Removal of Iron (III) from Ammonium Alum Solution of Coal Fly Ash H₂SO₄ + NH₄HSO₄ Leaching by Ion Exchange Sorption Using Purolite S957 and S950 Resins

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Abstract

In this research, the ammonium alum solution was purification from Fe (III) – the main impurity in the acid technology for alumina production using ion exchange method. The acid solution was obtained after Coal Fly Ash high-pressure leaching by 7.5M H_2SO_4 + 40% NH_4HSO_4 mixture at T = 210 ºC. Two type of resins, Purolite S957 and S950 were used in sorption tests. At optimum parameters (T = 25 ºC, sorption time 24 h and the resin to solution ratio - 1:25), it is possible to extract more than 90% of iron. Using the Langmuir model, the batch sorption values for iron were calculated: for Purolite S957 resin - 19.48 g/g; for Purolite S950 - 3.39 g/g. The dynamic sorption was studied for Purolite S957 resin at a solution flow rate of 0.4, 0.65, and 1.6 specific volumes ($V_{sp.vol.}$) per min. Using the Thomas model, dynamic capacity values were calculated for each of the rates: 0.4 $V_{sp.vol.}$/min - 6344.56 mg/g; 0.65 $V_{sp.vol.}$/min - 5590.23 mg/g; 1.65 $V_{sp.vol.}$/min - 1632.77 mg/g. The desorption of Fe(III) from resin using 2M H_2SO_4 at the rate of 0.4-0.65 $V_{sp.vol}$ was studied. The required volume of desorbent at both rates is 6 $V_{sp.vol.}$. The possibility of removal the Fe(III) from ammonium alum solution by 12 times (from 27.2 mg/L to 2.2 mg/L) using Purolite S957 resin was founded.

Keywords: Coal fly ash, High pressure leaching, Ammonium bisulfate, Resin sorption, Iron removal.

1.   Introduction

Currently, one of the main areas of utilization of solid waste from coal-fired thermal power plants, coal fly ash – CFA, is the extraction of non-ferrous metals. CFA is a promising raw material for alumina production due to the high content of aluminum oxide, from 20 to 40 wt. %, the absence of the need to grind the raw material and the low cost (basically, the cost of CFA includes only the cost of delivery to the consumer). Reftinskaya GRES the largest coal power plant in Russia, generates more than 4 Mt of CFA per year, and the total CFA store in the landfills exceeds 180 Mt. This CFA can be used at Russian alumina refineries in the Urals region, since Reftinskaya GRES is located 120 km and 490 km from the Urals and Bogoslovsky aluminum plants, respectively [1].

It is not effective to use alkaline methods (Bayer and sintering) to extract alumina from this type of CFA, due to the low content of aluminum oxide (25-28 wt. %) compared to CFA from the Inner Mongolia of China (40-45 wt. %) [2]; thus, resulting in high consumption of fluxes during the preliminary high-temperature treatment and alkali during the leaching process. For the CFA of the Reftinskaya GRES, it is efficient to use acid methods. Previously, it was shown that the hydrochloric acid method can be used to extract alumina [3]. An alternative is the bisulfate method, which allows the use of less aggressive reagents for CFA leaching – a mixture of ammonium bisulfate and sulfuric acid [4,5].

The main impurity in acid methods is iron, which, together with aluminum, is almost completely extracted into solution during the leaching process. Previously, a solvent extraction method for iron removing from Al-Cl liquor was studied to obtain of ferric chloride solution [6]. Another approach would be to use iron selective ion exchange resins. In this study, the purification of ammonium alum solution obtained after leaching of the CFA using Purolite S957 and S950 resins was investigated.

2.   Materials and Methods

2.1   Materials and Reagents

CFA was collected from the Reftinskaya GRES (Asbest, Russia). Ammonium sulfate CAS No. 7783-20-2, sulfuric acid CAS No. 7664-93-9 (both from SigmaTek, Russia) were used in the present study. Distilled water was used to washing and dilute of ammonium alum after CFA leaching and liquor cooling. The ion exchange resins S957 and S950 (Purolite, USA) were used for sorption research.

2.2   Experiments

The CFA was leached with a mixture of 40% ammonium bisulfate and 7.5M sulfuric acid at 210 °C, S:L = 1:10, with a duration of 180 min. After slurry filtration, the solution was cooled to room temperature and ammonium alum crystals ($NH_4Al(SO_4)_2 \cdot 12H_2O$) was precipitated. These crystals were 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. The loss of alum after washing was 14%. Next, the crystals were dissolved in distilled water for iron removal study.

Resins were placed in 2M sulfuric acid for 3 h to converted it into the H^+ form. Fe batch sorption experiments were performed by mixing a certain amount of Purolite S957 and S950 with an ammonium alum solution of a known concentration in a plastic Erlenmeyer flask. The Erlenmeyer flasks were agitated at 100 rpm with an ECROS PE-6300 laboratory shaker (LLC ECROSKHIM, Russia) at temperatures of 25, 40 and 55 ºC until pseudo-equilibrium was reached. For dynamic sorption, a column filled with resin was used; the solution was fed into the column using a peristaltic pump YW21-SP25 (YW FLUID, China).

2.3   Analytical Methods

The metals concentrations in liquor after CFA leaching and resin sorption were measured by inductively coupled plasma optical emission spectrometry (ICP-OES) using an atomic absorption spectrometer AA-240FS (Varian, Melbourne, Australia).

3.   Results and Discussion

3.1   Batch Sorption

Before carrying out experiments on batch sorption, the impurity composition of the solution after CFA leaching, water washing of precipitated ammonium alum and alum solution were analyzed (Table 1).

5.   References

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