

## Influence of inhibitors on corrosion and anodic behaviour of different grades of aluminium in alkaline media

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### Abstract

The electrochemical properties of different grades of aluminium are said to be dependent on the presence of minor amounts of iron and silicon impurities. This investigation aims mainly at addressing the influence of different inhibitors (e.g., sodium stannate, sodium citrate and calcium oxide combinations) on properties such as corrosion, anodic behaviour and anode utilization efficiency of different grades of aluminium in alkaline media.

### Introduction

The purity of aluminium metal varies from 99.0 to 99.9999%; the main impurities are iron and silicon. Aluminium and its alloys have been recognized as an excellent fuel material for electrical power production [1, 2], especially in an alkaline battery. Though the theoretical Ah capacity and theoretical open-circuit potential of aluminium and its alloys are as high as  $2980 \text{ Ah kg}^{-1}$  and  $-2.7 \text{ V}$  (versus standard hydrogen electrode, SHE), respectively, these values have not been realized completely in practice. The main reasons are a high corrosion rate and an appreciable anodic polarization. These effects cause a low anodic utilization efficiency of aluminium and its alloys in alkaline media [3–5]. The presence of silicon and iron in aluminium are major contributors to this decline in performance. Since this aspect has not been studied systematically, the work reported here is aimed at elucidating the role of the two elements by studying their electrochemical characteristics in NaOH solution both in the presence and in the absence of inhibitive and complexing agents. In order to employ aluminium as a galvanic anode in alkaline electrolyte, it is essential that its corrosion due to local-cell action is suppressed, without any hindrance to the anodic dissolution. This can be achieved by incorporating inhibitors, addition agents or complexing agents in the alkaline electrolytes [6–17]. Alternatively, different alloys of aluminium can be developed with improved electrochemical characteristics [18–20]. A large number of research papers have appeared on both these approaches. Not one of these papers, however, deals systematically with the role of silicon and iron in altering the electrochemical characteristics of different grades of aluminium.

This paper deals with the influence of the main impurities (such as iron and silicon) on the above-mentioned electrochemical properties of different grades of

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aluminium (99.0, 99.5 to 99.7, 99.8 and 99.9999%) in some of the successful alkaline battery electrolytes developed in the Central Electrochemical Research Institute (CECRI), India [13], namely:

- (i) 4 M NaOH solution with 0.2 to 0.5 wt.% CaO, 15 to 25 wt.% sodium citrate;
- (ii) 4 M NaOH solution with 0.2 to 0.5 wt.% CaO, 15 to 25 wt.% sodium citrate, 0.01 to 0.04 M sodium stannate;
- (iii) 4 M NaOH solution with 0.01 to 0.04 M sodium stannate;
- (iv) 4 M NaOH solution with 0.01 to 0.04 M sodium stannate, 0.2 to 0.5 wt.% CaO.

## Experimental

### *Preparation of electrode materials*

The specimens were prepared from the following grades of aluminium: (i) 99.0% pure (commercial purity); (ii) 99.5 to 99.7% pure (EC grade); (iii) 99.8 pure (commercial high purity); (iv) 99.9999% pure (spectroscopically pure).

The specimens (except those of spectroscopically pure aluminium) used for polarization studies were cylindrical rods of uniform diameter (0.6 cm) and cross-sectional area (0.8 cm<sup>2</sup>). They were cast by melting pieces of the respective grades of aluminium in a clean and fresh graphite crucible in a coke-fired furnace. The rods were embedded in Teflon rods, such that only a cross-sectional area of 0.8 cm<sup>2</sup> was exposed to the test solution. A copper rod of diameter 0.6 cm was inserted into the other end of the Teflon and made a permanent contact with the embedded aluminium. This copper rod served as the electrical lead for the working electrode. Spectroscopically pure aluminium rods were embedded directly in Teflon. The exposed side of the aluminium specimen was polished on a cloth wheel that was charged with pumice powder. The resulting polished surface was degreased with trichloroethylene before immersion in the test solution.

### *Chemicals*

Both the sodium hydroxide and the sodium citrate were of GR grade. The calcium oxide was of high purity. It was ignited for an extensive period to decompose any CaCO<sub>3</sub> contained in the sample, and then cooled and kept in a desiccator prior to use. The sodium stannate was of LR grade. The electrolyte solutions were prepared in conductivity water. The experiments were performed at 30 ± 0.2 °C. All the potentials were IR compensated.

