



Original Article

Organic compound as inhibitor for corrosion of aluminum in sulphuric acid solution

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ABSTRACT

The corrosion behavior of aluminum in H_2SO_4 solution was investigated by weight loss method in absence and presence of organic compound (glutaraldehyde) as inhibitor. Inhibition efficiency (I.E) was found to increase with increasing inhibitor concentration from 0.02 to 0.10 M and decrease as temperature increased. The activation energy value was 36.91 kJ/mol in uninhibited acid solution which increased to 61.80 kJ/mol in the presence of the inhibitor. The values of activation energy were found to increase with increase in inhibitor concentration due to the adsorption of more inhibitor molecules on the metal surface. The positive and negative values of enthalpy and entropy signified the endothermic adsorption process and formation of an ordered system. The negative value and low values of Gibbs's free energy of adsorption indicate the spontaneous and physical adsorption mechanism of the adsorption process. The adsorption of inhibitor on the metal surface was found to accord with Langmuir adsorption isotherm. Fourier transform infrared spectroscopic analysis, scanning electron microscopic analysis, surface and functional group characterization supported the inhibitive effect of the glutaraldehyde at the metal surface.

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1. Introduction

Aluminum is the second most abundant metal after iron used in different human activities and many of important applications. It has a low atomic mass and negative value of standard electron potential, aluminum potentially serves as an anodic material for power sources with high energy density. It is used in packing, construction and transportation. It's largely uses in electronics due to its electrical conductivity, strength and super purity [1]. Aluminum has an adhesive protective passive oxide film which is hard and tough the film is amphoteric and dissolved readily in acidic and alkaline solutions [2]. Therefore, study of aluminum corrosion in different acidic and alkaline medium has attracted considerable attention in view of its important application. H_2SO_4 is among the acids commonly used for pickling and etching purposes due to its low cost in comparison to other acids [3].

The cost of replacing metals due to corrosion is economically unbearable and often exorbitant. In making an effort to mitigate electrochemical corrosion of aluminum, the main strategy is to isolate the metal from

corrosive agents and this can be achieved by corrosion control. However, several options to control corrosion of metals has been adopted in industries, including anodic/cathodic protection, electroplating, painting and galvanizing, but the use of corrosion inhibitors has been emerged to be one of the most effective and practical method in combating corrosion problems [4].

Inhibitors are compounds that reduce the rate of corrosion of metals by being absorbed on the metal surface either through physical adsorption, which involve the transfer of charge from inhibitor molecule to charged metal surface, or by chemical adsorption which involve electron transfer from the inhibitor molecule to the vacant d-orbital of the metal [5].

Numerous investigations have been carried out to find effective inhibitors for aluminum corrosion in harsh environments and most of the well-known and suitable inhibitors are organic compounds [6]. Organic compounds with oxygen, sulfur, and/or nitrogen as polar groups and conjugated double bonds in their structures have been

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reported to be good corrosion inhibitors for many metals and alloys in corrosive media [7, 8]. The inhibiting action of these organic compounds is usually attributed to their interactions with the metallic surfaces via their adsorption. Polar functional groups are regarded as the reaction centers that stabilizes the adsorption process. However, the adsorption of an inhibitor on a metal surface depends on a few factors, such as the nature and surface charge of the metal, the adsorption mode, the inhibitor's chemical structure, and the type of the electrolyte solution [9].

This study was aimed to study the effect of organic compound (glutaraldehyde) as inhibitor for the corrosion of aluminum in H_2SO_4 solution, using weight loss method and characterization by Fourier transform infrared spectroscopic (FTIR) and scanning electron microscopic (SEM) analysis.

The choice of glutaraldehyde as inhibitor was on the basis of its effectiveness at wide range of temperatures, good solubility in water, compatibility with protected materials, low costs, relatively low toxicity and its simplicity to adsorb on the metal surface to form protective film which displace water and protect it against deteriorating [10]

2. Materials and Methods

2.1. Sample preparation

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

2.2. Preparation of solutions

A stock solution of analytical grade H_2SO_4 with percentage purity 97% and density 1.84 g/cm³ was prepared by using double distilled water. The acid solution of required concentrations of 1.4 M were prepared by appropriate dilution. Glutaraldehyde with percentage purity 50 % and density of 1.06 g/cm³ was used as the inhibitor. The concentrations of the inhibitor used for the study were 0.02, 0.04, 0.06, 0.08 and 0.1 M. Each of this concentration was diluted in the prepared desired concentrations of acid for use as test solution during weight loss experiment.

