



A mixed collector system for phosphate flotation



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ABSTRACT

Flotation has been used in industry for more than a half century as the primary technique for upgrading phosphate. While the flotation of phosphate was inefficient when oleic acid was used alone as a collector, therefore a mixed collector of oleic acid (HOI), linoleic acid (LA) and linolenic acid (LNA) was employed to improve the recovery of phosphate flotation. The batch flotation results showed that the optimal composition of the mixed collector was 54 wt.% HOI, 36 wt.% LA and 10 wt.% LNA. Additionally, the effect of pH on the mixed collector application was studied while considering the surface tension, contact angle and micro-flotation. The results showed that the mixed collector should be used at a pH of 9.5. Above a pH of 9.5, the adsorption of fatty acids dimers on the apatite surface hindered phosphate flotation. The influence of the mixed collector assembly on apatite flotation was also investigated. It was demonstrated that due to its low critical micelle concentration, a sufficiently hydrophobic apatite surface could be generated at a collector concentration of 60 mg/L. In addition, zeta potential experiments suggested that collector adsorption was governed by chemisorption. FTIR and XPS spectra studies further indicated that the chemical reaction involved the carboxyl groups of fatty acids and Ca species at the apatite surface for each fatty acid in the mixed collector.

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1. Introduction

Phosphorous is one of the most common elements on Earth and is essential for life in all organisms (Sharpley et al., 1997). Due to its high reactivity, phosphorus is primarily found in its oxidized state (i.e. phosphate) in nature. Phosphates form the backbone of adenosine triphosphate, which stores the chemical energy used in biological systems, and they are also the primary components of the bones and teeth in humans (Arnold and Gaengler, 2007; Helbig et al., 1998a). In its synthetic form, phosphorous is used as a fertilizer and is one of the three primary nutrients for plants.

Phosphate ore is the primary natural resource from which phosphorous is extracted. While apatite is the most prevalent phosphate, other gauge minerals commonly found in phosphate ore are dolomite, calcite and silicates. Increasing global demand for food has increased consumption of phosphate ore to an alarming rate over the past few decades; the year of 2005 boasted a world phosphate production of 148 Mt, which was more than twice the production of 1965 (Herring and Fantel, 1993; Van Vuuren et al., 2010). The USA is the largest phosphate producer and produced approximately one third of the world's 35.8 Mt of phosphate in

2002 (USGS, 2003). It was also estimated that over 70% of the production in the USA occurred at plants in Florida (Pittman, 1990).

To meet the requirement of phosphoric acid production, phosphate ore should be enriched to near 30% P₂O₅ (Abouzeid et al., 2009). Generally, natural phosphate ores fail to meet this market grade and must be upgraded with further processing. For certain high-grade phosphate ores, a satisfactory concentration could be obtained simply using gravity separation methods (Abouzeid, 2008). With the depletion of high-grade phosphate ore, low-grade phosphate ore with more impurities is becoming the primary resource for phosphate; thus, more efficient methods are required for upgrading. Flotation, which uses fatty acids as collectors, is the predominant method for the processing of low-grade ore. Today, it is estimated that more than half of the marketable phosphate in the world undergoes flotation techniques (Sis and Chander, 2003c).

Despite successful and economic recovery of phosphate via flotation, its efficiency using fatty acids as collectors is still not satisfactory. Oleic acid is the most common and traditionally used collector for apatite flotation. One concern in the use of oleic acid is its tendency to adsorb onto a slime surface. A large slime surface area also favours the adsorption of oleic acid (Hernández et al., 2004) and thus reduces the flotation selectivity. A significant amount of calcium ions introduced into the pulp by the dissolution of Ca-bearing minerals and from recycle water (Zhang et al., 1999)

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can react with the oleic acid and precipitate out calcium dioleate salt, leading to high consumption of the collector. Additionally, the use of oleic acid requires high temperatures, resulting in high processing cost (Ge et al., 2008; Sis and Chander, 2003a).

To mitigate the disadvantages of oleic acid mentioned above, many new collectors have been developed. It was reported that a hydroxamic acid–alcohol collector was an efficient and selective collector for phosphate flotation and was suitable for coarse feed (Miller et al., 2002; Wang et al., 2006). Amino acids could also be used for apatite flotation (Hirva and Gordejiev, 1999). The *ab initio* simulation study revealed that sarcosine anion could bind tridentately or bidentately with Ca^{2-} (Hirva and Gordejiev, 1999). It was also demonstrated that sarcosine anion could selectively adsorb onto an apatite surface with a bidentate binding but not onto calcite and dolomite surfaces (Hirva and Gordejiev, 1999; Hirva and Tikka, 2002). Though the newly developed collectors showed improved performance for phosphate flotation but have been found limited use in industry due to their high cost and complex processes required for synthesis.

