



Original Article

Corrosion inhibition of aluminium in phosphoric acid solution using glutaraldehyde as inhibitor

Musa Husaini*, Umar Yunusa, Hussaina Aminu Ibrahim, Bishir Usman and Muhammad Bashir Ibrahim

Department of Pure and Industrial Chemistry, Faculty of Physical Sciences, College of Natural and Pharmaceutical Science, Bayero University Kano (BUK), P.M.B. 3011, Kano, Nigeria

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ABSTRACT

The effect of glutaraldehyde as corrosion inhibitor for aluminum in phosphoric acid solution was studied by using weight loss method and characterized by fourier transform infrared spectroscopic and scanning electron microscopic analysis. The inhibition efficiency increases with increase in inhibitor concentration and decreases with increase in temperature while the corrosion rates increases with rise in temperature and decreases with increase in inhibitor concentration. The value of activation energy (ΔE_a) was found to be 32.61 kJ/mol in uninhibited acid solution and increased to 47.33 kJ/mol in the presence of 0.1 M inhibitor concentration. The positive values of the enthalpy of activation (ΔH_a) suggest an endothermic nature of the reaction and negative values of activation entropy (ΔS_a) shows that the activation complexes in the rate determining step represent an association rather than dissociation. The values of free energies of adsorption were all negative which indicate spontaneity of the process. The phenomenon of physical adsorption is proposed from the values of activation energy and free energy of adsorption. The results also shows that the adsorption of inhibitor molecules on the surface of aluminum is accord with Langmuir adsorption isotherm as to the values of correlation coefficient (R^2) obtained were very close to unity. The result of the SEM analysis in the presence of inhibitor shows how the aluminium surface was protected against the aggressive effect of the acid. The new functional group in the FTIR analysis of the corrosion product is an indication of thin layer formation on the aluminium surface.

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1. Introduction

Corrosion is the gradual degradation of materials (usually metals), due to chemical reaction with its environment. In the most commonly use word, this stand for electrochemical oxidation of metallic substances in reaction with a particular oxidant such as oxygen. Metallic materials play an important role in the construction of many equipment used in petrochemical, oil and gas, agriculture, process and allied industries and also in medical services. In these industries, the metallic material losses integrity over a period of time as a result of interaction with its environment [1]. However, corrosion of stainless steels, structural grades of cast iron and some

aluminium alloys occur speedily unless the metal is protected against corrosion. Aluminium is reactive metal and prone to corrosion. Its corrosion immunity in many environments can be explained due to the formation of a compact, adherent passive oxide layer. When in contact with air or immersed in solution, aluminium is always spontaneously covered by an oxide film which protects the metal from further oxidation [2].

Aluminium and its alloys are exposed to the action of acid like hydrochloric acid, phosphoric acid and sulphuric acid in many industrial processes where acid play an important role such as acid pickling, oil well acidizing,

* Corresponding author. Tel.: +2348036283936

E-mail address: musahusaini36@gmail.com

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acid cleaning and descaling, this can lead to substantial metal loss due to corrosion [3]. Efforts have been made to protect the integrity of aluminum surface in an aggressive acid medium or other corrosive environment. In recent decades, the addition of inhibitors has been considered to be the most common approach to hinder the corrosion of aluminum [4].

Large numbers of organic compounds were studied to investigate their corrosion inhibition potential activity. All these investigation reveal that organic compounds having electron density on their heteroatoms like O, N, S, shows significant efficiency i.e they have the tendency to resist corrosion [5, 6]. The heteroatoms served as the active center for the process of adsorption on metal surfaces, in addition, compounds with π -bonds, electronegative groups, conjugated double or triple bonds, usually exhibit good inhibitive property. They reduces the corrosion by supplying electrons from the electron-rich bonds to the metal surface which they are effectively adsorbed [7, 8].

The aim of this study was to evaluate the corrosion inhibition effect of glutaraldehyde through weight loss method and to carryout surface and functional group characterization by using scanning electron microscopic and Fourier Transform Infrared Spectroscopic analysis

2. Materials and Methods

2.1. Sample Preparation

The aluminum sample with a chemical composition of 99.500 % Al, 0.0007 % Pb, 0.3202 % Fe, 0.0024 % Sn, 0.0030 % Mn, 0.0015 % Cu, 0.0018 % Mg, 0.0074 % Ti, 0.0041 Cr, 0.0045 % Ni, 0.0060 % Zn and 0.0085 % Si was used in the current study. The metal sample was press cut in to 3 x 2 x 0.1 cm dimensions. The metal sample was polished by different grade of emery paper. It was then degreased in ethanol, dried in acetone and kept in a desiccator to use during the weight loss experiment.

