



## Reaction behavior of silicon-rich diasporic bauxite with ammonium sulfate during roasting

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**Abstract:** The separation of Al from the silicon-rich diasporic bauxite is of great significance in alumina production. Herein, we proposed a low-temperature ammonium sulfate roasting-water leaching process to extract aluminum from silicon-rich diasporic bauxite. Parameters including roasting temperature, dosage of ammonium sulfate, roasting time, and particle size of ore were investigated. Under the condition of roasting temperature of 400 °C, roasting time of 5 h, ammonium sulfate dosage of 2.5 times of the theoretical value and ore particle size of 80–96 μm, more than 98% leaching rate of aluminum was obtained. The phase transformation and mechanism during the roasting process were revealed by using X-ray diffraction, thermogravimetric analysis, differential thermal analysis, and scanning electron microscope methods. The diaspore and kaolinite phases in the silicon-rich diasporic bauxite could react with ammonium sulfate to form corresponding sulfates  $(\text{NH}_4)_3\text{Al}(\text{SO}_4)_3$ ,  $\text{NH}_4\text{Al}(\text{SO}_4)_2$  and  $\text{Al}_2(\text{SO}_4)_3$ . The proposed technology could provide an effective method for the direct and separation of aluminum from silicon-rich diasporic bauxite.

**Key words:** silicon-rich diasporic bauxite; ammonium sulfate; roasting; separation

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## 1 Introduction

The silicon-rich diasporic bauxite is the main type of bauxite in China, more than 80% of which is of medium and low grade, with a high content of

aluminum and silicon [1, 2]. While it has low alumina-silica ratio and the aluminum and silicon present an occurrence of dissemination. The fine-grained dissemination of aluminum and silicon creates difficulties during the beneficiating and washing to the silicon-rich diasporic bauxite [1–4].

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The traditional Bayer method requires harsh conditions such as high temperature and concentrated alkali to process the silicon-rich diasporic bauxite, leading to a low recovery of alumina and high costs.

Many attempts have been tried in the alumina industry in China, such as combined method, sintering method, lime Bayer method, and mineral processing Bayer method [5, 6]. At present, the combined method and sintering method are mainly used to treat silicon-rich diasporic bauxite. Compared with single Bayer process, these two processes have achieved better economic benefits. However, these are still some problems, such as long production process, high energy consumption, and high production cost, which impedes the promotion and utilization of these methods [8, 9]. Thus, developing a new process to treat silicon-rich diasporic bauxite resources is of great significance for the rational utilization of bauxite resources.

Low-temperature ammonium sulfate roasting method is a potential new technology for separating valuable elements from minerals with relatively lower energy consumption, less pollution, and high sulfation of target metals [10]. It has been widely used in the treatment of high-sulfur bauxite [11], coal fly ash [12, 13], low-grade kaolin [14], laterite nickel ore [10], and other minerals [15]. It is found that during the roasting process of mineral and ammonium sulfate, the aluminum in the mineral can react with ammonium sulfate at the temperature range of 300–500 °C to form soluble salt, while silicon remains insoluble. In this way, aluminum and silicon can be separated in the following leaching and filtration process efficiently [11–14].

In this work, the ammonium sulfate roasting-water leaching process was used to separate aluminum from the silicon-rich diasporic bauxite. The reaction system of silicon-rich diasporic bauxite and ammonium sulfate is novel, and no relevant report is available until now. By this method, the efficient separation of aluminum and silicon can be achieved at a lower temperature. The tail gas generated during the roasting process was absorbed with water to regenerate ammonium sulfate. After evaporation and crystallization, the ammonium sulfate can be reused in the roasting process to achieve a closed production process. Therefore, it is a clean and efficient process with a good application

prospect. The process could provide a new idea and research basis for the utilization of high silicon diasporic bauxite.

The effects of roasting temperature, roasting time, the dosage of ammonium sulfate, and the particle size of silicon-rich diasporic bauxite on the extraction of aluminum were investigated. The mechanism of roasting and transformation rules of the mineral phase was determined using X-ray diffraction, thermogravimetric-differential thermal analysis, and scanning electron microscope method.

## 2 Experimental

### 2.1 Materials

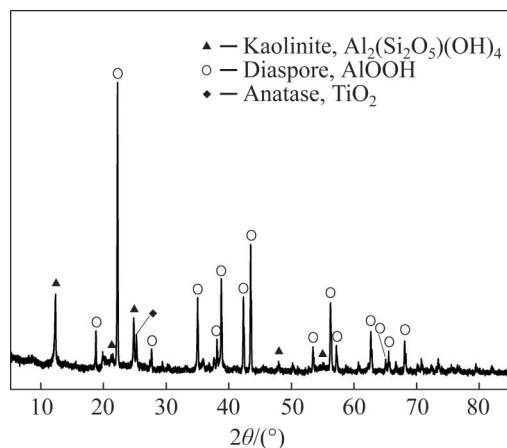
The silicon-rich diasporic bauxite used in this study was sourced from Shanxi province, China. According to the common particle size of industrial bauxite [6, 10–16], the ore samples were crushed, ground, and sieved. The chemical compositions and particle sizes are shown in Table 1. The particle size of the ore in the experiment is within the range of 84–96 μm. Table 1 shows that the Al/Si ratio of the bauxite is low, ranging from 4.9 to 5.0.

