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Inhibitive Effect of Glutaraldehyde on the Corrosion of Aluminum in Hydrochloric Acid Solution

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Abstract: The inhibition effect of glutaraldehyde on the corrosion of aluminum in 1.4 M HCl solution at 308-318 K was investigated by using weight loss method and characterized by Fourier Transform Infrared Spectroscopy (FTIR) & Scanning Electron Microscopic Analysis (SEM). The results show that the inhibition efficiency increases with increasing inhibitor concentration and decreases with rising temperature. The activation energy value was 20.48 kJ mol⁻¹ for the uninhibited acid solution which increased to 33.79 kJ mol⁻¹ in the presence of 0.1 M inhibitor concentration. Langmuir adsorption isotherm was found to provide an accurate description of the adsorption behavior of the investigated inhibitor. The thermodynamic parameters such as adsorption equilibrium constant (K_{ads}), free energy of adsorption (ΔG_{ads}), heat of adsorption (ΔH_{ads}) and entropy of adsorption (ΔS_{ads}) have been calculated and discussed in detail.

Keywords: Aluminum, glutaraldehyde, weight loss, FTIR analysis, SEM analysis, thermodynamic parameters, adsorption isotherm.

1. Introduction

Corrosion is obviously a multidisciplinary field of science and engineering. Corrosion is a natural process, which can be considered as either chemical or electrochemical in nature. It is an inevitable and serious problem for many industries. Corrosion phenomenon degrades the properties of metal/alloys and renders them to be rejected from the special structures of industrial importance. Most industries utilize metals and their ores (such as aluminum, zinc, mild steel, and copper) in the fabrication of their installations. In most cases, these metals are prone to corrosion when exposed to aggressive medium/media (Fouda, 2000). Since metals have a high electric conductivity, corrosion is usually electrochemical process that gradually returns the metal to its natural state in the environment. Acid solutions are extensively used in a variety of industrial processes such as oil well acidizing, acid pickling and acidic cleaning, which generally lead to serious metallic corrosion (Yadav *et al.*, 2011).

Aluminum owes its widespread use after steel, due to its excellent corrosion resistance to the air formed film strongly bonded to its surface. This film is actually stable in aqueous solutions over a pH range of 4–8.5 (Cabot *et al.*, 1991). Acid solutions mainly HCl and H₂SO₄ are generally used for the removal of undesirable scale and rust in several industrial processes. The effect of Cl⁻ ions (which can be generated by hydrolysis of HCl) on the corrosion of aluminum and its alloys has been the subject of several studies (Lin and Chiu, 2005). For this reason, some sorts of inhibitors are generally used to control metal dissolution as well as acid consumption. Most of the well-known acid inhibitors are organic compounds containing polar nitrogen, oxygen or sulfur atoms in a conjugated system have particularly been reported to exhibit corrosion inhibiting efficacy (Vrsalovic *et al.*, 2005; Ekpe *et al.*, 2001; Maayta *et al.*, 2004).

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Inhibition of metal corrosion by organic compounds occur as a result of adsorption of organic molecules or ions forming a protective layer on the metal surface. This layer prevents or reduces the corrosion of the metal. The extent of adsorption depends on the nature of the metal, the condition of the metallic surface, the mode of adsorption, the type of corrosive environment, and the chemical structure of the inhibitor (Arora *et al.*, 2007).

The aim of the present work was to examine the inhibitive action of glutaraldehyde as inhibitor towards the corrosion of aluminum in hydrochloric acid solutions. The choice of the inhibitors was based on the presence of donor atoms and π -electrons in the inhibitor molecule that induce greater adsorption of the compounds on the surface of aluminum which can lead to effective inhibition. The weight loss method was used in this study as its quantitative and possibly most accurate method in monitoring and measuring corrosion of metallic structures.

2. Experimental

2.1 Sample Preparation

Aluminum sheet with purity of 99.95% obtained from Metal Focus Fabrication Technology Incubation Centre Kano State, Nigeria was used in the current study. The sheet was 0.1 cm in thickness and was mechanically press cut into 3 cm \times 2 cm coupons. The coupons were polished with different grade of emery paper. After polishing, the samples were degreased by washing with ethanol, dried in acetone and preserved in a desiccator.

