

Extraction of alumina from fly ash by ammonium hydrogen sulfate roasting technology

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Abstract: NH_4HSO_4 roasting technology was used for preparing Al_2O_3 from fly ash. First, Al and Fe were extracted from fly ash by NH_4HSO_4 roasting and deionized water leaching. Then, the Al and Fe in the leached liquid were precipitated by adding NH_4HCO_3 solution. After the mixed precipitations of $\text{Al}(\text{OH})_3$ and $\text{Fe}(\text{OH})_3$ were leached by NaOH solution, the $\text{NaAl}(\text{OH})_4$ solution was decomposed by carbonation. Finally, the pure $\text{Al}(\text{OH})_3$ was calcined to $\alpha\text{-Al}_2\text{O}_3$. The optimal conditions of the whole technology were determined by experiments. The quality of $\alpha\text{-Al}_2\text{O}_3$ product is up to the technical indicator of YS/T 274-1998 standard.

Key words: fly ash; ammonium hydrogen sulfate; alumina; extraction

1 Introduction

Fly ash is a fine ash separated from the flue gas of coal-fired power plant burning pulverized coal [1], and it is the main by-product from power plant. By 2020, total amount of fly ash in China will reach more than 3 billion tons. A large proportion of fly ash is used by the building industry, highway road bases and grout mixes [2–6]. However, despite these positive uses, the production rate of fly ash is much greater than its consumption [7]. There is still a proportion which is disposed of in ponds or landfill, so fly ash has become the main waste of power plant. Thus, it will be necessary to improve value-added utilization of fly ash. Al_2O_3 industry has been developed rapidly in China in recent years. Therefore, there is an increasing focus on extracting Al_2O_3 from fly ash, along with the reduction of bauxite reserves and the decrease of minable ore grade.

At present, the typical metallurgical methods for extracting Al_2O_3 from fly ash include soda-lime sintering [8–10] and acid leaching [11–13]. Soda-lime sintering is considered to have great potential for industrial use, as it is similar to the major technology currently used in the Al_2O_3 plants nowadays [14]. Whereas, this method has some defects, such as high energy consumption [15],

large amount of waste residue and more Al_2O_3 loss. Acid leaching method can effectively separate Al and Si, but due to the high requirement for equipment [16] and high cost, this method has not been applied in industrial practice.

In this work, a novel technology for extracting Al_2O_3 from fly ash by NH_4HSO_4 roasting technology was established. Compared with the above methods, NH_4HSO_4 roasting technology has lower energy consumption, higher Al extraction rate and better operating environment. The technology includes roasting process, aluminum precipitation, alkali dissolution, carbonation decomposition and calcination. First, Al and Fe were extracted from fly ash by NH_4HSO_4 roasting and deionized water leaching. Second, the leached liquid containing Al and Fe was used for aluminum precipitation by NH_4HCO_3 solution. Third, the mixed precipitations of $\text{Al}(\text{OH})_3$ and $\text{Fe}(\text{OH})_3$ were leached by NaOH solution to remove Fe. Fourth, the $\text{NaAl}(\text{OH})_4$ solution was decomposed by carbonation. At last, the pure $\text{Al}(\text{OH})_3$ was prepared for $\alpha\text{-Al}_2\text{O}_3$ by calcination. The Al_2O_3 -extracted slag was used as raw material to recover SiO_2 . The present work mainly focuses on preparing $\alpha\text{-Al}_2\text{O}_3$ product and optimizing conditions of the whole process. Moreover, the optimal operation parameters were determined by experiments.

2 Experimental

NH_4HSO_4 and NaOH were industrial grade; ZnSO_4 , EDTA-2Na , $\text{K}_2\text{Cr}_2\text{O}_7$ and other reagents were analytical grade. Deionized water was used throughout the experiments whenever needed. Fly ash used in this study was got from a coal-fired power plant in Inner Mongolia, China, the size fraction of which mainly ranged from 61 to 74 μm . The detailed chemical composition was examined by ICP. The analytical result, which is listed in Table 1, indicates that the main components of fly ash are Al_2O_3 and SiO_2 . The XRD analysis was performed using $\text{Cu K}\alpha$ radiation ($\lambda=1.5406$ nm) at 40 kV and 30 mA. From the XRD result shown in Fig. 1, it can be seen that the major mineral phases of fly ash are mullite and $\alpha\text{-SiO}_2$.

