Technical note

Evaluation of collector performance at the bubble-particle scale

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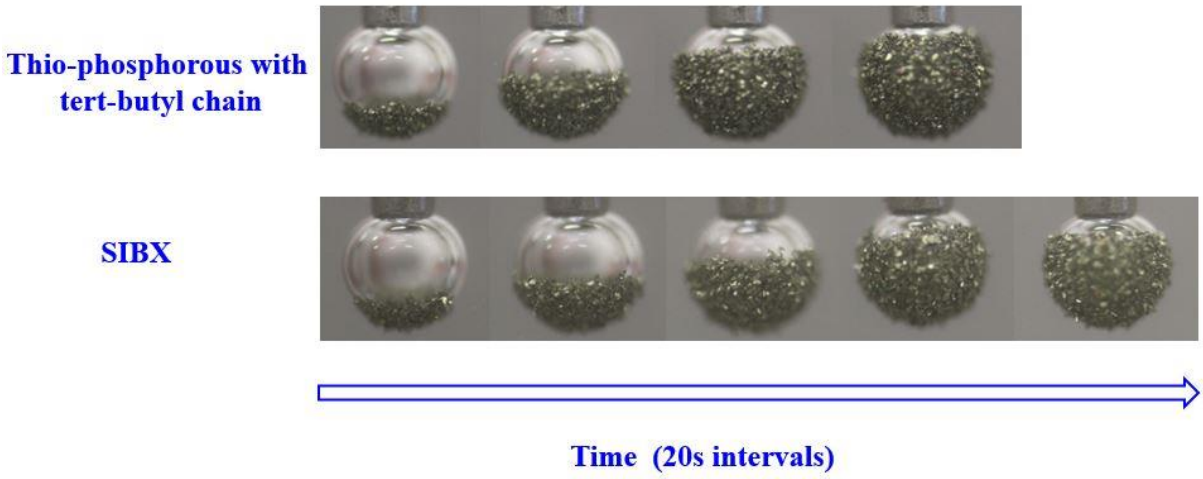
ABSTRACT: Particle attachment and detachment in froth flotation are complex processes and their measurement presents many challenges. Of particular interest is the effect of collectors at the bubble-particle scale, in order to assess the strength or collecting ability of these reagents. However, studies of the effect of collectors on particle attachment at the bubble-particle scale are scarce. In this work, we propose a methodology to characterise collector strength by measuring the attachment rate of particles to a capillary-pinned bubble. An image processing technique was developed to quantify bubble surface coverage over time, which was then used to determine particle attachment kinetics. The image analysis strategy is based on the sessile drop method and uses curve fitting to determine accurately the particle coverage. The methodology was used to assess the collecting ability of three chalcopyrite collectors. Interestingly, although very similar contact angle measurements were found for two of the collectors, they showed distinctly different particle attachment kinetics. It is proposed that this particle-bubble attachment method can be used to gain additional information not currently available from either contact angle measurements or bulk collector performance tests.

Graphical abstract:



Keywords:

Flotation; Collector performance; Image analysis; Sessile drop method; Particle attachment; Bubble loading; Surface coverage; Contact angle.

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1. Introduction

Collectors are used to render mineral particles hydrophobic and therefore play a key role in froth flotation. Different experimental approaches exist to evaluate collector performance, which include assessing their impact in terms of bulk separation performance indicators (recovery and selectivity) and measuring the mineral surface contact angle after exposure to the collector.

At the bubble-particle scale, induction time measurements (Verrelli and Albijanic, 2015) can be performed to study the time required for a particle to attach to a bubble for a particular

collector. This technique requires quiescent conditions and individual particle-bubble collision events.

A dynamic system where multiple particles attach to a single bubble in a turbulent environment will allow the collector performance to be assessed under conditions closer to those in a flotation cell. One of the challenges of such a system is to determine accurately the extent of particle coverage on the bubble.

The angle formed by the particle bed on a capillary-pinned bubble has been suggested by Chu et al. (2014) as a proxy for determining the mass of attached particles, as it was shown that the angle correlates linearly to the mass of the particles. Ata (2009) quantified the particle coverage on a capillary pinned bubble by assuming it as a half-sphere and half-ellipsoid, and solving a surface integral in spherical coordinates to determine surface coverage. The true bubble shape can be determined using the pendant drop method or the sessile drop method (Arashiro and Demarquette, 1999; Yang et al., 2017), but this has not previously been used to determine particle coverage of the surface of a bubble.

