

Effect of Anisaldehyde as Corrosion Inhibitor for Aluminium in Sulphuric Acid Solution

Musa Husaini^{1*}

¹Department of Pure and Industrial Chemistry,
Faculty of Physical Sciences, College of Natural and Pharmaceutical Sciences,
Bayero University Kano (BUK), P.M.B. 3011, Kano, NIGERIA

*Corresponding Author

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Abstract: The inhibition effect of anisaldehyde on the corrosion of aluminium in sulphuric acid was investigated by weight loss method and characterized by Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) analyses. The effect of different inhibitor concentrations was studied at varying temperature (308, 313 and 318 K). It was found that the percentage of inhibition efficiency (% I.E.) increases with increase of inhibitor concentration and decreases with increase in temperature. The values of activation energy and half-life were found to be higher in inhibited acid solution compared to uninhibited acid solution, while that for the rate constant reduced from uninhibited to inhibited acid solution. The negative values of Gibbs free energy of adsorption confirmed the spontaneity of the process. Other thermodynamic parameters such as enthalpy and entropy were also evaluated and discussed. The investigation of adsorption isotherm model indicates that the inhibitor fitted to Langmuir isotherm model. Result of SEM analysis shows how the inhibitor reduced the acid attack on the aluminium surface, and FTIR analysis result of the corrosion product proves the formation of thin layer of inhibitor molecules on aluminium surface.

Keywords: Aluminium, anisaldehyde, corrosion, FT-IR, kinetic parameters, SEM, thermodynamic parameters

1. Introduction

Aluminium has been known to show widely different electrochemical properties in various aqueous electrolytes. Aluminium is widely used in many industrial applications such as chemical batteries, food containers, reaction vessels, and pipes [1, 2]. Aluminium has an outstanding economic, industrial usage and attractive material for engineering applications due to its high thermal and electrical conductivity, light weight and low cost. The resistance of aluminium against corrosion in aqueous environment can be attributed to a rapidly formed compact, strongly adherent invisible protective oxide layer on its surface [3, 4]. However, this protective oxide layer dissolved and consequently lead to rapid corrosion of aluminium at low or high pH values (usually less than 4 and above 9). Mineral acids and strong alkaline solutions are known to be very corrosive for aluminium. Acidic solutions are usually used for pickling, cleaning, chemical and electrochemical etching of aluminium [5, 6].

Corrosion is the slow destruction of materials, normally metals, by chemical reaction with its medium especially in corrosive environment. When it occurs in materials other than metals such as polymers or ceramics, the term degradation is usually employed. Corrosion destroyed the useful properties of materials and structures like appearance, strength and

*Corresponding author: musahusaini36@gmail.com

their ability to contain vessel's contents; therefore, control of aluminium corrosion in acidic environment has great importance [7]. It is imperative to monitor the interaction of the metal with the environment since corrosion lead to deterioration of metallic substances upon reaction with the environment, especially in the presence of oxygen. Several methods have been used to prevent corrosion and the rates at which it propagates in view to improve the lifetime of metallic substances. Examples of such methods include changing interfacial potential, protective coating, changing the environment, and addition of inhibitors [8]. However, the use of inhibitors for the prevention of corrosion of metallic substances which are in contact with aggressive media is one of the acceptable and practical method to control corrosion [9].

Organic compounds containing functional groups and heteroatoms such as phosphorus, sulphur, nitrogen, and oxygen together with π electrons in their structures have been found to inhibit corrosion by forming an adsorptive film/layer on the metal surface to prevent direct attack of the corrosion agent on the metal. The aim of the present work is to determine the inhibitive effect of anisaldehyde on the corrosion of aluminium in sulphuric acid solution using weight loss method. Scanning Electron Microscopy and Fourier Transform Infrared Spectroscopy analyses were used as surface characterization techniques to ascertain the nature of the protective film.

Many researches have been done for the protection of metallic materials against corrosion effect through the inhibition process. Some researchers used different substances as anti-corrosive agent such as inorganic compounds, organic compounds, Schiff bases, polymers, plant extract, nano particles and so on. Several organic compounds have been widely reported as corrosion inhibitors of aluminium in acid solution such as aliphatic compound, aromatic compound and nitrogen heterocyclic molecules [10]. In this work, the anisaldehyde was used to prevent the corrosive effect of aluminium in sulphuric acid. Use of anisaldehyde as corrosion inhibitor served as a good contribution that improves the field of corrosion protection of metallic substances as there were not many studies reported earlier using the studied compound as corrosion inhibitor in any environmental condition.

