



Role of organic compound as corrosion inhibitor for aluminium in acidic solution

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Abstract

The inhibition action of aniline towards the corrosion behavior of aluminium in nitric acid has been studied using weight loss method and characterized by scanning electron microscopic (SEM) analysis. The compound was found to be good inhibitor for corrosion of aluminium in HNO_3 . The corrosion inhibition efficiency of the inhibitor increases with increase of the concentration of inhibitor and decreases with increase in temperature. The inhibitor showed the highest corrosion inhibition efficiency of 73.50, 70.65 and 66.66 % at 308, 313 and 318 K in the presence of 0.1 M inhibitor concentration. The activation energy increased for that of the uninhibited HNO_3 from $42.91 \text{ kJ mol}^{-1}$ to $52.74 \text{ kJ mol}^{-1}$ in the presence of 0.02 M inhibitor. The values of enthalpy are positive indicating the endothermic nature of the process. The entropy values are negative showing that the activated complex in the rate determining step represents an association, rather than dissociation. The adsorption process indicated feasible and spontaneous process from the large negative values of free energy of adsorption. The adsorption data were consistent with Langmuir adsorption isotherm. The results of SEM analysis showed that the inhibitor was adsorbed on the metal by forming a protective film over the surface as proposed by the kinetics and the thermodynamic data.

Keywords: Aluminium, Aniline, Corrosion, SEM, kinetic and Thermodynamic Parameters.

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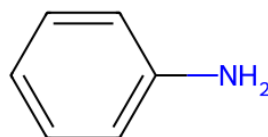
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1. Introduction

Corrosion is a natural degradation process that compromises the structural integrity and functionality of materials, particularly metals, when exposed to aggressive environments such as acids (Rosliza *et al.*, 2010; Zarrouk *et al.*, 2012 Ajanaku *et al.*, 2015; Arrousse *et al.*, 2021). Among the various metals susceptible to corrosion, aluminum stands out due to its widespread use in industries ranging from aerospace to automotive, owing to its lightweight and high strength-to-weight ratio (Paul and Koley 2016; Bazzi *et al.*, 2003). However, aluminum is prone to corrosion in acidic media, particularly in the presence of nitric acid, which is commonly encountered in many industrial processes (Mihit *et al.*, 2010; Zarrouk *et al.*, 2011; Ating *et al.*, 2012; Husaini *et al.*, 2018; Adams *et al.*, 2021).

In response to the challenge of metal corrosion, corrosion inhibitors have emerged as a critical line of defense. These substances are capable of reducing the rate of corrosion by forming a protective layer on the metal surface, preventing direct interaction between the metal and corrosive species (Fouda *et al.*, 2016; Husaini and Ibrahim, 2019; Hammouti *et al.*, 2011). One such class of inhibitors is organic compounds, which have shown significant promise due to their ability to adsorb onto metal surfaces, offering both chemical and physical protection (Husaini *et al.*, 2020a; A *et al.*, 2015; A *et al.*, 2015; Barouni *et al.*, 2014).

Aniline, an organic compound containing a primary amine group, has garnered attention as a potential corrosion inhibitor for aluminum in nitric acid solutions. The basicity and the nucleophilic nature of the amine group enable it to interact with the metal surface, forming complexes that hinder corrosion processes. Despite its potential, the efficiency and mechanism of aniline as a corrosion inhibitor for aluminum in nitric acid solution are not fully understood, necessitating further exploration (Husaini *et al.*, 2020a).



This paper aims to investigate the role of aniline as a corrosion inhibitor for aluminum in nitric acid solutions. Through an in-depth analysis of its adsorption behavior, effectiveness, and mechanism of inhibition, the study will contribute valuable insights into the application of aniline as a cost-effective and environmentally friendly solution for mitigating corrosion in acidic environments. Understanding these interactions is crucial for the development of more efficient and sustainable corrosion protection strategies for aluminum-based materials in industrial applications.

