Original Research Article

*Acalyph*a *wilkesiana* acid extract as a potential green inhibitor for corrosion of mild steel in acidic medium: Thermodynamics and kinetics study

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Abstract
The use of synthetic compounds as corrosion inhibitor is diminishing due to strict environmental regulations and toxic effects of the compounds on human beings and the environment. Consequently, there exists the need to develop a new class of corrosion inhibitors with low toxicity, eco-friendliness and good efficiency. Therefore, this study investigates the inhibitory potential of *Acalypha wilkesiana* (AWAE) extract on mild steel in 1M HCl using weight loss method. Experiments were performed by varying immersion period, concentration of the inhibitor and temperature. The results obtained showed a decrease in the corrosion rate of mild steel as the extract concentration increases while the inhibition efficiency increases with extract concentration. Effect of temperature on the inhibition efficiency showed an increase in inhibition efficiency with rise in temperature. The adsorption of the inhibitor on the mild steel surface in acid solution was found to obey the Langmuir adsorption isotherm. Calculated thermodynamic parameters revealed that the mechanism of the corrosion inhibition may be due to the chemical adsorption of the phyto-constituents on the surface of mild steel. Kinetic treatment of the data followed a first order reaction and the half-life values of the metal increase with increase in the extract concentration. Preliminary investigation of the phytochemical constituents showed that *Acalypha wilkesiana* contains tannin, flavonoid, saponin, alkaloid, steroid , terpenoid and some other compounds in trace quantity. FTIR results indicate that AWAE contains O and N atoms in functional groups and aromatic ring which meet the general consideration of a typical corrosion inhibitor.

***Keywords*:** Corrosion inhibition, Mild steel, Thermodynamic

INTRODUCTION
Corrosion is a process that occurs as a result of the electrochemical reaction between the anodic and the cathodic sites of the metals or alloys with corrosive media causing the metal surface to corrode[1]. Due to the relatively low cost and material properties of mild steel, it is used for various applications in the area of food, petroleum, chemical and power production [2]. Meanwhile, one of the challenges facing the industries on the use of mild steel is its susceptibility to corrosion when exposed to corrosive media [3]. Thus, to mitigate the rate of corrosion of mild steel for safety

use in the environment and industries, various corrosion inhibitors have been employed. Several studies have been carried out on the use of organic and inorganic compounds that can inhibit corrosion of mild steel. In spite of their high inhibition efficiencies, these groups of inhibitors have been found to be carcinogenic and mutagenic, hence, they are not environmentally friendly [4]. Due to these apparent shortcomings in the use of inorganic inhibitors, attention is now focused on the use of non-toxic, readily available, low-cost and biodegradable natural occurring materials [5]. The use of naturally occurring substances to inhibit the reaction and rate of corrosion of metals in acidic and alkaline environment has been well documented. Some of these are: lignin [6] *Azadirachta Indica*[7] *Hibiscus Sabdariffa*[4], *Delonix regia*[8] and *Opuntia* extract[9]. These inhibitors mitigate the rate of corrosion of a metal or alloy by decreasing the diffusion rate for reactants to the surface of the steel and the electrical resistance of the steel surface[10]. The efficiency of organic corrosion inhibitors is related to the presence of some functional groups such as O-H, N-H, C=C, C=N, C=O and aromatic ring in their structure. Reports have also shown that inhibition efficiency of an inhibitor can be improved by the addition of halide ions to the inhibitor, a process referred to as synergism [11]. In this study, we report the use of acid extract of *Acalypha wilkesiana* (AWAE) as an efficient corrosion inhibitor of mild steel in 1M HCl solution. *Acalypha wilkesiana* is an evergreen shrub which has a stem erected with many branches having a closely arranged crown. Previous reports have shown that *Acalypha wilkesiana* contains some phytochemicals which contain hetero-atoms in functional groups (O-H, N-H, C=C, C=N, C=O, C-O) and aromatic ring which could make it function as a good corrosion inhibitor [12 -14]. To the best of our knowledge, no work has been reported in the literature on the kinetics and thermodynamic study of *Acalypha wilkesiana* as corrosion inhibitor for mild steel in acidic medium. Therefore, we have investigated the kinetics and thermodynamic properties of *Acalypha wilkesiana* as corrosion inhibitor for mild steel using gravimetric method.

MATERIALS AND METHODS

Materials preparation

Mild steel having the composition (wt %): 0.2150 C, 0.2580 Si, 0.4665 Mn, 0.0090 S, 98.9880 Fe. was used for this study. The sheet was cut to form different coupons of dimension 18 x 15 x 5 mm and polished with different grades of Emery paper. The samples were thoroughly washed with de-ionized water and acetone (AR grade) and allowed to dry in the air before preservation in a desiccator. Accurate weight of the samples was taken using an electronic balance. All chemicals used for this study were of analytical grade and de-ionized water was used for the sample preparation.