2.2. Preparation of Solutions

Phosphoric acid with percentage purity (85 %) and density (1.685 g/L) was used as corrosive solution. The stock solution was used to prepare 1.4 M concentration by diluting analytical grade of H_3PO_4 using double distilled water. The inhibitor used was glutaraldehyde having a percentage purity (50 %) and density (1.06 g/cm³). The concentrations of the inhibitor used for the study was 0.02, 0.04, 0.06, 0.08 and 0.1 M. Each of these concentrations was diluted in the prepared desired concentration of acid for used as test solution in weight loss experiment.

2.3. Weight loss measurement

The method used by Husaini et al. [9] was adopted in weight loss experiment, where the prepared aluminium

coupons were each suspended completely in 1.4 M H_3PO_4 solutions without and with different concentrations of inhibitor (glutaraldehyde) with the help of glass hooks at temperature of 308 K and 3 hrs immersion time. The volume of solution was 100 cm³, the aluminium coupons were retrieved after 3 hrs, washed by distilled water, dried well and reweighed. From the weight loss which is an average of triplicate measurements, the weight loss in gram of aluminium in phosphoric acid solution without and with different inhibitor concentration (0.02, 0.04, 0.06, 0.08 and 0.10 M) for an immersion time of 3 hrs was calculated from the weight loss data obtained by the equation given below;

$$\text{Weight loss } (\Delta W) = W_1 - W_2 \dots \dots \dots (1)$$

Where W_1 and W_2 are the weight of coupons before and after immersion

$$\text{Corrosion rate (mg/cm}^2\text{h}^{-1}\text{)} =$$

$$\frac{\text{Weight loss (g)}}{\text{Metal surface area (cm}^2\text{)} \times \text{Time (hrs)}} \dots \dots \dots (2)$$

The degree of surface coverage (Θ) and inhibition efficiency (I.E.) for different concentration of the inhibitor in acidic solution was evaluated from weight loss experiments using the equations below:

$$\text{Surface coverage } (\Theta) = \frac{W_0 - W}{W_0} \dots \dots \dots (3)$$

$$\text{Inhibition efficiency (I.E)} = \frac{W_0 - W}{W_0} \times 100\% \dots \dots \dots (4)$$

Where W_0 and W are the weight of the aluminium coupons in the absence and presence of inhibitor.

2.4. Temperature effect

In the study of temperature effect on corrosion rate, the prepared aluminium coupons were completely immersed in phosphoric acid solution without and with different concentrations of the inhibitor at 308, 313 and 318 K for 3 hrs.

2.5. Fourier Transform Infrared Spectroscopic Analysis

Fourier transform infrared spectroscopic analysis of the fresh inhibitor and that of the corrosion product obtained after the reaction of aluminium immersed in phosphoric acid solution in the presence of 0.1 M inhibitor at 308 K were carried out using Fourier Transform Infrared Spectrophotometer, Agilent Technology, FTIR (Cary 630). 650 – 4000 cm⁻¹ wave number was used to scan the sample during the analysis.

2.6. Scanning Electron Microscopic Analysis

Surface morphologies of the aluminium samples before and after inhibition were studied by using PRO: X: Phenom World with 800-07334 model number, manufactured by Phenom World Eindhoven, Netherlands. Scanned images of un-reacted aluminium sample, aluminium sample immersed in 100 cm³ of 1.4 M H₃PO₄ without inhibitor and aluminium sample immersed in 100 cm³ of 1.4 M H₃PO₄ with 0.1 M inhibitor concentration at temperature of 308 K and 3 hrs immersion time were taken at an accelerating voltage of 15.00 kV and x2000 magnification.

3. Results and Discussion

3.1. Effect of inhibitor on corrosion rate and inhibition efficiency

Presented result in figure 1 is the plot of corrosion rate against inhibitor concentration for aluminium corrosion in 1.4 M H₃PO₄ at different temperatures. The figure reveals that the corrosion rate of aluminium decreased with increase in concentration of the inhibitor at all studied temperature. This indicate that the protection potential of the inhibitor was concentration dependent [9]. The decrease in corrosion rate in the presence of inhibitor indicate the inhibition ability of the glutaraldehyde as inhibitor. This shows that as the inhibitor concentration increase, there was an increase in surface coverage due to the adsorbed molecules of the inhibitor on the aluminium surface which formed a barrier between the aluminium surface and corrosive media. Similar work was reported by Olasehinde et al. [10] during investigation of the inhibitive properties of alchornea laxiflora leaves on the corrosion of mild steel in HCl and the result showed that corrosion rate decreases with increase in inhibitor concentration.