Several methods have been reported in the previous studies for measuring the rate of corrosion of metallic substances in different environmental condition. These include weight loss method, thermometric method, titrimetric method, electrochemical impedance analysis, gravimetric method and potentiodynamic polarization measurement [11]. However, the weight method was used in this study as it is quantitative and possibly most accurate method in measuring and monitoring the corrosion rate of metallic substances.

2. Experimental

2.1 Sample Preparation

The sample of aluminium 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 this study. The metal sheet was press cut to 3 x 2 cm² with 0.1 cm thickness. The metal sheet was polished by emery paper of different grade. It was then degreased in ethanol, dried in acetone and kept in a desiccator prior to use.

2.2 Solutions Preparation

A stock solution of analytical grade sulphuric acid with purity (97%) and density (1.84 g/cm³), was used as the corrosive environment. The required concentration of 1.4 M sulphuric acid was prepared by using double distilled water. The inhibitor used was anisaldehyde with percentage purity (98%) and density (1.11 g/cm³). The 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 solution in weight loss experiment.

2.3 Weight Loss Measurement

During the weight loss experiment, the prepared weighed aluminium sample was immersed in 100 mL beaker containing 50 mL of 1.4 M H₂SO₄, in absence and in presence of various inhibitor concentrations (0.02, 0.04, 0.06, 0.08 & 0.1 M). The beaker was then placed in the thermostat water bath at 308 K. The coupon was removed from the beaker after 3 hours of immersion and dipped immediately in saturated solution of ammonium acetate to stop the corrosion reaction. It was then washed several times with distilled water, dried in acetone and then kept in a desiccator to cool at ambient temperature and re-weighed. The experiment was done in replicate. The temperature effect was studied at a temperature range of 308, 313 and 318 K. This is because 308 K was the room temperature of the environment. The weight loss of aluminium was calculated in grams as the difference of the initial weight and the weight after the corrosion process. The weight loss, which is an average of triplicate measurements was calculated by Eq. 1:

$$\text{Weight loss } (\Delta W) = W_i - W_f \quad (1)$$

where W_i and W_f are the initial weight and weight after retrieval in grams. The corrosion rate was calculated following Eq. 2:

$$\text{Corrosion rate } (C_R) = \frac{W_i - W_f}{At} \quad (2)$$

where A is surface area cm^2 and t is the immersion time in hours. The surface coverage was calculated using Eq. 3 as follows:

$$\text{Surface coverage } (\theta) = \frac{W_1 - W_2}{W_1} \quad (3)$$

where W_1 and W_2 are the weight losses in grams of aluminium coupon in the absence and presence of the inhibitor at the same temperature. The inhibition efficiency is given by Eq. 4:

$$\text{Inhibition efficiency } (I.E) = \frac{W_1 - W_2}{W_1} \times 100\% \quad (4)$$

2.4 Fourier Transform Infrared Spectroscopy Analysis

Fourier Transform Infrared Spectroscopy (FTIR) analysis was carried out for the fresh inhibitor and that of the corrosion product obtained from the reaction of aluminium immersed in 1.4 M H_2SO_4 solution for 3 hours immersion time with the addition of 0.1 M anisaldehyde at 308 K. The machine used was Agilent Technology, FTIR (Cary 630) Fourier Transform Infrared Spectrophotometer. During the analysis, $650 - 4000 \text{ cm}^{-1}$ wave number was used to scan the sample. The FTIR spectrophotometer was used as it scans the samples directly without any preparation, unlike conventional IR spectrophotometer which requires sample preparation before running the analysis.

2.5 Scanning Electron Microscopy Analysis

Surface morphologies of the aluminium coupons before and after inhibition process was studied using (PRO: X: Phenom World 800-07334) model, manufactured by Phenom World Eindhoven, Netherlands. Scanned images of unreacted aluminium coupon, aluminium coupon immersed in 50 cm^3 of 1.4 M H_2SO_4 without inhibitor and aluminium coupons immersed in 50 cm^3 of 1.4 M H_2SO_4 with addition of 0.1 M inhibitor at 308 K for 3 hours were taken at an accelerating voltage of 15.00 kV and $\times 500$ magnification.

