Frother Dosage in Laboratory Flotation Testing

Federico Grandón, Jaime Álvarez and César Gómez
Universidad de Concepción, Chile

ABSTRACT

Frothers are mainly used in mineral flotation, particularly in the case of laboratory scale testing, to control bubble size. As bubble size has a significant impact on metallurgical performance, frother addition is dictated by the critical coalescence concentration (CCC), which is that value of concentration over which there are minor or no effects on bubble size. The CCC is not one of the physical or chemical properties of the frother in use, but rather a consequence of the combination of its characteristics and those of the flotation machine and operating conditions selected for the test.

Measurement of the CCC in a laboratory mechanical cell, by tracking bubble size for a series of frother solutions of increasing concentration, can be easily accomplished. One concern, however, is whether this value is a good indication for the selection of frother dosages in tests with mineral particles included. CCC measurements run in solutions of two analytical grade and three commercial frothers, with and without solids (ore sample supplied by one of the local mining companies), demonstrated practically no differences. Chemical analysis of the flotation products obtained in the tests with one of the frothers allowed assessment of metallurgical performance; the results showed a strong correlation between recovery and bubble surface area flux.

Keywords: bubble size; critical coalescence concentration; frothers; laboratory flotation cell; bubble surface area flux

INTRODUCTION

Mineral flotation is a process for selectively separating particles by attachment on the surface of bubbles. Once the particle size distribution and liberation characteristics are defined in the grinding stage, the efficiency of the flotation process depends on the size distribution of the generated bubble population. The bubble size generated in the machine depends on the gas dispersion technology installed and its operation. Frothers, dissolved in the pulp water around the bubble formation region, rapidly diffuse and adsorb on the fresh gas-liquid interface created until an equilibrium is reached. When two bubbles collide, if the concentration of frother molecules on their surfaces is high enough, then bubble coalescence will not occur and the formation size will be preserved.

Although flotation rate constants were initially related to bubble size (Ahmed & Jameson, 1985; Yoon, 1993), bubble surface area flux (BSAF) is currently considered the key variable to characterize gas dispersion in flotation cells, as integrates bubble size and superficial gas velocity effects (Finch et al., 1999, 2000). BSAF, the bubble interfacial area generated and rising through the cell per unit time defines bubble-particle collision frequency, and a correlation with metallurgical performance is expected. Relationships between BSAF and recovery have been demonstrated in pilot units (Gorain, Franzidis & Manlapig, 1997; Gorain et al., 1998; Hernandez, Gomez & Finch, 2002).

Bubble surface area flux is calculated from the superficial gas velocity and an average bubble diameter estimated from the bubble size distribution. In the case of lab flotation machines, the gas flowrate delivered to the cell and its cross sectional area are known and the superficial gas velocity can then be estimated; the calculation of the BSAF requires only measurement of bubble size. Several techniques have been proposed to measure bubble size in industrial flotation cells (Jameson and Allum, 1984; Randall et al., 1989; Gorain, Franzidis & Manlapig, 1997; Yianatos et al., 2001; Grau & Heiskanen, 2002; Gomez & Finch, 2002; 2007). Measurement in laboratory flotation units have been demonstrated to be reliable and accurate (Girgin et al., 2006; Gomez et al., 2013; Alvarez, Gomez & Wen, 2014; Gomez, Castillo & Alvarez, 2014).

Operations routinely run and contract flotation tests in laboratory mechanical cells to adjust plant conditions based on feed flotation response or to establish the effect of new reagents and operating conditions. The results of these tests are certainly affected by changes in bubble size during the collection of concentrates, which if unnoticed, are then attributed to variations of other process variables. It has been demonstrated, in water tests, that the presence of surface-active agents has a significant impact on bubble size for single- and multi-hole spargers (Cho & Laskowski, 2002), in lab flotation columns (Azgomi, Gomez & Finch, 2007), and in lab and pilot mechanical cells (Gomez, Castillo & Alvarez, 2014; Nesset, Gomez & Finch, 2007). In all cases, there was a concentration over which there were minor or no effects on bubble size (introduced as critical coalescence concentration (CCC) by Cho & Laskowski, 2002). Measurement of the CCC in a laboratory mechanical cell, by tracking bubble size for a series of frother solutions of increasing concentration, can be easily accomplished. One concern, however, is whether this value is a good indication for the selection of frother dosages in tests with mineral particles included. The work described in this communication addresses this issue by comparing, for several frothers, results of CCC measurements in 2- and 3-phases. The occasion was also used to explore whether a recovery vs. BSAF relationship could be established.

