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Study of the inhibitory properties of 2-((benzylthio)methyl)-1H-benzo[d]imidazole with respect to the corrosion of aluminum in a nitric acid medium

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Abstract

Due to its massive use, the behavior of aluminum in a 1 M nitric acid solution was studied in this work; this study which is mainly based on the inhibitory properties of 2-((benzylthio)methyl)-1H-benzo[d]imidazole (2-BTM1HBI) was carried out using the mass loss technique of temperature varying from 298 to 338 K and concentration of 10^{-3} mM at 5 mM. The inhibitory efficiency of 2-BTM1HBI increases with the increase of the concentration and this up to 96.09% but decreases with the increase of the temperature. The study of isotherms shows that the adsorption of the molecule studied on the surface of aluminum obeys the modified Langmuir isotherm (villamil model). The thermodynamic adsorption quantities were determined and discussed. They show that the adsorption of 2-BTM1HBI is spontaneous and exothermic with an increase in disorder. Adsorption is done in two modes: physisorption and chemsorption with a predominance of physisorption. The thermodynamic quantities of activation have shown that the dissolution process is endothermic with an increase in disorder.

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1. Introduction

Pure aluminum is a remarkable metal with a special role in society. It is currently the most widely used non-ferrous metal [1] due to its low density and its electrical and thermal performance. Furthermore, it occupies a prominent place in industrial applications especially in construction, electronics, packaging, storage and transportation equipment and machinery [2-5]. However, in contact with aggressive environments (acids, bases, etc.), its thin protective layer (a few micrometers) is destroyed and the material undergoes alteration (corrosion) which leads to the loss of its physicochemical properties. Corrosion results from chemical or electrochemical action of an environment on metals and alloys. The consequences are significant in various fields and in particular in industry: production stoppage, replacement of corroded parts, accidents and pollution risks are frequent situations with sometimes serious economic impacts. The current effective fight against this phenomenon is undoubtedly the use of organic molecules (corrosion inhibitors) acting at the metal-environment interface [6]. Several organic molecules have been tested as inhibitors of aluminum corrosion in an acidic environment. Thus, azoles, amines, drugs [7-13], etc., have been used successfully. This work, which is a contribution to the study of the inhibition of corrosion of metals in an acidic environment, aims to study the behavior of 2-((benzylthio)methyl)-1H-benzo[d]imidazole (2-BTM1HBI) with respect to the corrosion of aluminum in a 1 M nitric acid environment.

2. Experimental

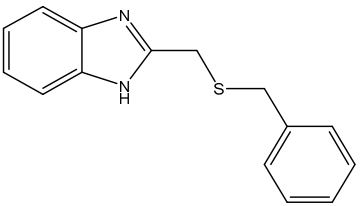
2.1. Aluminum specimens

The aluminum samples are in the form of a rod 10 mm long and 2.5 mm in diameter. It is a commercial aluminum with 99.6% purity.

2.1.1. Molecule studied

The molecule used as an inhibitor in this work, namely 2-BTM1HBI) with chemical formula C15H14N2S and molecular molar mass M = 254 g/mol was synthesized and characterized by a team at the Organic Chemistry and Natural Synthesis laboratory at Félix Houphouët Boigny University in Cocody (Ivory Coast) and its molecular structure was identified by 1H, 13C NMR spectroscopy. This molecule belongs to the

benzimidazole family. The molecular structure is shown in Scheme 1.



Scheme 1. Molecular structure of 2-((benzylthio)methyl)-1H-benzo[d]imidazole (2-BTM1HBI).

2.1.2. Solution

Analytical grade 65% nitric acid solution from Merck was used to prepare the corrosive aqueous solution. The solution was prepared by diluting the commercial nitric acid solution with distilled water. A molar solution of nitric acid without inhibitor then with inhibitor concentrations (2-BTM1-HBI) of 0.001, 0.005, 0.01, 0.05, 0.1, 0.5, 1.0 and 5.0 mM in a temperature range from 298 to 338 K.