Table 1 Composition of fly ash from a power plant in Inner Mongolia, China (mass fraction, %)

Al_2O_3	SiO_2	Fe_2O_3	CaO	TiO_2
38.53	45.41	2.38	2.73	1.85

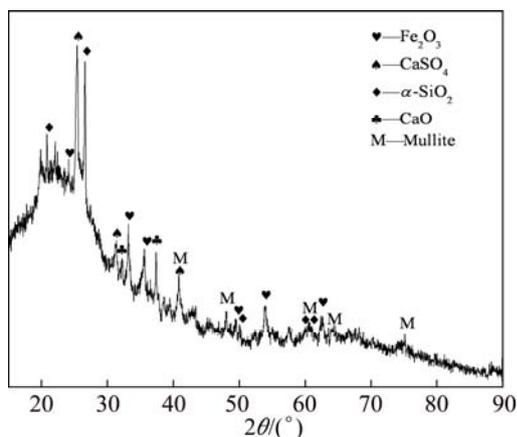
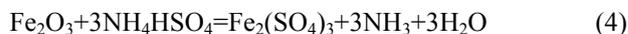
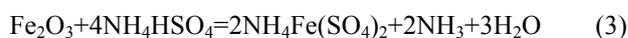
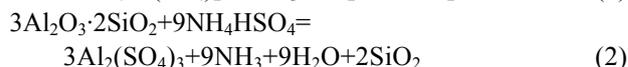
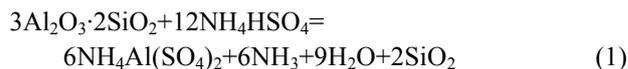


Fig. 1 XRD pattern of raw fly ash

2.1 Procedure of NH_4HSO_4 roasting

Fly ash was mixed with NH_4HSO_4 at a suitable mole ratio of Al_2O_3 in fly ash and NH_4HSO_4 . The resistance wire furnace was used, and the temperature was controlled by a programmable temperature controller with a precision of ± 2 °C. The reaction began at a required temperature. After a selected time, the roasted clinker was immediately taken out. It was leached by deionized water, and then filtered. The contents of Al and Fe in the leached liquid were detected by EDTA titration and $\text{K}_2\text{Cr}_2\text{O}_7$ titration, respectively. The Al_2O_3 -extracted slag was used for raw material of recovering SiO_2 .

The main chemical reactions taking place between fly ash and NH_4HSO_4 are as follows:



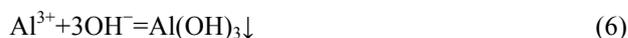
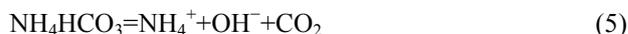
Three key roasting parameters, namely roasting temperature, mole ratio of $\text{Al}_2\text{O}_3/\text{NH}_4\text{HSO}_4$ and roasting time, were optimized by orthogonal experiment.

2.2 Procedure of aluminum precipitation

The concentrations of Al and Fe in the leached liquid were as follows: $C_{\text{Al}} \approx 5.5$ g/L, $C_{\text{Fe}} \approx 0.3$ g/L. 250 mL leached liquid was used for each experiment.

The leached liquid was heated from 25 to 99 °C. When the temperature was reached, 10% NH_4HCO_3 solution was dropped into the leached liquid under stirring, and then the Al and Fe were precipitated. When the pH value reached 4–7, the reaction began. After a certain time, the reaction solution was filtered. The filter cake, which was the mixed precipitations of $\text{Al}(\text{OH})_3$ and $\text{Fe}(\text{OH})_3$, was used for alkali dissolution. The filtrate can be used to prepare the crude product of $(\text{NH}_4)_2\text{SO}_4$ by the evaporation crystallization process.

The main chemical reactions are as follows:



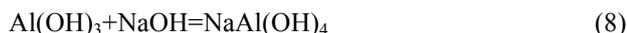
Three key parameters including temperature, pH value and holding time were optimized by single factor experiments.

2.3 Procedure of alkali dissolution

Fe, which is the impurity of strong interference, can decrease the quality of $\alpha\text{-Al}_2\text{O}_3$ product. Therefore, the key problem of preparing $\alpha\text{-Al}_2\text{O}_3$ product from fly ash is deironing. NaOH solution can be used to separate Al and Fe, because $\text{Al}(\text{OH})_3$ is dissolved while $\text{Fe}(\text{OH})_3$ is not dissolved in NaOH solution.

According to the Al content in the mixed precipitations of $\text{Al}(\text{OH})_3$ and $\text{Fe}(\text{OH})_3$, the dosage of NaOH (solid) can be calculated. The alkali dissolution process was investigated at $\text{Al}(\text{OH})_3$ to NaOH mass ratio of 1:1–1:5. These NaOH (solid) and 250 mL deionized water were mixed, and then heated to the temperature of 25–99 °C. When the temperature reached and remained stable, the mixed precipitations were added to the NaOH solution under the condition of continuous stirring. After a period of time, the reaction solution was filtered. The residue was the precipitation of $\text{Fe}(\text{OH})_3$, and the filtrate was $\text{NaAl}(\text{OH})_4$ solution.

