Original Research Article

Inhibitive Properties of *Acanthospermum Hispidum Dc* Leaves Extracts on Corrosion of Mild Steel in 1M HCl solution

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ABSTRACT

This study was conducted to investigate the suitability of *Acanthospermum hispidum Dc* leaves extracts for corrosion inhibitionof mild steel immersed in 1M HCl solution using gravimetric and potentiodynamic polarization methods. The study was conducted at room and elevated temperatures of 313, 323, 333 and 343K. The test period was varied from 24 to 168hrs at 24hrs interval, at room temperature and 3 hours at each elevated temperature. The experiments were performed with inhibitor concentrations of 0.1, 0.2, 0.3, 0.4 and 0.5 g/L. The results showed that the corrosion rates increased with time of immersion in the absence of inhibitor but decreased with inhibitor concentration at room and elevated temperature, while inhibition efficiency increased with increase in inhibitor concentration at room temperature but decreased with temperature rise. .Inhibition efficiency of 80 % for leaves extracts was achieved at 0.5 g/L at room temperature but decreased to 66.67 % at 0.5 g/L and temperature of 343K implying desorption of inhibitor at higher temperature. Potentiodynamic polarization method was used to see the influence of the extracts on the anodic dissolution of mild steel. The results showed that the extracts acted as mixed type inhibitor by moderating both the anodic and cathodic reactions and remarkably lowered corrosion rates from 10.889 to 0.365 mm/yr. The absorption of the extracts on the metal surface obeyed Langmuir and Temkin isotherms. The thermodynamic parameters evaluated gave negative values of ΔG°_{ads} in the range of -21.35 to – 22.82 kJ/mol. which is less than 40 kJ/mol indicating physical absorption of the extracts on the metal surface. The increasing values of E_a(kJ/mol) from 24.21 to 47.88 kJ/mol also confirms that absorption is by physisorption mechanism.

Keywords: Acanthospermum hispidum DC, adsorption mechanism, Gravimetric, inhibition, inhibitor concentration Mild steel corrosion, potentiodynamic polarization.

1. INTRODUCTION

Owing to relatively cheap prices and good characteristics,mild steel is one material that has found wide application in the construction industry. It is however, prone to corrosion when exposed to different corrosive environments. Environmental attack on metallic structures affect their physical and mechanical properties leading to failure in service, and consequently 3-4 % of GDP for developed countries like the U.S.A and Britain is lost to corrosion and its control annually in terms of production down time, man-hour losses and safety issues [1].

Efforts have been made to find ways of reducing its effects on mild steel. Several methods such as protective coatings, proper material selection, alloying, proper design, cathodic protection, use of inhibitors, etc. have been used in corrosion control and prevention, depending on such considerations as nature of the environment, efficiency required, cost and safety of the personnel involved [2]. Inorganic inhibitors such as sodium silicates, sodium phosphates, molybdates, chromates and tungstates have been effectively used as corrosion inhibitors. However, these compounds have been found to contain toxic substances that are hazardous to man and they pollute the environment after usage [3].

In view of the strict environmental regulations concerning the use of chemical substances which are capable of controlling corrosion but in turn release toxic chemicals in the environment, efforts are now geared towards the production of eco-friendly inhibitors. Hence plants have been observed to contain naturally occurring compounds with complex molecular structures and various physical, chemical and biological properties that could serve as replacement for the inorganic compounds. Inhibitors from plant origin are less toxic, biodegradable, available and cheap compared to the inorganic inhibitors [4].

These plant materials contain phytochemicals that have been found to contain bioactive substances such as alkaloids, Flavonoids, tannins, steroids, glycosides, etc. which have been proven to contain corrosion inhibitive properties for metals in various corrosive media [5, 6]. Organic compounds from plant extracts could block anodic or cathodic sites or both, thereby slowing down the dissolution of the metal or hydrogen evolution. The efficiency of these inhibitors is based on the fact that they contain heteroatoms from oxygen, nitrogen and sulphur with polar functional groups, non-bonding andπ – electrons that act as adsorption centres on the metal surface [7]. The use of corrosion inhibitors has gained attention in the past three decades due to ease of application, cheapness and availability. A good number of organic compounds have been studied and used for mild steel corrosion inhibition to improve its performance in corrosive environments [8]. Plant materials are not only environmentally friendly but also readily available and renewable sources with a wide range of chemicals in them. Due to eco-friendliness, biodegradability, low cost and easy availability, extracts of some common plants have been studied as inhibitors for metals under different environments [6].

