A dissolved-air flotation microcell for floatability tests with particulate systems

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A bench flotation apparatus was constructed and tested in the laboratory on pyrite fines. This was based on the conventional test-tube idea, but modified to permit the experimentation of floatability by dissolved-air. The various advantages of the new microcell were discussed.

Keywords: flotation; dissolved-air; Hallimond tube; Pyrite(s); modifiers

Introduction

Floatability tests are usually undertaken in order to evaluate the possibility of beneficiating a given mineral. For these tests, a small laboratory-scale apparatus is used. Indications of floatability and preliminary conclusions may be easily obtained and the results are extrapolated to semiscale or industrial-scale operating conditions, usually by performing experiments in larger scale apparatus.

For several decades now, the well-known Hallimond tube[1] has been extensively used and only very recently other such small vessels have been reported, e.g., the Emdee Microflot vessel[2] and others.[3] In the modified Hallimond tube, air is usually dispersed through a ceramic porous sparger at the bottom of the cell. The size of the bubbles in conventional cells usually lies between 0.6 and 1.0 mm; if smaller bubbles are required, a surfactant has to be added to the pulp.

Mineral processing plants are facing increasing needs to treat fine particle material, due to liberation problems, loss of valuable material, etc. However, when the size of the particles is too small, many problems are encountered in their separation and beneficiation: entrainment of gangue particles, increase of reagent consumption, etc.

Many of these problems could be alleviated by using air bubbles considerably smaller. One way of obtaining finer bubbles is to use pressurized water in which air has been dissolved; when pressure is released, using a needle valve, fine bubbles, usually smaller than 120 μm, "precipitate" out of the solution. The mean size of the bubbles is approximately 60–70 μm, whereas Water Research Centre (UK) has devised a proprietary nozzle, which produces even finer bubbles (approximately 40 μm).[4]

Dissolved-air flotation is based on this technique; it is a separation process extensively used in effluent treatment.[5] It is being used in flotation columns, with a specially designed pressurized water injector designed by the U.S. Bureau of Mines.[6] The main advantage of using smaller bubbles is an increase in recovery rates, which may be attributed to an improved bubble-particle collision efficiency.[7]

The present paper describes a novel flotation microcell. It is essentially based on the Hallimond cell, but modified so as to be used in dissolved-air flotation tests. This allows floatability tests to be performed in conditions similar to those encountered in larger scale flotation cells.

Experimental apparatus and materials

The experimental apparatus consisted of the novel dissolved-air flotation microcell, which was constructed in the laboratory, and a water saturator (*Figure 1*). The cell was a slightly altered Hallimond tube, with a vertical part height of approximately 265 mm, as compared with a height of 102 mm in the original design.[1] This further modification ensured that any fines carryover is avoided.[8] The Hallimond tube was placed on a magnetic stirrer to provide agitation.

A saturator, having a capacity of 10 L, was used to saturate water with air supplied from a compressor, having a gauge pressure of 400–500 kPa.

The tubing inside the connection (after the valve) ended in a piece of fine stainless steel tubing, with a nominal inside diameter of 0.595 mm, supplied by Coopers Needle Works Ltd (UK), so that fine air bubbles are produced (based on our experience).

Floatability tests were performed in the new cell with pyrite fines. Pyrite is a comparatively little studied sulfide mineral, usually associated with gold, which makes its beneficiation attractive, for obvious

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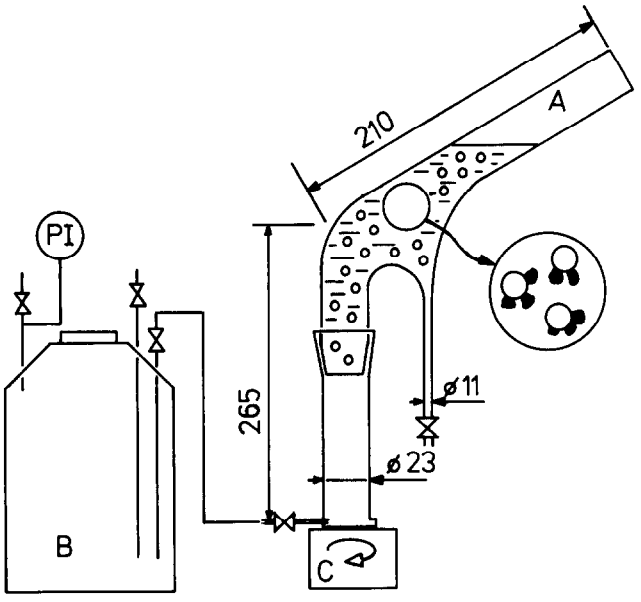

Figure 1 Dissolved-air flotation microcell apparatus. (A) Microcell. (B) Air saturator. (C) Magnetic stirrer

economic reasons. It is also associated with most sulfide ores. Therefore, its separation is important, since pyrite depression is required to produce a marketable-grade concentrate.[9] It appears also that, for environmental reasons, pyrite might have to be removed from coal used in power stations, so that sulfur emissions may be lowered. The pyrite used in the experiments came from the Hellenic Chemical Products & Fertilizers Co. Ltd., in the Stratoni area of Chalkidiki in Northern Greece. Its main component (approximately 95%) was found by chemical analysis to be FeS_2, with the remaining being carbonates and silicates; no other sulfide minerals were detected by X-ray diffraction.