The following reaction could occur in this process:



Three key parameters, which include temperature, mass ratio of $\text{Al(OH)}_3/\text{NaOH}$ and holding time, were determined by single factor experiments.

2.4 Procedure of carbonation decomposition

The NaAl(OH)_4 solution was heated to 25–99 °C. At the selected temperature, CO_2 was blew into the solution at a gas-flow rate of 20–100 mL/min. The reaction solution was stirred under atmospheric pressure. After a certain time, the reaction solution was filtered. The filter cake was the precipitation of Al(OH)_3 , and the filtrate was Na_2CO_3 solution in which CaO can be added for causticizing.

The chemical reactions occurred can be written as [17,18]:



There were two essential processes in carbonation decomposition. One was the neutralization reaction between NaOH and CO_2 (Reaction (9)); the other was the process of Al(OH)_3 precipitation (Reaction (10)).

Three key parameters, namely temperature, gas-flow rate of CO_2 and holding time, were examined by single factor experiments.

2.5 Procedure of calcination

Al(OH)_3 was calcinated at 1200 °C for 2 h, then the $\alpha\text{-Al}_2\text{O}_3$ product was obtained.

The chemical reactions occurred are listed in Table 2 [19,20]:

Table 2 Chemical reactions occurred at different temperatures

Temperature/°C	Reaction	Number
100–120	$2\text{Al(OH)}_3 + \text{H}_2\text{O}_{\text{attached}} = \text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O} + \text{H}_2\text{O}$	(11)
200–250	$\text{Al}_2\text{O}_3 \cdot 3\text{H}_2\text{O} = \text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O} + 2\text{H}_2\text{O}$	(12)
700	$\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O} = \gamma\text{-Al}_2\text{O}_3 + \text{H}_2\text{O}$	(13)
900	$\gamma\text{-Al}_2\text{O}_3 = \theta\text{-Al}_2\text{O}_3$	(14)
1150–1200	$\theta\text{-Al}_2\text{O}_3 = \alpha\text{-Al}_2\text{O}_3$	(15)

The whole technological flow of extracting Al_2O_3 from fly ash is shown in Fig. 2.

3 Results and discussion

3.1 Procedure of NH_4HSO_4 roasting

Table 3 lists the schedule of the orthogonal experiment, in which the key parameters including

roasting temperature (A), $\text{Al}_2\text{O}_3/\text{NH}_4\text{HSO}_4$ mole ratio (B) and roasting time (C) are selected as three factors. And every factor has three levels to be optimized. The Al extraction rate is taken as the index point to evaluate the roasting performance under different factors and levels.

The orthogonal experiment results are listed in Table 4. From the mathematical processing of the data, some results can be concluded. First, the factor of $\text{Al}_2\text{O}_3/\text{NH}_4\text{HSO}_4$ mole ratio shows the most notable influence on Al extraction rate. Then, roasting temperature is the relatively remarkable factor compared with roasting time. The optimal conditions of roasting process were determined as 400 °C, 1:8 and 45 min, for roasting temperature, $\text{Al}_2\text{O}_3/\text{NH}_4\text{HSO}_4$ mole ratio and roasting time, respectively. Moreover, 90.11% of Al extraction rate was achieved under the optimal roasting parameters.

The Al_2O_3 extracted slag was characterized by chemical analysis, XRD and SEM. The chemical compositions of Al_2O_3 -extracted slag are shown in Table 5. Compared with Table 1, it is evident that the Al_2O_3 content is lowered, while the SiO_2 content is improved. The purposes of extracting Al_2O_3 and cumulating SiO_2 from fly ash are realized. The XRD pattern of Al_2O_3 - extracted slag is shown in Fig. 3, indicating that the major mineral phase is $\alpha\text{-SiO}_2$. The morphologies of fly ash and Al_2O_3 -extracted slag were analyzed by SEM. It can be seen from Fig. 4 that the contact surface of fly ash is eroded by NH_4HSO_4 .

3.2 Procedure of aluminum precipitation

The effects of temperature, pH value and holding time on Al and Fe precipitation rates are shown in Fig. 5. These results manifest that increasing pH value and holding time while decreasing temperature could increase Al and Fe precipitation rates. The decomposability of NH_4HCO_3 is enhanced with the increase of temperature. Thereby, high temperature is unfavorable to the reaction. However, Al(OH)_3 and Fe(OH)_3 are amorphous precipitates, which need high temperature to accelerate the precipitation rate and decrease the adsorption. For this reason, 80 °C is chosen (Fig. 5(a)). It is found from Fig. 5(b) that the precipitation rate of Al at pH value of 7 is lower than that at pH value of 6. This is because a small amount of Al(OH)_3 is transformed into Al(OH)_4^- at pH value of 7, that is, Al^{3+} returns to the solution. As seen from Fig. 5(c), Al is precipitated completely after 40 min.