Extraction of plant

Acalypha wilkesiana leaves were obtained from teaching and research farm, Federal University of Technology Akure (FUTA), authenticated by Crop, Soil and Pest Management Department, FUTA. AWAE was dried in the oven at 80 °C for 2 h, after which, it was pulverized into fine powder using industrial grinding machine and sieved with 50 μm mesh. Thirty grams of the powdered sample was weighed into a 500 mL beaker and 300 mL dilute 1M HCl was added. The mixture was transferred into water bath at 90 °C for 3 h for extraction. After the extraction, the mixture was cooled overnight and filtered. The filtrate was stored in 250 mL amber bottles at room temperature. From the stock solution obtained, inhibitor test concentrations of 1 – 5% (v/v) were prepared by diluting it with 1M HCl solution. Experiments were performed by varying contact time, initial concentration of the inhibitor and temperature.

Weight loss measurement

Weight loss method was used to evaluate the corrosion rate and inhibition efficiency. For this study, mild steel coupons of a known mass were immersed in 100 mL of 1 M HCl blank and with the addition of the different concentration of AW extract at temperature ranges of 303–333 K in a thermostated water bath. The coupons were retrieved after 2 hours and the difference in weight was taken as weight loss. The weight loss values were used to calculate the corrosion rate (CR) and inhibition efficiency of the mild steel using equations 1 and 2 respectively [34] .

$$\textbf{I.E \%} = \left(1 - \frac{CR_{inh}}{CR_{blank}}\right) \times 100 \qquad \textbf{(1)}$$

Where CR_{inh} and CR_{blank} are the corrosion rates in the presence and absence of the inhibitor, respectively.

$$\textbf{CR (mgh}^{-1}\textbf{ cm}^{2}\textbf{)} = \frac{\Delta W}{AT} \qquad \textbf{(2)}$$

Where ΔW is the weight loss of mild steel in the absence and presence of the inhibitor, respectively, A is the area of the coupon in cm^2, and T is the period of immersion in hours.

Chemical Analysis

Phytochemical analysis of *Acalypha wilkesiana* extract was carried out according to the standard methods**.** Briefly, tannin determination was carried out according to the previously reported method [15] while the spectrophotometric method of [16] was used for Saponin determination. The procedure described by [17] was employed for the determination of cardiac glycoside and terpenoid in the sample. The total flavonoid content of the extract was determined using a colourimeter assay as previously reported [18].

Fourier Transform-Infrared Measurement

The AWAE was characterized by Fourier transform infrared (FT-IR) spectroscopy. Powdered mild steel specimen was immersed separately in solution of hydrochloric acid containing the extract for 2 hours to form the adsorption product. FT-IR spectrum for the extract and the adsorption product was recorded. The spectra were recorded using perkin –Elmer -1600 spectrophotometer using KBr pellects with the frequency ranging from $4000cm^{-1}$ to $350cm^{-1}$

RESULTS AND DISCUSSION

Weight loss measurement

Effect of extract concentration on corrosion rate of mild steel

The variation of corrosion rate of mild steel in 1 M HCl in the absence and presence of various concentrations of acid extract of AW was studied at 298 and 313 K, respectively. The result (Figure1) obtained showed that the corrosion rate of mild steel in 1 M HCl decreases with increase in the concentration of the extract at both temperature values under study. This is expected because there is an increase in the number of adsorption of the extract components on the surface of the mild steel as the concentration of the extract increases. This makes a barrier for mass transfer and prevents further corrosion.

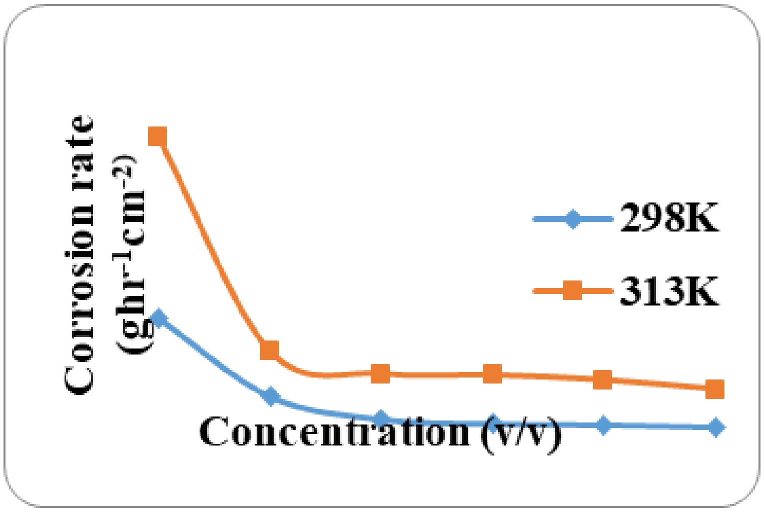

Figure 1: Effect of extract concentration on corrosion rate of mild steel at 298K and 313K.