Another approach to enhance the performance of oleic acid is using synergic reagents. Synergists could improve the adsorption of oleate ions at the liquid/air interface and to the phosphate surface (Sis and Chander, 2003a,b). In addition, synergists help to prevent the detrimental effects of slime and calcium ions on collector efficiency (Helbig et al., 1998b). Ethoxylated nonylphenol with four oxyethyl groups (NP-4) is a typical non-ionic synergist. It was reported that the critical micelle concentration (CMC) of a 2:1 mixture of oleic acid and NP-4 (50 mg/L) was significantly below the CMC of pure oleic acid (500 mg/L) (Sis and Chander, 2003b). The contact angle of apatite also improved 6–7° using a 2:1 mixture of oleic acid and NP-4 (Sis and Chander, 2003a).

In phosphate plants today, vegetable oils are the dominant collectors that are replacing oleic acid due to their low costs and high efficiencies at normal temperature. Vegetable oils, including corn oil, soybean oil and rice bran oil, have been successfully used as collectors for apatite flotation in Brazil (Guimaraes et al., 2005). These vegetable oils are primarily composed of oleic acid (HOI), linoleic acid (LA) and linolenic acid (LNA), which are not stable even if they are derived from the same vegetable. The content change of each component can affect the flotation property of the vegetable oil; however, the influence of changes in the composition of the vegetable oils on phosphate flotation is still unknown. Guimaraes et al. reported that LA was as effective as HOI, and a high LA fraction in the mixed collector was not necessary for apatite flotation (Guimaraes et al., 2005). Conversely, few reports currently exist in the literature about phosphate flotation with the LNA collector.

Thus, the optimal composition of a mixed fatty acid collector must be determined for phosphate flotation. In addition, the flotation behaviour of this type of mixed collector also must be investigated in detail. Therefore, HOI, LA and LNA were selected as components for a mixed collector in this study. The suitable fraction for each component was first determined directly with the flotation tests using run-of-mine phosphate ore. Fundamental surface chemistry experiments were conducted to reveal the adsorption state of the mixed collector on the apatite surface. The adsorption mechanism for the mixed collector was also determined using FTIR and X-ray photoelectron spectroscopy (XPS).

2. Experimental

2.1. Reagents and materials

AR-grade oleic acid (HOI), linoleic acid (LA) and linolenic acid (LNA) were purchased from TCI of Japan. The vegetable oils used

were all provided by the Institute of Multipurpose Utilization of Mineral Resources, China.

The phosphate ore sample for the batch flotation tests was obtained from the Wenfu Phosphate Company located in Guizhou Province, China. A chemical composition analysis showed that the ore contained 25.50% P_2O_5 , 18.98% of acid insoluble (A.I) and 6.83% of loss on ignition (L.O.I), as shown in Table 1. XRD and MLA analyses indicated phosphate existed in the form of fluorapatite, and primary gauge minerals were hydromica, quartz and dolomite in the ore (Table 2).

The apatite ($\text{Ca}_5(\text{PO}_4)_3\text{F}$) pebbles with a purity of 98%, which was confirmed by XRD analysis, were used for the surface property analysis. The apatite pebbles were pre-treated in an ultrasonic cleaner for 5 min and dried before the surface tests began. All experiments were conducted at 23 °C. Tap water was used in all batch flotation tests, and deionized water (DI water) was used for the pure apatite study.

2.2. Batch flotation test

A 1.5 L flotation cell was prepared for the flotation tests. For each test, 500 g of phosphate ore was wet ground to 80 wt.% passing 200 mesh with a ball mill. The phosphate pulp was transferred into the flotation cell and stirred for 1 min. 6 kg/t of sodium carbonate was added into the pulp to achieve a pH of 9.5 with 3 min of conditioning time. Next, 3 kg/t of soluble glass was added, and the solution was conditioned for another 3 min. All types of collectors were added with the same dosage of 600 g/t, and the slurry was stirred for 3 min. Before froth collection, 20 g/t of pine oil was added with 1 min of conditioning time. All conditioning processes were conducted in the absence of airflow. After completion of the flotation test, the float and sink products were filtered, dried, weighed and analysed. In addition, the fatty acids were mixed with different weight fractions and mass ratios to obtain various mixed collectors.

2.3. Surface tension measurement

The Du Noüy ring method was used to measure the surface tension of the collector solutions. In the solution preparation, the glassware was cleaned with chromic acid and rinsed with ample DI water. The platinum ring first was flushed with acetone, followed by methanol and DI water. After removing the remaining water, the ring was flame treated to remove any organic contamination. Before each measurement, the surface tension of the DI water was tested as a control. In each test, a 20 mL sample of solution was used. The pH was maintained at a desired value with dilute NaOH and HCl solutions.

2.4. Contact angle measurement

The contact angle on the apatite surface was measured with the sessile drop method using a GBX 3S tensiometer. An apatite crystal was dry polished first with 500 grit and then 1000 grit and 4000 grit Al_2O_3 sandpapers. The crystal was then air cleaned and plasma treated for 5 min to obtain a fresh surface. The crystal was conditioned with the collector solution at the desired concentration and pH for 15 min and then air dried. A DI water droplet (2–3 mm) was then introduced onto the apatite surface. After the three-phase contact line was observed, the syringe was moved upward, and the shape of the free droplet was recorded by a CCD camera system. The image of the droplet was analysed with the software to obtain a contact angle value.

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