



Flotation and adsorption of a new mixed anionic/cationic collector in the spodumene-feldspar system



Houqin Wu^a, Jia Tian^c, Longhua Xu^{b,c,*}, Shuai Fang^a, Zhenyue Zhang^d, Ruan Chi^d

^a School of Civil Engineering and Architecture, Southwest University of Science and Technology, Mianyang, Sichuan, PR China

^b School of Chemical Engineering, Sichuan University, Chengdu, Sichuan, PR China

^c Key Laboratory of Biohydrometallurgy, Ministry of Education, School of Resources Processing and Bioengineering, Central South University, Changsha, Hunan, PR China

^d Key Laboratory for Green Chemical Process of Ministry of Education, Wuhan Institute of Technology, Wuhan, Hubei, PR China

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ABSTRACT

The flotation separation of spodumene from feldspar was investigated using a new type of mixed anionic/cationic collector, sodium oleate/tributyl tetradecyl phosphonium chloride (NaOL/TTPC). Microflotation experiments, fluorescence spectroscopy measurements, microcalorimetric analysis, and Fourier transform infrared analysis were performed. Microflotation experiments show that the highest flotation separation performance can be achieved at the NaOL:TTPC molar ratio of 5:1 at the pH of 4.0. Fluorescence spectroscopy measurements show that the critical micelle concentration (CMC) of the mixed collector NaOL/TTPC is smaller than either of its components alone. Microcalorimetric results indicate that the difference in adsorption reaction heat between spodumene and feldspar is larger for the mixed collector than either single collector, confirming the former's separation advantage. Fourier transform infrared analyses show that in the mixed collector, both NaOL and TTPC adsorb on the spodumene surface through chemical interaction and electrostatic attraction, respectively; while only trace amounts of them are adsorbed on the feldspar surface.

1. Introduction

Lithium has remarkable physicochemical properties, such as high conductivity and high chemical activity, thus it is widely used in many fields, especially in energy storage (Huang et al., 2017; Tan et al., 2017). Spodumene ($\text{LiAlSi}_2\text{O}_6$) is one of the main sources of lithium, and is generally found in pegmatite deposits (Xu et al., 2016a). Since these deposits contain silicates as associated minerals, such as feldspar, flotation is generally employed for the selective separation of spodumene (Barbosa et al., 2014; Tian et al., 2017a).

To realise successful flotation separation, numerous researchers have studied the surface properties of spodumene and feldspar (Moon and Fuerstenau, 2003; Xu et al., 2016b; Xu et al., 2018). Moon and Fuerstenau (2003) found that the surface Al site on {110} cleavage plane of spodumene was the most favourable site for the selective chemisorption of oleate. However, in other pegmatic aluminosilicates, these Al sites are buried deep inside the crystallographic unit cells of the minerals, making them unavailable for oleate adsorption (Moon and Fuerstenau, 2003). Xu et al. (2017a, 2017b) studied the surface chemistry of spodumene and feldspar particles in different sizes. Their results confirmed that the Al content on the feldspar and spodumene

surfaces varied with the particle size, explaining the different flotation performance depending on particle sizes (Xu et al., 2017a, 2017b).

However, the surface properties of aluminosilicate minerals are only one factor in the flotation separation. Many researchers have also attached great importance to the selection of suitable collectors for the flotation separation process. Due to the similar surface properties of aluminosilicate minerals, it is difficult to separate spodumene from gangue minerals using traditional single fatty acid collectors during flotation (Liu et al., 2015a; Moon and Fuerstenau, 2003). The use of mixed collectors is considered a possible solution to this problem. Especially, the flotation of oxide and silicate minerals using mixed collectors has attracted more and more attention (Hosseini and Forssberg, 2007; Vidyadhar and Hanumantha Rao, 2007; Wang et al., 2014). Mixed collectors used in optimum conditions may have the characteristics of both single collectors and thus can satisfy multiple requirements (Xu et al., 2017c; Zhang and Somasundaran, 2006). For example, Yang et al. (2016) found that the mixed collector of sodium oleate–benzohydroxamic acid (NaOL–BHA) at a molar ratio of 4:1 exhibited excellent performance in the flotation separation of ilmenite from titanite (Yang et al., 2016). Yan et al. (2017) found that the mixture of oxidised paraffin soap (OPS) and BHA demonstrated a higher

* Corresponding author at: School of Chemical Engineering, Sichuan University, Chengdu, Sichuan, PR China.