**Table 1** Main chemical compositions of ore powders at different particle sizes (wt%)

Particle size/μm	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	Other
140–173	63.90	12.85	2.91	3.76	0.68	14.15
117–140	63.92	12.86	2.90	3.82	0.67	14.20
107–117	63.85	12.91	2.80	3.90	0.62	14.16
96–107	63.89	12.88	2.87	3.86	0.70	14.18
84–96	63.93	12.90	2.89	3.78	0.69	14.20
74–84	63.87	12.81	2.95	3.88	0.71	14.10

The phases of the mineral were determined by XRD (Rint-2000, Rigaku). The results are shown in Figure 1. The main phases in this silicon-rich diasporic bauxite include kaolinite (Al<sub>2</sub>(Si<sub>2</sub>O<sub>5</sub>)(OH)<sub>4</sub>), diasporic (AlOOH) and anatase (TiO<sub>2</sub>).

Figure 2(a) shows the SEM images of silicon-rich diasporic bauxite (ZEISS Sigma 500 Scanning Electron Microscope). As seen in Figure 2(a), the particle of the silicon-rich diasporic bauxite presents regular, fine granular, plate, and layered structure. Figures 2(b)–(f) show the EDS mapping of the mineral (Al, Si, O, Ti and Fe elements). Figures 2(b)–(d) show that diasporic and kaolinite

