



Original Article

Study of corrosion inhibition performance of Glutaraldehyde on Aluminium in nitric acid solution

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ABSTRACT

The inhibition effect of glutaraldehyde on the corrosion behavior of aluminium in 1.4 M nitric acid solution was studied using weight loss method, adsorption studies and characterized instrumentally by Fourier Transform Infrared Spectroscopic (FT-IR) analysis. The effects of inhibitor concentration, temperature and surface coverage were all investigated. The effect of inhibitor concentration and other parameters were evaluated for different inhibitor concentrations and the probable mechanism for the studied inhibitor was also proposed. The results showed that glutaraldehyde possess an excellent inhibiting effect toward the corrosion of aluminium in nitric acid solution with highest inhibition efficiency of 84.68, 81.34 and 76.87 % at 308, 313 and 318 K in the presence of 0.1 M inhibitor concentration. The values of the activation energy, rate constant, half-life and rate constant in uninhibited acid solution were $31.84 \text{ kJ mol}^{-1}$, $3.9 \text{ k} \times 10^{-3} \text{ hr}^{-1}$ and $1.76 \times 10^2 \text{ hr}$ which changed to $57.64 \text{ kJ mol}^{-1}$, $0.60 \text{ k} \times 10^{-3} \text{ hr}^{-1}$ and 11.55 hr in the presence of 0.1 M inhibitor concentration. The positive values of enthalpy reflect the endothermic nature of the reaction. The negative values of entropy (ΔS) signified that the activated complex in the rate determining step represented an association, rather than dissociation. The adsorption of the inhibitor on the metal surface followed Langmuir adsorption isotherm.

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1. Introduction

Corrosion refers to an irreversible interfacial reaction of a material usually a metal, polymer and ceramic, on exposure to an aggressive environments, affects the performance efficiency of a material which also leads to reduction of its mass [1, 2]. Many technological efforts have and are currently being developed to reduce corrosion of service materials by proper selection of the materials, change in design philosophies and more importantly exercising of varied prevention techniques. Considerable attention has been received on Corrosion studies of Aluminium and Aluminium alloys by researches as a result of its technological importance and industrial applications. They find applications in household appliances, automobiles, food-handling containers, building, aviation and electronic devices because of their advantages; lightness, low cost

and good corrosion resistance at moderate temperatures [3, 4]. It has been reported that the passive oxide film formed on aluminium surface is responsible for its protection against corrosion, but the surface film is amphoteric that dissolves substantially whenever the metal is exposed to high concentrations of acids or bases.

Aluminium dissolves readily in acid to form Aluminium salt and hydrogen. The reaction is more vigorous when the acid becomes hotter and more concentrated. The attention on the protection of metals against corrosion has being on, without abating, because of enormous losses of natural resources and finances associated with corrosion effect. Apart from the economic outlook, corrosion control is also important from environmental and aesthetical angles [5-7]. One of the most importance methods normally employed to

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combat corrosion is the application of corrosion inhibitors and majority of the well-known inhibitors are organic compounds. Organic compounds have been accepted as effective inhibitors for metal corrosion in various solutions. Most of the effective and most efficient organic inhibitors are those with heteroatoms containing elements such as oxygen, nitrogen, sulphur, and phosphorus, in their structures, which permit them to be adsorbed onto the metal surface [8 -11], thereby interacting with anodic and/or cathodic reaction sites and cause the oxidation and

reduction corrosion reaction, and prevent transportation of water and corrosion active species onto the surface of metal. In this work, the inhibition effect of glutaraldehyde on corrosion reaction of Aluminium was investigated at temperature range of 308, 313 and 318 K in 1.4 M HNO₃ medium. Adsorption mechanisms were deduced through the data obtained from weight loss method. This submission is in furtherance of contribution toward the sustained global interest in the search for less toxic inhibitors for corrosion of metals.

2. Methodology

2.1. Sample Preparation

Aluminium metal (Al, 99% purity) was obtained from Metal Focus Fabrication Technology Incubation Centre Kano State, Nigeria. The metal was pressed cut into 2 x 3 x 0.1 cm coupon. The coupons were polished with 240, 640, 800 and 1000 grades of different emery paper respectively. The coupons were degreased in ethanol, washed with acetone, and then kept in desiccator prior to use.