2.2 Solutions Preparation

A stock solution of analytical grade hydrochloric acid, HCl (36.5%, 1.18g/cm³), was prepared by using double distilled water. The acid solutions of required concentrations of 1.4 M was prepared by appropriate dilutions. The used inhibitor was glutaraldehyde (50 %, 1.06 g/cm³), and 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 concentrations of acids for use as test solutions in weight loss experiment.

2.3 Weight Loss Measurement

During weight loss experiment, the prepared aluminum coupons were each suspended completely in 1.4 M HCl solutions without and with different concentrations of glutaraldehyde with the help of glass hooks at 308 K for 3 h. The volume of solution was kept at 50 cm³. The coupons were retrieved after 3 h, washed by distilled water, dried well, reweighed and recorded. From the weight loss data, corrosion rate in mg/cm² was calculated.

2.4 Temperature Effect

To study the effect of temperature on corrosion rate, the prepared aluminum coupons were completely immersed in 50 cm³ of 1.4 M HCl solution without and with different concentrations of glutaraldehyde at 308, 313 and 318 K for 3 h.

2.5 Characterization

2.5.1 Fourier Transform Infrared Spectroscopic Analysis

Fourier Transform Infrared Spectroscopic Analysis (FTIR) was carried out for the fresh inhibitor and that of the corrosion product obtained from the reaction of aluminum immersed in 1.4 M HCl for 3 h immersion time in the presence of 0.1 M glutaraldehyde at 308 K using Agilent Technology, FTIR (Cary 630) Spectrophotometer. The analysis was conducted at 650 – 4000 cm⁻¹ wave number to scan the samples.

2.5.2 Scanning Electron Microscopy

Surface morphologies of the aluminum coupons before and after inhibition was studied with Scanning Electron Microscopy (SEM) using PRO: X: Phenom World 800-07334 model, manufactured by Phenom World Eindhoven, Netherlands. Scanned images of un-reacted aluminum coupon, aluminum coupon immersed in 50 cm³ of 1.4 M HCl without inhibitor and aluminum coupons immersed in 50 cm³ of 1.4 M HCl 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 Experiment

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 was calculated from the weight loss data using the equations given below;

$$\Delta W = W_1 - W_2 \tag{1}$$

$$\text{Corrosion Rate (mg cm}^{-2}\text{h}^{-1}\text{)} = \frac{\text{Weight loss (g)}}{\text{Metal surface area (cm}^2\text{)} \times \text{Time (h)}} \tag{2}$$

The degree of surface coverage (θ) and inhibition efficiency (I.E.) for different concentrations of the inhibitor in acidic media had been evaluated from weight loss experiment using the equations below (Singh and Singh 2010). Where W_i and W_f are the weight of the coupon in the absence and presence of inhibitor.

$$\theta = \frac{W_i - W_f}{W_i} \tag{3}$$

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

3.2 Effect of inhibitor on Inhibition Efficiency and Corrosion Rate

The effect of inhibition efficiency (% I.E) of the inhibitor for the corrosion of aluminum at different temperature was studied and the inhibition efficiency was found to increase with increasing inhibitor concentration. This trend was observed due to increase in surface coverage (θ) of adsorbed species on the surface of the metal as a result of increase in inhibitor concentration. Increase in inhibition efficiency with increasing inhibitor concentration indicates that more inhibitor molecules are adsorbed on the metal surface thus providing wider surface coverage as reported by Fouda *et al.*, (2014). Similar work was reported by Ladha *et al.*, (2013) during investigation of cumin (*cuminum cyminum*) extract as an eco-friendly green corrosion inhibitor for pure aluminum in acidic medium and result for the inhibition efficiency was found to increase with increase in inhibitor concentration.

Fig. 1 shows the variation of corrosion rate with inhibitor concentration for the corrosion of aluminum in HCl solution at different temperature. It can be seen from the figure that the corrosion rate decreases with increase in inhibitor concentration at all given temperatures. 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 reflects the inhibition action of the compound as inhibitor. This shows that increase in inhibitor concentration leads to the increase in surface coverage as a result of the adsorbed molecules of the inhibitor on the aluminum surface which provides a barrier (film) and reduces further corrosion. Similar result was reported by Vashi and Prajapati (2017) during corrosion inhibition of aluminum in HCl using *Bacopa monnieri* leaves extract as green inhibitor and the result of the corrosion rate was found to decrease with increase in inhibitor concentration.