2.3. Weight loss measurement

In weight loss experiment, the prepared aluminum sample were each suspended completely in 1.4 M H_2SO_4 solution without and with different concentrations of inhibitor (glutaraldehyde) with the help of glass hooks at 308 K for 3 h. The volume of solution was kept 50 cm³, the sample was removed from the corrosive environment after 3 h, and they were then washed by distilled water, dried, reweighed and recorded. From the weight loss data which is the

average of triplicate measurement, the corrosion rate in mg/cm² was calculated.

2.4. Temperature effect

To study the effect of temperature on corrosion rate, the prepared aluminum samples were immersed completely in 50 cm³ of 1.4 M H_2SO_4 solution in the absence and presence of different inhibitor concentrations at 308, 313 and 318 K for 3 h.

2.5. Characterization

2.5.1. Fourier Transform Infrared Spectroscopic (FTIR) Analysis

Fourier transform infrared spectroscopic analysis was carried out for the fresh inhibitor and the aluminum coupon that was immersed in 1.4 M H_2SO_4 for 3 h immersion time in the presence of 0.1 M glutaraldehyde at 308 K. The coupon was retrieved, and scraped with a sharp razor blade. The scrap was collected and used as the corrosion product for the analysis. The analysis was done using Agilent Technology, Cary 630, fourier transform infrared spectrophotometer by scanning the sample through a wave number range of 650 – 4000 cm⁻¹; 32 scans at 8 cm⁻¹ resolution.

2.5.2. Scanning Electron Microscopic (SEM) Analysis

Surface morphologies of the aluminum samples before and after inhibition was studied using PRO: X: Phenom World 800-07334 model, manufactured by Phenom World Eindhoven, Netherlands. Scanned images of un-reacted aluminum sample, aluminum sample immersed in 50 cm³ of 1.4 M H_2SO_4 without inhibitor and aluminum sample immersed in 50 cm³ of 1.4 M H_2SO_4 in the presence of 0.1 M inhibitor at 308 K for 3 h were taken at an accelerating voltage of 15.00 kV and x500 magnification

3. Results and Discussion

3.1. Weight loss experiments

The weight loss and corrosion rate for the corrosion of aluminum in 1.4 M HCl solution without and with 0.02, 0.04, 0.06, 0.08 and 0.10 M concentration of the inhibitor for 3 h immersion time the weight loss (ΔW) was calculated according to Eq 1.

$$\Delta W = W_1 - W_2 \quad (1)$$

Where W_1 and W_2 are the weight of aluminum sample before and after immersion.

$$\text{Corrosion Rate (mg cm}^{-2}\text{h}^{-1}) = \frac{\text{weight loss } (\Delta W)}{\text{Area (A)} * \text{Time (t)}} \quad (2)$$

The degree of surface coverage (Θ) (Eq 3) and inhibition efficiency (I.E.) (Eq 4) for different inhibitor concentration was evaluated from weight loss experiments using the Equation 2.

$$\theta = \frac{W_i - W_f}{W_i} \quad (3)$$

$$I.E(\%) = \frac{W_i - W_f}{W_i} * 100 \quad (4)$$

Where W_i and W_f are the weight of the sample in the absence and presence of inhibitor.

3.2. Effect of inhibitor Concentration on Inhibition Efficiency and Corrosion Rate

The effect of inhibitor concentration on I.E for the corrosion of aluminum at 308 K was studied and presented in figure 1. The inhibition efficiency was found to increase with increase in concentration of the inhibitor. This trend was observed due to the increase in θ of adsorbed specie on the surface of aluminum as a result of increase in inhibitor concentration. Increase in inhibition efficiency with increase in inhibitor concentration indicated that more inhibitor molecules are adsorbed on the surface of the metal, thus providing wider surface coverage as reported by Olasehinde *et al.* [11]. Similar work was reported by Husaini *et al.* [12] in the study of inhibitive effect of glutaraldehyde on the corrosion of aluminum in hydrochloric acid solution and the result of inhibition efficiency was found to increase with increase in concentration. Another similar work was also reported by Ladha *et al.* [13] during investigation of cumin (*cuminum cyminum*) extract as an eco-friendly green corrosion inhibitor for pure aluminum in acid medium and result of

the inhibition efficiency of aluminum was increased from 79.30 to 90.20 % in the presence of 0.04 and 0.13 % inhibitor concentration.

