



Dissolution of sodium, aluminum and caustic compounds from bauxite residues



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ABSTRACT

Bauxite residues consist of solid particles and a strongly alkaline liquid. Depending on the selected disposal method, the solids content of washed bauxite residues varies from 30 to 70 w-%. The objective of this study is to provide better understanding of the leaching of metals and caustic compounds from bauxite residues. These issues are receiving more attention in the alumina industry, not only due to the need to recover the valuable compounds more effectively, but also due to the environmental impacts caused by leaching of soluble metals and alkali from bauxite residue disposal areas. Three different bauxite residues of industrial origin were investigated by straightforward leaching and dilution methods by using water as the diluent. In the analysis stage, dilution with a nitric acid solution was also tested. The main dissolved compounds were analyzed with atomic absorption spectroscopy and thermometric titration. The results showed that the sodium balance was influenced by the applied *L/S* ratio, which implies that the solid particles were partially dissolved. Comparison of the dissolution behavior of sodium, aluminum and total caustic (i.e. the hydroxyl ion content) revealed changes in the Al/Na and caustic/Na ratios, which deserve to be studied further in the future. Grinding the particles did not increase the liberation of sodium from the solids, i.e. the role of captured sodium did not seem to be important.

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1. Introduction

The production of alumina from bauxite ore by the Bayer process generates large quantities of highly alkaline waste called bauxite residue, which is of serious environmental concern. The desanded bauxite residue is separated from the sodium aluminate liquid by a series of gravity thickeners with polymer addition to increase the settling velocity (Kirwan, 2009; Sidrak, 1997). These thickeners are typically operated according to the counter-current principle (Pejcinovic et al., 2007), in order to minimize the loss of alkali and aluminate with the bauxite residue. The composition of the bauxite residue depends on many factors, such as the quality of the bauxite ore and the Bayer process parameters (Jankovic et al., 2013; Mayes et al., 2011; Zhang et al., 2009).

It is estimated that approximately 120 million tons of bauxite residue are produced in the world annually (Milacic et al., 2012; Power et al., 2011; Rubinos and Barral, 2013). Thus it is evident that bauxite residue is currently a major concern in the production of alumina (Liu et al., 2013). The disposal methods of bauxite residue have improved greatly during the last decades. The disposal of

the slurry into the marine environment has practically ended, wet stacking in large ponds is still applied, and dry disposal methods have gained popularity (Boger, 2012; Cooling, 2007; Fourie, 2009; Green and Boger, 1997; Jones and Boger, 2012; Nguyen and Boger, 1998). Dry stacking methods are currently the recommended practices for the disposal of bauxite residues (Kirwan et al., 2013; Hind et al., 1999). In spite of the benefits of dry stacking, leaching of harmful compounds from the disposed residue remains a challenge which can be partially resolved by washing the bauxite residue carefully prior to disposal. Understanding of the dissolution phenomena should therefore be increased.

A number of different utilization methods for bauxite residues have been proposed. The most potential applications are the use as construction material (Samal et al., 2013; Pontikes and Angelopoulos, 2013), ceramic materials (He et al., 2012), and various water treatment applications (Atun and Hisarli, 2000; Liu et al., 2009; Poulin et al., 2008). In these cases, neutralization of the residue is typically required to make it more compatible with the environment (Wang et al., 2008; Agatzini-Leonardou et al., 2008). The recovery of valuable compounds, such as metal oxides, has also been considered (Brunori et al., 2005). Although bauxite residues seem to be potentially applicable for many purposes, their utilization has not become commercially significant (Clifton et al., 2007;

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Lee and Pandey, 2012). The neutralization can be performed with seawater (Johnston et al., 2010), CO₂ (Sahu et al., 2010; Bonenfant et al., 2008; Dilmore et al., 2008), or various acidic salts (Hamdy and Williams, 2001).

It has been observed that harmful components may also leach from bauxite residues. In addition to alkaline compounds such as NaOH (Liu et al., 2007), the leaching of heavy metals, such as Cr, Cu, Ni, and Pb, can cause problems (Ghosh et al., 2011; Rubinos and Barral, 2013), although the leachable alkali may be of a greater concern in some cases (Kutle et al., 2004). The removal of (heavy) metals can be carried out by chemical or biological leaching methods (Qu et al., 2013; Vachon et al., 1994). Leaching of alkalinity from the stock may continue for a long time, which makes it important to collect and treat the leachate properly (Kirwan et al., 2013).

The caustic compounds which are to be recovered as effectively as possible, are present either in the mother liquor or in the bauxite residue solids, desilication products in particular (Thornber and Binet, 1999). The recovery of the valuable caustic and dissolved aluminum compounds can be performed by dewatering and washing the bauxite residue in various filter units (Arslan et al., 2012; Borges et al., 2011; Kinnarinen et al., 2012). The main motivation for the present study comes from a previous pressure filtration study of the authors (Kinnarinen et al., 2013) and analytical procedures performed for two other studies (Kinnarinen et al., 2015, 2012), where it was observed that the total amount of sodium and aluminum removed from bauxite residue solids is significantly affected by the applied pressure difference, temperature, and wash ratio. On the basis of mass balances, it was clear that also the so called insoluble solids were partially dissolved in the process. This observation could be explained by liberation of soluble sodium from the pores of the particle flocs, dissolution of particles due to the reduction of the pH, and simply by changes in the tendency of the investigated compounds to remain adsorbed on the surfaces of the particles. The importance of these phenomena can be investigated by breaking down the particle flocs by mechanical shear and by dilution of the bauxite residue slurry with pure water.