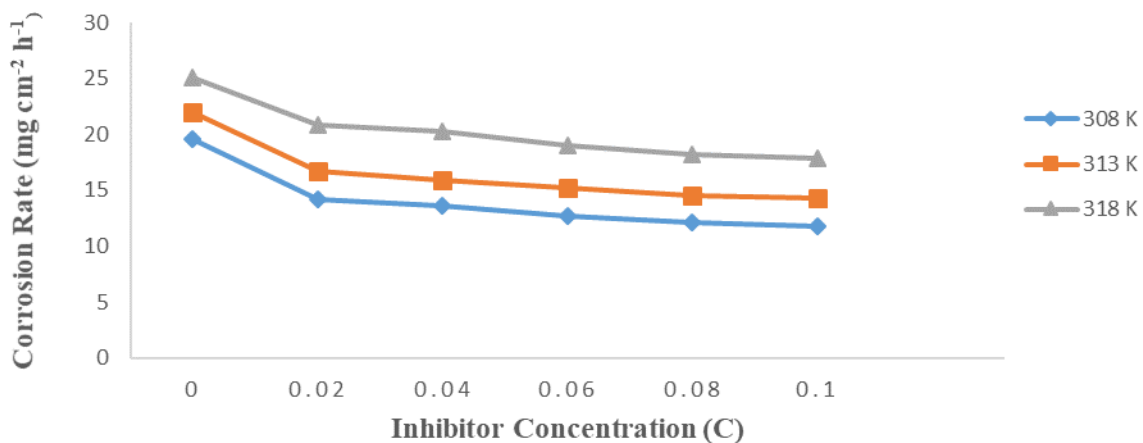


Fig. 1 - Variation of Corrosion Rate with Inhibitor Concentration for Al Corrosion in 1.4 M HCl.

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 acidic environment has been studied. The result shows that as the reaction temperature increases from 308-318 K, the inhibition efficiency decreases. It is suggested that the variation of surface coverage is due to the desorption of this inhibitor molecules on the surface of aluminum as a result of rise in temperature resulting in destabilizing the inhibitor molecule, which reduces the efficiency of physical adsorption activity. Similar result was reported by Jonathan *et al.* (2015).

Fig.2 shows the variation of corrosion rate with temperature for the corrosion inhibition of aluminum in acid solutions at a given temperature of 308, 313 and 318 K. From the result, the rate of corrosion of aluminum in inhibited and uninhibited acid solutions increases with increase in temperature. Observation of the result shows that the highest corrosion rate was at highest temperature (318 K). This observation is due to the fact that chemical reaction rates generally increases with increase in 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 (Ekpe *et al.* 2004; Musa *et al.*, 2018).

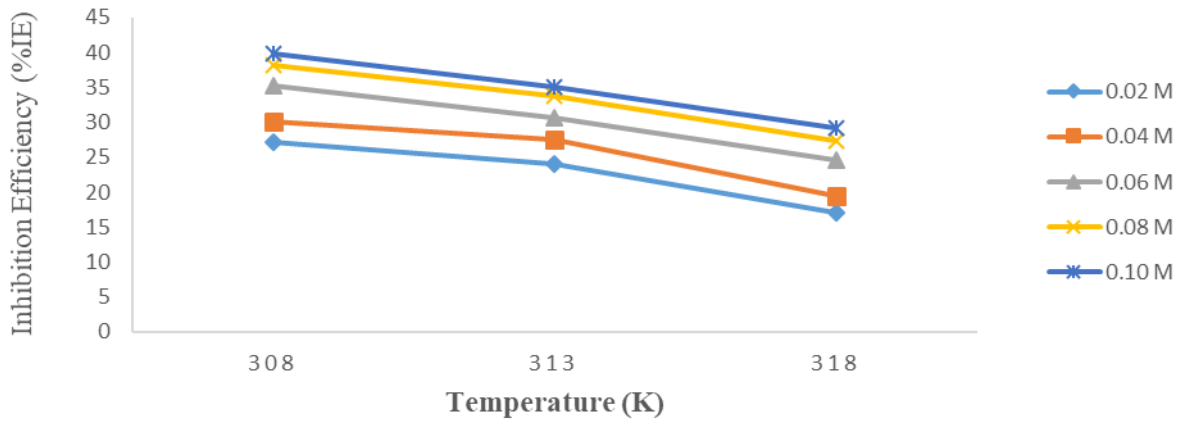


Fig. 2 - Variation of Inhibition Efficiency with Temperature for Al Corrosion in 1.4 M HCl.