The variation of corrosion rate with inhibitor concentration for the corrosion of aluminum in H_2SO_4 solution at 308 K is shown in figure 2. It can be seen from the figure that there is decrease in corrosion rate with increase in inhibitor concentration. Inspection of the result further indicates that the protection ability of the inhibitor was concentration dependent. The decrease of corrosion rate in the presence of inhibitor indicate the inhibition ability of the compound as inhibitor. This shows that increase in inhibitor concentration lead to the increase in surface coverage as a result of the adsorbed inhibitor molecules on the surface of aluminum which provided a barrier film/layer on aluminum surface and reduced further corrosion. Similar work was reported by Husaini *et al.* [14] during the study of effect of aniline as corrosion inhibitor on the corrosion of aluminum in hydrochloric acid solution and the result of corrosion rate was found to decrease with increase in inhibitor concentration.

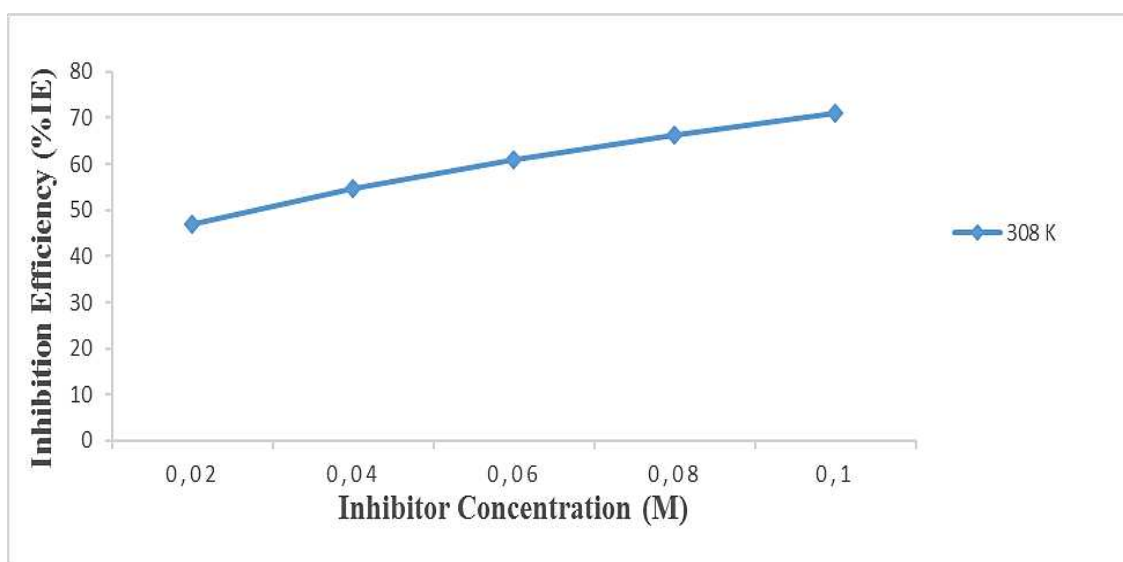


Fig. 1. Variation of Inhibition Efficiency with Inhibitor Concentration for Al Corrosion in H_2SO_4 Solution.

