



Corrosion and Kinetic Study of *Eucalyptus camaldulensis* Seeds Extract Percolated with Methanol on Aluminium Coupons in HCl

Ishaq Yahaya Lawan¹, Fatima Khalil Abdullah², Sani Idris³, Shinggu D. Yamta⁴ and Abdurrahman Hudu⁵

¹Department of Chemistry, Nigerian Army University, Biu, Nigeria

²Department of Chemistry, Kano University of Science and Technology, Wudil, Nigeria

³Department of Chemistry, Kano University of Science and Technology, Wudil, Nigeria

⁴Department of Chemistry, Nigerian Army University, Biu, Nigeria

⁵Department of Chemistry, Nigerian Army University, Kano, Nigeria

Abstract

This research discusses a detail optimization of *Eucalyptus camaldulensis* seeds extract as corrosion inhibitor for aluminum coupons in HCl using weight loss measurement and kinetic study. The result shows that the maximum inhibitor efficiency was obtained at a concentration of 2.0 (%W/V). However the highest inhibitor efficiency of 85% was obtained at 50°C and the least inhibitor efficiency of 29% was obtained at 30°C. Thermodynamic consideration revealed that adsorption of inhibitor of aluminum surface was exothermic and consistent with chemical adsorption mechanism.

Introduction

The significant of corrosion protection of carbon steel in acidic solutions has become necessary due to its propensity to be attacked in acidic media when deployed in services like acid electrochemical systems, oil recovery and also the petrochemical industry. The protection of carbon steel against corrosion in acidic environment can be achieved in various ways using corrosion inhibitors [1-3]. Several research have been carried out on

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the use of eco-friendly inhibitors to contain corrosion of carbon steel, aluminum and many other metals in both acid and alkaline solutions [4-7]. Corrosion inhibitors are commonly added in small quantity to pickling acids, acid stimulation fluids, cooling water, oil and gas production streams, either continuously or intermittently to control attacks. This inhibitor generally controls corrosion by forming various types of films either by adsorption or the formation of bulky precipitates, and/or the formation of a passive layer on the metal surface that modifies its corrosiveness [9-10]. The mechanism of most corrosion inhibitors on metal oxidation can be explained. Inhibitor on metal oxidation can be explained on the basis of molecular adsorption, which involves forming a thin, invisible film that coats the metal thereby protecting it from attack or a combination of adsorption films and corrosion products form a passive layer on the metal surface [11]. In acidic solutions, corrosion inhibitors subsist either as neutral molecules which adsorb on the metal/acid solution interface through electrostatic attraction between charged molecules and unshared π electrons in the inhibitor molecules and charged metal surface [12]. Apart from sea water, hydrochloric acid remains the main source of the aggressive chloride ions in aqueous solutions and occurrences of carbon steel corrosion in environments containing chloride ions are known [13]. Several reports claimed the use of green inhibitors as cheap, effective, renewable and non-toxic for metal corrosion [14-20]. The effectiveness of these bio-extracts as metal corrosion inhibitors mainly depends on its phytochemical functional groups having; steric effect, π -orbital character of donating electrons via adsorption at active sites and electronic density of donor atoms among other variables.

Inhibitors are substances which reduce metallic corrosion by changing the structure of the double layer, reducing the effective electrode area, decreasing metal reactivity (Abdallah et al. [18]).

Inhibitors are also substances which when added to the environment, usually in small quantity effectively reduce corrosion (Eddy [19]). Inhibitors are classified depending on the number of orientations.

Based on their chemical nature as organic and inorganic and based on the nature of their polarizing action into anodic and cathodic inhibitors.

Aluminum steel

Aluminum is a chemical element in the boron group with symbol Al and atomic number 13. It is a silvery-white, soft, non-magnetic, ductile metal. Aluminum is the third

most abundant element (after oxygen and silicon), and the most abundant metal in the earth's crust, it makes up about 8% by mass of the earth crust, though it is less common in the mantle below. Aluminum metal is so chemically reactive that native specimen and rare and limited to extreme reducing environments. Instead, it is found combined in over 270 different minerals (Saraswathi et al. [22]). The cheap ore is bauxite.

