EXTRACTION OF ALUMINA FROM COAL-DERIVED PYRITE FLOTATION TAILING BY PRE-DESILICATION–BAYER PROCESS

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Abstract

A large amount of tailing from the flotation processing of pyrite ore are accumulated and landfilled, resulting in serious environmental pollution and a waste of valuable elements such as Al, Ti, Si, etc. Aiming to extract alumina from coal-derived pyrite flotation tailing, the process has been proposed based on activation roasting-alkali leaching for desilication, followed by alumina extraction via Bayer process. Proper parameters of the desilication system and Bayer process were investigated in this work. Under the optimized conditions of desilication in the pilot scale experiment, an alumina concentrate with 69.49% Al₂O₃ and alumina to silica mass ratio (A/S) of 5.93 was obtained from a pyrite flotation tailing bearing 46.22% Al₂O₃ and 28.33% SiO₂, and the corresponding desilication ratio was 73.06%. Then, under the appropriate conditions of Bayer process, the digestion of alumina was 78.42%. The results provide a new way for utilization of pyrite tailings.

Introduction

In China, a vast majority of sulfuric acid production is derived from pyrite concentrate as the raw material, which generates vast tailing accumulation from the processing of pyrite ore, and usually creating serious land and water pollution problems, due to the release of acids and toxic substances [1, 2, 3]. Meanwhile, the pyrite tailings contain a significant amount of valuable elements such as Al, Ti, Si, RE, etc. but without effective treatment measures for recycling. Thus, research on efficient utilization of pyrite tailings resources are of great theoretical value and practical significance.

In recent years, some laboratorial researches on exploiting pyrite tailing had been reported. Mainly includes: Production the glass-ceramics or heat-insulating sintered porous brick by utilization pyrite tailings as major raw material [4]; Preparation of polymeric aluminum ferric chloride, reclaim sulfur or diaspore concentrate from pyrite tailing [5, 6, 7]; General utilization all of components in the tailings for the preparation of porous-SiO2, aluminum hydrate and iron oxide vellow pigment [8]. Furthermore, previous studies indicate that, an integrated thermochemical process has been developed for the combined utilization of gypsum and pyrite wastes by converting them into useful products including lime, sulfur, and direct reduction iron (DRI) [9, 10, 11].

For the pretreatment of bauxite ores during alumina production, roasting-leaching process is the major of technology to remove silica and improve the A/S ratio because of remarkable advantages of high alumina recovery [12, 13]. Technique of desilication from bauxite ores by roasting-leaching process is: firstly, the aluminosilicate minerals, such as pyrophyllite, kaolinite, illite, etc., are activated and transformed into amorphous SiO₂ by thermal treatment. And then, the amorphous SiO₂ in activated ore is dissolved by alkali leaching [12-16]. Based on the fact that pyrite flotation tailing mainly contains diaspore and aluminosilicates including kaolinite, pyrophyllite etc., the process of activation roasting followed by alkali leaching for desilication was proposed to achieve the comprehensive utilization of

alumina components from pyrite tailing.

In this study, the feasibility and suitable process conditions for preparation of alumina concentrate, as well as the alumina extraction by Bayer process were studied. This research will provide an improving thought for the use of resource of pyrite tailings.

Experimental

Materials

The chemical and mineralogical compositions of the coal-derived pyrite flotation tailing are shown in Table I and Table II, respectively. Mineralogical analysis of the initial sample (XRD, Fig.1) was performed by using a D/Max2500 X-ray diffractometer with scanning angle 2θ from 5 to 70 degree. The sample mainly contains diaspore, pyrophyllite, illite-montmorillonite, kaolinite, etc., and is characterized by high alumina content (44.22%) and high silicon content (28.33%), but low mass ratio of alumina to silica (A/S 1.63). Moreover, the tailing is also associated with 5.02% titanium dioxide. Incidentally, the particle size of the tailing is 80% undersize 74 µm, approximately 40% undersize 38 µm.