3. Results and Discussion

3.1 Effect of Inhibitor Concentration on Corrosion Rate and Inhibition Efficiency

Fig. 1 shows the variation effect of inhibitor concentration for aluminium corrosion in sulphuric acid solution at different temperatures. The figure reveals that the rate of corrosion of aluminium in sulphuric acid decreases with increase of inhibitor concentration at all the studied temperatures. The corrosion rate value of uninhibited acid solution was higher than the values obtained in inhibited system which depicts the inhibitive effect of the inhibitor. Increase in the concentration of the inhibitor lead to the decrease in the interaction that occurs between surface of the metal and the corrosive media which also retards the corrosion rate [12]. Similar work was reported by Omotoma and Onukwuli [13] in the evaluation of pawpaw leaves extract as anti-corrosion agent for aluminium in hydrochloric acid medium, and the result of corrosion rate was found to decrease with increase in inhibitor concentration.

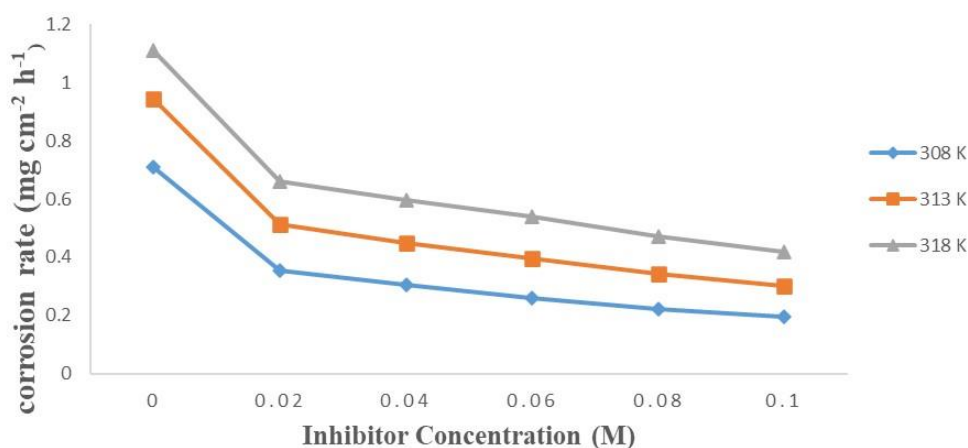


Fig. 1 - Plot of corrosion rate against inhibitor concentration for Al corrosion in sulphuric acid

The result presented in Fig. 2 shows the variation in the effect of inhibitor concentration against percentage inhibition efficiency (% I.E). The percentage inhibition efficiency (% I.E) was found to increase with increase in inhibitor concentration due to increase in the inhibition action of the compound as inhibitor. The increase in percentage inhibition efficiency (% I.E) suggests that more inhibitor molecules were adsorbed on the metal surface thus providing wider surface coverage [14]. Similar work was reported by Rao and Deepa [15] during the study of corrosion inhibition of 6063 aluminium alloy by *Coriandrum sativum L* seed extract in phosphoric acid medium, and the result of inhibition efficiency was found to increase with increase in inhibitor concentration.

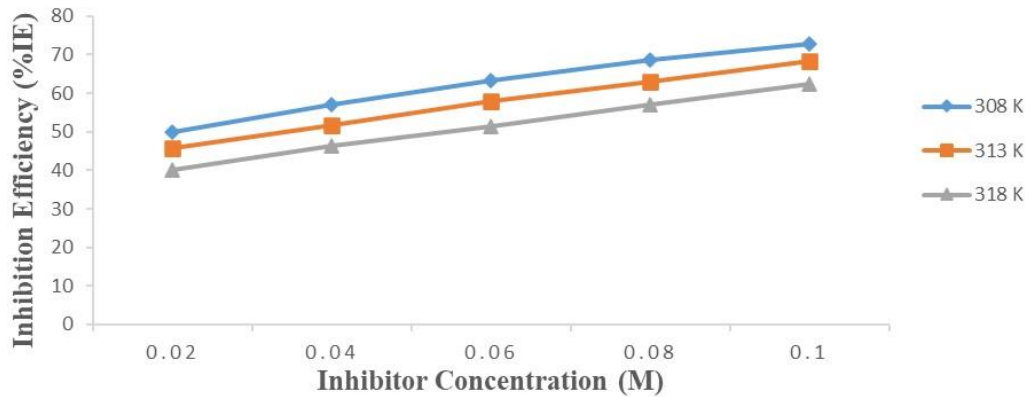


Fig. 2 - Plot of inhibition efficiency against inhibitor concentration for Al corrosion in sulphuric acid