E-mail addresses: neuxulonghua@163.com, xulonghua@swust.edu.cn (L. Xu).

selectivity for the flotation of scheelite from calcite at pH 9 than individual OPS and BHA (Yan et al., 2017). According to the types of components, mixed collectors are classified into categories such as the mixed anionic–cationic, anionic–non-ionic, and cationic–non-ionic collectors (Rao and Forssberg, 1997). Among them, the mixed anionic–cationic collectors are most extensively investigated due to the special interaction between the differently charged components (Wang et al., 2014). Studies show that there is synergistic adsorption of the mixed anionic–cationic collectors on the mineral surfaces (Cui et al., 2010; Xu et al., 2017c), because the electrostatic repulsion between the similarly charged head groups in one collector is reduced by the presence of another collector with head groups carrying the opposite charge. The result is a closer packing of the first collector on the mineral surface. At the same time, the second collector can co-adsorb on the mineral surface by forming a neutral complex with the first collector (Rao and Forssberg, 1997). Xu et al. (2013) found that the adsorptions of dodecylamine acetate (DDA) and NaOL on muscovite were enhanced in the mixed systems in comparison to those of either single collector (Xu et al. 2013). Ejtemaei et al. (2011) found that the concentration of anionic collector potassium amyl xanthate (KAX) affected the adsorption of Armac C, a cationic surfactant amine salt, on the smithsonite surface and further influenced the grade of Zn ore (Ejtemaei et al., 2011). Tian et al. (2018) also showed that, during the beneficiation of a lithium pegmatite ore, the application of mixed anionic–cationic collectors SX (laurylamine and oxidised paraffin wax soap) and MOD (laurylamine and naphthenic acid soap) could improve the grade and recovery of Li₂O in spodumene concentrates to 6.02% and 87.34%, respectively (Tian et al., 2018).

In this study, we identified a new mixed cationic/anionic collector by combining a quaternary phosphonium salt (tributyltetradecylphosphonium chloride, TTPC) and NaOL for the flotation separation of spodumene from feldspar. Compared with ammonium salt cationic collectors used in similar studies, the substitution of phosphorus atoms for nitrogen atoms in quaternary phosphonium salts greatly enhanced the charge on of reactive groups, which further enhanced their adsorption on the surface of negatively charged silicate minerals (Chen, 2013). The activity of this mixed collector (NaOL/TTPC) and its adsorption mechanism on the surface of minerals are also clarified using new analytical methods. This investigation will help guide the development of flotation separation of pegmatite aluminosilicate ores.

2. Experimental

2.1. Materials and reagents

The spodumene and feldspar samples were supplied from the JiaJika Lithium Mine, Ganzi District of Sichuan Province (China). After hand picking, crushing, grinding, and screening, powder samples with a particle size of -0.074 mm were obtained for the microflotation tests and microcalorimetry studies. The crushing and grinding processes were completed in a TXPE series zirconia clean jaw crusher and a GMS series jar mill (both from Changsha Mitu Instrument and Equipment Co., Ltd, China), respectively. The chemical composition of spodumene and feldspar was measured by chemical analysis. The content of Li₂O, K₂O, and Na₂O in spodumene was measured by flame atomic absorption spectrometric method. The content of SiO₂ was measured by gravimetric molybdenum blue photometric method. The contents of Al₂O₃ and Fe₂O₃ were measured by EDTA (ethylenediamine tetra acetic acid) compleximetric method. The results are shown in Table 1. Analyses show that the purity of the as-prepared spodumene and feldspar powders was more than 90%, and that the as-prepared spodumene contained 7.86% Li₂O. The suppliers and functions of reagents are presented in Table 2. The mixed collector NaOL/TTPC was freshly prepared to minimise the experimental error. Deionised (DI) water with a resistivity of 18.3 M Ω ·cm was used in all the experiments.