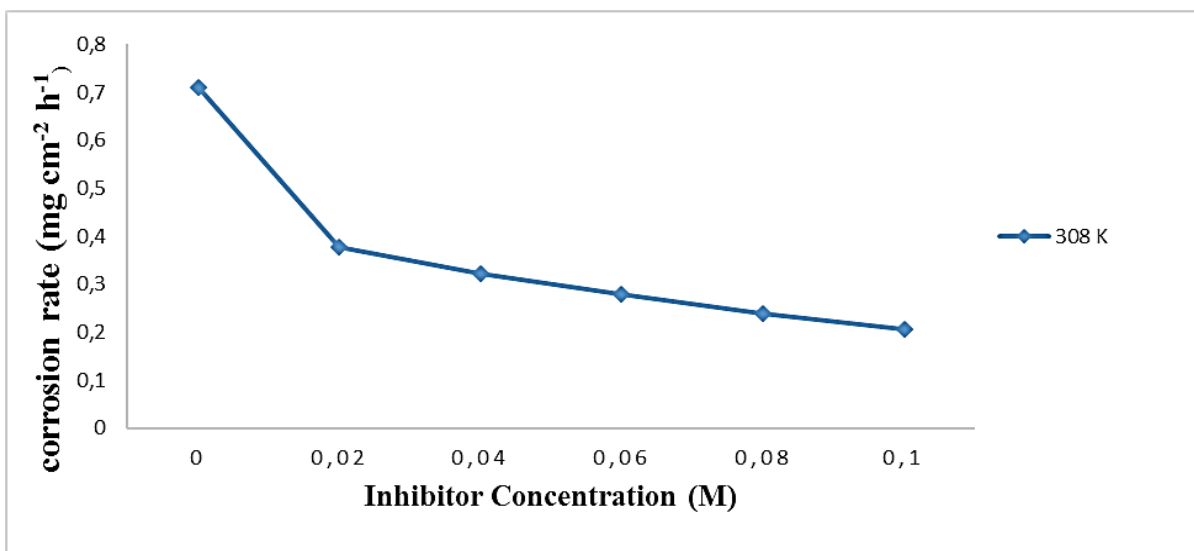


Fig. 2. Variation of Corrosion Rate with Inhibitor Concentration for Al Corrosion in H₂SO₄ Solution.

3.3. Effect of Temperature on Inhibition Efficiency and Corrosion Rate

The effect of temperature on the inhibition efficiency for the corrosion of aluminum in H₂SO₄ has been studied and the result is presented in figure 3. The result shows that inhibition efficiency decreases with increase in reaction temperature from 308 – 318 K. It is suggested that the variation of surface coverage is due to the desorption of inhibitor molecules on the aluminum surface as a result of rise in temperatures resulting in destabilizing the molecules of inhibitor molecule, this results in decreasing the efficiency of physical adsorption activity. Similar work was reported by Husaini *et al.* [15] during thermodynamic and kinetic study on the corrosion of aluminum in hydrochloric acid using benzaldehyde as corrosion inhibitor and the result of inhibition efficiency was found to decrease with increase in temperature.

Figure 4 shows the variation of corrosion rate with temperature for the corrosion inhibition of aluminum in acid solution at a given temperatures of 308, 313 and 318 K. From the result it can be seen that the rate of corrosion of aluminum in absence and presence of different inhibitor concentration increases with increase in temperature. Observation of the result shows that the highest corrosion rate was obtained at highest temperature. This observation is due to the fact that rate of chemical reaction generally increases with rise in temperature. Increase in temperature lead to increase in the average kinetic energy possessed by the reacting molecules thereby making the molecules to overcome the energy barrier faster [16]. Similar work was reported by Husaini *et al.* [17] during the study of corrosion inhibition of aluminum in nitric acid solution using Anisaldehyde (4 – methoxy benzaldehyde) as inhibitor and the result of the corrosion was found to increase with increase in temperature.

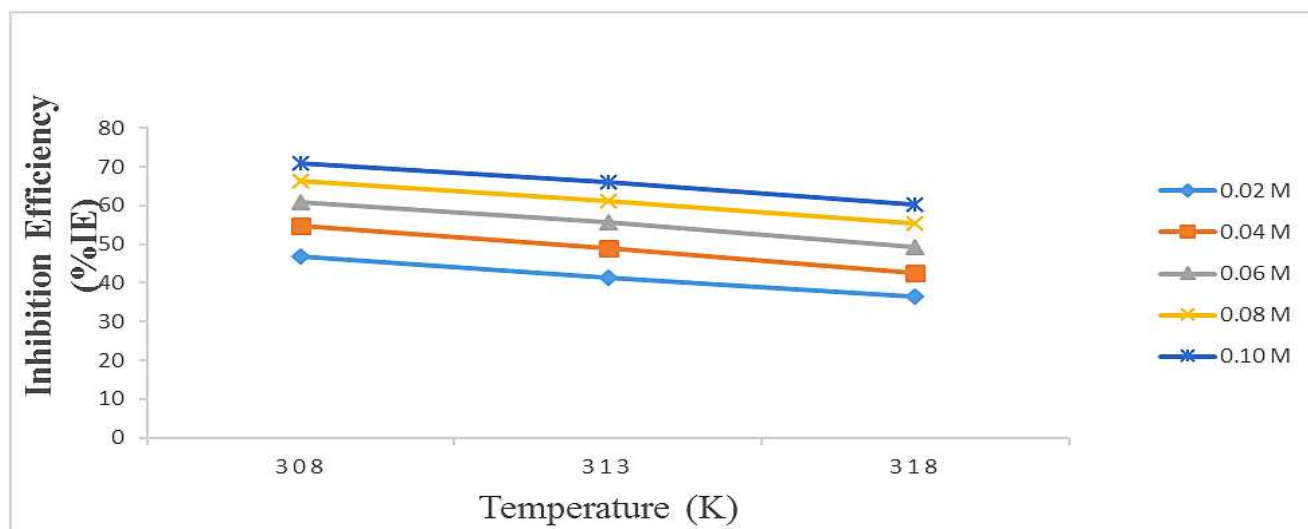


Fig. 3. Variation of Inhibition Efficiency with Temperature for Al Corrosion in H₂SO₄ Solution