Effect of Temperature on Corrosion rate of mild steel

The effect of temperature on the corrosion rate of mild steel with and without *Acalypha wikesiana* extract was studied in the temperature range of 303K to 333K as shown in Figure 2. It was found that the rate of corrosion of mild steel with and without different concentrations of the extract in acid solution increases with temperature ranging from 1.1679 ghrcm^2 at 303 K to 5.340 ghrcm^2 at 333K. However, it can be observed from the Figure that the corrosion rate of mild steel in the inhibited acid solution is much more decreased than the corrosion rate in the uninhibited acid solution. This is plausible because decrease in the corrosion rate of the inhibited acid solution is indicative of the mitigating effect of inhibitors on the corrosion rate of mild steel [19, 20]. Thus, as the temperature increases, the average kinetic energy of the reacting molecules increases which leads to an increase in the corrosion rate.

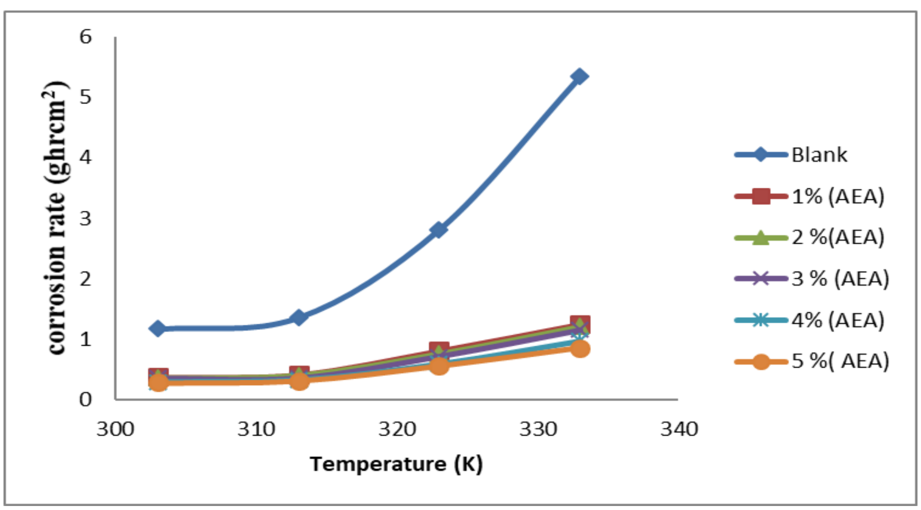

Figure 2: Effect of Temperature on the corrosion rate of mild steel.

Effect of Extract concentration on inhibition efficiency.

The results of the effect of extract concentration on the inhibition efficiency of the mild steel at 298 and 313 K is as shown in Figure 3. The inhibition efficiency of the extract increases with increase in the concentration of the inhibitor as the temperature increases. This is probably due to the increase in the fraction of the mild steel surface covered by the adsorbed extract components as the concentration of the plant extracts increases. The inhibition efficiency increases gradually as the concentration of the extracts increases up to about 5% (v/v), after which further increase in extract concentration did not cause any significant change in the performance of the extract. It was observed that the inhibition efficiency increases as the reaction temperature increases. Previous studies have shown that increase in inhibition efficiency with increase in inhibitor concentration can be explained on the basis of increased adsorption of the extract molecules on the metal surface. [20, 21]. Thus, the results obtained showed that the adsorption of the phytochemical constituents on the surface of the mild steel follows a chemical adsorption mechanism.