The optimal conditions for the process of aluminum precipitation are temperature of 80 °C, pH value of 6 and holding time of 40 min. Under these conditions, the precipitation rates of Al and Fe are up to 99% and 98%, respectively.

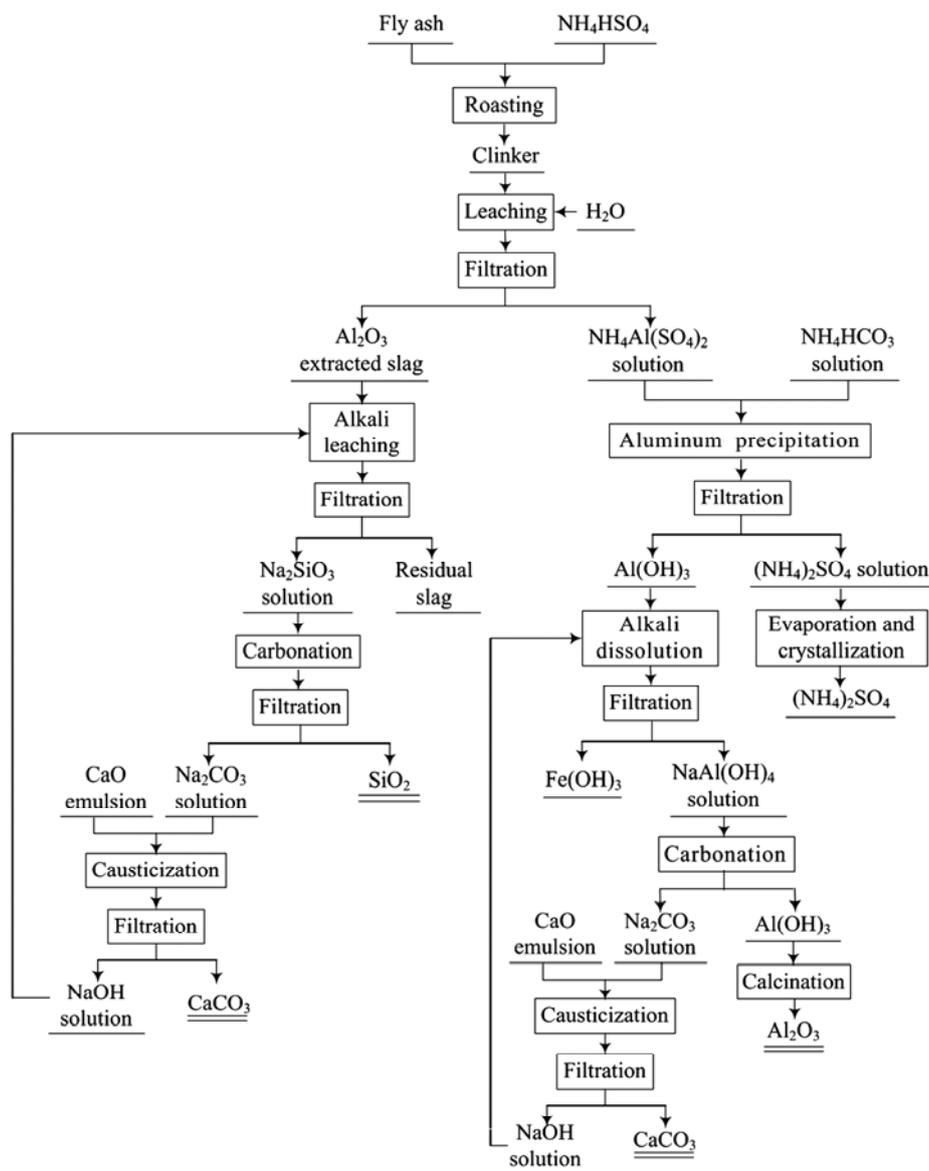


Fig. 2 Flow diagram of extracting Al₂O₃ from fly ash

Table 3 Factors and levels selected for orthogonal experiment

Level	Factor		
	Roasting temperature, A/°C	Al ₂ O ₃ /NH ₄ HSO ₄ mole ratio, B	Roasting time, C/min
1	300	1:6	30
2	350	1:7	45
3	400	1:8	60

