African Peach Leaves as a Mild Steel Corrosion Inhibitor in 0.3M HCl Solution

Abstract

The inhibition effect of African peach leaves methanol extract on the corrosion of mild steel in 0.3 M HCl aqueous solution at 303K was investigated by weight loss and polarization resistance. The results show inhibition efficiency decreased with temperature rise but increased with increase in plant extract concentration. Values obtained from polarization studies was relatively higher than that of weight loss at the same temperature. Also the activation energy (E_a), enthalpy and entropy for African peach methanol extract values were evaluated and found to favour adsorption of the plant extract onto the mild steel surface. Langmuir isotherm provided more accurate description of the adsorption behavior of the African peach leaves extract because values obtained R^2 were closer to unity when compared to others. The evaluated thermodynamic parameters show the process of the inhibition to be spontaneous. The polarization Tafel plot of the extract inhibition process proved it to be a mixed type inhibitor and the process of inhibition obey a first order kinetics with an improved half-life. The studied plant extract can be concluded to possess phytochemicals responsible for the inhibition of mild steel in 0.3M HCl solution.
Key words: Mild steel, African Peach, Corrosion Inhibition, Thermodynamics, Kinetics.

Introduction

Corrosion can be viewed as a universal phenomenon that occurs almost everywhere, be it in air, water, soil, and in every environment we encounter. It is known to a layman as rust (Burak and Gavgali, 2014). Corrosion is an unbearable phenomenon which destroyed the beauty of the materials and diminishes their life span (Geethamani, 2019). Corrosion is aided most a times by chemical cleaning, pickling and rescaling of the metals with corrodent reagents such as acid and other medium that corrode the metals in oil and gas industries (Ayuba and Abubakar, 2021). There is no single life on earth that is not affected by corrosion; those in developed countries suffered the most in the effect of corrosion structurally and economically, hence their life depends on developmental technology (Eddy et al. 2018; El-Etree et al. 2015). Corrosion phenomena, control and prevention are inevitably major scientific issues of concern that must be addressed daily as far as there are increasing needs of metallic materials in all phases of technological development (Ayuba and Aminullahi, 2020).

The use of organic molecules consisting of heteroatoms with unsaturated bonds and polar atoms like S, N, O, P etc. due to the lone pair of electrons present on them are potential corrosion inhibitors. They adsorbed on to the metal surface, and therefore loss of electrons from the metal surface can be avoided. Adsorption is one of the characteristic measures used by the inhibitors to be able to inhibit the activities of most corrodents on the surface of metals in different media (Dominic and Monday, 2016). In as much as inhibition of corrosion of metals need urgent approach to save the metallic world, polar atom compounds with high toxicity must be avoided by all means to safe guard the ecosystem. As such green plant materials of biological origin are inevitably and have found application in this respect. Extracts of plant sources are considered as an extremely wealthy source of various phytochemical compounds like alkaloids, flavonoids, phenolic, sugars that can be derived by relatively easy and cheap methods (Umaru and Ayuba, 2020). Both experimental and theoretical approaches have been used on numerous extract of this green natural product for the investigation of corrosion inhibition processes (Fouda et al. 2017).

In this research, leaves extract at varying concentrations of African peach was used for the inhibition of mild steel coupon at different temperatures in 0.3M HCl solution.

MATERIALS OF METHODS

Analysis and Preparation of Mild Steel Coupons

The composition of mild steel used in the experiment is as shown in Table 1. This shows that the mild steel coupon contains 81.3% iron, indicating a rich iron mild steel suitable for corrosion and corrosion inhibition studies.

Table 1. Elemental composition of the studied mild steel coupon

Element	Fe	Na	Al	Zn	Si	Mn	Nd	Cr	Ti
Composition (%)	81.37	9.3	5.76	2.48	0.40	0.27	0.16	0.08	0.07

The mild sheets were mechanically press-cut into coupons with each of the coupon having a dimension of 4cm×3cm×0.5cm. The coupons were then polished with different grades of SiC abrasive paper (#400 to #1200), degreased by washing with ethanol, dipped in acetone and allowed to dry in air before they were place in a desiccator to avoid contamination with environment for further use.

Collection, preparation and extraction of the plant samples

The sample of African peach leaves were collected from Apkuuna village Mbapenda, Mbazum Mbaterem Ukum in Ukum Local Government Area of Benue State, Nigeria and was transported to Chemistry Laboratory for further processes. 500g of the sieved powdered leaves of African peach sample that passes through 75µ meshed size were soaked in 2.5 litres of 95% methanol for two (2) weeks with constant agitation. The mixture was then filtered using Whatmann No 1 filter paper. To free the extract from ethanol, Rotatory (BUCHI labortechnik AG/ 9230 flawil/Switzerland) was used in order to concentrate the extract for quick drying. The concentrated extract was then air dried until a constant weight was recorded. The extract was crushed using mortar and pestle to be able to dissolve quickly while forming a solution of it with the corrodent (Hussaini et al. 2019).