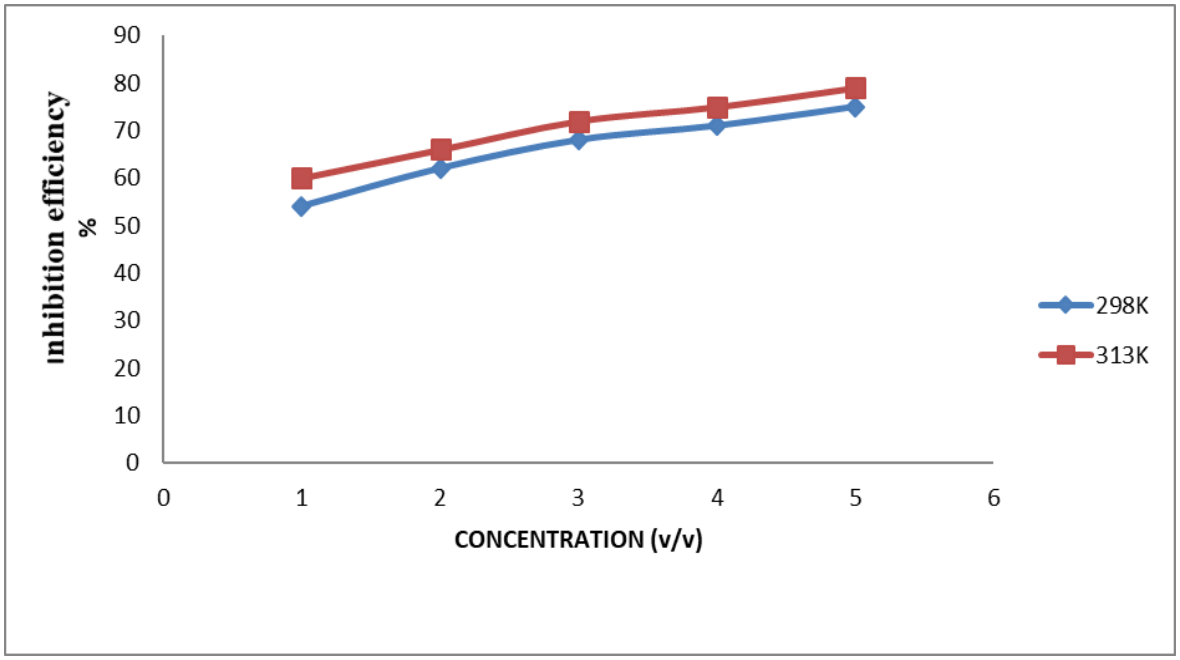

Figure 3: Effect of concentration on the inhibition efficiency of AWAE on mild steel in 1M HCl.

Effect of temperature on inhibition efficiency

The effect of temperature on the inhibition efficiency of AWAE on mild steel in 1M HCl is shown in Figure 4. According to the Figure, the inhibition efficiency of the extract increases with increase in the temperature at different extract concentration. Previous investigators have described increase in inhibition efficiency with temperature as being suggestive of chemical adsorption mechanism while decrease in inhibition efficiency with analogous increase in temperature indicates physical adsorption mechanism [19]. In this study, it can be inferred that the mechanism of adsorption of AWAE on mild steel is chemical adsorption due to an increase in inhibition efficiency with increasing temperature. At the highest concentration of the extract employed in this study, the inhibition efficiency increases from 76.45 % at 303K to 83.99 K at 333K.

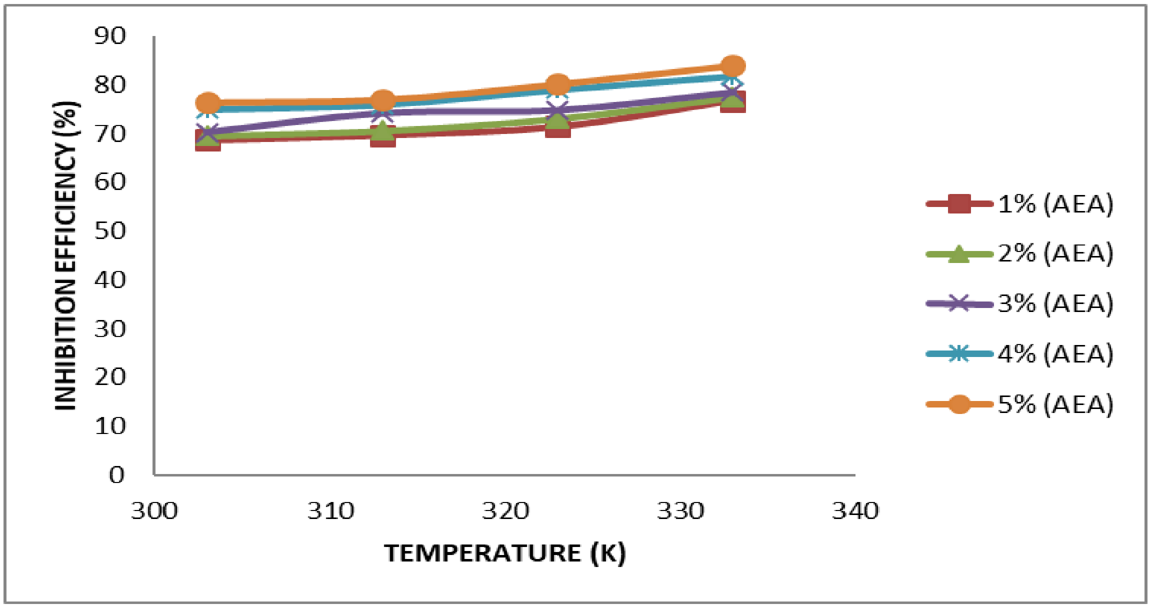

Figure 4: Effect of temperature on inhibition efficiency of AWAE in 1 M HCl

Adsorption isotherms

The nature of inhibitor interaction on the corroding surface during corrosion inhibition of metals has been deduced in terms of adsorption characteristics of inhibitor. The inhibition of the corrosion of mild steel in acidic solution with addition of various concentrations of the extract can be explained by the adsorption of the components of the plant extract on the metal surface. The percentage inhibition efficiency (I.E%) of mild steel is directly proportional to the fraction of the surface covered by the adsorption molecule (θ). Therefore, the adsorption isotherm provides important clues regarding the relationship between the surface of the metal covered with the adsorbed species and the concentration of extract in solution [19-22]. The data obtained in this study were fitted graphically into different isotherms and the results showed that Langmuir isotherm was found to be most suitable for the experimental data. Langmuir isotherm is based on the assumption that all adsorption sites are equivalent and that bindings occur independently from nearby site [23]. In order to obtain the adsorption isotherm, the values of the surface coverage (θ) at various concentrations of the inhibitor in 1M HCl solution for the extract were plotted as C/θ vs C (Eqn. 3.) and the adsorption parameters derived from the plot are listed in Table 1.