2. Materials and methods

2.1. Sample Preparation

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

2.2. Preparation of Solutions

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

2.3. Weight loss Measurement

The prepared weighed samples were immersed in 100 ml beaker containing the acid solution in the absence and presence of various inhibitor concentrations (0.02, 0.04, 0.06, 0.08 & 0.1 M) at 308 K and 3 hours immersion time, after which they were retrieved, washed, dried, re-weighed and recorded respectively. The experiments were performed in replicate. The effect of temperature was studied at a temperature range of 308, 313 and 318 K. The weight loss of aluminium was calculated in grams as the difference between the initial weight and the weight after the removal of the corrosion product. The weight loss (Δw) corrosion rate (C.R), inhibition efficiency (I.E) and degree of surface coverage (Θ) were calculated using the Equations 1, 2, 3 and 4 respectively (Husaini, 2020b; Zarrouk *et al.*, 2010).

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

$$C.R = \frac{w_i - w_f}{At} \quad 2$$

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

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

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

2.4. Scanning Electron Microscopic Analysis Analysis

The surface morphology of aluminium coupons, both before and after the application of the inhibitor, was analyzed using the PRO: X model (800-07334) from Phenom World, Eindhoven, Netherlands. Scanning was performed on three untreated aluminium coupons, as well as on coupons immersed in acidic solution with and without inhibitor. The images were captured at an accelerating voltage of 15.00 kV with a magnification of 1000x.

3. Results and discussion

3.1. Effect of inhibitor concentration on corrosion rate

The results presented in Figure 1 for the effect of inhibitor on the corrosion rate of aluminium in HNO_3 , in the presence of 0.02 - 0.1 M inhibitor shows the gradual decrease in corrosion rate of aluminium in nitric acid solution. The values of corrosion rates of uninhibited system were found to be higher when compared to the inhibited systems. Examination of the results revealed that lower corrosion rate was obtained at the highest concentration of the inhibitor. The corrosion rate was inversely proportional to the concentration of inhibitor at the same temperature. The corrosion rate in the presence of inhibitor was reduced indicating the effectiveness action of the compound as corrosion inhibitor (Husaini and Ibrahim, 2020). This suggests that as the concentration of inhibitor increases, there was an increase in surface coverage of the adsorbed molecules on the aluminium surface which provided a barrier (film) and reduced further corrosion. Similar results was reported by Vashi and Prajapati (2017)

