inhibitors is slow, indicating a strong inhibitive action of phytocompounds of alkaloids, flavonoids and tannins presence in *PNS* extracts, which leads to increasing the energy barrier for the corrosion process (Omotioma & Onukwuli, 2017). Actually, 2-heptanone and 2tridecene molecules (the main compounds of *PNS* extracts are easily protonated and existed in 0.5 M H_2SO_4 medium in cationic form. Indeed, it is logical to assume that in this study, the electrostatic cat ion adsorption is responsible for the good protective properties of these compounds.

Table.2 Adsorption parameters for the corrosion inhibition of Zinc in H_2SO_4 by PNS extract

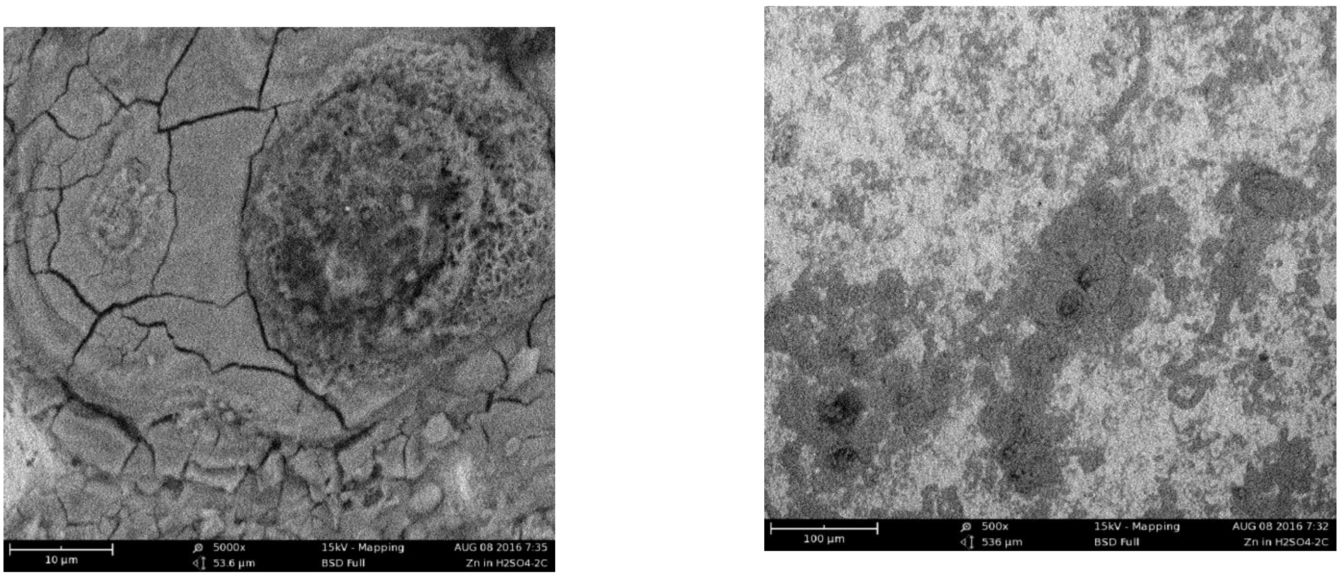
Adsorption isotherm	Temperature (k)	R^2	Log K	K	ΔGads (KJ/mol)	Isotherm property	
Langmuir isotherm	303 323	0.974 0.968	-0.205 -0.152	0.6237 0.7047	-8.930 -9.847		
Frumkin isotherm	303 323	0.977 0.981	-0.9566 -0.9805	0.1105 0.1046	-4.569 -4.723	α	1.822 1.987
Temkin isotherm	303 323	0.910 0.944	-3.1898 -1-4249	0.006 0.0376	8.572 -1.975	a	-2.262 -2.659
Flory-Huggins isotherm	303 323	0.745 0.848	0.358 0.407	2.2803 2.5527	-12.196 -13.304	x	0.903 1.444

Table 3. Activation Energy and Heat of Adsorption for the Corrosion Inhibition of Zinc in 0.5M H_2SO_4 at various Inhibitor Concentrations

Inhibitor concentration (gL^{-1})		Ea (kj mol^{-1})		$-\Delta G_{ads}$ (kj mol^{-1})
0.2		11.81		13.01
0.45		3.47		2.19
0.70		4.59		2.39
0.95		37.09		23.01
1.20		39.31		23.90

Morphological examinations of zinc surface immersed in 0.5 M H2SO4 and in the presence of plant extract containing $1.2gL^{-1}$

Fig.5 (a) Shows the SEM image of zinc coupon immersed in a solution of 0.5 M H_2SO_4 for 12h. The SEM image revealed that the surface was badly corroded as a result of aggressive attack by the sulphuric acid. Fig.(b) shows SEM image of the zinc specimens immersed for the same period of time interval in 0.5M H_2SO_4 solution containing 1.2 gL^{-1} of PNS extract. These images show that the adsorbed inhibitor film present on the zinc surface mitigated the dissolution of the base metal with high degree of efficiency. It is also observed that there is significant morphological variation between the protective barrier formed on the zinc surface at $1.2gL^{-1}$ of the extract and the one immersed in the solution free inhibitor.. This observation is in clear agreement with the findings of (Ezeugo et al, 2018)



(a) (b)

Figure 5: (a) The SEM image of zinc coupon immersed in a solution of 0.5 M H_2SO_4 for 12h in absence of PNS extract. **(b)** SEM image of the zinc specimens immersed for the same period of time interval in 0.5M H_2SO_4 solution containing 1.2 gL^{-1} of PNS extract.

Conclusion

i. The *Picralima nitida* seed extract acts as a good inhibitor for corrosion of zinc in 0.5 M H_2SO_4 solution. The IE increases with increasing extract concentration.

ii. The inhibitory action was carried out through adsorption of the extract compounds on zinc surface. The adsorption process is spontaneous, stable and obeys Langmuir adsorption isotherm.

iii. The adsorption process is physical as various studies techniques points towards physisorption. More so, the increase in temperature decreases the IE of the extract.

iv. The presence of the extract increases the activation energy of the corrosion reaction.

v. The *Picralima nitida* seed extract provide strong protection against corrosion of zinc in presence of chloride ions. The extent of protection increases with increasing extract concentration.

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