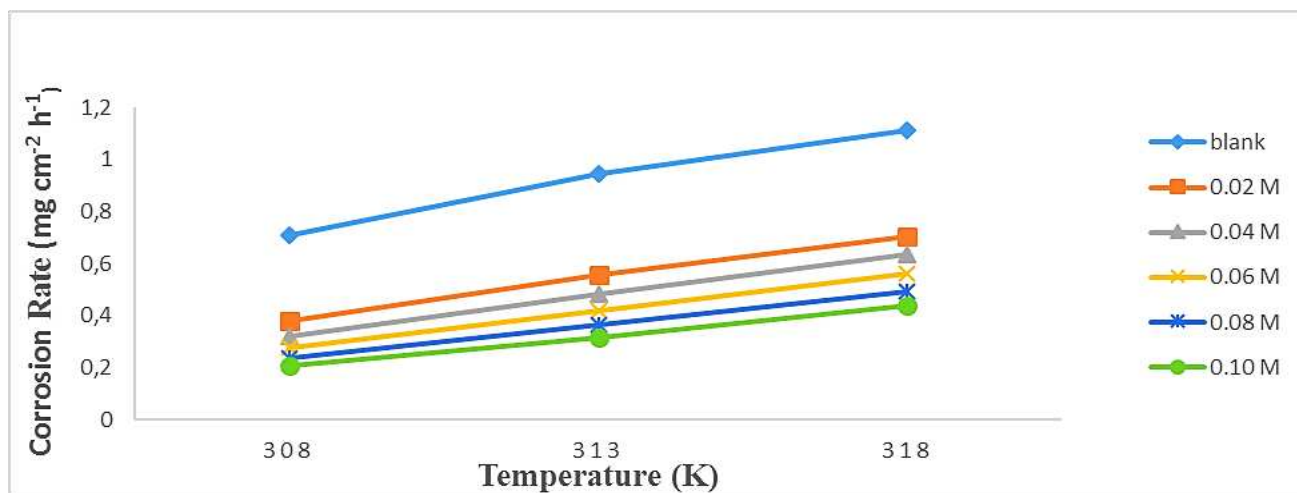


Fig. 4. Variation of Corrosion Rate with Temperature for Al Corrosion in H₂SO₄ Solution.

3.4. Activation Parameters

3.4.1. Activation Energy (E_a)

Elucidation of the inhibitive properties of the inhibitor and the temperature dependence on the corrosion rates requires the evaluation of activation energy (E_a) for the corrosion process in the absence and presence of the inhibitor. The activation energy was calculated from Arrhenius-type plot according to the equation given below.

$$\ln(C_R) = \ln B - \frac{E_a}{RT} \quad (5)$$

From the above equation, E_a is the apparent activation energy in kJ mol⁻¹, R is the universal gas constant, T is the absolute temperature and B is a constant. Values of apparent activation energy for the corrosion of aluminum in blank and inhibited H₂SO₄ with various concentrations of the inhibitor are shown in Table 3.1. These values were obtained from the slope of lnC_R versus 1/T plots. Activation energy values were found to be 36.39 kJ mol⁻¹ in blank acid solution which was increased to 61.80 kJ mol⁻¹ in the presence of highest inhibitor concentration (0.1 M), this shows that the activation energy values in the presence of inhibitor is higher than that for blank acid solution, suggesting that there was a slow dissolution of aluminum in the presence of the inhibitor [18, 19]. It is known that from Arrhenius equation the higher values of activation energy lead to the lower corrosion rate, as a result of film formation on the aluminum surface serving as a protective barrier that reduces the aluminum corrosion.