Table 1

Chemical compositions of purified samples (in mass fraction, %).

sample	Li ₂ O	Na ₂ O	K ₂ O	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃
Spodumene	7.86	0.15	0.043	62.477	27.434	0.133
Feldspar	–	11.60	0.144	66.432	20.584	0.253

Table 2

Suppliers and functions of the reagents.

Reagent	Supplier	Function
NaOL	Sinopharm Chemical Reagent Co., Ltd	Collector
TTPC	Aladdin Industrial Corporation	Collector
NaOH	Sinopharm Chemical Reagent Co., Ltd	Regulator
HCl	Sinopharm Chemical Reagent Co., Ltd	Regulator
Pyrene	Sinopharm Chemical Reagent Co., Ltd	Fluorescence probe
Ethanol	Sinopharm Chemical Reagent Co., Ltd	Solvent

2.2. Flotation tests

Microflotation tests were carried out on both single and mixed mineral samples, using a flotation machine of XFG type (Changchun, China) with an impeller speed of 1700 rpm. Firstly, 2 g of mineral sample was placed inside a 40-mL flotation cell containing 38 mL DI water. After stirring for 2 min, the pH regulator (NaOH or HCl) was added to the pulp to achieve the desired pH value. Then, the collector (4×10^{-4} , 2×10^{-4} , and 4×10^{-4} mol/L for NaOL, TTPC, and NaOL/TTPC, respectively) was added, and each flotation test was performed for 5 min. Through artificial scraping, filtering, drying, and weighing, the recovery was determined by the weight of the froth products and tails. The mixed ores consisted of 1 g of spodumene and 2 g of feldspar, and the pH value of the minerals mixed NaOL, TTPC, and NaOL/TTPC were 8.5, 6.0, and 4.0, respectively. After applying similar flotation procedures as for single minerals, the froth products and tails were collected for recovery and grade measurement. Each experiment was repeated three times and the average was reported as the final value.

2.3. Fluorescence spectroscopy measurement

The pyrene fluorescence emission spectra of the samples were obtained by using a Hitachi F4500 fluorescence spectrophotometer (Japan). The excitation wavelength of pyrene was 335 nm, and the widths of excitation and emission slits were 6 and 5 nm, respectively.

A saturated pyrene solution was prepared with 0.01 g pure pyrene and 100 mL ethanol (95%) in a volumetric flask. The collector (NaOL, TTPC, or NaOL/TTPC) was freshly prepared in accordance with the microflotation tests. For the fluorescence measurements, 0.5 g of the weighed sample and 0.1 mL pipetted pyrene solution were added into a test tube. Then, the surfactant at diluted concentrations (10^{-2} – 10^{-5} mol/L) was added into each tube to the 25 mL tick mark. Afterwards, the suspension was oscillated for 24 h using a constant temperature (30 °C) oscillator to obtain the fluorescence test samples.

2.4. Microcalorimetry studies

Microcalorimetric measurements of the collector adsorption on the mineral surface were performed at 30 °C in a SETARAM C80 calorimeter (Paris, France), which was capable of maintaining a baseline of ± 0.12 μ W with a temperature stability of ± 0.0001 °C. Before each measurement, 100 mg of the mineral sample and 2.0 mL of collector (4×10^{-4} , 2×10^{-4} , and 4×10^{-4} mol/L for NaOL, TTPC, and NaOL/TTPC, respectively) were put into two compartments of the calorimetric cell that were separated by a circular poly tetra fluoroethylene (PTFE) thin membrane. After complete stabilisation of the calorimeter baseline, the membrane was broken by a movable rod to enable reaction

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