



Leaching of rare earths from bauxite residue (red mud)



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ABSTRACT

Bauxite residue (or red mud) is a waste generated during the Bayer process of alumina production. Its storage is a spatial and environmental concern. Currently, there are no bulk applications of bauxite residue except for minor use in cements and ceramics. Nonetheless, some types of bauxite residues are rich in rare-earth elements (REEs), and the extraction of scandium in particular is of special interest. Leaching experiments on Greek bauxite residue were performed with different acids at different concentrations, liquid-to-solid ratios, leaching times and temperatures. Extraction of the REEs was high for leaching in HCl solutions compared to other acids, but the dissolution of iron was high as well (~60%). The maximum extraction of the REEs was around 80%. Sodium and calcium were completely dissolved during leaching. Dissolution of aluminum, silicon and titanium was between 30% and 50%. The leaching data show a very close association of scandium with the iron oxide phases.

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1. Introduction

Bauxite is the major ore for aluminum production. Alumina is extracted from bauxite by the Bayer process. The waste slurry generated in this process is called red mud and its solid fraction is called bauxite residue. About 1–2 metric tonnes of bauxite residue is generated for each tonne of alumina produced (Kumar et al., 2006). The global annual generation of bauxite residue is around 120 million tonnes and 2.7 billion tonnes of this material has already been stockpiled (Klauber et al., 2011). The pH of the wet red mud slurry is about 12. It is stored in huge tailing ponds and this poses a significant problem, since it not only occupies vast land areas, but it can also lead to environmental pollution (Power et al., 2011). Hence, there is a need for better management strategies (preferably utilization) of the bauxite residue. However, currently there are not many large-volume applications of bauxite residue besides minor use in cement and ceramic production (Binnemans et al., 2013; Klauber et al., 2011; Kumar et al., 2006; Pontikes and Angelopoulos, 2013). Nonetheless, bauxite residue contains several interesting minor or trace elements, among which the rare-earth elements (REEs). More than 90% of the trace metal value in bauxite residue can be attributed to the presence of one of these REEs, namely scandium (Binnemans et al., 2013). Considering the revived interest on REEs

due to supply concerns, novel research in the area is likely to take place.

Bauxite ores are classified in three different ways based on: (1) genetic principles: (1a) bauxites on igneous and metamorphic rocks; (1b) bauxites on sediments: carbonate rocks and clastic strata; (2) geological age: (2a) Palaeozoic; (2b) Mesozoic; (2c) Cenozoic; and (3) mineralogical composition: (3a) gibbsite bauxites; (3b) boehmite bauxites; (3c) diasporic bauxites (Valeton, 1972). The bauxites on carbonate rocks (karst) have higher REE concentrations compared to other bauxites (Mordberg, 1993). Karst bauxites are mainly located in Europe, Jamaica, Russia and China. These bauxites account for 14% of total bauxite reserves (Bárdossy, 1982). The REEs report to bauxite residue in the Bayer process. The enrichment factor of the REEs in bauxite residue compared to bauxite is about two (Ochsenkühn-Petropulu et al., 1994). REEs are present in bauxite as mineral phases or as ions that are either absorbed on the surface of minerals or replacing similar ions in the lattice of some matrix minerals (Li et al., 2013).

Some literature data on the leaching of REEs from bauxite residue are available (Fulford et al., 1991; Ochsenkühn-Petropulu et al., 1996; Ochsenkühn-Petropoulou et al., 2002; Qu and Lian, 2013; Smirnov and Molchanova, 1997; Wang et al., 2011, 2013; Xue et al., 2010; Yatsenko and Pyagai, 2010; Zhang et al., 2005). Fulford et al. (1991) developed a process for the extraction of REEs from Jamaican bauxite residue using sulfur dioxide, whereby REEs are selectively dissolved while leaving iron and titanium substantially undissolved in the bauxite residue. Ochsenkühn-Petropulu et al.

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(1996) compared metal leaching extraction with different acids, different acid concentrations and different leaching conditions. Their results showed that 0.5 N HNO₃ leaching at 25 °C for 24 h with a liquid-to-solid (*L/S*) ratio of 50:1 recovered 80% of scandium, 90% of yttrium, 70% of the heavy lanthanides (Dy, Er, Yb), 50% of the middle ones (Nd, Sm, Eu, Gd) and 30% of the light lanthanides (La, Ce, Pr). The leaching process with dilute HNO₃ was performed at pilot scale and optimized for the extraction of scandium from bauxite residue (Ochsenkühn-Petropoulou et al., 2002). Xue et al. (2010) found that at a reaction temperature of 90 °C and an *L/S* ratio of 3:1, over 80% of the scandium in bauxite residue could be leached by H₂SO₄. Zhang et al. (2005) recovered 80% of scandium by HCl leaching at acid concentration of 6 mol/L HCl, *L/S* ratio of 4:1, temperature of 50 °C and a reaction time of 1 h. Wang et al. (2010) studied the extraction of scandium from bauxite residue by using HCl as leaching agent, with an *L/S* ratio of 5:1, an acid concentration of 6 mol/L, a reaction temperature of 50 °C and a reaction time of 1 h. Qu and Lian (2013) leached REEs and radioactive elements from bauxite residue by bioleaching.

