



Flotation experimental research of calcareous–siliceous phosphorite



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ABSTRACT

Based on the analysis of occurrence states and properties of a calcareous–siliceous phosphorite, the effects of GZ₁ collector dosage, regulator dosage, pulp density and particles size distribution of sample on concentrate indexes were investigated by reverse flotation technology. The rare earth oxidize (REO) grade and recovery of concentrate were also analyzed in the work. Two-stage reverse flotation technology was applied for the beneficiation of a calcareous–siliceous phosphorite. Under the integrated optimal single factor conditions of GZ₁ collector 0.9 kg/t, phosphate regulator 14 kg/t and pulp density 35 wt.%, mass fraction of particles size of $-74\ \mu\text{m}$ of 85 wt.%, flotation time of 11 min and flotation pH of 5.5, the P₂O₅ grade increases from 14.64 wt.% in raw ore to 26.82 wt.% in the concentrate, and the recovery is 72.09 wt.%, while REO grade increases from 0.08 wt.% in the raw ore to 0.175 wt.% in the concentrate, and the recovery is 86.08 wt.%.

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1. Introduction

Phosphate ores are vital nonrenewable resources and essential components in agricultural fertilizers and phosphorous-based chemicals (Mohammadkhani et al., 2011). Phosphate bedrock in Zhijin Xinhua of Guizhou is a large composite ore deposit with phosphate ore containing REE. The reserve of explored phosphate ore is 1.33 billion tons, which accounts for about a half of the total phosphate ore reserves of Guizhou. While, the amount of rare earth oxidize (REO) is 1.45 million tons. The calcareous–siliceous phosphate ore in Zhijin Xinhua holds an important position in explored P₂O₅ and rare earth reserve of China (Zhang et al., 2003; Zhang et al., 2006; Zhang et al., 2007; Zhang & Zhang, 2008). Reasonable P₂O₅ extraction and comprehensive utilization of other valuable elements are equally important because the special electronic structure of REE is the basis of light, electricity and magnetism. In addition, many researchers around the world want to make breakthrough in the aspects of new properties, functions, materials and devices for the excellent properties of REE.

Phosphate rock resources are very rich in Guizhou province. The calcareous–siliceous types of phosphate ores are extremely difficult to be concentrated by flotation, because the similar physicochemical characteristics of surfaces of the main constituents, carbonates and phosphates. For the last few decades, flotation was popularly used to separate carbonates from phosphates. These studies mainly focused

on modification of the techniques, controlling pulp environments, and finding new reagents to separate carbonates from phosphate ore.

The calcareous–siliceous phosphate ore has not been reasonable developed and utilized yet for the difficult separation of carbonates and silicates. The processing of calcareous–siliceous phosphate ore still represents a continuous industrial worldwide challenge (Al-Fariss et al., 2013; Boulos et al., 2014). The presence of carbonate minerals affects the flotation of phosphate and silica, particularly calcite (Guo et al., 2011). Although the process had been implemented on a commercial scale perhaps, yet there is a lot of discrepancy about the best strategy to achieve selectivity. That was attributed to the similarities in the surface properties of both the phosphate and the carbonate minerals (Abdel-Khalek, 2000). The calcareous–siliceous phosphate ore is affected not only by the phosphate's own solution chemistry but also by the dissolved Ca²⁺, Mg²⁺ from other salt-type minerals in the system (Amankonah & Sumasundaran, 1985).

Based on the above discussion, the collectors of calcareous–siliceous phosphate ore should show good selectivity and strong ability of collecting. To improve the grade and recovery of P₂O₅, GZ₁ mixed collector was developed considering dolomite and apatite surface properties. In addition, the key constraining factors of exploitation of calcareous–siliceous phosphate ore are occurrence states of the CaO and SO₂ in the minerals and separation process (Lu, 2006; Jin et al., 2011; Abouzeid et al., 2009; Tian et al., 2011). So, it is very important to find out the existing form of P₂O₅, CaO and SiO₂ in phosphate ore, independent minerals or isomorphism. The separation of calcareous–siliceous phosphate ore was studied by flotation through analyzing occurrence states of P₂O₅, CaO and SO₂ in the work.

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2. Materials and methods

2.1. Component analysis of the sample

The sample is a light-blue calcareous–siliceous phosphorite ore with biomass dolomite and granular structure. The XRD analysis results are shown in Fig. 1. Phosphate minerals mainly contain fluorapatite and a little microcrystal carbonate-fluorapatite. Associated gangue minerals have dolomite, quartz and calcite, etc. Analysis results of sample components are shown in Tables 1, 2.

As Table 1 lists, P_2O_5 grade is 14.64 wt.%, \sum REO grade is 0.08 wt.%, and the grade of CaO, SiO_2 , MgO, Al_2O_3 and Fe_2O_3 is 46.46 wt.%, 10.32 wt.%, 7.86 wt.%, 0.62 wt.%, 0.86 wt.%, respectively. Therefore, the grade and recovery of P_2O_5 should be considered in separation technology of phosphate ore. ICP-MS analyses of REO are shown in Table 2, the grade of La_2O_3 , CeO_2 and Nd_2O_3 is 236×10^{-6} , 138.17×10^{-6} and 178.73×10^{-6} , respectively. The grade of other single REO is less than 81.93×10^{-6} .