Umoren *et al.* [9] studied leaves and stem extracts of *Sida acuta*, Kumar *et al.* [10] investigated the inhibitive action of *oxystelma Esculentum* stem extract, Uwah *et al.* [6], studied inhibitive action of *costus afer* stem extracts, Bamaiyi and Peni [11] investigated the effects of locus beans (*Pakiabiglobosa*) and banana sap extracts, Oparaku *et al.* [12] studied Alchornea *cordifolia* leaf extract, Oloche *et al.* [13] evaluated the inhibitive ability of kolanut leaves extract, and Ekeke *et al.* [14] studied inhibitive properties of *Magnifera indica* seed extract. All these studies showed that plants extracts performed well as corrosion inhibitors in various media. Similar investigations were carried out by Okewale and Adesina [3], Olasehinde *et al.* [8], and Agboola *et al.* [15], and they confirmed different plant extracts as good inhibitors for mild steel corrosion in acidic solution.

Acanthospermum hispidum DC is a common weed that grows freely in cultivated farmlands in Nigeria. Preliminary studies showed that this plant contains bioactive compounds that have potentials for corrosion inhibition [5]. Hence, this paper looks at the possibility of using *Acanthospermum hispidum* DC leaves extracts as corrosion inhibitors for mild steel in 1M HCl solution.

2. MATERIAL AND METHODS

Pls kindly see highlighted wordsfor necessary action

The mild steel sheet of composition (wt %): C (0.04), Si (0.004), Mn (0.225), P (0.004), Cu (0.002), Ti (0.007) and the rest Fe was mechanically cut into coupons of 40 × 30 × 0.5 mm. A small hole of 3 mm was drilled at the end of each coupon for its suspension in HCl solution with the help of flexible copper wire. The coupons were degreased, polished using different grades of silicon carbide impregnated with the emery paper of 120, 400, 800 and 1000 grits; cleaned with acetone, dried and stored in a desiccator.

Fig. 1. *Acanthosperum hisipidum DC* Leaves (AHL) and Stem with Flowers

The leaves and stem materials (Figure 1) were collected from *Antsah* village, North Bank, Makurdi, Nigeria; washed under running tap water and then separated into leaves and stem parts. The separated materials were then dried under shade for four weeks to a constant weight and pounded in a mortar to reduce it to smaller sizes which were later blended to powder form and stored for subsequent use.

The extracts were prepared from dried powdered leaves materials at the biological laboratory Joseph Sarwuan Tarka University Makurdi (JOSTUM) using cold extraction process as stated by Umoren *et al*.[9]. Fifty grams (50 g) of the prepared material was weighed using a digital weighing scale and soaked in 500 ml of absolute ethanol for 48 hours with occasional agitation. The solution was double filtered with cheese cloth and the filtrate collected in a conical flask. The extract was dried using water bath at 45 °C,~~and~~ collected in a sample bottle and stored in a refrigerator at 4 °C for subsequent use.

The acid solution was prepared by diluting 83.61 ml of concentrated hydrochloric acid of 37 % purity in distilled water to make 1M HCl solution. To achieve this, 83.61 ml of HClwas diluted with 900 ml of distilled water and made up to 1000 ml of HCl solution. From this volume of 1000 ml, 200 ml was measured and used as the corrosive media in six 250 ml beakers. Inhibitor concentrations of 0.1, 0.2, 0.3, 0.4 and 0.5 g/L were then prepared. Each extract weighing 0.5 g~~0.5 g of each extract~~ was added to 200 ml of HCl solution to give the stock solution with concentration of 2.5 g/L.

