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# Extraction of aluminium as aluminium sulphate from thermal power plant fly ashes



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**Abstract:** Valuable metal extraction technology from thermal power plant fly ash is limited. In the present study, aluminium is extracted from fly ash as highly pure aluminium sulphate (>99.0%) by leaching with sulphuric acid, followed by pre-concentration and successive crystallization. Two types of fly ashes from different sources, i.e., Talcher Thermal Power Station (TTPS) and Vedanta Aluminium Company Limited (VAL) were chosen for comparative study on the extraction of aluminium as aluminium sulphate. The product is characterized by powder X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and thermogravimetric analysis (TGA). Purity of aluminium sulphate was also investigated by inductively coupled plasma–optical emission spectrometry (ICP–OES). The extraction efficiency of aluminium depends on the varied solid-to-liquid ratio (fly ash : 18 mol/L  $H_2SO_4$ , g/mL) and particle size of fly ashes. Physico-chemical analysis indicates that the obtained product is  $Al_2(SO_4)_3 \cdot 18H_2O$ , having low iron content (0.08%).

Key words: fly ash; sulphuric acid; aluminium sulphate; leaching; solid-to-liquid ratio; particle size

### **1** Introduction

Thermal power plant fly ash linked to various environmental problems, particularly its accumulation posed a great threat to air, water and soil pollution. Besides bulk utilization like the brick, cement and non-cement uses, high-value utilization of fly ash is also essential to conserve the natural resources. Fly ash contains major components such as silica (around 60%), alumina (around 30%) and iron oxides (4%-20%) along with various other impurities such as calcium oxides (1%-12%), magnesium oxides (1%-2%), titanium oxides, sodium oxides and potassium oxides. The extraction of aluminium and other strategic metals has specific relevance in view of safe disposal and residue utilization. The recovery of aluminium is amenable through metallurgical processes such as direct acid leaching or soda-lime sinter processes. Although a large number of processes have been developed for gainful utilization and safe management of fly ashes [1-5], constraints still persist for the high-value applications (metal recovery) due to process difficulty. Several methods for the recovery of aluminium from coal fly ashes have been proposed [6-20], but a definite process has not been established for its commercialization. The obvious reason is that the physical properties and precise composition of fly ashes vary depending on the source of the coal being burned, type and uniformity of the coal, the operating parameters of the power plant and the collection methods employed to separate the ashes.

Nitric acid and hydrochloric acid leaching processes for the recovery of alumina and other minerals have little practical applications due to low recovery. Low cost and stability have prompted sulphuric acid as lixiviant by several investigators for the recovery of alumina [21,22]. According to MATJIE et al [22], the aluminium extraction efficiency of 85% was achieved when the sintered pellets of fly ash, mixed with calcium oxides, were leached with sulphuric acid using an acid concentration of 6.12 mol/L at 80 °C for 4 h. Many researchers have reported about the extraction of aluminium from ores of clay, shale and aluminosilicate by using H<sub>2</sub>SO<sub>4</sub> as leachant at different temperatures for different leaching time [23,24]. HUANG [25] investigated the potential of hydrofluoric acid (HF) in the extraction of aluminium by treating coal ash with 52% HF at 25 °C for 1 h. In another process, a coarse

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y-Al<sub>2</sub>O<sub>3</sub> powder was prepared by acid leaching, and the extraction rate of Al<sub>2</sub>O<sub>3</sub> was found to be 87% [26]. Pressure acid leaching method achieved 82.4% extraction efficiency by using 74 µm particle size of coal fly ash with 50% sulphuric acid in a pressure reaction kettle at 180 °C for 4 h [8]. The extraction of alumina from coal fly ash using an acid leach-sinter-acid leach technique has been proposed by SHEMI et al [27], based on the dissimilar response characteristics of the amorphous and mullite- alumina phases present in coal fly ash. The reaction behaviour of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> in high alumina coal fly ash under various alkali hydrothermal conditions was explored by JIANG et al [28]. The results showed that the Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> leaching efficiencies of coal fly ash were dependent on the dissolution of glass phase and the precipitation of zeolite phase. DING et al [29] has extracted alumina from high alumina pulverised coal by using high alkali sodium aluminate solution through a reactor in two stages. Alumina extraction efficiency of 96.03% has been achieved. SIBANDA et al [30] have reviewed the hydrometallurgical extraction processes of different workers to assess the available opportunity and prospects of commercial exploitation of the aluminium extraction from coal fly ash.

