

## Use of Attapulgite as a Corrosion Inhibitor for Industrial Metals and Alloys: Case of Aluminum, Zinc and Aluzinc in a 0.5 M Hydrochloric Acid Solution

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The study deals with the use of attapulgite as a corrosion inhibitor of two metals, namely aluminum and zinc, and their alloy (aluzinc), in a 0.5 M hydrochloric acid medium. The study was carried out by monitoring the free-corrosion potential and performing dynamic polarization measurements (Tafel curves) at different concentrations of inhibitor in solution. The measurements suggested that in a 0.5 M acidic medium, the surface of each of the three materials was attacked. The decrease in current density was very noticeable in aluminum and zinc at a level of 2 g·L<sup>-1</sup>. A weak tendency to passivity was noticed for zinc and aluzinc. The experimental results obtained showed a decrease in the current intensity in the presence of the corrosion inhibitor in the acidic medium. The maximum inhibitory efficiency rate reached for the metals and their alloy was higher than 99 %. This percentage was obtained for aluminum and zinc in the presence of 4 g·L<sup>-1</sup> of attapulgite and 3 g·L<sup>-1</sup> for aluzinc.

*Keywords:* attapulgite, corrosion inhibitor, dynamic polarization.

### 1. INTRODUCTION

Aluminum and its alloys are increasingly used, especially because of their good strength-to-weight ratio, tensile properties, fatigue strength, fracture toughness, good suitability for engineering processes, etc [1, 2]. In addition, they are more resistant to corrosion, which makes them better materials for military, aerospace, marine and automotive applications [3]. Zinc and aluzinc, used interchangeably, are mainly used in the construction industry as roofing and cladding materials. Because of its durability and environmental friendliness, Aluzinc requires, like zinc and aluminum, protection against corrosion by using environmentally-friendly inhibitors. Among the ecological inhibitors, we can mention green inhibitors such as gum arabic, essential oils and mineral inhibitors such as clays [4–9]. Corrosion is an electrochemical phenomenon and any study done on it can only be made in the context of an electrochemical representation of the processes involved [10]. Corrosion control can be achieved in different ways: organic or inorganic coating, anodization of the metal, protection by organic or mineral inhibitors and cathodic protection by the applied current. The choice of one of these processes depends on ecological and economic considerations and the conditions of use.

The use of the chemicals identified as corrosion inhibitors for metals in aqueous media requires prior verification of their toxicity. These are usually substances containing XO<sub>4</sub><sup>2-</sup> oxo-anions such as chromates, molybdates, tungstate, etc., or other oxo-anions such as

nitrites, phosphates, borates and silicates [11, 12]. Because of toxicity concerns, an alternative class of inhibitors called green or ecological inhibitors has emerged, including a wide variety of plant extracts [13, 14] and clays [15–17]. In this context, tuffs [8, 9] and montmorillonite [8] have recently been studied in our laboratory as corrosion inhibitors for steel as suspended solids in aqueous solutions. We obtained maximum efficiencies of 62 % for montmorillonite and 70 % for tuffs.

In this work, we studied the effect of attapulgite used as a corrosion inhibitor for aluminum, zinc and aluzinc in a corrosive environment of 0.5 M hydrochloric acid (HCl). We chose attapulgite which, in contrast to tuffs and montmorillonite, is rich in Mg. This is expected to lead to higher inhibition efficiencies. The electrochemical methods used to study the behavior of these materials in a corrosive environment are respectively open circuit potential measurements and dynamic polarization.

### 2. MATERIALS AND METHODS

Aluminum and aluzinc with mass percentages of 55 % Al, 43.4 % Zn and 1.6 % Si, were supplied by the Senegalese company of transformation of materials (CSTM SAFOR), Dakar, Senegal. Zinc was supplied by the hot-dip galvanizing plant in Sebikotane, Senegal. Aluzinc, also known as Al-Zn, is a carbon steel sheet coated by dipping prepared products in the melted mixture containing 55 % Al, 1.6 % Si and 43.4 % Zn and with a weight of 185 g/m<sup>2</sup> on both sides.