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

An alternative 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 linear form of transition state given below: [20].

$$\ln\left(\frac{C_R}{T}\right) = \ln\left(\frac{R}{Nh}\right) + \left(\frac{\Delta S_a}{R}\right) - \left(\frac{\Delta H_a}{RT}\right) \quad (6)$$

(6)

Where h is the Planck's constant, N is Avogadro number, ΔS_a 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 graph with a slope of (-ΔH_a/R) and intercept of (ln R/Nh + ΔS_a/R) from which the value of enthalpy change (ΔH_a) and entropy change (ΔS_a) of activation were calculated and presented in Table 3.1. It can be seen that from the result all the values of enthalpies are positive and the positive signs of the enthalpies prove the endothermic nature of the aluminum dissolution process [21]. The entropy of activation in the presence and absence of the inhibitor was large and negative. A knowledge of the entropy of activation, and hence the entropy of the adsorbed material, gives some indication of the extent to which the molecules are capable of unhindered translation on the surface. A negative value of ΔS_a suggests that the adsorption process involves an association rather than dissociation mechanism [22]. The adsorption leads to order by the formation of an activated complex between the inhibitor and the metal surface. Furthermore the negative values of ΔS_a reflect that there is no significant change in the adsorbent internal structures during the adsorption process due to decreased in disorder at the solid/liquid interface during the adsorption process as reported by Saha and Shamik [23].

Table 3.1: Activation Parameters for the Corrosion of Aluminum in H₂SO₄ Solution

Inhibitor Conc. (M)	E _a (kJ mol ⁻¹)	ΔH _a (kJmol ⁻¹)	-ΔS _a (JK ⁻¹ mol ⁻¹)
Blank	36.39	33.78	195.58
0.02	50.92	48.32	153.61
0.04	55.08	52.47	141.46
0.06	57.29	54.69	135.55
0.08	59.28	56.68	130.31
0.10	61.80	59.20	123.40

3.5. Adsorption Isotherm

The most convenient way of expressing adsorption quantitatively is deriving the adsorption isotherm that characterizes the metal/inhibitor/ environment system. Basic information on the interaction between the inhibitor and the surface of the metal can be obtained by adsorption isotherm. The values of the degree of surface coverage (θ) were evaluated at different inhibitor concentrations in 1.4 M H_2SO_4 solution. Various adsorption isotherm models were applied to fit the degree of surface coverage (θ) values, but the best fit was found to be Langmuir adsorption isotherm. Langmuir adsorption can be expressed by the equation given below [24].

$$\frac{C}{\theta} = \frac{1}{K_{\text{ads}}} + C \quad (7)$$

Where C 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/θ produced a straight line graph with slope equals to 1 and intercept equal to $1/K_{\text{ads}}$. The correlation coefficient (R^2) values obtained are presented in Table 3.2.

Langmuir isotherm model describes the relationship between surface coverage of an adsorbed inhibitor molecule over the surface of its adsorbent at constant pressure. The model assumed a monolayer adsorption of the adsorbate at fixed number of definite localized adsorption sites. The molecules were assumed to be identical and equivalent with no lateral interaction or steric hindrance between them. All the sites on the adsorbent were also assumed to possess equal affinity for the adsorbate, and possess constant sorption activation energy and enthalpy.

The values of K_{ads} obtained from the reciprocal of the intercept on the ordinate of the Langmuir isotherm decreases with increase in temperature due to the desorption of adsorbed inhibitor molecules. The correlation coefficient (R^2) is another parameter that also use to determine the best fitting isotherm to the experimental data. In this study the values of R^2 in Langmuir isotherm were correspondingly found to be higher than the values in other tested isotherm. This suggests that Langmuir isotherm is the model that fit to the experimental data.

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

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

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

From equation 8, value of 55.55 is the concentration of water in the solution in mol/L. The values of free energy of adsorption calculated by using equation 8 are all negative as presented in Table 3.2. The negative values of ΔG_{ads} indicate the spontaneous adsorption of the inhibitor molecules on the aluminum surface. Generally, values of ΔG_{ads} around -20 kJ mol^{-1} or lower are consistent with physical adsorption (physisorption) mechanism, while those around -40 kJ mol^{-1} or higher involves chemical adsorption (chemisorption) mechanism. In the present study the highest value of free energy of adsorption obtained at 318 K is $-20.37 \text{ kJ mol}^{-1}$, considering this result the adsorption of the inhibitor on the aluminum is associated with physical adsorption (physisorption) mechanism i.e. it involved electrostatic interaction between the molecule of the inhibitor and the metal surface [25].

Table 3.2: Adsorption Parameters Deduced from Different Adsorption Isotherm for Corrosion Inhibition of Aluminum.