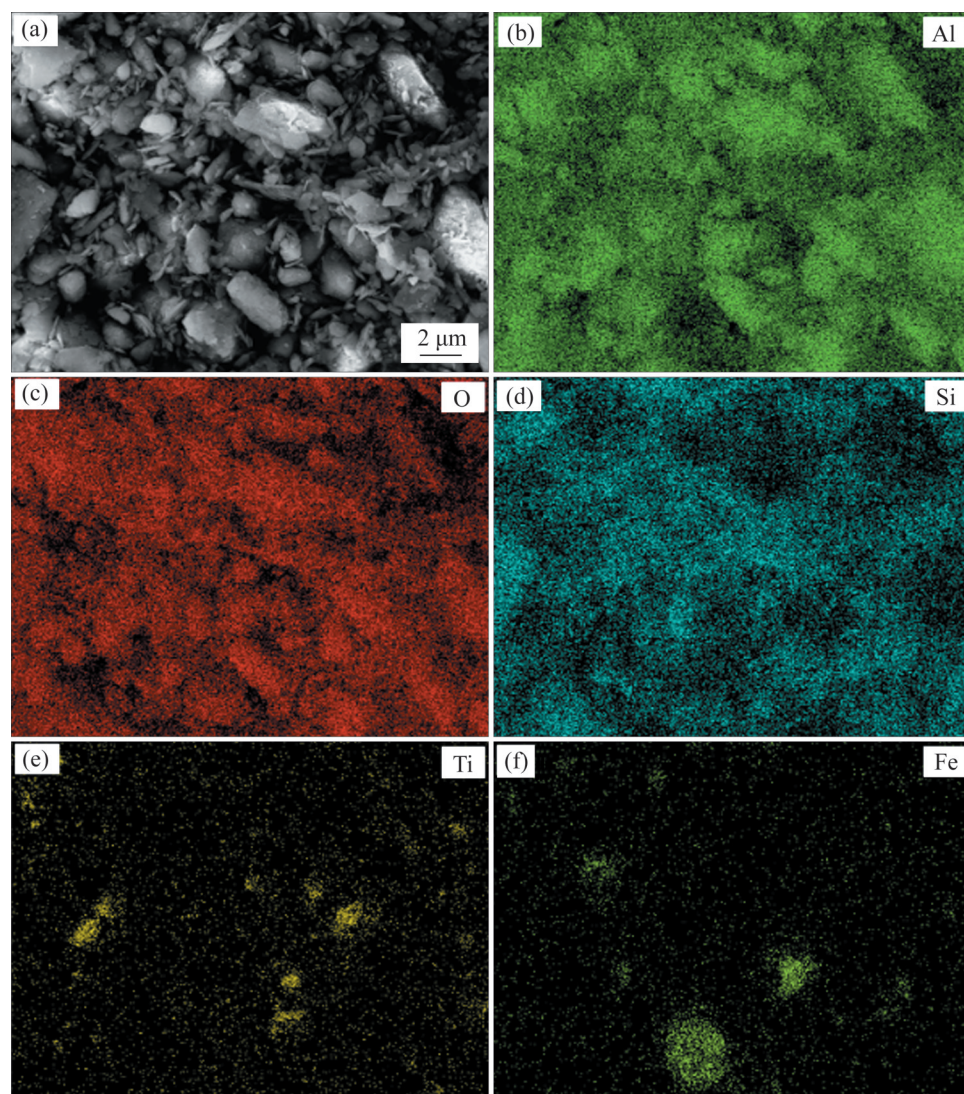


**Figure 1** XRD pattern of silicon-rich diasporic bauxite

are highly disseminated. Combined with the XRD results, we could speculate from Figure 2(e) that Ti is independent as anatase in the silicon-rich diasporic bauxite.

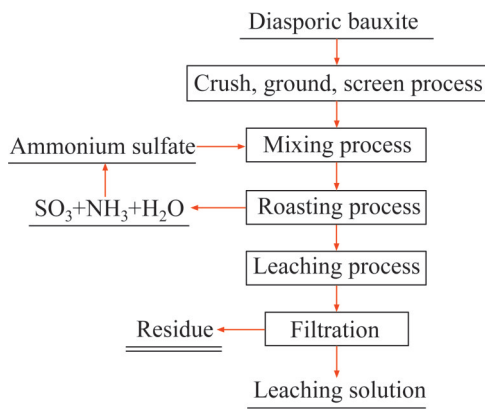
## 2.2 Methods and procedure

10 g of silicon-rich diasporic bauxite and ammonium sulfate were mixed according to the set proportion. And a certain amount of the mixture was then placed in a corundum crucible and roasted in a vertical furnace. The tail gas produced during the roasting process was absorbed by an absorption equipment. The heating rate of the vertical furnace was kept at 10 °C/min, after reaching the set temperature, holding for the desired time after reaching the needing temperature, then drop the temperature to room temperature in air. And the crucible was then taken out from the vertical furnace, and the roasted sample was put into the water according to the solid-liquid ratio of 1:5 g/mL. The mixture was stirred at 75 °C with a stirring rate of 400 r/min. During the process, the soluble aluminum salt enters into water. While silica is insoluble in water. By filtration, aluminum could



**Figure 2** SEM images of silicon-rich diasporic bauxite (a) and scanning maps (b)–(f)

be separated from silicon in the mineral. The flow sheet is shown in Figure 3.



**Figure 3** Flow sheet of roasting and water leaching process

### 2.3 Characterization

The phase compositions of the solid samples were determined by X-ray diffraction (XRD, Rigaku-Smartlab, Cu/K $\alpha$ , Japan). The micromorphology and surface composition of samples were obtained using scanning electron microscopy (SEM, Zeiss-Sigma 300, German). The thermogravimetric analysis (TG, Setsy Evolution, France) was conducted at a heating rate of 10 °C/min to characterize the decomposition behavior of silicon-rich diasporic bauxite. The contents of alumina in the solution were tested by EDTA titration. The alumina extraction efficiency was calculated using Eq. (1).

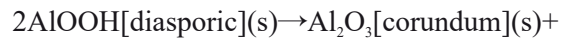
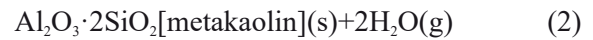
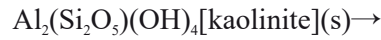
$$x_i = \frac{c_i M_i V_i}{w_i m} \times 100\% \quad (1)$$

where  $x_i$  (%) is the extraction of alumina element,  $c_i$  (mol/L) is the concentration of alumina ion in the leaching solution,  $M_i$  (g/mol) is the molar mass of alumina,  $V_i$  (L) is the volume of leaching solution,  $w_i$  (wt%) is the content of alumina in silicon-rich diasporic bauxite, and  $m$  (g) is the mass of the ore powder.

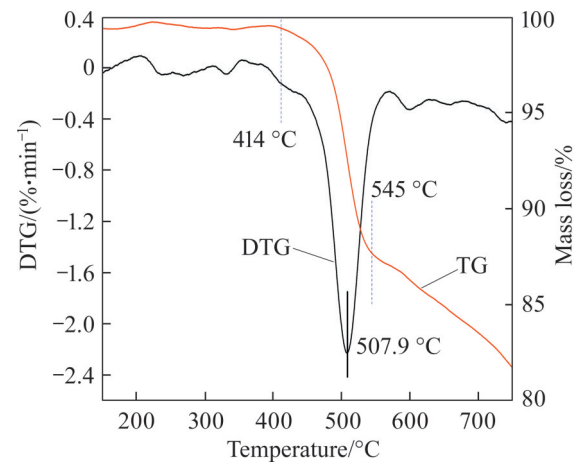
### 3 Thermal decomposition behavior of silicon-rich diasporic bauxite

The main phases of aluminum in the silicon-rich diasporic bauxite are kaolinite and diaspore. Both phases decomposed below 750 °C and the phase transformation routes were reported previously. The reactions occur as shown in Eqs. (2)

and (3) [17–19].