3.3 Procedure of alkali dissolution

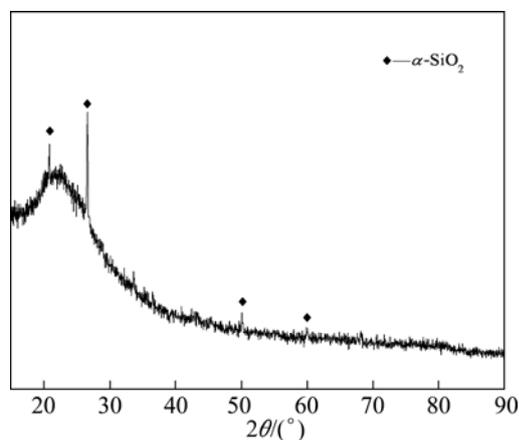
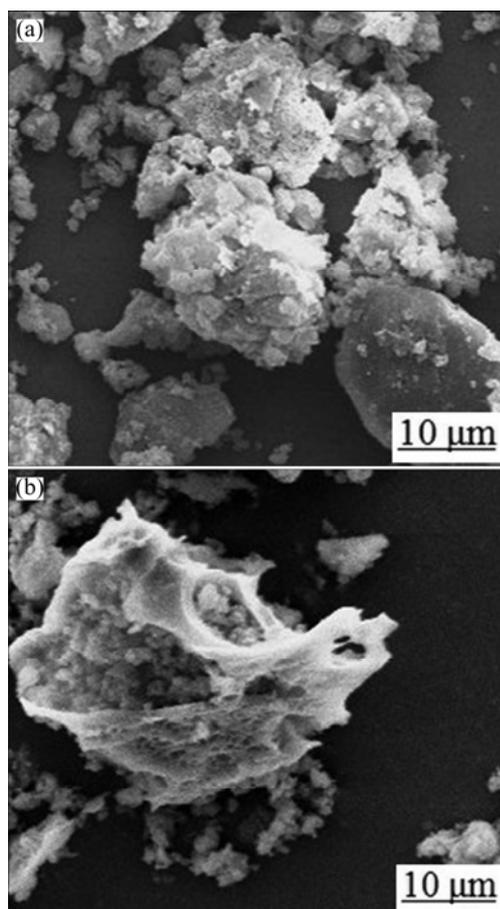
The influences of temperature, Al(OH)₃/NaOH mass ratio and holding time on Al alkali dissolution rate and deironing rate are shown in Fig. 6. It is evident that increasing temperature, Al(OH)₃/NaOH mass ratio and holding time increase the alkali dissolution rate of Al and deironing rate. The increase of temperature would

Table 4 Orthogonal experiment results

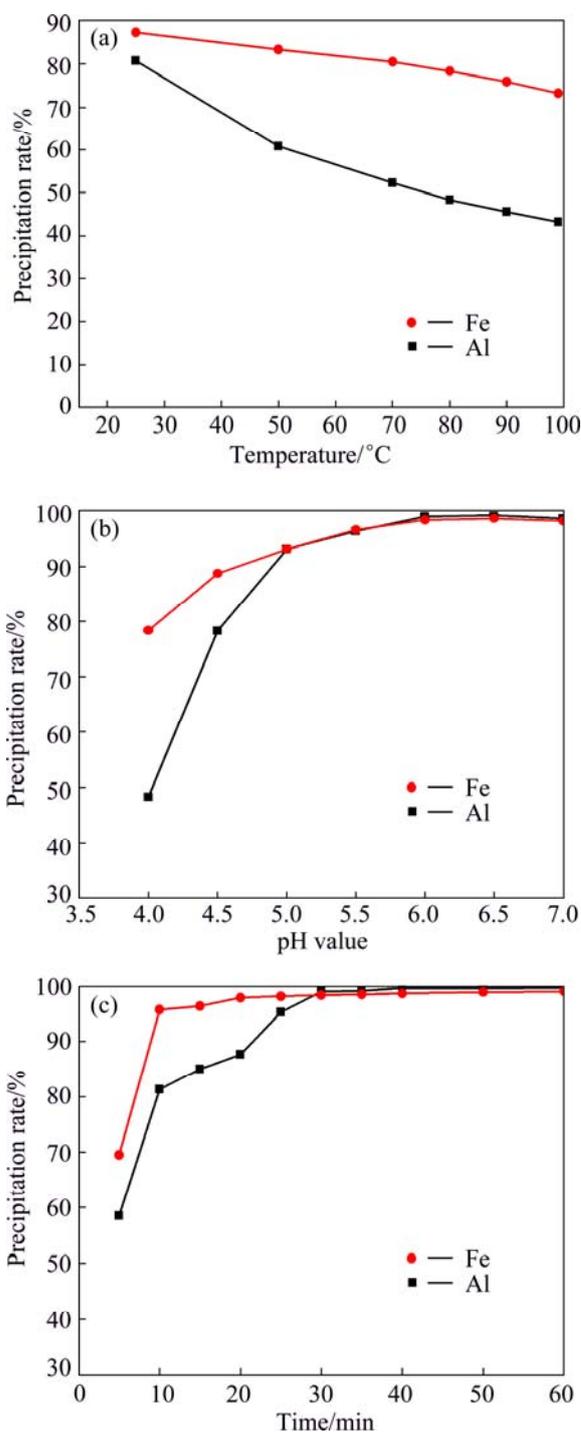
No.	Level of A	Level of B	Level of C	Al extraction rate/%
1	1	1	1	70.48
2	1	2	2	83.87
3	1	3	3	84.95
4	2	1	2	81.39
5	2	2	3	87.96
6	2	3	1	86.92
7	3	1	3	83.19
8	3	2	1	86.30
9	3	3	2	90.11
<i>d</i> _{1,j} /%	79.767	78.353	81.233	
<i>d</i> _{2,j} /%	85.423	86.043	85.123	
<i>d</i> _{3,j} /%	86.533	87.327	85.367	
<i>R</i> /%	6.766	8.974	4.134	