T (K)	Adsorption isotherms					
	Langmuir R^2	K_{ads}	ΔG (kJ mol^{-1})	Temkin R^2	Freundlich R^2	Frumkin R^2
308	0.997	45.24	-20.04	0.982	0.984	0.988
313	0.996	42.91	-20.23	0.979	0.986	0.986
318	0.998	39.98	-20.37	0.960	0.982	0.974

3.7. Role of Glutaraldehyde as Corrosion Inhibitor

Glutaraldehyde undergoes protonation in H_2SO_4 solution and the protonated molecule gets adsorbed on to the anodic side of aluminum via the pre-adsorbed SO_4^{2-} ions. Sulphate ions have a small degree of hydration and due to this, they first get adsorbed on the positively charge aluminum surface. The adsorption of sulphate ions creates an excess negative charge towards the solution side of the aluminum and favors a higher adsorption of the inhibitors cationic form. This results in electrostatic interaction between the negatively charged aluminum surface and the positively charged glutaraldehyde molecules forming a protective

adsorbed layer. The protonated glutaraldehyde molecule are also adsorbed at cationic sites of the aluminum in competition with hydrogen ions that are reduced to H_2 gas.

3.8 Infrared Spectroscopy Analysis Results

Figures 5 & 6 show the result of FT-IR analysis of the inhibitor (glutaraldehyde) and the corrosion product of aluminum in 1.4 M H_2SO_4 with 0.1 M inhibitor concentration. The analysis of the inhibitor in figure 5 shows C-H aldehyde bending frequency at 3361 cm^{-1} and C=O carbonyl stretching frequency at 1640 cm^{-1} . The

analysis of the corrosion product presented in figure 6 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 suggest that the adsorption of the

inhibitor take place through the functional group of the inhibitor molecules.

