Note

Research on mineralogy and flotation for coal-series kaolin

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ABSTRACT

The iron mineral was separated by froth flotation for the coal-series kaolin. The mineralogy of the coal-series kaolin was studied by several methods such as X-ray diffraction (XRD), X-ray fluorescence (XRF), particle size distribution and SEM/EDS analyses. The mineralogy demonstrated that pyrite was the main occurrence of iron in the coal-series kaolin. The influence of grinding time and pH on the flotation was researched so as to determine the optimal flotation conditions. The results of froth flotation showed that the mass fraction of pyrite in kaolin concentrate was 0.11% at 1.12% pyrite in the flotation feed, and hence 90.72% of total pyrite was separated by froth flotation. The findings in this study suggest that the effective pyrite separation from coal-series kaolin was achieved by grinding moderate time and controlling pH weak acid and neutral.

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1. Introduction

The studied coal-series kaolin (from Yichang city, Hubei province, China) is the hard kaolin ore associated with the coal, which is commonly known as gangue that is color mineral of gray and dark. When the coal is to be mined and processed, a great deal of the gangues is discarded into the tailing dam which waste mineral resources and pollute the environment (Ji et al., 2013). The main valuable mineral of the gangue is kaolinite and the production of kaolinite produced by coal gangue in China is about 0.3 million tonnes annually (~10% of total kaolin production in China) and mainly used in paint, paper, rubber, plastics, ceramics and chemicals industries (Wu and Wang, 2005). Additionally the potential use of coal-series kaolin in cement production as cementitious material has been studied by many researchers due to the enhancement of early strength and improvement of durability by incorporation of calcined coal-series kaolin into mortar and concrete (Siddique and Klaus, 2009; Zhang et al., 2009). However, the impurities such as iron, titanium, carbonaceous matters have a detrimental influence on the applications of kaolin (Raghavan et al., 1997). There are limited standards and published commercial specifications in China for the different industry products which specify the chemical components of kaolin and other process parameters such as whiteness, particle size,

viscosity etc. (Raghavan et al., 2004; GB/T 14563, 2008) and commercial specifications in world can be also found in Le Berre and Berton (1983) and Harben (1998). To remove the impurities and meet the limited standards and specifications is significant and essential for the comprehensive utilization of the coal-series kaolin.

There are several separation methods to remove the impurities from kaolin such as size classification (Chandrasekhar and Ramaswamy, 2006), froth flotation (Raghavan et al., 2007, 2008), high gradient magnetic separation (Ambikadevi and Lalithambika, 2000; Santos et al., 2012), bleaching (Veglio', 1997), leaching (Lee et al., 2006; Martínez-Luévanos et al., 2011), chlorination (Orosco et al., 2011) and selective flocculation (Poorni and Natarajan, 2013) etc. at present. In this study, the froth flotation was used to remove the pyrite in coal-series kaolin.

Pyrite, the Fe sulfides content in coal-series kaolin, is usually associated with organic matter contents which limited ceramic use in fast firing because of usual excessive organic matter content. This fact is remediable by heat treatments (De la Torre et al., 1995; Lores et al., 1997; Bastida et al., 2006) but ceramic use may remain limited by excessive Fe sulfides contents which is required the pyrite removal in coal-series kaolin.

An important problem is the comminution of kaolin that leads to the argillization which is not advantageous to the froth flotation. The effectiveness of the forth flotation decreases with the decline of particle size (Mathur, 2002). Therefore, this study focused on the influence of particle size on pyrite separation by froth flotation for coal-series kaolin. Furthermore the influence of pH and the mineralogy of coal-series kaolin were also studied.

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2. Materials and methods

2.1. Materials and reagents

The crude coal-series kaolin sample was prepared by crushing and sieving with jaw crusher and oscillating screen. The particle size of the obtained crude coal-series kaolin sample was lower than 2000 μm.

All the reagents such as sulfuric acid, sodium carbonate and butyl xanthate were analytically pure. The deionized water with the electrical resistivity over 10 MΩ·cm was used.

2.2. Flotation experiments

The crude coal-series kaolin samples were ground using the porcelain ball mill (~40% ball loading rate with 320 balls in 15 mm diameter and 180 balls in 10 mm diameter) for different time to liberate locked pyrite and simultaneously added dispersant (sodium silicate, 3.75 kg/t). The single flotation cell was used for flotation to separate pyrite. The sulfuric acid or sodium carbonate was added to regulate the pH of the pulp. The pyrite was floated as adding the collector (butyl xanthate, 80 g/t) and the frother (No. 2 oil, 20 g/t) in turns. To increase the removal rate of iron, the concentrating was adopted by adding the half dosage of collector and frother when the rougher flotation finished. The froth products (pyrite tailing) and bottom products (kaolin concentrate) in the cell were weighed respectively after suction filtering and drying. The yield of concentrate was calculated and the mass fraction of iron in concentrate was assayed. Afterwards the flotation performance was evaluated by the yield of kaolin concentrate and the removal rate of iron. This froth flotation adopted the one-factor experimental design to evaluate the influence of grinding time and pH on the flotation performance.