3.4 Thermodynamic Study

3.4.1 Determination of Activation Energy (E_a)

Elucidation for the inhibitive properties of the inhibitor and the dependence of the temperature on the corrosion rates requires the activation energy (E_a) for the corrosion process in the absence and presence of the studied inhibitor which was evaluated from Arrhenius-type plot according to the equation given below (Eddy *et al.*, 2009).

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

From equation 5, E_a is the apparent activation energy, 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 1.4 M HCl without and with various concentrations of inhibitor are shown in Table 1 (refer Section 3.4.2). These values were determined from the slope of $\ln C_R$ versus $1/T$ plots. Activation energy values were found to be 20.48 kJ mol⁻¹ in uninhibited acid solution which was increased to 33.79 kJ mol⁻¹ in the presence of highest inhibitor concentration (0.1 M). This shows that the activation energy values for inhibited solution is higher than that for uninhibited solution, suggesting that dissolution of aluminum is slow in the presence of the inhibitor. From Arrhenius equation it is known that the higher values of activation energy leads to the lower corrosion rate. This is due to the formation of a film on the aluminum surface serving as an energy barrier for the aluminum corrosion.

3.4.2 Enthalpy change (ΔH_{ads}) and Entropy change (ΔS_{ads})

Transition state equation is an alternative formulation of Arrhenius equation. Enthalpy change (ΔH_{ads}) and entropy change (ΔS_{ads}) of activation process were evaluated from the transition state equation (Ahmed *et al.*, 2011).

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

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 $(-\Delta H/R)$ and intercept of $(\ln R/Nh + \Delta S_{ads}/R)$ from which the value of ΔH_{ads} & ΔS_{ads} are calculated and presented in Table 1. From the result, all values of enthalpies (ΔH_{ads}) are positive. The positive signs of the enthalpies (ΔH_{ads}) confirmed the endothermic nature of the aluminum dissolution process. The entropy of adsorption in presence and absence of the inhibitor was large and negative. A knowledge of the entropy of adsorption, 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_{ads} suggests that the adsorption process involves an associative mechanism. The adsorption leads to order through the formation of an activated complex between the adsorbate and adsorbent. Also, a negative value of ΔS_{ads} reflects that no significant change occurs in the internal structures of the adsorbent during the adsorption process.

Similar work was reported by Zarrouk *et al.*, (2013) during the evaluation of N-containing organic compound as corrosion inhibitor for carbon steel in phosphoric acid and the result of enthalpy was positive ranging from 81.84 to 87.94 kJmol⁻¹ in 0.5 to 5 mM inhibitor concentration while that of entropy was large and negative.

Table 1 - Activation Parameters for the Corrosion of Aluminium in 1.4 M HCl.

Inhibitor Conc. (M)	E_a (kJ mol ⁻¹)	ΔH_{ads} (kJmol ⁻¹)	$-\Delta S_{ads}$ (JK ⁻¹ mol ⁻¹)
Blank	20.48	17.88	219.86
0.02	30.95	28.35	188.56
0.04	32.06	29.46	185.34
0.06	32.91	30.31	183.14
0.08	33.69	31.09	181.01
0.10	33.79	31.19	180.89

3.5 Adsorption Isotherm

Deriving the adsorption isotherm that characterizes the metal/inhibitor/environment system is one of the most convenient ways of expressing adsorption quantitatively. Adsorption isotherm provides the basic information on the interaction between the inhibitor and the metal surface. The values of the degree of surface coverage (θ) were evaluated at different concentrations of the inhibitor in 1.4 M HCl solution. Various adsorption isotherms were applied to fit surface coverage (θ) values, but the best fit was found to obey Langmuir adsorption isotherm. Langmuir adsorption isotherm may be expressed by equation given below (Macfarlane *et al.*, 2009).

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

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 gave a straight line with slope equals to 1 and intercept equal to $1/K_{ads}$. The correlation coefficient (R^2) values obtained are presented in Table 2 (refer Section 3.6).

Langmuir isotherm model explains the relationship between surface coverage of an adsorbed 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 were correspondingly found to be higher than the values in another 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 (Samide *et al.*, 2014).

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

The values of free energy of adsorption calculated by using equation 8 are all negative as presented in Table 2. The negative values of ΔG_{ads} indicates 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, while those around -40 kJ mol^{-1} or higher involves chemical adsorption. In the present study, the highest value of free energy of adsorption obtained at 318 K is $19.08 \text{ kJ mol}^{-1}$. Based on this result, the adsorption of the inhibitor on the aluminum is associated with physical adsorption mechanism (physisorption) i.e. it involved electrostatic interaction between the inhibitor molecule and the metal surface.