Preparation of Corrodent Solution

The corrodent solution used for this study is 0.3M HCl and was prepared using equation (1):

$$V_{stock}\,(ml) = \frac{M \times C \times V}{10 \times \%purity \times density} \qquad (1) \qquad 1$$

Where (M) is Molar mass of the corrodent (C) is the concentration of the initial concentration of the corrodent V_{stock} is the volume of the stock solution. %purity and Density of HCl. The concentrated solution was further diluted using standard procedures to obtain the required concentration of 0.3M HCl (Ayuba and Abubakar, 2021; Hussaini et al. 2019).

Weight loss Measurements

In the course of this experiment, analytical weighing balance was used throughout for weighing the mild steel samples, before and after immersion in the acid medium. A previously weighed metal (mild steel) coupon was completely immersed in 100 cm^3 of the various test solutions of 0.0, 0.2, 0.4 and 0.6g/L extract prepared in 0.3M HCl concentration in each beaker. All the beakers were inserted in a vacuum thermostatic water bath at various temperatures of 303K, 313K and 323K. At an hour interval of total four hours, the test sample was retrieved from the corroded media, washed with distilled water and then dried in Acetone after another film clean up in methanol using a hard brush. The washed coupon was rinsed in acetone and air dried before reweighing. Corrosion rates (CR), degree of surface coverage (θ) and inhibition efficiency (%I.E) were obtained from the change in weights of the coupon's specimens of the inhibited and uninhibited experiment and were calculated using equations (2, 3, 4) respectively.(Zarrouk et al. 2012; Jyothi and Rathideri, 2017)

$$\%IE = (1 - \frac{Wi}{Wf})\times 100 \qquad (2)$$

$$\theta = 1 - \frac{Wi}{Wf} \qquad (3)$$

$$CR\ (g/hcm^{-2}) = \frac{\Delta W}{At} \qquad (4)$$

Where W_i and W_f are weight loss in grams (g) of mild steel in in absence and presence of inhibitor in HCl solutions respectively, (θ) is the degree of surface coverage (CR) is the corrosion rate, (ΔW) is the weight loss in (g), A is the area of one face of the spacemen (cm^2) and (t) is the period of immersion in hours (h).

Fourier transform infrared (FT-IR) analysis

Identification of major functional groups present in the African peach leaves extract was carried out using FTIR Instrument of model: Cary 630 FTIR Spectrophotometer (Agilent Technologies) before and after inhibition of mild steel coupons by the leaves extract in 0.3M HCl. All the coupons were separately dipped in 100cm^3 of 0.3M HCl of acid-inhibitor concentration respectively for 4 hours to enable the inhibitor form a layer on the surface of each coupon except the un-corroded coupons which was a fresh coupon from the desiccator. They were dried and the samples were scraped using a sharp razor blade which were taken for analysis. The analysis was done by scanning the sample through a wave number range of 650-4000 cm^{-1} (Jyothi and Rathideri, 2017; N'guessan et al. 2018).

Scanning Electron Microscopy

Morphology of inhibited with different extract concentration from 0.0-0.6g/L at the interval of 0.2g/L and the uncorroded blank mild steel surface was studied for one week at 303K by taking Prox scanning electron microscope (Phenom World Eindhoven). In the sample preparation analysis, a thin layer adhesives serving as carbon glue was attached onto a stub, and very small amount of the materials to be viewed spread on the stub materials and subsequently viewed in the instrument to obtain micrograph at an accelerating voltage of 15.00KV and x500 magnification (N'guessan et al. 2018).

Polarization Resistance Measurement

To obtain the electrochemical results of the corrosion effect on the mild steel in 0.3MHCl, mild steel coupons of dimension 1cm by 1cm were mechanically press-cut to fit into the Potentiostat. The coupons were first polished with abrasive of size 400 and 1200 to bring out the luster surface of the coupon and was letter degreased using methanol washed in distilled water and dry in the acetone before preserving them in a desiccator. The polarization resistance was carried out in an acid solution 0.3M (HCl) at 298K in the potential region of -10 to +10 mV at the scan rate of 0.333mV/s while 0.2g/L,0.4g/L and 0.6g/L respectively where the concentrations of the African peach leaves extract inhibitor used. Three electrode were used as usual; platinum counter electrode (CE), saturated calomel with Ag/AgCl reference electrode (RE) and working electrode (WE) which mild steel coupon was attached. Before carrying out the electrochemical measurements on the samples a 13.33 min call OCP was allowed to stabilization the solution, hence proven to give stable values of E_{corr}. Princepton Applied Research VersaSTAT4 model potentiostat was used for the analysis. Electrochemical parameters derived from polarization curves including corrosion potential (E_{corr}), corrosion current (i_{corr}), anodic and cathodic Tafel slopes (β_a and β_c) were measured and Tafel extrapolation conducted. The i_{corr} of both uninhibited and inhibited was calculated from the relation in equation (5), while the inhibition obtain from the electrochemical inhibition was calculated using the Stern-Greary equation (6) (Merchers and Wells, 2018).