The different working concentrations of 0.1 to 0.5~~(0.1 to 0.5)~~ grams per liter were obtained using the dilution principle, equation(1)

$$C_1V_1 = C_2V_2 \tag{1}$$

where $C_1$is the initial concentration of the extract, $V_1 = \frac{C_2V_2}{C_1}$ is the initial volume needed to obtain desired concentration, $C_2$is the final concentration (working concentration) and V_2 is the final volume (working volume).

Weight loss measurements were obtained according to ASTM G31-72 standard procedure. The polished coupons were then weighed using a digital weighing balance. These weighed coupons were then fully immersed in six beakers of 250 cm^3 containing 0.1 to 0.5 g/L extract concentration in 200 cm^3 HCL solution. The sixth beaker contained only the acid solution without inhibitor in it. Two coupons were immersed in each beaker containing the media. Weight loss measurements were carried out after every 24 hours of immersion progressively for 168 hours at room temperature. The coupons were removed from the solution after 24 hours, washed with distilled water, cleaned with acetone, dried in the sun and reweighed and average weight loss recorded. This procedure was repeated to test the coupons at elevated temperatures of 313, 323, 333 and 343 K but with replacement of coupons and exposure time of 3 hours in a thermostatic water bath capable of temperature regulation.

The difference in weight of the coupons before and after exposure in acidic solution was recorded as weight loss (W) of the metal as presented in equation 2 [16].

$$W = W_o - W_i \quad (2)$$

where W = weight loss in grams (g), W_o = weight before immersion (g) and W_i = weight after immersion (g).
The corrosion rate was determined using equation 3.

$$C.R. = \frac{87.6W}{DAT} \quad (mm/yr) \quad (3)$$

where W = weight loss in (g), D = density of the metal, (g/cm^3), A = total surface area of the coupon (cm^2),
87.6 is the unit conversion constant, and T = time of exposure (hours).

The inhibition efficiency of the samples was determined using equation 4.

$$I.E. = \frac{CR_{0-CR_1}}{CR_0} \times 100\,\% \quad (4)$$

where *I.E.* = inhibition efficiency (%), CR_o and CR_1 are corrosion rates in the absence and presence of inhibitor respectively.The surface coverage was determined usingequation 5.

$$\theta = \frac{CR_{0-CR_1}}{CR_0} \quad (5)$$

where θ is the degree of surface coverage, CR_o and CR_1 are corrosion rates in the absence and presence of inhibitor respectively.

The influence of temperature on the corrosion behavior of mild steel in 1M HCl in the absence and presence of the various inhibitor extracts was also investigated by weight loss method at 313, 323, 333 and 343K. Twelve (12) pre-cleaned mild steel coupons were weighed separately on analytical digital weighing balance and each suspended with a string and two immersed in each beaker. 0.1 – 0.5 g/L, extract concentrations were added to five beakers containing 200 ml of the acid solution, while the sixth beaker contained 1M HCl solution without inhibitor as a control. The six beakers with and without inhibitor solutions containing the coupons were kept at the desired temperature in a thermostatic water bath for a period of three (3) hours. At the end of this time, the specimens were carefully removed, washed in doubly distilled water, dried and reweighed. The weight loss was recorded for the purpose of calculating the corrosion rate due to effect of elevated temperature using equation 3.

Potentiodynamic polarization investigations were conducted in the absence and presence of the plants extracts to evaluate its effect on the corrosion potential ($E_{corr.}$), corrosion current density ($I_{corr.}$) and the anodic (β_a) and cathodic (β_c) Tafel constants. The electrochemical experiments were performed using a computer controlled potentiostat instrument, VERSASTAT 4. The set up consisted of a three – electrode corrosion cell made up of mild steel coupons as the working electrode (WE), with surface area of 1cm^2, saturated silver/silver chloride as reference electrode, and platinum rod as counter electrode. The test electrolyte was 1M HCl solution in the presence and absence of AHL and AHS extracts in varying concentrations of 0.1, 0.2, 0.3, 0.4 and 0.5 g/L and the blank.

