Physicochem. Probl. Miner. Process., 61(4), 2025, 204689

http://www.journalssystem.com/ppmp

ISSN 1643-1049 © Wroclaw University of Science and Technology

# Hot alkali mill pretreatment for flotation desulfurization of highsulfur bauxite

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**Abstract:** Oxidative acidification of high-sulfur bauxite can cause great problems in the production of alumina by the Bayer process. In this study, sodium hydroxide was used as a hot alkali solution to pretreat high-sulfur bauxite by hot alkali milling, which inhibited the effect of oxidative acidification on flotation, and then flotation treatment was carried out, to improve the grade of alumina and reduce the sulfur content of high-sulfur bauxite, and finally meet the production requirements of Bayer alumina. The results showed that under the optimal conditions of sodium hydroxide solution concentration of 40%, temperature of 60°C, and grinding time of 4 minutes, the alumina grade increased from 57.18% to 87.45% and the sulfur grade decreased from 5.46% to 0.69% by pretreated flotation. XRD, SEM, EDS, and AFM were used to analyze the pretreatment mechanism of the hot alkali mill, and it showed that the pretreatment of the hot alkali mill would corrode the surface of diaspore monohydrate and expose more surface sites. The hot alkali solution pretreatment restrained the acidification and oxidation of pyrite. The hot alkali solution pretreatment makes the surface of pyrite rougher and enhances the floatability of pyrite.

Keywords: high-sulfur bauxite, flotation, desulfurization, hot alkali mill pretreatment

# 1. Introduction

Aluminium is the world's second-largest source of metal after steel (Yi et al., 2022), while bauxite is the main industrial feedstock for alumina production (Cheng et al., 2023). About 800 million tons of high-sulfur bauxite (with a sulfur content higher than 0.7%) (Chai et al., 2018) in China has not been effectively utilized. In recent years, with the rapid development of China's alumina industry, the supply of domestic bauxite resources has been tight, forcing us to develop and utilize high-sulfur bauxite. High sulfur content in high-sulfur bauxite can have various adverse effects on the Bayer process, such as increasing the sulfur and iron content in the product, corrosion of accelerated reaction equipment, reducing the particle size of the product, and increasing alkali consumption (Liu and Yan et al., 2020). Therefore, it is necessary to desulfurize the high-sulfur bauxite to meet the requirements of the Bayer production process. The desulfurization treatment of high-sulfur bauxite is significant to the stable and safe supply of bauxite resources in China.

There are many desulfurization methods for high-sulfur bauxite, including pre-roasting (Lou et al., 2016), flotation (Zhu et al., 2023), bioleaching (Han et al., 2023), electrolytic desulfurization (Gong et al., 2015), wet oxidative desulfurization (Long and Dixon, 2004) and precipitator desulfurization (Li et al., 2016). Flotation has the advantages of flexible operation and low cost (Li and Zhang, 2024), so flotation is an economical and effective high-sulfur bauxite desulfurization technology (Zhu et al., 2023). However, during the storage and sorting of high-sulfur bauxite, the sulfides in it will be oxidized to sulfuric acid, resulting in an acidic solution, and the sulfate sludging in the slurry will cause the equipment to clog. Therefore, before flotation, bauxite needs to be pretreated to change the surface properties of mineral particles and improve the floatability of minerals (Xia et al., 2013).

