



Simultaneous removal of sulfur and iron by the seed precipitation of digestion solution for high-sulfur bauxite

Xuejiao Zhou^{a,b}, Jianguo Yin^{b,*}, Yongli Chen^b, Wentang Xia^b, Xiaoyan Xiang^b, Xiaoli Yuan^b

^a School of Metallurgy and Environment, Central South University, Changsha 410083, China

^b School of Metallurgical and Materials Engineering, Chongqing University of Science and Technology, Chongqing 401331, China



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ABSTRACT

Sulfur in bauxite ore results in a series of notable negative effects in Bayer process. It is necessary to remove sulfur for the utilization of high-sulfur bauxite resources. A novel process involving seed precipitation of digestion solution was adopted to simultaneously remove sulfur and iron from the sodium aluminate solution for the high-sulfur bauxite. Seed was prepared from the digestion solution of pyrite in a water bath. X-ray diffraction, a scanning electron microscope and chemical analysis were used to characterize the prepared seed. The utilization of the seed precipitation to remove sulfur and iron for the sulfur-containing sodium aluminate solution was studied. The various parameters included seed dosage, agitation speed and temperature. The results indicated that seed dosage and temperature had a remarkable effect on the sulfur and iron removal. Under the optimum condition, the removal percentages of sulfur and iron from the digestion solution can achieve to 76% and 87%, respectively, and the loss of alumina is lower than 2%. The research shows that the sulfur and iron seed prepared from pyrite can be used effectively for the sulfur and iron removal. A cyclic flowsheet for the simultaneous removal of sulfur and iron for high-sulfur bauxite is proposed.

1. Introduction

Bauxite is a primary mineral to produce alumina. In the last 16 years, China was the world's leading producer of metallurgical-grade alumina, nearly operating at full capacity. The operational yield of Chinese alumina industry in 2016 has exceeded 60 million tons, which results in a great challenge for Chinese bauxite resources (Li et al., 2017). There are > 560 million tons of high-sulfur bauxite (sulfur content is higher than 0.7%) in China and will be expected to reach 2 billion tons in the future, which are widely distributed in Guizhou Province and Chongqing Municipality (Li et al., 2015; Yin et al., 2011). Comprehensive utilization of the high-sulfur bauxite is one of the most important measures to prolong the service life of bauxite resource for Chinese alumina industry (Lou et al., 2016; Yin et al., 2011).

In the Bayer digestion process, sulfur can react with aluminate solution to form S^{2-} and then enter into the sodium aluminate solution, which leads to a series of notable negative effects, such as increasing the alkali consumption, raising the iron concentration in the alumina production, corroding the steel equipment, affecting the evaporation process and so on (Ge et al., 2015; Li et al., 2016; Liu et al., 2018). Consequently, it is necessary to desulfurize for the utilization of high-sulfur bauxite. Much attention has been paid to the desulfurization technology

of high-sulfur bauxite, which can be classified into flotation desulfurization (Chai et al., 2018; Huang et al., 2018), roasting desulfurization (Lou et al., 2016; Yin et al., 2015), electrolysis desulfurization (Gong et al., 2017; Lü et al., 2016) and wet desulfurization (Liu et al., 2015b) and so on. The first two desulfurization methods belong to pretreatment desulfurization, which remove sulfur before Bayer digestion and they are usually the prior methods. But flotation desulfurization generates a large amount of wastes and it is hard to achieve a good desulfurization percentage for a complex sulfur bauxite ore only using this single desulfurization technology, roasting desulfurization increases the energy consumption and environmental contamination (Liu et al., 2015a; Lou et al., 2016; Yin et al., 2011). Electrolysis desulfurization provides a new idea and method for the pretreatment of high-sulfur bauxite, but further study is still needed. Besides, sulfur inevitably enters in the hydrometallurgical process and accumulates in circulation, which has a negative effect on the alumina production (Yin et al., 2015). Therefore, it is necessary to study the process desulfurization while taking measures to intensify the effect of pretreatment desulfurization. Wet desulfurization belongs to the process desulfurization. This method is a very flexible and efficient approach to remove sulfur. But it is usually needed to expend precipitators/oxidants and brings new impurities to the digestion liquor (Yin et al., 2011). New economic and effective

* Corresponding author.

