AA23 - Preparation of Metallurgical Grade Alumina from Coal Fly Ash

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Abstract

Alumina recovery from coal fly ash is being pursued at CSIR-Institute of Minerals and Materials Technology (CSIR-IMMT), Bhubaneswar. As alumina is associated mainly in mullite phase, it becomes very difficult to recover alumina from such matrix. Extraction of alumina from fly ash therefore is a challenging task. Further, the silica content of fly ash is higher than that of alumina, and therefore, directly getting aluminate solution through Bayer digestion method is difficult without employing stringent conditions and pre-treatments. One of the processes developed at CSIR-IMMT goes through partial desilication of fly ash followed by direct leaching in acidic solution in the presence of fluoride ions. The process was developed primarily to produce non-metallurgical grade alumina. Large-scale utilisation of fly ash will be possible if metallurgical grade alumina is produced. Therefore, at CSIR- IMMT, the production of metallurgical grade hydroxides is being developed from the direct acid leached solution through precipitation-soda leaching and conventional seeding route to precipitate out the metallurgical grade hydroxide from the purified aluminate liquor. The characterisation of the produced aluminium hydroxide is being compared with the properties of the aluminium hydroxide produced through Bayer's process which showed positive results. Recycling of the spent solution to the mixed precipitate leaching circuit is also being tested. Further testing is continuing to ascertain the results.

Keywords: Coal fly ash, leaching, fluoride ions, aluminium hydroxide (ATH), metallurgical grade alumina.

1. Introduction

Countries with developed aluminium industry like China, United States, European Union and CIS countries heavily depend on imports of bauxite ore from countries like Australia, Indonesia, Guinea, Brazil, and India as feedstock for the production of metallurgical grade alumina [1]. The depletion of bauxite ore across the globe, made the researchers to think about different innovative methods for the recovery of alumina values from the secondary resources; amongst them, coal fly ash is one of the most prominent ones because of its greater availability.

The annual world generation of fly ash is nearly 800 million tons of which India alone produces more than 200 million tons [2]. Storing such a huge quantity of fly ash requires land. In India around 43 % of fly ash is presently utilised in different sectors including cement (major portion) leaving a vast quantity unutilised [3]. In China and USA utilisation rates are 70 % and 50 %

respectively. Fly ash contains different valuable minerals such as mullite, quartz, hematite, magnetite, alpha-alumina, CaO, TiO_2, etc. including rare earth elements in a matrix of aluminosilicate glass. Nearly 50 million tons of alumina, the major value material which is present in fly ash is getting wasted due to unavailability of suitable technologies in India. The disposal of such a huge quantity of fly ash also creates environmental problem. The greatest disadvantage of fly ash processing is its high silica content, due to which various conventional methods cannot be employed. Furthermore, the main alumina bearing mineral is mullite which is refractory in nature and difficult to solubilize.

Many authors have tried to extract alumina from fly ash using different techniques such as lime stone sintering [4], [5] soda lime sintering [6], calsinter process [7], [8] ammonium sulphate sintering process [9] and acid leaching processes [10], [11]. However, this process consists of many disadvantages. Sintering process generates huge quantity of residue which is few times higher than the original fly ash. In ammonium sulphate process, the requirement of ammonium sulphate is huge (10:1, ammonium sulphate: alumina). The sulphuric acid method requires higher temperature of 200-210 °C, with a ratio of acid to fly ashes 5:1 (v/wt.) and extraction efficiency of 85 %. Bhattacharya et al., [12] patented a process to recover alumina by using sulphation roasting and water leaching technique, where the alumina produced is non-metallurgical grade.

Tripathy et al., [13], has studied the use of sodium fluoride to solubilise the alumina values of fly ash in sulphuric acid medium. Interesting results were obtained, where the leaching efficiency was found to be greater than 90 %. The study was extended beyond leaching to produce the metallurgical grade alumina by employing precipitation, dissolution and further precipitation methods. A tentative flow sheet was developed to produce metallurgical grade alumina and is presented in this paper. Further optimization work is under progress.

2. Materials and Methodology

2.1 Materials

Coal fly ash was collected from the thermal power plant of National Aluminium Company Limited (NALCO), Angul, India. All the chemicals used in the present work were of analytical grade (Merck, India). The phase analysis was carried out using the X-Ray diffraction patterns generated from an X-ray diffractometer (Philips, PW 1710). Analyses of the solutions were carried out using ICP-OES and AAS.

2.2 Methodology

All the leaching experiments were carried out in batches. An autoclave of volume 1L was taken for conducting the experiments for silica removal. NaOH solution (500mL) of different concentrations and fly ash (50 g) were added in the reactor. Hydrothermal leaching operations were carried out at various temperatures from 150 °C to 180 °C. The experiments were of 3 h duration. After the experiment, the slurry was filtered. The residue was dried and again leached with sulphuric acid in the presence of NaF varying different concentrations to recover alumina. A standard double walled borosilicate glass reactor of volume 250 mL was used for conducting alumina leaching at a regulated temperature, controlled by a circulatory thermostatic water bath through inlet and outlet ports. The reactor was kept on a magnetic stirrer and agitation was made with the help of a magnetic paddle. The temperature during leaching was varied from 40-90 °C and the leaching time was varied from 15 min to 5 h. In each experiment, 100 mL solution was taken with 10 % solids concentration. The leach liquor thus obtained was then analysed for aluminium value and the recovery percent of alumina was calculated (Tripathy et al., 2019) [13]. This study was tested in higher scale (100 g fly ash) to carry out further study on the precipitation aluminate solution from fly ash and to produce the smelter grade alumina. In this process, by-products have been generated which could minimise environmental problem posed by fly ash while conserving the Bauxite resources. However, the main concern in this process is the contamination of sulphate ions in the product, alumina. After 5 cycles of the present study, very minimum contamination of SO_4^{-2} ion was noticed. Options are being tried at CSIR-IMMT to avoid the contamination of sulphate. One of the options could be to incorporate a bleeding circuit to keep the sulphate ion concentration in the solution to minimum for obtaining proper hydroxide/alumina product. Some of the properties of the produced alumina were compared with the product of the Bayer's process and found satisfactory. Optimization of the flow sheet, complete characterization of alumina and to compare with the smelter grade is underway. If successful, bench studies will be carried out for generating the mass and energy balance.

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