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Surface dissolution of spodumene and its role in the flotation concentration of a spodumene ore



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ABSTRACT

The effects of sodium hydroxide and sodium carbonate on the flotation of spodumene were investigated by flotation tests, XPS and SEM measurments and thermodynamic analysis. A collector YOA (a mixture of 90% oleic acid and 10% dodecyl amine) was used in the flotation. It was found that sodium hydroxide showed an activation effect whereas sodium carbonate showed a depression effect on the flotation of spodumene. This difference in flotation performance was consistent with the measured contact angles of spodumene treated in sodium hydroxide and sodium carbonate solutions, respectively. XPS results indicated that the pretreatment of spodumene surface by sodium hydroxide gave larger relative contents of Al and Li on spodumene surface than that of sodium carbonate, and the scanning electron microscopy images showed rougher surfaces indicating leaching of the mineral surface. The spontaneity of possible reactions associated with the surface dissolution of spodumene in alkaline solutions was also examined, and the results could largely account for the observed difference in surface dissolution of spodumene in sodium hydroxide and sodium carbonate solutions.

1. Introduction

Spodumene is an important resource for extraction of lithium. Recovering spodumene from spodumene-rich ore is often achieved by flotation. The effective collectors for the spodumene flotation practice are oleic acid, oxidized paraffin soap, naphthenic acid soap and tall oil (Felix and Reiner, 2010; Yu et al., 2015; Menéndez et al., 2004; Wang and Yu, 2005; He 2009; Wang et al., 2018). Flotation of spodumene is commonly conducted in alkaline solutions with NaOH and Na₂CO₃. Na2CO3 was mainly used as depressant for silicates while NaOH was used as activator for spodumene flotation. The flotation of spodumene can also be changed by using different dosage of alkalis and stirring time (Zhang et al., 1983; Wang and Yu, 2007; Yu et al., 2014). Moon and Fuerstenau (2003) pointed out that the flotation recovery of spodumene with sodium oleate was greatly improved when the mineral sample was pretreated by an alkaline solution at pH 12. And the XPS analysis suggested that NaOH would allow more Al sites of spodumene surface to be exposed to the aqueous solution. It has also been found that spodumene was preferentially leached by conditioning in a concentrated NaOH solution (Yu et al., 2015). For the selective separation of spodumene from other silicates, the surface Al site on {110} cleavage plane of spodumene is considered the most favorable site for the selective chemisorption of oleate, while those in other pegmatic alumino-silicates are buried deep inside the crystallographic unit cells of the minerals which makes them unavailable for oleate adsorption (Moon and Fuerstenau, 2003; Zhu et al., 2015).

It is widely recognized that many minerals will dissolve in aqueous solutions, and at certain pHs, the dissolved species can undergo various reactions, such as hydrolysis, complexation, adsorption, and even surface or bulk precipitation, which can significantly affect the interfacial properties of the mineral particles and their flotation behavior (Somasundaran and Wang, 2006). Minerals of phosphates (apatite and francolite), tungsten (wolframite and scheelite) and associated carbonates (calcite and dolomite) are sparingly soluble in aqueous solution, and the dissolved mineral species in bulk solution can interact with mineral solids, often resulting in surface conversion of the minerals. In addition, these dissolved species can interact with various floation reagents to form surface or bulk precipitation, leading to loss of flotation selectivity (Somasundaran et al., 1991). For example, quartz can be activated by dissolved Zn(II) ions and floated by using oleate in alkaline system, making it difficult to separate smithsonite (ZnCO₃) from quartz (Majid et al., 2012). In the flotation of ilmentite, surface dissolution of ilmentite was carried out by acid pretreatment to convert Fe²⁺ ions on the ilmenite surface to Fe3+ ions, which results in an increase in the

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adsorption of oleate ions on the ilmentite surface (Parisa et al. (2016); Zhu et al., 2011). Ultrasonic pretreatment on the oxidized pyrite can remove the oxidation products from the pyrite surface and increase the flotation of pyrite (Cao et al., 2017). High intensity conditioning can produce an ultra clean surface of coal particles and effectively improve the hydrophobicity of coal surface (Yu et al., 2017).

In the present work, the effects of sodium hydroxide and sodium carbonate on surface properties and flotation behavior of spodumene were systematically studied by means of flotation tests, contact angle, XPS and SEM meassurments. The role of surface dissolution in the flotation concentration of a spodumene ore was also investigated. The results have significant implications for flotation of spodumene and similar minerals.

