Preparation of Alumina from Aluminum Ash by Sintering with Sodium hydroxide

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Abstract. Aluminum ash is an industrial waste residue produced during alumina electrolysis and aluminum processing and casting. In the experiment, alumina powder with a purity of 98.97% was prepared by sintering with sodium hydroxide and the process conditions affecting the dissolution rate of Al₂O₃ from aluminum ash were studied. The best process conditions for the dissolution of Al₂O₃ are the sintering temperature of 700°C, the alkali-ash ratio of 1.2:1, and the time of 2h using orthogonal experiments. The phases of the aluminum ash and alumina were determined by X-ray diffraction(XRD) analysis. The chemical compositions of the aluminum ash and alumina were determined by X-ray fluorescence (XRF) spectroscopy. The microstructure and morphology of the aluminum ash and alumina were studied with scanning electron microscopy (SEM). Alumina prepared can be used as a metallurgical material.

1. Introduction
Aluminum ash is an industrial waste residue produced during alumina electrolysis and aluminum processing and casting. Aluminum ash is a complex oxide material containing Al₂O₃, SiO₂, Na₂O, Fe₂O₃, ZnO, and other compounds[1]. However, it contains 40%~60% alumina, so it may be an alternative source of alumina[2]. At present, Aluminum ash was treated with acidic and alkaline to prepare alumina, such as NaOH, H₂SO₄, HCl and HNO₃ having been used as leaching agents[3-5]. Aluminum ash is also used to prepare β-Sialon-15R ceramics, insulation bricks, calcium aluminate cement, mullite/zirconia, hexagonal mesoporous aluminophosphate, aluminum isopropoxide, (Mg,Si)Al₂O₄ Spinels and Alpo₄-5 type zeolitic materials[6-13]. Most of these composites are only used for experimental synthesis, and there is no market application.

Alumina has attracted a lot of attention because of high elastic modulus, high abrasion resistance, chemical stability and oxidation resistance. It is widely used in industries such as abrasives, ceramics, electronics, semiconductor, metallurgy, catalysts and artificial gemstones. There are also many ways to prepare alumina, such as Bayer method, sintering method, Ammonium aluminum carbonate (AACH) precursor and aluminum ammonium sulfate method[14-17]. Currently most of the world’s commercial alumina is produced by the Bayer and sintering method using bauxite material. AACH and ammonium aluminum sulfate process produce sulfur trioxide, ammonia gas that harmful to environmental during sintering. Microwave pyrolysis, chemical vapor deposition method (CVD method), and hydrothermal method also prepare alumina[18-20]. However, they have the disadvantages of high equipment requirements, technical difficulties, and poor safety performance.

In this work, desalted aluminum ash, CaO and NaOH were used as starting material to synthesize α-Al₂O₃. The dissolution rate of α-Al₂O₃ in aluminum ash was studied by using sodium hydroxide sintering method. The problem that alumina does not react with alkali solution and acid solution was
solved, thereby the utilization rate of alumina in aluminum ash was improved. The influence of the extraction process condition of α-Al₂O₃ in aluminum ash were investigated.

2. Experimental

2.1 Material
The main raw material used is aluminum ash from Jiayuguan city Chengyu Metal Material Company. The phases of the aluminum ash were determined by XRD and the photograph is shown in Figure 2. Raw aluminum ash was characterized by SEM and the photograph is shown in Figure 1. From the Figure 1 it can be seen the surface of the aluminum ash has an irregular shape and contains blocks, rods, flakes and fine particles with smooth surfaces. The main components of aluminum ash are 61.84% Al and Al₂O₃, 20% NaCl and Na₂O, and some other substances. The raw materials were pretreated to obtain desalted aluminum ash. The composition of the raw materials was as shown in Table 1. The content of Al and Al₂O₃ in the desalted aluminum ash was increased from 61.84% to 78.91%. It was found by XRD that there was 48% α-Al₂O₃ in the desalted aluminum ash.

2.2 Experimental procedures
25 g aluminum ash 170 mesh in a beaker, 125 g water was slowly added while stirring to prepare desalting aluminum ash at 40℃for 2 h. Approximately 21 g desalted aluminum ash after filtration and drying, 25.2 g granular NaOH were mixed with 0.85 g CaO in a 200 mL crucible. The crucible was heated in a muffle furnace at 700 ℃ for 2 h to prepare NaAl(OH)₄ clinker, then cooled and crushed. The obtained powder and 5 times water at 100℃for 2 h in a pyrex three-neck round-bottom flask equipped with a reflux condenser. Then 0.85 g 0.025% Polyacrylamide (PAM) was added to precipitation. The filtered solution and 2.2 g CaO were added at 100℃for 2 h in the pyrex three-neck round-bottom flask. Then 0.85 g 0.025% PAM was added to settled again. The filtered NaAl(OH)₄ solution was concentrated to Nk (concentration of NaOH in NaAl(OH)₄ solution) between 160 g/L and 280% Al(OH)₃ were slowly added to the concentrated solution at 50℃for 50 h in the pyrex three-neck round-bottom flask. Then filtered and washed the filtrate pH≤7. Dried Al(OH)₃ was heated in a muffle furnace at 1200℃for 2 h, the α-Al₂O₃ powder was obtained.

