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Importance of collector chain length in flotation of fine particles

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ABSTRACT

Flotation of fine ore is usually carried out with collectors that adsorb specifically, such as the derivatives of carboxylic acids in phosphate processing plants. Industrial flotation processes that use conventional cationic collectors, generally, have a stage of desliming to discharge the fines, as in reverse flotation of iron ore in Brazil. This study aimed to understand why the fines respond well to flotation with oleate (carboxylic collector), but do not have the same performance with usual cationic collectors such as dodecylamine (DDA).

The flotation tests were carried out in a modified Hallimond Tube using a pure dolomite sample. DDA, a cationic collector which adsorbs through electrostatic interaction (physical and non-specific adsorption), and sodium oleate (SO), an anionic collector which adsorbs via chemisorption (specific adsorption) were used as flotation collectors. The effect of adding a chain extender was tested and the influence of the size of the amine chain in the floatability of the fines was also observed.

The initial results showed that flotation with DDA is strongly affected by particle size. The floatability decreases from 82.6% to 52.6% when d_{50} decreases from 74 µm to 14 µm. In turn, particle size variation had little effect on the floatability with oleate. The better performance of the fines flotation with oleate was attributed to the hydrophobic aggregation during conditioning which increases the d_{50} by 43%. The floatability of fines increases with the hydrophobic aggregate size which, in turn, increases with the chain length of the collector. This explains the better performance of the oleate (17C), if compared with DDA (12C), and suggests the use of long chain collectors for the floatabil of fines.

1. Introduction

Flotation is the result of three key steps involving solid (mineral), liquid (water) and gas (air): (1) collision of mineral particles with air bubbles; (2) adhesion or formation of air bubble-particle aggregate, and (3) transport of the aggregate to the surface of the liquid, where the particles are collected.

The flotation of particles about 15 μ m, or smaller, is not an easy task. The problem is related to inherent characteristics of the fine particles such as small mass and high surface area, that impair the steps of collision and adhesion (King, 1982; Sivamohan, 1990; Miettinen et al., 2010). Due to the low momentum the fines tend to follow the liquid streamlines around the bubbles avoiding the collision (Gontijo et al., 2007). In turn, the probability of adhesion and formation of bubble-particle aggregate depends fundamentally on the surface hydrophobicity, which is usually measured in terms of contact angle (Crawford and Ralston, 1988; Muganda et al., 2011). The degree of hydrophobicity depends on the adsorption density of the collector on the surface (Muganda et al., 2011). Trahar (1981) and Crawford and

Ralston (1988) showed that flotation of a particle is made possible only from a certain minimum contact angle. The higher the surface area of the fines, the lower is the density of collector adsorption and smaller is the contact angle. So, the flotation of fines is characterized by a low speed which results in low recovery.

The flotation of fines seems to be even more complicated when using conventional cationic collectors that adsorb through electrostatic interactions. This fact has been attributed to the high surface energy of the fines, due to higher proportion in edges and corners, besides cracks and other lattice imperfections arising from size reduction. The high surface energy contributes to a non-selective adsorption of he collector (Trahar and Warren, 1976). Fuerstenau (1980) and Trahar (1981) suggested the use of chemisorbing collectors to minimize this problem, since they promote a more selective adsorption.

In fatty amines, used in cationic flotation, the hydrocarbon radical is saturated and contains no heteroatoms. The chain has 8–12 carbons. A smaller chain does not impart the required hydrophobicity to the surface, whereas a higher chain presents solubility problems. Amines with 12 carbons chain (dodecylamine) are the most commonly used. The

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functional group (NH_2) is the part of the molecule common to all amines used in flotation. In this group, the nitrogen presents a pair of free electrons that determines the basic character of the amines.

The amines ionize by protonation according to the reaction: $\text{RNH}_2 + \text{H}_2\text{O} \leftrightarrow \text{RNH}_3^+ + \text{OH}^-$. In an amine aqueous solution there are the ionic specie (RNH_3^+), the molecular specie (RNH_2), the dimers ($\text{RNH}_2\text{RNH}_3^+$) and (RNH_3)₂²⁺, besides the insoluble species in the form of colloidal precipitate (Gao et al., 2015; Liu et al., 2015). The distribution of the species depends on the pH and the total concentration of the amine. The amines have a limited solubility in water. Due to this, they are usually partially neutralized and used in the form of soluble salts (Araújo et al., 2005). Acetic acid (CH₃COOH) and hydrochloric acid (HCl) are the most used to neutralize the organic base. Until the formation of the monolayer, the main adsorption mechanism is based on the electrostatic interaction between the cationic species (RNH₃⁺) and the negative sites of the mineral surface. Therefore, it is a physical nature adsorption.