Aluminum is remarkable for the metal's low density and for its ability to resist corrosion due to the phenomenon of passivation (Frank [14])

Corrosion resistance can be excellent due to a thin surface layer of aluminum oxide that forms when the metal is exposed to air, effectively preventing further oxidation (Vargel [13]). The strongest aluminum alloys are less corrosion resistance due to galvanic reaction with alloyed copper. (Polmear [10]) this corrosion resistance is also often greatly reduced by aqueous salts, particularly in the presence of dissimilar metals. In high acidic solutions, aluminum reacts with water to form hydrogen, and highly alkaline one to form aluminates-protective passivation under these condition is negligible-also, chlorides such as common sodium chloride are well known sources of corrosion of aluminum (Beal [15]).

Eucalyptus camaldulensis

Eucalyptus camaldulensis, the river red gum is a tree of the genus eucalyptus. It is one of around 800 in the genus. It is a plantation species in many parts of the world, but is native to Australia, where it has the most widespread natural distribution of *Eucalyptus* in Australia (Colloff [9]); especially beside inland water courses. Oddly, it is named for a private estate garden near camaldoli monastery near Naples, from where first specimen came to be described. Material from this tree was used by Frederick Delinhardf chief gardener at the botanic gardens in Naples to describe this species in 1832 (Slee et al. [8]).

Eucalyptus camaldulensis seed

The seeds are yellow or yellowish brown, cuboids and smooth (Walsh and Entwisle [23]) and can be difficult to distinguish from chaff (Bonney [11]). They have long ripening period, usually maturing during the summer months, about one year after flowering (Ralph [21]). Valves on maturing fruit will turn from green to brownish-red when ripe (Bonney [11]). Temperature and seasonal condition will detect when seed is ready to pick and seed release can occur quickly once matured (Bonney [11]).

In this present work, corrosion study was made using Aluminium coupons immersed in aerated acid solutions with *Eucalyptus camaldulensis* seed.

Materials/reagents

Volumetric flasks (250ml), Measuring cylinder (100ml), Glass Beakers, Thermometers (0-360°C), Water baths, Reagent bottles, Filter paper, Sieve, Funnel, Electric oven, Stop watch, Aluminium coupons, Hydrochloric acid (HCl), Absolute Methanol (CH₃OH), Potassium hydroxide (KOH), Acetone, Hook.

Preparation of inhibitor

The *Eucalyptus camaldulensis* seeds were spread out in a laboratory for two weeks for dried and were transferred to an electric oven at 100°C for an hour in other to be properly dried, and then ground to powder. The powdered seeds were added to Methanol and soaked in a beaker at room temperature for 48 hours and the dark brown sample was filtered and the resulting filtrate was evaporated by using rotator evaporation for some hours in other to leave the sample free from methanol, the extracts obtained was used to prepared various concentration of inhibitor.

Preparation of standard solution of hydrochloric acid

A standard solution is a solution of known concentration. The amount of a solution is measure in various ways. The amount of substance dissolve per unit volume of the solution has a unit of mol dm⁻³ and is called molarity. Also, the mass concentration is the mass of solute per unit volume of solution. It has a unit of g dm⁻³ or g cm⁻³.

Several concentration of hydrochloric acid could be effectively prepared, by dissolving the suitable weight in 500 ml of distilled water in 500 cm³ volumetric flasks.

From commercial acid whose bottle was labeled as follows:

Percentage purity = 36.5%

Specific gravity = 1.2 g cm⁻¹

Molar mass = 36.46 g cm⁻¹.

Using the relation to calculate the concentration in mol dm^{-3}

$$\begin{aligned}\text{Concentration in mol dm}^{-3} &= \frac{\text{percentage purity} \times \text{specific gravity} \times 1000}{\text{molar mass}} \\ &= \frac{36.5 \times 1.2 \times 1000}{36.46} \\ &= 12.01 \text{ mol dm}^{-3}.\end{aligned}$$

To convert $12.01 \text{ mol dm}^{-3}$ of the commercial acid to 1 mol dm^{-3} by dilution, using the relation

$$C_1 V_1 = C_2 V_2,$$

where

C_1 = concentration of acid in $12.01 \text{ mol dm}^{-3}$

C_2 = concentration of dilute acid in mol dm^{-3}

V_1 = initial volume of the acid in cm^3

V_2 = first volume of the dilute acid in 250 cm^3 .

Substituting

$$12.01 \text{ mol dm}^{-3} \times V_1 = 1 \text{ mol dm}^{-3} \times 250 \text{ cm}^3$$

$$V_1 = 250 \text{ cm}^3 \times 1 \text{ mol dm}^{-3} / 12.01 \text{ mol dm}^{-3}$$

$$V_2 = 20.81 \text{ or } 21 \text{ cm}^3 \text{ (approximation).}$$

21 cm^3 of a commercial acid was taken into a beaker which; contain about 100 cm^3 of distilled water and the solution was started. The solution was transferred into 250 cm^3 volumetric flask, the beaker was rinse with more distilled water and it was pour into the solution in volumetric flask until it reaches the meniscus level of the flask. That was how to prepare to 1 mol dm^{-3} HCl. The same procedure was repeated in the preparation of 2, 3, and 4 mol dm^{-3} solutions.