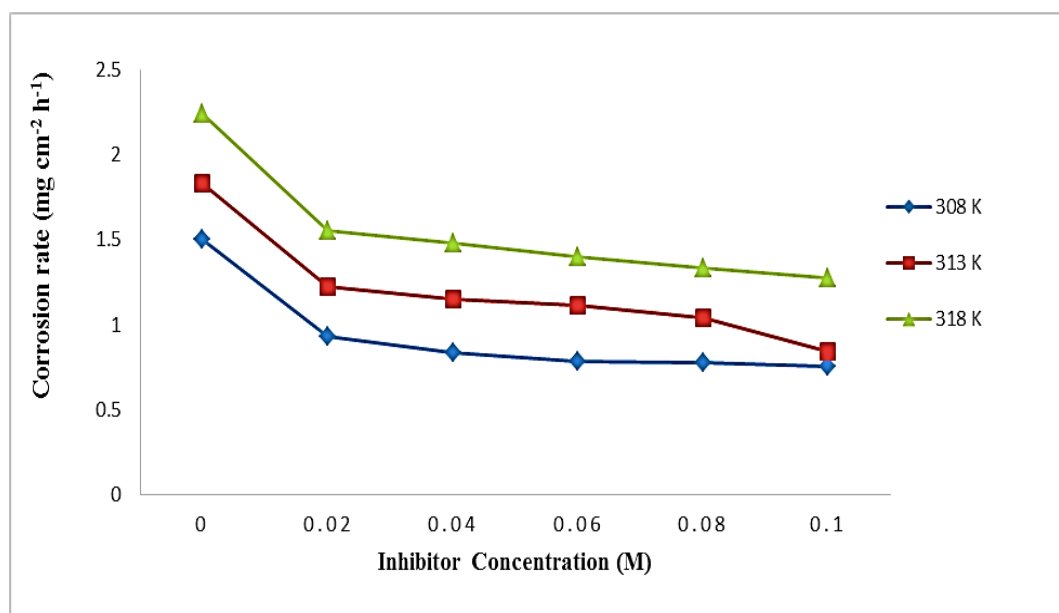


Figure 1. Variation of Corrosion Rate with Inhibitor Concentration for Al Corrosion in H₃PO₄.

The surface coverage (Θ) and inhibition efficiency (% IE) were found to increase with increase in concentration of the inhibitor. The plot for the variation of inhibition efficiency against inhibitor concentration is presented in Figure 2. The highest inhibition efficiency was observed at 0.1 M inhibitor concentration and at 308 K temperature. This trend was observed as a result of increase in surface coverage (Θ) of adsorbed inhibitor molecule on surface of the aluminium, due to increase in inhibitor concentration [11]. This shows that more inhibitor molecules were adsorbed on the metal surfaces, which lead to increase in inhibition efficiency. Similar work was

reported by Husaini *et al.* [12] during the study of effect of aniline as corrosion inhibitor on the corrosion of aluminium in hydrochloric acid solution and the result of inhibition efficiency was found to increase with increase in inhibitor concentration.

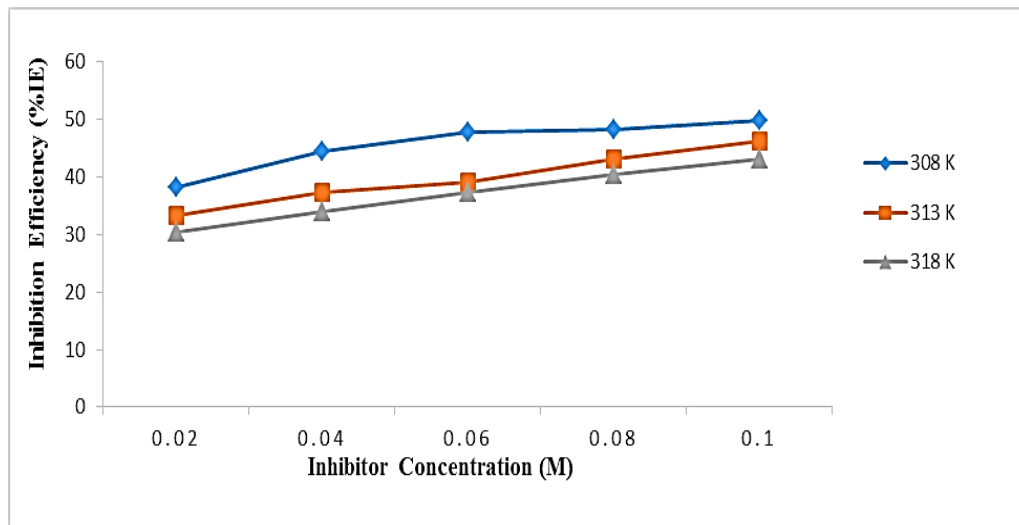


Figure 2. Variation of Inhibition Efficiency with Inhibitor Concentration for Al Corrosion in H_3PO_4 .