Surface pretreatment methods include grinding pretreatment (Chen et al., 2017), crushing and refining large ore blocks into smaller particles improves ore surface area and dispersion, making it easier for chemical reactions to occur inside and on the surface, which not only optimizes ore sorting efficiency but also reduces energy consumption. Ultrasound pretreatment (Tong et al., 2024), the mechanical effect of ultrasonic waves can be used to act on ore, which can cause collision and crushing between mineral particles, which has the advantages of environmental protection and no heat source, and has many applications in the field of flotation beneficiation. Microwave pretreatment (Su et al., 2011), by improving the physical and chemical properties of the target minerals, this method has attracted attention for its advantages of cleanliness, low contamination, high efficiency, cost-effectiveness, and uniform heating, magnetic pretreatment (Zheng et al., 2018), it is usually applied to ores containing magnetic minerals, which can separate a part of non-magnetic impurities from the ore, thereby reducing the impurity content in the flotation process, reducing the amount of flotation waste and improving the economy of flotation. Electrochemical pretreatment (Liu et al., 2024), the physicochemical properties of the collector to improve the flotation efficiency, its environmental protection characteristics are remarkable, and no secondary pollution will be generated. Pressure pretreatment (Tominaga, 2009), typically used for metal sulfide minerals that are difficult to float, this pretreatment allows the sulfide minerals to be oxidized into oxides or oxide-sulfide mixtures that are easier to float. Thermal pretreatment (Kenzhaliyev et al., 2025), changing the structure, chemical properties and surface properties of ore is a relatively simple method of mineral oxidation, which is usually applied to ores with poor flotation effect at low temperatures, etc.

The hot alkali mill pretreatment in this study is easier to change the surface properties of minerals, the process flow is relatively simple, and the cost is low, and the aluminum concentrate with good desulfurization effect can be obtained by flotation. Hot alkali pretreatment has certain applications in mineral processing, such as microwave heating and alkali lignin reduction combined with pretreatment of manganese ore (Hu et al., 2023) and hot alkali solution pretreatment of low-grade high-sulfur bauxite (Zhou et al., 2019).

In order to improve the effect of flotation desulfurization of high-sulfur bauxite, the surface pretreatment of high-sulfur bauxite was studied by hot lye, and the more suitable experimental conditions of hot alkali pretreatment were determined, and the mechanism was studied. It is hoped that this study will provide a reference for the economical and efficient development and utilization of high-sulfur bauxite.

#### 2. Materials and methods

#### 2.1. Materials

The actual high-sulfur bauxite ore used in the test was purchased from a place in Guizhou. The main chemical composition analysis results of raw ore are shown in Table 1.

Elements	$Al_2O_3$	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaO	MgO	K <sub>2</sub> O	S
Content/%	57.18	6.17	18.11	3.72	1.96	1.23	0.82	5.46

Table 1. Elemental analysis results of experimental high-sulfur bauxites

As can be seen from Table 1, the main chemical components of the test ore samples are  $Al_2O_3$ ,  $SiO_2$ ,  $Fe_2O_3$ ,  $TiO_2$ , and S, of which the content of  $Al_2O_3$  is 57.18%, the content of A/S is 9.27, the S content is 5.46%, and the sulfur content is greater than 0.7%, which belongs to high-sulfur bauxite.

In order to preliminarily determine the main mineral species in the ore, the qualitative analysis of the mineral sample phase by X-ray diffraction analyzer is carried out, and the analysis results are **s**hown in Fig. 1.

As can be seen from Fig. 1, diaspore monohydrate is the main useful mineral, and gangue minerals include silica-bearing minerals (muscovite) and sulfur-bearing minerals (pyrite). To find out the particle size composition of the raw ore and the distribution of sulfur in the high-sulfur bauxite particles of

different grain sizes, the raw ore was screened and analyzed, and the contents of Al<sub>2</sub>O<sub>3</sub> and S in each particle size interval after screening were determined. The test results are shown in Table 2.

As can be seen from Table 2, the proportion of particle size greater than 0.074 mm is 79.03%, showing a high S grade, indicating that sulfur-bearing minerals mainly exist in larger particle sizes. The particle sizes greater than 0.315 mm and less than 0.045 mm accounted for 64.83% and 18.25%, respectively, and the  $Al_2O_3$  content of these two particle sizes was higher. According to the distribution of S grade and  $Al_2O_3$  content in each particle size, sulfur-bearing minerals are mainly enriched in flotation, proper grinding is required.