2.3. Determination of iron mass fraction (Fe_2O_3)

The mass fraction of iron was determined using ultraviolet spectrophotometer based on the Chinese standard (GB/T 14563, 2008). Initially the standard solution of Fe_2O_3 (0.1 mg·mL^{-1}) was prepared by dissolving Fe_2O_3 (spectroscopic pure) with the 6 mol·L^{-1} hydrochloric acid solution and then diluting with deionized water. Afterwards the calibration curve was constructed by determining the absorbance of the working standard solutions at 420 nm adsorption wavelength which adopted the sulfosalicylic acid as the color agent in the ammonia solution and the concentrations ranged from 0 μg·L^{-1} to 10 μg·L^{-1}. Finally the absorbance of kaolin samples was determined according to above-mentioned method and then the mass fraction of iron (% Fe_2O_3) was calculated based on the calibration curve.

2.4. Characterizations

Mineralogical composition was determined by powder XRD using a rotation anode high power X-ray diffractometer (RU-200B/D/MAX-RB, Rigaku Corporation, Japan) with CuKα radiation (λ = 0.154 nm) (40 kV, 50 mA) over the scanning range 2θ = 5–70° with a step width of 2°/min.

The chemical compositions of the kaolin were determined using the Axios advanced spectrometer (PANalytical.B.V Dutch).

The particle size distribution was measured by a laser particle size analyzer (BT-9300S, Bettersize Instruments Ltd., China) using a dilute suspension conditioned with dispersants (sodium hexametaphosphate, analytically pure). Additionally the particle size distribution of crude coal-series kaolin sample was combined with dry sieving to determine the coarse fraction (45 μm–2000 μm).

The element mapping and energy spectrum was detected by the field emission scanning electron microscope (Zeiss Ultra Plus, Carl Zeiss, Germany) which assembled the energy dispersive spectrometer.

3. Results

3.1. Mineralogy of crude coal-series kaolin

The major valuable mineral of crude coal-series kaolin sample observed by XRD pattern was kaolinite and the gangue minerals were pyrite, quartz, anatase, boehmite and feldspar (Fig. 1). Pyrite (maximal intensity reflection at d-value 2.71 Å) was the only iron-bearing mineral in the crude coal-series kaolin sample. Meanwhile, the Hinckley index of crystallinity calculated based on the XRD pattern at the (02.11) band (Nahdi et al., 2002; Franco et al., 2004) for the studied kaolinite was 0.56 which belonged to the disordered kaolinite with high defects. The chemical components of the crude coal-series kaolin sample detected by XRF analysis were 47.30% SiO_2, 33.89% Al_2O_3, 0.93% Fe_2O_3, 1.49% SO_3, 2.15% TiO_2, 0.28% K_2O, 14.07% LOI (loss on ignition) and other minor elements. The mass fraction of pyrite was approximately 1.12% calculated by XRF analysis, which indicated that 80.11% of the total iron was present as pyrite. The difference (19.89% of the total iron, 0.18% Fe_2O_3) must correspond to Fe oxide as oxidated pyrite and to Fe content in kaolinite structure which was invisible in the XRD pattern (Fig. 1).

3.2. Influence of grinding time

The median diameter D_{50} (the size of 50% cumulative passing) obtained from the particle size distribution (Fig. 2) of the crude coal-series kaolin sample and flotation feed with different grinding time was 380 μm (crude sample), 17.07 μm (7 min), 11.36 μm (8 min), 9.944 μm (9 min), 8.963 μm (12 min) and 7.076 μm (15 min). In Fig. 2 the grinding process resulted in the remarkably decrease of the particle size for the coal-series kaolin and as the grinding time increased, the median diameter D_{50} decreased. The decrease rate of particle size was fast before grinding time of 9 min, while after 9 min, the decrease rate became slow.

The influence of the median diameter D_{50} on the flotation performance was presented in Fig. 3(a). As the median diameter D_{50} increased, the removal rate of iron decreased, while the yield of concentrate shown a counter trend with respect to removal rate of iron. The optimal point corresponding to the excellent flotation performance was median diameter of 8.963 μm and meantime the grinding time was 12 min. The particle size distribution of pyrite tailing at the optimal grinding time (12 min) was also displayed in Fig. 2 and hence median diameter D_{50} of pyrite tailing was 29.81 μm. Comparing with the flotation feed, the size of pyrite tailing was much coarser than flotation feed and the passing rate of 8.963 μm (D_{50} for flotation feed) was almost 75%. Additionally there were also a slight amount of fine particles (lower than 10 μm), which indicated that the fine particles of kaolinite were entrained into the pyrite tailing.

The kaolinite is generally recognized as the 1:1 clay which consists of a tetrahedral silica sheet alternating with an octahedral alumina sheet. The end tetrahedral silica sheet connects with the adjacent octahedral alumina sheet by the bonding oxygen atom, and forms a layer. The two adjacent layers are held by the weak hydrogen bond. As the naturally layered mineral, kaolinite is easily over-ground into fine particle size and expresses an unsatisfactory flotation performance.

In this experiment, to avoid the overgrinding, the moderate time (12 min) was selected to grind the coal-series kaolin and liberate the locked pyrite. In Fig. 2 pyrite was coarse and well-liberated and performed a perfect separation efficiency. Therefore, controlling the suitable grinding time could liberate the pyrite effectively and avoid the overgrinding, which was significant and essential for the pyrite separation.

3.3. Influence of pH

The sulfuric acid and sodium carbonate was used to shift the pulp to acid and alkaline, respectively. The influence of pH on flotation