2. Materials and methods

2.1. Mineral samples and reagents

Bulk spodumene samples were collected from Xinjiang Koktokay Rare Metal Mine, China. Then it was crushed with hammer and hand-picked to remove a small proportion of impurity minerals, ground in a porcelain mill with agate balls, and then sieved with stainless steel standard screens in deionized water to obtain the -0.104 + 0.038 mm fraction samples. These samples were then subjected to high intensity magnetic separation with field strength of 1.5 T to remove iron containing impurities, and stored in glass bottles after vacuum-drying. The analyses of XRD and chemical components on spodumene samples were also carried out, and the results showed that spodumene samples had high purity with the Li₂O content being 7.81% by weight. More details on the XRD results and chemical components of the spodumene sample were shown in Table 1 and Fig. 1.

A pegmatite ore (with the $\rm Li_2O$ content being 1.48% by weight) was provided by Ganzi Rongda lithium Co., Ltd. in Sichuan, China. The valuable mineral in the ore is spodumene, and the gangue minerals are quartz, feldspar and mica. More details on the chemical composition and XRD results of the ore can be found in Table 2 and Fig. 2.

Deionized water (resistivity $> 18.2~M\Omega\cdot cm$) was used throughout the experiments. Analytical sodium hydroxide (NaOH) and sodium carbonate (Na₂CO₃) were used as regulators. YOA (a mixture of 90% oleic acid and 10% dodecyl amine) was used as collector without the addition of frother. Analytical pure calcium chloride was also adopted as regulator in the flotation of spodumene ore according to the practice of spodumene flotation.

2.2. Flotation tests

Microflotation tests for the spodumene samples were carried out using an XFGII-type self-aeration flotation machine with a 40 ml cell at room temperature. In each test, the flotation cell was loaded with 5 g of the samples and deionized water, and the pulp was then conditioned at an agitation speed of 1890 rpm for 30 min with addition of alkalis and for 3 min with addition of YOA. After the pulp conditioning, the flotation lasted for 4 min. The concentrates (froth products) and tailings (residue) were individually filtered, dried and weighed to calculate the mass recovery. For each test condition, three experimental runs were conducted and the mean value of the data was reported with the recovery deviation less than 1%.

The spodumene ore was ground in a $\Phi200 \times 240$ mm XMB-type steel bar mill to 70% passing 0.074 mm. The pulp was then transferred to a XFD-type self-aeration flotation machine with a 1500 ml cell. Various

 Table 1

 Chemical components of spodumen single-mineral samples (%).

Componetes	SiO_2	Al_2O_3	Li ₂ O	K_2O	Na ₂ O	Fe ₂ O ₃	CaO	MnO	Cr ₂ O ₃
Content	61.09	27.43	7.81	0.22	0.32	0.94	0.15	0.16	0.05

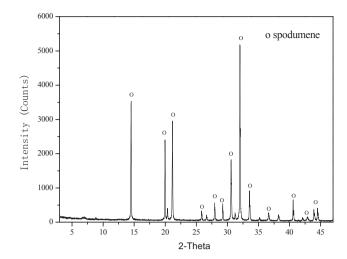


Fig. 1. XRD diffraction pattern of the spodumene.

Table 2
Chemical composition of the spodumene ore (%).

Elements	Al_2O_3	SiO_2	Li_2O	BeO	Nb_2O_5	Ta_2O_5	Fe_2O_3	CaO	MgO
Content	14.43	73.29	1.48	0.01	0.011	0.009	2.01	0.21	0.13

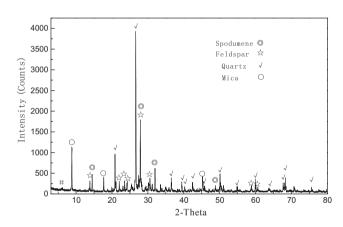


Fig. 2. XRD diffraction pattern of the spodumene ore.

reagents were added successively, and the pulp was conditioned for a fixed period of time before starting flotation at an agitation speed of 1900 rpm and 2.5L/min aeration rate. All the flotation tests were carried out at room temperature. The concentrates (froth products) and tailings (residue) were separately filtered, dried, weighed and analyzed (by chemical method) to determine the flotation performance. Three experimental runs were conducted for each test condition and the weighted average of the data with the deviation less than 1% was adopted.

2.3. Contact angle meaurements

The advancing contact angles of the single spodumene sample were measured using the sessile air bubble method. A clean mineral crystal was placed in the reagent solution of YOA at room temperature. A 3S contact angle meter (GBX Company, France) was used to measure the contact angle 5 times, and the average of the data was reported. The measuring uncertainty was $\pm\,2^\circ$. Between each measurement, the crystal was cleaned with acetone first, then polished with sandpaper (abrasive size of 2.6 μm), and washed with deionized water.

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