### *Procedure*

#### *Measurement of open-circuit potential*

The open-circuit potentials (OCP) of different grades of aluminium in different electrolytes were measured by keeping the specimens in the respective solutions for about 30 min. During this time, the potential became steady and was measured using a Hg/HgO, 4 M NaOH reference electrode with the help of a digital multimeter (HIL 2161) with sensitivity of 0.1 mV.

#### *Determination of corrosion rate and overall corrosion-kinetic parameters by galvanostatic polarization technique*

The anodic and cathodic polarization characteristics of different grades of aluminium were determined by using the galvanostatic technique. The working electrode consisted

of a Teflon embedded, cloth buffed and degreased aluminium specimen, and the auxiliary electrode was a cylindrical gauze of stainless steel. A Hg/HgO-OH<sup>-</sup> electrode was used as reference electrode. All the three electrodes were held in their respective positions in a PVC lid. The electrode setup was kept immersed in 200 cm<sup>3</sup> of the test solution in a 250 cm<sup>3</sup> baker. The electrolyte solution was subjected to constant stirring by means of a magnetic stirrer. The polarization measurements were commenced after 30 min of immersion when the working electrode started to register a steady OCP.

Anodic and cathodic current densities in the range 0.03 to 300 mA cm<sup>-2</sup> were impressed on the working electrode. The corresponding (closed circuit) potentials of the working electrode with respect to the Hg/HgO-OH<sup>-</sup> electrode were measured 5 min after the application of each current density. All the experiments were repeated twice, or until good reproducibility of the current-potential data was established. The potential ( $E$ ) versus log current ( $i$ ) curves were plotted. From the above curves,  $E_{\text{corr}}$  and  $i_{\text{corr}}$  were computed using the Tafel extrapolation method. The anodic and cathodic Tafel slopes were also determined.

#### *Chemical analysis of different grades of aluminium*

Different grades of aluminium were analysed by means of an atomic absorption spectrometer (model 380 Perkin-Elmer, USA) using air/acetylene and nitrous oxide/acetylene flames. The percentage contents of iron and silicon in different grades of aluminium are given in Table 1.

## Results and discussion

### *Anodic and cathodic polarization data*

The polarization data pertaining to the four grades of aluminium in 4 M NaOH solution is shown in item A, Table 2. It is observed that the OCP value of aluminium decreases (i.e., becomes more positive) as the purity of aluminium decreases. Furthermore, both the  $E_{\text{corr}}$  and the  $i_{\text{corr}}$  values decrease with decrease in aluminium purity. The cathodic and anodic Tafel slopes indicate that the corrosion of all the grades of aluminium in 4 M NaOH is predominantly under anodic control. Further, it is seen that the anodic current sustaining capacity of different grades of aluminium increases as the purity of aluminium increases. This last parameter is listed in Table 2 as the anodic current density sustained by the aluminium electrode at -1.0 V (versus Hg/HgO-4 M NaOH).

Item B, Table 2 shows the electrochemical characteristics of different grades of aluminium in 4 M NaOH solution that contains sodium citrate and calcium oxide

TABLE 1

Content (wt.%) of silicon and iron in different grades of aluminium

Aluminium grade	Silicon	Iron
Spectroscopically pure	0	0
NALCO	0.055	0.08
EC Grade	0.078	0.17
Commercial 2S	0.16	0.80

TABLE 2

Electrochemical characteristics of different grades of aluminium in different electrolytes

Serial number	Aluminium grade	OCP (V)	$i_{\text{corr}}$ (mA cm <sup>-2</sup> )	$E_{\text{corr}}$ (V)	$b_a$ (mV/decade)	$b_c$ (mV/decade)	Current sustained by the anode at -1.00 V (mA cm <sup>-2</sup> )
A: 4 M NaOH							
1	Spec. pure 99.9999% pure	-1.8	3	-1.75	200	-190	282
2	NALCO 99.8% pure	-1.471	6	-1.465	130	-80	167
3	EC grade 99.5 to 99.7% pure	-1.437	9	-1.365	135	-90	168
4	Commercial aluminium-2S 99% pure	-1.350	13	-1.350	80	-65	164
B: 4 M NaOH solution with 0.4% CaO and 20% sodium citrate							
1	Spec. pure 99.9999% pure	-1.67	2.0	-1.78	145	-110	106
2	NALCO 99.8% pure	-1.5	2.5	-1.450	360	-120	83
3	EC grade 99.5 to 99.7% pure	-1.491	3.2	-1.430	190	-130	80
4	Commercial aluminium-2S 99% pure	-1.425	6.0	-1.410	120	-85	73
C: 4 M NaOH solution with 0.02 M sodium stannate							
1	Spec. pure 99.9999% pure	-1.63	2.5	-1.63	175	-80	230
2	NALCO 99.8% pure	-1.530	4.0	-1.475	105	-80	159
3	EC grade 99.5 to 99.7% pure	-1.344	8.0	-1.360	190	-85	162
4	Commercial aluminium-2S 99% pure	-1.305	12.0	-1.290	140	-85	145
D: 4 M NaOH solution with 0.4 wt.% CaO and 0.02 M sodium stannate							
1	Spec. pure 99.9999% pure	-1.806	1.5	-1.81	215	-140	176
2	NALCO 99.8% pure	-1.597	3.8	-1.545	160	-150	158

