Boehmite-Kaolinite Bauxite Treatment by Ammonium Bisulfate Method: Study of $Al(OH)_3$ Precipitation from Ammonium Alum Solution

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Abstract

The precipitation of aluminum hydroxide ($Al(OH)_3$) from ammonium alum solution by ammonia water was studied. Ammonium alum solution was obtained after high-pressure leaching of boehmite-kaolinite bauxite with a mixture of 3M H_2SO_4 + 40% NH_4HSO_4 at T = 170 ºC. Fe(III) was extracted from the ammonium alum solution by ion-exchange sorption using Purolite S957 resin. Analyze the precipitation experiments, the influence of seed addition to shape of particle, the phase composition, the content of impurities, and the size of the $Al(OH)_3$ particles were investigated. The $Al(OH)_3$ calcination process at T = 900 ºC to obtain alumina powder was studied.

Keywords: Bauxite, High-pressure leaching, Ammonium bisulfate method, $Al(OH)_3$ precipitation, Sand grade alumina.

1. Introduction

Currently, two deposits located in the Urals are used in Russia for alumina production – North Urals and Middle Timan. The bauxite of these deposits has a low silica (SiO_2) content – 3-5 wt.%, therefore, alkali methods (Bayer and sintering) are used for its treatment [1]. However, the total amount of sandy grade alumina that produced in Russia is not enough to compare to the requirements of Aluminum Smelters. Therefore, it is necessary to look for new sources of alumina. In the Arkhangelsk region, there is an explored bauxite of Severoonezhsk deposit. Annually about 900 kt are mined, this bauxite is not used to produce alumina, due to the high content of silica (up to 20 wt. %) and chromium oxide Cr_2O_3 (up to 1 wt. %) [2].

The most promising methods for obtaining alumina from such raw materials are acidic, which allow the extraction of almost all aluminum, while silica remains in the solid residue [3]. Previously, the leaching process of bauxite with a mixture of sulfuric acid and ammonium bisulfate was studied [4]. In this article, the process for alumina production from acid liquor was studied and the behavior of the main impurities was analyzed.

2. Materials and Methods

2.1 Materials and Reagents

Raw bauxite sample was collected from the Severoonezhsk Bauxite Mine (N62.573349º, E39.719039º). Analytical grade ammonium sulfate CAS No. 7783-20-2, sulfuric acid CAS No. 7664-93-9 (both from SigmaTek, Russia) were used in the bauxite leaching process. Distilled water was used to washing bauxite, washing and dilute of ammonium alum after bauxite leaching and liqour cooling. The ion exchange resins S957 (Purolite, USA) were used for Fe removal from

alum solution by resin sorption method. The gibbsite powder ($Al(OH)_3$) from Urals Alumina refinery was used as seed in the precipitation process.

2.2 Experiments

Bauxite was leached by 40% NH_4HSO_4 + 3M H_2SO_4 mixture in a 50 mL high-pressure reactor (Deschem, China). The leaching time at T = 170 ºC was 90 min. The liquid to solid ratio (L:S) was 10. Pulp after leaching was filtered, the solid residue was washed by heat water (90 ºC). The solid residue dried at 110 ºC for 2 h and analyzed by physical and chemical methods. The liquor after filtration was analyzed for major and minor metals content.

After liquor colling the ammonium alum crystals ($NH_4Al(SO_4)_2 \cdot 12H_2O$) were precipitated. These crystals washed with cold distilled water (3 ºC), since at this temperature the solubility of ammonium alum is minimal: 5 g / 100 mL of H_2O. Next, the crystals were dissolved in distilled water for iron removal by Purolite S957 resin. Resins were placed in 2M sulfuric acid for 3 h to converted it into the H^+ form. Fe batch sorption was performed by mixing a Purolite S957 resin with an ammonium alum solution at the ratio 1:100 in a plastic Erlenmeyer flask. The Erlenmeyer flask was agitated at 100 rpm with an ECROS PE-6300 laboratory shaker (LLC ECROSKHIM, Russia) at a temperature of 55 ºC for 2 h.

After filtration from resin, the solution was used for gibbsite precipitation by two methods: using ammonia water (NH_4OH) with and without seed addition. The alum solution was heated in laboratory glass on a magnetic stirrer to 90 ºC, stirring rate was 350 rpm. The precipitation process time was 2 h. Ammonia was added to the alum solution using peristaltic pump YW21-SP25 (YW FLUID, China) at a rate of 1 mL/min. Precipitation was completed at a solution pH = 7, then the solution was filtered, washed, and dried at 110 ºC for 4 h. Calcination gibbsite samples was carried out in a PM-1 muffle furnace (Plavka.Pro, Russia) at T = 900 ºC at 1 h. The samples of gibbsite and alumina were analyzed by physical and chemical methods.

2.3 Analytical Methods

Phase composition of the samples were measured by X-ray diffraction (XRD) using a Difrei-401 X-ray diffractometer (JSC Scientific Instruments, Russia) using a Cr-Ka radiation source and a 2θ range from 5º to 140º with 30 min exposure time. The operating mode of the X-ray source was set to 25 kW/4 mA. The mineral phases were analyzed by Match! 3 software. The surface morphology and elemental composition of samples were investigated by scanning electron microscopy energy dispersive X-ray spectroscopy (SEM-EDX, Vega III, Tescan, Czech Republic). The metals concentrations in solutions and solid samples were measured by inductively coupled plasma optical emission spectrometry (ICP-OES) using an atomic absorption spectrometer AA-240FS (Varian, Melbourne, Australia). The particle size distribution of samples was determined by laser diffraction method (LD) using Bettersizaer ST (Bettersize Instruments Ltd., China) and Zetasizer Nano ZS (Malvern, UK). The specific surface area of samples analysed by the Brunauer–Emmett–Teller method (BET) using NOVA 1200e (Quantachrome Instruments, FL, USA).

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5. References

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