DETERMINATION OF THE IRON CONTENT OF ALUMINATE LIQUORS FROM THE BAYER ALUMINA PROCESS BY NEUTRON ACTIVATION ANALYSIS

E. Bujdosó, M. Miskei

Research Institute for Non-Ferrous Metals, Budapest (Hungary)

(Received August 27, 1971)

The behaviour of iron in the Bayer process has been investigated by neutron activation analysis and Ge(Li) spectrometry, using bauxite samples of various origin. The amount of iron impurities in alumina was found to be independent of the origin of the bauxites. Alumina is contaminated by iron in the process of decomposition of aluminate liquors; regardless whether iron is present in 'dissolved' form or as a floating impurity, it will pass into the alumina almost completely.

Introduction

One of the main impurities in alumina produced by the Bayer process is iron, whose concentration varies between 0.02 and 0.06 %. Taking into account the characteristics of the domestic technologies and assuming that the impurity of alumina originates solely from the iron content of the aluminate liquor, the above value corresponds to 12–72 mg/l of Fe₂O₃ in the aluminate liquor before decomposition.

According to generally accepted views, the iron content of aluminate liquors consists of two components in different states. Part of the iron is present in the form of floating red mud particles due to imperfect separation of the liquid and solid phases. In principle, this can be removed by careful filtration. The residual iron content after filtration is in practice referred to as 'dissolved' iron. As to its chemical form and nature, only assumptions are available at present.

There are no reliable experimental data on the factors determining the amounts and ratio of these two kinds of iron impurity. The determination of dissolved iron is particularly uncertain because its amount barely exceeds the sensitivity of chemical analysis.

Practical observations indicate that the Fe₂O₃ content of the settled aluminate liquor differs even in plants using identical technologies. This fact is attributed, besides differences in the sedimentation technology, to the varying quality of bauxites processed. For example, it is assumed that the amount of dissolved iron in the aluminate liquor increases with the increasing FeO content of the bauxite.

The presence of dissolved iron in aluminate liquors was ascribed by Dunay and coworkers¹ to the possibility of ferrate formation. However, this assumption...
has not been proved and, anyway, the formation of ferrate ions under the conditions of digestion is unlikely and doubtful. The preparation of Fe(VI) ions requires special conditions and their existence in the aluminate liquor containing various organic substances is highly unlikely.\textsuperscript{5}

On the other hand, iron is known to form complex ions in alkaline media. For example, in carbonate solutions the complex $\text{Na}_2\text{Fe(CO}_3\text{)}_3$ is assumed to be present,\textsuperscript{3} whereas in solutions of alkali the $\text{Fe(OH)}_\text{4}$ species may be formed.\textsuperscript{4} Dissolved iron in aluminate liquors cannot be attributed to the very slight solubility of either ferrous or ferric hydroxide.\textsuperscript{5} However, no data are available on the formation and stability of iron complexes in the aluminate liquor.

The aim of the present work was to determine the amount of floating and dissolved iron from various types of bauxites by means of the highly sensitive neutron activation technique, and to follow the iron content of aluminate liquors in various phases of the alumina production.

Digestion of bauxite, sedimentation of the red mud and decomposition of the aluminate liquor are performed in highly concentrated solutions of alkali. Direct activation analysis of the samples taken from the above phases is rendered impossible by the high activity of $\text{Na}^{24}$ formed in the nuclear reactions $\text{Na}^{23}(n, \gamma)\text{Na}^{24}$ and $\text{Al}^{27}(n, \alpha)\text{Na}^{24}$. Therefore, bauxite samples taken prior to digestion were irradiated in the reactor. Thus it was ensured that the chemical and mineralogical composition of $\text{Fe}^{56}$ were identical with those of the iron containing minerals in the bauxite.

**Experimental**

**Sample preparation**

4—7 g bauxite samples of Hungarian and foreign origin were irradiated at a neutron flux of $2 \times 10^{13} \text{n} \cdot \text{cm}^{-2} \cdot \text{sec}^{-1}$ together with an iron standard. In order to achieve the decay of short-lived isotopes and of $\text{Na}^{24}$ formed in the reaction $\text{Al}^{27}(n, \alpha)\text{Na}^{24}$ from the Al content of bauxite, the samples were cooled for about 800 hrs, then brought to a total weight of 30—35 g by adding inactive bauxite.

The bauxite samples were digested in autoclaves at 240 °C for 1 hr, with an aluminate liquor containing 204 g/l of $\text{Na}_2\text{O}$ and 96.5 g/l of $\text{Al}_2\text{O}_3$.

After digestion, the suspension of red mud was diluted to 145 g/l of $\text{Na}_2\text{O}$, then the usual flocculating agent was added and the suspension settled in a tube. After sedimentation, the aluminate liquor was filtered off. Half of the solution obtained was filtered repeatedly through a cotton-wool plug. Decomposition was carried out by adding crystalline aluminium hydroxide (technical grade) at a seed ratio of 2, and the liquor was decomposed isothermally at 40 °C. Samples were taken from the liquid phases both at the beginning and end of each operation. Thus the samples permitted to determine the concentration of floating + dissolved and of dissolved iron in the aluminate liquor before and after the decomposition.

*J. Radioanal. Chem.* 11 (1972)