$$icorr. = \frac{\beta a \beta c}{2.303(\beta a+\beta c)} \times \frac{1}{Rp} \quad (5)$$

$$IE(\%) = \frac{icorr - i^{\circ}corr}{icorr} \times 100 \quad (6)$$

Where, i_{corr}, i°_{corr}, β_a, β_c and R_p are the corrosion current density in (μAcm^{-2}) of uninhibited, inhibited, anodic Tafel plot, cathodic tafel plot and polarization resistance (ohms) respectively.

Results and Discussion

Weight loss Experiment

The corrosion inhibition of African peach leaves on mild steel was carried at various temperatures of 303K, 313K and 323K respectively in different concentration of the plant extract ranging from 0.2g/L to 0.6g/L at the interval of 0.2g/L in 0.3M concentration of HCl. The results obtain as presented in Table 2 shows the decrease in weight loss as the concentration of the inhibitor increased from 0.2g/L to 0.6g/L. The corrosion rate also decreases as the concentration of the inhibitor increase, but increased as the temperature rises. The inhibition efficiency (IE) of the inhibitor on the surface of the metal decrease as the temperature of the system increase but increase as the concentration of the inhibitor increase from 29.32% to 67.69%. This inhibition efficient could be as a result of the increase in the adsorption of African peach leaves extract on the metal/electrolyte boundary as the concentration increases (Jyothi and Rathideri, 2017; N'guessan et al. 2018). From the result obtained, the trend in corrosion rate is in good agreement with the concentration of the inhibitor and that of the corrodent, HCl.

Results also from Table 2 how that the weight loss of both inhibited and uninhibited mild steel increase with increase in temperature in all the concentration of the corrodent. The corrosion rate of the blank and the highest inhibited extract 0.6g/L of 0.3M HCl at 303K are $0.708 \times 10^{-4} g/hcm^2$ and $0.229 \times 10^{-4} g/hcm^2$ respectively. While at 323K the corrosion rate increase to $3.980 \times 10^{-4} g/hcm^2$ and $2.130\times 10^{-4} g/hcm^2$ for blank and higher inhibited extract under the same condition. The observation above shows that increase in temperature is in good relation with the rate of corrosion in the process (Jyothi and Rathideri, 2017; Chahul et al. 2015).

Table 2. Parameters obtained from weight loss experiment at varying temperatures.

Temp (K)	Conc. of Extract (g/L)	0.3M HCl ΔW (g)	$CR\times10^{-4}$ ($g/cm^2.h$)	θ	IE (%)
	0.0	0.0034	0.708	-	-
303	0.2	0.0018	0.375	0.4706	47.06
	0.4	0.0015	0.313	0.5588	55.88
	0.6	0.0011	0.229	0.6765	67.65
	0.0	0.0098	2.040	-	-
313	0.2	0.0066	1.370	0.3265	32.65
	0.4	0.0058	1.210	0.4082	40.82
	0.6	0.0047	9.790	0.5204	52.04
	0.0	0.0191	3.980	-	-
323	0.2	0.0135	2.810	0.2932	29.32
	0.4	0.0115	2.400	0.3979	39.79
	0.6	0.0102	2.130	0.4659	46.59

Fourier Transform Infrared (FTIR) Spectrometry

FTIR spectra were used to determine the functional groups present in African peach leaves extract and also the corrosion protective film formed on the surface of the mild steel after adsorption. The infrared spectra of uninhibited corroded mild steel, inhibited mild steel and methanol extract of African peach leaves are shown in the figures (1a-c). From the spectra in figure 1c, the un-inhibited corroded mild steel is infrared inactive, because there are no functional groups on it that could result in absorption bands. Comparing the absorption bands in spectra of both the inhibited corrosion product in figure 1c and that of the methanol plant extract before corrosion in figure 1b, shows that the bands position on the spectrum in figure 1b shifted from one position to another when compared to that of figure 1c as follows: 3301 to 3316 cm^{-1} (-OH stretching), 1701cm^{-1} with only intensity change (-C=O stretching), 1603 to 1622 cm^{-1} (C=C) 1103 to 1622cm^{-1} (C-O stretching). The above shifts in absorption bands reveal the adsorption of different phytochemical components of the African peach leaves extract on the mild steel surface. The surface analysis of the inhibited corroded mild steel using FTIR reveals that functional groups including –OH, –COOH, -C=C, =N-H, -C=N, C=O, are present in the extract may be involved in adsorptive interaction on the mild steel surface via hydrogen bonding and/or weak Van der Waals forces (Jyothi and Rathideri, 2017). Hence, the FTIR spectra studies reveal the interaction of the inhibitor on the metallic surface for adsorption in corrosion protection (Jyothi and Rathideri, 2017; N'guessan et al. 2018; Merchers and Wells, 2018; Felix et al. 3021).