3.2 Effect of Temperature on Corrosion Rate and Inhibition Efficiency

Fig. 3 shows the variation effect of corrosion rates of aluminium corrosion in blank and presence of various inhibitor concentrations at different temperatures. From the result, it can be seen that there is an increase in the rate of corrosion with increase in temperature for both inhibited and uninhibited acid solution. The highest corrosion rate of aluminium was obtained at the highest temperature as can be seen from the result. This is due to the fact that chemical reaction rates usually increase when the temperature increase. 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 and react faster [16, 17]. Similar work was reported by Olasehinde *et al.* [18] during the investigation of the inhibitive properties of *Alchornea laxiflora* leaves on the corrosion of mild steel in HCl and the result of corrosion rate was found to increase with increase in temperature.

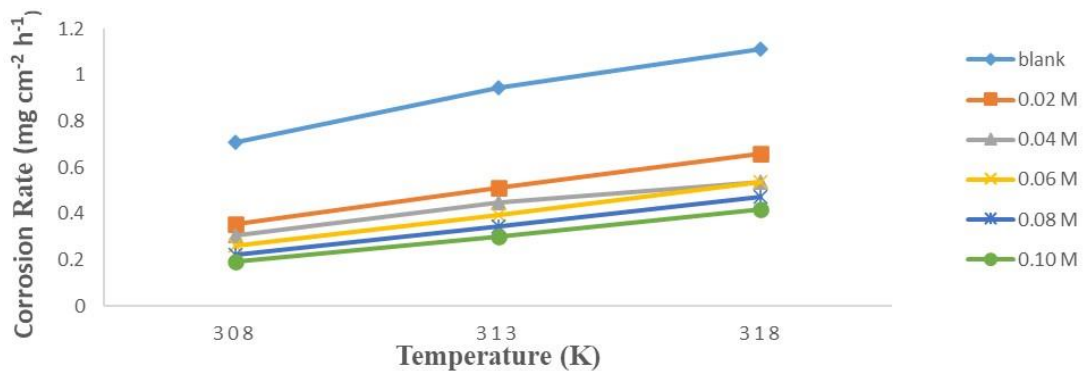


Fig. 3 - Plot of corrosion rate with temperature for Al corrosion in sulphuric acid

The variation effect of inhibition efficiency against temperature at different inhibitor concentration is presented in Fig. 4. The result shows that the inhibition efficiency decreases with increase in the temperature. This is due to the desorption of adsorbed molecules of the inhibitor from surface of aluminium because of increase in temperature resulting in destabilization of the inhibitor and that reduced the efficiency of physical adsorption. The difference between values of inhibition efficiency of the given inhibitor obtained at studied temperatures suggests that the adsorption mechanism on aluminium surface is consistent with physical adsorption (physisorption) mechanism in which the inhibition efficiency decreases with increasing the temperature of the reacting species [19, 20]. Similar work was reported by Eddy [21] during the study of ethanol extract of *Phyllanthus amarus* as a green inhibitor for the corrosion of mild steel in H₂SO₄ and the result of inhibition efficiency was found to decrease with increase in temperature.

