

## EXTRACTING ALUMINA FROM COAL FLY ASH USING ACID SINTERING-LEACHING PROCESS

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Keywords: Acid Process, Alumina Production, Coal Fly Ash

### Abstract

Local shortages of bauxite in recent years present a challenge to the development of sustainable aluminum industry in China. Coal fly-ash from coal-fired power plants is rich in  $Al_2O_3$  content with potential use as a resource for alumina refinery. This paper will describe recent investigation in extracting  $Al_2O_3$  from coal fly-ash using an acid sintering-leaching process. Thermal weight loss testing against temperature is first carried out to find out an appropriate sintering temperature so as to avoid heavy volatilization of the coal fly ash -  $H_2SO_4$  (98 %) mixture. Then, the mixture is subjected to sintering at 220 °C, dissolving in water at 85 °C, adjusting alkali level in solution by  $CaCO_3$ , removing iron by  $KMnO_4$  and  $MnSO_4$ , precipitating  $Al_2(SO_4)_3 \cdot 17H_2O$  particles, and heating-treated at 850 °C to produce  $Al_2O_3$  powders. The rate of recycling  $Al_2O_3$  can reach 70 – 90 % with relative lower processing temperature and less solid residues.

### Introduction

China has developed rapidly in the aluminum industry in recent years. In 2010, 29.9 mt of alumina were produced in China, an increase of 23 % compared to 2009 as well as an import of 4.3 mt in 2010. The reason lies in the fact that domestic alumina consumption continues to increase while the bauxite reserves declines quickly. It is a huge challenge to maintain the sustainable development of aluminum industry, so it is necessary to develop rich-aluminum substituted resources of bauxite such as coal fly ash and nepheline.

By the end of 2010, the installed capacity of electric power in China had reached 962 MKW and the largest proportion, 73.47 %, was the thermal power which was 706.6 MKW. By 2020, China's total amount of coal fly ash will reach about 3 billion tons, which will represent a reserve of alumina about 1.2 billion tons [1-2].

The main approaches of extracting alumina from coal fly ash are alkali process, acid process, acid and alkali combined process. Among them the alkali process is considered to have great potential for industrial use, as it is similar to the major technology currently used in the alumina plants nowadays. It includes soda lime sintering [3-4] or lime sintering process [5-7], which can generate a considerable amount of calcium silicate residue but is not utilized in an effective way except for producing cement. At the same time, alkali process demands an operating temperature of above 1000 °C while it consumes a great deal of energy and alkali. The silica content of the ore is not used as a product, so the cost of the process is high. Acid and alkali combined process [1, 8] activates the alumina and silica content together in the soda sintering process and extracts them using acid leaching, in which the major disadvantages are high energy consumption and excessively complex process. Acid leaching process [9-13] has been investigated for alumina extraction from coal fly ash directly using many types of acid such as hydrochloric acid, sulfuric acid,

or organic acid. The recovery rate of alumina in these processes was low, and by-product compound like calcium sulfate induced self inhibiting effect to mass transfer at the leaching sites of the coal fly ash particle. Furthermore, fluoride was used in the process as processing aid including ammonium fluoride, calcium fluoride to increase the extraction rate of alumina, but the fluoride was hazardous to environment.

Alumina is dissoluble in sulfuric acid and mullite can be decomposed by concentrated sulfuric acid at a high temperature [14-15]. In this paper, we use acid sintering-leaching process to extract alumina from coal fly ash so as to avoid the disadvantages in traditional process.

### Experiments

#### Materials and Chemical

The coal fly ash samples used in this research were gathered from a power plant in Inner Mongolia, China. The size distribution shows that 60 % particles of the coal fly ash are minus 150 mesh. The chemical composition of the coal fly ash is listed in Table I, where the content about 90 % is presented as silica and alumina. Figure 1 is X ray diffraction graph of raw coal fly ash that mullite and quartz are the main phases of raw sample.