All electrochemical experiments were conducted at room temperature (30 ± 2°C) using 100 ml of the inhibitor solution in a stationary condition. The working electrode was immersed in a test solution for 30 minutes until a stable open-circuit potential (OCP) was attained for corrosion to reach a steady/equilibrium state.Polarization measurements were carried out at a scan rate of 1.0 mV/s at a potential initiated at -250 mV to +250 mV. The test sample and electrolyte were replaced after each experiment. The linear portion of the Tafel plot of the anodic and cathodic curves were extrapolated to obtain the corrosion current densities ($I_{corr.}$) and corrosion potential (E_{corr}), anodic Tafel slope (β_a) and cathodic Tafel slope (β_c) were determined from the experimental curve.
The inhibition efficiency was calculated using the values of current densities in the absence and presence of inhibitor using equation 6.

$$IE_{Icorr} = \frac{I^{o}{}_{corr} - I_{corr}}{I^{o}{}_{corr}} \, x\, 100 \qquad (\%) \qquad (6)$$

where $I^{o}{}_{corr}$ and I_{corr} are the current density values in the absence and presence of inhibitor respectively.

3. RESULTS AND DISCUSSION

Based on weight loss measurements, the corrosion rates, surface coverage and inhibition efficiency for the various extract concentrations of AHL were calculated and presented in Figures 2 to 5. This can be clearly seen in the plot of corrosion rate against inhibitor concentration which shows a decline in corrosion rate with increase in extract concentration as presented in Figures 4 and 5. The same observation was made at elevated temperature as the corrosion rate increased with increase in temperature but decreased with increase in inhibitor concentration in Figures 6 to 7. The reduction in corrosion rate in the presence of inhibitor could be attributed to phytochemical constituents of AHL and AHS which contain sulphur, nitrogen and oxygen atoms that act as centers of adsorption on the metal surface.

There was also increase in inhibition efficiency with increased concentration (Figures 8 and 9), but decreased with rise in temperature. This implies that more surface coverage is achieved with higher concentration creating a barrier film on the metal surface which blocks the corrosion sites, thereby reducing corrosion rate. However, with rise in temperature some of the extracts could be dissolved, hence reduction in inhibition efficiency. This trend is indicative of physical adsorption mechanism of phyto-constituents on the metal surface and similar to the findings of Nwosu *et al.* [16], Ebenso *et al.* [17], Adejo *et al.* [18] and Iroha and Chidiebere [19].

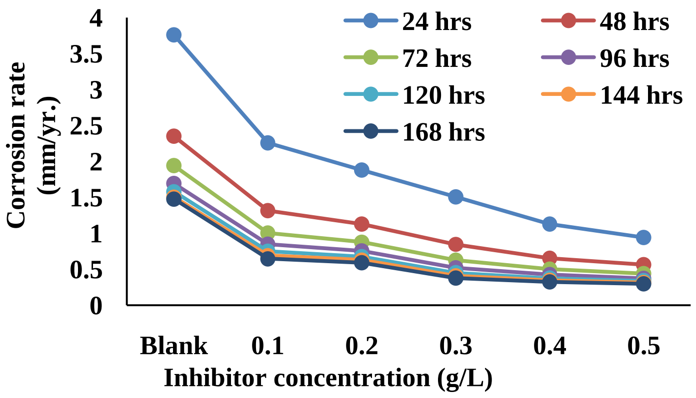

Fig. 2. Corrosion Rate against AHL Inhibitor Concentration in 1 M HCl Solution

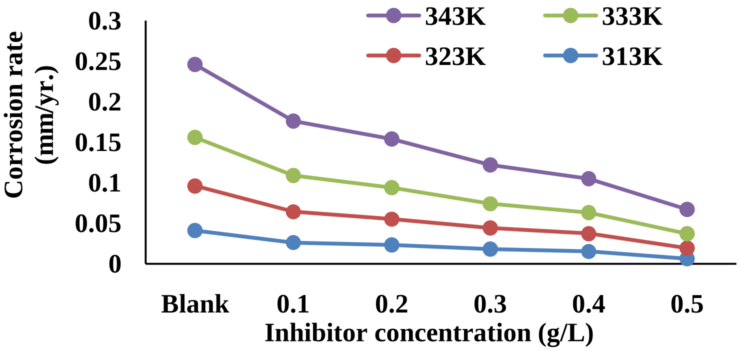

Fig. 3. Variation of corrosion rate with AHL concentration at elevated temperatures