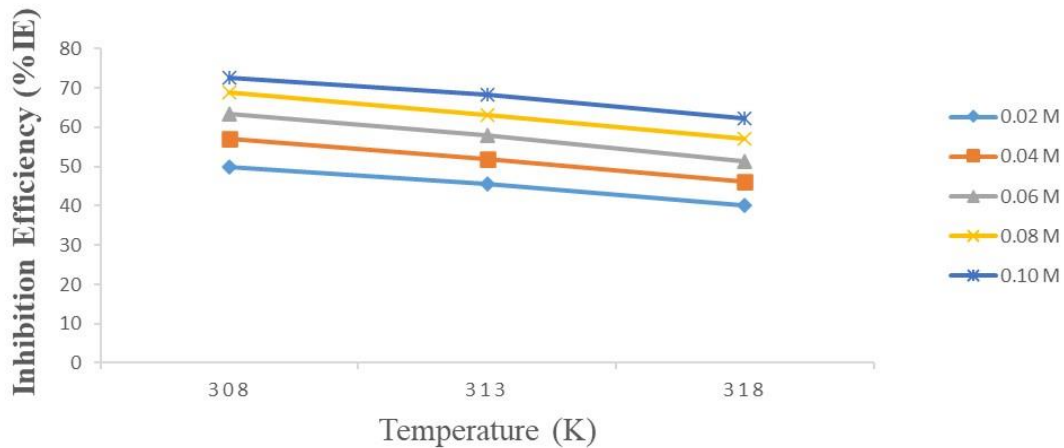


Fig. 4 - Plot of inhibition efficiency with temperature for Al corrosion in sulphuric acid

3.3 Kinetics Study

3.3.1 Activation Energy

The rate of many chemical reactions increases with temperature obeying Arrhenius equation. In the situation of electrochemical reactions, temperature favors the kinetics of corrosion process and more specifically, the dissolution of the metallic material at anodic site. The activation energy of the corrosion reaction can be evaluated from the Arrhenius plot according to the following Eq. 5:

$$C_R = A_{exp} \left(\frac{-E_a}{RT} \right) \quad (5)$$

The linear form of the above equation would yield Eq. 6:

$$\ln C_R = \ln A - \frac{E_a}{RT} \quad (6)$$

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 corrosion process, and T is the absolute temperature. The plot of $\ln(C_R)$ against reciprocal of absolute temperature ($1/T$) gave a straight line with slope = $-E_a/R$, from which the activation energy values of the corrosion process were obtained.

From Table 1, the values of activation energy in the presence of the inhibitor is higher than the activation energy value in the absence of inhibitor. This indicates 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 increase 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 [22, 23].

Table 1 - Kinetic Parameters for aluminium corrosion with and without various inhibitor concentrations

| Inhibitor Concentration (M) | Activation Energy (kJ mol ⁻¹) | Rate Const. (k ×10 ⁻³) (hour ⁻¹) | Half-life (10 ²) (hours) |
|-----------------------------|---|--|--------------------------------------|
| Blank | 36.39 | 5.81 | 1.19 |
| 0.02 | 46.28 | 2.14 | 3.23 |
| 0.04 | 50.54 | 1.83 | 3.76 |
| 0.06 | 59.04 | 1.57 | 4.41 |
| 0.08 | 61.42 | 1.33 | 5.18 |
| 0.10 | 62.02 | 1.16 | 5.92 |

3.3.2 Rate Constant

The kinetics of the corrosion reaction possess 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 Eq. 7.

$$\ln(W_i - W_L) = -k_1 t + \ln W_L \quad (7)$$

According to Eq. 7, the plots of $\ln(W_i - W_L)$ against time showed a linear variation as shown in Fig. 5 with correlation coefficients close to 1 which confirmed first - order kinetics for the corrosion of aluminium in inhibited and uninhibited acid solution. From Table 1, the value of rate constant (k_i) for the corrosion of aluminium was found to be higher in uninhibited acid than inhibited acid solution. This confirms the inhibition of aluminium corrosion in acid solution by the presence of the studied inhibitor.