2.1.3. Mass loss method

Mass loss measurements were carried out by total immersion of the pre-weighed aluminum sample in 100 mL capacity beakers containing 50 mL of the test solution maintained at a temperature of (298 to 338 K). The samples were recovered one hour later and rinsed thoroughly with distilled water, cleaned, dried in acetone and reweighed using a balance with a sensitivity of ±0.1 mg. All tests were performed in triplicate to ensure reliability of the results. Weight loss was considered as the difference between the initial weight and the weight after 1 h of immersion. The average values of the mass loss data were used to calculate parameters such as corrosion rate, inhibition efficiency and surface coverage using Equations 1-3.

$$W = \frac{\Delta m}{St} \tag{1}$$

$$EI(\%) = \frac{W_0 - W}{W_0} \times 100 \tag{2}$$

$$\theta = \frac{W_0 - W}{W_0} \tag{3}$$

where W_0 and W are the corrosion rate in the absence and presence of the inhibitor, respectively. Δm is the mass loss, S is the total surface area of the copper sample and t is the immersion time.

3. Results and Discussion

3.1. Mass loss experiment

Mass loss data were determined at the end of a 1-hour time interval in the absence and presence of different concentrations of 2-((benzylthio)methyl)-1H-benzo[d]imidazole and were used to calculate corrosion rates, inhibition efficiency and degree of surface coverage, according to Equations 1-3. Figure 1 shows the evolution of the corrosion rate with concentration and temperature respectively for 2-BTM1HBI.

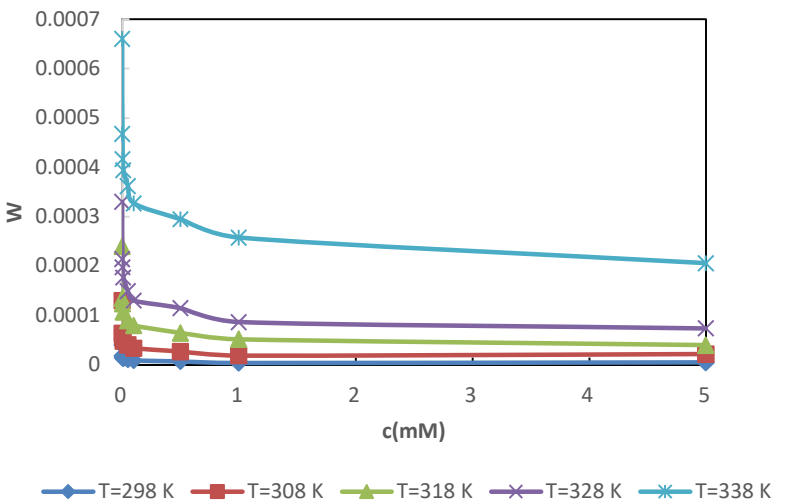


Figure 1. Corrosion rate versus 2-BTM1HBI concentration curve at different temperatures.

Examination of **Figure 1** shows that the corrosion rate increases with temperature for all concentrations and decreases as the inhibitor concentration increases. In the absence of inhibitor, the corrosion rate is very high, which shows that the addition of 2-BTM1-HBI in the corrosive medium delays the corrosion of aluminum. In addition, the presence of 2-BTM1-HBI promotes the formation of a protective layer due to the adsorption of the molecule on the surface of the metal. This protective layer prevents the aluminum from

losing enough electrons or undergoing strong dissolution in acid. These results indicate that the molecule studied has a good performance in inhibiting the corrosion of aluminum in a nitric acid solution. **Figure 1** respectively represents the evolution of the inhibitory efficiency as a function of the concentration of 2-BTM1-HBI at different temperatures.

Analysis of **Figure 2** shows that inhibitory efficiency increases with inhibitor concentration, but decreases with increasing temperature. This trend could be explained by the increasing adsorption of the inhibitor to the metal as temperature rises, as their concentration in the solution increases. However, inhibitory efficacy decreases with increasing temperature. This behavior could be linked to a breakdown in the adsorption-desorption equilibrium in favor of inhibitor desorption as temperature rises.