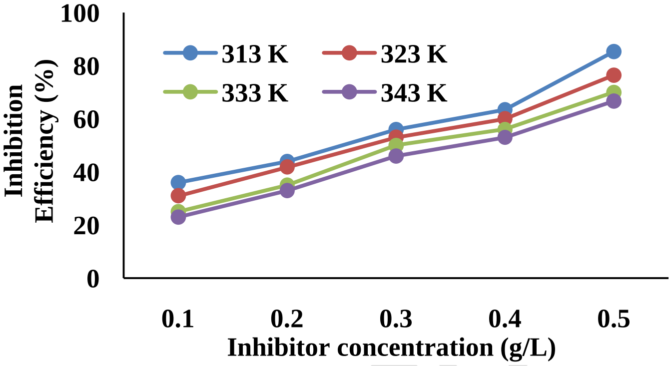

Fig. 4. Variation of inhibition efficiency at various AHL inhibitor concentration at various temperatures

The variation of inhibition efficiency with temperature at various concentrations as presented in Figure 4 indicate that inhibitor efficiency decreases with increase in temperature but increases with increase in concentration of the extract. This could be attributed to the fact that at higher temperatures there may be desorption of inhibitor phytochemical constituents from the metal surface thereby leading to increased corrosion rate and consequently lower inhibition efficiency [20, 21].

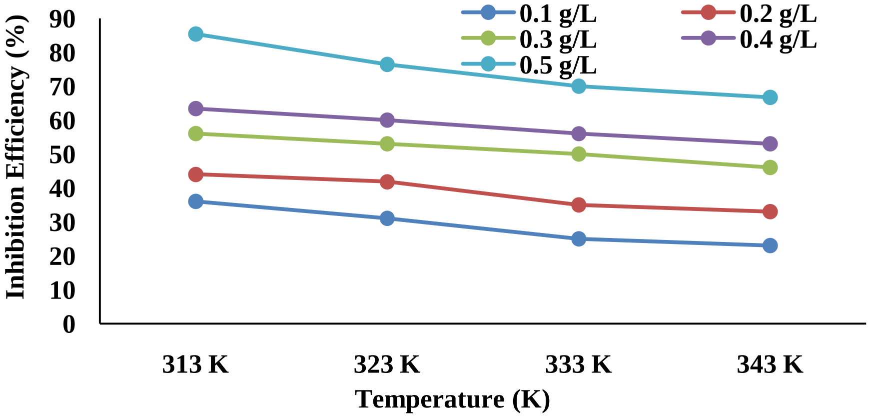

Fig. 5. Variation of inhibition efficiency of AHL inhibitor with temperature at various concentrations

The results of potentiodynamic polarization shows the anodic (β_a) and cathodic (β_c) Tafel constants presented in Figure 6 as Tafel plots. It can be observed from the polarization plots that in the presence of the inhibitors, there is a decrease in anodic and cathodic current densities and the corrosion potential (E_{corr}) for both extracts concentrations compared to the uninhibited. This implies that the extracts have inhibitory effects on the mild steel corrosion [22]. The higher values of β_c~~(β_c)~~compared to those of β_a~~(β_a)~~ shows that the cathodic reaction was the predominant factor and that inhibitors acted as mixed type but with more cathodic influence [19].

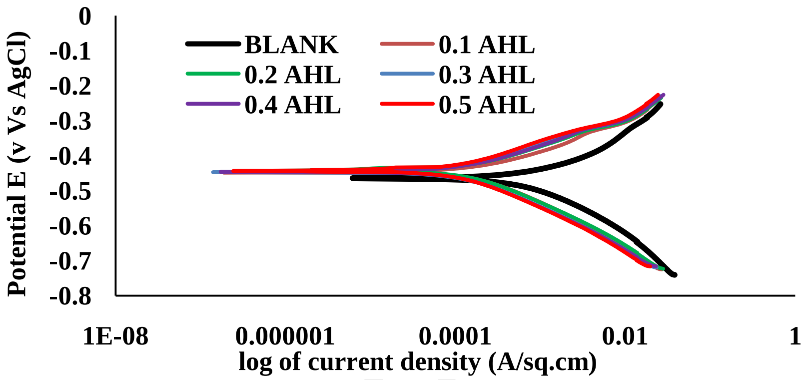

Fig. 6. Potentio-dynamic Polarization Curves for Mild Steel Corrosion using various Concentrations of AHL Extract