(continued)

TABLE 2 (continued)

Serial number	Aluminium grade	OCP (V)	$i_{\text{corr}}$ (mA cm <sup>-2</sup> )	$E_{\text{corr}}$ (V)	$b_a$ (mV/decade)	$b_c$ (mV/decade)	Current sustained by the anode at -1.00 V (mA cm <sup>-2</sup> )
3	EC grade 99.5 to 99.7% pure	-1.325	7.5	-1.308	205	-185	125
4	Commercial aluminium-2S 99% pure	-1.305	9.0	-1.300	160	-150	110
E: 4 M NaOH solution with 0.4 wt.% CaO, 20% sodium citrate and 0.02 M sodium stannate							
1	Spec. pure 99.9999% pure	-1.820	1.0	-1.38	150	-105	159
2	NALCO 99.8% pure	-1.597	1.5	-1.250	130	-120	158
3	EC grade 99.5 to 99.7% pure	-1.580	3.6	-1.238	230	-140	125
4	Commercial aluminium-2S 99% pure	-1.284	5.0	-1.205	140	-140	110

(i.e., alkaline citrate solution). The data show that the OCP of aluminium decreases as the percentage contents of iron and silicon increase. The general trend is exhibited by the different parameters (i.e.,  $i_{\text{corr}}$ ,  $E_{\text{corr}}$ , OCP,  $b_a$  and  $b_c$ ) of different grades of aluminium in alkaline citrate solution.

As the content of iron and silicon in aluminium increases, the OCP values decrease in both 4 M NaOH and the alkaline citrate solutions. In all but the spectroscopically pure grade of aluminium, the OCP values are higher in the alkaline citrate solution than in 4 M NaOH. With the increase of iron and silicon contents,  $E_{\text{corr}}$  values decrease both in 4 M NaOH and in the alkaline citrate solution. The  $i_{\text{corr}}$  values also follow the same trend. For all the grades of aluminium, the  $i_{\text{corr}}$  values are higher in 4 M NaOH than in alkaline citrate solution. The  $b_a$  and  $b_c$  values of all the grades of aluminium in the alkaline citrate solution reveal a predominant anodic control in the overall kinetics of corrosion, as observed in the case of 4 M NaOH solution.

It is quite clear from Table 2 that the anodic current sustaining capacity decreases as the amount of silicon and iron impurities increase in aluminium. In other words, spectroscopically pure aluminium is found to sustain a higher anodic current density than the 99.0% pure aluminium in alkaline citrate medium. While comparing the two media, each grade of aluminium shows a much lower anodic current sustaining capacity in the alkaline citrate solution than in the plain 4 M NaOH solution.

Previous work [11] has shown that a combination of calcium and sodium citrate is a synergistic inhibitive system for aluminium in 4 M NaOH. In fact, it has been proposed that calcium forms a complex with citrate and that this complex, in turn, maintains the pH in the layer of the solution near to the aluminium electrode at

about 11.5 to 11.8. In short, a buffer theory has been proposed for the actual mechanism. (Adsorption of calcium in presence of citrate is practically zero [21] and, hence, an adsorption theory is not valid for the inhibition offered by the calcium citrate synergistic system.) The same buffer theory can be extended to the observed inhibition of corrosion of different grades of aluminium in alkaline citrate solution.

Kato and Kerichi [22] used sodium stannate as a successful inhibitor for preventing the corrosion of aluminium (99.99% pure) in highly alkaline (about 4 M NaOH) media. In the USA, researchers working on the development of the aluminium/air battery have reported [23] that a 4 M NaOH based alkaline medium containing aluminium oxide and sodium stannate is the most successful electrolyte.

It was thought appropriate that the combination of  $\text{Ca}^{2+}$  ions and sodium stannate could be tried as a possible synergistic system similar to the calcium citrate system. Hence, experiments were carried out to determine the electrochemical characteristics of different grades of aluminium in 4 M NaOH solution containing  $\text{Ca}^{2+}$  or stannate ions alone, as well as  $\text{Ca}^{2+}$  and stannate ions in combination.