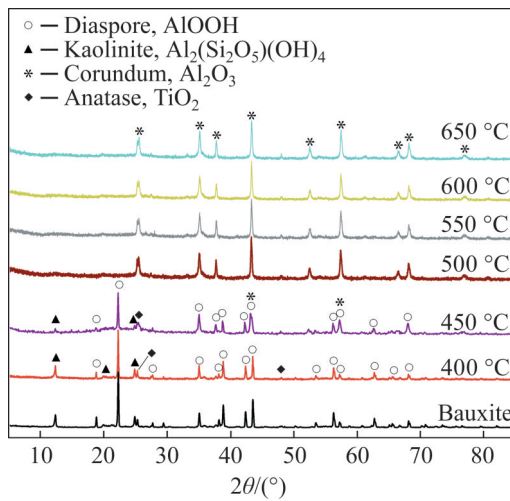
The thermogravimetric analysis of silicon-rich diasporic bauxite was used to determine the roasting temperature of mixed silicon-rich diasporic bauxite and ammonium sulfate. The thermal gravity (TG) analysis was tested from 100 to 750 °C at a heating rate of 10 °C/min, and the results are presented in Figure 4.



**Figure 4** TG curve of silicon-rich diasporic bauxite

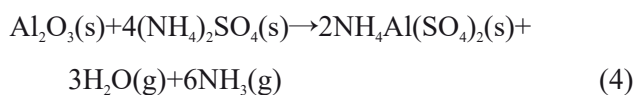
In the temperature range of 414–545 °C, the weight loss of the sample is 11.56%, indicating that silicon-rich diasporic bauxite decomposed in this temperature range. To determine the phase transformation of the sample in this temperature range, the sample was calcined at every 50 °C from 400 to 600 °C for 5 h and the calcined materials were analyzed by XRD. The results are shown in Figure 5, it is known that the decomposition of diaspore and kaolinite occurs in the range of 450–500 °C. At 500 °C, the kaolinite phase and diaspore phase disappear, and the corundum phase appears in the materials. It could be concluded that, in the process of roasting, kaolinite decomposed into amorphous metakaolinite, and diaspore decomposed into corundum [20, 21].

As the diaspore phase is more active than the corundum phase [21], and the decomposition temperature of ammonium sulfate is between 260 and 500 °C [10, 22], the roasting temperature in this study is controlled below 500 °C.



**Figure 5** XRD patterns of silicon-rich diasporic bauxite calcined at different temperature

In the experiment, the dosage of ammonium sulfate is calculated according to Eq. (4). Alumina in the silicon-rich diasporic bauxite including diaspore and kaolinite reacts with ammonium sulfate to form ammonium aluminum sulfate [13].

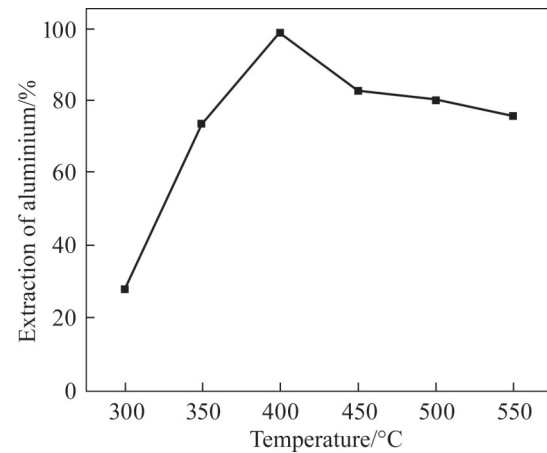


## 4 Results and discussion

### 4.1 Effect of roasting temperature

The effect of roasting temperature on the extraction of aluminum was investigated under the condition that the roasting time of 5 h, the dosage of ammonium sulfate of 2.5 times of the theoretical value calculated according to Eq. (4), and the mineral particle size of 84–96 μm. The roasting temperature was set in the range of 300–550 °C according to the thermal properties of ammonium sulfate and silicon-rich diasporic bauxite.

As shown in Figure 6, the roasting temperature has a great influence on the extraction of aluminum. The extraction of aluminum increases gradually when the temperature increases from 300 to 400 °C. The reason is that the activity of the ammonium sulfate increases with the elevating of temperature [13, 23]. When the temperature is higher than 400 °C, the extraction of aluminum slightly decreases with the enhanced roasting temperature due to the rapid decomposition of ammonium sulfate [10, 24].