In turn, carboxylic collectors are characterized by the carboxyl functional group (COOH) having the general formula RCOOH. The "R" represents a hydrocarbon radical, responsible for the hydrophobization induced of the mineral surface. The active carboxyl group has hydrophilic and solidophilic characteristics. So, it is responsible for collector solubility and adsorption on the mineral surface. The carboxylic acids ionize according to the reaction: $RCOOH \leftrightarrow RCOO^- + H^+$. The oleic acid with 17 carbons is the most commonly used collector of this family. In order to minimize the problem of the low solubility of the long chain collector, an unsaturated acid (oleic acid) in the form of salt (sodium oleate) is usually used. Saponification is obtained by reaction with a base generally sodium hydroxide: $RCOOH + NaOH \leftrightarrow$ RCOONa + H_2O . The aqueous solution of a carboxylic collector may contain the ionic species of carboxylate (RCOO⁻), molecular fatty acid (RCOOH), and those resulting from chain-chain associative interactions: the molecular-ion dimers (RCOOH.RCOO⁻) and ion-ion $(\text{RCOO})_2^{2-}$ (Pugh and Stenius, 1985). The concentration of each species depends on the pH and the total concentration of the reagent in solution (Yu et al., 2015, Atrafi and Pawlik, 2016). In alkaline media, where the ionic form (RCOO⁻) predominates, the carboxylic collectors are adsorbed by chemisorption, with the formation of a hydrophobic compound on the mineral surface. This mechanism prevails until the formation of the monolayer has been completed. Carboxylic collectors have high affinity for alkaline earths $(Ca^{2+}, Mg^{2+}, Ba^{2+} and Sr_2^+)$ with which form very stable compounds.

Therefore, while the dodecylamine has a smaller chain and adsorbs by a physical mechanism, the oleate has a long chain and a chemical adsorption based mechanism (Fig. 1).

In commercial production, the flotation of fines is usually performed with collectors that adsorb specifically, like in phosphate processing plants that use collectors derived from carboxylic acids (Guimarães et al., 2005). On the other hand, the processes that use conventional cationic collectors generally involve a desliming step to remove the fines (Colombo and Frommer, 1976; Filippov et al., 2010; Filippov et al., 2014), such as in the reverse flotation of iron ore in Brazil (Araújo et al., 2005). Houot (1983) reports processes of iron ore flotation without desliming using fatty acid and with desliming using an etheramine.

However, despite the evidences related to the advantage of using collectors that adsorb specifically (as SO) for fines flotation, the factors identified as responsible for the low efficiency of flotation of fine do not depend on the active group and the nature of the collector adsorption. Therefore, it does not explain the difficulty for flotation of fines with DDA.

The research aimed at contributing to understand why carboxylic collectors, such as SO, are usually more effective in flotation of fine particles than conventional cationic collectors (such as DDA). The dolomite was chosen for this study since it responds well to flotation with both collectors.

2. Experimental

2.1. Sample

A pure dolomite sample from *Pedra Lavrada* (State of Paraíba, Brazil) was used. Initially, the sample was ground in a porcelain ball mill and wet sieved to produce four size fractions. Then, each fraction was passed through a high intensity magnetic separator to remove contaminants.

2.2. Characterization

The purity of each sample fraction used in the flotation tests was checked by X-ray diffraction analysis (XRD) using a BRUKER diffractometer the D2 PHASER model, Cu- α radiation.

The average diameter of each fraction and the surface area were determined in a laser particle size analyzer Malvern, model Mastersizer 2000.

2.3. Flotation

The micro-flotation tests were performed in a modified Hallimond tube with 3.5 cm diameter and volume of 400 mL. An extender 20 cm high column was introduced in order to reduce entrainment. The tests were carried out with 2 g of sample conditioned for five minutes (oleate tests) or 1 min (amine tests) before introducing the nitrogen gas. The flotation time was 2 min, while the gas-flow rate was maintained at

COLLECTOR	CHAIN LENGTH	CHEMICAL STRUCTURE	ADSORPTION MECHANISM
Dodecilamine	12 C	Н Н Н Н Н Н Н Н Н Н Н Н Н 	PHYSICAL
Oleate	17 C	Н Н Н Н Н Н Н Н Н Н Н Н Н Н Н Н Н Н 	CHEMICAL

Fig. 1. Characteristics of the collectors used in the fine dolomite flotation.

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