Items C and D in Table 2 present the data obtained with 0.02 M sodium stannate and 0.4 wt.% CaO + 0.02 wt.% sodium stannate, respectively. The results show that the OCP and  $E_{\text{corr}}$  values of aluminium decrease as the impurity contents increase. The  $i_{\text{corr}}$  values of all grades of aluminium decrease in the presence of sodium stannate. The  $E_{\text{corr}}$  values show a similar trend. In the case of 99.8% pure aluminium, however, both the OCP and  $E_{\text{corr}}$  values are higher in 4 M NaOH in the presence of sodium stannate than in 4 M NaOH. The  $i_{\text{corr}}$  values increase as the impurities in aluminium increase.

In general, the  $b_a$  and  $b_c$  values decrease with increasing iron and silicon contents in aluminium in 4 M NaOH in the presence of sodium stannate (item C, Table 2). Furthermore,  $b_a \gg b_c$  for all the grades of aluminium in 4 M NaOH in the presence of sodium stannate. The anodic current sustaining capacities of all the grades of aluminium are only slightly different in 4 M NaOH in the presence and absence of sodium stannate. As in previous cases, it is generally observed that OCP values of aluminium in 4 M NaOH + stannate also decrease with increasing levels of iron and silicon. The OCP values of the four grades of aluminium in this medium are comparable with those in uninhibited 4 M NaOH medium. The  $i_{\text{corr}}$  and  $E_{\text{corr}}$  values show the same behaviour as that of OCP. In quantitative terms, however, the  $i_{\text{corr}}$  values of all grades of aluminium are lower in the stannate medium. By contrast, the extent of polarization (i.e.,  $b_a$  and  $b_c$ ) is larger. The differences between the actual  $b_a$  and  $b_c$  values of all grades of aluminium in the stannate medium, is smaller than in other media. The anodic current sustaining capacities at  $-1.0$  V are comparable with those in 4 M NaOH solution for the four grades of aluminium, and the actual anodic current densities at  $-1.0$  V are found to decrease as the impurities increase. Here, the synergism between sodium stannate and  $\text{Ca}^{2+}$  ions may be invoked because of the inhibitive nature of the CaO–sodium stannate combination in the case of all the grades of aluminium in 4 M NaOH solution. The calcium–stannate synergistic inhibitive combination reduces the corrosion of all the grades of aluminium in 4 M NaOH by a predominant anodic inhibition.

Since independently calcium–citrate and calcium–stannate are synergistic inhibitors for all grades of aluminium in 4 M NaOH, it is not unreasonable to expect that, by further combining these two systems, an even better inhibition efficiency will be achieved. With this aim, polarization experiments were performed to determine the electrochemical parameters of different grades of aluminium in 4 M NaOH containing CaO, sodium citrate and sodium stannate (item E, Table 2). The results show that there is a marked

synergism between the calcium citrate and calcium stannate systems with regard to the  $i_{\text{corr}}$  values.

Based on all the properties examined above, the order of performance among different grades of aluminium is: spectroscopic pure Al > 99.8% pure Al > EC grade Al > commercial grade Al. Since the highest purity of aluminium produced in India is 99.8% (from NALCO) and this gives relatively good performance as an anode for an alkaline battery, this grade of aluminium has been considered the best-suited material for anode development.

The following interesting observation has been made with regard to the influence of impurities on the  $b_a$  and  $b_c$  values of different grades of aluminium in 4 M NaOH, both in the presence and the absence of inhibitors such as CaO, sodium citrate and/or sodium stannate. For example, in 4 M NaOH,  $b_a > b_c$  for all the grades of aluminium. The difference between  $b_a$  and  $b_c$ , however, increases as the iron and silicon contents increase in the following order (with the exception of commercial aluminium):

$b_a - b_c = 200 - 190 <$	$b_a - b_c = 130 - 80 <$	$b_a - b_c = 135 - 40;$	$b_a - b_c = 80 - 65$
10 mV	50 mV	95 mV	15 mV
spectroscopically pure Al <	99.8% pure Al <	EC grade Al;	commercial Al

In the presence of sodium citrate and/or sodium stannate and CaO in 4 M NaOH, the  $b_a$  values are always greater than the  $b_c$  values for all grades of aluminium. The difference between the  $b_a$  and  $b_c$  values does not diverge with increase in the concentrations of impurities such as iron and silicon when the additives are present in 4 M NaOH.

