CHRYSOPHYLLUM ALBIDUM *(AFRICAN STAR APPLE)* FRUIT EXTRACT AS ENVIRONMENTALLY FRIENDLY INHIBITOR ON THE CORROSION OF ALUMINIUM IN ACIDIC MEDIUM

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ABSTRACT

Inhibition of aluminium corrosion in 0.1M H_2SO_4 in the absence and presence of Chrysophyllum albidum fruit extract (CAFE) at temperature range of 30 – 60ºC was studied using weight loss and thermometric techniques. The fruit extract acts as an inhibitor in the acid environment. The inhibition efficiency increased with increase in inhibitor concentration but decreased with increase in temperature. The inhibiting effect of the CAFE could be attributed to the presence of some phytochemical constituents in the fruit extract which is adsorbed on the surface of the aluminium. The CAFE was found to obey Temkin adsorption isotherm at all the concentrations and temperatures studied. Thermodynamic parameters reveal that the adsorption process is spontaneous.

Keywords: Aluminium; Chrysophyllum albidum*;* Inhibition; Corrosion; Sulfuric acid.

INTRODUCTION

Aluminium is a metal with a high electronegative potential (-1.67 V). It is also highly resistant to most acidic and neutral solutions due to the formation of a protective oxide film on its surface. This film is responsible for the corrosion resistance of aluminium in most environments (Umoren, *et al.,* 2008). Nevertheless, in some cases, aluminium may be exposed to high concentrations of acids or bases. These solutions dissolve the passive film. Under these circumstances, corrosion inhibitors should be used.

The use of chemical inhibitors for aluminium alloy has been established to be the most practical and cost effective means of preventing corrosion (Umoren, *et al.,* 2008; Umoren & Obot, 2006; Umoren, *et al.,* 2008). Most of the well known acid inhibitors are organic compounds containing nitrogen, sulphur and/or oxygen atoms. Moreover, many *N*-heterocyclic compounds have been proven to be effective inhibitors for corrosion of metals and alloys in aqueous media (Umoren & Obot, 2008; Ergun, *et al.,* 2008; Ozcan, *et al.,* 2008; Saliyan & Adhikari, 2008). The influence of organic compounds containing nitrogen on aluminium corrosion in acidic solutions has been investigated by several authors (Fiala, *et al.,* 2007; Hasanov, *et al.,* 2007; Al-Juaid, 2007; Allam, 2007; Ali, *et al.,* 2008). These organic compounds can absorb on the metal surface, block the active corrosion sites on the surface and thereby reduce the corrosion rate.

In this paper, weight loss and thermometric techniques were employed to study inhibition capability of CAFE on aluminium corrosion in 0.1M H_2SO_4. The effect of temperature is also reported.

EXPERIMENTAL

Aluminium metal (purity 99.0%) of the type AA 1050/1070 obtained from System Metals Industries Limited, Calabar, Nigeria was used for the investigation. Each sheet was 1.4mm in thickness and was mechanically press cut into 5cm x 4cm coupons. However, for surface treatment they were degreased in absolute ethanol, dried in acetone and stored in a desiccator devoid of moisture before use in corrosion studies.

Chrysophyllum albidum was obtained from Obollo, Udenu Local Government Area of Enugu State, Nigeria. The fruit extract was obtained by expression technique. The impurities in the CAFE, which are principally, sand, wood and some bark fibres were removed by dissolving the extract in hot 95% ethanol following the method of Ekpe *et al.,* (1999).

The concentrations of inhibitor (CAFE) prepared and used in the study were 0.1 – 0.5 g/l. The concentration of H_2SO_4 (BDH supplies chemicals, England) used was 0.1M.

Weight Loss Measurements

In the weight loss experiments, a clean weighed aluminium coupon was immersed completely in a 250 ml beaker containing the corrodent and inhibitors with the aid of glass rod and hooks. The beakers were placed in a constant thermostated bath maintained at 30 – 60^oC. The coupons were retrieved at 24 h interval progressively for 168 h (7 days), immersed in 70% nitric acid (specific gravity: 1.42) at room temperature for 2 minutes to remove the corrosion product, scrubbed with bristle brush under running water, dried in acetone and weighed (NACE, 1984). The differences in weight of the coupons were taken as the weight loss which was used to compute the corrosion rate given by NACE, (n. d.):

$$\text{Corrosion rate (mm/yr)} = \frac{876W}{\rho At} \quad \text{..............(1)}$$

where W is the weight loss (g), ρ is the density of specimen (g cm^{-3}), A the area of specimen (cm^2) and t the exposure time (h).