A sample of about 0.5 kg was selected by hand-sorting. Its size was reduced to -75 μm using a jaw crusher and a disk pulverizer; the material was then wet-sieved in order to collect the various size fractions. The $-25 + 10$ μm particle size range was selected for the experimental work (unless otherwise stated). A 200-ml pyrite suspension was prepared in deionized water, having a pulp density of 0.5% (w/w) in solids. This was mixed for 15 minutes; the pH of the solution was adjusted using sulfuric acid or sodium hydroxide. A further conditioning period was applied when modifiers were used: 10 minutes in the case of pentahydrate copper sulfate and 30 minutes for potassium permanganate.

Potassium O-ethyl-dithiocarbonate (KEX) was used as a collector, since xanthates are known to be good collectors for pyrite and the mechanism of pyrite flotation with xanthate collectors is well known.[10] It should be noted that the xanthic acid is unstable, giving in an acid medium

$$RX^- + H^+ \longrightarrow HRX(aq) \longrightarrow CS_2 + ROH$$

At time $t = 0$, the valve of the tube connecting the saturator to the Hallimond tube was opened, allowing a certain amount of pressurized water to enter the cell. The pressure release generated extremely small air bubbles; they were so fine that no individual bubbles could be distinguished, in contrast to dispersed-air bubbles, which can be clearly seen from other bubbles. Pyrite fines were floated. At the end of the experiment, the concentrate samples were analyzed gravimetrically and the flotation recovery was then calculated as a percentage of the original mineral, in the usual manner.

Experimental results and discussion

The amount of collector required for an efficient flotation of pyrite fines was investigated first (*Figure 2A*); a concentration of 15 ppm was found sufficient and this was kept constant throughout all experiments.

Preliminary experiments were also performed to determine the optimum amount of water saturated with air to be introduced in the cell; this is termed "recycle" and is defined by the following equation:

$$\text{Recycle} = \frac{\text{amount of pressurized water added}}{\text{original suspension volume}}$$

It was found that a recycle of 40% was adequate for an effective flotation (*Figure 2B*).

The effect of the particle size on (dispersed-air) flotation behavior has been investigated.[11] Both particle size ranges (of $-75 + 45$ and $-25 + 10$ μm) exhibited a (local) maximum recovery at pH 8; otherwise the recovery for both particle sizes was similar. The finer size exhibited in general a higher recovery of up to 20% more at pH values over 8, whereas in the acidic region the recovery of coarser particles was higher.

Modifiers are often added to the pulp in many flotation circuits since, when added in correct dosages, they may increase both grade and recovery and, more

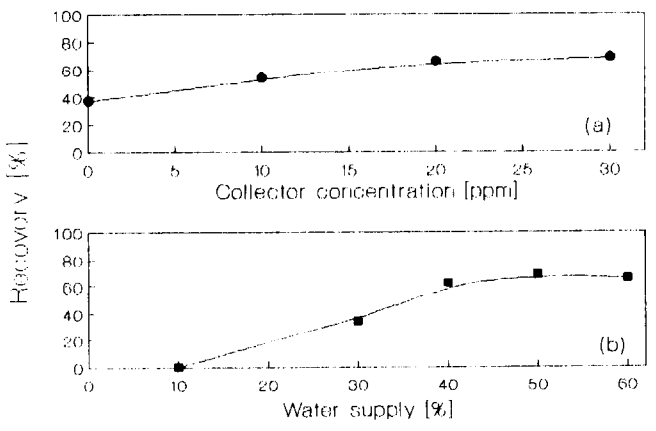

Figure 2 (a) Effect of collector concentration on pyrite recovery. (b) Effect of recycle on pyrite recovery. pH 8; size range $-25 + 10$ μm; [KEX] = 15 ppm; conditioning time 15 minutes; flotation time 10 minutes