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The metals were cut into square sheets of  $1 \times 1 \text{ cm}^2$  surface. One of the surfaces is exposed to the electrolyte and the rest was coated with an epoxy oxide layer. The surface in contact with the electrolyte solution was polished with abrasive paper of varying grit sizes (400, 600, 800 and 1000) and then rinsed with distilled water and dried.

Attapulgite extracted from the quarries of Mbodiène (Senegal), used as a corrosion inhibitor, is composed essentially of Si (42.90 %), Mg (13.20 %), Ca (17.12 %), Fe (15.16 %) and Al (6.97 %) [18]. Its chemical composition is given in Table 1.

**Table 1.** Chemical composition of attapulgite [18]

Compound, %									
SiO <sub>2</sub>	MgO	CaO	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	Cl
55.15	13.8	11.11	8.3	7.78	0.99	0.97	0.78	0.35	0.33

The electrolyte used was hydrochloric acid with a concentration of 0.5 M, prepared by appropriate dilution of the commercial acid (37 %, Sigma Aldrich). We used the solution without and with attapulgite, to study the electrochemical behavior of each material. Electrochemical measurements were performed using a three-electrode cell consisting of a counter electrode (platinum grid), a reference electrode (Ag/AgCl), and a working electrode made of the sample under study (aluminum, zinc or aluzinc). We used a  $\mu$ -Autolab III potentiostat-Galvanostat controlled by General Purpose for Electrochemical System (GPES) and Frequency Response Analyzer (FRA) software for electrochemical measurements. Before each experiment, the working electrode was kept immersed in the HCl solution containing the inhibitor at free-corrosion potential for one hour under magnetic stirring, in order to stabilize the suspension. We first investigated the free-corrosion potential or open-circuit potential (OCP), measured in the absence of current. In potentiodynamic mode, we performed a potential sweep between -0.25 and +0.25 V/Ag/AgCl relative to the OCP. The potential scan rate for all measurements was 0.5 mV/s. This value allowed testing under quasi-stationary conditions [13, 18]. The equilibration time was set to 2 h. The electrochemical parameters ( $i_{\text{corr}}$ ,  $E_{\text{corr}}$ ,  $R_p$ ,  $b_a$  and  $b_c$ ) were obtained using Tafel polarization curves. The calculation of these parameters was performed by fitting the experimental values into the theoretical model of Stern-Geary [19] by a linear regression by GPES software after correction for the ohmic drop. The inhibitory efficiency was calculated from the following equations [14].

$$IE = \frac{i_{\text{corr}}^0 - i_{\text{corr}}}{i_{\text{corr}}^0} \times 100, \quad (1)$$

where  $i_{\text{corr}}^0$  and  $i_{\text{corr}}$  being respectively the current densities without and with inhibitor.

This inhibitory efficiency was also evaluated by means of the polarization resistance.

$$EI = \frac{R_p - R_p^0}{R_p} \times 100, \quad (2)$$

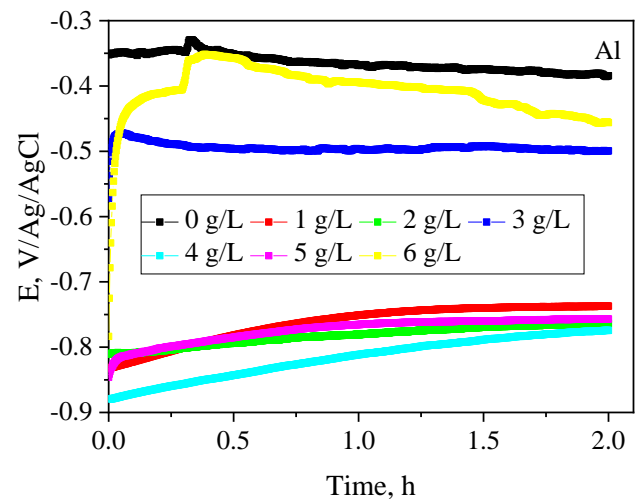
where  $R_p$  and  $R_p^0$  are respectively the polarization resistances with and without inhibitor.