OLOGY

Experimental aspects

Mineral samples preparation and characteristics

The samples used in the 3-phase testing were prepared from a 200-kg ore sample, 100 % below 3.36 mm (mesh 6), provided by a local mining operation. The sample was crushed in three stages using a jaw, a cone and a roll crusher; the product from the roll crusher was in close circuit with a 10-mesh (2 mm) screen. The crushed product was separated in one-kg flotation samples; two of these samples were used for chemical analysis (average feed composition: total Cu 0.77 %, soluble Cu 0.02 %, S 15.31 %, and insoluble material 80.07 %), and five were used to determine ore density by water displacement (average density: 3.17 g/cm3).

Laboratory mechanical cell

The laboratory cell utilized in this work (Figure 1) has been used and described before (Gomez et al., 2013; Alvarez, Gomez & Wen, 2014; Gomez, Castillo & Alvarez, 2014). The unit, an automated forced-air mechanical flotation machine (Edemet), had many features allowing programming, continuous tracking, and control of operating conditions and electrochemical variables. Features used in this work were the electronic control of impeller speed, the internal compressor to provide a steady flow of clean (filtered) and oil-free air, and the automated paddles in the 3-phase flotation tests. Air flow was manually controlled and monitored using one of two rotameters installed in the front panel (ranges 1-10 L/min and 2-20 L/min). All tests were run in a 2.7 L cell provided with an Agitair type impeller.

Figure 1 Flotation unit shown idle (left) and in 3-phase operation (right)

Test conditions and procedures

The tests with solutions were run with four of the same frothers and at the same conditions used in previous work (Gomez, Castillo & Alvarez, 2014), to allow comparisons, while those including solids were run at conditions specified in the protocol for the standard flotation test of the company that provided the mineral samples (Table 1). Gas flow rate was 10 L/min (Jg=0.7 cm/s), and the fastest paddling rate to remove concentrate was used (about 1 rotation/second), once every 5 s.

Table 1 Conditions used in 2- and 3-phase flotation tests

Test	Conditioning		Flotation	
	Impeller speed (rpm)	Time (min)	Impeller speed (rpm)	Time (min)
2-phase	600	1	1,000	10
3-phase	600	3	1,000	7

Reagents used

The frothers tested in this work, as well as the reagents required in the flotation of the mineral samples, are indicated in Table 2. The frother included in the standard flotation tests, which was not available at the time of the tests, was a commercial MIBC from a different supplier.

Table 2 Reagents used in flotation tests

Reagent	Function	Type	Supplier
1-heptanol	Frother	Analytical grade	Sigma- Aldrich
MIBC	Frother	Analytical grade	Sigma- Aldrich
MIBC	Frother	Commercial	Celanese
Flomin 500	Frother	Commercial	SNF
Hostaflot X-23	Collector (Cu)	Commercial	
Diesel	Collector (Mo)	Commercial	
Hydrochloric acid	pH modifier	Commercial	
Calcium oxide	pH modifier	Commercial	

Preparation of frother solutions

The procedure utilized for preparing frother solutions considered that frothers are in general slow to solubilize, may be volatile, and may decompose at room temperature if exposed to daylight. For every frother, test solutions at seven concentrations (2, 5, 10, 15, 30, 60 and 100 ppm) were necessary to model the bubble size variation as a function of frother concentration, and then to calculate the CCC. These test solutions were prepared by dilution of a 100-ppm (μg/g) stock solution; the required volume of this stock solution was made by dissolving 2.3 g of frother in 23 kg of water. The mixture was maintained, in the case of the commercial frothers, for 12 hr with a gentle agitation to ensure complete frother dissolution (only 4 hr were necessary for the analytical-grade frothers), and then sealed in the same container and stored in darkness (Figure 2, left). An effort was made to run the tests for one frother on the next day the stock solution was prepared, to minimize concentration changes by volatilization and decomposition, which would go unaware.

Preparation of mineral pulps

The one-kg samples were ground in a 5.4 L ball mill, Marcy type, using a balanced charge of steel balls. Collector (22 g/ton), diesel (8 g/ton), 0.35 g of calcium oxide and water to have a pulp density of 70 %, were added. A grinding time of 18 minutes, determined to ensure a product with 80 % passing 150 µm, was utilized. The grinding product was added to the flotation cell with 2,384 mL of water, to have a 30 % pulp density. The pH was adjusted, if necessary, to a value of 10.