Table I. Chemical Analysis of Coal Fly Ash Samples

Chemicals	Content(wt %)	Chemicals	Content(wt%)
$SiO_2$	54.55	MgO	0.82
$Al_2O_3$	33.54	$SO_3$	0.28
$Fe_2O_3$	3.59	$P_2O_5$	0.28
$TiO_2$	1.44	$K_2O$	0.98
CaO	3.80	$Na_2O$	0.31

Mullite was formed through thermal chemical reactions between alumina and silica during the coal combustion process. The alumina minerals usually present in kaolin within the raw coal, and the silica originates from quartz in the raw coal. The mullite diffraction peak of the range (20°-30°) is wide which indicates that vitreous phase exists in the coal fly ash particles [16]. Vitreous phase was formed in the coal fly ash particle's rapidly cooling process from high temperature, which enclosed the particles and reduced chemical activity of the coal fly ash. This is the reason why valuable component in the coal fly ash is hard to be extracted. The agents used in research are analytical reagents and commercially available such as  $H_2SO_4$ ,  $CaCO_3$ ,  $KMnO_4$ ,  $MnSO_4$ .

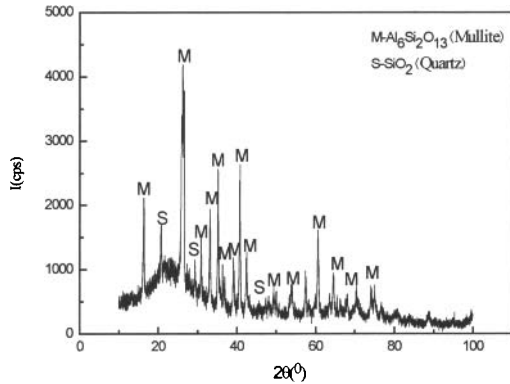


Figure 1. XRD spectrum of the raw coal fly ash.

### Analysis Methods

The raw materials were dried by oven and ground in a vibrating grinder. The energy dispersive X ray fluorescence analyzer (XRF-1800 X) was used to analyze chemical composition of raw and processed samples.

X-ray diffraction analyzer (Rigaku D/MAX-RB12KW) was used to estimate crystallographic phase characteristics of raw and generated samples. Concentrations of the iron in the processed solution was conducted with an ICP analyzer (OPTIMA 7000DV), and aluminum was determined by EDTA titrimetric analysis method.

### Process of Extracting Alumina from Coal Fly-ash

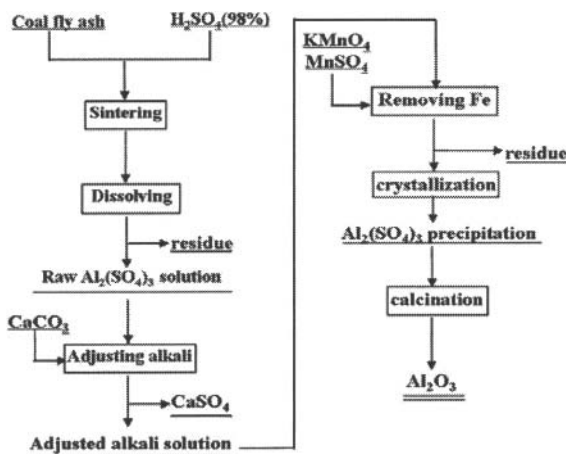


Figure 2. Schematic chart of extracting alumina from coal fly ash through acid sintering-leaching process.

Figure 2 shows the experimental process of extracting alumina from the coal fly ash, where it combines sulfuric acid sintering process and water dissolving as well as crystallization in aluminum sulfate and calcination of aluminum sulfate. A mixture of coal fly ash and concentrated sulfuric acid was sintered for a certain time in open atmosphere and the resulting solids were dissolved into hot water. With the addition of CaCO<sub>3</sub> in filtrate,

the alkalinity of the solution was raised, and iron ion was deposited and removed by adding KMnO<sub>4</sub> and MnSO<sub>4</sub>. The purified aluminum sulfate solution was then used for Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> precipitation which became Al<sub>2</sub>O<sub>3</sub> after calcinating at high temperature.