### 3. RESULTS AND DISCUSSION

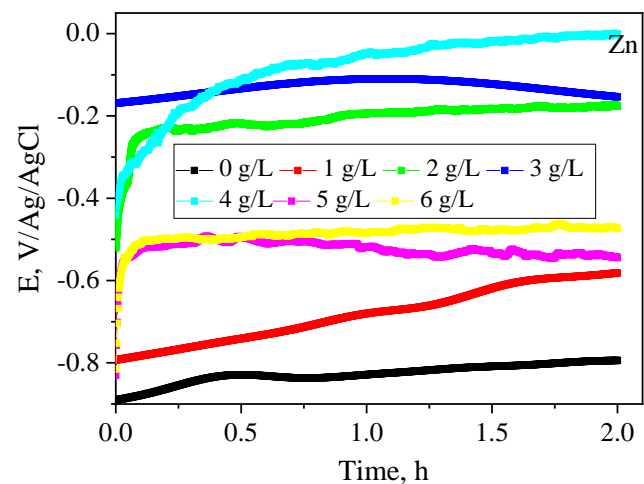
#### 3.1. Free-corrosion potential

The monitoring of the free-corrosion potential, or open-circuit potential (OCP) as a function of time makes it possible to appreciate the behavior of the metal surface in a corrosive environment and determine the equilibrium time necessary to obtain a stationary regime essential to the potentiodynamic plots. This behavior is reflected by the attack of the material and/or the formation of a protective layer.

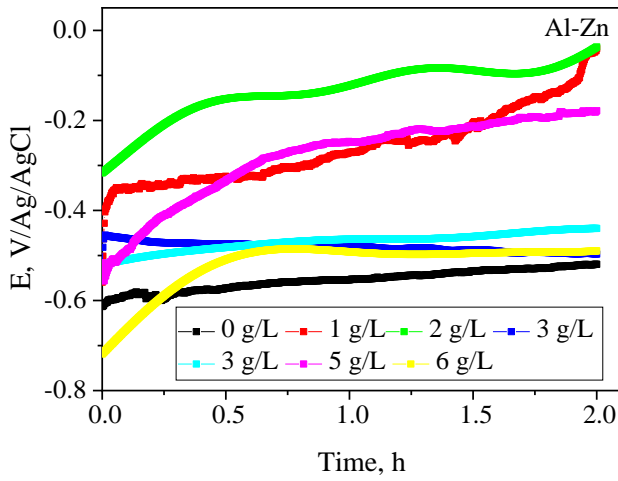
Chronopotentiometry at zero current does not allow to conclude on the evolution of the anodic and cathodic reactions. It is a preliminary test for electrochemical studies. The free-corrosion potential is the most immediately measurable electrochemical quantity. It is the only measurement that does not involve any disturbance of the state of the system to be studied [15]. Fig. 1, Fig. 2 and Fig. 3 show the evolution of the free-corrosion potential of aluminum, zinc and aluzinc, respectively, in a 0.5 M HCl solution at different inhibitor concentrations.



**Fig. 1.** Evolution of the free-corrosion potential of aluminum in 0.5 M HCl at different concentrations of inhibitors



**Fig. 2.** Evolution of the free-corrosion potential of zinc in 0.5 M HCl at different inhibitor concentrations



**Fig. 3.** Evolution of the free-corrosion potential of aluzinc, in 0.5 M HCl at different inhibitor concentrations

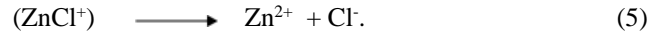
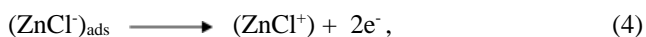
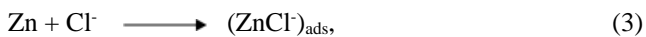
Table 2 presents the OCP values for the three materials in 0.5 M aqueous HCl solution without attapulgite.