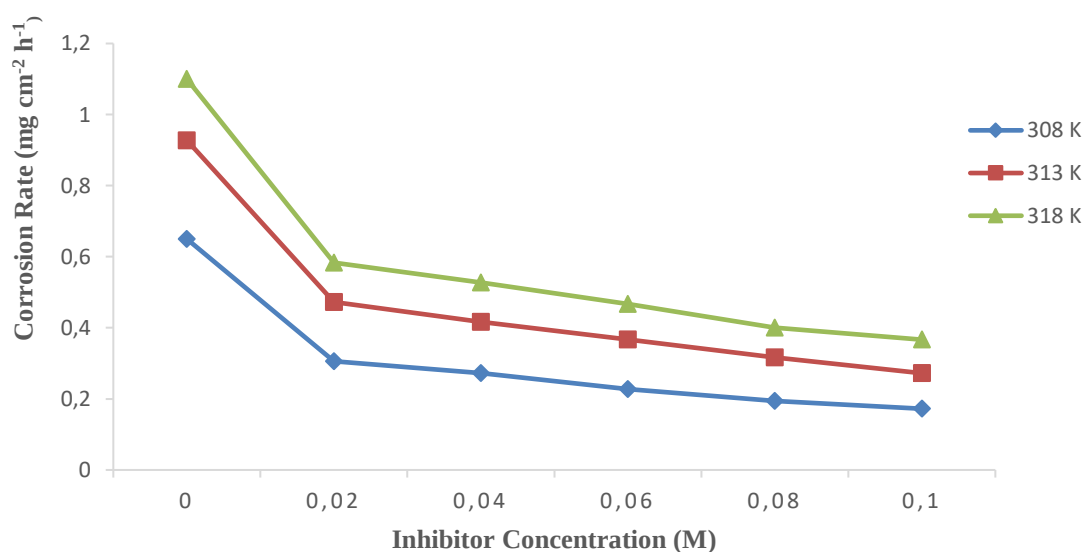


Figure 1. Variation of Corrosion Rate with Inhibitor Concentration

3.2. Effect of inhibitor concentration on inhibition efficiency

The effect of inhibitor concentration against inhibition efficiency was plotted in Figure 2. The inhibition efficiency (% IE) was found to increase with increasing inhibitor concentration due to increase in surface coverage (Θ) of adsorbed specie on surface of the metal. The % IE increases with increasing the inhibitor concentration indicated that more inhibitor molecules are adsorbed on the metal surface thus providing wider surface coverage (Husaini *et al.*, 2020c). The aniline molecule is spontaneously protonated in acidic medium to be cationic ion $C_6H_5-NH_3^+$ to facilitate the adsorption process.

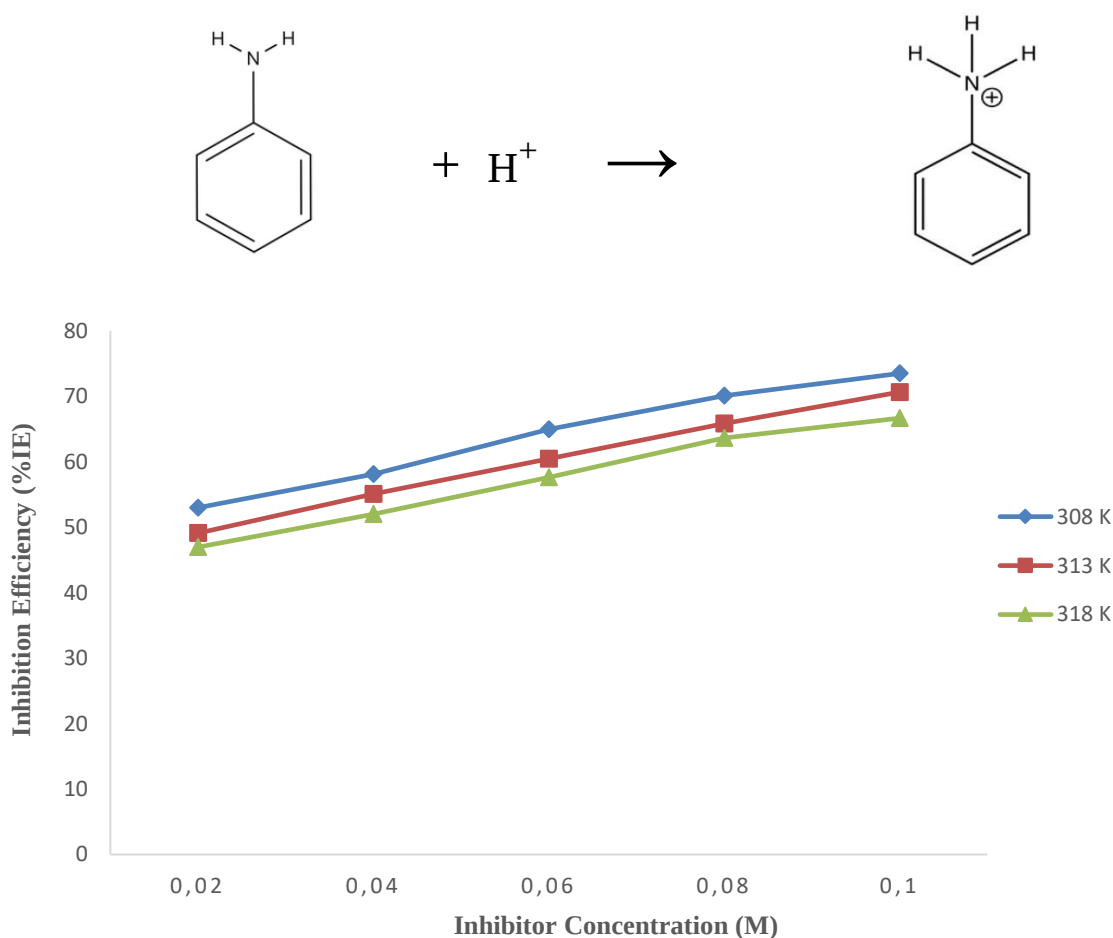


Figure 2. Variation of Inhibition Efficiency with Inhibitor Concentration

3.3. Effect of Temperature on Corrosion Rate

Figure 3 shows the variation of corrosion rate against temperature at different inhibitor concentration. The results showed that the corrosion rate increases with increase in temperature. Increase in temperature result in destabilizing the inhibitor molecule and decrease its activity. It can be observed from the results that the highest corrosion rate of aluminium was