## Results and Discussions

### Concentrated Sulfuric Acid Sintering and Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> Formation

Figure 3 shows the mass loss of mixed sample in the heating process, which mainly has three sections in heating process from ambient temperature to 500 °C. In the time range of 0 to 80 min, the temperature continues to rise as the weight of the mixture keeps approximately stable. In the time extent between 80 and 120 min, the sample finishes its heterogeneous mass loss reaction and has high mass loss speed. The mass of the mixture have gradually stable trend from the point at 120 min to the end of test. According to the obtained test results, it can be estimated that the temperature range matching the weight loss process is from 234 °C to 362 °C.

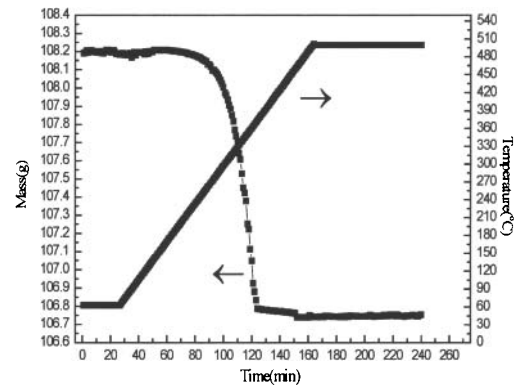


Figure 3. Mass loss image of mixture sintering process.

As the silicon content can not react with concentrated sulfuric acid and calcium component form insoluble matter in the sintering process, it only consider aluminum and iron content in the chemical reactions which form soluble product in the sintering process. Therefore, sintering process of the mixture of coal fly ash and concentrated sulfuric acid may involve the following chemical reaction equations:

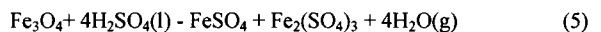
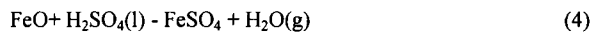
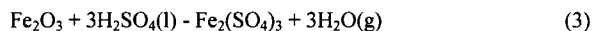
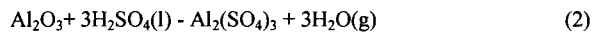
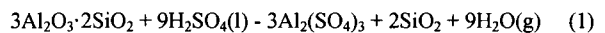


Figure 4 presents the thermodynamics calculated results of the relationship  $\Delta G$ -T for reactions (1)-(5) using universal thermodynamic data. It shows that reactions from (1) to (5) have evident possibility and tendency in the research temperature range, and forward reaction trend decreases with increasing

temperature. In the case of adding sufficient concentrated sulfuric acid, the major products should be  $Al_2(SO_4)_3$ ,  $Fe_2(SO_4)_3$  and  $FeSO_4$ . At the same time, the concentrated sulfuric acid volatilizes seriously at high temperature.

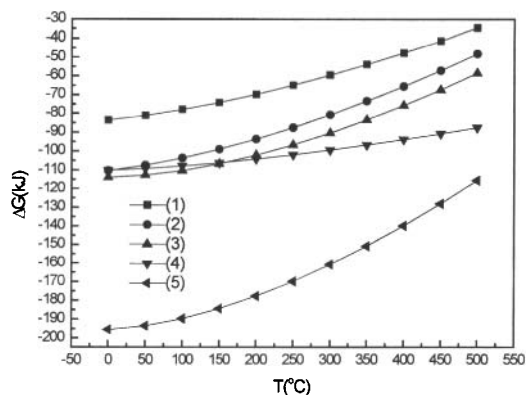


Figure 4. Relationship of  $\Delta G-T$  for reactions (1)-(5).