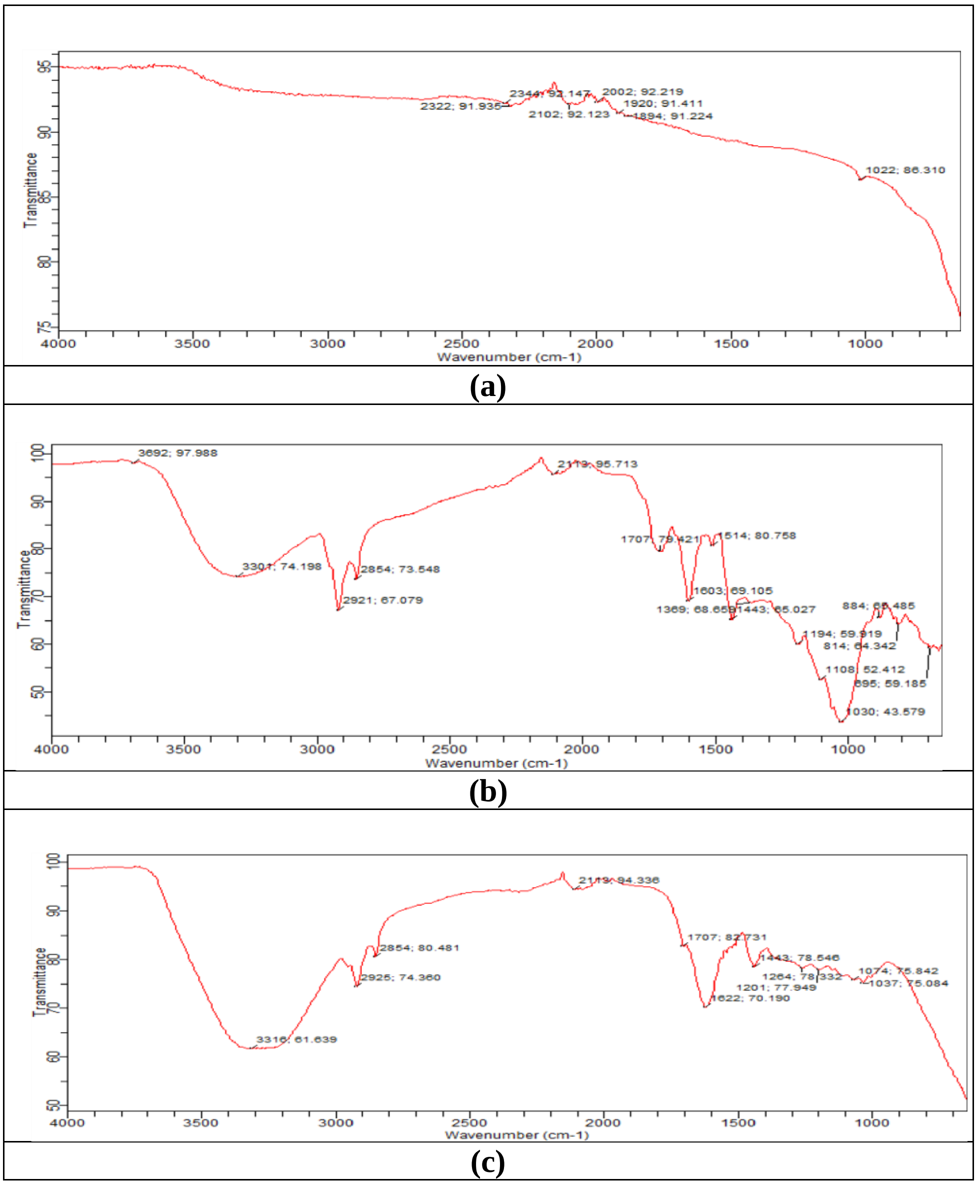
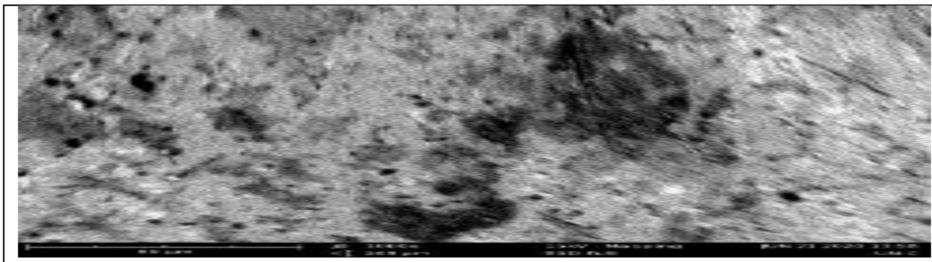