**Figure 6** Effect of roasting temperature on aluminum extraction

X-ray diffraction analysis was carried out to analyze the leaching residue obtained from 300 to 550 °C and the results are shown in Figure 7. It can be seen that both kaolinite phase and diaspore phase in silicon-rich diasporic bauxite disappeared at 400 °C. Combined with Figure 5, it can be concluded that both the kaolinite phase and diaspore phase can react with ammonium sulfate completely under these conditions. At 350 °C, the kaolinite phase and diaspore phase still exist in leaching residue, indicating that the reaction is not completed. At 450 °C, the kaolinite phase and diaspore phase still exist in silicon-rich diasporic bauxite, manifesting that part of the kaolinite or diaspore participates the sulfation reactions, but the reaction is not complete, causing by the rapid decomposition of ammonium sulfate at 450 °C [15, 23, 25]. Additionally, the diaspore phase decomposes before reacting with ammonium sulfate. There are both diaspore phase and corundum phase in the residue at 450 °C, indicating that the diaspore phase begins to decompose without reacting with ammonium sulfate completely at 450 °C and the diaspore phase decomposes slowly at this temperature and does not completely transform into corundum. At 500 and 550 °C, the corundum phase in the leaching residue is mainly resulted from the decomposition of the diaspore phase [11].

The roasted samples obtained at different temperatures were analyzed by X-ray diffraction, and the results are shown in Figure 8. As shown, the silicon-rich diasporic bauxite can react with the

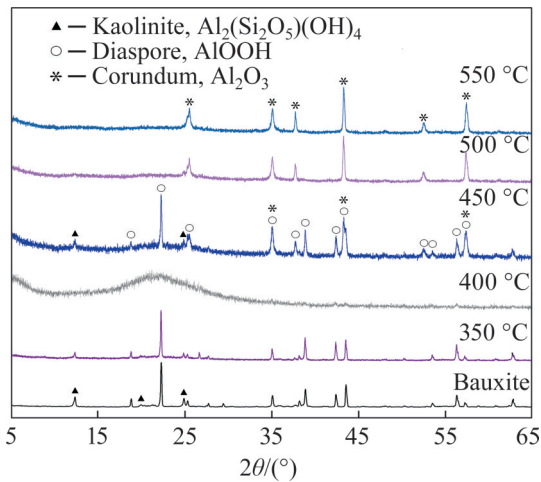


Figure 7 XRD patterns of residue at different temperature

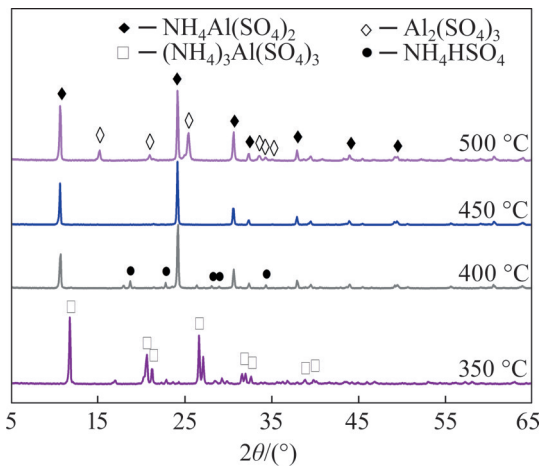
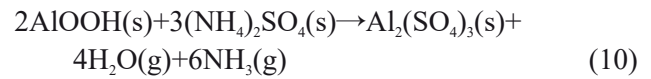
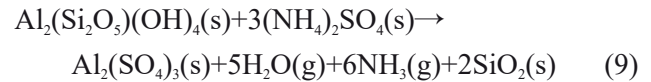
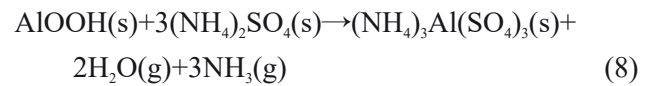
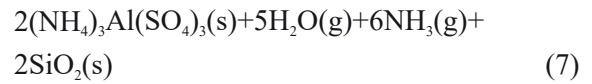
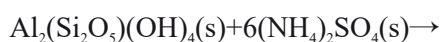
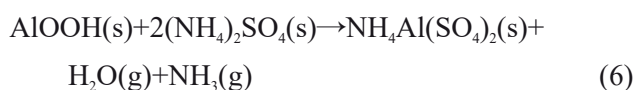
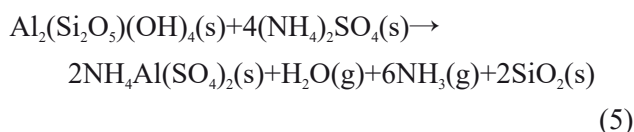