All of the above studies aimed at finding alternate routes to produce smelter grade alumina, but these processes have their own engineering difficulty for commercial viability. The present investigation is an attempt to convert a considerable amount of aluminium into environment friendly material for industrial uses. In the proposed process, selective recovery of aluminium has been tried in the form of aluminium sulphate from the leach liquor with acceptable iron and other impurities. To make the process more economical, direct acid leaching method has been employed with the commercial grade sulphuric acid and moreover, as-received fly ash samples have been used in leaching without further being subjected to energy intensive grinding, size classification or sintering.

## 2 Experimental

#### 2.1 Leaching of fly ash

Coal fly ash (CFA) samples were collected from two different thermal power stations, viz., Talcher Thermal Power Station (TTPS) and Vedanta Aluminium Company Limited (VAL). Both the samples were subjected to particle size, chemical and mineralogical analyses [31,32]. In continuation to our earlier reported works [15,33,34], leaching study of both the fly ashes has been undertaken systematically with varying solid-to-liquid ratio (1:1, 1:2, 1:3 and 1:4) to investigate the extent of aluminium extraction from the as-received coal fly ashes without further grinding and size reduction. Representative fly ash samples (100 g) from each TTPS and VAL were mixed separately with a specific volume of 18 mol/L  $H_2SO_4$  to produce suspensions with varying solid-to-liquid ratio (1:1, 1:2, 1:3 and 1:4). The resulting suspensions were heated up to 200 °C for 4 h at normal atmospheric pressure with the evolution of white fumes. The fly ash residues were filtered from the leached liquor by using G3 Buchner funnel followed by washing with hot water. The extraction efficiency of aluminium is determined using the following formula [35]:

$$E = \frac{C_{\rm bl}(\rm Al) - C_{\rm al}(\rm Al)}{C_{\rm bl}(\rm Al)} \times 100\%$$
(1)

where *E* is the extraction efficiency, and  $C_{bl}(Al)$  and  $C_{al}(Al)$  are the concentrations of aluminium present in fly ash before and after acid leaching, respectively.

#### 2.2 Precipitation of aluminium sulphate

The leached liquor obtained after filtration and washing was evaporated by using hot water bath. The liquor got concentrated on slow evaporation to a point where aluminium sulphate started precipitating. Then, the concentrated liquor was kept overnight at 8 °C in a cooling incubator. White coloured aluminium sulphate (gel) precipitated out, it was filtered in cold condition using G1 Buchner funnel. The same procedure was repeated for complete crystallization of aluminium sulphate. Since aluminium sulphate is almost insoluble in acetone, the precipitate obtained was washed with acetone and further dried by vacuum desiccation. As aluminium sulphate is highly hygroscopic, it was dried at 60 °C followed by keeping it in a desiccator and then subjected to physico-chemical analysis.

#### 2.3 Sample characterization

The elemental compositions of the aluminium sulphate, recovered from both fly ashes, were determined by ICP-OES (Perkin Elmer Optima 2100 DV) and the thermal behaviour of both samples was examined by means of TG/DTA (METTLER STAR<sup>e</sup> SW 9.01). The crystalline phases were characterized by powder X-ray diffraction (XRD) (Phillips diffractometer, PW–1710) using Cu K<sub>a</sub> radiation. The FTIR spectra of the samples were taken with FTIR spectrophotometer (IR Prestige–21, Shimadzu) in the range of 400–4000 cm<sup>-1</sup>. The particle size analysis of fly ash residues was carried out by MALVERN particle size analyzer (UK, Model–3600).

#### **3** Results and discussion

The TTPS and VAL fly ashes were characterized chemically and mineralogically to understand the chemical composition, phase mineralogy and particle

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