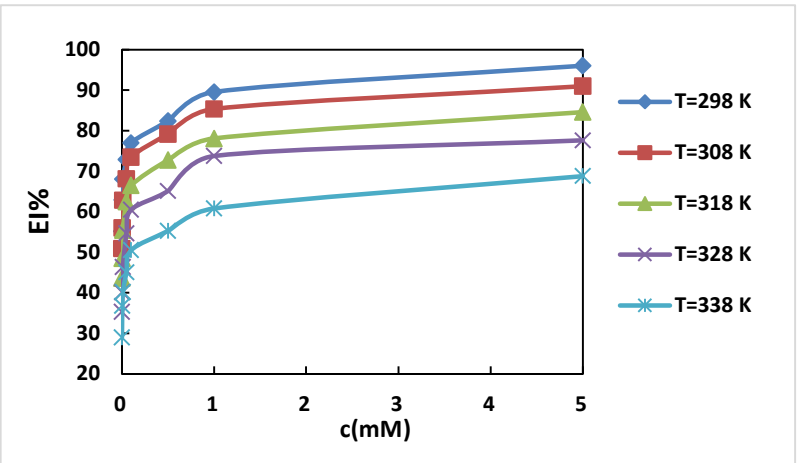


Figure 2. Inhibition efficiency versus 2-BTM1-HBI concentration at different temperatures.

3.2. Study of adsorption isotherms and thermodynamic adsorption quantities

The study of adsorption isotherms involved in the process of inhibiting metal corrosion by organic molecules shows how these compounds attach to the surface of a metal. Indeed, the adsorption of an organic adsorbate on the metal-solution interface can be likened to a chemical reaction in which water molecules adsorbed on the metal surface are replaced by the organic molecules from the solution. It is therefore a phenomenon of substitutional adsorption **[14, 15]** as shown by the following reaction:

$$Org_{(sol)} + xH_2O_{(ads)} \rightleftarrows Org_{(ads)} + xH_2O_{(sol)} \quad (4)$$

Several adsorption isotherms (Langmuir, Temkin, El-awady) were tested. However, the Langmuir adsorption isotherm (**Figure 3**) was found to provide the best description of the adsorption of the material studied. It best reflects the behavior of 2-BTM1-HBI on the aluminum surface. The equation of this isotherm is given in **Equation 5**:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh}. \quad (5)$$

Figure 3 shows the representation of these different isotherms for 2-BTM1-HBI.

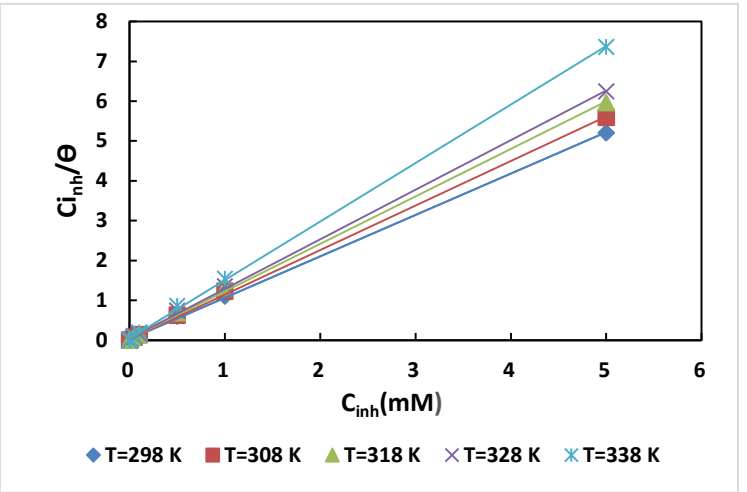


Figure 3. Langmuir adsorption isotherm plots of 2-BTM1-HBI on aluminium in 1M HNO_3.

Table 1 gives the different parameters of the isotherms studied.

Table 1. Langmuir isotherms parameters for different temperatures.