Fig. 1. XRD pattern of test ore

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Particle size/mm	Yield/%	S grade/%	Al <sub>2</sub> O <sub>3</sub> Content/%
+1	33.95	7.26	68.14
$0.315 \sim 1$	30.88	7.32	56.65
$0.15 \sim 0.315$	8.68	8.01	50.76
$0.074 \sim 0.15$	5.52	8.09	50.93
$0.045 \sim 0.074$	2.72	6.65	51.89
- 0.045	18.25	0.55	59.76
Total	100.00	5.62	57.56

# 2.2. Methods

#### 2.2.1. Oxidative acidification test of mineral samples

The pH value of the natural slurry was determined after the test ore sample was ground to  $77\pm7\% < 0.074$  mm and the slurry density was controlled at 50%. The SO<sub>4</sub><sup>2-</sup> concentration was determined by ion chromatography. The results are shown in Table 3.

Table 3. The natural pH and SO<sub>4</sub><sup>2-</sup> concentration of high sulfur bauxite slurry were tested

sample	pH value of natural slurry	SO <sub>4</sub> <sup>2-</sup> (mg/L)
Test ore samples	6.02	266.19

Table 3 shows that the bauxite has undergone a certain degree of acidification during natural storage, the pH of the slurry is 6.02, the  $SO_4^{2-}$  concentration is 266.19 mg/L, and the oxidative acidification of the high-sulfur bauxite is mainly caused by pyrite oxidation, resulting in the formation of Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> in the slurry, and the direct flotation will affect the desulfurization efficiency, so surface pretreatment is required.

#### 2.2.2. Hot alkali mill pretreatment experiment

150 g of high-sulfur bauxite was put into a conical ball mill with a grinding concentration of 50%, wet grinding for 4 min, and sodium hydroxide solution with a concentration of 50% and a temperature of 60 °C was added to prepare the slurry for flotation.

#### 2.2.3. Flotation test

The test process is shown in Fig. 2. In the test, a 1.5 L XFD single-tank flotation machine was used to perform reverse flotation desulfurization of the test ore. The flotation test ore weighed 150 g and the grinding fineness (-0.075 mm) was set at 50%. In the desulfurization test, the solid-liquid ratio of the slurry was 10 : 1, the pre-mixing time of the slurry was 2 min, the pH value of the slurry was 7.5, and the copper sulfate was 80 g/Mg. Modified starch 100 g/Mg, PXS-CTAB (isopropyl xanthate-cetyltrimethylammonium bromide) 240 g/Mg, Pineol oil (2 # oil) 100 g/Mg, scraping time 6 min. At the end of flotation, the resulting concentrate and tailings are filtered, dried, weighed, and analyzed. Considering that some of the Al<sub>2</sub>O<sub>3</sub> may float up and be removed with the foam during the flotation process, the S% and Al<sub>2</sub>O<sub>3</sub> recoveries in the concentrate are used as the key indicators to evaluate the flotation effect. The sulfur content was determined by the CLS microcomputer coulometric sulfur meter, and the alumina content was determined by EDTA complexation titration.



Fig. 2. Flotation process flow of test ore

#### 2.2.4. Characterization method

X-ray diffraction (XRD) of the sample was run on the D8 Advance (Bruker, DE) with Cu K $\alpha$  radiation. The samples were scanned at a scan rate of 2 min<sup>-1</sup> in a 2 $\theta$  range of 5° ~ 90°.

Scanning electron microscopy (SEM) was used to observe the microscopic morphological changes of pyrite surface before and after pretreatment, and the distribution of pyrite surface elements in the selected area was studied by using a randomly equipped energy dispersive X-ray spectroscopy (EDX) analyzer. In addition, the atomic concentrations of Al, Si, S, Fe, and O elements on the pyrite surface were determined. Test conditions: acceleration voltage 15 kV, pure platinum sprayed material.