Figure 8 XRD patterns of roasted sample at different temperature

ammonium sulfate system to form soluble ammonium sulfate salt between 350 and 500 °C; at 350 °C, the soluble ammonium sulfate salt is  $(\text{NH}_4)_3\text{Al}(\text{SO}_4)_3$ . At 400 and 450 °C, the soluble ammonium sulfate salt is  $\text{NH}_4\text{Al}(\text{SO}_4)_2$ ; at 500 °C, the soluble ammonium sulfate salts are  $\text{NH}_4\text{Al}(\text{SO}_4)_2$  and  $\text{Al}_2(\text{SO}_4)_3$ . Hence, the main reactions of the kaolinite phase and diaspore phase with ammonium sulfate during the roasting process are shown in Eqs. (4)–(10):



#### 4.2 Effect of ammonium sulfate dosage

Under the condition of roasting time of 5 h, the roasting temperature of 400 °C, the mineral particle size of 84–96 μm, the effect of ammonium sulfate dosage on aluminum extraction was investigated. The ammonium sulfate dosage was set 0.75, 1.0, 1.5, 2.0, 2.5, 3.0 times the theoretical value calculated according to Eq. (4). It can be seen from Figure 9 that the ammonium sulfate dosage has a great influence on the extraction of aluminum, the extraction of aluminum increases obviously with the enhanced dosage of the ammonium sulfate from 0.25: 1 to 1.5: 1, while it increases slowly after the dosage exceeds 2.5: 1, obtaining a maximum extraction of 98.86%.

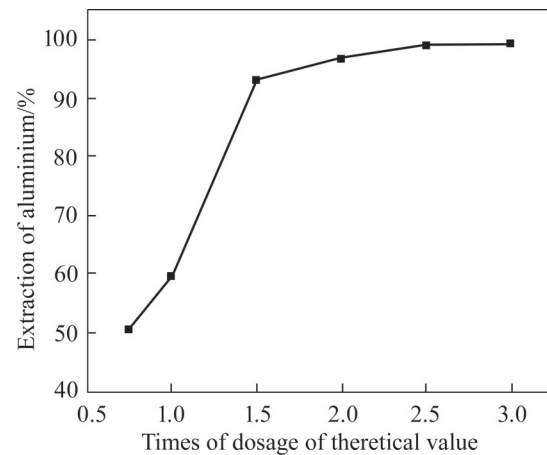
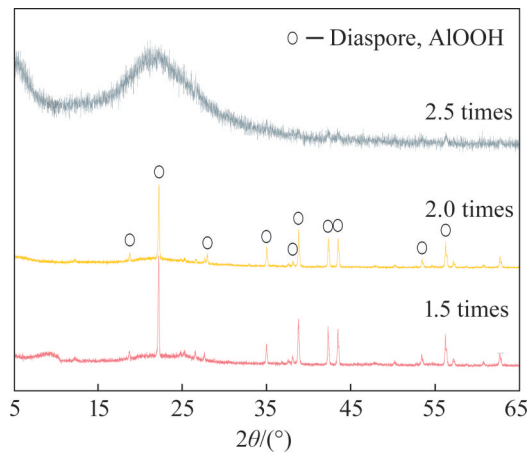


Figure 9 Effect of ammonium sulfate dosage on aluminum extraction

The leaching residues at different ammonium sulfate dosages were analyzed by XRD, and the results are shown in Figure 10. As seen, when the ammonium sulfate dosage is 1.5 and 2.0 times the theoretical value, the kaolinite phase in the silicon-rich diasporic bauxite disappears while the diaspore phase still exists, resulting from the part of the excess of ammonium sulfate decomposed into liquid



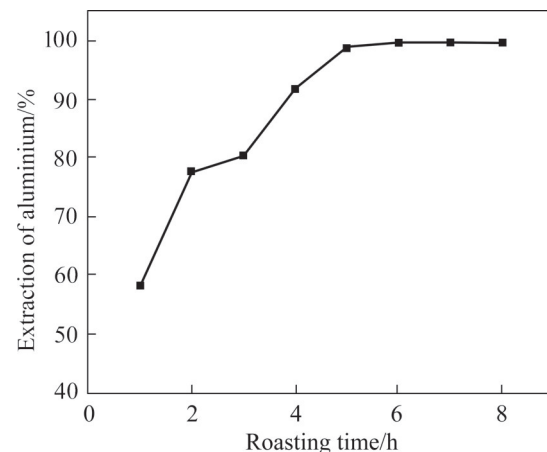
**Figure 10** XRD patterns of residue with different ammonium sulfate at 400 °C

ammonium hydrogen sulfate which can be decomposed into gases ( $\text{NH}_3$ ,  $\text{SO}_3$ ,  $\text{H}_2\text{O}$ ) at 400 °C [10, 26]. This leads to the decrease of the utilization ratio of ammonium sulfate [12, 27]. When the dosage of ammonium sulfate is 2.5 times of the theoretical value, the kaolinite and diaspore phases disappear, and the diffraction peaks of amorphous silica appears in the reaction leaching residue between 20° and 25°, indicating that the kaolinite and diaspore phase react completely with ammonium sulfate under these conditions. The formation of amorphous silica results from the andante crystal growth at low temperatures.