Inhibitor	T(K)	K_{ads} (M^{-1})	Slope	R^2	Intercept
2-BTM1-HBI	298	52631	1.038	0.9997	0.0275
	308	40000	1.1169	0.9996	0.031
	318	35714	1.1909	0.9994	0.0384
	328	33670	1.2454	0.9995	0.0415
	338	32467	1.4672	0.9997	0.0427

The values of the coefficient of determination (R^2) of the Langmuir isotherm are close to unity; However, the slopes of the curves are different from unity due **[16, 17]** to the interactions between the adsorbed species. Thus, the Langmuir adsorption isotherm cannot be rigorously applied; It is replaced by the modified Langmuir isotherm called the Villamil isotherm which is based on the **Equation 6**:

$$\frac{C_{inh}}{\theta} = \frac{n}{K_{ads}} + nc_{inh} \quad (6)$$

θ: Recovery rate

n: is a constant introduced to consider all factors, not taken into account in the derivation of the Langmuir isotherm

$n\theta$ represents the effective recovery rate.

The values of the standard free energy ΔG^0_{ads} were determined from the equilibrium constant K_{ads}according to the **Equation 7**:

$$\Delta G^0_{ads} = -RTln(55.5K_{ads}) \quad (7)$$

where R is the perfect gas constant, T is the absolute temperature and 55.5 is the concentration of water in mol.L^{-1} and K_{ads} is the adsorption equilibrium constant.

The other thermodynamic adsorption parameters (adsorption enthalpy ΔH^0_{ads} and adsorption entropy ΔS^0_{ads}) are calculated using the **Equation 8**:

$$\Delta G^0_{ads} = \Delta H^0_{ads} - T\,\Delta S^0_{ads} \quad (8)$$

The different curves are given in **Figure 4** below.

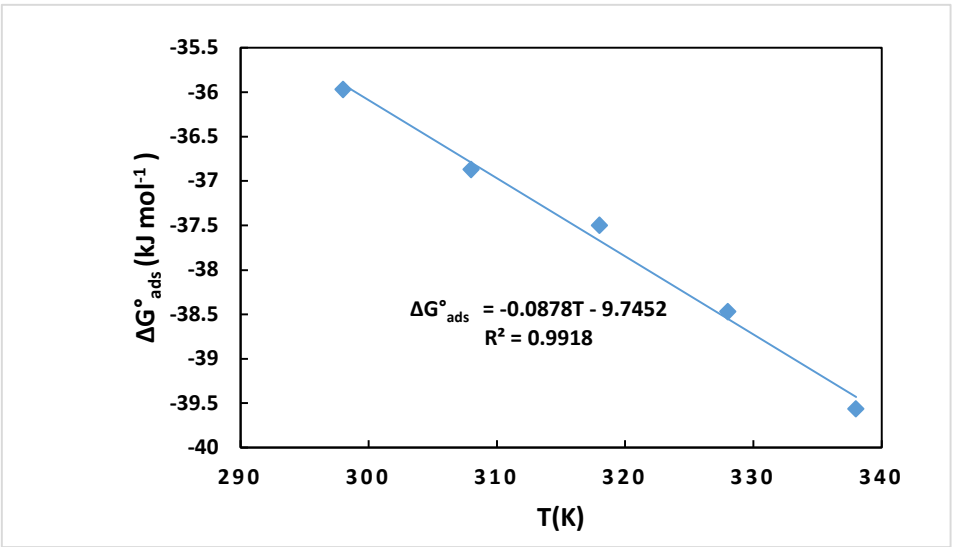


Figure 4. Variation of ΔG^0_{ads} versus temperature.

The values of these functions are presented in Table 2.

Table 2. Thermodynamic adsorption parameters for 2-BTM1-HBI.