Figure 5 shows various outcomes like sintered mixture, dissolved residue and aluminum sulfate crystal of sintering experiments conducted at 4 temperature conditions. Relatively speaking, the sample in sintering process at 220 °C forms more sintered mixture and less dissolved residue which indicates the sintering reaction between coal fly ash and concentrated sulfuric acid is more thorough and plenary at this sintering temperature.

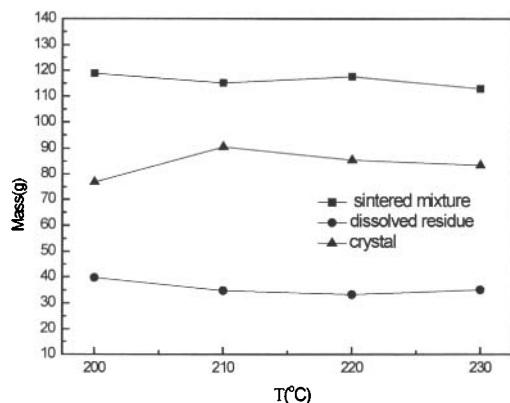


Figure 5. Curves of quantities of chemical products against processing temperature.

It is postulated that the probability of forming aluminum sulfate decreases with increasing temperature, high temperature leads to a rapid volatilization of sulfuric acid and reduction of energy consumption as much as possible, so the experimental research selects 220 °C as mixed material sintering temperature which is matched with the 1.5:1 of concentrated sulfuric acid to coal fly ash mass ratio and 4 h of sintering time.

The X ray diffraction graph of sintered sample of extracting alumina from coal fly ash at 220 °C is presented in Figure 6. Aluminum sulfate and silica phases are identified in the sintered sample and the intensity of aluminum sulfate diffraction peaks are very strong in the sintered material as showed in Figure 6. At the same time, the intensity of silica diffraction peaks is lowered comparing with the X ray diffraction graph of raw coal fly ash.

The declination of silica diffraction peaks in the sintered sample can be attributed to the mass substantial increasing of forming aluminum sulfate from alumina. The strong aluminum sulfate diffraction peaks confirm that coal fly ash and concentrated sulfuric acid mixture in sintering conditions can form water soluble aluminum sulfate product. This provides theoretical foundation to extracting alumina from coal fly ash using acid method and proves that it is feasible using the concentrated sulfuric in the first step phase transition process of acid sintering method.

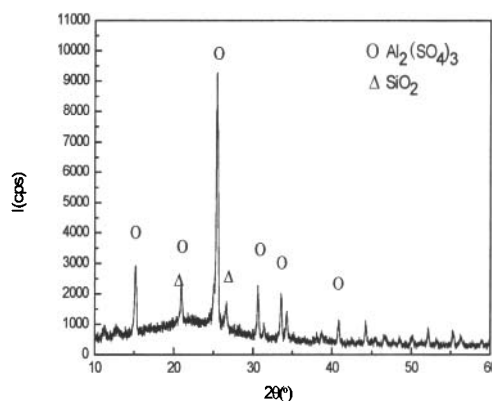


Figure 6. XRD analysis of the sintering product at 220 °C.

#### Dissolving in Aqueous Solution

The water temperature selection in the dissolving process is 85 °C. An overly high temperature of the water will increase evaporation capacity and energy consumption while a too low temperature will greatly affect aluminum sulfate dissolving efficiency in stirring hot water. The liquid to solid ratio of 6:1, the stirring speed of 500 rpm, dissolving time of 30 min are chosen to achieve the goal of dissolving aluminum sulfate formed in sintering process as much as possible.

