Minerals Engineering 112 (2017) 27–35

Contents lists available at ScienceDirect

Minerals Engineering

journal homepage: www.elsevier.com/locate/mineng

Effect of energy input on flocculation process and flotation performance of fine scheelite using sodium oleate

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A R T I C L E I N F O

Keywords:
Energy input
Fine scheelite
Flotation rate
Shear flocculation
Sodium oleate

A B S T R A C T

We have investigated the effect of energy input on the flocculation process and flotation behavior of fine scheelite of less than 10 μm size. Sodium oleate was used as a dual-function reagent, acting as flocculant and collector. Energy input in shear flocculation was controlled in a four-baffle cylindrical tank with a four-blade impeller by changing the agitation speed. The flocculation process was investigated by measuring continuous transformations in size distribution and observing floc shape. The results show that with increasing energy input, the size distribution of fine scheelite transforms from unimodal to bimodal. The flocs produced tend to possess more branches with low energy input and tends to become globule-like with high energy input. A parameter termed the flocculation degree was introduced to quantify the flocculation process as a function of energy input. The flocculation degree with increasing energy input reveals the aggregation order of different size fractions (all less than 10 μm) when forming flocs. The flotation rate of flocs formed with different energy input was studied. The results demonstrate that the flotation rate is closely related to energy input and also, exhibits an intimate correlation with flocculation degree. These results could potentially be used to routinely monitor the flotation performance of fine particles in operating plants when shear flocculation is used.

1. Introduction

Scheelite is the principle mineral of tungsten and is usually recovered by froth flotation. Froth flotation is a physico-chemical separation process that utilizes the difference in surface properties of the valuable minerals and the unwanted gangue minerals (Wills and Napier-Munn, 2005). Due to the brittle nature of scheelite and/or mineral liberation of low grade, finely disseminated scheelite ore, slimes (particles less than 10 μm) containing high grade of scheelite are often generated in the size reduction process. The fine scheelite in flotation feeds is difficult to recover and the loss of fine scheelite is generally regarded as a main reason for low flotation efficiency in flotation plants (Sivamohan, 1990; Ralston, 1992). It is reported that more than 1/5 of total tungsten in flotation feeds is presented as slimes (Gaudin, 1995). Therefore, promoting the recovery of fine scheelite is of great importance.

Common collectors for scheelite, such as fatty acids and their derivatives, have been widely demonstrated to be adsorbed onto scheelite through chemisorption, and this adsorption makes its surface hydrophobic (Atademir et al., 1981; Rao and Forssberg, 1991). The apparent size of particles is a significant factor during the process, as the adsorption exploits the interaction between collector and exposed particle surface. Fine particle characteristics, such as large specific surface area, often leads to high reagent consumption and low flotation rate in flotation (Sivamohan, 1990). Calculations on efficiencies of particle–-bubble collision have demonstrated that the low flotation rate of fine particles are attributed to the low efficiency of collision between fine particles and gas bubbles in flotation cells (Pyke et al., 2003). Increasing the apparent size of fine particles can improve the efficiency. The size increasing process is known as flocculation, coagulation, or aggregation and was firstly introduced in pre-treatment of fine scheelite flotation as "shear flocculation" in 1975 (Warren, 1975).

Shear flocculation is the process where charged hydrophobic fine particles are aggregated under high shear conditions. It has been proven that surface of scheelite is negatively charged in dilute alkaline medium and becomes more negative when sodium oleate is added (Arnold and Warren, 1974). Shear flocculation utilizes the energy of hydrophobic association that takes place when the hydrocarbon chains of the collector on particle surface come in contact (Subrahmanyam and Forssberg, 1990), but energy is required to be input into the system to allow the hydrophobic association to occur, which has electrostatic repulsion as a barrier to the process. In previous published research, shear flocculation for fine scheelite was performed in a cylindrical stirred tank with a one-blade agitator. At sufficient high agitating

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http://dx.doi.org/10.1016/j.mineng.2017.07.002
Received 25 September 2016; Received in revised form 10 June 2017; Accepted 11 July 2017
0892-6875/

speed, hydrophobic, negatively charged fine scheelite particles (about 1 μm) overcame their energy barrier and aggregated into flocs (larger than 4 μm), which were easy to be recovered by conventional froth flotation (Warren, 1975). It was reported that the pre-treatment via shear flocculation on a fine scheelite ore (70 wt.% less than 15 μm) could achieve a 9% improvement in recovery and a 5–6% improvement in scheelite grade (Koh and Warren, 1980).

Based on flocculation and flotation of fine scheelite by offering high shear conditions, shear flocculation can potentially upgrade deposits of finely-grained minerals, and, more generally, for separating different types of fine particles suspended in a fluid (Warren, 1992). Numerous papers about shear flocculation have been published, for example, aggregation of chromite fines using sodium oleate (Akdemir and Hiçyilmaz, 1996) and its separation from serpentine (Akdemir and Hiçyilmaz, 1998), flocculation-flotation of hematite (Akdemir, 1997; Yin et al., 2011) and its separation from quartz (Pascoe and Doherty, 1997), floc flotation of galena-sphalerite fines using potassium amyl xanthate (Song et al., 2001), shear flocculation of celestite using anionic surfactants (Ozkan et al., 2006), shear-induced flocculation of kaolinite (Mietta et al., 2009) and shear flocculation of colemanite (Ucbeyiay and Ozkan, 2011, 2014).

Though shear flocculation has always appeared promising for recovering valuable mineral particles that are not floatable due to their fine size, there are little large-scale applications of shear flocculation in flotation practice. However, the energy input range for forming optimal flocs with no destruction or re-dispersion is hard to determine (Bakker et al., 2002). It is thought that the limited industrial uptake of shear flocculation is attributed to lack of full understanding and characterization of shear flocculation (Forbes, 2011). So far, how to determine an appropriate energy input for realizing effective shear-flocculation hasn't been studied.