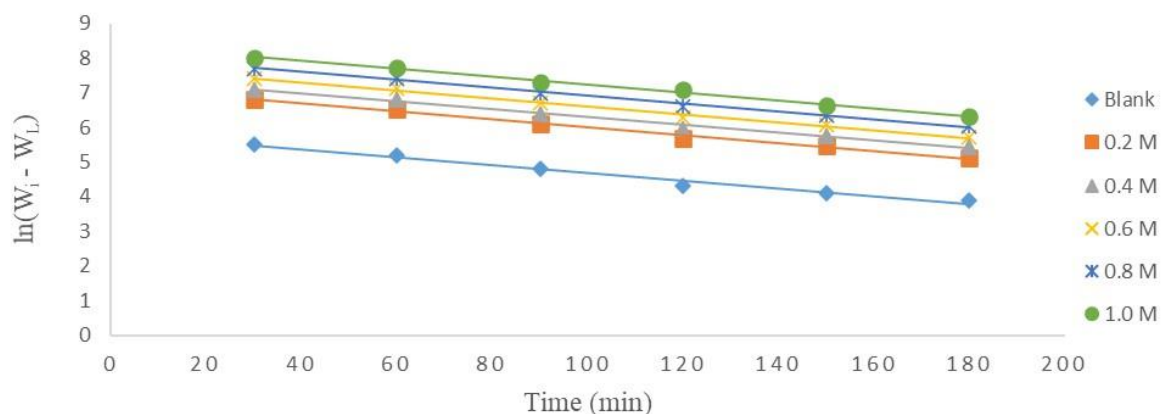


Fig. 5 - Plot of $\ln(W_i - W_L)$ against time for blank and at various inhibitor concentration

3.3.3 Half-life

The half - life ($t_{1/2}$) of the first order equation was calculated by using Eq. 8 as follows.

$$Half - life(t_{1/2}) = \frac{0.692}{K} \quad (8)$$

From Table 1, it can be seen that the value of the half-lives ($t_{1/2}$) increased from uninhibited acid to inhibited acid 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 acid solution. Higher half-life values in the presence of inhibitor is an indicative of protection ability of the studied inhibitor that reduces the rate of etching away of aluminium in sulphuric acid solution. It should also be noted that as inhibitor concentration increases the half-life also increases which results in a decrease in the corrosion rate, suggesting that more protection of the aluminium by the presence of higher inhibitor concentration has been established.

3.4 Thermodynamic Study

Enthalpy change (ΔH) and entropy change (ΔS) of reaction were calculated using the transition state equation. The linear form of transition state equation is given by Eq. 9:

$$\ln\left(\frac{C_R}{T}\right) = \ln\left(\frac{R}{T}\right) + \left(\frac{\Delta S}{R}\right) - \left(\frac{\Delta H}{RT}\right) \quad (9)$$

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 graph with slope $= -\Delta H/T$ and intercept $= \ln(R/Nh) + \Delta S/R$ from which the values of enthalpy and entropy were calculated.

From Table 2, the calculated values of enthalpy for the corrosion of aluminium in both inhibited and uninhibited acid solution are all positive indicating the endothermic nature of the reaction and 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} which proves that the adsorption of the inhibitor on the aluminium surface in sulphuric acid follows physical adsorption mechanism. The negative values of entropy confirmed that the corrosion process is 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 represents an association rather than a dissociation step as reported previously by Ating *et al.* [24].

Table 2 - Enthalpy and entropy change of the reaction process with various concentrations of the inhibitor

| Inhibitor Concentration (M) | ΔH_{ads} (kJ mol^{-1}) | $-\Delta S_{ads}$ ($\text{J mol}^{-1}\text{K}^{-1}$) |
|-----------------------------|---|--|
| blank | 33.78 | 195.58 |
| 0.02 | 43.68 | 155.40 |
| 0.04 | 47.94 | 170.35 |
| 0.06 | 56.43 | 130.41 |
| 0.08 | 58.82 | 123.96 |
| 0.10 | 59.49 | 122.9 |

3.4.1 Free Energy of Adsorption (ΔG_{ads})

The tendency for many chemical reactions to proceed, including the metal reaction with its environment is measured by Gibb's free energy change (ΔG). The more negative value of ΔG , it gives the greater tendency for the reaction to proceed. The free energy of adsorption (ΔG_{ads}) is related to adsorption equilibrium constant (K_{ads}) by the following Eq. 10:

$$\Delta G_{ads} = -RT \ln(55.5K_{ads}) \quad (10)$$

The value of 55.55 in the above equation is the concentration of water in the solution in mol/L. The values of free energy of adsorption are presented in Table 3. The calculated values of free energy of adsorption for the corrosion of aluminium in sulphuric acid in the presence of inhibitor are all negative. This is the fundamental criterion for spontaneity. The negative values are 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 as reported from the previous research of Loto *et al.* [25]. 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. The positively charge inhibitor molecule in the acid solution and negatively charge surface of aluminium were the charges of the molecules involved in this case, i.e. the positive and negative charges attracted each other electrostatically.