Atomic force microscopy (AFM) is a technique used to analyze the microscopic morphology of a surface by detecting changes in the interaction force between the probe and the sample surface and with the help of the forces between atoms, high-precision imaging of the sample surface. Before the measurement, a 1 cm1 cm pyrite sample was first ground using a semi-automatic polishing machine, followed by 500 mesh, 600 mesh, 1000 mesh, 2000 mesh, and 3000 mesh sandpaper from coarse to fine. After that, the samples are treated in a hot alkaline solution for 10 minutes according to the conditions

of the "hot alkali mill", then thoroughly rinsed and dried in an oven, and finally stored in a bag for later use. The samples to be measured were placed in the AFM sample stage for scanning, and the scanned images were analyzed, including surface morphology analysis, particle size measurement, surface roughness analysis, etc., to observe the surface morphology of pyrite before and after surface pretreatment.

#### 2.3. Reagents

In the experiment, 10% NaOH solution (Analytical pure) was used as alkali solution and pH adjuster, CuSO<sub>4</sub> solution (Analytical pure) was used as activator, modified starch was used as inhibitor (Analytical pure), PXS-CTAB (isopropyl xanthate-cetyltrimethylammonium bromide, Analytical pure) was used as the collector, and pineal oil (Industrial pure) was used as foaming agent.

# 3. Results and discussion

# 3.1. Effect of hot alkaline solution mass concentration on the flotation results

The effect of the mass concentration of hot aqueous alkali on the flotation index was studied using a 60 °C temperature of hot alkali. The results are shown in Fig. 3.

As can be seen from Fig. 3, when the hot alkali concentration increases from 10% to 40%, the recovery rate of alumina increases from 85.4% to 88.47%, and the grade increases from 55.33% to 58.76%. The results showed that the recovery rate of alumina increased with the increase of hot alkali solution in this range. When the concentration of hot alkali continues to increase, the recovery rate of alumina shows a decreasing trend, indicating that the sulfide ore may react under the action of high hot alkali solution to form hydrogen sulfide gas and other substances. These gases can adhere to the surface of the mineral, forming bubbles and affecting the flotation performance of the mineral. The sulfur grade in the concentrate under different hot alkali concentrations is between 0.68% ~ 0.73%, and the change range is not large, so the hot alkali concentration of 40% at the point with the highest recovery rate of alumina is used as the concentration of hot alkali mill pretreatment solution.



Fig. 3. Effect of surface pretreatment concentration on flotation test

#### 3.2. Effect of hot alkaline solution temperature on the flotation results

The effect of the temperature of hot aqueous alkali on the flotation index was studied using a 40% mass concentration. The results are shown in Fig. 4.

As can be seen from Fig. 4, the recovery rate of alumina is almost unchanged from 25 °C to 40 °C, and the recovery rate of alumina increases from 73.51% to 79.59% when the temperature increases to 60°C and decreases significantly with the increase of temperature when the temperature exceeds 60 °C. From 25 °C to 70 °C, the grade of sulfur in the concentrate decreases first and then increases, and the

lowest is 0.71% at 60 °C, indicating that the temperature is too high or too low is not conducive to the rise of pyrite, so 60 °C is used as the temperature of the hot alkali mill pretreatment solution.



Fig. 4. Effect of surface pretreatment temperature on flotation tests

# 3.3. Effect of hot alkaline solution grinding time on the flotation results

The effect of the grinding time of hot aqueous alkali on the flotation index was studied using a 40% mass concentration and 60 °C temperature of hot alkali. The results are shown in Fig. 5.