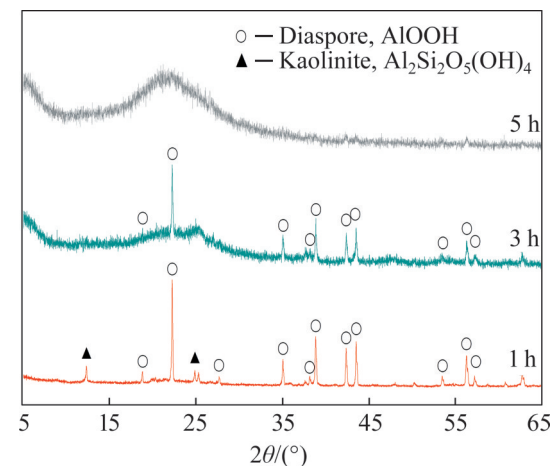
#### 4.3 Effect of roasting time

The effect of roasting time on the extraction of aluminum from silicon-rich diasporic bauxite was investigated under the condition of roasting temperature of 400 °C, ammonium sulfate dosage of 2.5 times of the theoretical value. The results are shown in Figure 11. As shown, the extraction of aluminum increases dramatically with the increase of roasting time. And the extraction of aluminum is almost constant when the roasting time is more than 5 h, suggesting that the roasting time of 5 h is sufficient.

The leaching residues obtained at different roasting time were tested by XRD. As shown in Figure 12, the kaolinite phase disappears and the diaspore does not completely react when the roasting time is 3 h. Only the amorphous silica phase exists in the residue when the roasting time is 5 h. This result is consistent with that of Figure 11.



**Figure 11** Effect of roasting time on aluminum extraction



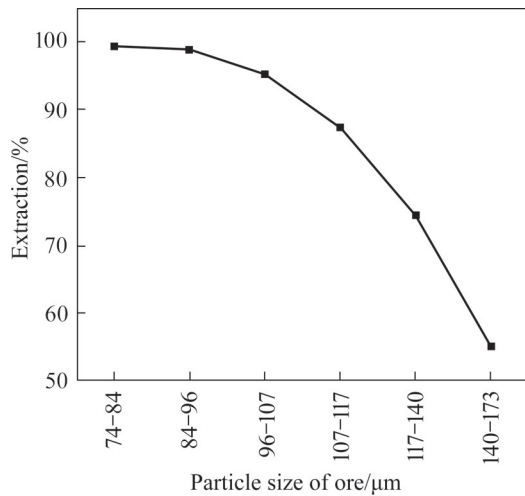
**Figure 12** XRD patterns of residue at different roasting time

#### 4.4 Effect of mineral particle size

The effect of mineral particle size on the extraction of aluminum was investigated at the roasting temperature of 400 °C, ammonium sulfate dosage of 2.5 times of the theoretical value and the roasting time of 5 h, the results are shown in Figure 13. The extraction of aluminum increases sharply with the decrease of mineral particle size. And the extraction of aluminum of silicon-rich diasporic bauxite can reach 98.86% when the mineral particle size is 84–96 μm. When the particle size of the mineral is smaller, the interaction between the mineral and ammonium sulfate will increase and the reaction probability will be increased [10, 23].

#### 4.5 Analysis of leaching residue

The roasted sample at the roasting temperature of 400 °C, ammonium sulfate dosage of 2.5 times of



**Figure 13** Effect of mineral particle size on aluminum extraction

the theoretical value, and the roasting time of 5 h was leached with water and filtered. The leaching residue was dried at 100 °C and the chemical composition of the dried residue was tested. The results are shown in Table 2. The aluminum content in leaching residue is very low, while the main composition of the residue is silicon dioxide, which is up to 95.94%.