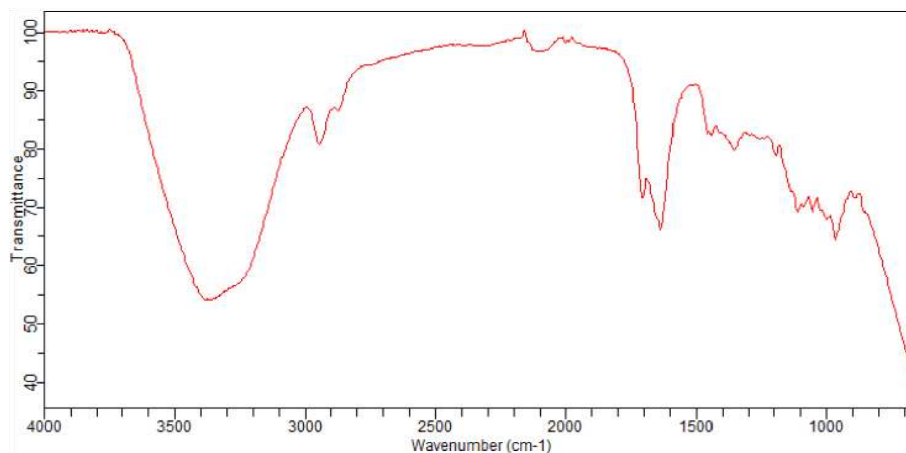


Fig. 5. FT-IR Spectrum of glutaraldehyde

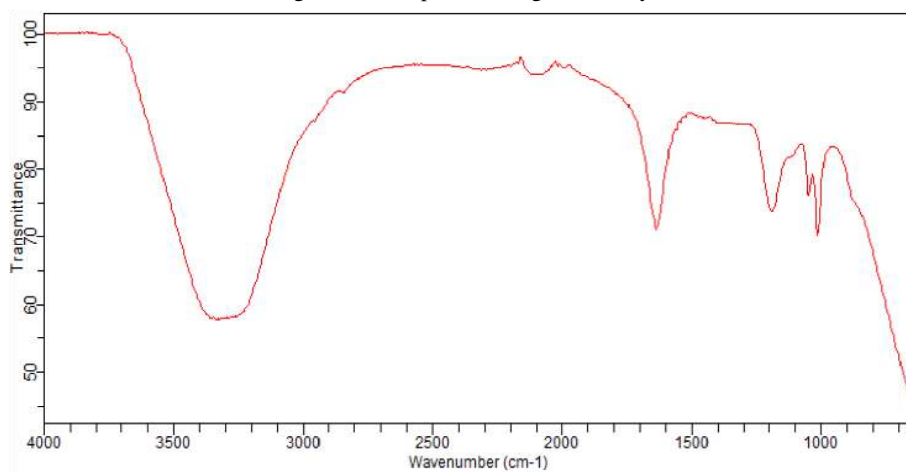


Fig. 6. FT-IR Spectrum of aluminum in 1.4 M H₂SO₄ with 0.1 M Glutaraldehyde

3.9. Scanning Electron Microscopy

Figure 7 presents the micrograph of fresh aluminum without subjecting it to acidic environment. Figure 8 shows that the aluminum immersed in 1.4 M H₂SO₄ was rough and highly damaged due to the attack of the aggressive acid. Figure 9 revealed how the aluminum surface was inhibited from H₂SO₄ attack by the presence of 0.1 M inhibitor. It can be concluded that the glutaraldehyde used, inhibited aluminum dissolution in H₂SO₄ by covering the surface area with protective film which was found absent in case of free acid interaction with aluminum.

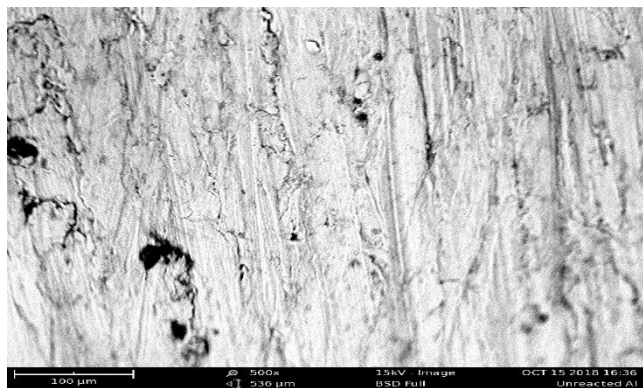


Fig. 7. Micrograph of Unreacted Aluminum.

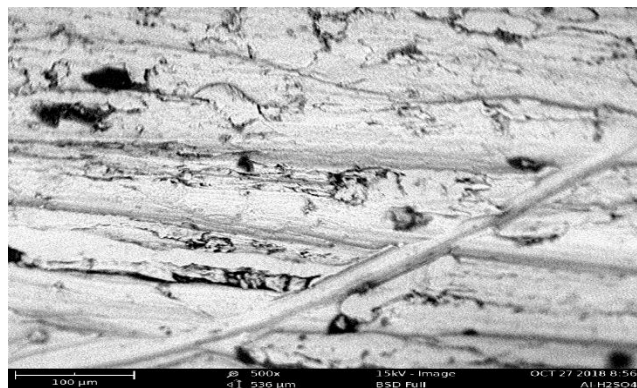


Fig. 8. Micrograph of Aluminum in 1.4 M H₂SO₄ Solution

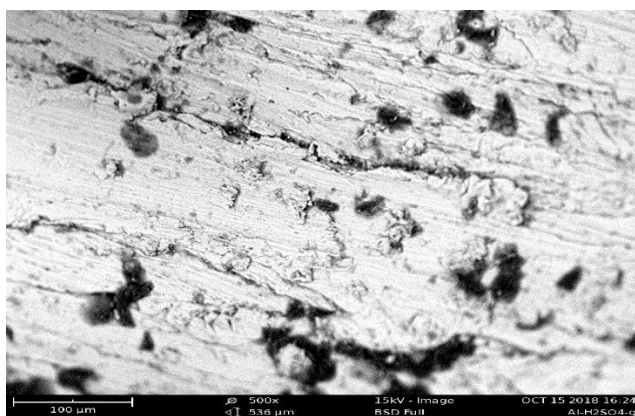


Fig. 9. Micrograph of Aluminum in 1.4 M H₂SO₄ with 0.1 M Glutaraldehyde.

4. Conclusion

The organic compounds act as effective and efficient inhibitor against aluminum corrosion in the acid solution at all studied temperatures with greatest efficiency at 308 K. Corrosion rate increases with increase in temperature, both in the blank and inhibited acid solution but decreased further significantly in the presence of the inhibitor. The corrosion inhibition is due to the adsorption of the inhibitor on to the aluminum surface and thus blocking the corrosion active sites of the aluminum by the physical adsorption mechanism. The adsorption of the inhibitor molecules on

the surface of aluminum was found to obey Langmuir adsorption isotherm model at all studied temperatures. The adsorption process is endothermic and spontaneous as inferred from positive values of enthalpy and negative values of free energy of adsorption. The negative values of entropy shows that there was an orderliness in the system as adsorption progressed.

Conflict of Interest

The authors declare that they have no conflict of interest.

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