The adsorption consideration gives us the idea of how the inhibitor molecules can be adsorbed on the metal surface through their polar functions. Adsorption isotherm values are used to explain the mechanism of corrosion inhibition of organo-electrochemical reactions. In this study, Langmuir and Temkin isotherms were investigated as presented in Figures 7 and 8 .Langmuir isotherm is given by the equation 8.

$$\frac{C}{\theta} = \frac{1}{Kads} + C \qquad (7)$$

where C = inhibitor concentration, θ =degree of surface coverage, and K_{ads} is the adsorptive equilibrium constant.

The plots of C/θ $against\ C$ are given in Figures 6 for AHL. Linear plots were obtained with good correlation coefficient of (R^2) close to unity which suggests that the adsorption of this inhibitor obeys Langmuir isotherm model. The adsorptive equilibrium constant K_{ads} decreases with increase in temperature of the environment (Table 1) which indicates that the interactions between the adsorbed molecules and the metal surface becomes weaker at higher temperatures and could lead to desorption.

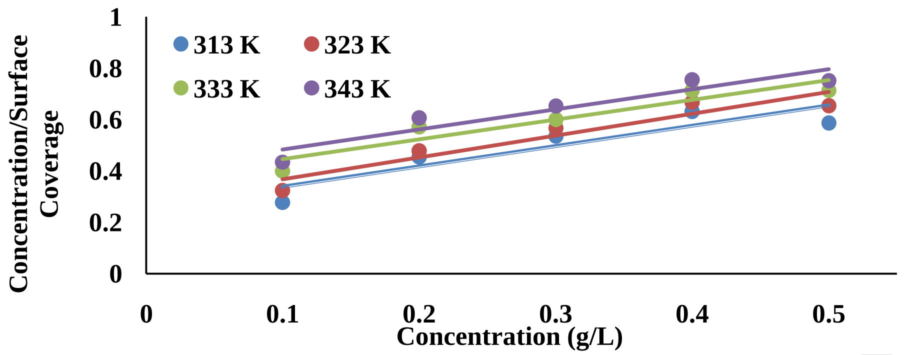

Fig. 7. Langmuir adsorption isotherm for mild steel in 1 M HCl solution in the presence of AHL extract

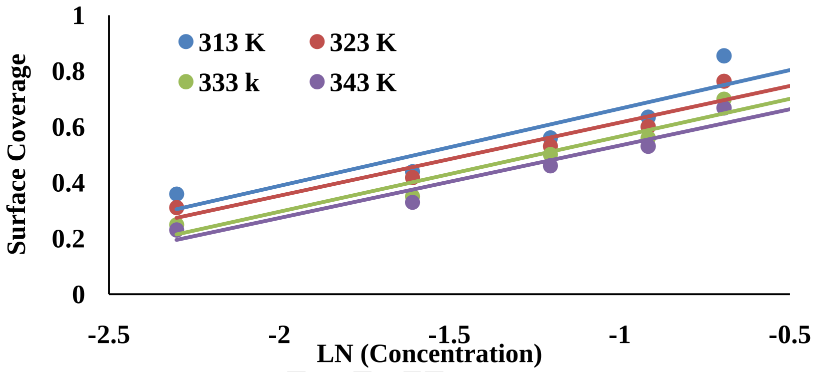

Fig. 8. Temkin Adsorption Isotherm for Mild Steel in 1.0 M HCl Solution in the Presence of AHL Extract

The plots of surface coverage against $ln(C)$ obtained from the expression (8)

$$\theta = \left(\frac{1}{f}\right) K_{ads}\, C) \tag{8}$$

where,f = factor that determines adsorbent - adsorbates interaction, θ = the degreeof surface coverage, K_{ads} = is the equilibrium constant and c = concentration of inhibitor ~~(Israel *et al.*, 2014)~~[This reference is not listed in Reference section andalso not in line with the journal reference style]; gives straight lines as shown in Figure 7 indicating that Temkin isotherm is also obeyed by AHL extract with R^2 values close to unity [23].