Table 5 Composition of Al₂O₃-extracted slag (mass fraction, %)

Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	CaO	TiO ₂
4.82	90.00	0.03	0.10	0.44

**Fig. 3** XRD pattern of Al₂O₃-extracted slag**Fig. 4** SEM images of fly ash (a) and Al₂O₃-extracted slag (b)

improve the solubility of Al(OH)₃. When the temperature is 80 °C, the alkali dissolution process can achieve a better effect (Fig. 6(a)). Additionally, excess NaOH can make the reactive molecules contact better and ensure sufficient reaction, so Al(OH)₃/NaOH mass ratio of 1:4 is

**Fig. 5** Effects of different conditions on Al and Fe precipitation rates: (a) Different temperatures at pH value of 4 for 30 min; (b) Different pH values at temperature of 80 °C for 30 min; (c) Different holding time at temperature of 80 °C and pH value of 6

chosen (Fig. 6(b)). It can be seen from Fig. 6(c) that the alkali dissolution rate of Al could reach the maximum when the reaction time is fixed at 30 min.

As stated above, the optimal conditions for the process of alkali dissolution are temperature of 80 °C, Al(OH)₃/NaOH mass ratio of 1:4 and holding time of 30

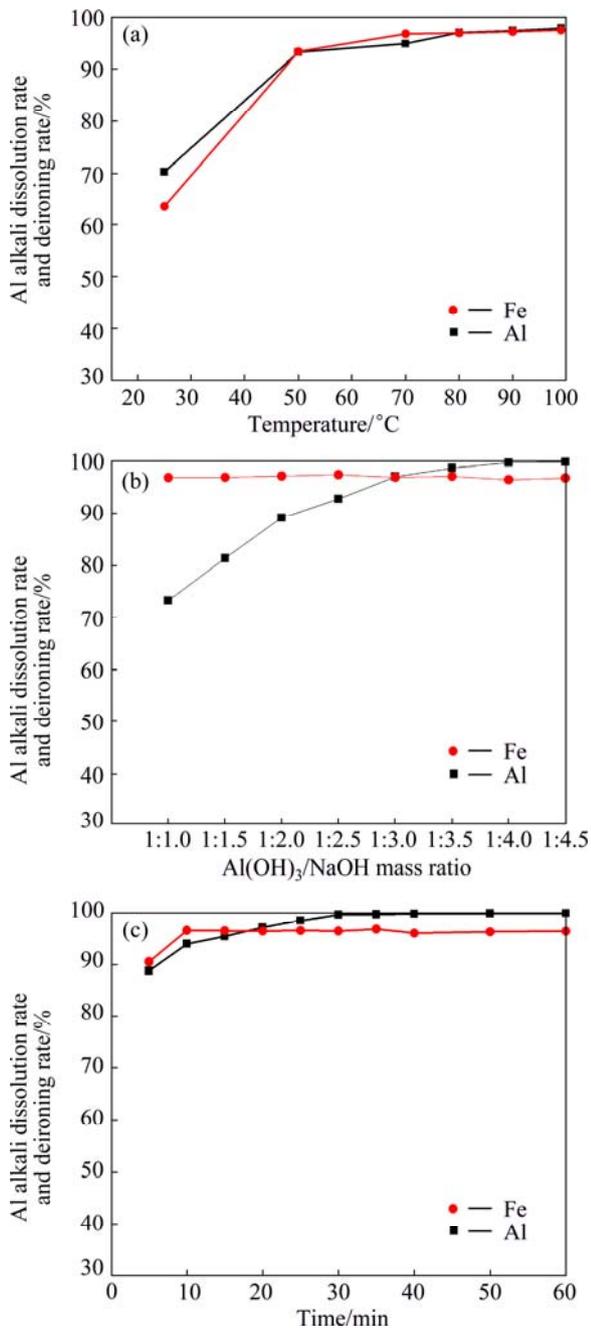


Fig. 6 Effects of different conditions on Al alkali dissolution rate and deironing rate: (a) Different temperatures with Al(OH)₃/NaOH mass ratio of 1:3 for 30 min; (b) Different Al(OH)₃/NaOH mass ratio at 80 °C for 30 min; (c) Different time with mass ratio of 1:4 at 80 °C

min. Under these conditions, the alkali dissolution rate of Al and the deironing rate are more than 99% and 96%, respectively.