Molecule	T(K)	$\Delta G^0_{ads}(kJmol^{-1})$	$\Delta H^0_{ads}(kJmol^{-1})$	$\Delta S^0_{ads}(Jmol^{-1}K^{-1})$
2-BTM1-HBI	298	-36. 88	-9.7452	87.8
	308	-37. 200		
	318	-37. 730		
	328	-38. 180		
	338	-38. 68		

Negative values of ΔG^0_{ads} indicate the stability of the adsorbed layer on the aluminium surface and the spontaneity of the adsorption process [18]. According to the literature [19], a value of ΔG^0_{ads} lower than -40 kJ.mol^{-1} would indicate a chemical adsorption process (chemisorption) whereas a value higher than -20 kJ.mol^{-1} would indicate a physical adsorption process (physisorption). For values between -40 kJ.mol^{-1} and -20 kJ.mol^{-1}, both types of adsorptions would exist. In view of the values contained in Table 2, we can deduce that the adsorption of 2-BTM1-HBI on aluminum takes place according to the two adsorption modes (physisorption and chemisorption).

The value of the standard adsorption enthalpy ΔH_{ads}^{0} is negative, which reflects the exothermic nature of the adsorption of 2-BTM1-HBI on aluminum. The value of the standard adsorption entropy ΔS_{ads}^{0} is positive, thus indicating an increase in disorder due to the desorption of water molecules.

3.3. Type of adsorption

In order to correctly justify the mode of adsorption of the molecule studied, we used the Adejo-Ekwenchi isotherm [20]. Indeed, this isotherm allows us to know the mode of adsorption of an organic compound. This model is based on the Equation 9:

$$log\frac{1}{1-\theta} = logK_{AE} + b\,logC_{inh} \tag{9}$$

where K_{AE} and b are isotherm parameters.

Figure 5 gives the curves obtained.

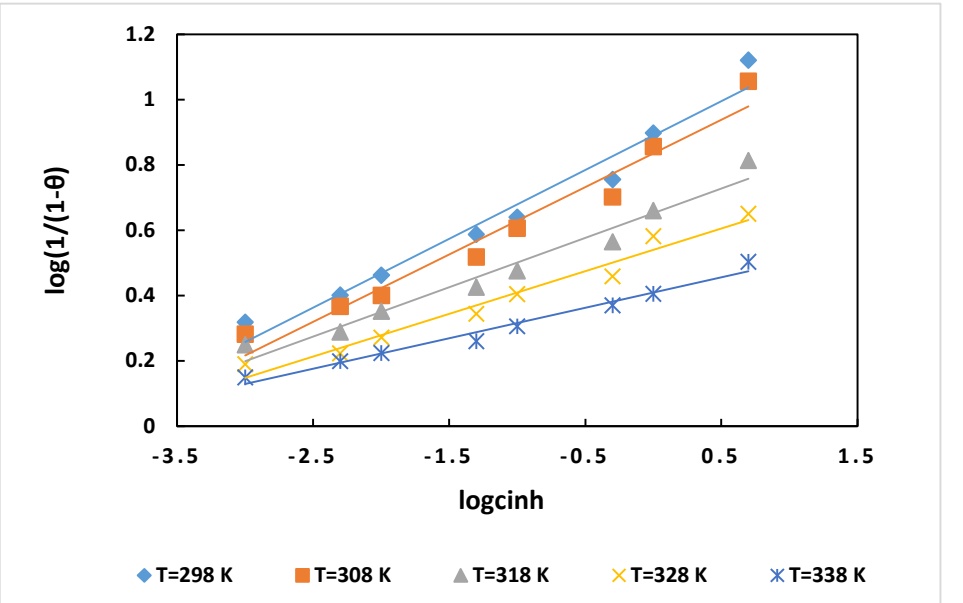


Figure 5. Adejo-Ekwenchi isotherm plots of 2-BTM1-HBI on aluminium in 1M HNO_3.

The parameters of this isotherm are given in **Table 3**.

Table 3. Parameters of the Adejo-Ekwenchi isotherm.