3.2. Effect of Temperature on Corrosion Rate and Inhibition Efficiency

The result presented in figure 3 shows the variation effect of temperature on the corrosion rate of aluminium for inhibited and uninhibited acid solution at 308, 313 and 318K. It can be seen that from the result the rate of inhibited and uninhibited solution increases with increase in temperature. The highest corrosion rate of aluminium was observed at 318 K which is the highest temperature. This observation is due to the fact that rising the reaction

temperature generally increases the rate of chemical reaction. Increase in temperature cause the increase in the average kinetic energy of the reacting molecules thereby making the reactant molecules to overcome the energy barrier faster [13]. Similar work was reported by Husaini and Ibrahim [14] during the study of inhibitive effect of glutaraldehyde on the corrosion of aluminium in hydrochloric acid solution and the result of corrosion rate was found to increase with increase in temperature.

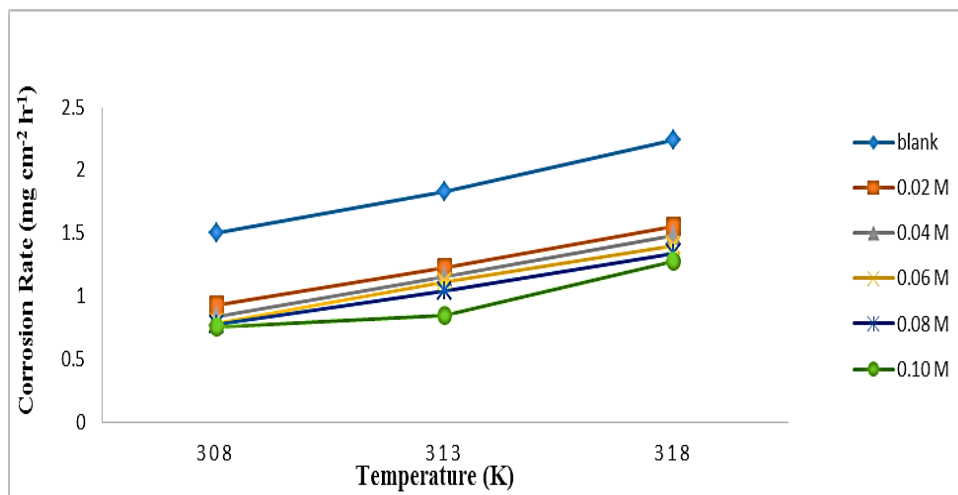


Figure 3. Variation of Corrosion Rate with Temperature for Al Corrosion in H_3PO_4 .

The result for the temperature variation effect for the inhibition efficiency of the inhibitor on the corrosion of aluminium at 308, 313 and 318 K in phosphoric acid solution is presented in Figure 4. The observation from the result shows the decrease in inhibition efficiency with

increase in temperature. Decrease in inhibition efficiency occurs as a result of desorption and destabilization of the adsorbed inhibitor molecules from the surface of metal due to the increase in temperature. This suggests that the molecules of inhibitor were physically adsorbed on the

surface of aluminium [15]. Similar result was reported by Husaini and Ibrahim [16] during the thermodynamic and kinetic study on the corrosion of aluminium in hydrochloric acid using benzaldehyde as corrosion inhibitor and the result of inhibition efficiency was found to decrease with increase in temperature.