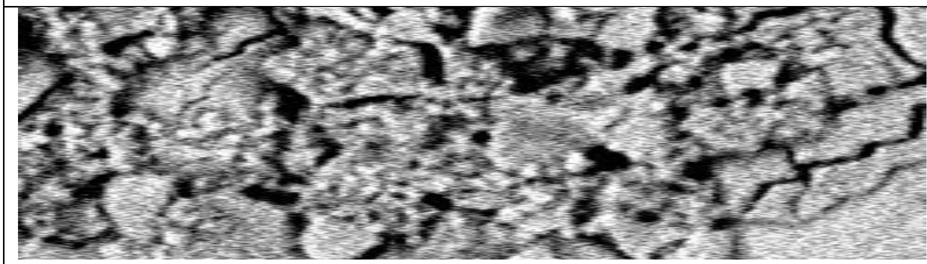
Figure 1. FTIR spectrum of (a) uninhibited corroded mild steel, (b) methanol leaves extract of African peach, (c) inhibited corrosion product of mild steel.

Scanning Electron Microscopy (SEM)

SEM images of magnification x1000 were recorded after one week (1wk) immersion of mild steel in 0.3M HCl and in the presence of varying extract concentration dissolved in 0.3M HCl solutions using Electron probe micro analyzer (Meften et al. 2017). The results are as presented in figure (2a-e). The snap shot with the most morphemically damaged surface is the one immersed in the uninhibited 0.3M HCl medium as presented in the figure (2b), where visibly presence of patches, cracks and pits could be observed all over the surface when compared to figure (2a) which is that of the uncorroded mild steel. Figures (2c-e) are the snap shots of the inhibited mild steel in 0.3M HCl with concentration of 0.2g/L extract figure (2c), 0.4g/L extract figure (2d), 0.6g/L extract figure (2e) respectively. It is clear from the snap shots that the surface coverage of the extract across the surface of the mild steel increases as the concentration of the extract increases having a higher coverage which also has the highest inhibition efficiency from the data in table 1.



(a)



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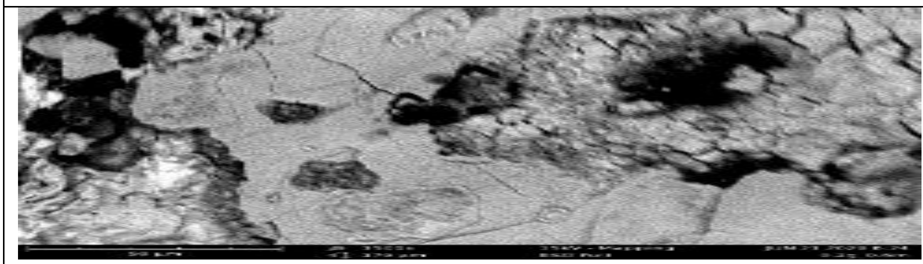
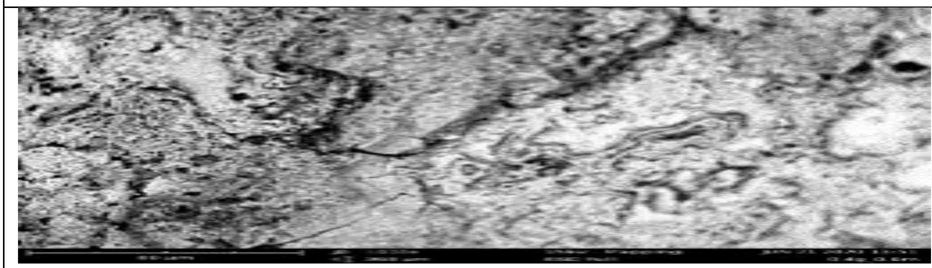


(c)



(d)

(e)

Figure 2. SEM micrographs of mild steel dipped in 0.3 M HCl of (a) Uncorroded, (b) the uninhibited 0.3MHCl, (c) 0.2g/L extract (d) 0.4g/L extract (e) 0.6g/L extract in 0.3M HCl solutions.

Electrochemical measurements

The result of the linear polarization resistance shows that, the inhibitor is a mixed type inhibitor as indicated in Figure 3 and has the highest inhibition of 80.00% and the least was 77.79% (Table 3). It can be observed that the increase in inhibition, increases with increase in concentration of the plant extract. Hence, Tafel plot as presented in figure 3 below is in a good relationship with the reaction in absence and presence of the inhibitor. The measurement further show that, the results obtained from linear polarization resistance is relatively better than that of weight loss measurements (Ayuba and Aminullahi, 2021; Elmi et al. 2019).