**Table 2.** The free-corrosion potential of the materials and time to reach a constant value, in 0.5 M HCl

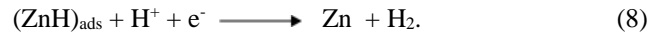
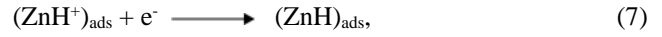
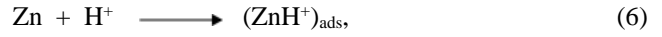
Materials	Aluminum	Zinc	Aluzinc
OCP, V/Ag/AgCl	-0.384	-0.820	-0.520
t, min	90	105	120

We noted in most cases an initial decrease of the free-corrosion or open-circuit potential (OCP) which may be due to the dissolution of the remaining oxides that are still present on the metal surface [20]. Table 2 shows that the free-corrosion potential of Aluzinc is between the value obtained for aluminum (-0.384 V) and that of zinc (-0.820 V). For a duration of 15 min, fluctuations are noted on the curves, which would seem to prove attacks on the substrate surfaces. Beyond this time, a stabilization of the curve is observed. This stabilization is justified by the destruction of the oxides and hydroxides and the possible formation of a thin layer of adsorbed inhibitor molecules. For a concentration of 6 g/L, the curve steepens after 15 min showing the effect of the self-protective layer. Like aluminum, there is for zinc the possibility to have a self-protective layer formed on the metal/electrolyte interface. In the absence of an inhibitor, the free-corrosion potential of zinc decreases significantly in the first few minutes. Its evolution eventually stabilizes over time after 15 min. (Fig. 2). We believe that at 2 g/L, the protective layer has broken down under the action of chloride ions. The Cl<sup>-</sup> ion is known to be destructive to passive films that can be formed at the metal/electrolyte interface [21, 22]. The chloride ion causes a localized corrosion phenomenon, such as pitting and stress corrosion cracking [23–25]. In the case of aluminum and aluzinc, OCP appears to be less fluctuating and is almost constant over time.

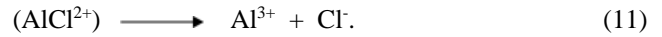
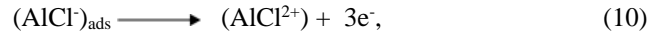
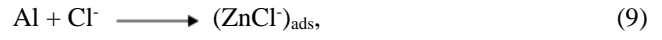
In hydrochloric acid solution, we can expect that the reaction mechanism of zinc or aluminum is similar to that of iron suggested by other authors [26, 27]. Therefore, the anodic oxidation of zinc by the Cl<sup>-</sup> can be represented by the following reactions:



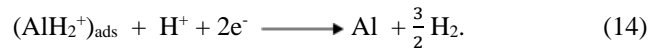
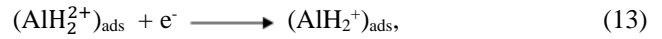
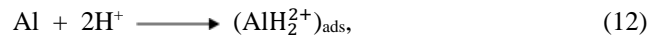
The cathodic reduction of hydrogen follows the following reactions:



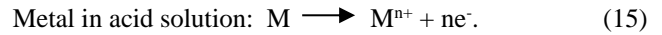
The same reaction schemes can describe the aluminum corrosion in protonated media. The anodic oxidation of zinc by the Cl<sup>-</sup>:



The cathodic reduction of hydrogen is given by the following reactions:



The introduction of attapulgite in the hydrochloric acid solution does not modify the mechanism of evolution and reduction of hydrogen on the surface [28] but acts simultaneously on the half-equations of oxidation and reduction by modifying the adsorption process.



The corrosion of Al, Zn and Al-Zn in HCl 0.5 M is globally manifested by the dissolution of the materials according to Eq. 15, any oxide or hydroxide being soluble in the highly acidic medium.

We reported in Table 2, for each material, the necessary time to reach a constant potential. The values are comprised between 90 and 120 min. Therefore, an equilibrium time of 2 h was chosen before dynamic polarization measurements.

### 3.2. Dynamic polarization

Corrosion is mainly an electrochemical process. The action of the inhibitor can only take place at one of the stages of the elementary reactions which are the transport of species in solution, the transfer of electronic charges, the absorption of species at the surface of solid phases, the formation of surface intermediates. The inhibitor acts according to the conditions in which it has been placed. Therefore, the additional inhibitor in hydrochloric acid must give such dynamic polarization behavior.

The dynamic polarization curve in the absence or presence of different concentrations of inhibitor is shown in Fig. 4, Fig. 5 and Fig. 6.