The inhibition efficiency of CAFE acting as inhibitor in 0.1M H_2SO_4 was calculated using the following expression:

$$I(\%) = 1 - \frac{W_i}{W_o} \times 100 \quad \text{...................... (2)}$$

where W_o and W_i are the weight losses of the aluminium coupons in the absence and presence of inhibitor respectively in H_2SO_4 at the same temperature.

The degree of surface coverage *(θ)*, which is defined as the fraction of the aluminium surface that was covered by the inhibitor was calculated from Equation (3):

$$\theta = 1 - \frac{W_i}{W_o} \quad \text{........................ (3)}$$

Thermometric Measurements

The reaction vessel and procedure for determining the corrosion behaviour by this method has been described elsewhere by other authors ((Oza and Sinha, 1982; Upadhyay and Mathur, 2007; El-Etre, 2001). In the thermometric technique, the corrodent (H_2SO_4) concentration was kept at 0.1M. The volume of test solution used was 50 ml. The initial temperature in all experiments was kept at 30^oC. The progress of corrosion reaction was monitored by determining the changes in temperature with time using a calibrated thermometer (0 – 100^oC)

to the nearest ±0.05°C. This method enabled the computation of the reaction number (*RN*) defined as:

$$RN \ (^oC \ min^{-1}) = \frac{T_m - T_i}{t} \qquad \text{……..…………. (4)}$$

where T_m and T_i are the maximum and initial temperatures respectively and t is the time (min) taken to reach the maximum temperature.

The Inhibition efficiency (%I) was evaluated from percentage reduction in the reaction number using Equation (5).

$$\%I = \frac{RN_{aq} - RN_{wi}}{RN_{aq}} \times 100 \qquad \text{…….………….. (5)}$$

where RN_{aq} is the reaction number in the absence of inhibitor (blank solution) and RN_{wi} is the reaction number of 0.1M H_2SO_4 containing studied inhibitor.

RESULTS AND DISCUSSION

Weight Loss, Corrosion Rate and Inhibition Efficiency

The effect of addition of CAFE at different concentrations on aluminium corrosion in 0.1M H_2SO_4 was investigated using weight loss technique at a temperature range of 30 – 60°C after 168 h of immersion period. Figure 1 shows the plot of weight losses versus time for aluminium in 0.1M H_2SO_4 in the absence and presence of different concentrations of CAFE at 30°C. Similar plots were obtained for other temperatures (40 – 60°C).

The figure clearly shows a reduction in weight loss of the metal coupons in the presence of CAFE compared to its absence. The figure further revealed that loss in weight of the coupons decreased as the concentration of the inhibitor (CAFE) increased. Similar trend was observed for other temperatures (40 – 60°C) but with greater values of weight loss.
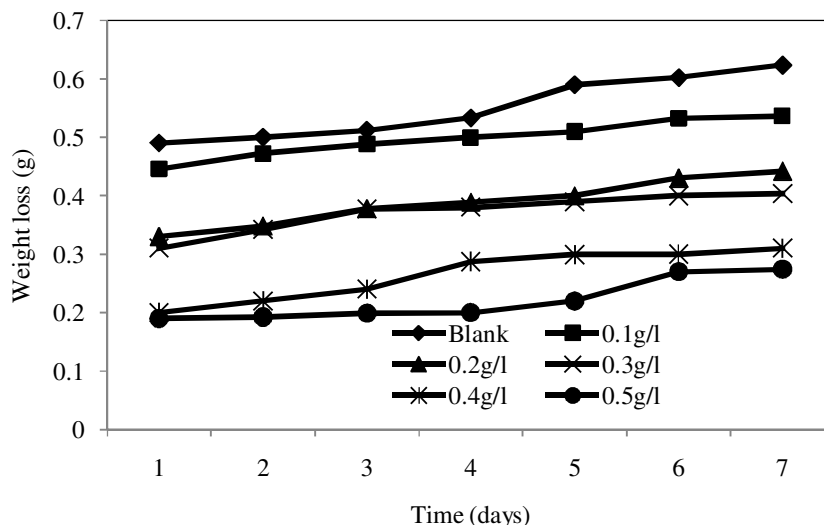

Figure 1. Plot of weight loss against time for aluminium corrosion in 0.1M H_2SO_4 devoid of and containing different concentrations of the CAFE at 30°C

Values of corrosion rate of aluminium coupons in 0.1M H_2SO_4 in the absence and presence of different concentrations of CAFE were determined and are presented in Table 1. From the table, it was found that the rate of corrosion decreased with increase in the concentration of

the CAFE. This indicates that the CAFE in the solution inhibits aluminium corrosion in H_2SO_4 and that the extent of corrosion inhibition depends on the amount of the extract present.