2.2. Preparation of Solutions

Required volume of the stock solution of acid was measured using a measuring cylinder and poured into a standard volumetric flask and then filled up to the mark with distilled water to obtain the desired concentration of 1.4M nitric acid solution. The used inhibitor was glutaraldehyde and concentrations of the inhibitor used for the study were 0.02, 0.04, 0.06, 0.08 and 0.1 M. Each of these concentrations was diluted in the prepared desired concentrations of acid for use as test solutions in weight loss experiment.

2.3. Weight loss Measurement

The prepared weighed aluminium metal coupons were immersed in 100 ml beaker containing 50 cm³ of the acid solution in the absence and presence of various inhibitor concentrations (0.02, 0.04, 0.06, 0.08 & 0.1 M) at 308 K and 3 hours immersion time, after which they were removed, washed, dried, re-weighed and recorded respectively. The experiment were performed in replicate. The effect of temperature was studied at a temperature range of 308, 313 and 318 K. The weight loss of

Aluminium was calculated in grams as the difference between the initial weight and the weight after the removal of the corrosion product. The weight loss (Δw), corrosion rate (C_R), inhibition efficiency (I.E) and degree of surface coverage (Θ) were calculated using the Equations 2.1, 2.2, 2.3 and 2.4 respectively.

$$\Delta w = w_i - w_f \quad (1)$$

$$C_R = \frac{w_i - w_f}{At} \quad (2)$$

$$\Theta = \frac{w_0 - w_1}{w_0} \quad (3)$$

$$I.E = \frac{w_0 - w_1}{w_0} \times 100 \quad (4)$$

Where w_i and w_f are the initial and final weight of Aluminium metal coupons, w_1 and w_0 are the weight loss values in presence and absence of inhibitor, respectively. A is the total area of the aluminium specimen and t is the immersion time.

2.4. Fourier Transform Infrared Spectroscopic Analysis

FT-IR analysis was carried out for the fresh inhibitor (glutaraldehyde) and that of the corrosion product obtained from the reaction of Aluminium immersed in 1.4 M HNO₃ solution for 3 hours immersion time in the presence of 0.1 M inhibitor (glutaraldehyde) at 308 K using Agilent Technology, FTIR (Cary 630) Fourier Transform Infrared Spectrophotometer. The analysis was done by scanning the sample through a wave number range of 650 – 4000 cm⁻¹; 32 scans at 8 cm⁻¹ resolution.

3. Result and Discussion

3.1. Effect of inhibitor concentration on corrosion rate

Figure 3.1 shows the result for the effect of inhibitor on the corrosion rate of aluminium in HNO₃, in the presence of 0.02 - 0.1 M inhibitor. The results shows the gradual decrease in corrosion rate of aluminium in nitric acid solution. The values of corrosion rates in the absence of

inhibitor was found to be higher when compared to the values in the presence of inhibitor. The corrosion rate was inversely proportional to the concentration of inhibitor at the same temperature. Examination of the results revealed that lower corrosion rate was obtained at the highest concentration of the inhibitor. The decrease of corrosion

rate in the presence of inhibitor indicates the effectiveness action of the compound as corrosion inhibitor. This suggests that as the concentration of inhibitor increases, there was an increase in surface coverage of the adsorbed inhibitor molecules on the aluminium surface which provided a barrier (film) and reduced further corrosion. Similar results was reported by Ansari *et al* [12] during the study for effect of three component (aniline–formaldehyde and piperazine) polymer on mild steel corrosion in hydrochloric acid medium and the result of the corrosion rate in uninhibited acid was $7 \text{ mg cm}^{-2}\text{h}^{-1}$. Likewise there was decreased in corrosion rate from 1.60 to $0.13 \text{ mg cm}^{-2}\text{h}^{-1}$ in the presence 25 to 100 mg/L inhibitor concentration.