In alkaline citrate with stannate medium, the  $b_a - b_c$  values show the following trend:

spectroscopically pure Al	99.8% pure Al	EC grade Al	commercial Al
150-105 = 45 mV	130-120 = 10 mV	230-140 = 90 mV	140-140 = 0 mV

Thus, there is no uniform rise or fall in the  $b_a - b_c$  values. It should be noted, however, that the commercial grade (which contains the maximum amount of iron and silicon) exhibits a very low  $b_a - b_c$  value, namely, 0 mV. In other words, the corrosion of this grade of metal is shifted from a predominant anodic control to one of mixed control. Perhaps this is one of the reasons for the better electrochemical properties of 99.8% aluminium in 4 M NaOH, especially in the combined presence of sodium citrate and sodium stannate and CaO.

In alkaline citrate, alkaline stannate + CaO and in alkaline stannate solutions, the  $b_a - b_c$  values display the following trend:

spectroscopically pure Al	99.8% pure Al	EC grade Al	commercial grade Al	
145-110 = 35 mV	360-120 = 240 mV	190-130 = 60 mV	120-85 = 35 mV	(1)
215-140 = 75 mV	160-150 = 10 mV	205-185 = 20 mV	160-150 = 10 mV	(2)
175-80 = 95 mV	105-80 = 25 mV	190-85 = 105 mV	140-85 = 55 mV	(3)

where (1) represents alkaline citrate solution; (2) represents alkaline stannate + CaO solution, and (3) represents alkaline stannate solution.

Thus, based on all the measured electrochemical properties, 99.8% pure aluminium can be chosen as the best grade of aluminium, and the best medium is 4 M NaOH solution that contains sodium stannate, sodium citrate and CaO. The most important

reason for this choice is that the  $b_a - b_c$  value is the least; this indicates a shift in the mechanism of corrosion of 99.8% pure aluminium from a predominantly anodic control in 4 M NaOH alone to one of mixed control in the same solution in the combined presence of  $\text{Ca}^{2+}$ , citrate and stannate ions. The iron and silicon contents of 99.8% pure aluminium are roughly the same, perhaps this is the reason for its better electrochemical characteristics as reflected, especially, by the  $b_a - b_c$  values. Hence, this study leads to the conclusion that the 99.8% pure aluminium is the most suitable starting material (especially under Indian conditions) for the preparation of better alloy anodes for alkaline batteries using alkaline citrate plus stannate electrolyte based on 4 M NaOH. This observation has been verified by the development of a special quaternary alloy of 99.8% pure aluminium with indium, gallium and lead for use as galvanic anode in the alkaline citrate plus stannate electrolyte of an aluminium/air battery [13, 24].

### Conclusions

1. The electrochemical characteristics of the four grades of aluminium show a decreasing trend as the concentration of impurities in the aluminium increases.

2. Spectroscopically pure aluminium shows the best electrochemical characteristics among the different purities of aluminium that have been examined.

3. Among the inhibitors studied for the different grades of aluminium in 4 M NaOH, the sodium citrate–sodium stannate–CaO combination exhibits by far the best electrochemical properties.

4. All the grades of aluminium, irrespective of their differences in iron and silicon contents, show that their overall corrosion kinetics are predominantly under anodic control in 4 M NaOH solution.

5. As the concentrations of iron and silicon increase in aluminium, the  $i_{\text{corr}}$  rate increases, the OCP and  $E_{\text{corr}}$  values shift towards more positive values, and the anodic and cathodic Tafel slopes decrease in plain 4 M NaOH solution.

6. In the presence of inhibitors,  $b_a$  and  $b_c$  are changed to such an extent that in some cases there is a shift in the mechanism of corrosion from a predominantly anodic control to a mixed control. This is very much so in the combined presence of sodium citrate, sodium stannate and CaO in 4 M NaOH, especially in the case of 99.8% pure aluminium.

7. The best grade of aluminium is the spectroscopically pure variety, but, from the practical point of view, the appropriate choice is a combination of 99.8% pure aluminium with an electrolyte based on 4 M NaOH containing stannate, citrate and  $\text{Ca}^{2+}$  ions.

8. The joint presence of  $\text{Ca}^{2+}$ , citrate and stannate ions in 4 M NaOH gives rise to a synergistic inhibitive system for 99.8% pure aluminium. This synergism is reflected in all the measured electrochemical characteristics, such as  $i_{\text{corr}}$ ,  $E_{\text{corr}}$ , OCP,  $b_a$ ,  $b_c$  and the anodic current sustaining capacity.

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