Table 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 | 51.81 | -20.39 |
| 313 | 0.994 | 50.02 | -20.63 |
| 318 | 0.997 | 48.98 | -20.90 |

3.5 Active Materials Present in the Inhibitor

Anisaldehyde known as 4-methoxybenzaldehyde is an aldehyde organic compound that contains an aldehyde functional group ($-\text{CHO}$) and methyl ($-\text{CH}_3$) substituent at the para position of the benzene ring. The active materials in the compound are the functional group itself, ($-\text{CHO}$) which contained oxygen as the heteroatom and π -electrons that are present in the benzene ring. These two active materials can easily supply the lone pair of electrons on the surface of the aluminium to form a thin layer that served as the barrier between the metallic surface of aluminium and acidic environment which leads to the protection of the aluminium surface against the corrosive effect of the acid.

3.6 Fourier Transform Infrared Spectroscopy Analysis Result

Fig. 6 shows the result of FTIR analysis of the fresh inhibitor (anisaldehyde) and the corrosion product of aluminium in sulphuric acid solution with 0.1 M concentration of the inhibitor. The analysis of the inhibitor (Fig. 6a) shows C-H aldehyde bending frequency, C-H aromatic bending frequency and C=O carbonyl stretching frequency. The analysis of the corrosion product (Fig. 6b) shows the appearance of the corresponding functional group of the inhibitor at different wave numbers. The appearance of these functional group in the FTIR analysis of the corrosion product suggests that the adsorption of the inhibitor takes place through the functional group of the inhibitor molecules.

Table 4 - Absorption bands of inhibitor molecule and corrosion product obtained from FTIR analysis

| Functional Group | Wave Number (cm ⁻¹) | |
|-------------------|---------------------------------|-------------------|
| | Inhibitor | Corrosion Product |
| -C=O | 1596 | 1640 |
| -OCH ₃ | 1180 | 1164 |
| -CH | 1257 | 1212 |
| -C=C | 1022 | 1052 |

The main differences from the FTIR analysis of the inhibitor and that of the corrosion product presented in Fig 6 and Table 4 is the changes observed in their absorption band. The -C=O, -OCH₃, -CH and -C=C stretching vibration were observed at 1596, 1180, 1257 and 1022 cm⁻¹ as presented in Fig. 6a which changed to 1640, 1164, 1212 and 1052 cm⁻¹ as presented in Fig. 6b. The shift and changes of the absorption band from the FTIR analysis of the inhibitor and that of the corrosion product is an indication of the adsorption of inhibitor on the aluminium surface.