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

Temperature (K)	R ²	K _{ads}	ΔG (kJ mol ⁻¹)
308	0.9923	26.13	-18.65
313	0.9946	24.87	-18.81
318	0.9777	24.57	-19.08

3.7 Role of Glutaraldehyde as Corrosion Inhibitor

Glutaraldehyde undergoes protonation in acid solution and the protonated molecule gets adsorbed on the anodic side of aluminum via the pre-adsorbed Cl⁻ ions. Chloride ions have a small degree of hydration and due to this, they get first adsorbed on the positively charge aluminum surface. The adsorption of chloride 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 molecule forming a protective adsorbed layer. The protonated glutaraldehyde molecules are also adsorbed at cationic sites of the aluminum in competition with hydrogen ions that are reduced to H₂ gas.

3.8 Infrared Spectroscopy Analysis Results f

Fig. 3 and 4 show the result of FTIR analysis of the inhibitor (glutaraldehyde) and the corrosion product of aluminum in 1.4 M HCl with 0.1 M inhibitor concentration. The analysis of the inhibitor in Fig. 3 shows C-H aldehyde bending frequency at 3361 cm^{-1} and C=O carbonyl stretching frequency at 1640 cm^{-1} . The shift in frequency has been observed in the analysis of the corrosion product presented in Fig.4. C-H bending shifted from 3361 to 3335 cm^{-1} and C=O stretching shifted from 1640 to 1637 cm^{-1} . The change in the adsorption band suggests that the adsorption between the inhibitor and aluminum take place through the functional group of the inhibitor molecule.

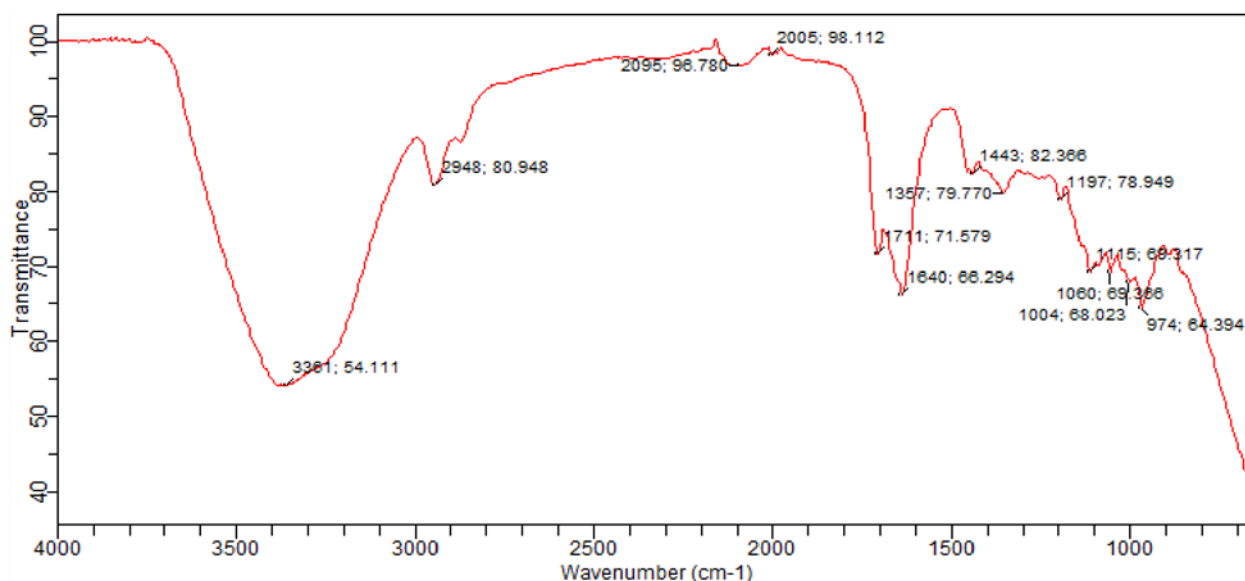


Fig. 3 - FTIR Spectra of Glutaraldehyde.