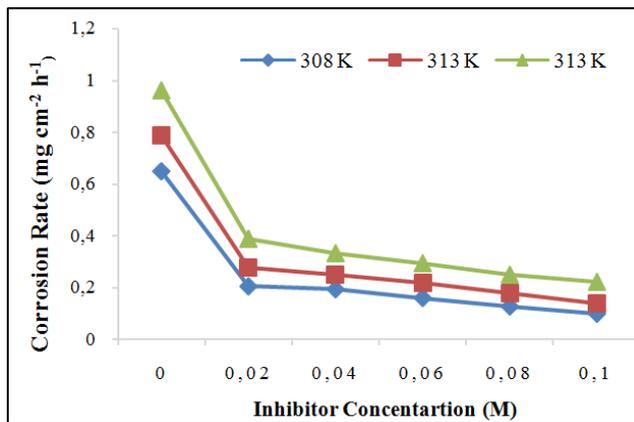


Fig 3.1. Variation of Corrosion Rate with Inhibitor Concentration for Al Corrosion in 1.4 M HNO₃

3.2. Effect of inhibitor concentration on inhibition efficiency

The result presented in Figure 3.2 shows the effect of inhibitor concentration against percentage inhibition efficiency (% IE). The percentage inhibition efficiency (% IE) was found to increase with increase in inhibitor concentration due to increase in surface coverage (Θ) of adsorbed specie on surface of the metal.

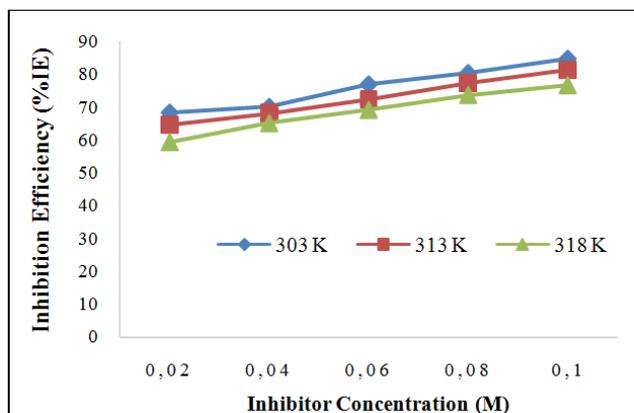


Fig 3.2. Variation of Inhibition Efficiency with Inhibitor Concentration for Al Corrosion in 1.4M HNO₃

The increase in percentage inhibition efficiency (% IE) indicated that more inhibitor molecules were adsorbed on the metal surface thus providing wider surface coverage. Similar work was reported by Omotoma and Onukwuli [13] during evaluation of pawpaw leaves extract as anti-corrosion agent for aluminium in hydrochloric acid medium and result for the inhibition efficiency of aluminium increased from 51.64 to 84.22 at 0.2 and 1.0 g/L inhibitor concentration.

3.3. Effect of Temperature on Corrosion Rate

The variation of corrosion rate against temperature at different inhibitor concentration has been shown in Figure 3.3. The results revealed that the corrosion rate increases with increase in temperature. Increase in temperature leads to destabilizing the inhibitor molecule and decrease its activity. The highest corrosion rate of aluminium was observed at the highest temperature as it can be seen from the result. This observation is due to the fact that chemical reaction rates generally increases with rising temperature. Increase in temperature leads to increase in the kinetic energy possessed by the reacting molecules thereby making the molecules to overcome the energy barrier faster [14]. Similar results was reported by Ansari *et al* [12] during the study for effect of three component (aniline–formaldehyde and piperazine) polymer on mild steel corrosion in hydrochloric acid medium and the result of the corrosion rate was found to increase from 0.13 to $3.06 \text{ mg cm}^{-2}\text{h}^{-1}$ at 308 to 338 K.

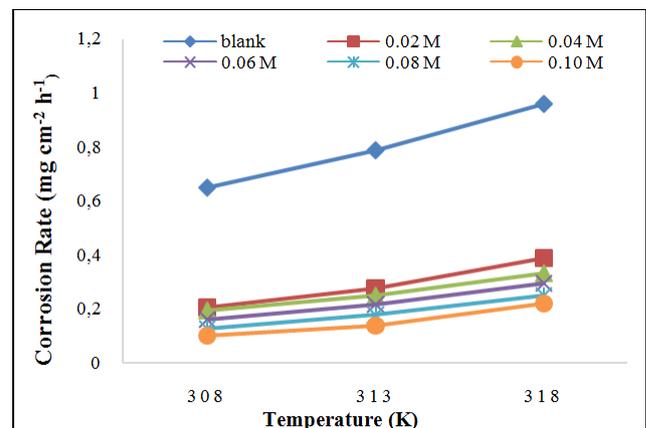


Fig 3.3. Variation of Corrosion Rate with Temperature for Al Corrosion in 1.4 M HNO₃.