Chemical composition	Mass /%
Al ₂ O ₃	46.22
SiO_2	28.33
TiO ₂	5.02
Fe _{total}	4.32
S	2.80
CaO	0.93
LOI*	10.28

Table I. Chemical Composition of Pyrite Tailing

*LOI: loss on ignition

Mineral composition	Content /%
Diaspore	44.53
Illite-montmorillonite	29.27
Kaolinite	8.08
Pyrophyllite	6.65
Anatase	5.04
Pyrite	5.21



Fig.1 XRD pattern of the pyrite tailing

Methods

Activation roasting experiment The tailing was granulated into small pellet with the size of 4~8 mm by the disc pelletizer with a diameter of 1000mm, the thermal activation experiments were carried out in a lab rotary kiln (Φ 300×1000 mm). The rotating speed of the kiln is 2 rpm, and the roasting temperature was adjusted by natural gas burner. Once the given roasting temperature attained, the pellet was loaded into the kiln and treated isothermally at uniform temperatures for a given period. After the roasting time elapsed, the roasted samples were cooled in air, and then ground to 90% undersize 74 µm.

Alkali leaching experiment Alkali leaching tests were conducted in a DY-8 autoclave. The autoclave was equipped with eight 150 mL stainless steel pots that can rotate end to end in a bath of glycerin. It was electrically heated, and the temperature of the glycerin bath was controlled by a thermostat. At the beginning of each trail, the fixed volume of caustic soda (NaOH) solution and fixed weight of activated ores were added to the test pots. The sealed pots were soaked in the bath and agitated at 20 rpm. The temperature of glycerin was maintained at a certain temperature. Filtration was performed immediately after leaching. The contents of SiO₂ and Al₂O₃ in dried alumina concentrate were measured.

Bayer process digestion experiment The digestion experiment was carried out in a GCF rotating stirred

autoclave. A certain quality of alumina concentrate and 150 mL spent liquor with Na₂O_k 250 g•L⁻¹, Al₂O₃ 118 g•L⁻¹ and the caustic molar ratio (α_k) of 3.5 was poured into the autoclave, and without CaO addition. It was treated isothermally at uniform temperatures for a given period, and the stirring speed was 150 rpm. After digestion, the digested slurry was separated into solution and red mud by filtration. The content of Al₂O₃ and SiO₂ in the red mud were determined by chemical analysis. The actual and relative alumina digestion ratios were calculated.



Fig. 2 Experimental flowsheet of utilization of pyrite tailing in principle

Results and Discussion

Desilication by activation roasting followed by alkali-leaching

<u>Effect of roasting temperature</u> The effect of roasting temperature varying from 1050 °C to 1250 °C, on the desilication ratio, Al_2O_3 content and A/S ratio of the alumina concentrate was evaluated, and the results were shown in Fig.3. The alkali leaching conditions were temperature of 140 °C, time of 30 min and NaOH

concentration of 140 g•L⁻¹. The results show that, desilication changes with the roasting temperature significantly, at lower roasting temperature (1050~1150 °C), the desilication ratio, Al_2O_3 content and A/S ratio of the alumina concentrate improves as temperature goes on. While at higher temperature (>1150°C), the desilication ratio, Al_2O_3 content and A/S of the alumina concentrate all decrease along with the temperature. The optimal roasting temperature of 1100~1150 °C is recommended.



Fig. 3 Influence of roasting temperature on desilication of tailing (Roasting time: 15 min)



Fig. 4 Influence of roasting time on desilication of tailing (Roasting temperature: 1150 °C)

Effect of roasting time Fig.4 presents the effect of roasting time varying from 10 min to 25 min on the desilication ratio, Al_2O_3 content and A/S of the alumina concentrate. The alkali leaching conditions were temperature of 140 °C, time of 30 min and NaOH concentration of 140 g•L⁻¹. It showed that, when the roasting time varies in the range of 10 min to 15 min, the desilication ratio, Al_2O_3 content and A/S ratio of the alumina concentrate improves as time goes on. However,

when the roasting time exceeds 15 min, the desilication indexes decline slowly along with the time. So, the optimum roasting time is 15 min.

Effect of NaOH concentration of alkali liquor The effect of NaOH concentration on desilication of activated tailing is shown in Fig. 5. The roasting conditions were temperature of 1150 °C, time of 15 min, and the alkali leaching conditions were temperature of 120 °C, time of 90 min. It can be seen that, with the increase of NaOH concentration, the desilication ratio, Al_2O_3 content and A/S ratio of the alumina concentrate increase initially, whereas too high NaOH concentration shows unfavorable effect on the desilication. The proper NaOH concentration is 140 g•L⁻¹.