Preparation of coupons

The aluminum coupons were cut into 3/5cm coupons. The coupons were cleaned and decreased in absolute methanol, dried in an electric oven and were stored in a desiccators prior to use.

Preparation of hydrochloric acid/inhibitor

Several concentrations of hydrochloric acid and inhibitor were prepared by dissolving adequate amount of inhibitor in 50 ml of 3 mol dm⁻³ hydrochloric acid.

For example,

To prepared 0.5 (%W/V) of the inhibitor in 3 mol dm⁻³ HCl

$$\begin{aligned} (\%W/V) &= [\text{weight of solute} / \text{volume of solution}] \times 100 \\ &= (0.25/50) \times 100 \\ &= 0.5 (\%W/V). \end{aligned}$$

The same procedure was used for 1.0 (%W/V), 1.5 (%W/V), and 2.0 (%W/V) respectively.

Experimental procedures

The first was carried out by introducing 1, 2, 3, and 4 mol dm⁻³ into four (4) sets of beaker respectively. The coupons were immersed in each of the test solution containing HCl. Each test was carried out at different temperature ranging from 30-50°C. The coupons were withdrawn from the acid after five hours and immersed in KOH solution to quench the reaction, scrub with bristle brush in distilled water and dried in acetone and then reweighed.

The second test, involved the introduction of 0.25 g, 0.5 g, 0.75 g and 1.0 g of extracts into 50 ml of 3 M in 400 cm³ beaker at 30, 40 and 50°C, respectively. The previously weighed coupons were introduce into each five set of beaker each coupons was withdrawn from their respective solution after five hours, washed as before and weighed.

At the end of the experiment, the weight loss, corrosion rate, inhibitor efficiency, degree of surface coverage, free energy, activation energy, half-life and heat of adsorption were calculated.

The weight loss was calculated by finding the difference between the initial weight of the coupons and the final weight after 5 hours. Using the relationship (Okafor et al. [26])

$$W = W_i - W_f, \quad (1)$$

where

W = weight loss

W_i = initial weight

W_f = final weight.

The corrosion rate was determined from standard expression for measurement of corrosion rate in millimeters per year (MMPY).

$$\text{MPY} = \frac{87.6W}{\text{DAT}}, \quad (2)$$

where

W = weight loss (mg)

D = density of material used (g cm^{-3})

T = time of exposure (hour)

A = total surface Area (cm^2).

Inhibitor efficiency (IE) % was computed using the relationship (Abiola and James [27]).

$$\text{Inhibitor efficiency (IE) \%} = 1 - \frac{W_1}{W_2} \times 100, \quad (3)$$

where

W_1 = corrosion rate with inhibitor

W_2 = corrosion rate without inhibitor.

Degree of total surface coverage (θ) was calculated using the relationship (Abiola and James [27]).

$$\text{Degree of total coverage} = 1 - \frac{W_1}{W_2}. \quad (4)$$

Kinetic study

The activation energy (EA) of the corrosion rate was calculated using the Arrhenius equation given by (Abiola and James [27])

$$\text{Log} \frac{R_1}{R_2} = \frac{Ea}{2.303R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right), \quad (5)$$

where R_1 and R_2 are the corrosion rates at any given two different temperatures T_1 and T_2 .

The free energy of adsorption (ΔG) was determined, using the given relationships (Abiola and James [27]).

$$\Delta G (\text{ads}) = -RT \ln (55.5k), \quad (6)$$

where

$$K = \frac{\theta}{c(1 - \theta)}$$

K = equilibrium constant,

θ = degree of surface coverage

C = concentration of the inhibitor.

Also the heat of adsorption (HADS) of the inhibitor was calculated using the relationship (Chetouani et al. [1])

$$\Delta H_{\text{ads}} = 2.303R \left[\left(\text{Log} \frac{\theta_2}{1 - \theta_2} \right) - \left(\log \frac{\theta_1}{1 - \theta_1} \right) \right] \times T_1 T_2 / T_2 - T_1 \quad (\text{Jmol}^{-1}), \quad (7)$$

where θ_1 and θ_2 are degree of surface coverage at any two temperature T_1 and T_2 .