3.4. Effect of Temperature on Inhibition Efficiency

Variation effect of temperature on the inhibition efficiency for the corrosion of aluminium at different inhibitor concentration has been shown in Figure 3.4. The result shows that the inhibition efficiency decreases with increase in temperature. It is suggested that the decrease of inhibition efficiency is due to the desorption of the inhibitor molecules on the surface of aluminium as a result

of increase in temperatures resulting in destabilizing the inhibitor, this results in reducing the efficiency of physical adsorption activity. Similar work was reported by Adejo *et al* [15] adsorption characterization of ethanol extract of leaves of portulaca oleracea as green corrosion inhibitor for corrosion of mild steel in sulphuric acid medium and the result of the inhibition efficiency was found to decrease from 68.19 to 32.72 % with increase in temperature from 305 to 315 K.

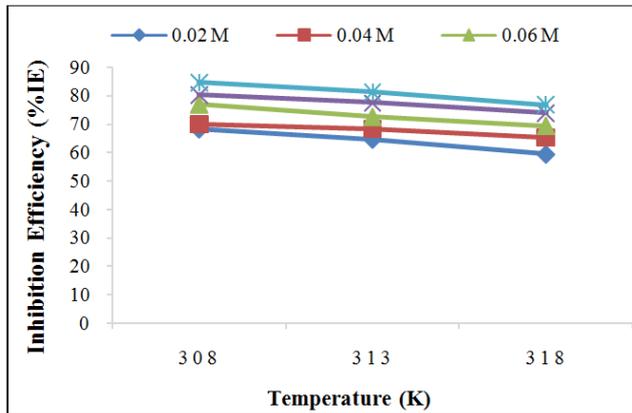


Fig 3.4. of Inhibition Efficiency with Temperature for Al Corrosion in 1.4 M HNO₃

3.5. Adsorption behaviour

The efficiency of a compound as a good inhibitor is mainly depend on its ability to get adsorbed on the metal surface, which consists the replacement of water molecules at the corroding interface. To ascertain the nature of adsorption, attempts were made to fit the experimental data into different adsorption isotherms. The result indicates that Langmuir adsorption isotherm model best described the adsorption properties of the inhibitor on the aluminium surface as it gives the highest values of correlation coefficient (R^2). Langmuir adsorption isotherm is the ideal adsorption isotherm for physical and chemical adsorption on a smooth surface, and it is valid for monolayer adsorption onto a surface containing a finite number of identical sites. According to this Langmuir adsorption isotherm, the surface coverage (Θ) is related to the inhibitor concentration C_{inh} by the equation given below:

$$\frac{C_{inh}}{\Theta} = \frac{1}{K_{ads}} + C_{inh} \quad (3.1)$$

Where K_{ads} is the adsorption equilibrium constant, C_{inh} is the inhibitor concentration in the solution, and Θ is the degree of surface coverage. The plot of C_{inh}/Θ against C_{inh} gave a straight line with slope equal to unity and R^2 close to 1 indicating that the adsorption of the inhibitor on the surface of Aluminium is consistent with Langmuir adsorption isotherm. The correlation coefficient (R^2) and adsorption equilibrium constant (K_{ads}) values are presented

in Table 3.3.

3.6. Kinetics Study

3.6.1. Activation Energy.

The rate of most chemical reactions increases with temperature obeying Arrhenius equation. In the case of the electrochemical reactions, temperature favors the kinetics of corrosion process and more specifically, the dissolution of the metal at anodic site. The activation energy of the corrosion process can be obtained from the Arrhenius plot according to the equation given below:

$$C_R = A \exp\left(\frac{-E_a}{RT}\right) \quad (3.2)$$

Taking logarithm of both sides of equation 3.2, would yield equation 3.3

$$\ln C_R = \ln A - \frac{E_a}{RT} \quad (3.3)$$

Where C_R is the corrosion rate of Aluminium. A is Arrhenius constant or pre-exponential factor, R is the universal gas constant, E_a is the activation energy of the process and T is the absolute temperature. The plot of $\ln(C_R)$ versus reciprocal of absolute temperature ($1/T$) gave a straight line with slope = $-E_a/R$, from which the activation energy values for the corrosion process were obtained.