Table II. Chemical Analysis of Dissolved Residues

Chemicals	Content(wt%)	Chemicals	Content(wt%)
SiO <sub>2</sub>	74.79	Fe <sub>2</sub> O <sub>3</sub>	2.43
Al <sub>2</sub> O <sub>3</sub>	10.50	TiO <sub>2</sub>	0.85
SO <sub>3</sub>	7.48	CaO	2.18

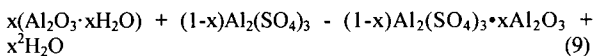
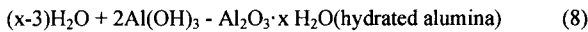
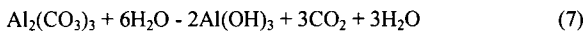
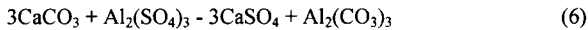
Table II shows the chemical contents in the coal fly ash dissolved residue of the investigation process, which presents higher silica and lower alumina comparing with the raw coal fly ash samples. The sharp rising of silica content indicates that the mullite phase containing most of alumina of coal fly ash is decomposed by concentrated sulfuric acid. The declination of alumina content proves that aluminum sulfate formed in the experiment research is dissolved in the hot water leaching process as well as the use of concentrated sulfuric acid sintering method to extract alumina from coal fly ash is feasible. Furthermore, the residue sample still have a little amount of alumina, which demonstrates that fraction of mullite is not decomposed by concentrated sulfuric acid in the sintering process. Sulfur content converted into sulfur trioxide content in the analysis results increases substantially, so insoluble

sulfate forms in the sintering process between minerals in the coal fly ash and concentrated sulfuric acid as calcium sulfate. The process will be optimized uninterruptedly in the posterior study.

### Removing Iron and Crystallization

KMnO<sub>4</sub> is a strong oxidizing agent so Fe<sup>2+</sup> in solution will be oxidized into Fe<sup>3+</sup> by MnO<sub>4</sub><sup>-</sup> and Fe<sup>3+</sup> rapidly hydrolyzes to form Fe(OH)<sub>3</sub> deposit, then it uses the right amount of MnSO<sub>4</sub> to remove excess KMnO<sub>4</sub> in the solution based on the theory that Mn<sup>7+</sup> and Mn<sup>2+</sup> can generate MnO<sub>2</sub> precipitate. It can achieve the aim of removing iron from solution because Fe(OH)<sub>3</sub> and MnO<sub>2</sub> form flocculation precipitate with a stoichiometric form Fe(OH)<sub>3</sub>·MnO<sub>2</sub>·H<sub>2</sub>O. When active manganese dioxide which is generated by the reaction of MnSO<sub>4</sub> and KMnO<sub>4</sub> is insufficient, iron removal rate is low, and excessive formation of manganese dioxide can not only increase the cost but also don't reach the best effect of iron removal.

The alkalinity of solution for iron removal effect is very large. It is more easy to form Fe(OH)<sub>3</sub>·MnO<sub>2</sub>·H<sub>2</sub>O and a small amount of Al(OH)<sub>3</sub> precipitation by increasing the solution alkalinity. At the same time, forming aluminum precipitation under the conditions of good iron removal effect influences the recovery rate of aluminum sulfate. Aluminum sulfate is an ionic compound and easily soluble in water, and the aluminum sulfate solution is acidity because Al<sup>3+</sup> hydrolyze. To add the CaCO<sub>3</sub> powder into the dissolved solution from the leaching process can generate moisture indefinite hydrated alumina Al<sub>2</sub>O<sub>3</sub>·xH<sub>2</sub>O, which can also be written in the form of Al<sub>2</sub>(OH)<sub>3</sub>. Alkalinity of the Al<sub>2</sub>O<sub>3</sub>·xH<sub>2</sub>O is slightly stronger than the acidity. The active component Al<sub>2</sub>O<sub>3</sub> has strong affinity to SO<sub>2</sub>, so it can form basic complex (1-x)Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·xAl<sub>2</sub>O<sub>3</sub> which often referred to as basic aluminum sulfate. The principle is clarified through chemical reaction equations (6)-(9).