Table 1. Adsorption parameters for AHL inhibitor at various Temperaturesfrom Langmuir and Temkin isotherm models

	AHL inhibitor				**AHS inhibitor**			
Temp. (◦K)	**K_{ads} (L/mol)**	**$\Delta G^0{}_{ads}$(kJ/mol)**	**R^2 (Langmuir)**	**R^2 (Temkin)**	**K_{ads}**	**$\Delta G^0{}_{ads}$**	**R^2 (Langmuir)**	**R^2 (Temkin)**
313	5.625	-22.47	0.8133	0.8466	3.661	-21.35	0.829	0.9514
323	4.493	-22.58	0.8973	0.9189	3.089	-21.58	0.8561	0.9251

333	3.333	-22.46	0.8894	0.9401	2.5	-21.66	0.9339	0.9344
343	2.987	-22.82	0.8857	0.938	2.005	-21.68	0.936	0.9375

The standard Gibb's free energy of adsorption $\Delta G^0{}_{ads}$ (kJ/mol) value at temperature range 313 - 343 K was determined according to equation 9.

$$\Delta G^0{}_{ads} = -RT\ In\ 55.5K \qquad (9)$$

where, R is the universal gas constant (8.3145 J/mol.K), T is the absolute temperature in Kelvin (K), $\Delta G°_{ads}$ = Gibbs free energy of adsorption (kJ/mol) and the value 55.5 is the molar concentration of water in the solution in mol./K and K_{ads} is the film attractive power [9]. The negative values of $\Delta G°_{ads}$suggests that there was spontaneous adsorption of the extracts onto the metal surface.The values of $\Delta G°_{ads}$obtained are in the range -21.35 to -22.82 kJ/mol which indicate that it is more of physical adsorption mechanism than chemical adsorption.

Table 2. Calculated Values of Activation Energy and Heat of Adsorption at 313K and 343K for AHL Extract Concentrations

Inhibitor type	Extract Conc. (g/L)	E_a (kJ/mol)	Q_{ads} (kJ/mol)
AHL	Blank	24.214	-
	0.1	29.155	-18.913
	0.2	29.536	-13.904
	0.3	30.212	-11.943
	0.4	31.701	-10.60
	0.5	47.878	-31.93

Generally, values of $\Delta G°_{ads}$less negative or equal to -20 kJ/mol are consistent with physical adsorption, while values more negative than - 40 kJ/mole signify chemical adsorption [16, 19, 24]. Since the values of $\Delta G°_{ads}$ are below 40 kJ/mol, it implies that the adsorption process is physical and spontaneous. This is corroborated by increase in activation energy (E_a) with temperature in the presence of inhibitor compared to the uninhibited (blank) as presented in Table 2. The negative values of heat of adsorption (Q_{ads}**)** is an indication that the degree of surface coverage decreases with increase in temperature and resulting to increased reaction rate due to greater surface area exposed to the acidic environment and consequent lower inhibition efficiency . This also supports the assertion that the mechanism of adsorption for AHL extracts is physisorption [19].

4. CONCLUSION

From the investigation of corrosion inhibition properties of leaves extracts of *acanthospermum hispidum* in 1M HCl solution, it was concluded that the extracts can be used as effective corrosion inhibitors for mild steel corrosion in acidic medium. The corrosion rates were found to decrease with increase in inhibitor concentration but increased with rise in temperature. The inhibition efficiency also increased with increase in inhibitor concentration due to formation of protective film on the metal surface but decreased with temperature rise. The extract acted as mixed – type inhibitor modifying both the anodic and cathodic reactions. The adsorption process obeyed Langmuir and Temkin isotherm models Negative values of $\Delta G°_{ads}$ indicate spontaneous adsorption and thermodynamic parameters obtained show that the mechanism of adsorption was physisorption.

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