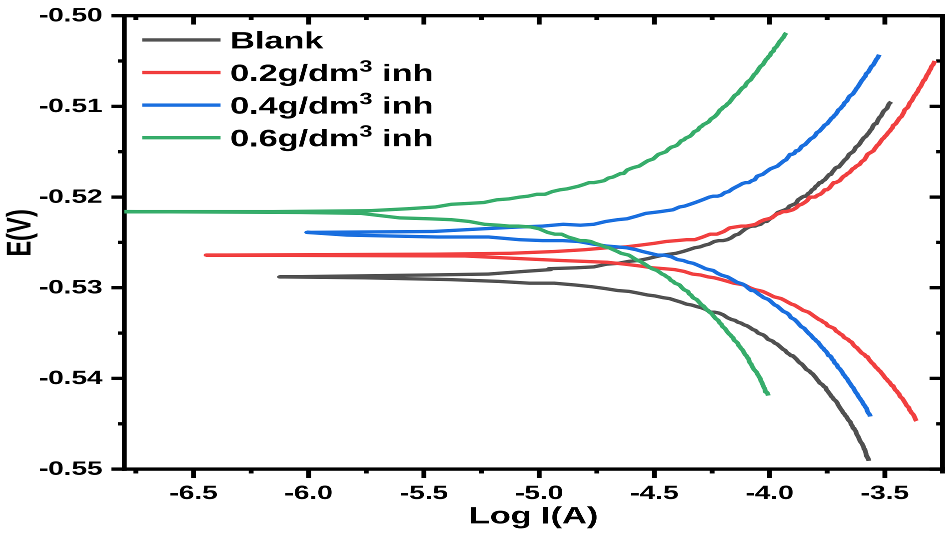


Figure 3. Overlayed Tafel polarization curves for mild steel corrosion inhibition in 0.3M HCl solution in varying concentrations of methanol leave extract

Table 3. Electrochemical parameters obtained from tafel polarization techniques for mild steel in 0.3M HCl solutions in varying concentrations of extracts at 298K

Extract (gL^{-1})	i_{corr} (μAcm^{-2})	E_{corr} (mV)	B_a ($mVdec^{-1}$)	B_c ($mVdec^{-1}$)	C.R ($mmpy^{-1}$)	%I
0.0	341.175	-528.8	73.768	59.611	1.1156	-
0.2	75.7459	-521.6	66.811	65.624	0.2477	77.79
0.4	70.8239	-523.9	67.35	62.848	0.2316	79.24
0.6	68.2698	-526.4	58.209	73.787	0.2232	80.00

Adsorption Isotherm Studies

Isotherms can generally be represented as shown in equation (7) from which equation (8) for Langmuir and equation (9) for Freundlich were generated:

$F(\theta, x)exp(-2a\theta) = Kads\ Cinh$ (7)

$C/_{\theta} = 1/_{Kads} + C$ (8)

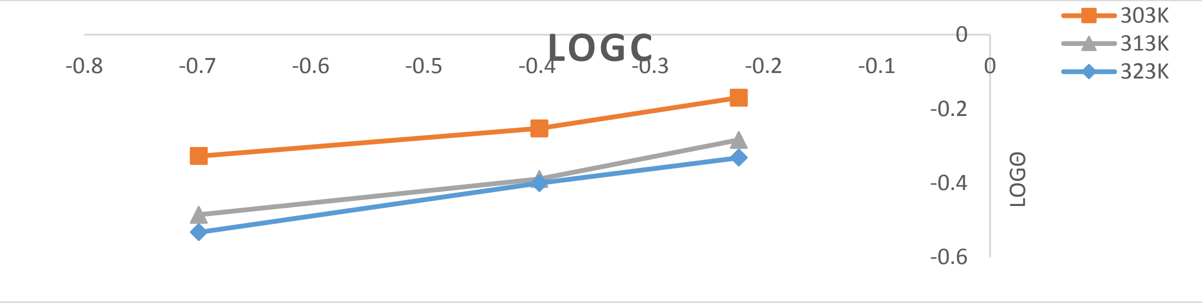
$log\ \theta = nlog\ C + ln\ Kads$ (9)

The values of K_{ads} obtained from the intercept of plots (Figure 4) for equations (8) and (9) were used to calculate the standard free energy (ΔG_{ads}) through relation giving in equation (10):

$\Delta Gads = -RTIn(Kads55.5)$ (10)

Where R is the molar gas constant, T is the absolute temperature and 55.5 is the concentration of water in solution expressed in M.

Langmuir equation is an ideal isotherm for physical and chemical adsorption (Beniken et al. 2018). From table 4, values of equilibrium constant K_{ads} were all positive, indicating a favorable adsorption (Ituen and Udo, 2012). The data obtain from the experiment have higher values of R^2 for Langmuir in relation to Freundlich. This prove the plant extract to form a monolayer adsorption on the mild steel surface (Sail et al. 2019).