Result and Discussion

Table 1. Corrosion rate with concentration of HCl without inhibitor at 30°C, 40°C and 50°C.

Concentration of HCl (mol dm ⁻³)	Corrosion rate (mils/year)		
	30°C	40°C	50°C
1	1081.48	1158.73	1313.23
2	1313.23	1467.72	1544.97
3	1467.72	1619.37	2085.71
4	1699.47	1931.22	2471.96

Table 2. Corrosion rate with concentration of inhibitor at 30, 40, 50°C.

Inhibitor concentration (% W/V)	Corrosion rate (mils/year)		
	30°C	40°C	50°C
0.5	1042.86	919.26	463.49
1.0	934.71	896.08	324.44
1.5	919.26	845.01	293.54
2	903.81	795.66	262.65

Table 3. Inhibitor efficiency with inhibitor concentration at 30, 40, 50°C

Inhibitor concentration (% W/V)	Inhibitor efficiency (%)		
	30°C	40°C	50°C
0.5	29	21	65
1.0	36	42	79
1.5	37	50	91
2.0	38	67	94

Table 4. Free energy (J mol^{-1}) with inhibitor concentration at 30, 40 and 50°C.

Inhibitor concentration (% W/V)	Free energy (J mol^{-1})		
	30°C	40°C	50°C
0.5	-9608.61	-11522.35	-16046.31
1.0	-8668.62	-9929.70	-15239.09
1.5	-7755.87	-9188.55	-14572.00
2.0	-7138.63	-8752.31	-14029.50

Table 5. Degree of surface coverage (θ) with inhibitor concentration.

Inhibitor concentration (% W/V)	Degree of surface coverage (θ)		
	30°C	40°C	50°C
0.5	0.29	0.43	0.78
1	0.36	0.45	0.84
1.5	0.37	0.48	0.86
2	0.38	0.51	0.87

Table 6. Heat of adsorption (Hads) for *Eucalyptus camaldulensis* seeds.

Concentration of <i>Eucalyptus camaldulensis</i> seeds (% W/V)	Heat of adsorption (Hads) (J mol^{-1})
	At 30 and 40°C
0.5	-8898.11
1.0	-4685.13
1.5	-8715.70
2.0	-11512.98

Table 7. Activation energy ($-E_a$) with concentration of inhibitor

Concentration of <i>Eucalyptus camaldulensis</i> seeds	Activation energy (E_a) (J mol^{-1}) at 40°C and 50°C
0.5	57,577.94
1.0	85,421.23
1.5	88,603.64
2.0	93,192.12

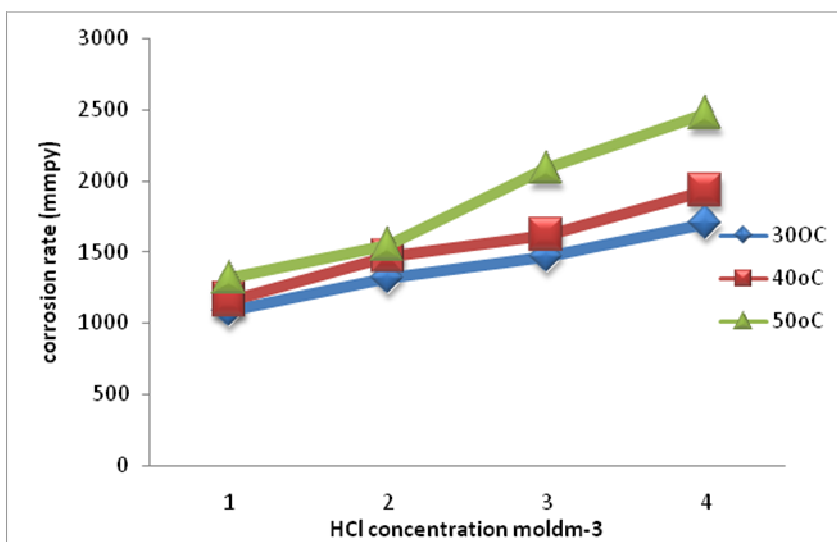


Figure 1. Variation of corrosion rate with concentration of HCl 30°C, 40°C and 50°C.