Molecule	T(K)	R^2	b	K_{AE}
2-BTM1-HBI	298	0.9641	0.2107	7.77
	308	0.9609	0.2062	6.85
	318	0.9646	0.1512	4.5
	328	0.966	0.1306	3.47
	338	0.977	0.0933	2.57
	328	0.9539	0.1393	4.30
	338	0.962	0.1157	3.51

The Adejo-Ekwenchi isotherm **[21-25]** indicates that a decrease in the value of b with increasing temperature means a predominance of physisorption, while an increase or a fairly constant value indicates a predominance of chemisorption. From Table 3, it can be seen that the adsorption parameters of the studied molecule 2-BTM1-HBI on the aluminum surface unambiguously explain the predominance of physisorption when b decreases with increasing temperature.

3.4. Effect of temperature and activation parameters of the corrosion process

In order to evaluate the apparent energy (E_a) in the absence and presence of molecules, the modified form of the Arrhenius equation **[23]** has been used (**Equation 10**)

$$logW = logk - \frac{E_a}{2.3RT} \tag{10}$$

where W is the corrosion rate, R is the perfect gas constant.

The values of the activation energies are deduced from the curves of logW as a function of 1/T are given in **Figure 6**.

The values of the variations of activation enthalpy ΔH_a^* and activation entropy ΔS_a^* are determined using the transition state equation (**Equation 11**):

$$W = \frac{R.T}{\aleph.h} exp\left(\frac{\Delta S_a^*}{R}\right).exp\left(-\frac{\Delta H_a^*}{R.T}\right) \tag{11}$$

This relationship leads to the relationship below (**Equation 12**):

$$log(\frac{W}{T}) = log(\frac{R}{\aleph.h}) + \frac{\Delta S_a^*}{2,3.R} - \frac{\Delta H_a^*}{2,3.R.T} \quad (12)$$

where h is Planck's constant and $\aleph$ is Avogadro's constant, E_a activation energy, ΔH_a^* activation enthalpy variation and ΔS_a^* activation entropy variation.

The parameters ΔH_a^* and ΔS_a^* are calculated from the slope $(-\frac{\Delta H_a^*}{2,3.R.T})$ and intercept $(log(\frac{R}{\aleph.h}) + \frac{\Delta S_a^*}{2,3.R})$ of the straight lines obtained respectively (**Figures 6 and 7**)

The values obtained are recorded in **Table 4**.