In this study, a technique is developed to assess collector performance at the bubble-particle scale that combines kinetic particle attachment tests with an image processing technique based on the sessile drop fitting model. This allows a more precise description of the bubble shape and loading. The technique is demonstrated for the attachment kinetics of chalcopyrite for three different collectors, and compared to contact angle results.

2. Experimental

2.1. Materials

For the particle attachment tests, samples of chalcopyrite were ground and screened to -125µm +75µm. Three collectors were tested; Sodium Isobutyl Xanthate (SIBX) and two thio-phosphorous collectors, one with tert-butyl and one with nonyl chains (supplied by BASF).

Thiol collectors have been reported to yield a high sulphide mineral recovery (McFadzean et al., 2012 and McFadzean and O'Connor, 2015). The thio-phosphorus collectors were mixed 1:1 by weight with 2-ethylhexan-1-ol. All experiments were performed at 19 °C under natural pH conditions.

2.2. Setup

A schematic diagram of the experimental setup is shown in Fig. 1. A stainless steel capillary (18 gauge needle), mounted on a 1 mL syringe, was placed in a transparent rectangular chamber (5 ×5 ×12.5 cm) and bubbles were generated from the tip of the capillary. The 1 mL syringe was connected to a 10 mL syringe driven by a programmable micro-syringe pump, which permitted generating bubble of a precise size by controlling the air injection into the capillary. The experimental rig was placed on a vibration-free table to avoid disturbances. Images of the bubbles, prior and after they were coated with particles, were recorded using a Canon EOS 600D camera with LED back-lighting for illumination.

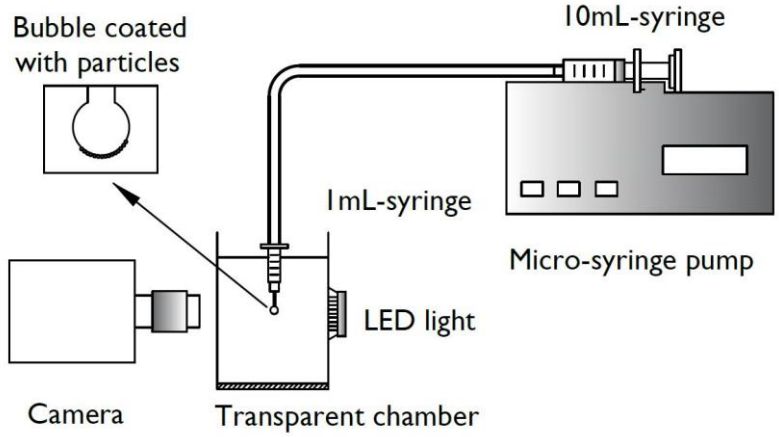


Fig. 1. Schematic diagram of the experimental setup to investigate the attachment of chalcopyrite particles to a capillary-generated bubble.

2.3. Experimental procedure.

One gram of chalcopyrite particles was added to the transparent chamber, which was filled with 250 mL deionised water. The collector was added to achieve a concentration of 0.08 mg/L and the solution was then mixed for 15 min using a magnetic stirrer. After the particles in

suspension settled, a bubble of approximately 2.4 mm in diameter was generated at the tip of the capillary, and an image of the uncoated bubble was captured. The system was agitated again to promote particle attachment at a magnetic stirrer speed of 350 rpm for consecutive, short periods of time. After the particles settled, an image of the particle-laden bubble was captured. The surface area of the bubbles and surface coverage, i.e. the percentage of the surface area covered by particles, were calculated using Matlab, using the image processing technique described in Section 3. The tip of the capillary was photographed as a reference dimension during image processing. Each experiment was carried out in triplicate.

2.4 Contact angle test

Contact angle tests were conducted using a Ramé-hart Model 500 Advanced Goniometer. Chalcopyrite sample plates (39 mm in diameter) were polished for 30 min with an 8 μm silica paste, to obtain a fresh surface. The polished chalcopyrite plate was then washed with DI water, prior to ultrasonic cleaning in a pure ethanol solution for 5 min, to remove any debris or organic contamination. The plate was conditioned for 15 min in a collector solution with the same concentration as the attachment tests. Finally, it was washed with DI water and then excessive water was removed using filter papers. After polishing and cleaning, the contact angle tests were performed immediately. To guarantee the accuracy of the measurements, ten points on the plate surface were chosen randomly to conduct contact angle tests for each collector.