From Table 3.1 the activation energy value obtained in the presence of the inhibitor is higher than the activation energy value in the absence of inhibitor indicating the deactivation of the acid molecule on collision with the metal surface by introduction of the inhibitor, thus reducing the rate of acid attack on the metal. The activation energy values increases with increase in inhibitor concentration. It is considered as good evidence supporting the higher Inhibition efficiency and lower corrosion rates of aluminium in higher inhibitor concentration. Activation energy served as another good tool in the determination and characterization of the type of adsorption taking place. The average activation energy values of ≤ 80 kJ/mol is consisted with physical adsorption, while those at ≥ 80 kJ/mol is consisted with chemical adsorption mechanism. The Calculated activation energies from experimental data of this current study are all below 80 kJ/mol. Therefore the adsorption of the studied inhibitor on the surface of aluminium is consistent with the mechanism of physical adsorption. The observed trend is similar to the one reported by Deepa and Rao [16] during Corrosion inhibition of 6063 Aluminium alloy by Coriandrum sativum L seed extract in phosphoric acid medium. The activation energy values were 50.24 and 75.99 kJ mol⁻¹ in the absence and present of 500 ppm inhibitor concentration.

3.6.2. Rate constant.

The kinetics of the corrosion reaction has the character of a diffusion process, in which the amount of inhibitor present on the metal surface is much reduced at higher temperature, than that present at lower temperature. In this present study, the initial weight of aluminium coupon at time t , is designated as W_i , the weight loss is W_L and the weight change at time t , is $(W_i - W_L)$. The first order reaction rate constants (k) was calculated from equation 3.4

$$\ln(W_i - W_L) = -k_1t + \ln W_L \quad (3.4)$$

According to equation 3.4 the plots of $\ln(W_i - W_L)$ against time showed a linear variation with correlation coefficients close to 1 which confirmed first - order kinetics for the corrosion of Aluminium in acid with and without the presence of inhibitor. From Table 3.1, the value of rate constant (k_1) for the corrosion of aluminium was found to be higher in uninhibited acid solution than inhibited acid solution. This confirmed the inhibition of aluminium corrosion in acid solution by the presence of the studied inhibitor. The value of the rate constant for the corrosion of aluminium in uninhibited HNO_3 was $3.92 \times 10^{-3} h^{-1}$ while the values of the rate constant for the corrosion of aluminium in HNO_3 with 0.02 M inhibitor concentration was $1.23 \times 10^{-3} h^{-1}$ which increased to $0.60 \times 10^{-3} h^{-1}$ at 0.10 M inhibitor concentration. Similar research was reported by Ezeokonkwo *et al* [17] during corrosion inhibition of eucalyptus citriodora on aluminium and mild steel in acidic

media and the result of the rate constant for aluminium and mild steel in uninhibited acid solution were 0.00585 and 0.0181 day^{-1} , while in the presence of inhibitor the rate constant reduced to 0.00226 and 0.0141 day^{-1} .

3.6.3. Half – life.

The half – life ($t_{1/2}$) of the first order equation was calculated by using equation given below:

$$\text{Half – life } (t_{1/2}) = \frac{0.693}{K} \quad (3.5)$$

From Table 3.1 it can be seen that the value of the half-lives ($t_{1/2}$) increased from uninhibited solution to inhibited solution The increase in half-life ($t_{1/2}$) in the presence of the inhibitor compared to the uninhibited solution further corroborate with the results reported earlier that corrosion rate decreases in the presence of the inhibitor compared to the uninhibited solution. It should also be noted that as inhibitor concentration increases the half-life also increases which results into a decrease in the corrosion rate, suggesting that more protection of the aluminium by the presence of higher inhibitor concentration has been established. Similar research was reported by Olasehinde *et al* [18] during investigation of the inhibitive properties of alchornea laxiflora leaves on the corrosion of mild steel in HCl and the result of the rate constant and half-life in uninhibited acid solution were 0.01 day^{-1} and 69.3 days, while in the presence of 0.1M inhibitor concentration the rate constant reduced to 0.05 day^{-1} and half-life increased to 138.6days.

