The Effects of Washed Seed on Sodium Oxalate Precipitation and Particle Size Distribution

Shao Shuai
Engineer
Zhengzhou Non-ferrous Metals Research Institute Ltd of CHALCO, Henan, China
Corresponding author: zyy_shsh@rilm.com.cn

Abstract

The precipitation of sodium oxalate in saturated liquor can affect the particle size distribution of the product in the precipitation process. Given this phenomenon, the behavior of sodium oxalate precipitation with washed seed was analyzed in the precipitation process. In addition, the internal theory, that the particle size of seed influenced by sodium oxalate would reduce, was also evaluated. In this paper, the results of using washed seed during three continuous precipitation cycles and the associated effect on particle size distribution is examined and discussed.

Keywords: Washed seed, Sodium oxalate, Particle size distribution, composition

1. Introduction

With China's domestic bauxite grade gradually reducing, some domestic alumina enterprises began to use overseas bauxite to produce alumina. The organic matter content was found to be higher in overseas bauxite than from the locally sourced bauxite. In the process of production, macromolecular organic matter was gradually transformed into soluble sodium oxalate under exposure to alkali and high temperature conditions [1].

When the concentration of sodium oxalate was high in the sodium aluminate solution [2], it precipitated in the process of seed precipitation and adsorbed on the surface of the alumina tri-hydrate seed. Also observed was an increase in secondary nucleation, a reduction in seed strength and a resulting reduction in the size of the product granularity [3]. The explosive precipitation of sodium oxalate significantly damaged the growth cycle of the seed and the product size was affected.

Over the past few decades, experts and scholars conducted in-depth research on the removal of organic matter in the process of alumina production. The main methods identified were bauxite and spent liquor roasting, precipitation, crystallization, ion exchange, seed washing, seawater neutralization, and wet oxidation [4].

Due to the different content and types of organic matter in the bauxite used by each aluminum oxide plant, as well as the different production processes and technical conditions, the level and types of organic matter were also different in the production systems. Therefore, studies geared towards seeking more adaptive, economically and operationally feasible methods of organic matter removal have been conducted.

In the aforementioned technologies, the seawater neutralization method required a large area and needed to be located near the sea. The solution combustion method has been used across the industry, but is cost intensive and the exhaust gases of combustion presents their own environmental problems. The wet oxidation method requires a high level of investment due to the amount of equipment and operational resources required [5]. Compared with the two-stage precipitation process used outside of China, the seed washing method is most easily adopted because of the small amount of fine seed required, and the crystallization method is relatively simple. The crystallization effect, however, is very different in different systems.

In this paper, the removal of sodium oxalate using the seed washing method was evaluated. Specifically, the precipitation behavior of sodium oxalate was studied in washed seed during the precipitation process. By managing the precipitation of sodium oxalate in the precipitation tank, the morphology of the seed was optimized, and the particle size of the product was controlled.

2. Equipment and Method

2.1 Test Equipment

The main types of equipment used for this test were:

1. Precipitation tanks with a mechanical agitators.
2. Water baths, developed by the Zhengzhou Research Institute, used to independently heat the system and maintain constant temperature
3. Analytical balances
4. Vacuum filter devices

The main equipment used for sample analysis during the test were:

1. X-ray Diffractometer (XpertProMPD) used to analyze the phase composition of the sodium oxalate impurities,
2. Ion Chromatography Analyzer (Metrohm930) for oxalate content in the solution
3. Scanning Electron Microscope (JSM6360LV) for seed morphology analysis
4. Particle Counter (Multisizer3) for seed particle size analysis
5. Chemical Titration for sodium aluminate solution composition analysis

2.2 Test Method

A sodium aluminate solution from the production process was added to the precipitation tank. After preheating to the set temperature, a specified mass of unwashed alumina tri-hydrate seed was added. The seed was initially added to the No. 1 precipitation tank, and washed seed was then added into the No. 2 precipitation tank. After the agitator was started, the system was operated under the established precipitation temperature regime. When the predetermined precipitation time was reached, separated seed from slurry. Some seed was taken for impurity composition analysis, and the rest was used as the precipitated seed for the next cycle (No. 2 precipitation tank only had one cycle of seed washing)

Three cycles of continuous precipitation tests were carried out in this time. The test conditions were as follows:

1. precipitation temperature was 52-65 ℃,
2. The initial solid content of precipitation was 600 g/L
3. The precipitation time was 45 hours
4. 25 ppm of CGM was added

The experimental conditions of seed washing:

1. the hot water temperature was 80 ℃,
2. The solid content was 600 g/L
3. The reaction time was 1 hour.

After the reaction, a solid-liquid separation was carried out with a suction filter bottle, and the washed seed was added to the No.2 precipitation tank for the precipitation test.

The method of analysis for oxalate in seed included the rinsing and drying of the seed with alcohol. Then 50 g of dried washed seed was added to 100 mL of ultrapure water and stirred at 80 ℃ for 20 minutes. Vacuum filtration was carried out after full dissolution. Then 200 mL of

5. References

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