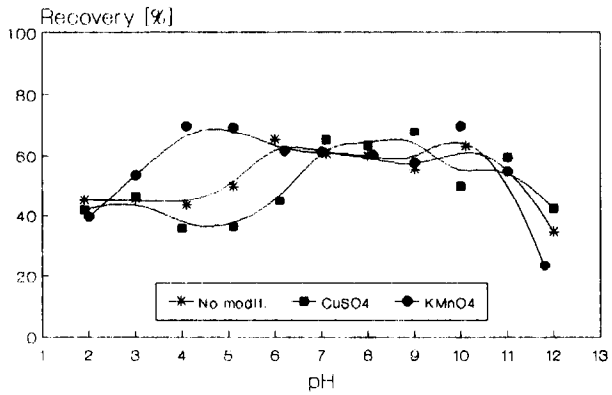

Figure 3 Effect of modifiers on dissolved-air flotation of pyrite. [KEX] = 15 ppm; [$CuSO_4$] = 10 ppm; conditioning time 10 minutes; [$KMnO_4$] = 100 ppm; conditioning time 30 minutes

importantly, achieve greater selectivity; copper sulfate is one such modifier.[12] The use of oxidizing agents for the depression of pyrite and arsenopyrite has also been reported, e.g., potassium permanganate.[13] Both modifiers were used in floatability tests. The optimal concentration for each one (10 ppm for copper sulfate and 100 ppm for potassium permanganate) was determined from preliminary tests.

Experimental results are presented in *Figure 3* and compared with results obtained without any modifier present in the pulp. The effect of the modifier addition is noticed mainly in the acidic region: potassium permanganate resulted in a considerable increase of pyrite recovery (up to 25% at a pH of approximately 4), whereas the addition of copper sulfate resulted in a considerable pyrite depression in the same pH region.

Finally, the performance of the dissolved-air cell was compared with that of the dispersed-air Hallimond tube, for identical experimental conditions. *Figure 4* illustrates the results obtained with the two cells; recoveries are similar over a wide pH range, and only in the extremely acidic region dispersed-air flotation gives higher recoveries.

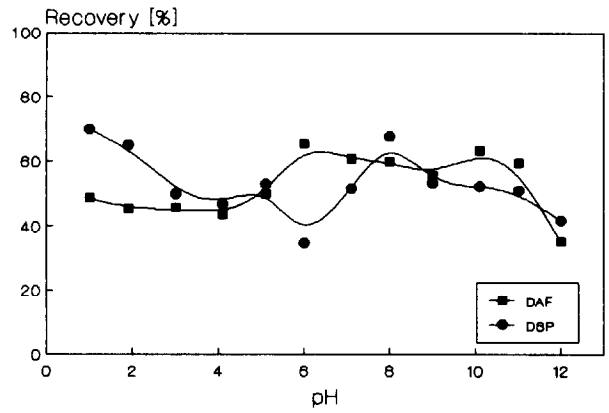

Figure 4 Comparison of the dissolved-air (DAF) and the dispersed-air (DSP) flotation techniques. [KEX] = 15 ppm; conditioning time 15 minutes; flotation time 10 minutes

Concluding discussion

Fines constitute a real problem in mineral beneficiation, and their processing remains opportune, with the continuously diminishing ore grades and their complex mineralogy (e.g., sulfides). The relationship between the physical and chemical properties of fine mineral particles and their behavior in flotation has been investigated.[14] As the particle size is reduced, different interfacial phenomena dominate, influencing either the flotation recovery, the concentrate grade, or both. This results in low flotation rates, rapid oxidation, high reagent consumption, undesirable particle (gangue) entrainment, nonspecific collector adsorption, etc.

New techniques are therefore required, e.g., flotation columns, which look promising for processing such systems.[15] Dissolved-air flotation, a process well established in water and wastewater treatment, offers also some advantages over the more conventional dispersed-air flotation, principally in the production of minute air bubbles, suitable for fines mineral processing.

The Hallimond cell has been modified, so as to use the dissolved-air technique for floatability tests of mineral particles. Its performance has been proved to be similar to the dispersed-air cell. The main advantage of the novel cell, however, lies in the fact that experiments are performed under conditions identical to those in the large-scale flotation cells. For example, laminar flow conditions prevail in the collection and flotation area of these cells, due to the relatively slow bubble rise velocity. This is beneficial to the rate of particle adhesion to the bubbles and hence to the flotation efficiency. Another advantage of the dissolved-air technique is used, namely the fact that several of the bubbles precipitate on the very surface of the solid particles themselves (using them as a nucleation seed), which means that there is no need for a bubble-particle collision to occur in order to achieve successful flotation. This is most clearly illustrated in *Figure 2A*, where a pyrite recovery of 36.8% was observed in the absence of any collector. All these reasons make this technique attractive for fines beneficiation; it has already been applied to the processing of magnesium carbonate fines,[16] but in a different cell design.

In summary, the novel dissolved-air flotation microcell has been found to offer several advantages over the conventional Hallimond cell design:

- It provides the capability of experimenting with small-size bubbles, which is essential if the floatability tests are to be performed under conditions similar to large-scale vessels equipped with small-size bubble generators, e.g., the pressurized-water injectors designed by the US Bureau of Mines.[6] The use of small gas bubble systems will probably spread in the years to come, especially in treating fine particles and effluent treatment.[17]

- At the same time, it maintains the advantages of the Hallimond tube, i.e., it allows for fast experiments with low mineral consumption, the latter being very important for pure minerals, due to their scarcity in large quantities.

Acknowledgment

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