The main objective of this study was to evaluate the dissolution of different bauxite residue solids by diluting the slurry with water to see how the dissolution is affected by the dilution ratio. This evaluation was primarily based on the concentration of dissolved sodium, but also on the concentrations of dissolved aluminum and total caustic and total soda as defined below in Section 2.3. The original and diluted bauxite residue samples were ground in a planetary ball mill to investigate the role of breakage of particle flocs in the dissolution of sodium from the solids. On one hand, the dissolution phenomena were evaluated with respect to the particle size distribution of solids, and on the other hand, in relation to the abovementioned dissolved compounds in the liquid phase. Atomic absorption spectroscopy and thermometric titration were applied for the analysis of the dissolved compounds. The results of this study help to understand the importance of appropriate washing of bauxite residue prior to disposal, and to illustrate the risks resulting from improper stacking of the residue.

2. Materials and methods

2.1. Characteristics of the slurries

Three bauxite residue slurries obtained from industrial alumina production sites were investigated.

In this paper, the slurries are called Slurry I, Slurry II, and Slurry III. The slurries were obtained from different origins in three continents. All the slurry samples were taken from the underflow of the last wash thickener of the residue washing train. The slurry

samples were pumped in air-tight plastic barrels, which were then stored at ambient temperature during transportation to the laboratory. The slurries were homogenized again in a baffled mixing tank equipped with a pitched-blade turbine and a recirculation pump. Small samples for the experiments were taken from the recirculation pipeline after several hours of mixing.

The caustic content of the slurries was relatively high, as the pH values shown in Table 1 indicate. Some other essential characteristics of the slurries are also presented in Table 1. The methods of analysis are described below.

The pH values of the slurries were measured from supernatants of clarified samples with a WTW pH 340i pH meter and WTW SenTix 41 electrode. The total solids contents (TS) of the slurries were measured by drying the slurry samples at 105 °C to dryness. The total dissolved solids contents (TDS) were measured by drying clear filtrate samples obtained by filtering the slurries through Whatman #42 filter paper at 180 °C to dryness. The density of each slurry was measured simply by measuring the mass of the sample with a certain volume in a graduated cylinder. The procedures of TS and TDS measurement were repeated twice and the averages were calculated.

The sodium concentrations c_{Na} were measured with atomic absorption spectroscopy (AAS) by using a Thermo Scientific iCE 3000 atomic absorption spectrometer. To prepare a sample for a standard Na analysis, the slurry sample was centrifuged, and the clear supernatant was filtered through a syringe filter with a nominal pore size of 0.2 μm. Another analysis procedure was also used in order to examine how much sodium is dissolved when the sample is diluted with a 14 w-% HNO₃ solution. The dilution ratio in these cases was 100 g of HNO₃ per one gram of original slurry in the sample. After this acid dilution stage, the analysis procedure was the same as for the standard Na analyses described above.

Particle size distributions (PSD) of the original slurries and the samples taken during the experiments were measured with a Malvern Mastersizer 3000 laser diffraction particle size analyzer. The particle size analyses were performed without sonication and without dispersing agents, at a constant stirring rate of 3000 rpm. The range of measurement, according to the manufacturer, was 0.01–3500 μm. Each sample was measured five times, and the analyzer calculated the average PSD automatically.

The elemental composition of the slurry was measured with a Jeol JSM-5800 SEM-EDS (scanning electron microscope with an energy-dispersive X-ray spectrometer). Prior to SEM-EDS analyses, the slurry samples were dried in an oven to obtain solid residues consisting of all suspended and dissolved solids present in the slurry. Each sample was analyzed at several different locations, and the average concentrations of each element were calculated. The results of the SEM-EDS analyses are shown in Table 2. It can be seen in Table 2 that the titanium content of Slurry I is significantly higher than that of Slurries II and III, whereas the iron content of Slurry I is correspondingly lower. Moreover, Slurry II has the lowest sodium content but the highest concentration of calcium. The mineralogical compositions of several different bauxite residues, analyzed by XRF, have been reported in the literature, for instance by Gräfe et al. (2011).

Table 1
Characteristics of bauxite residue slurries.

Slurry	pH (-)	TS ^a (g/kg)	TDS ^b (g/kg)	Density (kg/m ³)	c_{Na} ^c (g/kg)	D_{10} (μm)	D_{50} (μm)	D_{90} (μm)
Slurry I	13.20	442	52	1363	27.2	0.87	6.9	235
Slurry II	12.92	330	8	1290	2.3	0.90	2.6	17
Slurry III	13.46	483	86	1450	33.3	0.90	3.9	27

^a Total solids in slurry.

^b Total dissolved solids in the liquid phase of slurry.

^c Concentration of Na in the liquid phase of slurry.

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