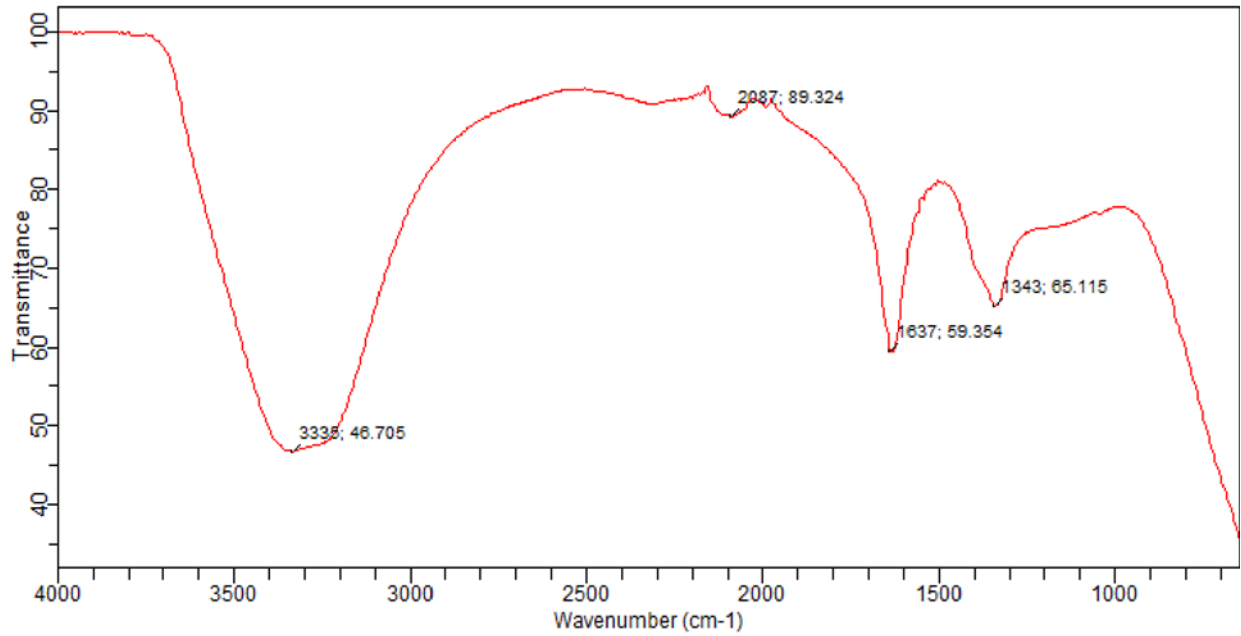


Fig. 4 - FTIR Spectra of Aluminium in 1.4 M HCl with 0.1 M Glutaraldehyde.

3.9 Scanning Electron Microscopy

Fig. 5 presents the micrograph of aluminum without subjecting it to acidic environment. Fig.6 shows that the aluminum immersed in 1.4 M HCl was rough and highly damaged due to the attack of the aggressive acid. Fig.7 reveals how the aluminum surface was inhibited from HCl attack by the presence of 0.1 M inhibitor. It can be concluded that when glutaraldehyde was used, it inhibited aluminum dissolution in HCl by covering the surface area with protective film which was found absent in case of free acid interaction with aluminum.