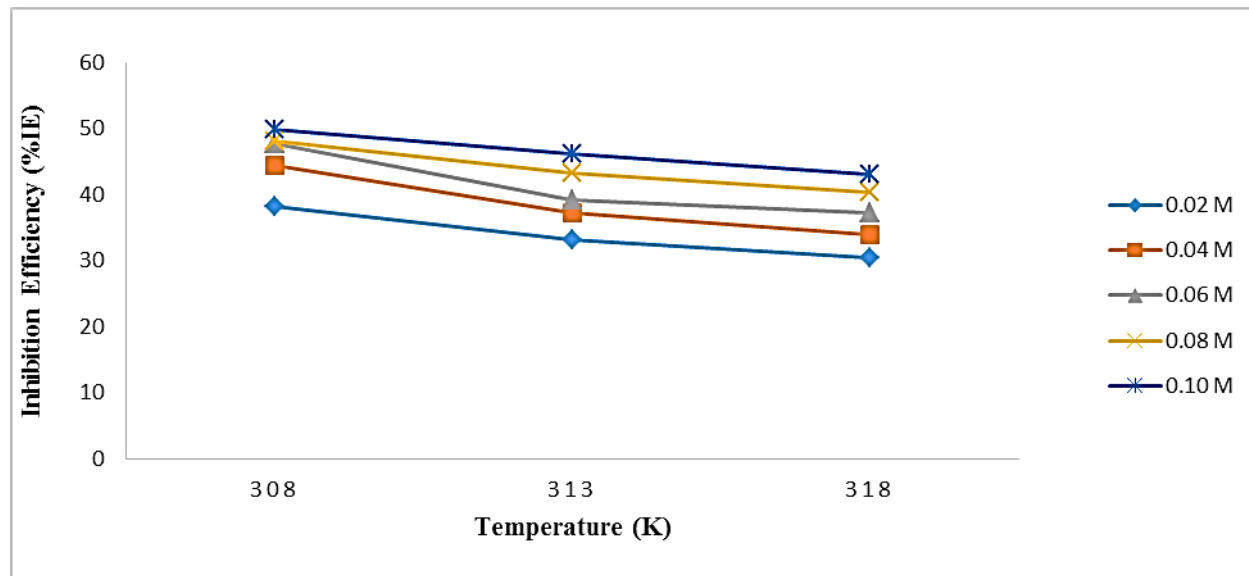


Figure 4. Variation of Inhibition Efficiency with Temperature for Al Corrosion in H_3PO_4 .

3.3. Thermodynamic Study

3.3.1. Determination of Activation Energy (E_a)

To elucidate the inhibitive properties of the studied inhibitor and temperature dependence on the corrosion rate, the activation energy (E_a) for the corrosion of aluminium in uninhibited and inhibited acid solution was calculated from Arrhenius-type. The linear form of Arrhenius equation is given in the equation below;

$$\ln(CR) = \ln A - \frac{E_a}{RT} \dots \dots \dots (5)$$

Where E_a is the apparent activation energy, R is the universal gas constant, T is the absolute temperature and A is constant. Values of apparent activation energy for the corrosion of aluminium in H_3PO_4 without and with different concentrations of inhibitor are shown in Table 1 and presented in Figure 5. These values were determined from the slope of $\ln CR$ versus $1/T$ plots. It can be seen that the activation energy values of inhibited solution are higher than that for uninhibited acid solution, suggesting slow dissolution of aluminium in the presence of the inhibitor [17]. It is known from Arrhenius equation that the higher values of activation energy lead to the lower corrosion rate, due to the formation of a thin layer film on the surface of aluminium serving as an energy barrier that protect the metal surface against effect of the acid.

3.3.2. Enthalpy change (ΔH_a) and Entropy change (ΔS_a)

The transformation of Arrhenius equation gives rise to transition state equation. Enthalpy change (ΔH_a) and entropy change (ΔS_a) of activation process were evaluated from the transition state equation. The linear form of transition state equation is given in the equation below;

$$\ln \left(\frac{C_R}{T} \right) = \ln \left(\frac{R}{Nh} \right) + \left(\frac{\Delta S_a}{R} \right) - \left(\frac{\Delta H_a}{RT} \right)$$

Where h is the Planck's constant, N is Avogadro number, ΔS_{aa} is the entropy change of activation and ΔH_a is the enthalpy change of activation. The plot of $\ln (C_R/T)$ against $(1/T)$ gave a straight line with a slope of $(-\Delta H_a/R)$ and intercept of $(\ln R/Nh + \Delta S_a/R)$ from which the values of ΔH_a & ΔS_a were calculated and presented in Table 1 and figure 5. It can be seen that from the result the values of all enthalpies (ΔH_a) are positive and thus reflect the endothermic nature of the aluminium dissolution process [18]. The entropy of activation values in uninhibited and inhibited acid solution was large and negative. This implies that there is an association rather than dissociation in the activated complex of the rate determining step, indicating that a decrease in disorder take place, going from reactant to the activated complex [19].

Table 1: Activation Parameters for the Corrosion of Aluminium in H₃PO₄ Solution

Inhibitor Conc. (M)	E _a (kJ mol ⁻¹)	ΔH _a (kJmol ⁻¹)	-ΔS _a (JK ⁻¹ mol ⁻¹)
Blank	32.61	30.01	201.81
0.02	42.09	39.49	176.48
0.04	42.29	39.69	174.97
0.06	43.90	41.30	170.56
0.08	46.67	44.07	160.94
0.10	47.33	44.73	159.26