According to the experimental results from Lin Geng [17] which reported the optimum ratio (2:1)-(4:1) for n (MnO<sub>2</sub>): n (Fe), it choose ratio 3:1 in the experiment research. The chemical reactions principle is showed in reactions (10)-(13).

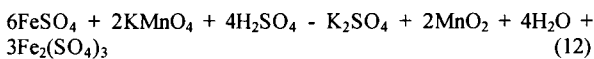
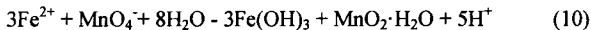


Figure 7 shows the iron concentration of the produced solution in the research such as dissolved solution, direct removing iron solution, solution adjusted alkali and removed iron. It can be seen that effect of direct removing iron from dissolved solution is

distinct, but the solution iron content still maintains at a higher level. After adding adjusting alkali step, solution iron content further substantially declines to 3 mg/L below. The result indicates that using the iron removal step matched adjusting alkali step is completely feasible. In addition, the coal fly ash treated through the conditions of sintered temperature 220 °C and acid to ash ratio 1.5:1 not only decomposes mullite to extract alumina but also controls the formation of ferric sulfate in the sintering process. It also can be seen that the increasing ratio of acid to ash results in more iron in coal fly ash into the solution, and the reaction between aluminum content and concentrated sulfuric acid is prior to the reaction of concentrated sulfuric acid and iron content in coal fly ash under the conditions of appropriate acid to ash ratio.

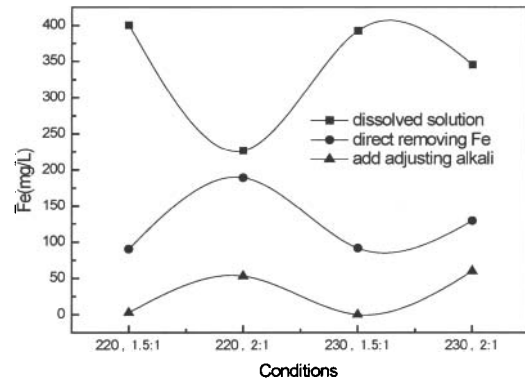


Figure 7. Fe concentrations of the produced solutions in process.

X ray diffraction graphs of adjusting alkali residue, iron removing residue and crystallized product after adjusting alkali and removing iron are presented in Figure 8 to 10.

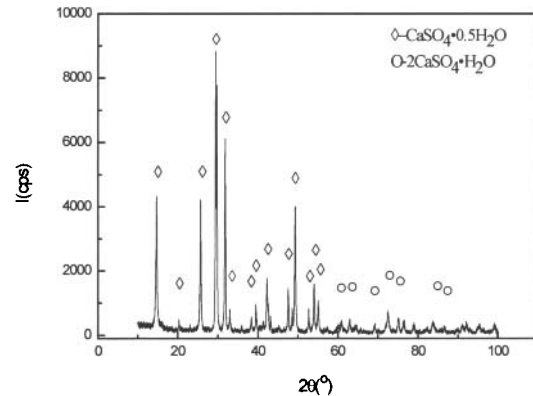


Figure 8. XRD analysis of the adjusting alkali residue.

Adjustable alkali residue composition accurately presents CaSO<sub>4</sub> and 2CaSO<sub>4</sub> which shows polymerization phenomenon of CaSO<sub>4</sub> in solution and can further prevent CaSO<sub>4</sub> from dissolving into solution again. The CO<sub>2</sub> gas generated by adding CaCO<sub>3</sub> into the solution can be recovered for two reasons, one is comprehensive utilization of resources, the other is that carbon dioxide can be used to generate the aluminum hydroxide from the sodium aluminate solution if the following purification step of Al<sub>2</sub>O<sub>3</sub> joins in the whole technical process. The Al(OH)<sub>3</sub> characteristic peaks

don't appear in X ray diffraction graph can demonstrate that the aluminum loss in the adjusting alkali process is very small.