Unfortunately, published papers related to flocs and energy input mainly focused on the size, shape and strength of flocs. It has been reported that size distribution of flocculated scheelite pulp is bimodal. The flocculation behavior of flocs and discrete particles can be described by mathematical models (Koh et al., 1986). The size distribution of flocculated suspensions in larger tanks and the growth of flocs during flocculation process can be predicated by population-balance model (Koh et al., 1989, 1987; Heath, 2006). The floc shape of fine particles can be characterized by parameters such as convexity, circularity (Vlieghe, 2014) and roundness (Koivuranta et al., 2013). The floc structure has been reported to be described by aspect ratio (Koivuranta et al., 2014), area fractal dimension (Gruy, 2011) and mass fractal dimension (Zhou and Franks, 2006). The strength of flocs induced by shear flocculation can be measured by its shear resistance capacity and interaction forces (Zhou et al., 2008; Liang et al., 2015). In addition, the process variables influencing the aggregation and breakage kinetics of flocculation has also been studied (Heath et al., 2009). In summary, the published work to date has put emphasis on the characterization techniques for flocs induced by shear flocculation, rather than the method of selecting the suitable energy input to achieve the most effective improvement in recovery of fine particles and the flotation rate.

It should be also noted that energy input on mineral pulp has considerable influence on its separation process and flotation rate in flotation. Energy input (represented by agitation intensity) in pilot-scale mechanical flotation cells has been reported to be a significant factor for the separation effect of platinum ores and gangue minerals (Deglon, 2005). Similarly, energy input in coal flotation (measured by shaft power and flotation time in cell) is also found to have a remarkable effect on the flotation grade and recovery of coal (Gui et al., 2014, 2013). Studies on the flotation kinetics of minerals using different energy input devices, such as oscillating grid flotation cell (Changunda et al., 2008; Massey et al., 2012) and a "Rushton turbine cell" (Newell and Grano, 2006), have demonstrated that flotation rates of galena, pyrite, pentlandite, apatite, hematite and quartz (Safari et al., 2016) are all energy input dependent. However, the influence of energy input on the flotation rate of shear flocculated pulps containing flocs and discrete particles is still unclear and has rarely been reported.

The main objective of this paper is to define the effect of energy input (EI) on the flocculation process and flotation rate of fine scheelite. A reagent scheme for shear flocculation was determined by basic flotation behavior of fine scheelite (less than 10 μm) in contrast to that of coarse scheelite (larger than 10 μm). A four-baffle cylindrical tank with a four-blade impeller was used to facilitate EI by changing agitation speed. Particle size measurement, optical observation and flotation tests with different EI were conducted. To quantify the flocculation process of coarse fines (CF, +7.5–10 μm), intermediate fines (IF, +2.5–7.5 μm) and fine fines (FF, −2.5 μm) of scheelite, a parameter termed the flocculation degree, was introduced. On the basis of calculating the flocculation degree of these size fractions with different EI, the flocculation process was described by an aggregation and growth model. Finally, the correlation between the flotation rate and flocculation degree as a function of EI was discussed.

2. Experimental

2.1. Materials and reagents

Hand-picked pure scheelite crystals were crushed to less than 1 mm by a laboratory jaw crusher and a laboratory roll crusher. The crushed products were concentrated on a concentrating table to remove the heavy particles and on a high-intensity magnetic separator several times to discard the magnetic minerals. The non-magnetic products were ground in a porcelain mill and classified into five narrow size fractions: −10, +10–38, +38–55, +55–74 and +74–106 μm.

Sodium oleate ($C_{18}H_{33}O_2Na$) used in this study was purchased from Tianjing Kermil Chemical Reagents Development Centre, Tianjin, China. Its molecular structure consists of a hydrocarbon chain and a carboxyl head group, as shown in Fig. 1. It is selected as the flocculant in shear flocculation operation because it is a traditional collector in flotation practice of scheelite. Hydrochloric acid (HCl) and sodium carbonate (Na_2CO_3) were used as pH regulators. Distilled water was used for all tests.

The effect of sodium oleate on the zeta potential of scheelite has been measured (Arnold and Warren, 1974). At the vicinity of pH 10 adjusted by Na_2CO_3, the zeta potential of scheelite is about −36 mV. Conditioned by 1×10^{-4} mol/L sodium oleate, the zeta potential becomes −42 mV. The decrease in zeta potential is attributed to the adsorption of the sodium oleate on the surface of scheelite as introduced in Section 1.

2.2. EI and its measurement

EI was realized using a four-baffle agitation tank with a four-blade impeller, as shown in Fig. 2. The tank was a cylinder with a height of 100 mm and a diameter of 60 mm respectively. There were 4 baffles (80 mm × 5 mm) in the tank. The whole diameter of the impeller was 30 mm and its blade (10 mm × 10 mm) was perpendicular to the axis. The distance between impeller and tank bottom was 30 mm. The impeller was driven by an IKA Eurostar power control-visc6000 agitator, which could control and adjust the rotational speed in a stepless manner in range of 150–6000 rpm. The torque to overcome the fluid resistance was recorded by a torque meter installed inside the agitator. When agitating with a slurry volume of 200 mL, it was found that air entrainment in the liquid was minimal even at 1500 rpm.

Mineral pulp was prepared by adding 10 g fine scheelite to 200 mL

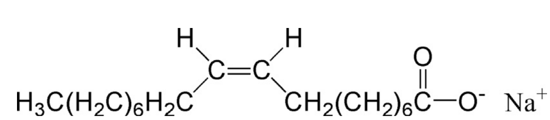

Fig. 1. Molecule structure of sodium oleate.