There are mainly two reasons responsible for incomplete deironing. First, a small part of Fe, which is absorbed by Al(OH)₃ during the process of aluminum precipitation, is released when this Al(OH)₃ is leached by NaOH solution; second, massive NaOH makes a small amount of Fe(OH)₃ dissolve. In this experiment, the

mixed precipitations of Al(OH)₃ and Fe(OH)₃ are leached directly by NaOH solution without process of drying. Thus, the mixed precipitations have a high reaction activity, which induce the relatively small dosage of NaOH (solid). Considering the above analysis, the reason of incomplete deironing is the first case.

3.4 Procedure of carbonation decomposition

The effects of temperature, CO₂ gas-flow rate and holding time on Al precipitation rate are shown in Fig. 7.

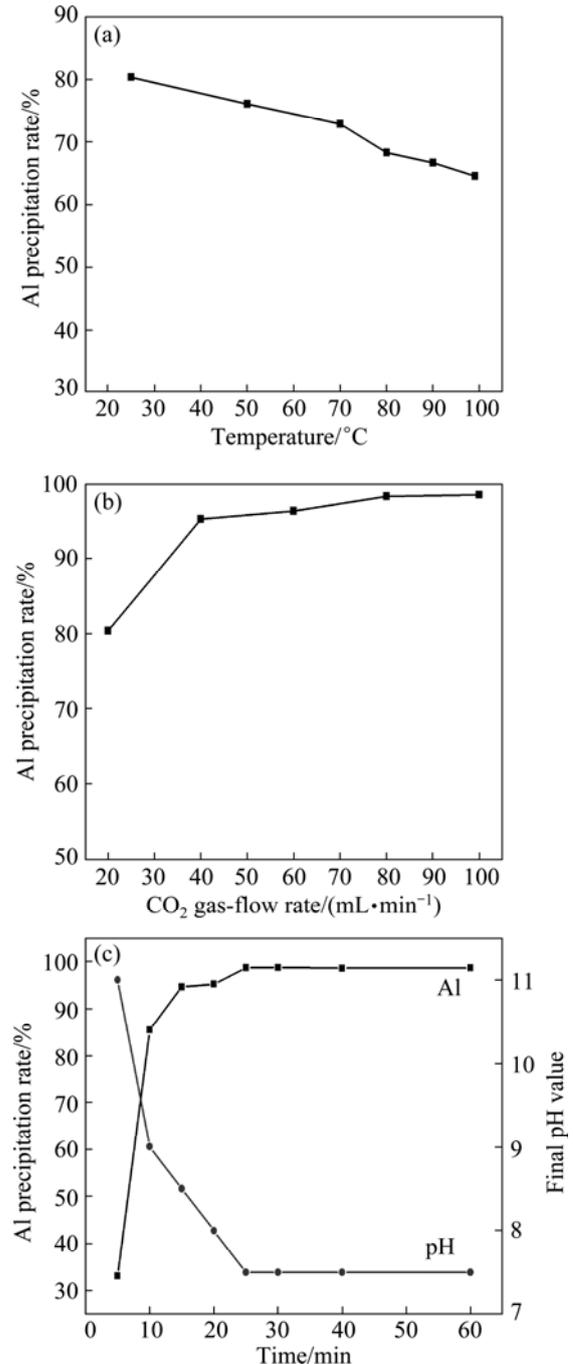


Fig. 7 Effects of different conditions on Al precipitation rate: (a) Different temperatures with CO₂ flow rate of 20 mL/min for 20 min; (b) Different CO₂ flow rates at 25 °C for 20 min; (c) Different time with CO₂ flow rate of 40 mL/min at 25 °C

The results show that increasing CO₂ gas-flow rate and holding time and decreasing temperature improve the Al precipitation rate. The solubility of CO₂ decreases with the increase of temperature, indicating that high temperature is adverse for the process of carbonation decomposition. Thus 25 °C is determined (Fig. 7(a)). It is evident from Fig. 7(b) that the reaction of carbonation decomposition is accelerated with increasing CO₂ gas-flow rate. However, excess CO₂ could affect the economy of the whole process, so CO₂ gas-flow rate is chosen as 40 mL/min. As shown in Fig. 7(c), Al precipitation rate can reach a maximum after 25 min. Also the final pH value decreases with the increase of Al precipitation rate. This manifests that the final pH value is also an index point to evaluate the Al precipitation performance.

According to the experiments result, the optimal conditions for the process of carbonation decomposition are determined as temperature of 25 °C, CO₂ gas-flow rate of 40 mL/min and holding time of 25 min. Under these conditions, Al precipitation rate is up to 98%.