E-mail address: yjqcqust@126.com (J. Yin).

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technology of desulfurization is urgently needed to develop to remove sulfur from the digestion solution.

The sulfur in bauxite mainly presents in the form of pyrite (Ablamoff, 1988; Hu et al., 2011; Liu et al., 2015a), others are marcasite, melnikovite, gypsum and so on. In the high-pressure digestion process, > 90% sulfur transfers into alkali solution, which will increase the solubility of iron (Li et al., 2013; Song et al., 2011). When temperature decreased, sulfur combines with iron to create compounds, such as $\text{Na}_2[\text{FeS}_2(\text{OH})_2 \cdot 2\text{H}_2\text{O}]$, FeS_2 , FeS , $\text{NaFeS}_2 \cdot 2\text{H}_2\text{O}$ and so on, which increases the sulfur and iron content in alumina and decreases the quality of alumina (Li et al., 2016; Song et al., 2011). We consider that cooling and diluting process will decrease the solubility of these substances and makes them precipitated from the digestion solution. Nevertheless, this process is very slow because of the difficulty of spontaneous nucleation. If these substances can be precipitated before the seed precipitation of sodium aluminate solution, simultaneous removal of sulfur and iron could be achieved. According to the classical “nucleation and growth” theory of the phase transformation process, there exists a critical nucleus size or a critical number n_c of atoms (or molecules, ions, etc.) in a nucleus. Nuclei containing fewer atoms than n_c dissolve into the solution, whereas larger ones grow continuously (Erdélyi et al., 2010; Kashchiev, 2000). Adding $\text{Na}_2[\text{FeS}_2(\text{OH})_2 \cdot 2\text{H}_2\text{O}]$, FeS_2 , FeS , $\text{NaFeS}_2 \cdot 2\text{H}_2\text{O}$ seed and mixed substance of them can effectively reduce the energy barrier of nucleation and intensify the removal of sulfur and iron. The present work provides an alternative scheme to remove sulfur and iron by seed precipitation. Seed containing sulfur and iron was prepared and characterized. The effects of seed dosage, agitation speed and temperature on the sulfur and iron removal were discussed. The loss of alumina in this process was evaluated. It was found that this novel process was an economic and effective technology for simultaneously removing sulfur and iron from the sodium aluminate solution. A new feasible and cyclic flowsheet is proposed for desulfurization and iron removal.

2. Experimental

2.1. Materials

Bauxite used in this work was from Nanchuan of Chongqing and pyrite (Fe 46.37%, S 50.07%) was obtained from Hunan province, China. The samples were dried, crushed, ground and screened to < 0.074 mm in particle size. Table 1 and Table 2 show the chemical components and mineral phases of this bauxite. Powder X-ray diffraction (XRD, Smartlab-9, Rigaku, Japan) has been used for phase identification of the bauxite and pyrite, as shown in Fig. 1.

Table 1 shows that the sample of the bauxite contains high silica, high sulfur with a low mass ratio of alumina to silica (A/S). This ore belongs to low-grade high-sulfur bauxite. The proven reserves of this bauxite are > 100 million tons and the prospective reserves are about 300 million tons in Nanchuan Chongqing, China (Yin et al., 2011). Generally, it is considered that sulfur in bauxite higher than 0.7% must be removed or reduced for the utilization.

Table 2 and Fig. 1a indicate that the aluminum in bauxite mainly exists as diaspore and a small amount of boehmite. In order to acquire a satisfactory digestion percentage of alumina, elevated temperature (> 240 °C) and higher caustic alkali concentration (> 240 g L⁻¹) in the Bayer process are needed for the diasporic bauxite (Le et al., 2017). The sulfur mainly exists in the form of pyrite. Other impurities in bauxite are kaolinite and illite as well as a small amount of anatase, rutile,

Table 1
Chemical components of bauxite sample (wt%).