Table 3.1: Kinetic Parameters for Aluminium Corrosion with and without various inhibitor concentrations

Inhibitor Concentration (M)	Activation Energy(kj mol ⁻¹)	Rate Const. ($k \times 10^{-3}$) (hour ⁻¹)	Half-life (10 ²) (hours)
Blank	31.84	3.92	1.76
0.02	43.87	1.23	5.60
0.04	49.09	1.16	5.92
0.06	51.89	0.96	7.15
0.08	54.64	0.76	9.02
0.10	57.64	0.60	11.55

3.7. Thermodynamic Study

The Thermodynamic parameters such as enthalpy (ΔH) and entropy of reaction (ΔS) were calculated using the transition state equation. The linear form of transition state equation is given by the equation below;

$$\ln\left(\frac{C_R}{T}\right) = \ln\left(\frac{R}{Nh}\right) + \left(\frac{\Delta S}{R}\right) - \left(\frac{\Delta H}{RT}\right) \quad (3.6)$$

Where h is Plank's constant and N is Avagadro's number. A plot of $\ln(C.R/T)$ vs $1/T$ gave a straight line with slope = $-\Delta H/T$ and intercept = $\ln(R/Nh) + \Delta S/R$.

From Table 3.2 the calculated values of enthalpy for the corrosion of Aluminium in uninhibited HNO_3 is 29.24 kJmol^{-1} which increased to 41.26 and 62.35 kJmol^{-1} in the presence of 0.02 and 0.10 M inhibitor concentration. The values of enthalpy are all positive indicating the endothermic nature of the reaction suggesting that higher temperature favors the corrosion process. Generally enthalpy values of 41.9 kJmol^{-1} are related to physisorption while those around 100 kJmol^{-1} or higher are attributed to chemisorption. All the values of enthalpy are less than 100 kJmol^{-1} this proved that the adsorption of the inhibitor on the Aluminium surface in HNO_3 , follows physical adsorption mechanism. Similar research was reported by

Udom *et al* [19] during the study of the effect of *Acanthus montanus* leaves extract on corrosion of Aluminium in hydrochloric acid medium and the result of enthalpy was positive ranging from 17.41 to 24.42 kJmol⁻¹ in 1×10⁻⁶ to 5×10⁻² M inhibitor concentration.

Table 3.2: Enthalpy and Entropy change of the reaction process with various concentrations of the inhibitor

Inhibitor Concentration (M)	ΔH_{ads} (kJ mol ⁻¹)	$-\Delta S_{ads}$ (J mol ⁻¹ k ⁻¹)
blank	29.24	211.26
0.02	41.26	182.29
0.04	46.49	166.86
0.06	49.29	155.77
0.08	52.04	150.77
0.10	62.35	119.51

The calculated values of entropy for the corrosion of aluminium in uninhibited HNO₃ from Table 3.2 is -211.26 JK⁻¹mol⁻¹ which decreased to -182.29 and -119.51 JK⁻¹mol⁻¹ in the presence of 0.02 and 0.10 M inhibitor concentration. The entropy values are all negative confirming that the corrosion process are entropically favourable. The values of ΔS decreased negatively from lower to higher inhibitor concentration. This reveals the formation of an ordered stable film of the inhibitor molecule on the aluminium surface, and the activated complex in the rate determining step represent an association rather than a dissociation step [20].

3.8. Free Energy of Adsorption (ΔG_{ads})

The tendency for many chemical reaction to proceed, including the reaction of a metal with its environment is measured by the Gibb's free energy change (ΔG). The more negative value of ΔG , gives the greater the tendency for the reaction to proceed. The free energy of adsorption (ΔG_{ads}) is related to adsorption equilibrium constant (K_{ads}) by the equation given below

$$\Delta G_{ads} = -RT \ln (55.5 K_{ads}) \quad (3.7)$$

The values of free energy of adsorption are presented in Table 3.3. The calculated values of free energy of adsorption for the corrosion of aluminium in HNO₃ in the presence of inhibitor are -21.64, -21.83 and -22.33 kJ mol⁻¹ at 308, 313 and 318 K respectively.