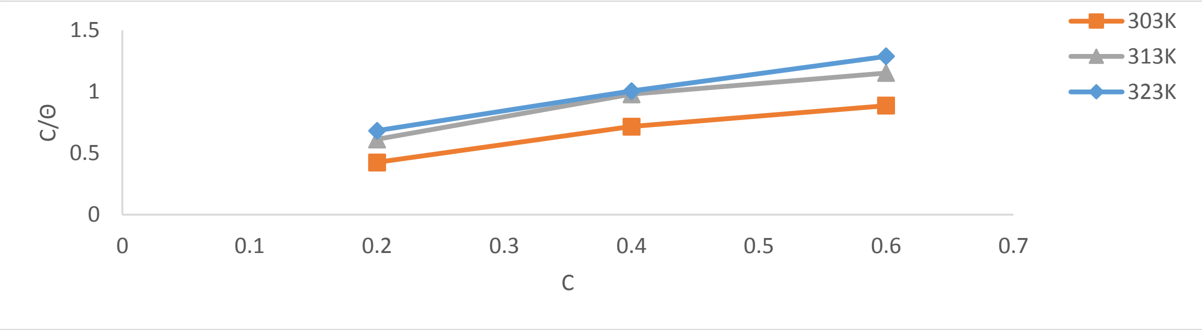


Figure 4: Plots of (a) Freundlich, (b) Langmuir isotherm for the adsorption of plant extract on the surface of mild steel.

Table 4 also presented the calculated values of ΔG_{ads} of the adsorption process. All the calculated values were negative for the range of temperatures analyzed ranging from -14.003 to –9.228 kJ/mol for the extract respectively. This shows that the adsorption process is spontaneous with values less than the threshold of -40 kJ/mol signifying a physical adsorption process (Sail et al. 2019).

Table 4. Calculated Gibb's free energy and K values of adsorption

Isotherm	Temperature (K)	Slope	ΔG_{ads} ($kJmol^{-1}$)	R^2	K_{ads}
Langmuir	303	1.15	-14.003	0.9781	4.67
	313	1.35	-13.011	0.9586	2.67
	323	1.51	-13.344	0.9905	2.59
Freundlich	303	0.32	-9.494	0.9677	0.78
	313	0.41	-9.228	0.9698	0.62
	323	0.42	-9.333	0.990	0.58

Effect of temperature

Activation energy of the inhibition process of African peach leaves extract on the surface of mild steel was obtained using Arrhenius equation (11). From the equation, a plot of lnCR against 1/T produced a slope equals the value of (−Ea/R) (Figure 5). Table 5 shows calculated values of Ea. An increase in the value of the calculated activation energy in relation to increase in plant extract concentration, further shows that the inhibitor has effect on the surface of mild steel by minimizing or preventing the corrosion of the mild steel surface by the studied corrodent (Anupama et al. 2015; Obot et al. 2019).

$$lnCR = lnA - Ea/RT \qquad (11)$$

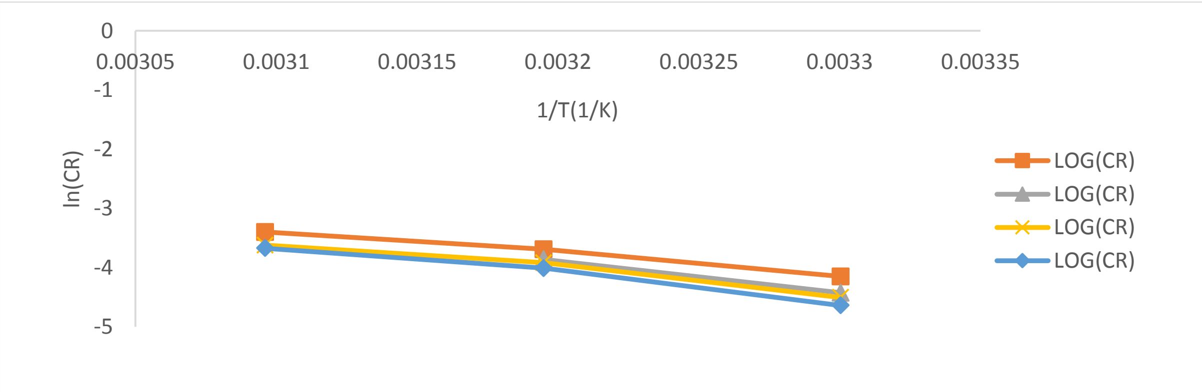


Figure 5. Arrhenius plots for inhibition of mild steel using different concentration of African peach methanol leave extract in 0.3 M HCl

To calculate thermodynamic parameters, change in enthalpy (ΔHa) and change in entropy (ΔSa) of adsorption of the plant extract onto the mild steel surface, equation (12) was used. By plotting ln(CR/T) against the inverse of T using equation (12), the results is as shown in Figure (6) with a slope equals to −ΔHa/R and an intercept equivalent to $ln(R/NAh + \Delta Sa/RT)$. Both values of ΔHa and ΔSa where obtained from the slope and intercept respectively. From the values of ΔSa obtained from the graph which is the entropy of the reaction process. The values of the calculated ΔHa increases with increase in the plant extract concentration, while that of ΔSa decease negatively with increase in plant extract concentration which shows that the degree of disorderliness of the plant extract on the metal surface reduces as the concentration of the inhibitor concentration increase (Menticelli, 2018). The thermodynamic values of the adsorption are as represented in table (5).