Al_2O_3	SiO_2	Fe_2O_3	TiO_2	K_2O	Na_2O	CaO	MgO	S_{Total}	C_{Total}	C_{Organic}
59.62	17.84	1.82	2.89	0.83	0.17	0.14	0.19	0.88	0.29	0.28

Table 2
Mineral phases of bauxite sample (wt%).

Diaspore	Boehmite	Kaolinite	Illite	Anatase	Rutile	Hematite	Pyrite
44	8	31	8	2.4	0.5	1	1.2

hematite and some trace of unidentified phases. Fig. 1b shows that all detected peaks can be attributed to pyrite, which demonstrates that pyrite used in experiments is of high purity.

Morphological features of the bauxite and pyrite have been analyzed using a scanning electron microscope (SEM, JSM-7800F, JEOL, Japan), as displayed in Fig. 2. It reveals that the bauxite and pyrite samples are irregular particles, but on the surface of bauxite has obvious agglomeration phenomenon. More details about the electronic mapping along with electronic dispersive spectra of the bauxite are shown in Fig. 3. The electronic dispersive spectra of the whole face make clear that the bauxite contains a mass of elements O, Al, Si and a certain amount of C, S, Fe, in which C is mainly derived from carbon coating applied to provide conductivity to the sample surface during image collection. The elemental mappings of Al, Si, Ti and Fe indicate that the concentration of Al is more compared to other elements and the mappings confirm again that the sulfur can combine iron to form pyrite.

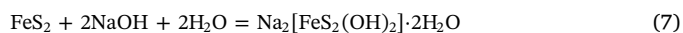
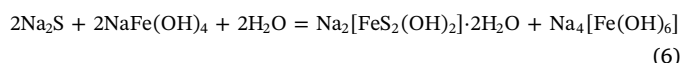
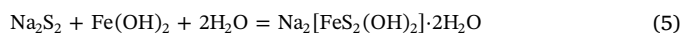
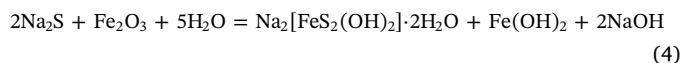
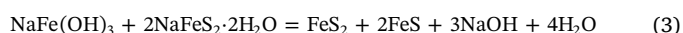
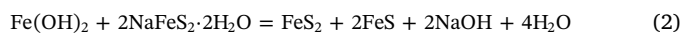
All chemicals used in the experiment were of analytical reagent and all aqueous solutions were prepared by ultrapure water.

2.2. Experimental procedure

2.2.1. Preparation of seed experiments

Before preparation of seed experiments, pressure alkaline digestion of pyrite was carried out in an autoclave and conditions controlled as follows: 260 g L⁻¹ caustic alkali (Na_2O_K), liquid to solid ratio of 100 mL g⁻¹, reaction temperature 270 °C, agitation speed 100 rpm and reaction time 2 h (after achieving the desired temperature 270 °C). After pressure digestion, the slurry was separated by vacuum filtration and the filtrate was used to the seed preparation experiments. Next, the filtrate poured into a beaker, then covered with a plastic wrap in a thermostat water bath controlled at 25 °C, as shown in Fig. 4. Photographs of the seed in the nature precipitation process were displayed in Fig. 5.

As can be seen in Fig. 5, the seed preparation process is very slow at first 3 days. But a large amount of dark green seed was aggregated in the bottom of the beaker with part of seed suspended in the solution on day 4, which precipitated completely on day 5. Possible chemical reactions during the seed precipitation process are as follows (Ablamoff, 1988; Li et al., 2015; Li et al., 2016):



Afterwards, the supernatant liquor was removed to get concentrated seed slurry, as shown in Fig. 5g. Then part of the seed slurry was dried for XRD analysis and SEM observation. The other part of seed slurry was prepared for sulfur and iron removal in subsequent experiments.

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