Also shown in Table 1 are the calculated values of inhibition efficiency for the CAFE. It is observed that the inhibition efficiency increased with increasing concentration of the CAFE. The maximum inhibition efficiency was observed to be 56.00% at 0.5g/l. The corrosion inhibition can be attributed to adsorption of molecules of phytochemicals present in the plant on the surface of the metal. The phytochemical composition of the CAFE was studied by Edem *et al.,* (1984).

Owing to the complex chemical composition of the CAFE, it is quite difficult to assign the inhibitive effect to a particular constituent. However, mutual effects of these compounds and other components present in the CAFE cannot be ruled out. The adsorption of these compounds on the aluminium surface reduces the surface area available for corrosion.

Table 1. Calculated values of corrosion rate (mm/yr), inhibition efficiency (%I) and degree of surface coverage (θ) for CAFE from weight loss data in 0.1M H_2SO_4

Conc.(s) of CAFE (g/l)	Corrosion Rate (mm/yr) $\times 10^{-2}$				Inhibition Efficiency (%I)				Degree of surface coverage(θ)			
	30^oC	40^oC	50^oC	60^oC	30^oC	40^oC	50^oC	60^oC	30^oC	40^oC	50^oC	60^oC
Blank	6.02	8.12	9.66	11.59								
0.1	5.18	7.17	8.64	10.62	13.90	11.70	10.50	8.37	0.14	0.12	0.11	0.08
0.2	4.27	6.30	7.82	9.70	29.10	22.41	19.10	16.30	0.29	0.22	0.19	0.16
0.3	3.90	5.52	6.77	8.30	35.21	32.01	29.90	28.40	0.35	0.32	0.30	0.28
0.4	3.00	4.86	5.96	7.40	50.21	40.11	38.35	36.20	0.50	0.40	0.38	0.36
0.5	2.65	4.18	5.12	7.02	56.00	48.50	47.00	39.45	0.56	0.49	0.47	0.40

Thermometric Studies

Thermometric methods have proved to be of considerable value and help in studying corrosion behaviour of a number of metals and alloys in various corroding environments (Umoren *et al*., 2006; Moussa *et al*., 1988). The technique is also useful in evaluating the inhibition efficiency of a number of organic substances (Ebenso, 1998; Umoren *et al*., 2006). Results obtained using thermometric methods have been confirmed by several research groups and by other well-established methods such as weight loss (Moussa *et al*., 1988; Umoren *et al*., 2006; James *et al*., 2007), potentiostatic (Maayta and Al-Rawashdeh, 2004), and polarization measurement (Abd El-Rehim *et al*., 1999).

Figure 2 shows the plot of temperature versus time for corrosion reaction of aluminium in 0.1M H_2SO_4 solution in the absence and presence of different concentrations of CAFE. The figure revealed that the dissolution of aluminium starts after a certain time from the immersion of the aluminium coupons in the test solution as evident in the constant temperature with time. It may be expected that this time corresponds to the period required by the acid to destroy the pre-immersion oxide film and is known as the 'incubation period'. After the consumption of the pre-immersion oxide film, the temperature of the system rises gradually due to the exothermic corrosion reaction to reach a maximum value, T_m.
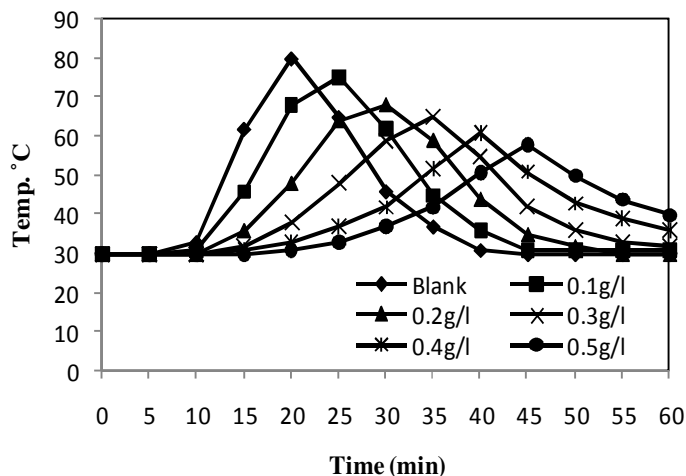
Figure 2. Temperature – time curves for aluminium corrosion in 0.1M H_2SO_4 in the presence of CAFE of different concentrations