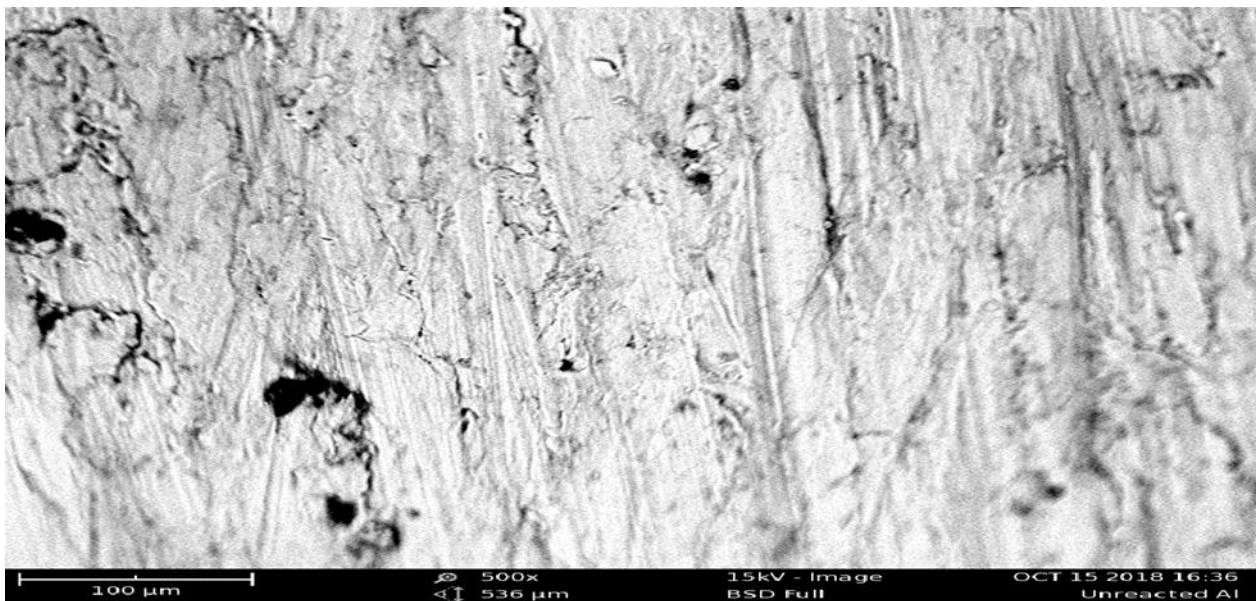


Fig. 5 - Micrograph of unreacted aluminum.

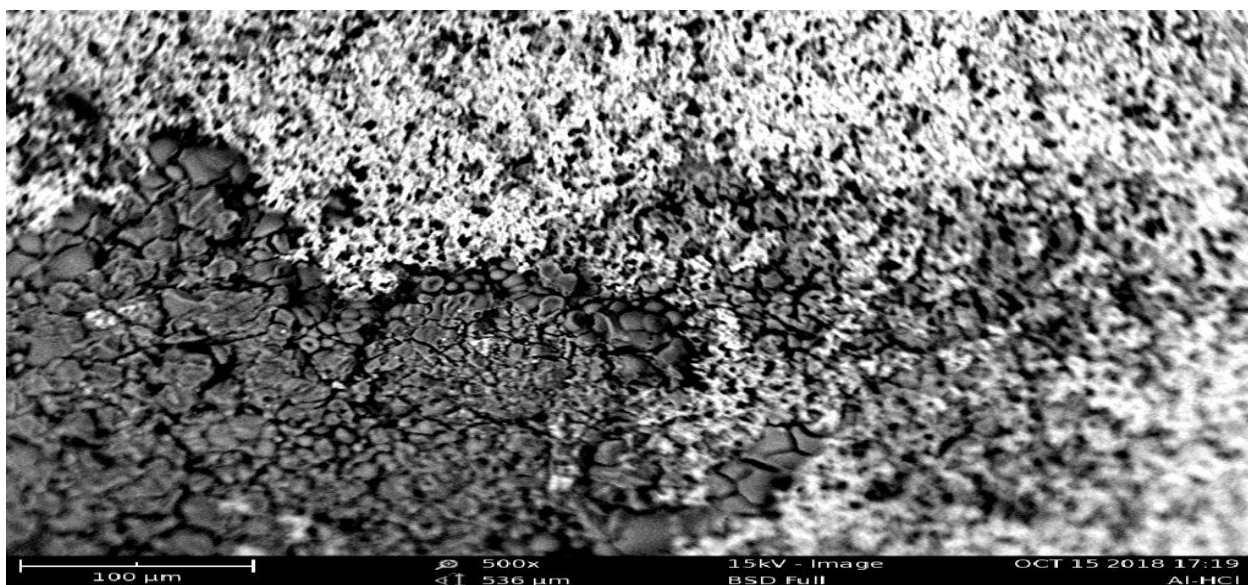


Fig. 6 - Micrograph of aluminum in 1.4 M HCl solution.



Fig. 7 - Micrograph of aluminum in 1.4 M HCl solution with 0.1 M glutaraldehyde.

4. Conclusion

The main conclusions drawn from these studies are; the inhibition efficiency increases with the increase in the inhibitor concentration, but decreases with increasing temperature for corrosion of aluminum in HCl solution. Thermodynamic parameters evidenced that the adsorption of the inhibitor on the surface of the aluminum is spontaneous and is consistent with the physical adsorption (physisorption) mechanism. The adsorption of different concentrations of the inhibitor on the surface of the aluminum followed Langmuir adsorption isotherm. The SEM analysis of the inhibited metal surface supported the adsorption of the inhibitor molecules on the surface of aluminum.

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