$$\ln CR/T = \ln(R/NAh + \Delta Sa/RT) - \Delta Ha/RT \qquad (12)$$

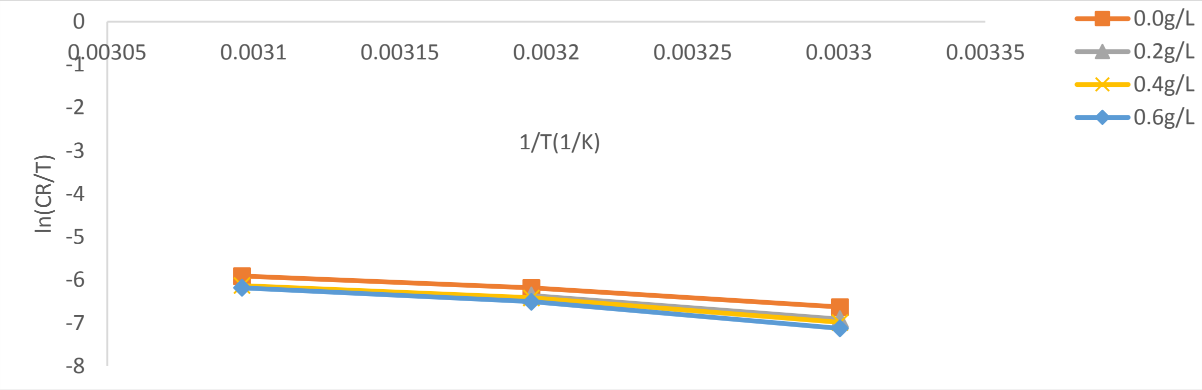


Figure 6. Transition state equation plot for the determination of thermodynamic parameters

Kinetic study

The kinetic studies of the interaction of the plant extract on mild steel corrosion in 0.3M HCl aqueous solution was studied by fitting kinetic data obtain from weight loss experiment into different kinetic model equations. These include zero, first and second order kinetic equations. The test revealed first order obtained by plotting –log (weight loss) against time to be relatively the best fit into the kinetic data generated with R^2 values of whose were closer to unity than others. Table (5) show the calculated half-life values for first order kinetics obtained using equation (13). The half-life of the corrosion and corrosion inhibition process of mild steel in relation to the plant extract shows that values obtained in the blank (0.3M HCl) uninhibited system is relatively lesser when compared to the half-life values of the mild steel obtain from the inhibited medium of the immersed coupons of the mild steel. It was also observed that as the concentration of the plant extract increases so also the half-life. Therefore, the methanol extract of the African peach leaves plays a role as an inhibitor of the mild steel in the 0.3M HCl aqueous medium by reducing the rate of corrosion of the mild steel in 0.3M HCl solution (Merchers and Wells, 2018; Barreto et al. 2017).

$$t_{1/2} = \frac{0.693}{k_1} \qquad (13)$$

Table 5: Tabulated values of activation energy (Ea) and half-life and some thermodynamic parameters of mild steel corrosion inhibition using African peach methanol extract

Extract (gL^{-1})	Ea ($kJmol^{-1}$)	R^2	k_1	$t_{1/2}$ (hrs)	ΔHα, ($kJmol^{-1}$)	ΔSα ($kJmol^{-1}$)
0.0	70.398	0.9876	0.0788	8.799	67.779	-0.100
0.2	82.218	0.9785	0.0587	11.800	79.618	-0.066
0.4	83.149	0.9718	0.0573	12.085	80.549	-0.064
0.6	90.891	0.9760	0.0559	12.383	88.291	-0.042

Conclusion

From the corrosion inhibition, kinetic and thermodynamics studies on the corrosion inhibition of mild steel in 0.3M HCl medium using different concentration of African peach leaves methanol extract, the following can be concluded:

- ✓ The inhibition efficiency of the African peach leaves extract on the surface of mild steel increase as the concentration of the extract increases from 0.2 to 0.6g/L with experimental inhibition efficiency of 67.65% when compared to that obtained from linear polarization resistance of 80.00% at 303K
- ✓ That the extract of African peach leaves was established to be a mixed type inhibitor and obeys first order kinetics model.
- ✓ The adsorption process of the plant extract on the surface of mild steel follows Langmuir isotherm which is indicator of monolayer kind of inhibitor. The adsorption was spontaneous and obeyed the mechanism of physical adsorption.
- ✓ The increase in activation energy and half-life of the inhibited system in relation to the uninhibited with increase in concentration of the plant extract further confirms the process of inhibition of the plant extract on mild steel surface.

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