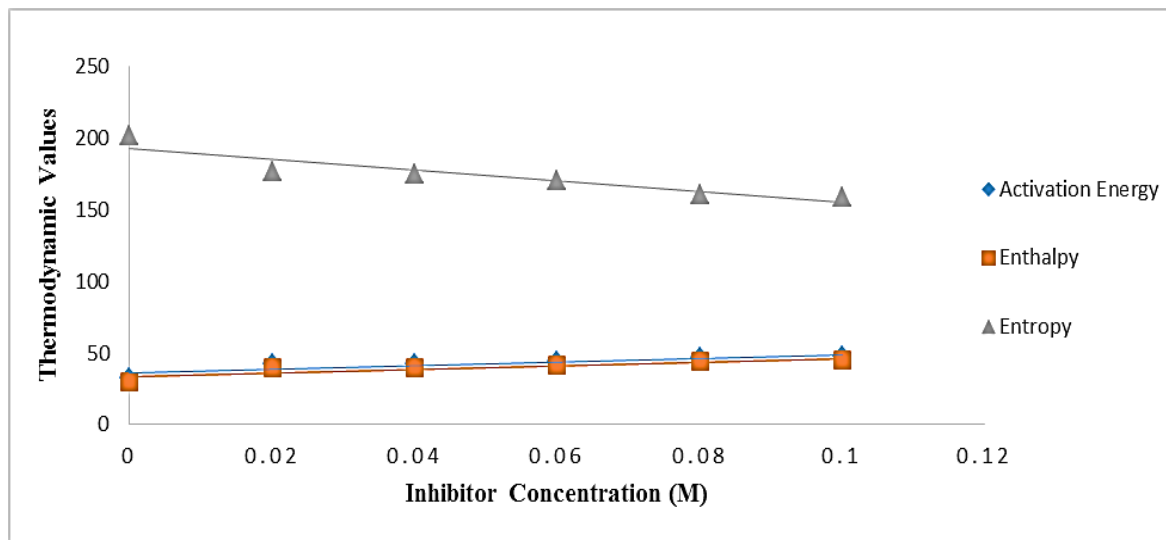


Figure 5. Variation of Activation energy, Enthalpy and Entropy values at different inhibitor concentration.

3.4. Adsorption Isotherm

One of the most convenient method of expressing adsorption quantitatively is deriving the adsorption isotherm that characterizes the inhibitor and metal surface. Adsorption isotherm is use to provide the basic information on the interaction between the metal surface and inhibitor molecule [20]. The degree of surface coverage (θ) values were evaluated at different concentrations of the inhibitor in H₃PO₄ solution. Various adsorption isotherms such as Temkin, Freundlich and Langmuir were applied to fit the surface coverage (θ) values, but the Langmuir adsorption isotherm was found to be the best fit among other tested isotherm. Langmuir adsorption isotherm can be expressed by the equation given below;

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \dots \dots \dots (7)$$

Where C_{inh} is the concentration of the inhibitor in the bulk electrolyte, θ is the degree of surface coverage, and

K_{ads} is the adsorption equilibrium constant. A plot of C_{inh}/θ against C_{inh} gave a straight line with slope equal to 1 and intercept equal to $1/K_{ads}$. The correlation coefficient (R^2) and adsorption equilibrium constant (K_{ads}) values were evaluated and presented in Table 2.

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

The adsorption equilibrium constant (K_{ads}) of the adsorption process is related to Gibbs free energy of adsorption (ΔG_{ads}) according to the equation given below;

$$\Delta G_{ads} = -RT \ln (55.5 \times K_{ads}) \dots \dots \dots (8)$$

From Equation 8, value of 55.55 is the concentration of water in the solution in mol/L. The calculated values of free energy of adsorption obtained by using equation 8 were all negative and range between 21.05 – 21.47 kJ/mol at 308 – 318 K as presented in Table 2. The negative values of ΔG_{ads} indicate the spontaneous adsorption of the inhibitor molecules on the aluminium surface [21]. The fact that ΔG_{ads} increases with the increase in temperature

indicates that the adsorption of inhibitor molecules on the surface of aluminium in H_3PO_4 are favored at high temperature and hence is considered to be predominantly physical adsorption.

Table 2. Adsorption Parameters Deduced from Langmuir Adsorption Isotherm for Corrosion Inhibition of Aluminium.

Temperature (K)	R^2	K_{ads}	ΔG_{ads} (kJ mol ⁻¹)
308	0.999	65.79	-21.05
313	0.998	62.50	-21.21
318	0.992	60.61	-21.47

3.6. Infrared Spectroscopy Analysis Results

Figures 6 and 7 shows the result of FT-IR analysis of the fresh inhibitor (glutaraldehyde) and the corrosion product of aluminum in H_3PO_4 solution with 0.1 M concentration of the inhibitor. The analysis of the inhibitor in Figure 6 shows C-H aldehyde bending frequency and C=O carbonyl stretching frequency. The analysis of the corrosion product presented in Figure. 7 shows the appearance of the corresponding functional group of the inhibitor at different wave number. The appearance of this functional group in the FT-IR analysis of the corrosion product suggests that the adsorption of the inhibitor take place through the functional group of the inhibitor molecules [22].