The majority of the literature studies focus upon scandium and very little on other REEs. Neither has much work been done on leaching of major elements from bauxite residue nor leaching of REEs by organic acids. Study on the leaching of major elements is, however, useful for the development of the further purification process. Therefore, in this study we systematically investigated the extraction of REEs as well as other major elements in organic and mineral acids (HCl, HNO₃, H₂SO₄, CH₃COOH, CH₃SO₃H and citric acid). The effect of different parameters such as acid type and concentration, leaching time, liquid-to-solid ratio and temperature were investigated.

2. Experimental

The bauxite residue studied in this work was provided by the Aluminium of Greece, which is located at Agios Nikolaos, Greece. It is originated from a mixture of karst and lateritic bauxites and is similar to the bauxite residue examined by Ochsenkühn-Petropulu et al. (1994). It was received from the alumina refinery after dewatering by filter presses and room temperature drying. The sample was further dried at 105 °C for 12 h. Next, the material was passed through a 500 µm size mesh before it was used in leaching studies. Analytical reagent grade nitric acid (65%) (Chem-lab), sulfuric acid (95–97%) (Sigma–Aldrich), hydrochloric acid (37%) (Fisher Scientific), acetic acid (100%) (VWR International), citric acid (99.6%) (Acros Organics) and methanesulfonic acid (98%) (Acros Organics) were used in the present study. Chemical analysis of major elements in bauxite residue was performed using wavelength dispersive X-ray fluorescence spectroscopy (WDXRF, Panalytical PW2400). Chemical analysis of minor elements was performed after complete dissolution of bauxite residue by alkali fusion and acid digestion in a 1:1 (v/v) HCl solution, followed by Inductively Coupled Plasma Mass Spectrometry (ICP-MS, Thermo Electron X Series) analysis. The alkali fusion was carried out by mixing 0.5 g of bauxite residue with 1.5 g of sodium carbonate and 1.5 g of sodium tetraborate decahydrate, followed by heating the mixture in a platinum crucible at 1100 °C for 30 min. The mineralogy of the samples was studied by X-ray diffraction technique (XRD, Philips PW1830). The powder morphology was investigated by scanning electron microscopy (SEM, Philips XL30). The particle size of the samples was measured by laser particle size analysis (Malvern Mastersizer 3000). Thermogravimetric analysis (TGA, Netzsch STA 409) experiments were carried out in nitrogen atmosphere from room temperature to 1000 °C at a heating rate of 10 °C/min.

The leaching of the bauxite residue was carried out in sealed polyethylene bottles by constant agitation using a laboratory shaker (Gerhardt Laboshake) at 160 rpm and 25 °C. High-temperature leaching experiments were carried out in a 500 mL glass reactor fitted with a reflux condenser and placed on a temperature-controlled ceramic hot plate with a magnetic stirring system. The leach solution sample was filtered using a syringe filter (pore size of 0.45 µm) and diluted with distilled water for ICP-MS analysis. Leaching experiments were repeated twice to assess the reproducibility and the error found was within the 10% range.

3. Results and discussion

3.1. Characterisation of the bauxite residue

The concentrations of the major elements in the form of oxides in Greek bauxite residue is given in Table 1. Iron oxide is the major oxide in the bauxite residue followed by alumina, silica, calcium oxide, titanium oxide and sodium oxide. The concentrations of the REEs is given in Table 2. The relative standard deviation was less than 20%. From the table, it is evident that the bauxite residue sample is richer in light REEs than in heavy REEs. It is also rich in scandium (121 g/tonne). The total REE concentration is around 0.1 wt%.

The following major minerals and compounds were identified by X-ray powder diffraction (XRD): the iron minerals hematite and goethite; the aluminum minerals gibbsite and diaspore; the calcium mineral calcite and the sodium compound cancrinite (Fig. 1). TGA–DSC curves of the bauxite residue show three slopes (TGA) and three peaks (DSC) (Fig. 2). The weight loss between 300 and 600 °C is due to decomposition of hydroxides in different

Table 1
Major chemical components in the bauxite residue sample.

Compound	wt%
Fe ₂ O ₃	44.6
Al ₂ O ₃	23.6
CaO	11.2
SiO ₂	10.2
TiO ₂	5.7
Na ₂ O	2.5

Table 2
Rare-earth elements composition of the bauxite residue sample. Standard deviations are based on triplicate measurements. The detection limit of a REE is 1 g/tonne.

Element	Concentration (g/tonne)
Sc	121 ± 10
Y	75.7 ± 9.6
La	114 ± 15
Ce	368 ± 68
Pr	28.0 ± 3.9
Nd	98.6 ± 7.0
Sm	21.3 ± 2.3
Eu	5.0 ± 0.9
Gd	22.0 ± 1.9
Tb	3.5 ± 0.6
Dy	16.7 ± 0.7
Ho	3.9 ± 0.6
Er	13.5 ± 1.8
Tm	1.9 ± 0.3
Yb	14.0 ± 1.9
Lu	2.4 ± 0.3

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