From the figure, the maximum temperature T_m was attained at a very short time (t) by the free acid solution. This corresponds to a reaction number (RN) of $2.50^{o}C$ min^{-1} (Table 2). Further inspection of the figure revealed that on addition of the CAFE, the maximum temperature attained decreased and the time required to reaching it increased correspondingly. This is an indication that the various additives inhibit the corrosion of Al in the acidic environment, probably by adsorption on the metal surface (Ekpe *et al.*, 1999). The extent of inhibition depends on the degree of coverage of the metal by the adsorbed molecules. Strong adsorption is noted at higher concentration of CAFE as depicted by further decrease in maximum temperature (T_m) attained and a corresponding increase in time (t) taken to reach it, and both factors cause a large decrease in the RN of the system (Table 2). Temperature of all the systems decreased after reaching their maximum values. This could be attributed to decreasing corrodent concentration with increasing reaction time, which in turn decreased the rate of corrosion, hence, a decrease in quantity of heat evolved. The results obtained in this study corroborate those of other studies (El-Etre, 2001; 2003).

Table 2. Calculated values of Reaction Number and Percentage Reduction in RN for Al dissolution in 0.1M H_2SO_4 containing CAFE from thermometric method

Conc.(s) of CAFE (g/l)	Reaction Number (RN) (^{o}C min^{-1})	% Reduction in RN (Inhibition efficiency)
Blank	2.50	0
0.1	1.80	28.00
0.2	1.26	49.60
0.3	1.00	60.00
0.4	0.78	68.80
0.5	0.62	75.20

Table 2 shows the calculated values of reaction number (RN) and the percentage reduction in reaction number (inhibition efficiency) for the various systems investigated. It is very clear from the table that reaction number decreased in the presence of CAFE compared to its absence (blank). Also, the percentage reduction in reaction number increased with increasing concentration of the CAFE. This assertion is also corroborated by weight loss measurements. Comparing the inhibition efficiency calculated from weight loss and thermometric methods for the CAFE (Tables 1 and 2), it can be seen that the values obtained from weight loss technique are lower while those obtained by the thermometric method are higher. This may be attributed to the difference in time required to form an adsorbed layer of the inhibitor on the metal surface that can inhibit corrosion. Also, values of corrosion rate from weight loss method represent average values while those of thermometric method represent the instantaneous values.

Adsorption/Thermodynamic Studies

Values of the degree of surface coverage θ were evaluated at different concentrations of the CAFE in 0.1M H_2SO_4 solution from weight loss measurements (Table 1). The values were fitted into Temkin adsorption isotherm model, which has the form (Sahin *et al.*, 2002).

$$\exp(-2a\theta) = KC \qquad \text{………………... (6)}$$

where a is molecules interaction parameter, θ is the degree of surface coverage, K is equilibrium constant of adsorption process and C is the concentration of CAFE. K is related to the free energy of adsorption by the equation:

$$K = (1/55.5) [\exp(-\Delta G^o_{ads}/RT)] \qquad \text{...…………… (7)}$$

The plot of surface coverage (θ) as a function of logarithm of concentration of CAFE is shown in Figure 3; from the plot, straight lines were obtained for CAFE suggesting that its adsorption on the aluminium surface follows Temkin adsorption isotherm. This result confirms the assumption that the corrosion inhibition of the CAFE is attributed to the adsorption of molecules of phytochemicals present in the fruit extract on the surface of the metal.
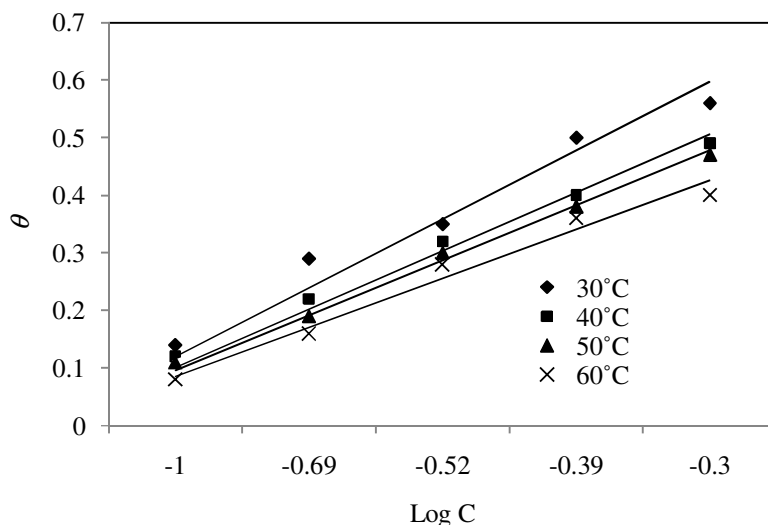