The main phase is  $MnO_2$  in the adjusted alkali and removed iron residue, which shows that removal of excess  $KMnO_4$  by adding  $MnSO_4$  is correct and feasible. The characteristic diffraction peak is not particularly evident in the Figure 9, which shows that the  $MnO_2$  presented in residue appears in amorphous form. The  $Fe(OH)_3$  characteristic peaks don't appear in X ray diffraction graph demonstrates that a small quantity of  $Fe(OH)_3$  is enclosed inside sphere bodies surfaced with a mass of  $MnO_2$  in the adjusted alkali and removes iron residue.

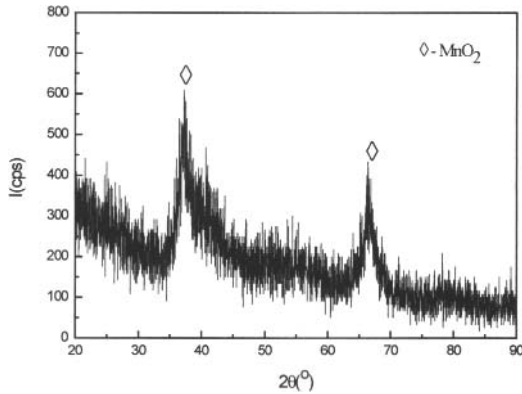


Figure 9. XRD analysis of the Fe removing residue.

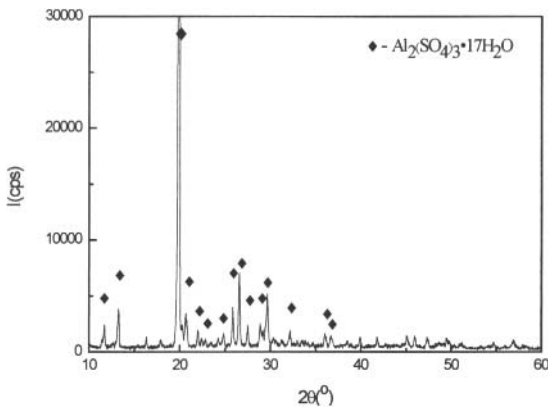


Figure 10. XRD analysis of the crystallized product.

Concentrated crystallization process of aluminum sulfate from solution removed iron is executed by the relationship of solution temperature and solubility of aluminum sulfate. When the temperature of the solution reaches 110 °C - 115 °C, the heating operation is terminated and aluminum crystals are gradually separated out through the natural cooling solution. Filtering solution and separating precipitate can obtain the aluminum sulfate crystal.

The diffraction peaks of pure crystal  $Al_2(SO_4)_3 \cdot 17H_2O$ , generated from the solution after removing iron, are outstanding and have great strength. This phenomenon indicates that the experimental research process of extracting  $Al_2O_3$  from coal fly ash is completely doable. Adding iron removal step in the experiments avoids the influences of main impurities Fe in the solution on the nucleation process of aluminum sulfate and crystal's morphology

and composition. Sintering process between concentrated sulfuric acid and raw coal fly ash can make accurate phase transformation from alumina to aluminum sulfate, then the adding adjusting alkali and iron removal step can eliminate the impurity interference from aluminum sulfate and ensure the purity of the product.

#### Calcinations of $Al_2(SO_4)_3 \cdot 17H_2O$

The calcination of aluminum sulfate crystal is described through chemical reaction equations (14)-(16).