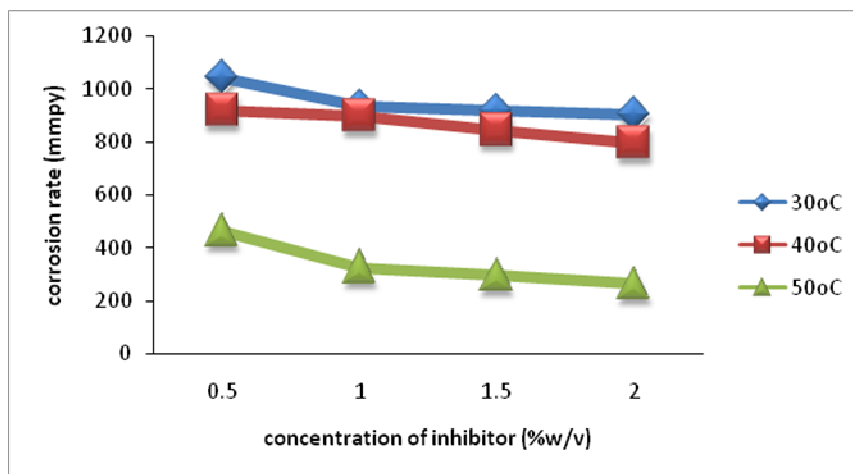


Figure 2. Variation of corrosion with of inhibitor at 30°C, 40°C and 50°C.

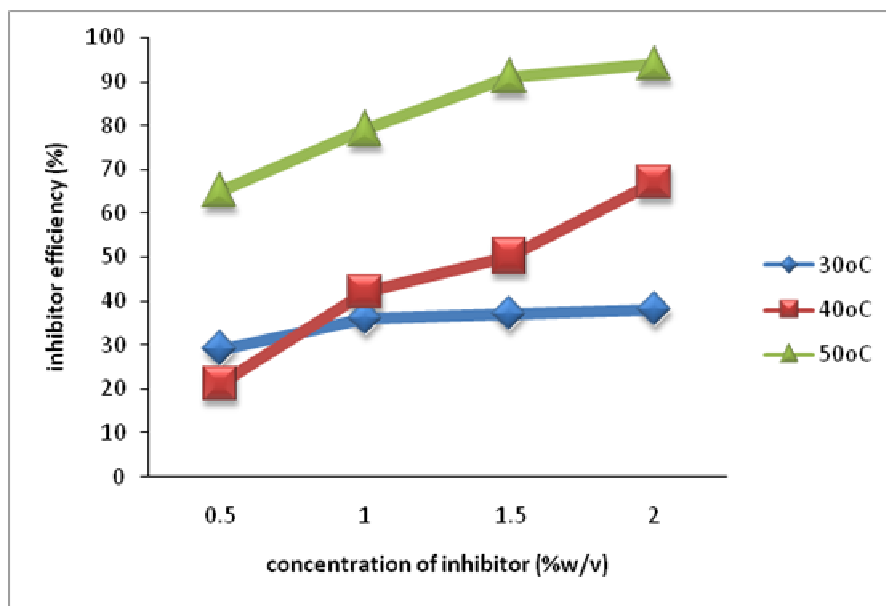


Figure 3. Variation of inhibitor efficiency with inhibition concentration at 30°C, 40°C and 50°C.

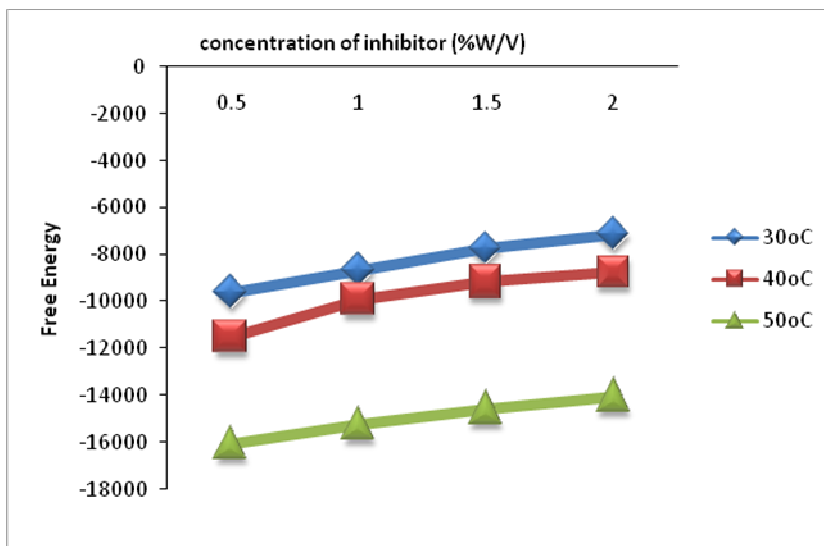


Figure 4. Variation of free energy with concentration of inhibitor at 30°C, 40°C and 50°C.