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad \textbf{(3)}$$

Where θ is the degree of surface coverage, C the molar inhibitor concentration in the bulk solution and K_{ads} is the equilibrium constant of the adsorption process. The plots are linear as revealed by the regression coefficient (r^2) with slope equal to unity. The r^2 obtained with strong positive values being close to unity suggests that the adsorption process of the extract on mild steel in 1M HCl solution conforms to Langmuir isotherm [35] . Thus, Langmuir adsorption isotherm (Figure 5) was found to best describe the adsorption mechanism for AWAE extract on the mild steel in 1 M HCl.

Table 1: Changes in adsorption equilibrium constant (K_{ads}) at different temperatures for AW extract

Temperature	Correlation (R^2)	Slope	K_{ads}	ΔG_{ads}
303	0.998	1.265	3.144	-13.01
313	0.999	1.249	3.235	-13.81
323	0.998	1.200	3.621	-14.01
333	0.999	1.175	4.606	-15.35

Table 1 showed the parameter obtained from the linear graph. Larger values of K imply more efficient adsorption and hence better inhibition efficiency [20]. The value of K obtained in this study increase with increase in temperature. This shows that the adsorption of *Acalypha wilkesiana* extract on mild steel is chemisorption and also indicates an increase in inhibition efficiency with temperature. The correlation efficiency and the slope obtained tend to unity indicating that the adsorption followed Langmuir isotherm.

The standard free energy of adsorption (ΔG_{ads}), was calculated from the adsorption equilibrium constant using the Eq 4: $\Delta G_{ads} = -2.303RT \log[55.5K_{ads}]$ **(4)**

The values of ΔG_{ads} obtained are presented in Table 1. The negative values of ΔG_{ads} ensure the spontaneity of adsorption process and stability on the adsorbed layer on the mild steel surface.

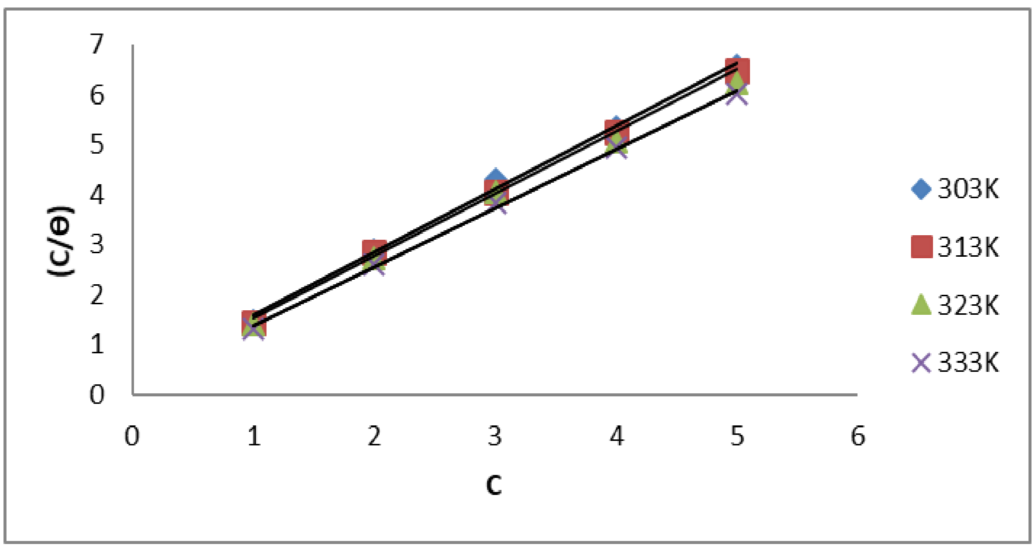

Figure 5: Langmuir Isotherm plot

Thermodynamic study

The effect of temperature on the corrosion rate of mild steel in the absence and presence of different concentrations of *Acalypha* extract were studied in the temperature range of 303 to 333 K using weight loss measurements. To evaluate the activation energy parameters, the logarithm of corrosion rate in the absence and presence of AWAE in 1 M HCl was plotted against temperature using equation 4. [24-27]

$$Log\ CR = \frac{-E_a}{2.303RT} + Log\ A \qquad \textbf{(4)}$$

Where *CR* is the corrosion rate, *E*a is the apparent activation energy; *R* is the molar gas constant, *T* is the absolute temperature and *A* is the frequency factor.