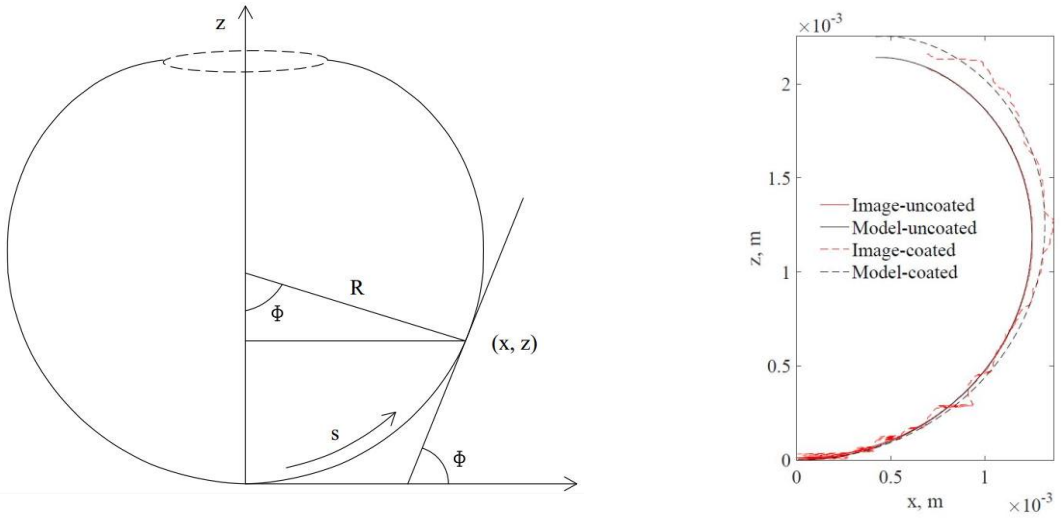
3. Image processing technique

This technique was used to calculate the bubble surface area as well as the bubble surface coverage. The bubble was assumed symmetrical, and a series of edge points were detected only on one half of the bubble image. The sessile drop equation was then fitted to these bubble edge points. The theoretical bubble shape was considered to be governed by the Young-Laplace

equation (Yang et al., 2007), and the pressure difference ΔP across the curved liquid/air interface is described using the radii of curvature $R_1$and R_2 and the surface tension σ:

$$\Delta P = (P_{int} - P_{ext}) = \sigma\left(\frac{1}{R_1} + \frac{1}{R_2}\right), \quad (1)$$

where $R_1 = R_2 = R$, P_{int} and P_{ext} are the internal and external pressure, respectively. This is shown schematically in **Fig. 2**. (a),



(a) (b)

Fig. 2. (a) Schematic for the derivation of the Young-Laplace equation to obtain a fit to a bubble edge. (b) Curve fitting to the uncoated and coated bubble edge points; the solid lines correspond to the uncoated bubble edge from the image (red) and the model (black) and the dotted lines to the coated bubble edge from the image (red) and the model (black).

Eq. (1) can be expressed as a group of three first-order differential equations for the spatial positions x and z and turning angle Φ of the interface as a function of the arc length s of the bubble shape, and then integrated with boundary conditions $x(s = 0) = 0, z(s = 0) = 0, \Phi(s = 0) = 0$, the equations were solved numerically:

$$\frac{d\Phi}{ds} = -\frac{sin\Phi}{x} + \frac{2}{R} + \frac{\Delta\rho g z}{\sigma} \quad (2)$$

$$\frac{dx}{ds} = cos\Phi \quad (3)$$

$$\frac{dz}{ds} = sin\Phi\,, \quad (4)$$

where g is the gravity acceleration constant, and the surface tension and the radii of the bubbles can thus be estimated from the numerical fit.

Fig. 2. (b) shows the sessile drop equation models fitted to the extracted bubble edge points. The fitting model was rotated around the vertical axis to achieve a 3D representation of the bubble. The surface area of the uncoated bubbles was then calculated. For the coated bubble, to take into account the influence of particle diameter on the calculation, an average of the coated model and the corresponding uncoated model was used for the calculation of the surface area. The surface area covered by particles was determined by considering the sections with different particle-coverage height and integrating over the corresponding surface, thus taking into account the 3D shape of the bubble. Surface coverage was then obtained by dividing the covered area by the total surface area of the bubble.