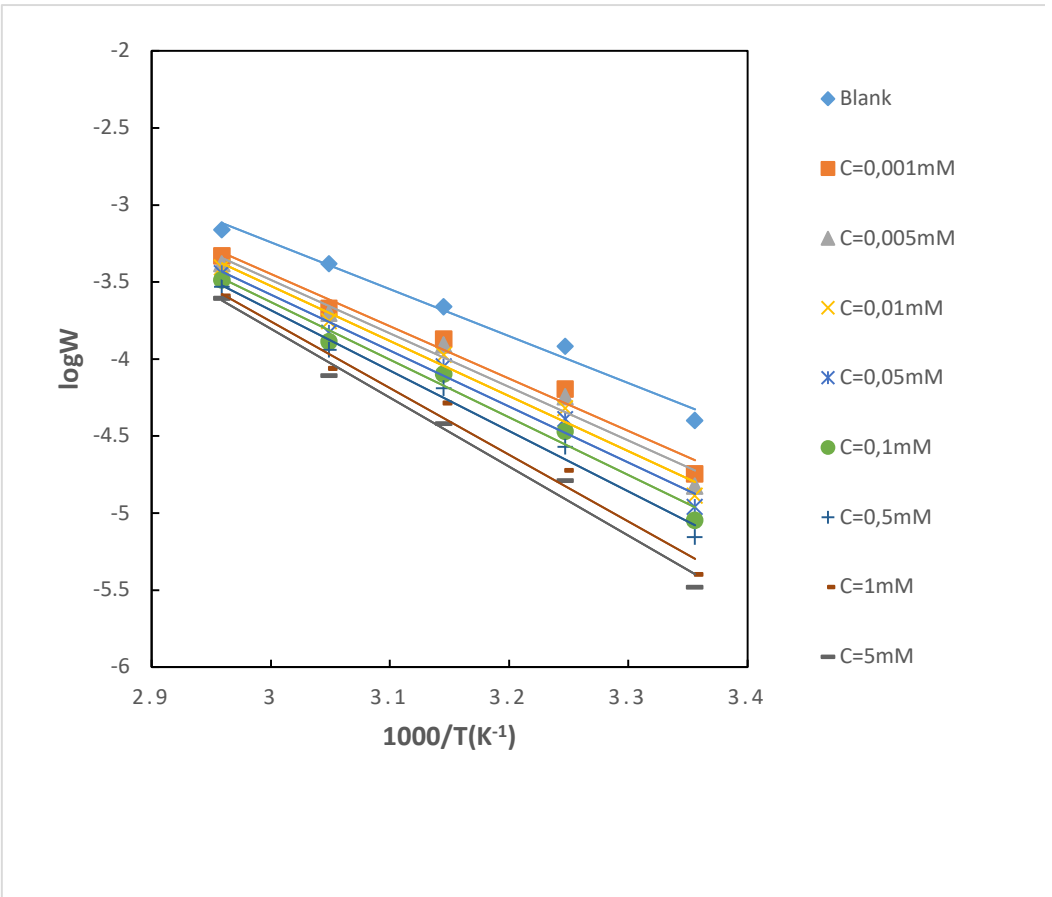


Figure 6. Arrhenius plots for aluminium in 1M HNO_3.