We observed that the cathodic branches are almost parallel, as well as the anodic ones. Therefore, a charge-transfer probably occurred between the electrolyte and the metal [27]. During this charge transfer, there is a shift in the corrosion potential from cathodic to anodic values. An inhibitor can be qualified as an anodic or cathodic type when the corrosion potential ( $E_{\text{corr}}$ ) shifts by  $\pm 85$  mV after adding an inhibitor.

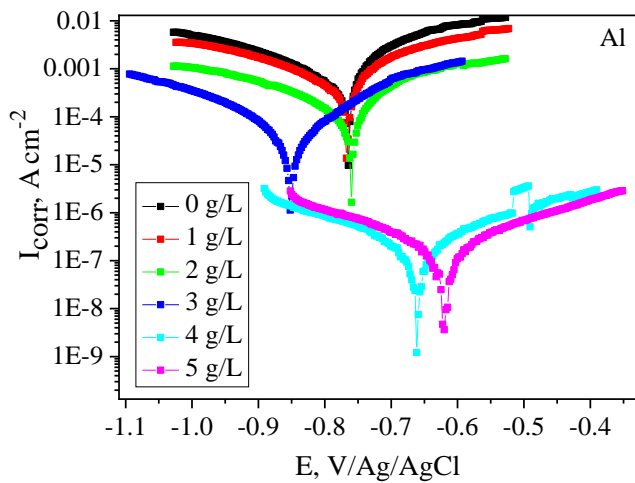


Fig. 4. Dynamic polarization curves of aluminum in a 0.5 M HCl solution

When the shift is less than 85 mV, the inhibitor is said to be mixed.

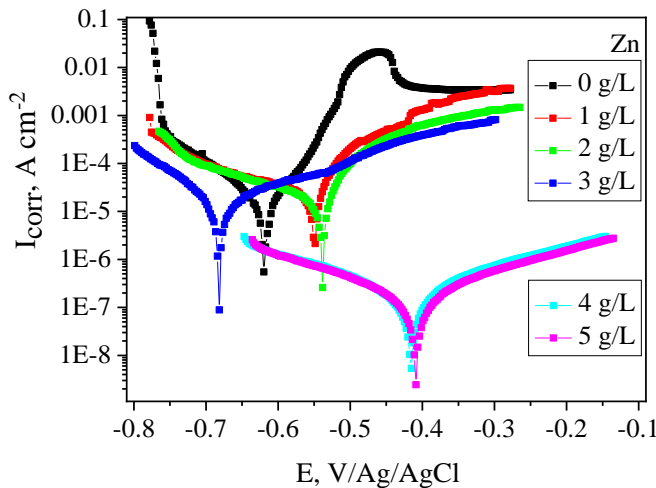


Fig. 5. Dynamic polarization curves of zinc in a 0.5 M HCl solution

In our case, there is a clear shift of the free-corrosion potential towards anodic values. The addition of 1 g/L of the inhibitor to the HCl solution causes a variation in corrosion intensity regardless of the smallest variation in the potential difference.

The variation of this current density can be due to the interactions between the  $Cl^-$  ions responsible for the attack and the Attapulgit responsible for inhibiting the progression of corrosion. In addition to the corrosion potential shift, the added inhibitor causes the corrosion current density to decrease significantly.

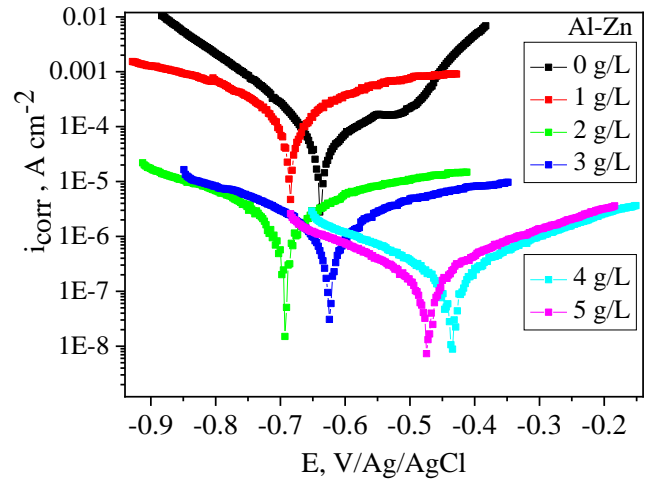


Fig. 6. Dynamic polarization curves of aluzinc in 0.5 M HCl solution

In the case of aluzinc, we noted a shift of the potential towards the more negative values for some inhibitor concentrations. This decrease in current density may be due to the formation of a protective film on the surface of the alloy rather than simple adsorption onto the active sites. The combined contribution of aluminum and zinc makes aluzinc good protection for attapulgit because it has almost the highest polarization resistance.