Figure 3. The plot of surface coverage (θ) as a function of logarithm of concentrations of CAFE at 30 – 60°C in 0.1M H_2SO_4

Calculated values of molecular interaction parameter '*a*' and equilibrium constant of adsorption proces *K*, obtained from Temkin's plot are shown in Table 3. The values of *a* are negative in all cases showing that repulsion exist in adsorption layer (Umoren *et al.,* 2007). Values of *K* decreased with increase in temperature suggesting that the inhibitor is physically adsorbed on the surface.

Generally, *K* denotes the strength between adsorbate and adsorbent. Large values of *K* imply more efficient adsorption and hence better inhibition efficiency (Refay *et al.,* 2004). The free energy of adsorption values ΔG^o_{ads} were obtained from Equation (7). The values obtained are presented in Table 3. Results presented in the table indicate that the values of ΔG^o_{ads} are negative in all cases indicating spontaneous adsorption of the inhibitor molecules. Values of ΔG^o_{ads} also indicate that the inhibitor function by physically adsorbing on the surface of the metal.

Table 3. Some thermodynamic parameters and adsorption coefficients for aluminium corrosion of CAFE in 0.1M H$_2$SO$_4$

Inhibitor	Temp. ^oC	Temkin		
		ΔG^o_{ads} (KJ mol^{-1})	a	k
CAFE	30	− 4.45	− 0.758	1.05
	40	− 4.60	− 0.909	1.05
	50	− 4.74	− 0.943	1.05
	60	− 4.88	− 1.087	1.04

Generally, values of ΔG^o_{ads} up to − 20 kJ mol^{-1} are consistent with electrostatic interaction between charged molecules and a charged metal (which indicates physical adsorption) while those more negative than − 40 kJ mol^{-1} involves charge sharing or transfer from the inhibitor molecules to the metal surface to form a co-ordinate type of bond (which indicates chemisorption) (Dehri & Ozcan, 2006; Popova *et al.,* 2003). Physical adsorption is a result of electrostatic attraction between charged metal surface and charged species in the bulk of the solution. Adsorption of negatively charged species is facilitated if the metal is positively charged. Positively charged species can also protect the positively charged metal surface acting with a negatively charged intermediate such as acid anions adsorbed on the metal surface (Popova *et al.,* 2003).

Values of enthalpy of activation ΔH^o and entropy of activation ΔS^o were obtained from Figure 4, the transition state Equation (8):

$$CR = (RT/Nh) \exp (\Delta S^o/R) \exp (-\Delta H^o /RT) \dots\dots\dots\dots\dots\dots \quad (8)$$

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*) as a function of 1/*T* was made and straight lines were obtained. ΔH^o and ΔS^o were computed from the slope and intercept respectively from the linear plot. The average values for ΔH^o and ΔS^o obtained in the presence of the CAFE were 7.871 kJmol^{-1} and −0.0291 kJmol^{-1}K^{-1} respectively.

The positive sign of the ΔH^o reflects the endothermic nature of the aluminium dissolution process in the presence of the fruit extract, while the negative sign of ΔS^o shows increase in the system order (El-Etre, 2003).

Effect of Temperature

The effect of temperature on corrosion rate of aluminium in free acid and in the presence of different concentrations of CAFE was studied in the temperature range of $30 - 60^oC$ using weight loss measurements. In examining the effect of temperature on the corrosion process in the presence of the fruit extract, the Arrhenius equation below was used:

$$\text{Log } CR = \frac{-E_a}{2.303 \, RT} + \log A \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (9)$$