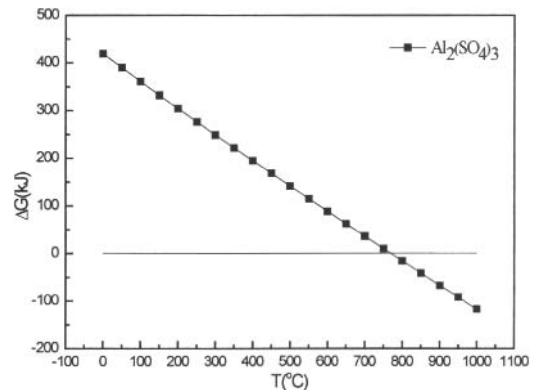
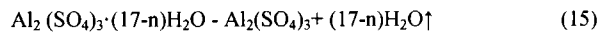
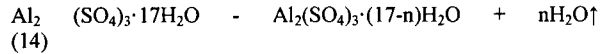


Figure 11. Relationship of  $\Delta G-T$  for reactions (16).

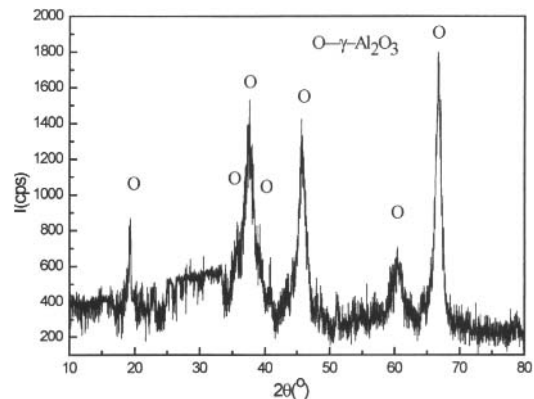


Figure 12. XRD analysis of the calcinations product.

Figure 11 is the  $\Delta G-T$  relationship of reaction (16), which theoretically expound that the formation of  $Al_2O_3$  generated by decomposition of  $Al_2(SO_4)_3$  at 750 °C. The aluminum electrolysis process mainly uses sandy alumina which contains only 10-15%  $\alpha-Al_2O_3$  and the rest is  $\gamma-Al_2O_3$ . The transformation temperature from  $\gamma-Al_2O_3$  to  $\alpha-Al_2O_3$  is the range of 1100 °C to 1200 °C. We operate the calcination process of  $Al_2(SO_4)_3 \cdot 17H_2O$  at 850 °C for 2 h. The X ray diffraction graph of product  $\gamma-Al_2O_3$  is showed in

Figure 12, which is observed that aluminum sulfate crystal has transformed into  $\gamma$ - $\text{Al}_2\text{O}_3$  completely by calcination process.

$$\eta = \frac{M_2 \times C_2}{M_1 \times C_1} \times 100\% \quad (17)$$

The alumina extracting rate in coal fly ash is calculated by formula (17). The parameters such as  $M_1$ ,  $M_2$ ,  $C_1$ ,  $C_2$  in the formula respectively represents the dosage of coal fly ash, yield of alumina, alumina content percentage in coal fly ash, purity of alumina. By calculating statistics data of many experiments, the alumina extracting rate of the experimental process is from 70 % to 90 %.

### Conclusions

1. The alumina in coal fly ash can be extracted in the form of hydrosoluble aluminum sulfate using the concentrated sulfuric acid sintering method at 220 °C for 4 h. The selected operating temperature can avoid heavy volatilization of concentrated sulfuric acid and ensure a complete reaction of raw materials.
2. In the leaching process, the liquid to solid ratio of 6:1 and dissolving temperature 85 °C can ensure that aluminum sulfate generated in the sintering step more thoroughly dissolved into the solution. Adjusting alkali process with  $\text{CaCO}_3$  and removing iron through  $\text{KMnO}_4$  and  $\text{MnSO}_4$  can make Fe level down to 3 mg/L in solution, thus avoiding Fe impurity in the final alumina product.
3. The quality of  $\gamma$ - $\text{Al}_2\text{O}_3$  product demonstrates the technical feasibility of the process of extracting alumina from coal fly ash developed in this investigation, which may avoid the disadvantages of a relative large amount of calcium silicate slag generated in the alkali treatment, as well as the complex long process of the acid-alkali combination method. The processing method is still under development and optimization for potential industrial application.

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