observed at the highest temperature. This observation is due to the fact that chemical reaction rates generally increases with rising temperature. Increase in temperature leads to increase in the kinetic energy possessed by the reacting molecules thereby making the molecules to overcome the energy barrier faster (Ladha *et al.*, 2013; Husaini, 2020b). The increase in corrosion rate as temperature increases in a given inhibitor concentration can be explain on the basis of desorption of the inhibitor molecules on the metal surface. Similar work was reported by Udom *et al.* (2017)

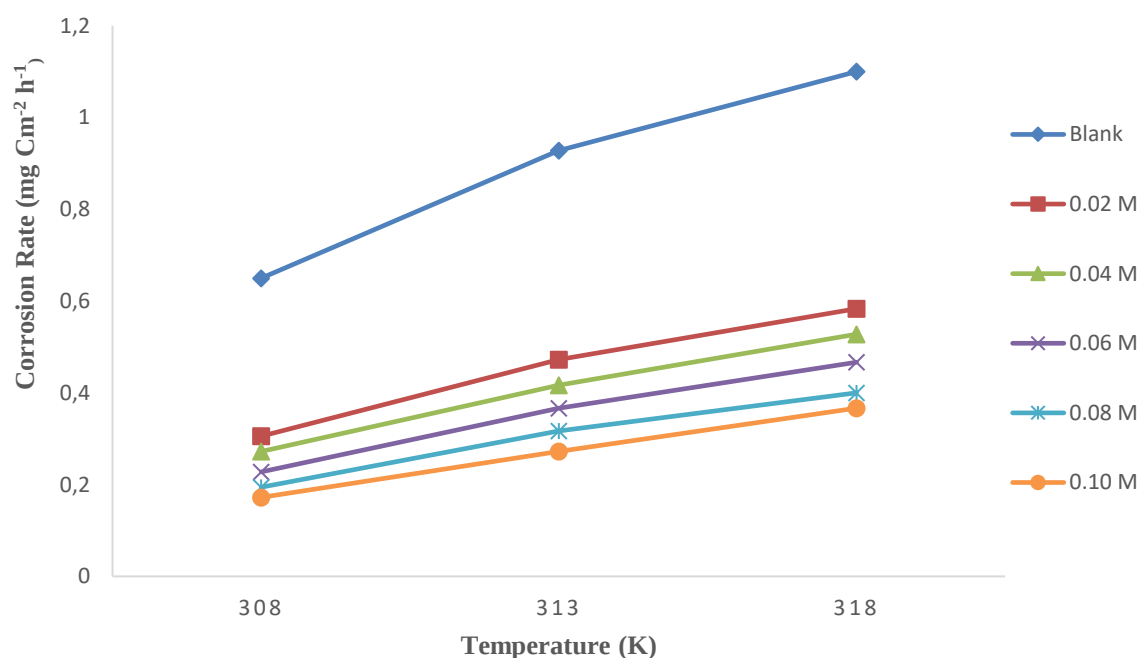


Figure 3. Variation of Corrosion Rate with Temperature

3.4 Effect of Temperature on Inhibition Efficiency

Figure 4 shows the variation effect of temperature on the inhibition efficiency for the corrosion of aluminium at different inhibitor concentration. The result shows that the inhibition efficiency decreases with increase in temperature. It is suggested that the decrease of inhibition efficiency is due to the desorption of the inhibitor molecules on the surface of aluminium as a result of rise in temperatures resulting in destabilizing the inhibitor, this results in reducing efficiency of physical adsorption activity (Husaini *et al.*, 2019a). Similar work was reported by Namrata *et al.* (2015) and Husaini *et al.*, (2025).