The corrosion parameters obtained by dynamic polarization of the three materials in a 0.5 M HCl solution at different inhibitor concentrations are grouped in Table 3.

Table 3. Dynamic polarization corrosion parameters of aluminum, zinc and aluzinc in 0.5 M HCl solution at different inhibitor concentrations. The results are obtained from Fig. 4, Fig. 5 and Fig. 6

$C, \text{g/L}$		0	1	2	3	4	5
$E_{\text{corr}}, \text{V}$	Al	-0.805	-0.851	-0.761	-0.777	-0.662	-0.621
	Zn	-0.621	-0.681	-0.538	-0.550	-0.416	-0.409
	Al-Zn	-0.638	-0.685	-0.694	-0.623	-0.422	-0.481
$i_{\text{corr}}, \mu\text{A}/\text{cm}^2$	Al	27.500	15.910	15.820	10.100	0.0561	0.0603
	Zn	8.739	3.842	2.377	2.301	0.773	0.0491
	Al-Zn	40.460	26.090	1.074	0.476	0.102	0.0804
$R_p, \Omega.\text{cm}^2$	Al	13.31	23.08	23.22	25.91	27100	10100
	Zn	59.33	124.5	234.2	239.0	45900	40300
	Al-Zn	32.89	272.1	5273	6302	46030	42000
$b_c, \text{mV}/\text{dec}$	Al	16	73	64	48	62	77
	Zn	27	75	30	52	85	64
	Al-Zn	34	54	114	84	112	75
$b_a, \text{mV}/\text{dec}$	Al	36	74	106	132	52	72
	Zn	22	49	88	229	97	67
	Al-Zn	23	42	114	81	97	106
$EI, \%$	Al	-	42.3	43.6	48.6	99.1	99.8
	Zn	-	75.2	52.3	74.6	99.87	99.84
	Al-Zn	-	87.9	99.3	99.4	99.93	99.92

The efficiency of the inhibitor is obtained from (Eq. 2). The reported values of the corrosion current density ( $I_{\text{corr}}$ ) are the average of three values obtained by drawing anodic and cathodic lines. Before 2 g/L of attapulgite, there is an attack activity justified by a value of the corrosion current density which varies in an increasing way. After 2 g/L passivation seems to be observed with a decrease of the corrosion current density. At 2 g/L the inhibitory efficiencies are 43.6 % for aluminum, zinc 52.3 % for and 99.3 % for aluzinc. For Zn and Al, attapulgite concentration should be equal or higher than 4 g/L to reach inhibitor effectiveness of 99 %. The very high percentage for aluzinc shows the existence of two combined effects. The effect due to the sacrificial anode protection played by zinc combined with the inhibiting effect of Attapulgite. For a concentration of 1 g/L for aluzinc the value 87.9 % of the inhibitory efficiency is due to the sacrificial oxidation of the anode.

#### 4. CONCLUSIONS

In hydrochloric acid environments, attapulgite provided fairly significant protection on these Al, Zn and aluzinc with optimum inhibitor concentrations of 2 g/L for aluzinc, and 4 g/L for Al and Zn in order to reach 99 % effectiveness. Partial blocking of the anodic areas by adsorption of the inhibitor can lead to an increase in the local current density. On the other hand, if the unblocked portions of the anode surface are not passivated, a pitting corrosion process is observed. This increase in current density cannot be due to a local attack on the metal surface. Attapulgite, which is not harmful to the environment, is a very effective inhibitor to protect these three metals. The experimental study showed that the inhibitory efficiency reached a maximum rate of about 99 % for these three materials.

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