



Comparative studies of flotation and adsorption with cetyl pyridinium chloride on molybdenite and fluorapatite



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ABSTRACT

In this study, flotation and adsorption of cetyl pyridinium chloride (CPC), a novel cationic collector for molybdenite and fluorapatite, were comparatively investigated by flotation tests, zeta potential measurements, adsorption tests and fluorescence spectroscopic analysis. The flotation results demonstrated a large difference in molybdenite and fluorapatite recovery, from 33.5% at pH 2 to 40.7% at 8, showing a possibility for the separation of molybdenite from fluorapatite in the range of pH 2–8. A higher amount of adsorbed CPC on molybdenite than on fluorapatite was dramatically revealed by Zeta potential measurements as well as adsorption tests, which can explain the better floatability of molybdenite than fluorapatite with CPC as the collector. Adsorption isotherm showed the adsorption of CPC on both minerals are mainly attributed to polar group–surface electrostatic attraction at lower CPC concentration and chain–chain hydrophobic interaction at CPC concentrations above its critical hemimicelle concentration (CHC, 6.47×10^{-4} mol/L). The four region model for adsorbed layer microstructure of CPC on both minerals is also acquired from adsorption isotherm, in addition, further determined from fluorescence emission spectroscopy measurements.

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1. Introduction

Molybdenum, widely used in the fields of energy (Prasad et al., 2008), aerospace (Aouadi et al., 2008), national defense industry (Godbole et al., 2013) and so on, is an important strategic metal. However, molybdenum industry is currently facing a problem of declined reserves for molybdenum sulfide resource, which provides an overwhelming majority of molybdenum before and now (Juneja et al., 1996; Braga et al., 2014). Therefore, the utilization of molybdenum oxide ores becomes more and more important. Generally, leaching of molybdenum from molybdenum oxide ores is primarily conducted and then ammonium molybdate is obtained after the extraction of molybdenum from molybdenum-containing solution (Liu et al., 2011). The major shortcomings of the hydrometallurgical technique are the high reagent dosages (Aylmore, 2001) and energy consumption (Zhao and Stanforth, 2000), which are mainly ascribed to the adsorption and reaction between reagents and a mass of gangue minerals. The preconcentration (Du and Luo, 2013) of molybdenum oxide ores thus makes important sense in both economic and environmental aspects, because most of the gangue minerals are depressed in tailings during preconcentration. It is pretty obvious that the acid leaching of the high grade rough concentrates with few or without acid-consuming minerals

such as calcite and dolomite, will be less reagent consumptive thus more economical.

Molybdenite (MoO_3), belonging to orthorhombic system (Solferino and Anderson, 2012), is one of the molybdenum oxide minerals, of which if we take full advantage, the resource gap of molybdenum supply will be bridged to some extent. However, the natural hydrophilicity of molybdenite surface results in its poor floatability, so that it is difficult to float molybdenite from gangue minerals. Fortunately, the author has found an effectively selective collector, a cationic surfactant CPC, for molybdenite flotation as a preconcentration method of molybdenum oxide ores. The advantages of using cationic collectors in systems where the predominant gangue minerals are fluorapatite, pyrite and quartz have been reported such as good adaptability to pulp chemistry, low dosage, and low water content in concentrate (Gonzalez-Caballero et al., 1989). The interaction between cationic surfactants and silicates has been intensively studied over the years (Xu and Boyd, 1995; Song et al., 2014; Patakfalvi and Dékány, 2001), and many constructive theoretical and practical achievements were obtained. Although the flotation and adsorption of cationic surfactants at the solid (silica, alumina etc.)–liquid interface have been studied in depth, far few studies exist for the case of adsorption of cationic collector on molybdenite.

The objective of this work is to understand the underlying adsorption mechanism and adsorbed layer microstructure of the cationic collector CPC on molybdenite and fluorapatite. Flotation tests, zeta-potential measurements, adsorption and the fluorescence spectroscopic

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tests were carried out. This study focused on elucidating the role of cationic collector CPC on molybdenite flotation and its separation from fluorapatite.

2. Material and methods

2.1. Materials

Molybdenite and fluorapatite were bought from a Mineral Specimens Co., LLC in Beijing. The minerals were crushed by a hammer and grounded in a porcelain mill, the -0.074 mm and -0.038 mm fractions were used for the flotation tests and adsorption tests, respectively. Chemical analysis results showed that the P_2O_5 content is 40.86% for fluorapatite and Mo content is 62.79% for molybdenite, respectively. The artificial mixture mineral was prepared through mixing fluorapatite with molybdenite with the mass ratio of 1:1.

Cetyl pyridinium chloride (CPC) (Fig. 1) of guarantee reagent from Tianjin Damao Chemical Reagent Factory was used as collector. KCl of analytical purity was employed as background electrolyte solution. Pyrene was bought from Sigma-Aldrich (Shanghai) Trading Co., Ltd., China. Pyrene was recrystallized twice by anhydrous ethanol to obtain pyrene crystals of higher purity. Analytical grade sodium hydroxide and hydrochloric acid were used for pH regulation. Double distilled water was used in all tests.