In this study, the proportion of mineral particle size less than 0.074 mm was selected as the criterion for evaluating grinding fineness. The duration of the grinding test was set to 2, 3, 4, 5 and 7 minutes, and the test results are shown in Figure 5(a), after 4 minutes of grinding, the proportion of mineral particles less than 0.074 mm reached 76.96%. When the grinding time increased to 5 minutes, the proportion increased to 86.12%; When the grinding time is further extended to 7 minutes, the proportion of mineral particles smaller than 0.074 mm increases to 92.96%, although the proportion of fine particles continues to increase, and the growth rate slows down compared to before. According to the results of Fig. 5(b), when the grinding time is 4 min and the grinding fineness is less than 0.074 mm and 76.96%, the sulfur removal effect of reverse flotation is optimal, the sulfur grade is reduced to 1.24%, and the recovery rate of alumina reaches 86.89%.



Fig. 5. Grinding time versus ore size (a). Effect of flotation fineness on flotation test (b)

#### 3.4. Flotation experiments

Figure 6(a) shows a comparison of flotation alumina recoveries for hot and non-hot alkali pretreatments. Figure 6(b) shows a comparison of flotation sulfur grades for hot and non-hot alkali pretreatments.

The recovery rate of alumina from flotation was 81.02% and the sulfur grade was 1.25%, and the recovery rate of flotation alumina was 87.45% after hot alkali pretreatment, which increased by 6.43 percentage points, while the sulfur grade decreased to 0.69%, and sulfate dissolution or conversion of the alkali solution may occur during the grinding process, thereby reducing the sulfur content. In addition, some sulfides on the surface of the ore may react with alkaline solutions to form soluble compounds, which can also lead to a decrease in sulfur grades.



Fig. 6 Effect of pretreatment Al<sub>2</sub>O<sub>3</sub> recovery (a), sulfur content (b) before and after pretreatment

# 4. Analysis of mineral surface mechanism before and after surface pretreatment of "hot alkali mill"

# 4.1. Corrosion of diaspore monohydrate

Fig. 7(a) shows the XRD profile of high-sulfur bauxite after terrazzo pretreatment. Fig. 7(b) shows the XRD profile of high-sulfur bauxite after hot alkali mill pretreatment. Table 4 shows Integration of XRD crest area of diaspore monohydrate before and after hot alkali mill pretreatment.

As can be seen from Fig. 7(a) and (b), the peak intensities of pyrite, silicate minerals and titanium minerals did not change much after pretreatment with hot alkali solution, and it can be seen from From Table 4, it can be seen that the peak area of diaspore monohydrate before and after pretreatment was 5924.79 and 5117.68 a.u., respectively. The results show that the pretreatment of hot alkali mill will corrode the surface of diaspore monohydrate, expose more surface sites, reduce the grinding time, enhance the adsorption effect of the inhibitor, and improve the recovery rate of alumina.

Table 4. Integration of XRD crest area of diaspore monohydrate before and after hot alkali mill pretreatment



Fig. 7. (a) XRD plots of high sulfur bauxite after pretreatment with water-milled ore; (b) XRD plots of high sulfur bauxite after pretreatment with hot alkaline solution

# 4.2. Increases the active sites of iron and aluminum on the surface of the particles

Scanning electron microscopy (SEM) was used to analyze the appearance and morphology of pyrite before and after pretreatment by hot alkali milling, and the distribution and content of O, Al, Si, S, Fe and other elements in pyrite were studied by energy scattering spectroscopy (EDS). Fig. 8(a) shows the spectra of the test minerals without pretreatment. Fig. 8(b) shows the spectrum of test minerals after hot alkali mill pretreatment. The corresponding results are shown in Fig. 8.



Fig. 8. SEM images and EDS energy spectra of test minerals before and after pretreatment ((a) test minerals without pretreatment, (b) test minerals after hot alkali pretreatment)

As can be seen from Fig. 8(a), the surface of the test without hot alkali pretreatment is relatively smooth, and the presence of tiny particles on the surface should be the result of slight oxidative acidification of some pyrite. As can be seen from the surface scanning analysis in Fig. 8(b), the purple active aluminum sites and green active iron sites are significantly increased, which increases the active sites of aluminum and iron on the mineral surface during reverse flotation desulfurization, facilitates pyrite floating, inhibits diaspore monohydrate, and improves the recovery rate of alumina. After the pretreatment, the S and O elements on the surface were reduced, indicating that the addition of hot alkali solution restrained the acidification and oxidation of pyrite.