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.
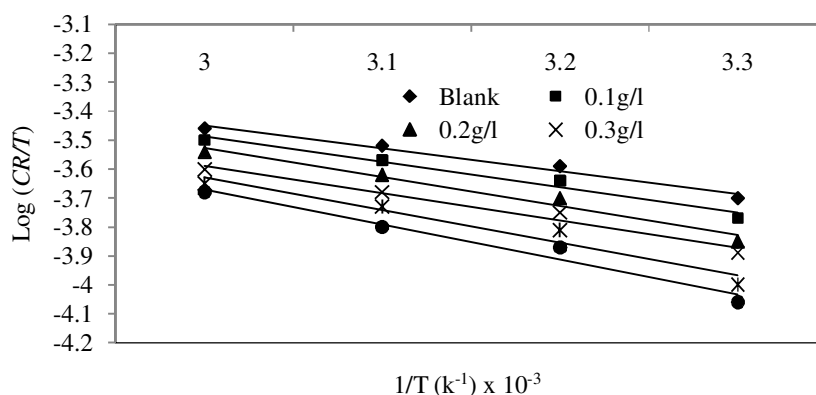

Figure 4. Transition state plot for CAFE in 0.1M H_2SO_4

Figure 5 represents Arrhenius plot (as Log CR versus $1/T$) for aluminium corrosion in 0.1M H_2SO_4 in the absence and presence of various concentrations of CAFE. Straight lines were obtained with slope equal to $- E_a / 2.303R$. The values of E_a for the corrosion reaction in the absence and presence of CAFE were calculated and are presented in Table 4. Inspection of the table shows that the presence of the fruit extract increased the value of E_a when compared to its absence (blank) indicating the adsorption of the CAFE on the surface of the metal.

The higher value of the activation energy of the process in an inhibitor's presence when compared to that in its absence is attributed to its physisorption, while the opposite is the case with chemisorptions (Elayyachy *et al.,* 2005; Morad *et al.,* 2006).

Table 4. Values of Activation Energy for aluminium corrosion in 0.1M H_2SO_4 in the absence and presence of CAFE

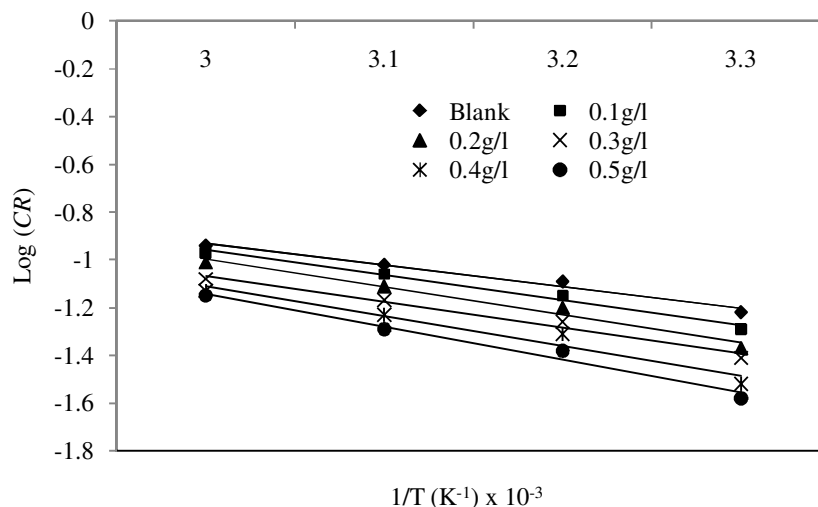| Conc.(s) of CAFE (g/l)	Activation Energy E_a (KJ mol^{-1})
Blank	16.59
0.1	19.47
0.2	21.06
0.3	18.83
0.4	22.02
0.5	24.89

Figure 5. Arrhenius plot (as Log CR versus $1/T$) for aluminium corrosion in 0.1M H_2SO_4 in the absence and presence of various concentrations of CAFE

CONCLUSIONS

The experimental results obtained in the present study can be summarized as follows:

1. CAFE acts as an inhibitor for aluminium corrosion in 0.1M H_2SO_4 solution.

2. The inhibition efficiency increased with increase in the concentration of CAFE but decreased with increase in temperature.

3. The inhibition is due to the presence of some phytochemical constituents in the fruit extract which is adsorbed on the surface of the aluminium.

4. The fruit extract was found to obey Temkin adsorption isotherm from the fit of experimental data and values of ΔG^o_{ads} are negative suggesting that the CAFE strongly adsorbed on the aluminium surface and the values also support the physical adsorption mechanism.

5. Thermodynamic parameters revealed that adsorption process is spontaneous.

6. The present study provides new information on the inhibition characteristics of CAFE under the specified conditions.

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