**Table 2** Main chemical compositions of ore powders at different particle sizes (wt%)

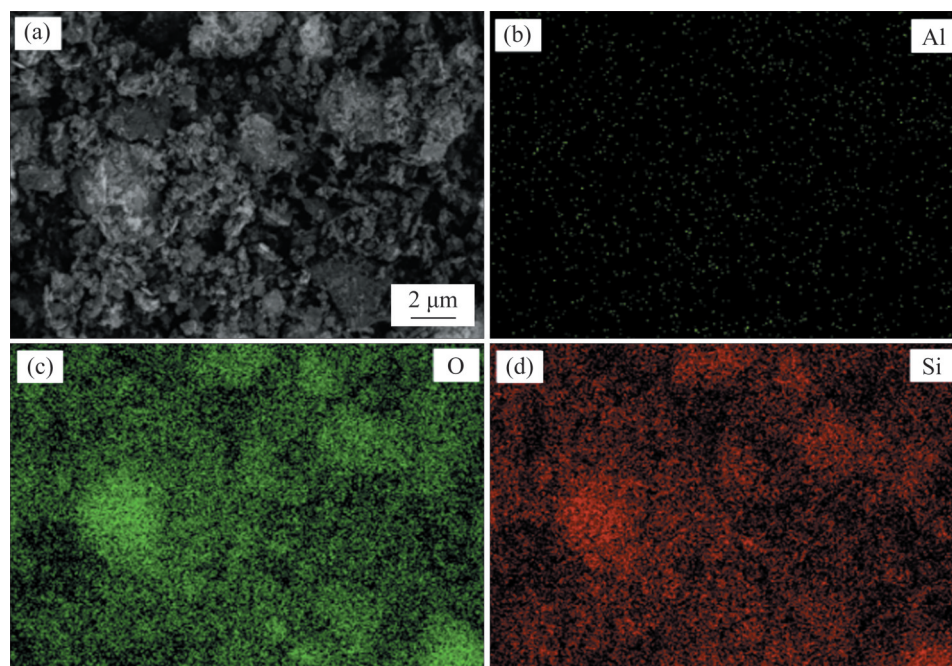
SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	Other
95.94	0.88	0.70	0.30	0.31	1.97

The SEM analysis of the leaching residue is shown in Figure 14. It can be seen that the particle size of the residue becomes smaller than the silicon-rich diasporic bauxite. And the layered structure in the silicon-rich diasporic bauxite disappears. Figures 14(b)–(d) show that the silicon and oxygen in the leaching residue are enriched and other components disappear, which is consistent with the chemical composition results in Table 2.

### 5 Conclusions

1) The ammonium sulfate roasting-water leaching process was used to process the silicon-rich diasporic bauxite. Alumina could be successfully separated from the silicon by roasting the silicon-rich diasporic bauxite with ammonium sulfate. When the silicon-rich diasporic bauxite was roasted under the following conditions: roasting temperature of 400 °C, roasting time of 5 h, ammonium sulfate dosage of 2.5 times of the theoretical value and ore particle size of 80–96  $\mu\text{m}$ , more than 98% aluminum could be extracted.

2) The phase transformation behavior of the kaolinite phase and diasporic phase in silicon-rich diasporic bauxite during the roasting process was thoroughly investigated. Both the diasporic phase and kaolinite phase in silicon-rich diasporic bauxite can react with ammonium sulfate. At 350 °C, the ammonium sulfate salt is  $(\text{NH}_4)_3\text{Al}(\text{SO}_4)_3$ ; at 400



**Figure 14** SEM images of residue (a) and Al, O, Si element mapping (b)–(d)



and 450 °C, the ammonium sulfate salt is mainly  $\text{NH}_4\text{Al}(\text{SO}_4)_2$ ; at 500 °C, the ammonium sulfate salts are  $\text{NH}_4\text{Al}(\text{SO}_4)_2$  and  $\text{Al}_2(\text{SO}_4)_3$ .

3) The leaching residue mainly consists of amorphous silica with a purity of 96.43%.

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## 中文导读

### 富硅一水硬铝石型铝土矿与硫酸铵焙烧反应行为机理

**摘要：**一水硬铝石型铝土矿中铝硅的分离在氧化铝生产中具有重要意义。硫酸铵低温焙烧技术能有效提取矿物中的金属元素，本文以硫酸铵为提取剂，将一水硬铝石型铝土矿与硫酸铵共同焙烧提取铝。实验考察了焙烧过程中铝提取的主要影响因素：焙烧温度，硫酸铵用量，焙烧时间和矿物粒度。结果表明，在400 °C下保温2 h、硫酸铵用量为理论值的2.5倍、矿物粒度在80~96 μm的条件下，铝的提取率可达到98%以上。通过X射线衍射、热分析，扫描电镜等研究焙烧过程中不同物相铝的反应行为和反应机理，发现在不同条件下，一水硬铝石和高岭石均可以与硫酸铵反应生成相应的硫酸盐： $(\text{NH}_4)_3\text{Al}(\text{SO}_4)_3$ 、 $\text{NH}_4\text{Al}(\text{SO}_4)_2$ 和 $\text{Al}_2(\text{SO}_4)_3$ 。硫酸铵与一水硬铝石型铝土矿混合焙烧—水溶出技术为铝土矿中铝硅直接分离提供了一种有效的方法。

**关键词：**铝土矿；硫酸铵；焙烧；分离