As shown in Figure 6, a plot of log CR versus 1/T for mild steel corrosion in 1 M HCl in the absence and presence at various concentrations of AW extract was linear with slope equal to $-E_a/_{2.303R}$. The values of E_a for the corrosion reaction in the absence and presence of AW extract were calculated and are presented in Table 2. The results from the Table show that the values of Ea in the absence of inhibitor were higher than those in the presence of the extract. Similar results were reported by [28]. It has been established that the higher value of the activation energy of the adsorption process in the presence of inhibitor when compared to its absence can be attributed to physisorption, while the reverse is attributed to chemisorption [29]. In this study, it can be concluded that the mechanism of adsorption of AWAE onto the surface of the mid steel is chemisorption.

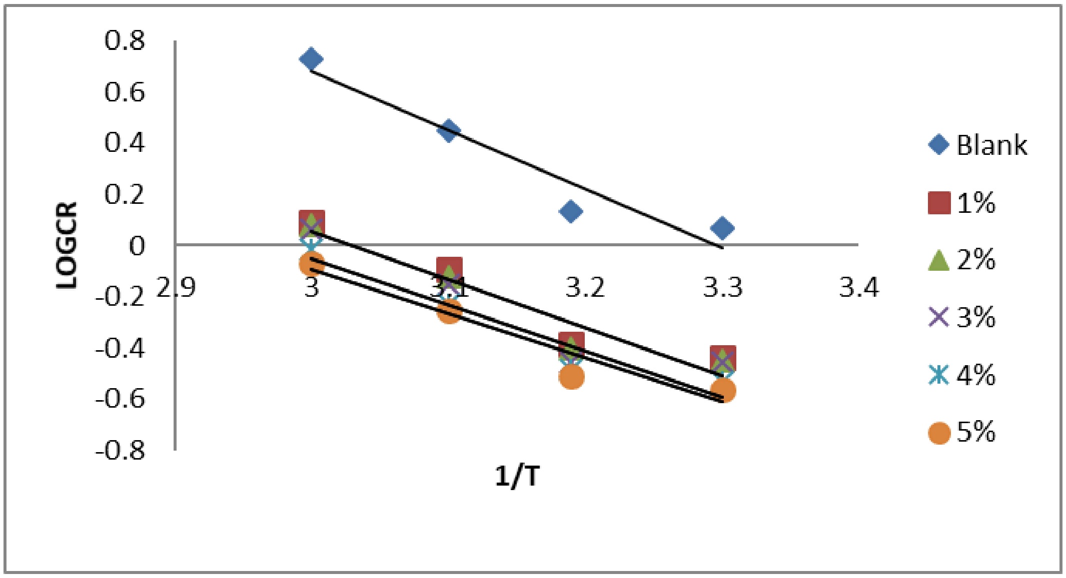

Figure 6: Arrhenius plot (Log *CR* versus 1/*T*) for mild steel corrosion in 1 M HCl in the absence and presence of various concentrations of AW extract.

Thermodynamic parameters such as enthalpy (ΔH) and entropy (ΔS) of activation of corrosion process may be evaluated from the effect of temperature and are calculated using equation 5.

$$CR = \left(\frac{RT}{Nh}\right) exp\left(\frac{\Delta S^{\circ}}{R}\right) exp\left(\frac{-\Delta H^{\circ}}{RT}\right) \quad \textbf{(5)}$$

where *h* is the Planck's constant, *N* is the Avogadro's number, *T* is the absolute temperature and *R* is the universal gas constant. A plot of Log (*CR/T*) vs 1/*T* is a straight-line graph as shown in Figure 4 with a slope of (- ΔH/ 2.303R) and intercept of (logR / nh + Δs / 2.303R) from which $\Delta H°$ and $\Delta S°$ were calculated and presented in Table 2. The results in Table 2 showed that the enthalpy of activation values are all positive which reflects the endothermic nature of the mild steel dissolution process. The negative values of entropy (ΔS^o) implies that the activated complex in the rate determining step represent association rather than dissociation, implying that orderliness increases on going from reactant to activated complex [20, 21].

Table 2: Activation and thermodynamics parameter for the corrosion inhibition of mild steel in the absence and presence of *Acalypha wilkesiana*.

Concentration (v/v)	Ea (kJ/mol)	ΔH (kJ /mol)	ΔS (kJ/mol/K)
Blank	43.987	41.34	-108.79
1	35.988	33.34	-144.51
2	32.817	33.17	-145.36
3	30.561	33.23	-146.71
4	28.872	30.49	-150.14
5	25.130	26.33	-156.22

Kinetic Study

Determination of rate constant and half-life

The effect of time on the inhibitive behavior of the AWAE in the absence and presence of the extract at 303 K was studied. The plot of logarithm of final weight loss (W_f) against time (t) in days (Fig. 7) with respect to mild steel in 1 M HCl solution in the absence and presence of the extract showed a linear variation with slope (k_1) suggesting a first order reaction kinetics. The rate constant of the reaction process at different concentrations of the inhibitor was obtained from the slope of the graph, and the half-lives were calculated from the rate constant using equation 6.

$$t_{1/2} = \frac{0.693}{K_1} \qquad \textbf{(6)}$$

The values of the rate constant and the calculated half-lives are listed in Table 3. The results showed that the half-lives of the metal increase as the concentration of the extract increases suggesting that inhibition efficiency increases with increase in the concentration of the extract.