3.5 Characterization of product

After the process of calcination, the α -Al₂O₃ product was obtained, and it was characterized by chemical analysis, XRD and SEM. From the XRD pattern in Fig. 8, it can be seen that the diffraction peaks of the product match with the standard diffraction peaks of α -Al₂O₃, which shows that the product is α -Al₂O₃. As shown in Fig. 9, the SEM image indicates that the fine particles of α -Al₂O₃ product are globular. Table 5 lists the chemical composition of α -Al₂O₃ product. It is found from Table 6 that the Al₂O₃ content of the product is more than 99%, and the quality of α -Al₂O₃ product is up to the technical indicator of YS/T 274-1998 standard.

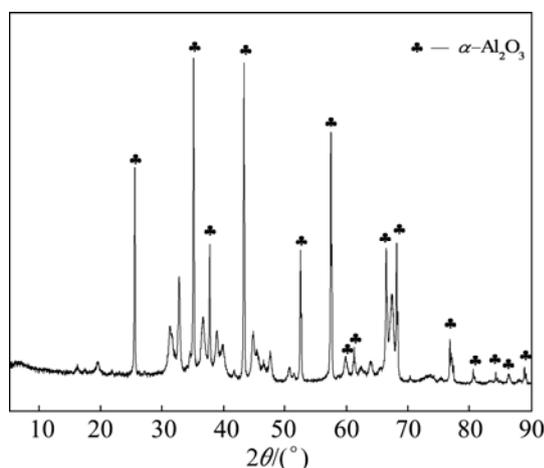


Fig. 8 XRD pattern of α -Al₂O₃ product

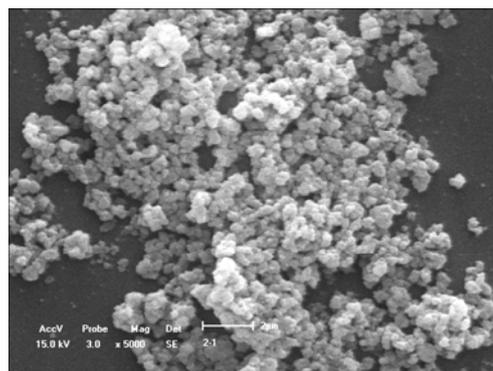


Fig. 9 SEM image of α -Al₂O₃ product

Table 6 Composition of α -Al₂O₃ product (mass fraction, %)

Item	Composition/%					
	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	Na ₂ O	TiO ₂	CaO
YS/T 274-1998	>98.3	0.06	0.04	0.65	-	-
AO-3	>99.0	0.06	0.02	<0.01	<0.01	0.11

4 Conclusions

1) A novel technology was proposed for comprehensive utilization of fly ash, which provides a new route for exploitation and utilization of fly ash.

2) The technology includes five parts: roasting process, aluminum precipitation, alkali dissolution, carbonation decomposition and calcination. The optimal conditions of the whole technology were determined. The Al extraction rate during the roasting process is up to 90.11% at 400 °C in mole ratio of Al₂O₃/NH₄HSO₄ of 1:8 for 45 min. The precipitation rates of Al and Fe are more than 99% and 98%, respectively, under the conditions of temperature 80 °C, pH value 6 and holding time 40 min. The alkali dissolution rate of Al and the deironing rate are 99% and 96%, respectively, and the optimal conditions are as follows: temperature 80 °C, Al(OH)₃/NaOH mass ratio 1:4 and holding time 30 min. The carbonation decomposition rate of Al could reach 98% under normal temperature and pressure, CO₂ gas-flow rate of 40 mL/min and holding time of 25 min.

3) The α -Al₂O₃ product was prepared, the quality of which was up to the technical indicator of YS/T 274-1998 standard.

4) The whole technology has many merits, such as low cost, low energy consumption and good operating environment. It turns waste into useful product and has good social benefit.

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硫酸氢铵焙烧粉煤灰提取氧化铝

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摘要: 采用 NH_4HSO_4 焙烧法从粉煤灰中提取 Al_2O_3 。首先, 通过 NH_4HSO_4 焙烧和去离子水浸出法提取粉煤灰中的 Al 和 Fe; 然后, 加入 NH_4HCO_3 溶液沉淀浸出液中的 Al 和 Fe, 利用 NaOH 溶液浸出得到的 $\text{Al}(\text{OH})_3$ 和 $\text{Fe}(\text{OH})_3$ 混合沉淀, 所得铝酸钠溶液经碳酸化分解得到纯净的 $\text{Al}(\text{OH})_3$; 最后, 煅烧纯净的 $\text{Al}(\text{OH})_3$ 制备 α - Al_2O_3 产品。通过实验确定各工艺流程的最佳条件。制备的 α - Al_2O_3 产品达到 YS/T 274-1998 标准的工艺指标。

关键词: 粉煤灰; 硫酸氢铵; 氧化铝; 提取

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