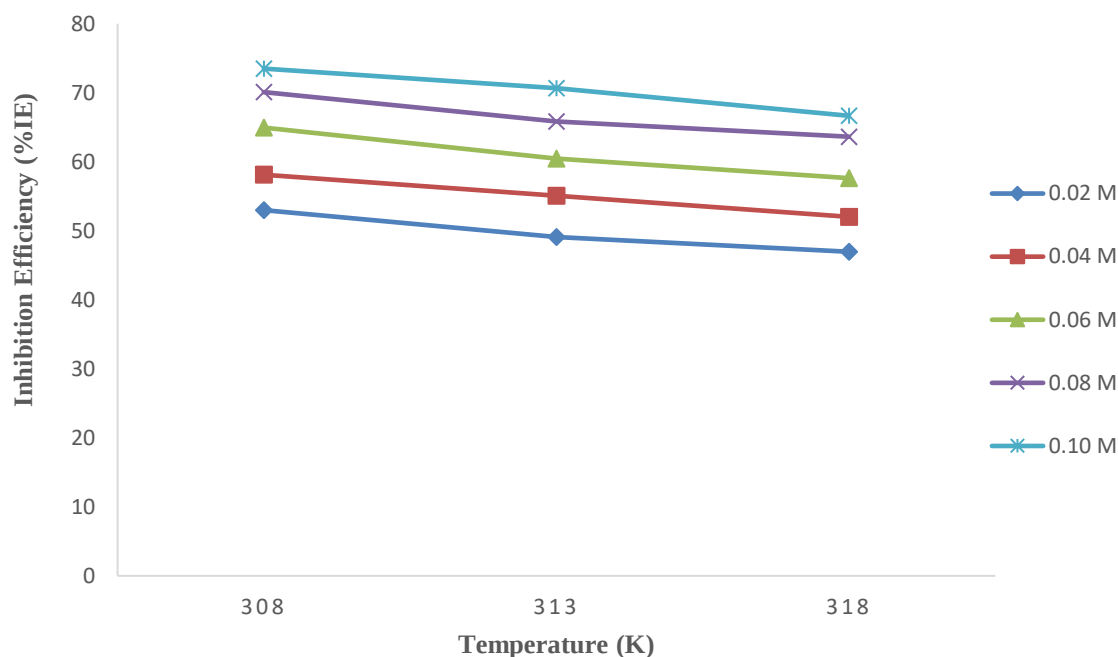


Figure 4. Variation of Inhibition Efficiency with Temperature

3.5 Adsorption behavior

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

$$\frac{C_{inh}}{\Theta} = \frac{1}{K_{ads}} + C_{inh} \quad 5$$

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

coefficient (R^2) and adsorption equilibrium constant (K_{ads}) values are presented in [Table 3.3](#)

3.6. Kinetics Study

3.6.1. Activation Energy.

The rate of most chemical reactions increases with temperature following Arrhenius equation. In the case of the electrochemical reactions, temperature favours the kinetics of corrosion reactions and more specifically, the anodic dissolution of the metal. The activation energy of the corrosion process can be obtained from the Arrhenius plots according to the following equation

$$C_R = A \exp \left(\frac{-E_a}{RT} \right) \quad 6$$

Taking logarithm of both sides of equation 6, would yield equation 7

$$\ln (C_R) = \ln A - \frac{E_a}{RT} \quad 7$$

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

From [Table 1](#) the calculated activation energy value obtained in the presence of the inhibitor is higher than that of the uninhibited solution indicating the deactivation of the acid molecule on collision with the metal surface by introduction of the inhibitor, thus reducing the rate of acid attack on the metal ([Husaini et al., 2023](#)). The E_a values increases from 0.02 M to 0.10 M inhibitor concentration. It is considered as good evidence supporting the higher Inhibition efficiency and lower corrosion rates of aluminium in higher inhibitor concentration. Activation energy is also another good tool in the determination and characterization of the type of adsorption taking place. It has been reported earlier that, for an adsorption to be considered as physisorption, it is expected that, the average value of E_a be ≤ 80 kJ/mol. For chemisorption, E_a should be ≥ 80 kJ/mol. The Calculated apparent activation energy from experimental data are all below 80 kJ/mol. Therefore the adsorption of the studied inhibitor on the surface of aluminium is consistent with the mechanism of physical adsorption. The observed trend from the data is consistent with investigations reported by [Patel et al. \(2012\)](#).