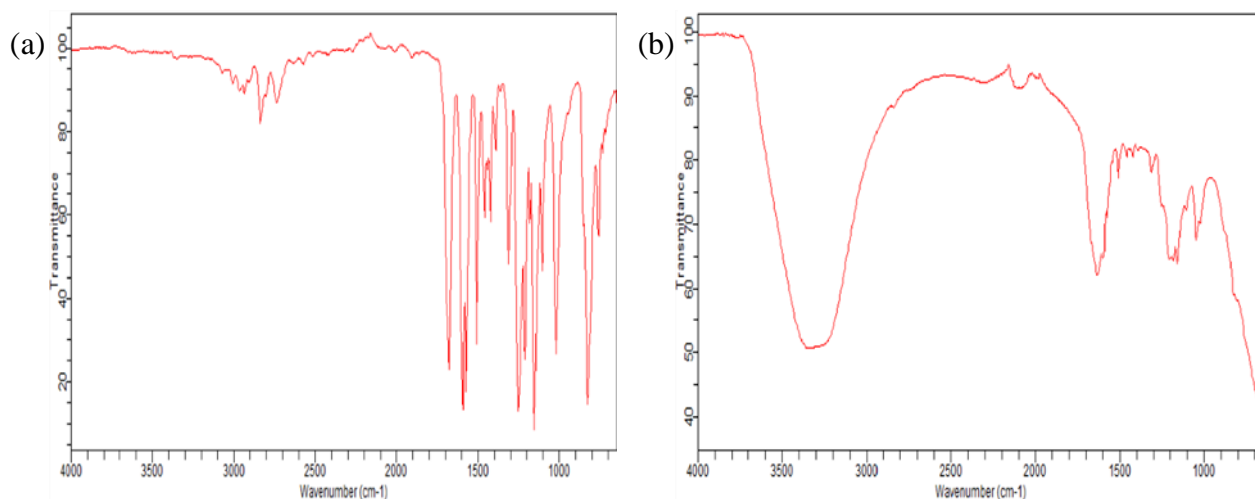


Fig. 6 - FTIR spectra of (a) anisaldehyde; (b) corrosion product

3.7 Scanning Electron Microscopy Analysis Result

Fig. 7a presents the micrograph of aluminium without subjecting it to acidic environment. Fig. 7b shows that the aluminium immersed in 1.4 M H₂SO₄ was rough and highly damaged due to the corrosion in the absence of the inhibitor. Fig. 7c reveals how the aluminium surface was inhibited from sulphuric acid attack by the presence of 0.1 M inhibitor as there is much smaller damage on the surface. This is attributed to the formation of a good protective film on the aluminium surface. It can be concluded that the anisaldehyde used inhibited aluminium dissolution in sulphuric acid by covering the surface area with protective film which was found absent in case of free acid interaction with aluminium.

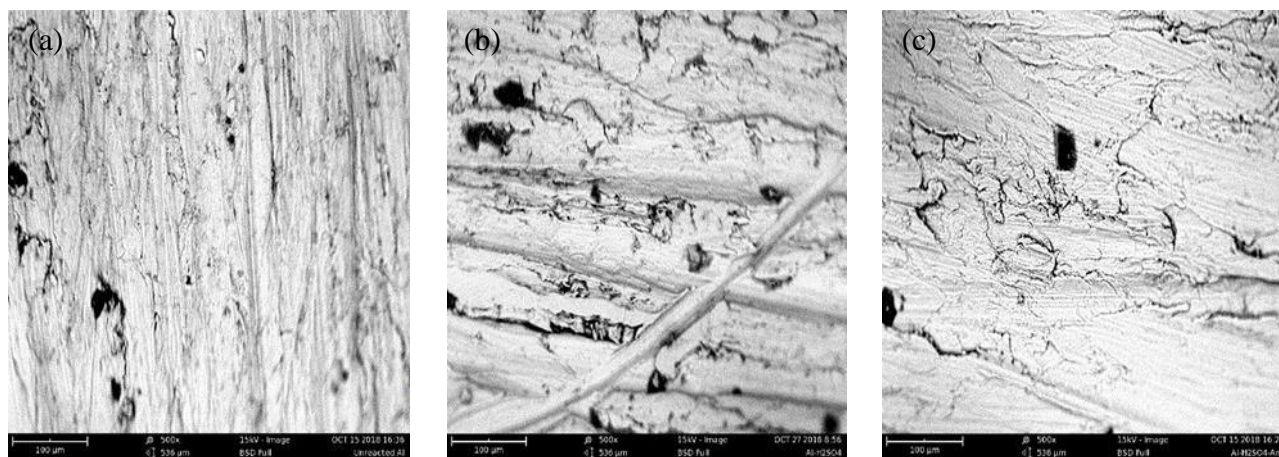


Fig. 7 - Micrograph at x500 of (a) unreacted aluminium; (b) Al in H₂SO₄ without inhibitor; (c) Al in H₂SO₄ with inhibitor

4. Conclusion

Results from the weight loss measurement show that; anisaldehyde served as an effective inhibitor for the corrosion of aluminium in sulphuric acid solution. Increase in temperature increases corrosion rate in both inhibited and uninhibited acid solution but decreases the inhibition efficiency. Inhibition efficiency was found to increase with increase in concentration of the inhibitor due to the adsorption of more inhibitor molecules on the metal surface. The positive values of enthalpy indicate the endothermic nature of the process. The negative values of entropy indicate that the activated complex in the rate determining step represents an association rather than a dissociation step. The negative values of Gibbs free energy of adsorption confirmed the spontaneity of the reaction. The adsorption of the inhibitor molecule on the aluminium surface was proposed to be physical adsorption by the values of enthalpy and Gibbs free energy of adsorption.

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