



Adsorption behaviors and mechanisms of dodecyltrimethyl ammonium chloride and cetyltrimethyl ammonium chloride on illite flotation

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ABSTRACT

Collecting performance and adsorption behaviors and mechanisms of two quaternary ammonium salts, dodecyltrimethyl ammonium chloride (DTAC) and cetyltrimethyl ammonium chloride (CTAC), on illite particles have been investigated through flotation tests, adsorption analysis, zeta potential, FTIR measurements and fluorescence spectroscopy. Adsorption analysis revealed that the adsorbed amount of surfactants on mineral surface increases by increasing the pulp pH or by increasing the length of carbon chains of surfactants. Therefore, at same concentrations, CTAC due to its longer carbon chains has a larger adsorption density on illite surface than DTAC, which was measured by FTIR analysis and zeta potential measurements. As compared to DTAC, CTAC showed much better collecting properties as it has higher I_3/I_1 value and a lower critical micelle concentration (CMC) value, which was analyzed by pyrene fluorescence observations. However, decrease in recovery by CTAC in an alkaline solution was mainly due to the complete adsorption of CTAC molecules on mineral particles, hence, its insufficient concentration in the pulp was unable to stabilize the flotation froth. From the results of flotation behaviors and adsorption mechanisms, it is concluded that CTAC is much more efficient surfactant than DTAC in relation to flotation separation of illite.

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1. Introduction

Illite, a non-metallic mica-type clay mineral, has been mined since the Stone Age and now it is among the most important industrial minerals used by many manufacturing industries. As it is mainly enriched with potassium and aluminum, hence natural illite is used for the synthesis of potassic fertilizer through physical and chemical processing. Illite has various superior properties such as excellent heat and noise insulation, chemical stability, natural nanoparticles and good dispersion, making it widely used as filler materials in the rubber, plastic, and cosmetic industries [1,2]. In recent years, application field of illite has widened continuously and the demand of it has grown after realizing its novel properties.

In natural ore deposits, illite commonly occurs as a predominant gangue mineral in diasporite-type bauxite ores. During industrial processing, these gangue minerals are usually floated out to improve the mass ratio of Al_2O_3 to SiO_2 (A/S) and meet the basic requirements of Bayer process (the A/S is higher than 10) [3]. Worldwide, reverse flotation method is considered as a proven and effective technique for obtaining silicate minerals, such as kaolinite, pyrophyllite and illite [4–6]. After electrostatic adsorption of cation surfactants such as alkyl quaternary ammonium salts [7–9], amino amides, polyamine [10] and ether amine [11] on the surfaces of silicate minerals, they can be

effectively floated out [12]. Among these surfactants, quaternary ammonium salts show more and more attention in froth flotation of silicate minerals due to their good characteristics of low toxicity, light irritation and low corrosion. They also have good stability in aqueous solution as most quaternary ammonium salts can be completely ionized. Besides this, the collecting properties of the quaternary ammonium salts are much stronger than other cationic collectors, such as aliphatic primary amine and the aliphatic tertiary amine. Therefore, based on our previous studies [4,13–15], two quaternary ammonium salts with different chain lengths were chosen as collectors in flotation of illite minerals in present study.

Literatures pertaining to cationic surfactants as collectors indicated that the different silicate minerals, due to difference in surface properties, showed different flotation behaviors when cationic surfactants are used as collectors. The adsorption of cationic surfactants on phyllosilicate minerals has been studied and the results suggested that silicates flotation is dependent largely on the adsorption behaviors of these surfactants [13,16]. Studies showed that, during flotation of phyllosilicate minerals, the quaternary ammonium salts mainly contribute to the flotation response from two aspects. First, a portion of the quaternary ammonium salts adsorbed on the mineral surface and rendered particles more hydrophobic. Thus, hydrophobic particles attached to bubbles will float to the top of the flotation cell where they can be separated. Second, the remaining portion in the pulp acts as a frother to stabilize the flotation froth [17]. This means that the adsorbed amount and the residual concentration of collectors (the concentration

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which has not been adsorbed on minerals) are of equal importance in the flotation response. The flotation behaviors of quartz, a typical oxidized mineral, have been investigated by many researchers. Results showed that the flotation recovery of quartz using cationic collectors, such as cetyltrimethylammonium chloride (CTAC), increased with rising pH [18]. However, some phyllosilicate minerals, such as kaolinite, illite and pyrophyllite, exhibited better flotation at lower pH [5,19,20].

To interpret such anomalous behaviors, much attention has been paid to the crystal structure of phyllosilicate minerals [16]. The flotation behaviors and adsorption mechanisms between kaolinite and collectors have been studied. It is found that the anomalous flotation behavior of kaolinite is connected with the crystal texture of kaolinite and its different states of aggregation in acidic and alkaline solutions [20]. However, there is no reasonable explanation for the anomalous flotation behaviors of the other phyllosilicate minerals, such as illite and pyrophyllite. Therefore, the adsorption behaviors and mechanisms deserve further investigations.

In present work, two cationic salts, namely dodecyltrimethyl ammonium chloride (DTAC) and cetyltrimethyl ammonium chloride (CTAC), have been studied as surfactants in illite flotation. Various Flotation tests, adsorption analysis, zeta potential and FTIR measurements were carried out to understand and underlined their collecting performances and adsorption behaviors. Along with this, the crystal structure of illite mineral was studied and is discussed in this paper.

