



# Flotation separation of quartz from collophane using an amine collector and its adsorption mechanisms



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## ARTICLE INFO

### Article history:

Received 18 January 2017

Received in revised form 12 May 2017

Accepted 1 June 2017

Available online 02 June 2017

### Keywords:

Quartz

Collophane

Flotation

Amine collector

Adsorption mechanism

## ABSTRACT

Quartz is the dominant gangue mineral in siliceous phosphate ore. To obtain efficient concentration of phosphate minerals, the flotation separation of quartz and collophane using AY as a collector was studied in this paper. The adsorption mechanism of the collector on the surface of quartz and collophane were investigated by measuring zeta potential, contact angle, adsorption density, surface tension and analyzing FTIR spectra. It reveals that AY displays a stronger collecting ability for quartz relative to collophane at pulp pH 7, resulting in a separation window for the two minerals. When the dosage of collector was 50 mg/L, phosphate minerals could be concentrated from 23.96% to 30.18% P<sub>2</sub>O<sub>5</sub> with a great recovery of 98.24%. Mechanism studies show that the interaction of the amine collector with quartz and with collophane is mainly through electrostatic attraction, whereas the cationic collector prefers adsorbing on the negatively charged quartz. This is also supported by measurement of minerals contact angle and collector's adsorption density. AY as a collector has a selective absorption on quartz rather than collophane.

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## 1. Introduction

Phosphate ore is a vital non-renewable resource, and so far there is no substitute for phosphate ore as the main raw material in the production of phosphatic fertilizers and phosphorus-based chemicals [1–2]. About 85% of the total phosphate ore that reserves in China can be classified as low to middle grade ore due to containing a considerable amount of gangue minerals. It generally has 12% to 25% P<sub>2</sub>O<sub>5</sub> [3], the processing thus requires very intensive separation of phosphate minerals and gangue minerals [4]. When the type of phosphate ore is siliceous, quartz is the dominant gangue mineral. The separation of quartz and phosphate ore is difficult as their co-existence is very complicated [5].

Flotation is the most widely used treatment for the concentration of these siliceous phosphate ore [6]. In flotation separation, the choice of collectors plays an important role [7]. When the cationic collectors are used, phosphate ore would experience a reverse flotation, in which quartz attaching to bubbles a collected while the most valuable mineral stays in the sink like the tailings [8].

Many researchers studied the reagents toward enhancing flotation performance of quartz. Such as collector of  $\alpha$ -Bromodecanoic acid was

reported as a potential one for flotation of quartz, the quartz recovery could be 99.5% when pulp pH was higher than 11.5 [9]. Similarly, collector DLG-2 was reported that it can recover 99.5% of quartz at pH 10 [10]. Flotation selectivity for quartz against collophane has not been further verified though. It is this kind of ore that requires the use of flotation reagents that has disparity to quartz and collophane. Currently, when referring to flotation selectivity, most topics will be about quartz from hematite. For example, researchers made functional amine surfactants to benefit quartz recovery from hematite [11–15]. Liu et al. [13] reported that the collecting performance of *N*-dodecyl ethylenediamine for flotation of quartz from hematite could be enhanced by adding butanol. A few studies reported the flotation performance of cationic collector to separate quartz from phosphate ore. Hanna [16] found that in comparison to cetyltrimethyl ammonium bromide (CTAB) quartz is readily floated by quaternary ammonium salts and primary amine. Salah et al. [17] conducted a comparison study to anionic and cationic collectors in flotation of a siliceous phosphate rock, the results show that amine collectors obtained a better metallurgical performance relative to sodium oleate.

The aim of this work is to investigate the flotation behaviors and adsorption mechanisms of quartz and collophane by using an amine collector AY. The collecting capacity and selectivity of the collector were evaluated by micro-flotation of pure mineral and artificially mixed minerals. Furthermore, the adsorption mechanisms of the collector on quartz

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and collophane were systematically investigated by measuring zeta potential, adsorption density, contact angle and surface tension plus the FTIR spectra analysis.

## 2. Experimental

### 2.1. Minerals and reagents

The hand-picked samples of pure quartz and collophane were obtained from a phosphate mine in Guizhou province, China. The mineral samples were crushed and ground in an agate mortar and then screened to obtain fine particles with a size fraction of 45 to 75  $\mu\text{m}$ . This ground sample is used for micro-flotation and adsorption experiments. The X-ray diffraction (XRD) (X'Pert PRO, PANalytical Company, Netherlands) analysis (Fig. 1) and X-ray fluorescence (XRF) (Axios mA, PANalytical Company, Netherlands) data reveal that the purity of quartz is 99.04% and the purity of collophane is 94.51%. The specific surface areas (BET) of quartz and collophane are 0.621  $\text{m}^2/\text{g}$  and 0.783  $\text{m}^2/\text{g}$ , respectively, which was measured by instrument of F-Sorb 3400, Gold APP, China. The artificially mixed minerals were prepared by mixing quartz and collophane samples at a mass ratio of 2:3, of which the  $\text{P}_2\text{O}_5$  content is 23.96%.

The cationic collector AY which is a mixture of various amines was supplied by Guizhou Key Lab of Comprehensive Utilization of Non-metallic Mineral Resources, China. The collector solution was prepared by mixing equimolar mixtures of AY and hydrochloric acid (HCl), and it was used as a 1 wt% solution in all the flotation experiments. HCl with an analytical purity of 37% was purchased from Chongqing Chuandong Chemical (Group) Co., Ltd. (Chongqing, China); sodium hydroxide (NaOH) with a purity of 96% was obtained from Tianjin Kemiou Chemical Reagent Co., Ltd. (Tianjin, China). Solutions of HCl and NaOH were used to adjust the pH. The distilled water was used for all the experiments.