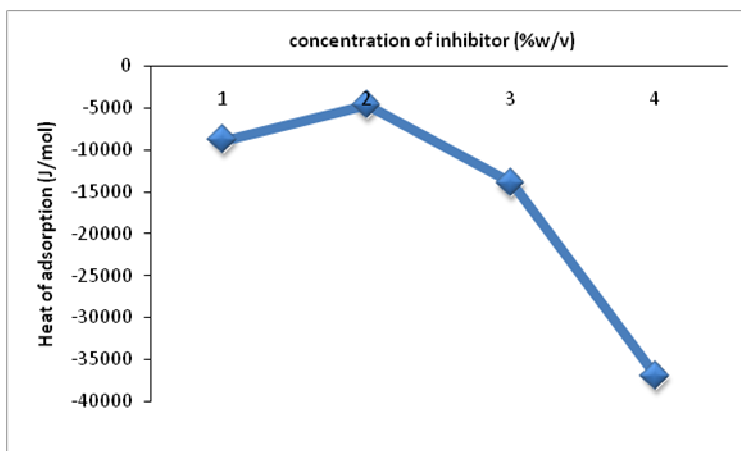


Figure 5. Variation of heat adsorption (H_{ad}) ($J\ mol^{-1}$) with of inhibitor at 30 and 40°C.

Discussion

The successful of *Eucalyptus camaldulensis* seeds extract as an inhibitor of the corrosion of aluminum coupons in acidic medium was carefully examined using weight loss measurement. Also, adequate observation of coupons in the absence and presence of inhibitor after 5 hours of exposure shows changes in colour of coupons.

Figure 1 shows values of corrosion rate with concentration of HCl in the absent of inhibitor at 30°C, 40°C and 50°C, the trend shows an increase in corrosion rate with variation in concentration from 1.0 to 4.0 ($\text{mol}\ \text{dm}^{-3}$) at 30°C, 40°C and 50°C. The highest rate of corrosion was observed at 50°C in concentration of 4 $\text{mol}\ \text{dm}^{-3}$, while the least corrosion rate was observed in 1.0 $\text{mol}\ \text{dm}^{-3}$ concentration. This means the higher the concentration of HCl the higher the corrosion rate also the lower the HCl concentration the lower the corrosion rate.

Figure 2 shows that the corrosion rate for the corrosion of aluminum coupons at 0.5 to 2.0 in 3 $\text{mol}\ \text{dm}^{-3}$ HCl in the presence of aluminum coupons at temperature 30°C, 40°C and 50°C. It may be observed from the data in Figure 2 that an increased in concentration of the inhibitor generally retard the corrosion rate of aluminum coupons in the acid solution. This is also seen and supported from the decreasing change in mass loss taking place at a particular acid concentration corresponding with an increase in inhibitor concentration.

Figure 3 shows that the inhibitor efficiency of *Eucalyptus camaldulensis* extract varies with its concentration. Optimum value of the inhibitor efficiency was obtained at a concentration of 2.0 (%w/v). The highest inhibitor efficiency of 85% was obtained at 50°C. While the least value were obtain at an extract concentration of 0.5 (%w/v), with inhibitor efficiency of 29% which was obtain at 30°C. The difference between the trends for inhibitor efficiency of eucalyptus camaldulensis extract obtain at 30°C, 40°C and 50°C at the acid concentration over the range of *Eucalyptus camaldulensis* concentration presently studied strongly suggest that the mechanism of adsorption of the inhibitor on the aluminum coupon surface is predominantly by chemical adsorption.

Figure 4 shows the variation of free energy with inhibitor concentration at 30°C, 40°C and 50°C. The graph shows that the highest free energy was obtain in inhibitor concentration of 2.0 (%w/v) at 30°C while the least was observe at 0.5 (%w/v) at 50°C. The negative value of free energy of adsorption suggests a strong interaction of the inhibitor molecules on the surface of the aluminum coupons (Monticelli et al. [16]).

In Figure 5, the graph shows that the value of heat adsorption were negative at all *Eucalyptus camaldulensis* extract concentration studied and ranged from -36990.71 to -8898.11 (J/mol). Indicate that the adsorption of the extract on aluminum coupon surface is exothermic. The negative value shows that the adsorption and hence the inhibitor efficiency decreases with rise in temperature.

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