2. Experimental work

2.1. Materials and chemicals

Illite samples used in present investigation were received from Ouhai, Zhejiang province, China. The lump samples were hand-picked, crushed and ground in a laboratory porcelain mill to obtain a maximum amount of sample with size range between 45–75 μm . The cumulative particle size distribution of illite samples is shown in Fig. 1. As noted from distribution of illite samples, the substantial amount of 75–45 μm size fraction was received which accounts for 70%, whereas other fractions with size $-45 \mu\text{m}$ and $+75 \mu\text{m}$ account only 16% and 14%, respectively. The BET surface area of illite was measured as 4.09 m^2/g . Chemical composition and X-ray diffractometry (XRD) were carried out to examine the purity of illite samples. The results given in Table 1 and Fig. 2 show that the purity of samples is approximately 90%, which satisfied the experimental demands in this work.

Dodecyltrimethyl ammonium chloride (DTAC) and cetyltrimethyl ammonium chloride (CTAC) of analytical grade were taken from Nanjing Robiot Co., Ltd. China. Bromothymol blue (BTB) of analytical

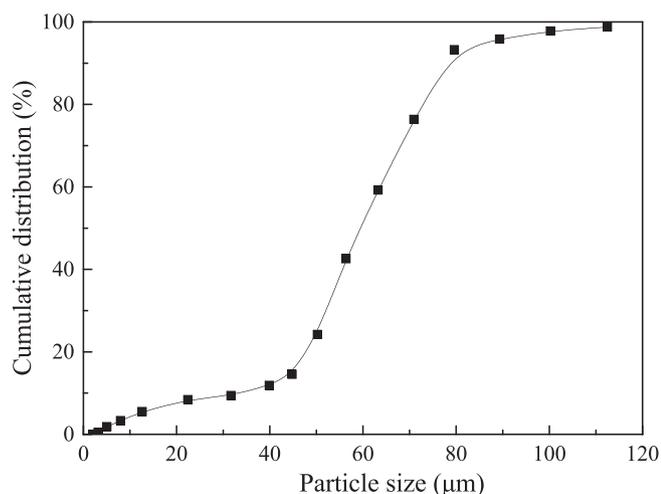


Fig. 1. Cumulative particle size distribution of illite sample used for flotation and adsorption experiments.

Table 1
Chemical composition of the purified illite sample (mass fraction, %).

Al_2O_3	SiO_2	K_2O	NaO_2	TiO_2	Fe_2O_3	CaO_2	MgO
35.54	46.27	10.37	0.81	0.17	0.10	0.06	0.077

grade from Damao Chemical Reagent Factory China was used as an indicator in the adsorption tests. Pyrene from Sigma-Aldrich Trading Co., Ltd., China, was recrystallized twice by ethanol before using. HCl and NaOH of analytical grade from Damao Chemical Reagent Factory China were used to control the pH. It is worth noting that pH regulators were added through glass dropper bottles to eliminate the influence of the volume change of slurry. Different drops of HCl (3%, volume concentration) or NaOH (5%, mass concentration) were added to control the pH to the desired value. Deionized water was used in all tests.

2.2. Flotation tests

Micro-flotation experiments were performed in a 40 mL flotation cell with a fixed impeller speed of 1500 r/min. 2 g of the illite sample with 35 mL of deionized water was used in each test. The pulp was first conditioned for 1 min to uniformly disperse the illite particles. Then, HCl or NaOH was added to adjust the pH to desired values. After that, the pulp was conditioned with the cationic collectors (DTAC and CTAC) for 3 min. The flotation was conducted for 3 min. The froth products and tails were weighed after filtration and drying. The recovery was calculated based on the mass of products. The flotation tests were conducted three times with same experimental conditions and the average was reported as a final value. In flotation, factors and parameters affecting mineral recovery include different kinds of reagents, their concentration, reaction time, pH, inflatable time and temperature etc. It should be kept in mind that even species that exist in small amounts can play a major role in affecting flotation. In this study, pH and the concentration of collector are seen the factors of flotation. Besides, the carbon chain length of collector is also investigated systematically according to factors of adsorption behaviors and mechanisms.

2.3. Zeta potential measurements

Zeta potential measurements were carried out by a Coulter Delsa 440sx Zeta potential analyzer (USA) equipped with a rectangular electrophoresis cell. Mineral samples were ground to $-2 \mu\text{m}$ and the suspension was prepared by adding 40 mg of mineral samples to 80 mL desired solutions containing 1 mmol/L KNO_3 as supporting electrolytes. Then, the suspension was conditioned by a magnetic

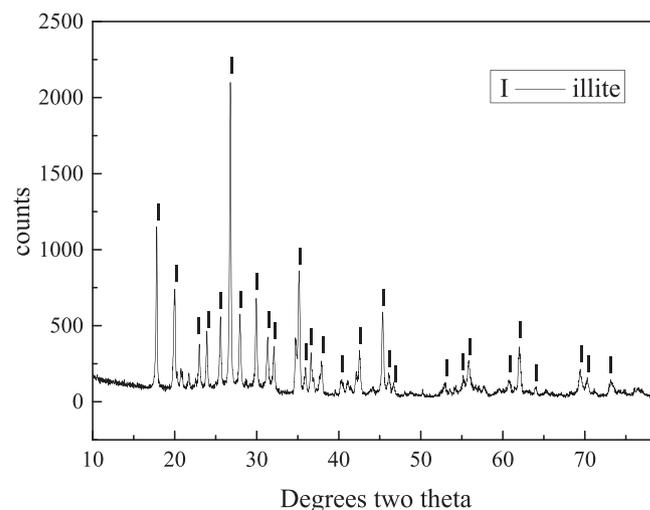


Fig. 2. X-ray diffraction patterns of illite.

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