### 2.2. Methods

#### 2.2.1. Micro-flotation tests

Micro-flotation tests on pure minerals and artificially mixed minerals were carried out as a function of the collector concentration and pulp pH in a XFGCII flotation machine (mechanical agitation) with a volume capacity of 40 mL. Firstly, 2.0 g of mineral samples were placed in a plexiglass cell in which 40 mL distilled water were filled. After 3 min agitation at 1992 rpm, HCl or NaOH solution was added to adjust the pH of slurry. The collector was added into the cell with 3 min conditioning time, and then flotation was conducted for another 3 min. The froth and sink products were separately filtered, dried, weighed and assayed. Data from assay is to calculate the flotation recovery.

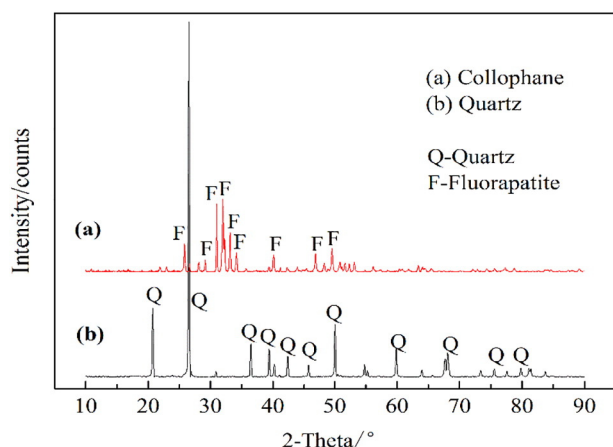


Fig. 1. X-ray diffraction patterns of collophane (a) and quartz (b).

#### 2.2.2. Adsorption density measurements

Collector adsorption on the mineral surface was measured by the solution depletion using an automated total organic carbon (TOC) analyzer (TOC-LCPH, Shimadzu, Japan). When there is only AY in the solution, TOC concentration and the real AY concentration shows a perfect linear correlation. The linear correlation coefficient  $R^2$  is 0.999. The AY concentration of the sample solution was determined by comparing the TOC concentration to the standard curve. Placed 2.0 g of pure mineral sample in a 100 mL conical flask, then added 40 mL of the collector solution. After that the pH of suspension was adjusted by HCl or NaOH solution. Solid and liquid adsorption was conducted for 30 min at 200 rpm on a thermostated shaker (TS-100C, China). Completing adsorption, the solid particles were separated by a centrifuge for 15 min. The concentration of the collector in the supernatant was analyzed by TOC analyzer. The adsorption density of the collector on mineral surface was calculated using Eq. (1).

$$\Gamma = \frac{(C_i - C)V}{mA} \quad (1)$$

where  $C_i$  and  $C$  are the initial and supernatant concentrations (mg/L), respectively,  $V$  is the solution volume (L),  $m$  is the amount of the pure mineral (g), and  $A$  is the particle specific surface area ( $\text{m}^2/\text{g}$ ).

#### 2.2.3. Zeta potential measurements

Zeta potential measurements were conducted with zeta potential analyzer (Delsa™ Nano C, Beckman Coulter, USA). The mineral samples for flotation were further ground to 5  $\mu\text{m}$  in an agate mortar. A suspension containing 0.1 wt% of the solid content in the  $1 \times 10^{-3}$  mol/L  $\text{KNO}_3$  background electrolyte solution was agitated for 15 min with a magnetic stirrer so that the mineral particles could be fully dispersed. The pH value of the suspension was adjusted to a desired value using HCl or NaOH solution. Measurements were performed in the absence and the presence of the collector (50 mg/L).

#### 2.2.4. Contact angle measurements

Experiments on the contact angles of quartz and collophane before and after treatment with the collector were carried out using the Washburn method [18] with the contact angle measurement instrument (XG-CAME, China). The cleaned mineral samples were immersed in distilled water and the collector solution at different pH values for 10 min, the collector concentration was 200 mg/L, and then the suspension was filtered, wet cake was dried at room temperature. Finally, the contact angle measurements were performed.

#### 2.2.5. FTIR spectra analysis

The mineral samples of pure quartz and collophane were further ground to  $-5 \mu\text{m}$  in an agate mortar. Then, 2.0 g of  $-5 \mu\text{m}$  mineral samples were added to 40 mL aqueous solution which was prepared initially with or without 50 mg/L collector at pH 7. After 10 min conditioning, the mineral samples were filtered, thoroughly rinsed with distilled water and dried in a vacuum oven at 40  $^\circ\text{C}$  for 12 h. Then the mineral samples were mixed with KBr and the spectra were recorded in the range of 4000–400  $\text{cm}^{-1}$  at room temperature.

#### 2.2.6. Surface tension measurements

The surface tension of flotation solution was measured by an automatic tension apparatus (JK99D, China). 2.0 g of  $-75 + 45 \mu\text{m}$  size fractions samples were added to 40 mL aqueous solution with different collector concentrations. Solution of HCl or NaOH was used to adjust the pH value of the suspension. Surface tension of flotation solution was measured following 10 min conditioning. The platinum plate was burned after washing in alcohol flame to completely remove the adsorbed surfactants before each measurement. Then, it was dipped in the solution to measure its surface tension.

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