



Technical note

Ammonium sulphate and/or ammonium bisulphate as extracting agents for the recovery of aluminium from ultrafine coal fly ash



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ARTICLE INFO

Keywords:

Coal fly ash
Thermochemical processing
Aluminium extraction
Ammonium sulphate
Ammonium bisulphate

ABSTRACT

We recently showed that the selective extraction of aluminium from the amorphous phase of a South African ultrafine coal fly ash can be achieved via thermochemical treatment with ammonium sulphate for 1 h followed by aqueous dissolution, as an alternative to conventional hydrometallurgical processes. In this study, insight gained from the previous work was applied to investigate and compare total vs selective aluminium extraction efficiencies using ammonium sulphate or ammonium bisulphate either on its own, or as a mixture of the two salts as extracting agents during a 2 h thermal treatment process. The effects of (i) ash-to-extractant mass ratio and (ii) temperature during thermal treatment on extraction efficiency was investigated. While a maximum, but non-selective, recovery of 46.6% total aluminium was achieved using ammonium bisulphate at 400 °C, the most technically appropriate results for selective recovery yielded 37.3% aluminium, with only 0.3% silicon, 0.1% titanium and 3.9% iron having been co-extracted when using ammonium sulphate at a processing temperature of 600 °C. Extraction of most of the calcium and magnesium could not be prevented. Using mixtures of ammonium salts as extracting agents during thermochemical treatment may however introduce technical difficulties on large scale. Our results indicate that any of the two ammonium salts could be used on their own during thermochemical treatment. Thermochemical treatment of coal fly ash using ammonium salts may therefore represent a promising technology for extracting aluminium from South African coal fly ash.

1. Introduction

The recovery of aluminium (Al) from coal fly ash (CFA) is of particular relevance to South Africa because the country has no economically viable bauxite deposits. Although South Africa has been actively involved in CFA beneficiation and/or utilisation research, the recovery of alumina as the precursor for aluminium production has not been widely pursued (Matjie et al., 2005; Shemi et al., 2012; Shemi et al., 2014; Shemi et al., 2015). This is rather surprising since the alumina content of domestic fly ashes generally exceeds 30%.

Due to the long distance between the power stations and the industrial hub of the country it is customary in South Africa to process and beneficiate CFA at source prior to being transported to the market. Air-classification is used to extract the particular size fractions required by the market. One of these is a commercial-grade, ultra-fine fly ash that is used as a high-performance pozzolan in the cement industry and

as a filler for polymers (plastics). This fraction of ash was used in the present study because it is slightly higher in alumina than other South African coal ash resources, and has an elevated amorphous content and a high specific surface area.

The pioneering work by Grzymek (Grzymek, 1976), demonstrating that alumina could be recovered from CFA, provided the mineralogy was altered by high temperature sintering, precipitated extensive research into the efficacy of various sintering agents in forming soluble aluminium compounds. In particular, lime sinter, lime soda sinter, and Calsinter (Egan et al., 1980) processes, along with high temperature chlorination, received global attention. Acid leach methodologies were shown to be less effective in selectively extracting aluminium (Yao et al., 2015; Yao et al., 2014).

Our recent study indicated that thermochemical treatment using ammonium sulphate ((NH₄)₂SO₄) may represent a promising technology for extracting Al from fly ash (Doucet et al., 2016). One of the

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advantages of this process resides in the fact that $(\text{NH}_4)_2\text{SO}_4$ is widely available at low cost and can be easily recycled (Highfield et al., 2012). The most promising processing conditions were determined to be a temperature of 500 °C and a fly ash to $(\text{NH}_4)_2\text{SO}_4$ weight ratio of 2:6 when a reaction time of 1 h was used (Doucet et al., 2016). It was also found that water leaching of the reaction products obtained under these conditions resulted in the selective recovery of 95.0% Al from the amorphous phase, with < 0.6% Si extracted. Results indicated that mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$) was unlikely to have reacted with the extracting agent when a reaction time of 1 h was used.

Since ammonium bisulphate (NH_4HSO_4) is known to yield better elemental extraction efficiencies from serpentine minerals than $(\text{NH}_4)_2\text{SO}_4$ (Romão et al., 2016), the present paper tested and compared, for the first time, the potential of these two agents, used either singularly or as mixtures in various mass ratios, for the extraction of Al from the same classified ultrafine South African CFA. The selectivity of these two extracting agents for Al vs other major elements has also not been previously examined. The second objective was therefore to compare the selectivity of these agents for Al recovery with minimal extraction of other major elements (*i.e.* Ca, Fe, Si, Ti and Mg). Thirdly, the duration of the thermochemical treatment was increased from 1 h to 2 h to examine the extent of reactivity between the mullite component of CFA and the two extracting agents. An outline of the multi-stage extraction process proposed is illustrated in Fig. 1. Potential chemical reactions taking place during the thermochemical treatment of CFA and the ammonium salts used in this study have already been described in the literature (Wang et al., 2014a; Wang et al., 2014b).