4. Results and discussion

The rate at which particles attach to bubbles is a key parameter in flotation kinetics and is strongly influenced by the collector used. The attachment rate can be quantified in terms of the surface coverage as a function of time, as shown in Fig. 3. The thio-phosphorous collector with the tert-butyl chain exhibited a high collecting ability, rapidly reaching a high surface coverage and out-performing both SIBX and the thio-phosphorous collector with nonyl chain. Thio-phosphorous with tert-butyl chain resulted in 92% surface coverage after 160 s, while after the same time period, only 71.6% and 59.5% surface coverage were obtained from particles conditioned with SIBX and thio-phosphorous with nonyl chain, respectively. It should be noted that at very short times, i.e. within 20 s, the results for thio-phosphorous with nonyl chain and

SIBX were similar. Confidence intervals shown are at 95%, estimated using the three repeats performed for each experiment.

For comparison, the contact angle of chalcopyrite after being conditioned with the different collectors was determined. The chalcopyrite plate conditioned with SIBX and thio-phosphorous with tert-butyl chain showed a very similar contact angle (114°±6.8° and 114°±3.7°, respectively), and higher than when conditioned with thio-phosphorous with nonyl chain (contact angle of 94.4°±8.4°).

This indicates that the kinetic attachment tests not only provide more information that cannot be obtained from single contact angle tests alone, but are also able to distinguish between collectors when the contact angle alone cannot. The technique provides a convenient way to characterise the performance of collectors by considering the kinetics of attachment in a flotation system.

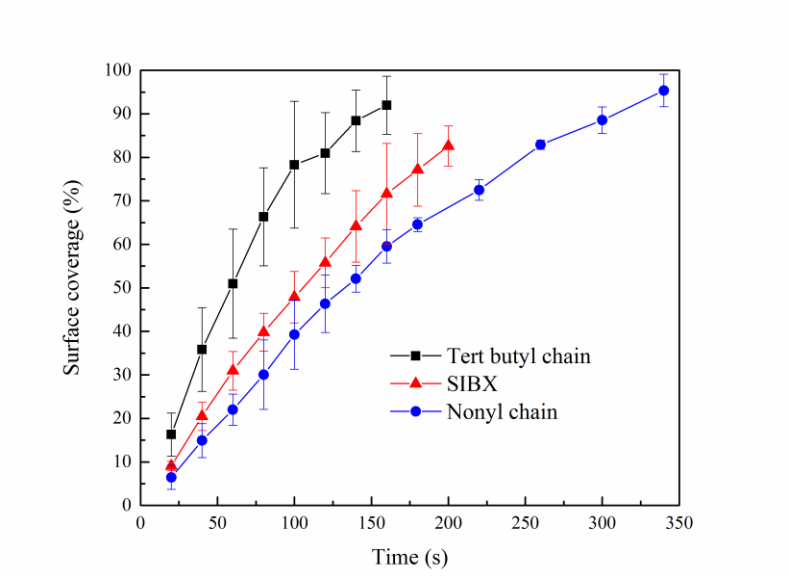


Fig. 3. Surface coverage as a function of time in the chalcopyrite attachment experiments, using SIBX and two novel thio-phosphorous based collectors with tert-butyl and nonyl chains. The data are shown with 95% confidence intervals.

5. Conclusions

An image analysis technique to determine the surface area of bubbles covered by particles was developed and used to study the rate at which chalcopyrite particles attached to a bubble on a capillary. The surface coverage after different agitation times is proposed as a proxy for collector strength that can be useful to understand phenomena at the bubble-particle scale. This method was used to compare three different collectors, including two thio-phosphorous based formulations.

The attachment results showed that the thio-phosphorous collector with tert-butyl chain promoted a more rapid rate of attachment of chalcopyrite particles to the bubble than with nonyl chain. This was in line with a higher contact angle observed for the former. Interestingly, the contact angle for SIBX was very similar to that of the thio-phosphorous collector with tert-butyl chain despite the latter yielding a higher rate of attachment. This suggests that the kinetics of attachment play also an important role in characterising and comparing collectors, providing information that would not be obtained from contact angle tests alone. The technique presented in this work can be used to provide such additional information to assess collector performance.

Future work to expand the scope of this technique may include assessing different ranges of particle sizes and different levels of agitation intensity, as well as adapting the experimental setup to collect the attached particles in order to determine their weight.

Acknowledgments

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