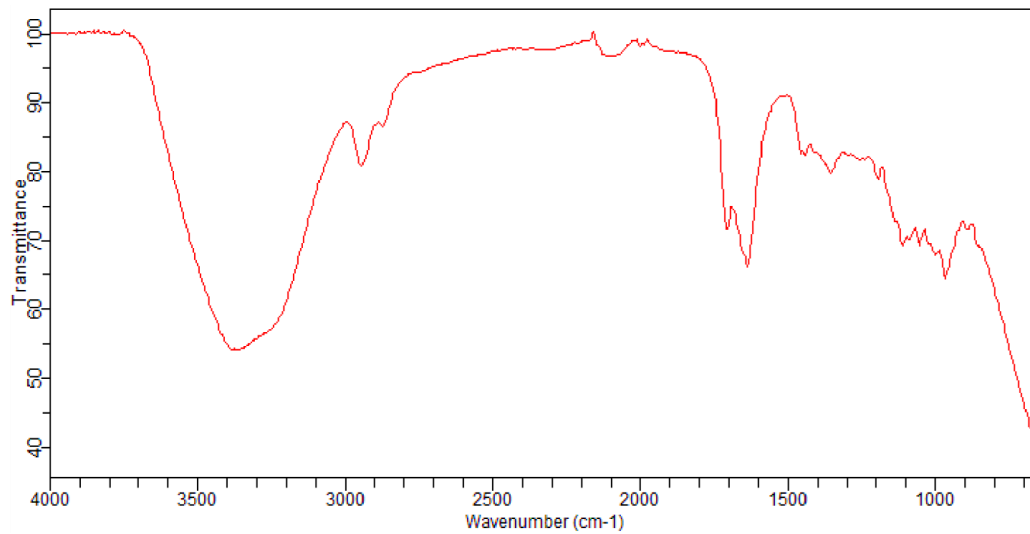


Figure 6. FT-IR Spectrum of glutaraldehyde.

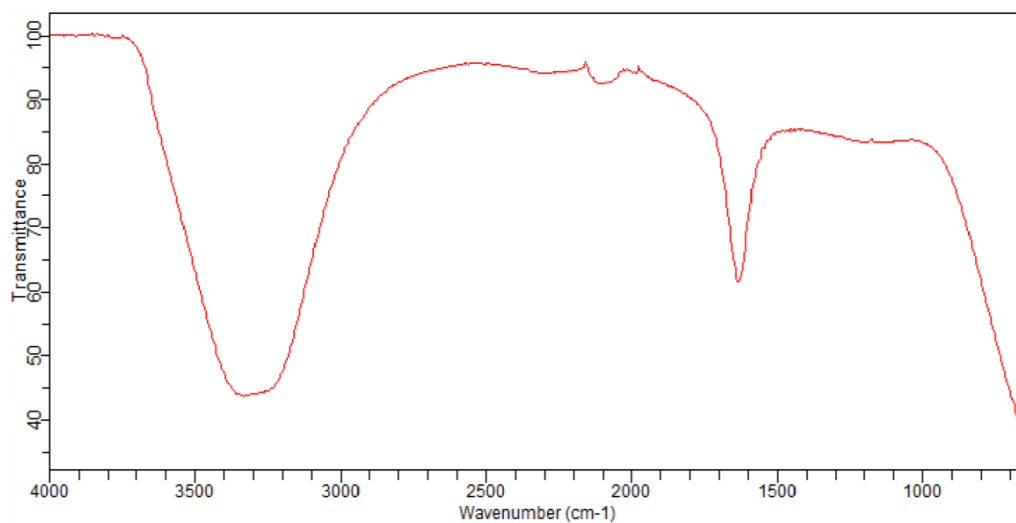


Figure 7. FT-IR Spectrum of Aluminum in 1.4 M H_3PO_4 with 0.1M Glutaraldehyde.

3.7. Scanning Electron Microscopy

The micrograph of aluminium without subjecting it to acidic environment is presented in Figure 8. The micrograph of aluminium immersed in 1.4 M H_3PO_4 was rough and highly damaged due to the attack of the aggressive acid as can be seen in figure 9. The presented Figure 10 shows how the surface of aluminium was

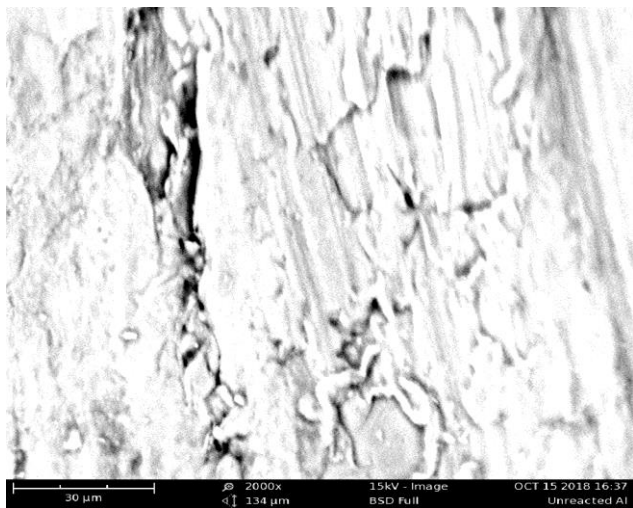


Figure 8. Micrograph of Unreacted Aluminium.