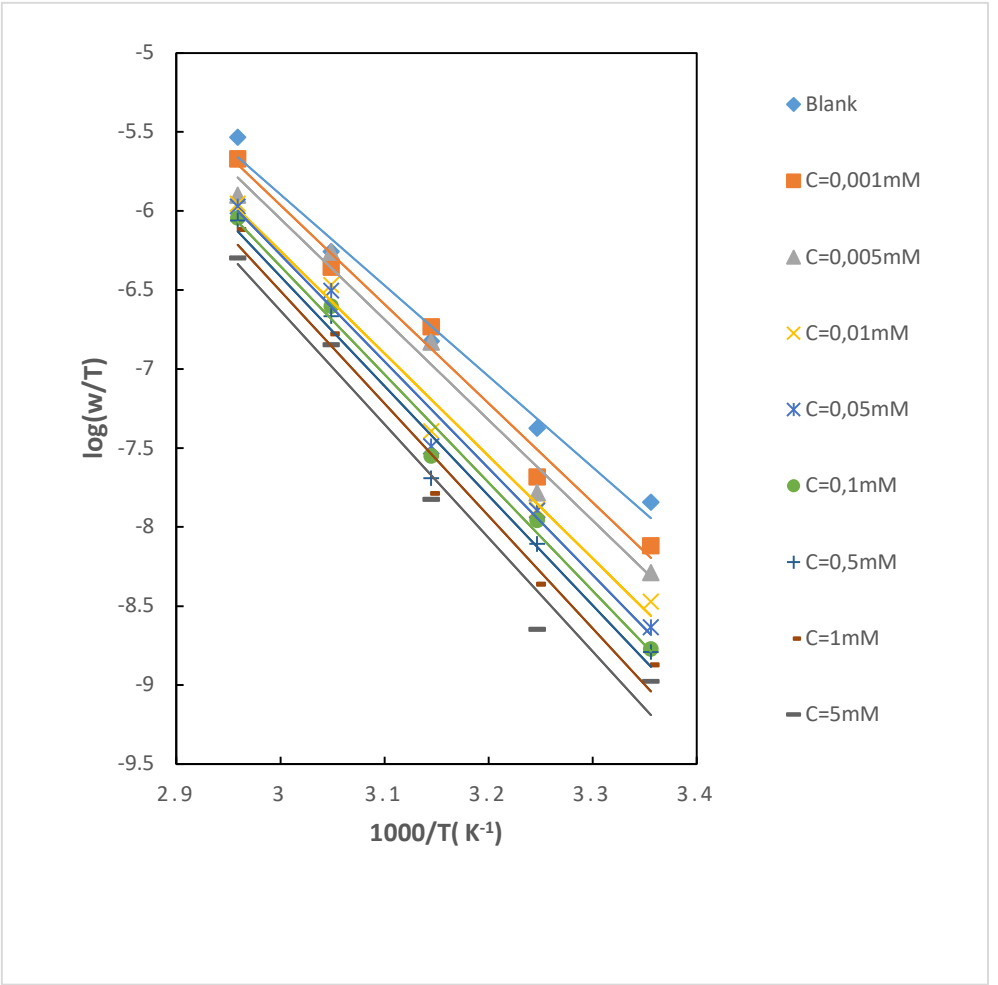


Figure 7. Transition state plots for aluminium in 1M HNO_3.

Table 4. Activation parameters of molecule studied.

Molecule	C (mol/L)	Arrhenius slopes			Transition states equations		
		$logk$	$E_a(kJmol^{-1})$	R^2	ΔH_a^* (kJ/mol)	ΔS_a^* (J. mol-^{1}K^{-1})	R^2
2-BTM1-HBI	0	5.8945	59.70	0.9851	10.200	20.4	0.9866
	0.001	6.7345	64.99	0.9786	120.070	48.44	0.9840
	0.005	6.9315	66.49	0.9809	121.870	52.19	0.9828
	0.01	7.1890	68.39	0.9820	124.200	55.27	0.9855
	0.05	7.3229	69.60	0.9819	129.400	70.43	0.9846
	0.1	7.6030	71.69	0.9863	131.200	74.49	0.9877
	0.5	8.0782	75.07	0.9790	132.840	78.14	0.9805
	1	9.2300	82.88	0.9848	136.270	86.64	0.9776
	5	9.6176	85.66	0.9972	137.620	88.33	0.972

Comparison of the activation energies obtained in the presence of inhibitor $E_a(inh)$ or without E_a **[26-27]** allows us to know the nature of the adsorption:

- $E_a(inh) > E_a$ (blank): the inhibitor s adsorbs on the substrate by electrostatic bonds. These connections are weak. This type of bond is sensitive to temperature and does not effectively combat corrosion at high temperatures;

- $E_a(inh) < E_a$ (blank): in this case the protective power of the inhibitor increases with temperature. The inhibitor adsorbs on the metal surface by strong bonds (chemisorption).

- $E_a(inh) = E_a$ (blank): This category does not show any change in protective power with temperature.

The analysis of our results shows that, for the three molecules, $E_a(inh) > E_a$ (blank) **[28-29]** thus reflecting the predominance of physisorption. The activation energy increases when the inhibitor concentration increases, thus favoring the formation of electrostatic bonds, which are weak. These connections are sensitive to temperature and do not effectively combat corrosion at high temperatures. The positive signs of the

activation enthalpy variations (ΔH_a^*) reflect the endothermic nature of the aluminum dissolution process. The values of the activation entropy variations (ΔS_a^*) are all positive, which reflects an increase in disorder during the dissolution of aluminum which could be the cause of the decrease in inhibitory effectiveness when the temperature increases.

4. Conclusion

The mass loss technique was used to evaluate the corrosion inhibition of aluminum by 2-BTM1-HBI of the benzimidazole family in 1M HNO3. 2-BTM1-HBI acts as a good inhibitor of aluminum corrosion in 1M nitric acid medium. The effectiveness of 2-BTM1-HBI inhibition depends on concentration and temperature. The 2-BTM1-HBI molecule adsorbs on aluminum according to the modified Langmuir isotherm (Villamil). The calculated thermodynamic parameters linked to adsorption and activation for 2-BTM1-HBI show the existence of two types of adsorption (physisorption and chemisorption) with a preponderance of physisorption.

5. Conflicts of Interest

There are no conflicts to declare.

6. Acknowledgement

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