3.6.2. Rate constant

The kinetics of the corrosion phenomenon has the character of a diffusion process, in which

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

$$\ln (W_i - W_L) = -k_1t + \ln W_L \quad 8$$

According to equation 3.4 the plots of $\ln (W_i - W_L)$ against time showed a linear variation with correlation coefficients close to 1 confirmed first - order kinetics for the corrosion of aluminum in acid with and without the presence of inhibitor.

From Table 1, the value of rate constant (k_1) for the corrosion of aluminium was found to be higher in the case of uninhibited acid solution than inhibited acid solution. This confirmed the inhibition of aluminium corrosion in acid solution by the presence of inhibitor. Similar work was reported by Ezeokonkwo *et al.*, (2015).

3.6.3. Half – life

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

$$\text{Half – life } (t_{1/2}) = \frac{0.693}{K} \quad 9$$

The value of the half-lives ($t_{1/2}$) increased from uninhibited solution to inhibited solution as can be seen from Table 1. The increase in half-lives ($t_{1/2}$) in the presence of the inhibitor compared to the uninhibited solution further corroborate with the results reported earlier that corrosion rate decreases in the presence of the inhibitor compared to the uninhibited solution (Husaini, 2021). It should also be noted that as going from 0.02 to 0.10 M inhibitor concentration, the half-life also increases which results into a decrease in the corrosion rate, suggesting that more protection of the aluminium by the presence of higher inhibitor concentration has been established. Similar work was reported by Olasehinde *et al.*, (2015).

Table 1. Kinetic Parameters for Aluminium Corrosion with and without various inhibitor concentrations

Inhibitor Concentration (M)	Activation Energy (kJ mol ⁻¹)	Rate Const. ($k \times 10^{-3}$) (hour ⁻¹)	Half-life (10^2) (hours)
Blank	42.91	3.92	1.76
0.02	52.74	1.83	3.76
0.04	53.98	1.63	4.23
0.06	58.50	1.36	5.05
0.08	58.84	1.16	5.92

0.10

61.59

1.03

6.69

3.7 Thermodynamic Study

The Thermodynamic parameters like enthalpy of activation (ΔH_a) and entropy of activation (ΔS_a) were calculated using the transition state equation. The linear form of transition state equation is given by the equation below

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

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

From Table 2 the calculated values of enthalpy are all positive indicating the endothermic nature of the reaction suggesting that higher temperature favours 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 (Husaini, 2023). All the values of enthalpy are less than 100 kJmol^{-1} this proved that the adsorption of the inhibitor on the aluminium surface in HNO_3 , follows physical adsorption mechanism.

Table 2. Enthalpy and Entropy change of the reaction process with various concentrations of the inhibitor

Inhibitor Concentration (M)	ΔH_{ads} (kJ mol^{-1})	$-\Delta S_{\text{ads}}$ ($\text{J mol}^{-1}\text{k}^{-1}$)
blank	40.31	175.05
0.02	50.14	149.36
0.04	51.38	146.34
0.06	55.90	133.29
0.08	56.24	133.11
0.10	58.99	125.28

The calculated values of entropy for the corrosion of aluminium in HNO_3 from Table 2 is - $175.05 \text{ JK}^{-1}\text{mol}^{-1}$ which decreased to -149.36 and -125.48 $\text{JK}^{-1}\text{mol}^{-1}$ in the presence of 0.02 and 0.10 M inhibitor concentration. The entropy values are all negative confirming that the corrosion process are entropically favourable. The values of ΔS_{ads} decreased negatively from lower to higher inhibitor concentration. This reveals the formation of an ordered stable film of the inhibitor molecule on the aluminium surface, and the activated complex in the rate determining step represent an association rather than a dissociation step (Husaini *et al.*, 2020d).