2.2. Sieve analysis of the sample

Raw ore was crushed to -2 mm and grinded in the XMQ-240 \times 90 conical ball mill. Then the sieve analysis was carried out in bushing screen. The results are listed as Table 3. The particles size of sample is divided into five grades. As can be seen from Table 3, the proportion of $-75 \mu m$ – $+44 \mu m$ is 4.62 wt.%, while the grade of \sum REO and P_2O_5 is 0.088 wt.% and 16.86 wt.%, respectively. The proportion of $-44 \mu m$ size grade is 10.95 wt.%, while the \sum REO and P_2O_5 grade is 0.074 wt.% and 16.53 wt.%, respectively. The \sum REO and P_2O_5 grade of $-75 \mu m$ – $+44 \mu m$ grade is 0.079 wt.%, and 14.21 wt.%–14.73 wt.%, respectively. By comprehensive consideration of P_2O_5 grade, \sum REO grade and their distributions, the liberation degree of phosphate minerals in $-75 \mu m$ – $+44 \mu m$ grade is best, so the optimal grinding fineness is 80 wt.% of $-75 \mu m$ – $+44 \mu m$, which is used in the following single factor experiment.

2.3. Separation of calcareous–siliceous phosphate ore

Two-stage reverse flotation technology is shown in Fig. 2, which is applied to separate phosphates from carbonate gangues in the study. Pulp consisting of mineral particles and water were blended with appropriate regulator. Then the pulp was poured into flotation cell and

Table 1

Analysis results of sample component (wt.%).

Components	P_2O_5	CaO	SiO_2	MgO	Al_2O_3	Fe_2O_3	\sum REO	Others
Mass fraction	14.64	46.46	10.32	7.86	0.62	0.86	0.08	19.16

stirred with collector. This leads to the hydrophobic difference increase of hydrophobic minerals and hydrophilic minerals. Therefore, the minerals containing P_2O_5 remain in the pulp and other gangue minerals adhered to bubbles float to the pulp surface, which makes separation of valuable minerals containing P_2O_5 from gangue minerals.

2.4. Flotation reagents

The flotation reagents are shown in Table 4. The reverse flotation collectors of calcareous–siliceous phosphate ore should have the following features. One hand, it has good collecting effect toward carbonate, and senior fatty acid soap has the features. On the other hand, the collectors of calcareous–siliceous phosphate ore should contain ligand oxygen and nitrogen because P_2O_5 doesn't react with hard alkali oxygen with electron, as well as nitrogen. The GZ₁ is a commercial mixed collector, and its major ingredients are 90 wt.% of senior fatty acid soap (paraffin soap) and 10 wt.% of nonionicsurfactant reagents. Based on the above mechanism and surface properties of the apatite and dolomite, GZ₁ mixed collector is chosen as collector. GZ₁ collector can selectively act on the dolomite surface, increase dolomite particles surface hydrophobicity, and possess foaming ability to realize separation of the dolomite and apatite. In addition, GZ₁ mixed collector is also frother. The strong hydrogen bond is formed between the phosphate ion and water molecules around phosphate ore particles when using H_3PO_4 , and generate soluble $CaHPO_4$, which can enhance hydrophilicity of phosphate ore particles, thus H_3PO_4 is chosen depressant and pH regulator.

3. Results and discussion

3.1. Influence of collector dosage

The conditions of flotation process are that collector dosage is 0.8 kg/t, 0.9 kg/t, 1.0 kg/t and 1.1 kg/t, respectively, regulator dosage is 16 kg/t of phosphoric acid, pulp density is 30 wt.%, flotation time is 11 min and flotation pH is 5.5 (Jin et al., 2011; Abouzeid et al., 2009). The result is presented in Fig. 3.

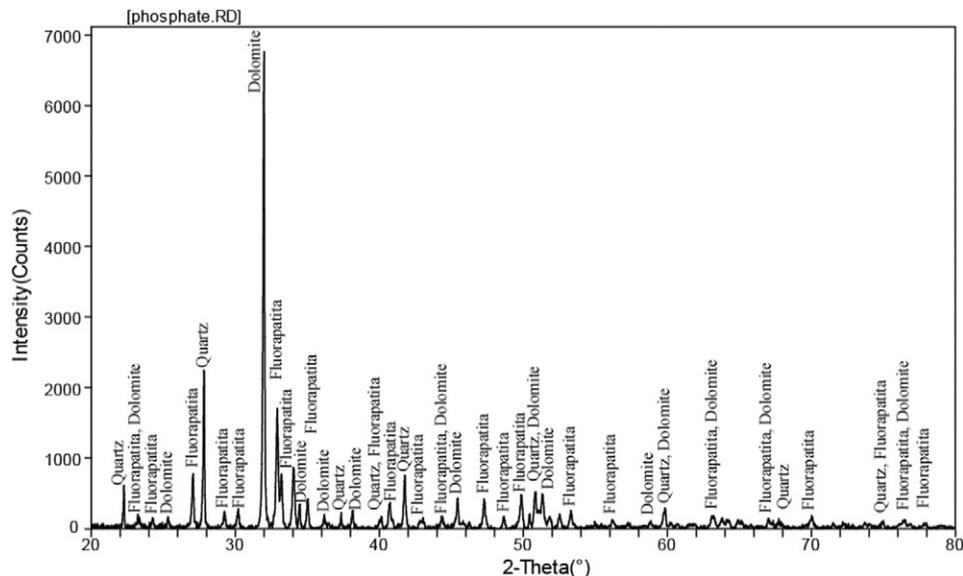


Fig. 1. XRD pattern of calcareous–siliceous phosphorite ore.

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