2.2. Flotation tests

Flotation tests were carried out in a micro-flotation cell with an effective volume of 40 ml. For each test, mineral particles prepared (2.0 g) were placed in the cell after their surfaces being cleaned for 5 min using an ultrasonic cleaner to clean the surfaces of particles, and then filled with 35 ml distilled water. After 1 min of agitation, the pH value was adjusted and then the collector was added. The conditioning time was 2 min and 3 min, respectively. The flotation time was 4 min. And the floated and non-floated fractions were filtered, dried and weighed for the recovery calculation.

2.3. Electrokinetic potential measurement

A 0.1 g sample of pure mineral was first grounded to -2 μm , and then transferred into a 100 ml breaker containing 50 ml of 1×10^{-3} mol/L KCl. The conditioning procedure is the same as those aforesaid in flotation tests. The zeta-potentials were measured using a Malvern Zetasizer Nano ZS90 (England) equipped with a rectangular electrophoresis cell. The conductivity and pH of the suspension were monitored continuously during the measurement and the environmental temperature was maintained at 25.0 ± 0.5 °C.

2.4. Adsorption tests

By using an ultra-violet (UV-3100) spectrometer to measure the absorbance of CPC at a wave length of 259 nm, the adsorbed amount

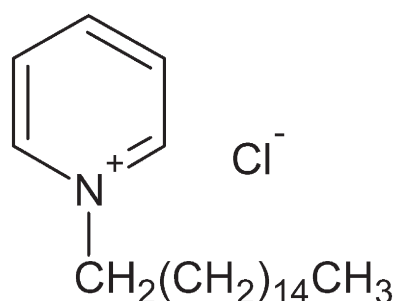


Fig. 1. The molecular structure of CPC.

of CPC on minerals was determined over the pH range 7 ± 0.5 . Several 0.5 g of -0.038 mm sample was pulped with 40 mL of distilled water in an appropriate volume of dry volumetric flasks, to which a known amount of desired reagents had been added and then, the suspension was agitated for 1.5 h using a stirrer, followed by centrifugation at 4500 rpm for 15 min using a high speed refrigerated centrifuge. The suspensions were then filtered and then the treated solution was measured by UV spectrometer to get its absorbance. Finally the residual CPC concentration was obtained according Absorbance–CPC concentration standard curve (Fig. 2). The amount of CPC adsorbed on a mineral was calculated from the difference between the initial and residual CPC concentrations in the suspension/solution using the following equation (Beaussart et al., 2009):

$$\Gamma = (C_0 - C) \cdot V / (1000 \cdot m). \quad (1)$$

Where C_0 and C are the initial and residual concentrations (mol/L), respectively, V is the solution volume (mL), and m is the weight of the particles per sample (g).

2.5. Fluorescence emission spectroscopy

The pyrene stock solution was first prepared by dissolving pyrene in hot water until saturation, then cooled to 25 °C, and filtered. The concentration of pyrene in the solution was determined to be 6.53×10^{-7} mol/L. And then the samples for fluorescence emission spectroscopy measurements were prepared through mixing a pyrene stock solution with CPC and mineral pulp, and allowing it to stand for 1.5 h to equilibrate. The pyrene steady-state emission spectra were obtained by using a Hitachi F-4500 fluorescence spectrophotometer. The excitation wave length of pyrene was 335 nm.

3. Experimental results and discussion

3.1. Flotation

Fig. 3 shows the recoveries of molybdenite and fluorapatite with CPC (4×10^{-4} mol/L) as the collector. Over the entire pH range tested, molybdenite recovery is more than 70%, while the recovery of fluorapatite varies widely from 36.8% to 73.3%. A dramatic increase in fluorapatite recovery is observed at $\text{pH} > 8$. As we can see, in weakly alkaline, neutral and acidic pH range, there are great differences in the recovery between the two minerals, from 33.5% at pH 2 to 40.7% at 8, which is enough for the flotation separation of molybdenite from fluorapatite. That is, molybdenite can selectively be floated from fluorapatite with CPC as the collector in this pH range.

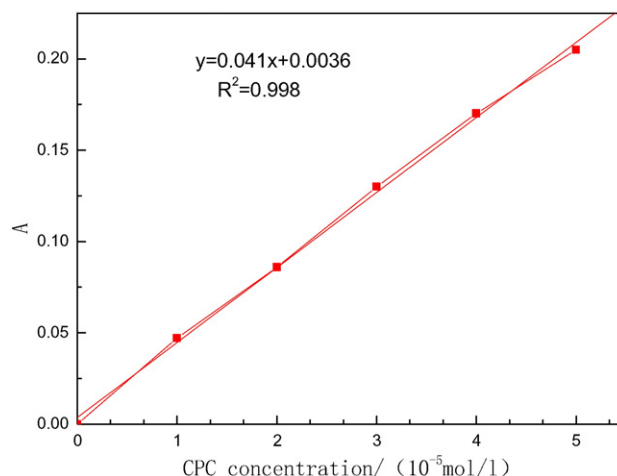


Fig. 2. Absorbance-concentration standard curve of CPC.

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