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

The tendency for many chemical reaction to proceed, including the reaction of a metal with its

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

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

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 HNO_3 in the presence of inhibitor are -20.85, -20.63 and -20.85 kJ mol^{-1} .

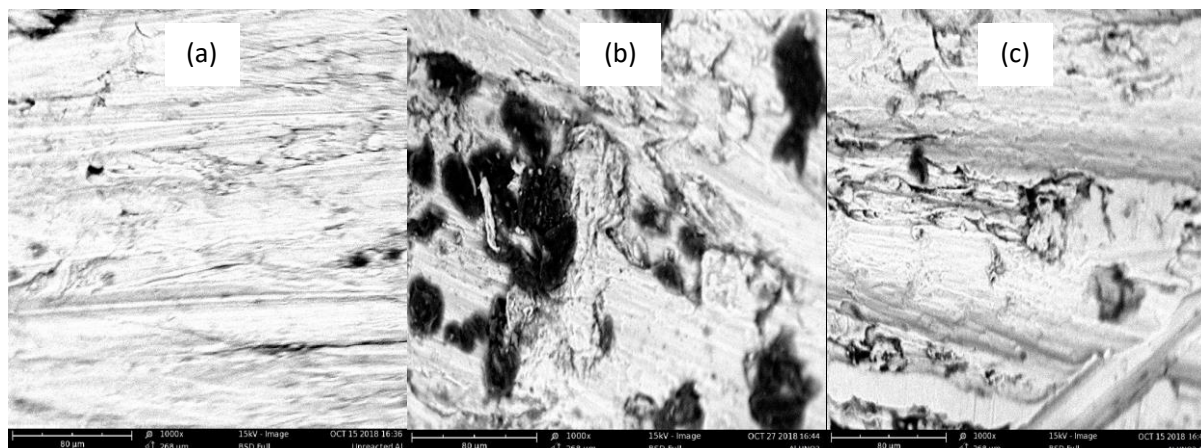
Results obtained indicated that the values of free energy of adsorption are negative. This is the fundamental criterion for spontaneity. The negative values is showing that the reaction is spontaneous. Generally, the values of ΔG_{ads} below or around -20 kJmol^{-1} are consistent with electrostatic interaction between charged molecules and charged metal surface which indicates physisorption 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 chemisorption mechanism (Rabiu *et al.*, 2023; Husaini *et al.*, 2019b). In this study all the values of ΔG_{ads} 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.

Table 3. Adsorption Parameters Deduced from Langmuir Adsorption Isotherm

Temperature (K)	R^2	K_{ads}	ΔG_{ads} (kJ mol^{-1})
308	0.994	54.00	-20.50
313	0.991	50.00	-20.63
318	0.991	48.07	-20.85

3.9 Scanning Electron Microscopic Analysis Result

Figure 5a displays the micrograph of aluminium that has not been exposed to an acidic environment. In contrast, Figure 5b illustrates the surface of aluminium after immersion in acidic medium, showing significant roughness and damage caused by the aggressive acid attack. Figure 5c demonstrates the protective effect of inhibitor, which reduced the extent of acid damage. These observations suggest that aniline effectively inhibited aluminium corrosion in nitric acid by forming a protective film over the surface, a film that was notably absent when aluminium interacted with the acid alone.



Conclusion

In this study the aniline was found to be good inhibitor for the corrosion of aluminium in nitric acid solution. The corrosion rate was found to increase with increasing temperature but decrease with increasing inhibitor concentration. The inhibition efficiency increases with increasing inhibitor concentration but decreases with increase in the temperature. The adsorption process of the inhibitor was feasible and spontaneous with large and negative values of ΔG_{ads} . The enthalpy of the adsorption (ΔH_{ads}) indicate that the adsorption process was endothermic, therefor increase in the reaction temperature of the medium will decrease the inhibition efficiency. The adsorption of the inhibitor on the surface of aluminium is consistent with Langmuir adsorption isotherm.

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