2. Experimental

2.1. Materials description and characterisation

A representative sample of an ultrafine siliceous coal fly ash (CFA) was obtained from the Ash Resources Pty Ltd.'s ash beneficiation site at Eskom's Lethabo Thermal Power station, South Africa. This commer-

cial-grade CFA is specified to have > 90% of its particles with a diameter smaller than 11 μm (vol%). Detailed surface and bulk characterisation of the sample, which included XRF, XRD, PSD, FE-SEM, Raman, FTIR and XPS analyses, was reported elsewhere (van der Merwe et al., 2014). Briefly, the sample consisted of an amorphous glass phase (64.3%), which co-existed with two primary crystalline phases, mullite ($\text{Al}_6\text{Si}_2\text{O}_{13}$; 30.2%) and quartz (SiO_2 ; 5.5%). It was made up of six major chemical constituents (SiO_2 , 52.9%; Al_2O_3 , 33.5%; CaO, 5.0%; Fe_2O_3 , 3.3%; TiO_2 , 1.7%; MgO, 1.1%).

Reagent grade (99.5%, Merck) ammonium sulphate and ammonium bisulphate (hereafter referred to as “AS” and “ABS” respectively) were used in all experiments.

2.2. Thermochemical processing and aqueous dissolution

The first step of the staged process involved processing CFA by thermochemical treatment using either AS, ABS or a mixture of both in various mass ratios as extracting agents. CFA and the ammonium salts were thoroughly mixed in fused quartz crucibles at CFA:AS:ABS mass ratios of 2:6:0; 2:4:2; 2:3:3; 2:2:4; and 2:0:6. These ratios were chosen to give a stoichiometric excess of SO_4 over the total amount of extractable elements (*e.g.* Al, Ca, Fe), to ensure that the amount of the ammonium salt did not represent a limiting factor during extraction. The mixtures were thermally treated in a static muffle furnace at 400, 500 or 600 °C for 2 h. The reaction products (CFA_{tct}) were then subjected to aqueous dissolution in deionised water. The chemical composition of the leachates was determined by ICP-MS, while that of CFA_{tct} , and the filtered solid residues (CFA_{res}) were characterised by XRD and FE-SEM. The ICP data was used to calculate elemental extraction efficiencies, *i.e.* the mass fraction of elements extracted as sulphates into the aqueous solution, was compared to the mass of these elements initially present in untreated CFA (determined by XRF). The procedures followed for dissolution experiments and solid residue characterisation were described previously (Doucet et al., 2016).

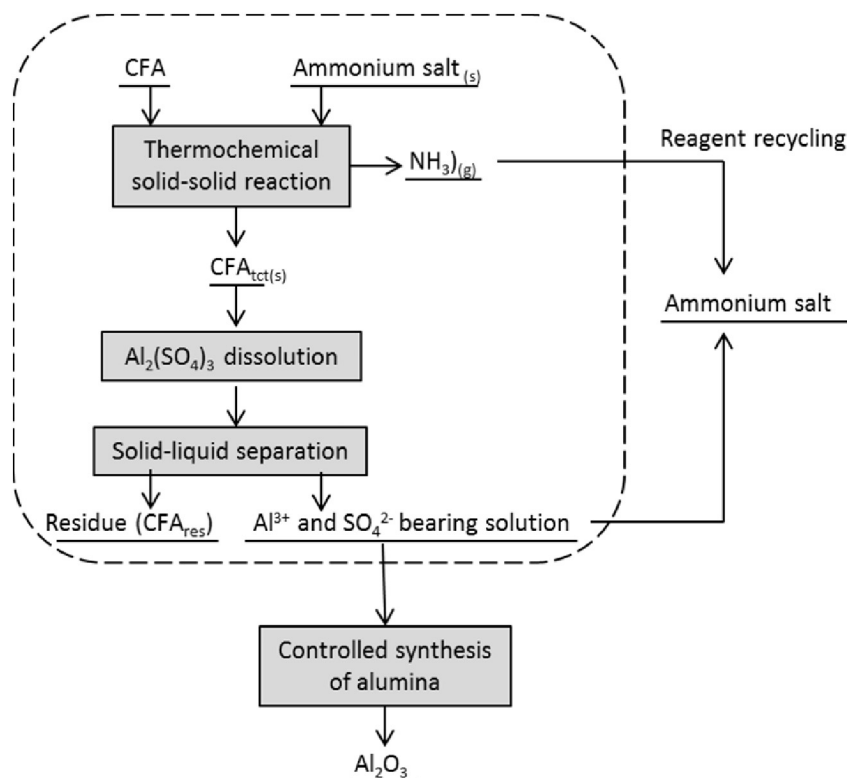


Fig. 1. Process flow diagram for the multi-stage extraction of Al from coal fly ash.

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