| Sample            | Na₂O | MgO  | Al₂O₃ | SiO₂ | CaO | FeO  | ZnO  | Cl  | Other |
|-------------------|------|------|-------|------|-----|------|------|-----|-------|
| Aluminum ash      | 17.25| 2.60 | 61.84 | 3.56 | 0.52| 0.39 | 3.78 | 6.64| 3.42  |
| After desalted    | 4.02 | 2.10 | 78.91 | 4.24 | 0.78| 0.55 | 6.13 | 0.17| 3.10  |

Figure 1. SEM micrograph of aluminum ash
2.3. Characterization of alumina powders
The chemical compositions of the alumina powders were determined by XRF. The XRF instrument is PANalytical Magix PW2403. The crystalline phase and microstructure were characterized by XRD and SEM. XRD data was obtained using a Rigaku D/Max-2400 diffractometer with Cu Kα radiation and the SEM instrument is JSM-6701F.

3. Results and discussion

3.1. Effects of various parameters on alumina extraction

3.1.1. Effect of the mass ratio of alkali to desalted aluminum ash. The effect of mass ratio of alkali to desalted aluminum ash on the extraction efficiency of alumina was studied at 700℃ for 2 h. The results are shown in Figure 3, it can be seen that the extraction efficiency of alumina increases with the increase of mass ratio gradually. When the mass ratio of alkali to desalted aluminum ash increased from 0.6 to 1.0, the extraction efficiency of alumina increased from 63.84% to 82.18%. Because of the addition of sodium hydroxide, the opportunities of colliding between NaOH and desalted aluminum ash increase, which leads to the augmentation of reactive area. However the mass ratio is too large, so that the concentration of sodium hydroxide in the sodium aluminate solution is increased, which is not conducive to the hydrolysis of the later sodium aluminate solution, so the mass ratio of alkali to desalted aluminum ash is established at 1.2:1.

Figure 2. XRD pattern of the aluminum ash and after desalted

Figure 3. Effect of alkali to desalted aluminum ash mass ratio on the extraction or efficiency alumina

Figure 4. Effect of sintering temperature on the extraction or efficiency alumina
3.1.2. Effect of sintering temperature. The effect of different sintering temperatures on the extraction efficiency of alumina was studied at sintering time 2 h, and sodium hydroxide to desalted aluminum ash mass ratio 1.2:1. Shown in Figure 4, as the sintering temperature increases, the extraction efficiency of alumina increases first and then decreases slowly. The increase in the loss of sodium hydroxide after 700°C causes the extraction rate of alumina to decrease, so the sintering temperature is established at 700°C.

3.1.3. Effect of sintering time. The effect of different sintering time on the extraction efficiency of alumina was carried out in experiments conducted at 700°C, 1.2:1 of sodium hydroxide to desalted aluminum ash. Shown in Figure 5, it can be obviously seen that the sintering time is 2 h and the extraction efficiency of alumina is up to 82.60%. However, as time passes, the extraction efficiency of alumina decreases, mainly because the clinker is agglomerated for a long time, which is not conducive to the dissolution of alumina, so the sintering time is established at 2 h.

3.2. Characterization of Al₂O₃ powders

3.2.1. XRD analysis. The XRD pattern of α-Al₂O₃ prepared is shown in Figure 6, The crystalline phase was identified by comparison with ICDD files(α-Al₂O₃, PDF NO.83-2080), the result shows that the peak position in the Figure 6 is consistent with the peak position of the PDF NO.83-2080. The result shows that a single crystal phase alumina product is obtained.

![Figure 5](image1.png)  Effect of sintering time on the extraction efficiency of alumina

![Figure 6](image2.png)  XRD pattern of the α-Al₂O₃

3.2.2 SEM analysis. Figure 7 (a, b) displays the SEM micrograph of α-Al₂O₃. It can be seen from the Figure 7(a) that the alumina particles are relatively uniform and there is no obvious agglomeration. Figure 7(b) shows the growth on the surface of the added seed crystal.

3.2.3 XRF analysis. The chemical analysis of α-Al₂O₃ prepared was determined by XRF, shown in Table 2 the purity of alumina reaches 99%, and it has reached the industry standard for metallurgical grade alumina, which can be used as a raw material for electrolytic aluminum.
Figure 7. SEM micrograph of production α-Al2O3

Table 2. Chemical components of α-Al2O3

| Item     | Al2O3 | SiO2 | Fe2O3 | Na2O | Loss on Ignition |
|----------|-------|------|-------|------|-----------------|
| Content  | 98.97 | 0.019| 0.0024| 0.59 | 0.92            |

4. Conclusions

In the paper, the alumina content in the raw material is up to 80% by pretreatment of aluminum ash. Desalted aluminum ash, calcium oxide and sodium hydroxide are mixed and sintered at high temperature to prepare sandy alumina. The experimental result shows that the alumina in the aluminum ash can be extracted by high temperature during the sintering process. The optimum extraction conditions obtained by the experiment are as follows: the mass ratio of sodium hydroxide to desalted aluminum ash is 1.2:1, the sintering temperature is 700°C, and the sintering time is 2 h. The experiment uses a period of sintering desiliconization and deep desiliconization to obtain less impurities in the product, and the purity of the alumina reaches 98.97%. This process is a simple, environmentally friendly and efficient approach for the preparing sandy α-Al2O3.

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