The obtained results indicated that the values of free energy of adsorption are all negative. This is the fundamental criterion for spontaneity. The negative values is showing that the reaction is spontaneous. Generally, the values of ΔG_{ads} below or around -20 kJmol⁻¹ are consistent with electrostatic interaction between charged molecules and charged metal surface which indicates physical adsorption mechanism, while those at -40 kJmol⁻¹ or above involved charge sharing or transfer from the inhibitor molecule to the metal surface to form a coordinate type of bond which indicates chemical adsorption mechanism. In this study all ΔG_{ads} values are around -20 kJmol⁻¹, therefore the adsorption of the inhibitor on the surface of aluminium is consisted with electrostatic attraction between charged metal surface and charged species in the bulk solution. This result is similar to the one reported by Ebenso *et al* [21] who reported the values of ΔG_{ads} ranges from -5.8193 to -14.5648 kJmol⁻¹ in the study of corrosion inhibitive properties and adsorption behaviour of ethanol extract of *Piper guinesis* as a green corrosion inhibitor for mild steel in H₂SO₄.

Table 3.3: Adsorption Parameters Deduced from Langmuir Adsorption Isotherm for Corrosion Inhibition of Aluminium.

Temperature(K)	R ²	K _{ads}	ΔG_{ads} (kJ mol ⁻¹)
308	0.994	97.08	-21.64
313	0.994	90.90	-21.83
318	0.997	84.03	-22.33

3.8. Infrared Spectroscopy Analysis Results

Figures 3.5 & 3.6 shows the result of FT-IR analysis of the inhibitor (glutaraldehyde) and the corrosion product of Aluminium in 1.4 M HNO₃ with 0.1 M inhibitor concentration. The analysis of the inhibitor in figure 3.5 shows C-H aldehyde bending frequency and C=O carbonyl stretching frequency. The adsorption of both C-H aldehyde bending and C=O carbonyl stretching frequency has been observed in the analysis of the corrosion product presented in Figure 3.6. The appearance of these adsorption band suggest that the adsorption between the inhibitor and Aluminium take place through the functional group of the inhibitor molecule.