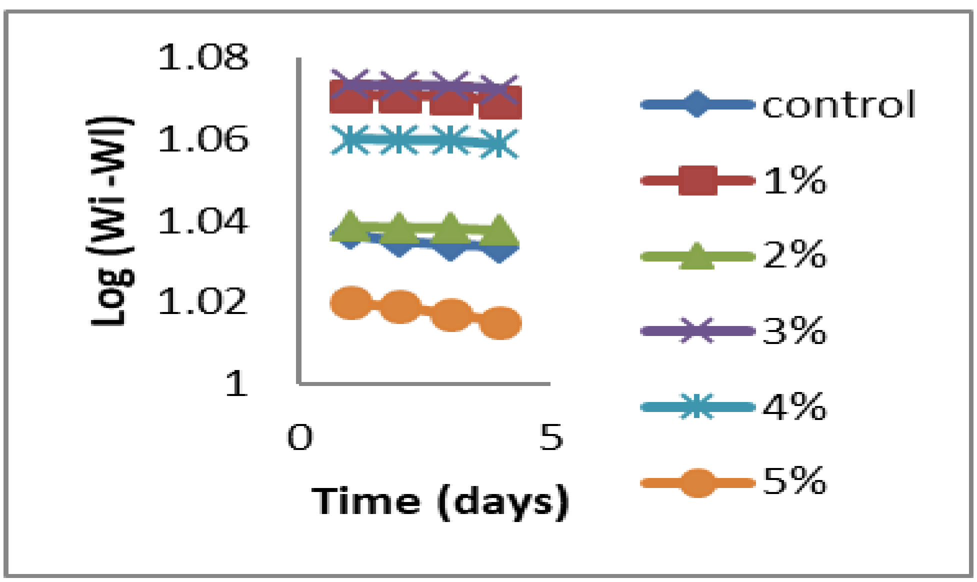

Figure 7: Variation of ln (Wi - W_L) with time (days) for mild steel coupons in 1M HCl solution at 303 K.

Table 3: Kinetic data for mild steel corrosion in 1 M HCl in the absence and presence AW

Concentration (v/v)	Rate constant (day^{-1})	Half-life (days)
Control	0.002327	298
1ml	0.000533	1300
2ml	0.000476	1455
3ml	0.000433	1598
4ml	0.000418	1657
5ml	0.000373	1856

Phytochemical Constituents

The major phytochemical constituents present in *Acalypha wilkesiana* are listed in Table 4 . As earlier reported [16-18]. The result obtained indicates that saponin, tannin, alkaloids, cardiac glycoside, terpenoid, steroid, phlobatannin were present in the acid extract. The corrosion of mild steel in HCl solution containing plant extracts can be inhibited due to the adsorption of phytochemicals present in plant extracts through their lone pair of electrons and p-electrons with the d-orbitals on the mild steel .The polar functions with S, O or N and pi-electrons of the organic compounds are usually regarded as the reaction center for the establishment of the adsorption process.

Table. 4: Phytochemical constituents of acid extract of AWAE

Phytochemicals	Acalypha extract
Tannin	+++
Saponin	+
Flavonoid	-
Alkaloid	+
Terpenoid	+
Steroid	++
Phlobatannin	++

FT-IR of AWAE

The inhibition performance of plant extract is normally attributed to the presence of heteroatoms such as oxygen, nitrogen and sulphur which make them to have high electron density. However, availability of non-bonded (lone pair) and pi -electrons in their molecules also facilitate electron transfer from the inhibitor to the metal thus make them to be useful as inhibitors for metal corrosion [30]. Fig. 8 and Fig. 9 show the IR spectra of AWAE and the corrosion product on the surface of mild steel in the presence of the inhibitors. The stretching bands at 3361cm^{-1} is due to N-H stretch, while the bands at 2066cm^{-1}, 1643cm^{-1}, and 1234 $cm^{-1}$are related to the C≡C, C═ C and C-N stretching frequency. The broad medium band centered at around 1643 cm^{-1} is related to C=N stretch and aromatic rings C=C stretch. The band AWAE shifted from 3361 cm^{-1} to 2066 cm^{-1} when adsorbed on the surface of the mild steel. This shifting in the frequencies may imply that the active phytochemical constituents present in the inhibitor molecules bind to the metal surface to form a protective metal inhibitor complex to reduce further dissolution of metal in the aggressive media. There was another shift from 1643 cm^{-1} to 1234cm^{-1} [31-33]. The FT-IR results indicated that the AWAE contains nitrogen and oxygen functional groups that can act as adsorption centers on the surface of mild steel.