Fig. 5 Influence of NaOH concentration on desilication of tailing

Effect of leaching temperature and time The roasting conditions were temperature of 1150 °C, time of 15 min, and NaOH concentration was 140 g•L⁻¹. As can be seen in Fig. 6, it is clearly shown that, at the same leaching temperature, as the leaching time increases all of the desilication ratio, Al_2O_3 content and A/S ratio of the alumina concentrate appear in the trend of firstly increased and then decreased. When the leaching temperature is 120 °C or 130 °C, the time of the desilication ratio attaining the maximum is about 60 min, however, the time merely needs 30 min when the leaching temperature goes up to 140 °C. Therefore, leaching time of 30 min and leaching temperature of 140 °C is recommended under the experimental conditions.



Fig. 6 Influence of leaching temperature and time on desilication of tailing

Based on the above results, as far as the sample of this investigation is concerned, the suitable roasting temperature and roasting time is 1150 °C and 15 min, alkali leaching temperature of 140 °C and time of 30 min and NaOH concentration of 140 g•L⁻¹ is recommended. The main composition of the alumina concentrate is shown in Table III. Under the optimized conditions, the desilication ratio, Al₂O₃ content and A/S ratio of the alumina concentrate were 73.06%, 69.49% and 5.93, respectively. The alumina concentrate can be used as the raw material for Bayer process.

Table III. Chemical Composition of the Alu	imina
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Concentrate		
	Chemical composition	Mass /%
	Al ₂ O ₃	69.49
	SiO ₂	11.71
	TiO ₂	7.15
	Fe ₂ O ₃	9.27
	CaO	1.55

Bayer digestion of the alumina concentrate

Effect of digestion temperature The alumina concentrate is digested at 240~280 °C without lime addition via Bayer process.



Fig. 7 Influence of digestion temperature on digestion of alumina concentrate via Bayer process (Digestion time: 60min)

The results in Fig. 7 show that digestion temperature has a great influence on Al_2O_3 digestion. The higher digestion efficiency can be achieved at above 270°C. At the moment, A/S ratio of red mud was 1.11, and the actual and relative alumina digestion ratios go up to 79.13% and 97.40%, respectively.

Effect of digestion time Fig.8 shows the effect of digestion time on digestion of alumina concentrate. It indicates that the alumina dissolution of concentrate increases as digestion time goes on. When the digestion temperature was 270 °C and digestion time was 40 min, the relative alumina digestion ratio was as high as 96.53%. While the relative alumina digestion ratio increased a little with the enhancing time was 60 min, the Al₂O₃ content and A/S ratio of the red mud were increased. Thus, the digestion time of 40 min is recommended.



Fig. 8 Influence of digestion time on digestion of alumina concentrate via Bayer process (Digestion temperature: 270 °C)

Consequently, the optimal processing parameters of digestion are: digestion temperature and digestion time is 270°C and 40 min. In this case, the actual and relative alumina digestion ratios go up to 78.42% and 96.53% and A/S ratio of red mud was 1.15.

Conclusions

Aiming to extract alumina from coal-derived pyrite flotation tailing, the process of activation roasting-alkali

leaching for desilication, followed by alumina extraction via Bayer process was developed in a pilot-scale test in this work, the following conclusions are obtained in the pilot scale test.

(1) The suitable activation conditions for desilication of pyrite flotation tailing are: roasting temperature of $1100 \sim 1150$ °C and time of 15 min, alkali leaching temperature of 140 °C and time of 30 min, NaOH concentration of 140 g•L⁻¹. An alumina concentrate with 69.49% Al₂O₃ and A/S of 5.93 was prepared from a tailing sample bearing 46.22% Al₂O₃ and 28.33% SiO₂, and the corresponding desilication ratio was 73.06%.

(2) The optimal Bayer process digestion conditions for the extraction of alumina from the alumina concentrate are: the digestion temperature of 270°C, the digestion time of 40min. Under the optimized condition, the actual and relative alumina digestion ratios are 78.42% and 96.53%, respectively.

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