# 4.3. Enhance the floatability of pyrite

AFM imaging uses a mica substrate that is cut with scotch tape to remove the surface of the substrate to obtain an atomically smooth, pure surface. The target mineral particles are then transferred to a mica matrix for AFM experiments. The obtained images were analyzed using Nano Scope Analysis software. Atomic force microscopy (AFM) imaging was used to characterize the morphological differences of pyrite adsorption of collectors before and after surface pretreatment. The scan size is set to 5  $\mu$ m and 5  $\mu$ m, and the left part of each image represents the 2D scan image and the right part represents the 3D image, as shown in Fig. 9 and Fig. 10. The effects of different treatment methods on the surface roughness of pyrite are shown in Table 5.

As can be seen from Fig. 9 and Table 4, the Ra of pure pyrite is 8.69 nm and Rq is 12.3 nm. As can be seen from Fig. 10 and Table 4, after hot alkali pretreatment, the adsorbed PXS-CTAB covers part of the crystal plane in the presence of the combined collector PXS-CTAB, and the spinel plane is more densely convex and there are many small pits, which is due to the adsorption of PXS-CTAB on the pyrite surface and the dissolution of the surface is reduced, which reduces the surface smoothness. Ra and Rq were 27.9 nm and 36.5 nm, respectively, which were much larger than those of pure pyrite, which confirmed that the combined collector PXS-CTAB was strongly adsorbed on the surface of pyrite after hot alkali solution pretreatment, making the surface rougher and enhancing the floatability of pyrite.



Fig. 9. AFM topography image of pyrite surface



Fig. 10. AFM morphology image of pyrite adsorbed PXS-CTAB surface after hot alkali pretreatment

Table 5. Effect of different treatment methods on sphalerite surface roughness

Parameter	Pure pyrite	Pyrite + combined collector after pretreatment PXS-CTAB
Ra	8.69	27.9
Rq	12.3	36.5

Ra: Surface Arithmetic Average Roughness

Rq: Root Mean Square Roughness

## 5. Conclusions

In this study, it was determined that after the pretreatment of hot alkali grinding with a grinding concentration of 50%, a temperature of 60 °C, and grinding time of 4 minutes, the grinding fineness of the sodium hydroxide solution was -0.075 mm, 50%; Slurry pH = 7.5; inhibitor 100 g/Mg; activator 80 g/Mg; collector 240 g/Mg; Under the flotation conditions of 100 g/Mg foaming agent and 6 min scraping time, the alumina grade increased from 57.18% to 87.45%, and the sulfur grade decreased from 5.46% to 0.69%.

The study of the mechanism of hot alkali pretreatment shows that the pretreatment of a hot alkali mill will corrode the surface of diaspore monohydrate, causing it to expose more surface sites. After the pretreatment with a hot alkali solution, the active aluminum sites and active iron sites increased significantly, which enhanced the difference between the active sites of aluminum and iron on the mineral surface during reverse flotation desulfurization, facilitating the upward movement of pyrite, and inhibited diaspore monohydrate. Hot alkali pretreatment restrained the acidification and oxidation of pyrite. After hot alkali pretreatment, the collector has strong adsorption on the surface of pyrite, making its surface rougher and enhancing the floatability of pyrite. In the course of this study, no specific funding support was obtained.

### Acknowledgments

The authors gratefully acknowledge the financial support of the National Natural Science Foundation of China (Grant No. 51464007), SRT Program for College Students of Guizhou University (No. 2024SRT263), and Educational Innovation Project Fund (No. 2024YJSKYJJ136).

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