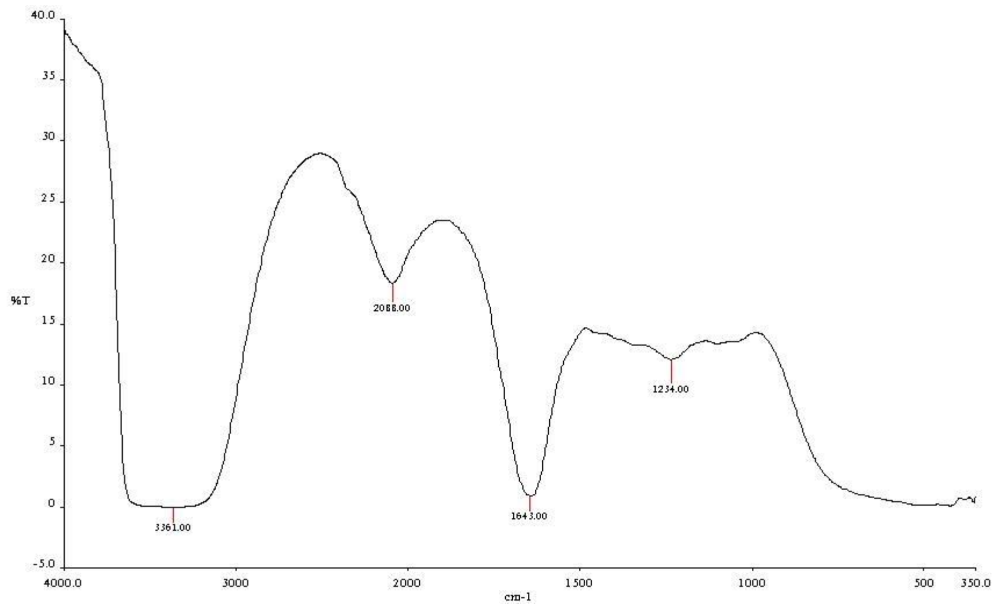

Figure 8: FTIR spectrum of AWAE

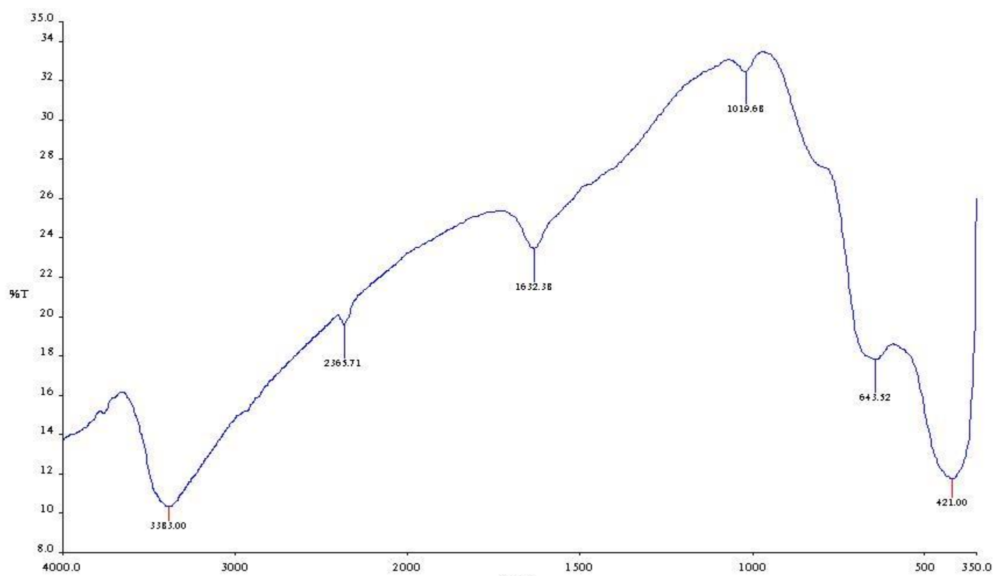

Fig 9: FT-IR spectrum of the dried solid adsorption product in 1M HCl solution containing AWAE and mild steel.

CONCLUSION

The present study provides new information on the inhibition characteristics of *Acalypha wilkesiana as* an inhibitor of mild steel corrosion in 1 M HCl solution. The inhibition efficiency increased with increase in inhibitor concentration and also increases with temperature. The inhibiting effect of the extracts could be attributed to the presence of some phytochemical constituents in the plant extracts which are adsorbed on the surface of the mild steel. The adsorption of AWAE on the surface of the mild steel in 1M HCl obeys Langmuir adsorption isotherm. The activation and thermodynamic parameters obtained support that the adsorption of AWAE on the surface of the mild steel is chemisorption. The FT-IR spectra indicate that AWAE contains O and N atoms in functional groups (O-H, N-H, CO, C=O, C-H) and aromatic rings which meet the general consideration of a typical corrosion inhibitor.

Conflict of Interest

The authors of this research hereby declare no conflict of interest in this work

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