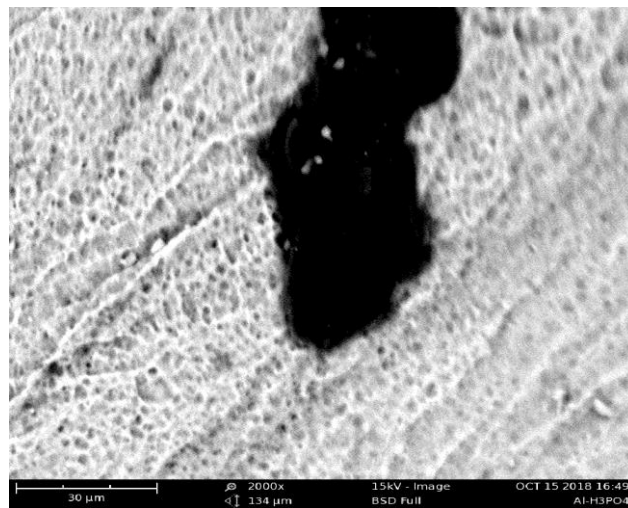


Figure 9. Micrograph of Aluminium in 1.4 M H_3PO_4 Solution.

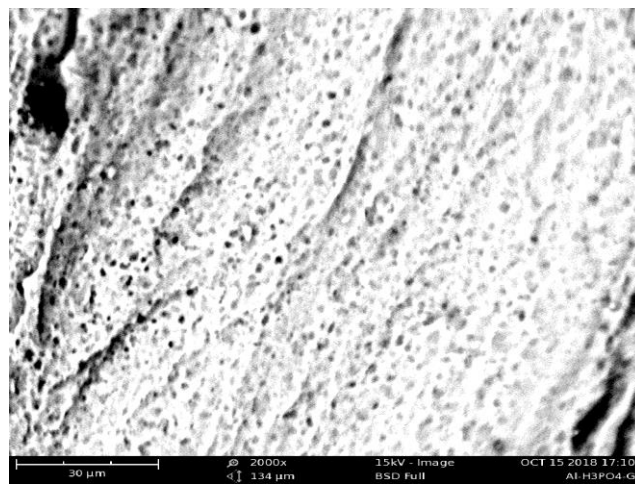


Figure 10. Micrograph of Aluminium in 1.4M H_3PO_4 solution with 0.1M Glutaraldehyde.

3.8. Protection Ability of the Inhibitor (Glutaraldehyde)

Glutaraldehyde known as 1,5-Pentaneione is an aldehyde organic compound, its structure contained two aldehyde functional group (-CHO) at first and fifth carbon. Those two functional group are the active materials in the compound which contained oxygen as the heteroatom and π -electrons. These two active functional group can easily supplies the lone pair of electrons on the surface of

aluminium to form a thin layer that served as a barrier between the metallic surface of aluminium and acidic environment which lead to the protection of the aluminium surface against the corrosive effect of the phosphoric acid.

3.9. Distinction of the Current Research and Recommendation on Feature Studies

The use of studied organic compound (glutaraldehyde) as corrosion inhibitor will be much significance as its nontoxic, friendly and commercially available. Though the

studied compound can compete with eco-friendly inhibitors. Therefore this research provided a knowledge by which the investigated organic compound will serve as corrosion inhibitor in both domestic and industrial purposes. The current research was only limited to weight loss method, so its recommended that the future research should contained other method of determining the corrosion process in order to get more information from the protecting ability of the inhibitor as well as properties and applications in various discipline.

4. Conclusion

The effect glutaraldehyde compound as corrosion inhibitor for aluminum in 1.4 M H_3PO_4 solution was studied. The corrosion rate increased with increase in temperature and decreased with increase in inhibitor concentration. The adsorption processes of the inhibitor molecules followed the Langmuir adsorption isotherm due to the linear graph obtained from the plot of C_{inh}/θ against C_{inh} and the values of correlation coefficients (R^2) that were all close to unity. The positive values of enthalpy of

activation (ΔH_a) in blank and inhibited acid solution confirmed the endothermic nature of the entire process. The negatives and large values of activation entropy (ΔS_a) shows that the entire process were entropically favorable. The calculated values of G_{ibbs} free energy of adsorption (ΔG_{ads}) were negative and their range indicated that the inhibitor was spontaneously absorbed on the aluminum surface through the mechanism of physical adsorption. The surface morphologies images obtained from scanning electron microscopic analysis were good proof for the reduction of dissolution of aluminum metal ascribed to the protective film formation on the metal surface. The appearance of functional group in the fourier transform infrared spectroscopic result of the corrosion product confirmed the formation of thin layer of inhibitor molecules on the aluminium surface.

Conflict of Interest

The authors declare that they have no conflict of interest

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