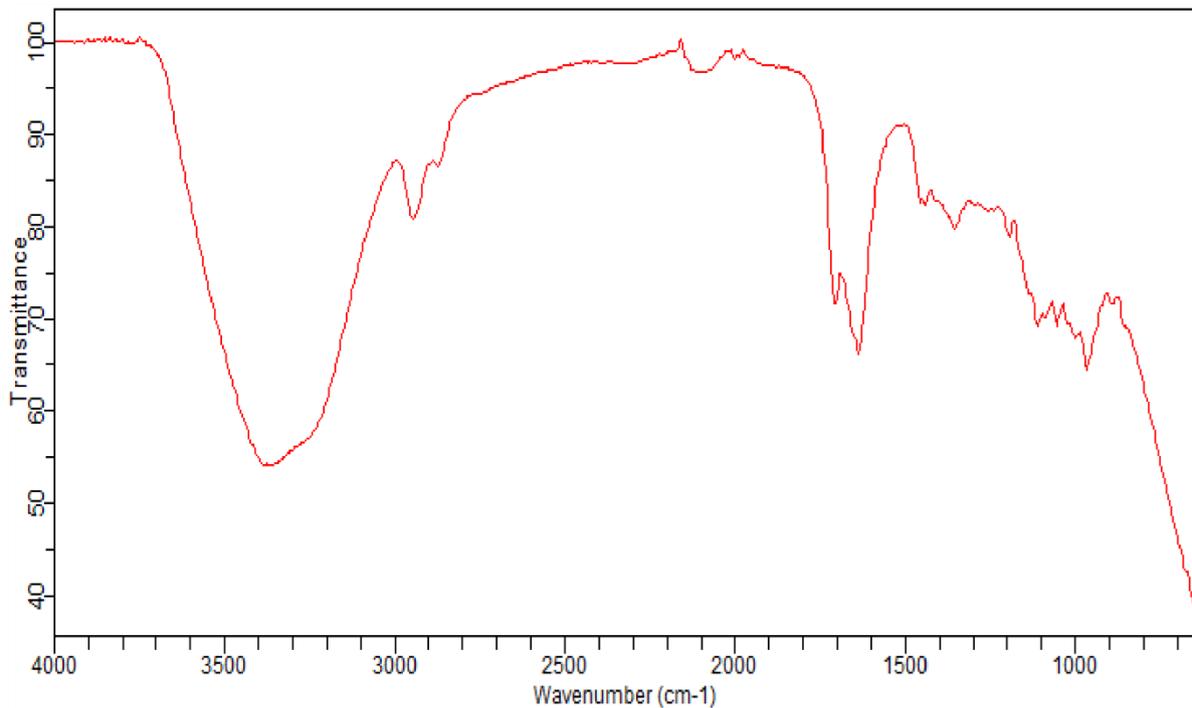
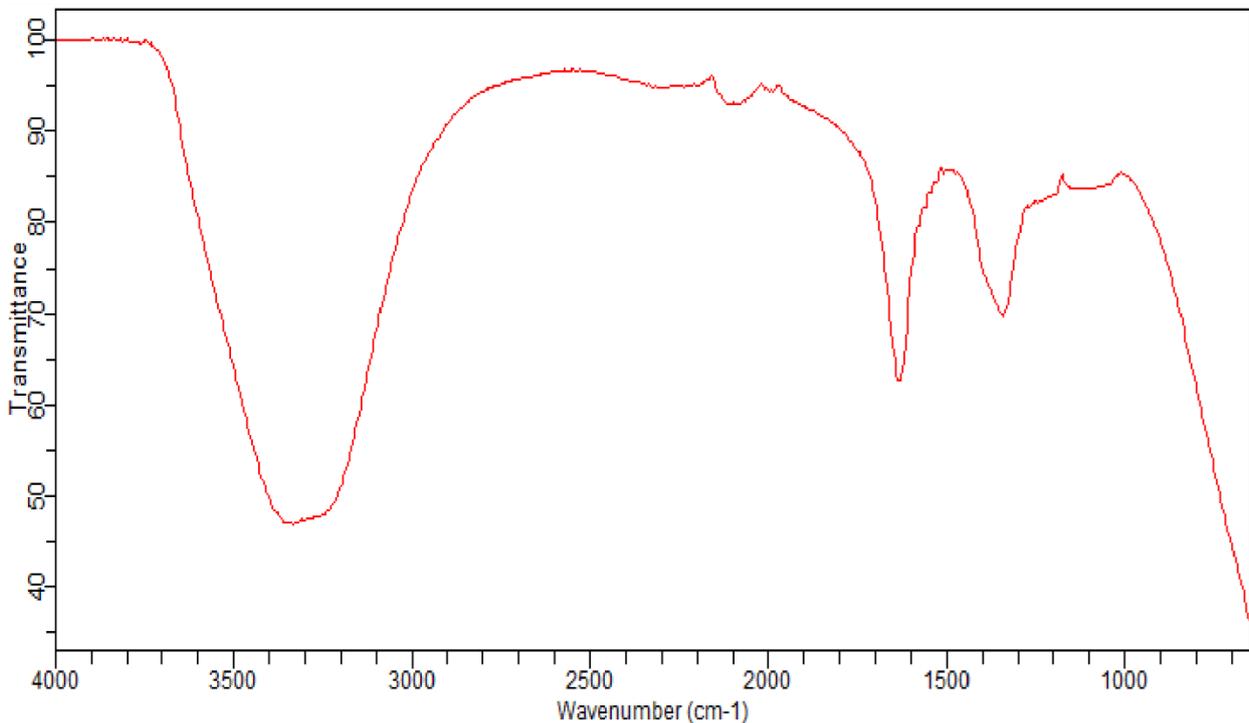


Fig 3.5. FT-IR Spectra of Glutaraldehyde

Fig 3.6. FT-IR Spectra of Aluminium in 1.4M HNO₃ with 0.1M Glutaraldehyde

4. Conclusion

Results from the weight loss method employed showed that; glutaraldehyde served as an effective inhibitor for the corrosion of aluminium in nitric acid solution. Increase in temperature increases corrosion rate but decreases the inhibition efficiency. Inhibition efficiency was found to increase with increase in concentration of the inhibitor. The values of activation energy, enthalpy and Gibbs free energy

of adsorption indicate that adsorption occur through the mechanism of physical adsorption. The large and negative values of Gibbs free energy of adsorption confirmed the spontaneity of the reaction. The results of FT-IR spectra showed that the inhibitor was adsorbed on the metal surface through physisorption mechanisms as proposed by the kinetics and the thermodynamic data.

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