Bubble size measurement

Bubble size measurement was accomplished using the McGill bubble size analyser (Gomez & Finch, 2002; 2007), which is based on the processing of bubble images collected using backward illumination, in a device for bubble sampling and imaging (Figure 2, right). A digital camera (Nikon model D5100) fitted with a macro lens (Nikon 60-mm), a combination that produced high magnification (4928x3264 pixels) and resolutions (between 170 and 180 pixels/mm), was used to capture bubble images at a rate of one every second for avoiding imaging of the same bubbles twice.

Figure 2 Arrangements used for preparation and storage of frother solutions (left), and for installation of apparatus for measuring bubble size (right)

The measurement was initiated by filling the cell and the bubble imaging device with the selected frother solution. The cell was started with the selected gas flow rate and conditioning impeller speed. After conditioning, the stopper closing the bubble collection tube was removed for bubbles to rise and reach the exposing area. After 3 minutes, to allow the development of a steady flow of rising bubbles, 200 to 300 images were collected one every second. The images were processed using the program Image J, which calculated area and pixel value statistics for every object (group of neighboring pixels darker than a threshold) in the image; area was used to calculate bubble size as the diameter of the circle of the same area, and circularity was used as criterion to accept objects as bubbles (larger than 0.8). Bubble clusters, if present, showed circularities lower than 0.8 and were eliminated. Sizes of the individual bubbles were used to calculate the average Sauter mean diameter D32 (same volume and interfacial area) for the distribution.

RESULTS AND DISCUSSION

Inspection of bubble size measurements in frother solutions and in mineral pulps (2- and 3-phases, respectively), at the same conditions and frother concentrations, consistently showed little difference with lower sizes when solids were present, as previously found by O'Connor, Randall and Goodall (1990); a comparison of sizes collected in this work resulted in values about 7 % lower (Figure 3).

As expected, the differences increased for larger sizes (obtained at lower frother additions), as concentration variations in solution preparation or during measurements may significantly affect bubble size. It is not clear whether these lower values in the presence of solids were the consequence of a more effective preservation of bubble size or just the effect of bubbles missing the imaging area because of reduced buoyancy when loaded with mineral particles.

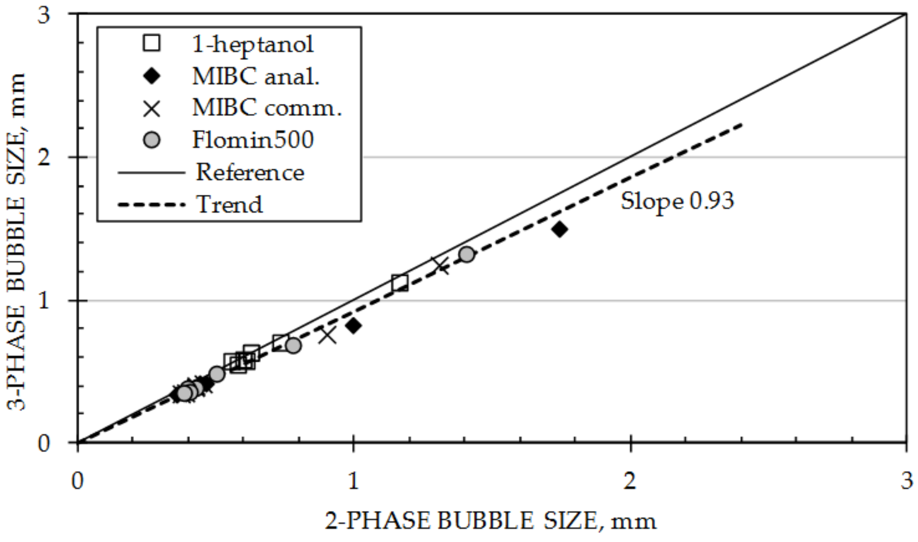

Figure 3 Comparison of bubble sizes measured in 2- and 3-phases for the frothers tested in this work

Bubble size changes in frother solutions and mineral pulps as a function of frother concentration (Figure 4), for two of the frothers tested in this work, demonstrated that the expected bubble size response to changes in frother concentration (a rapid decrease at low concentrations followed by a region with minor size reductions) was not affected by the presence of solids particles. Although a logarithmic scale axis was used for bubble size to expand the measurement range and make differences more evident, the plots demonstrated as before, for the same frother concentration, not much difference between 2- and 3-phase measurements.

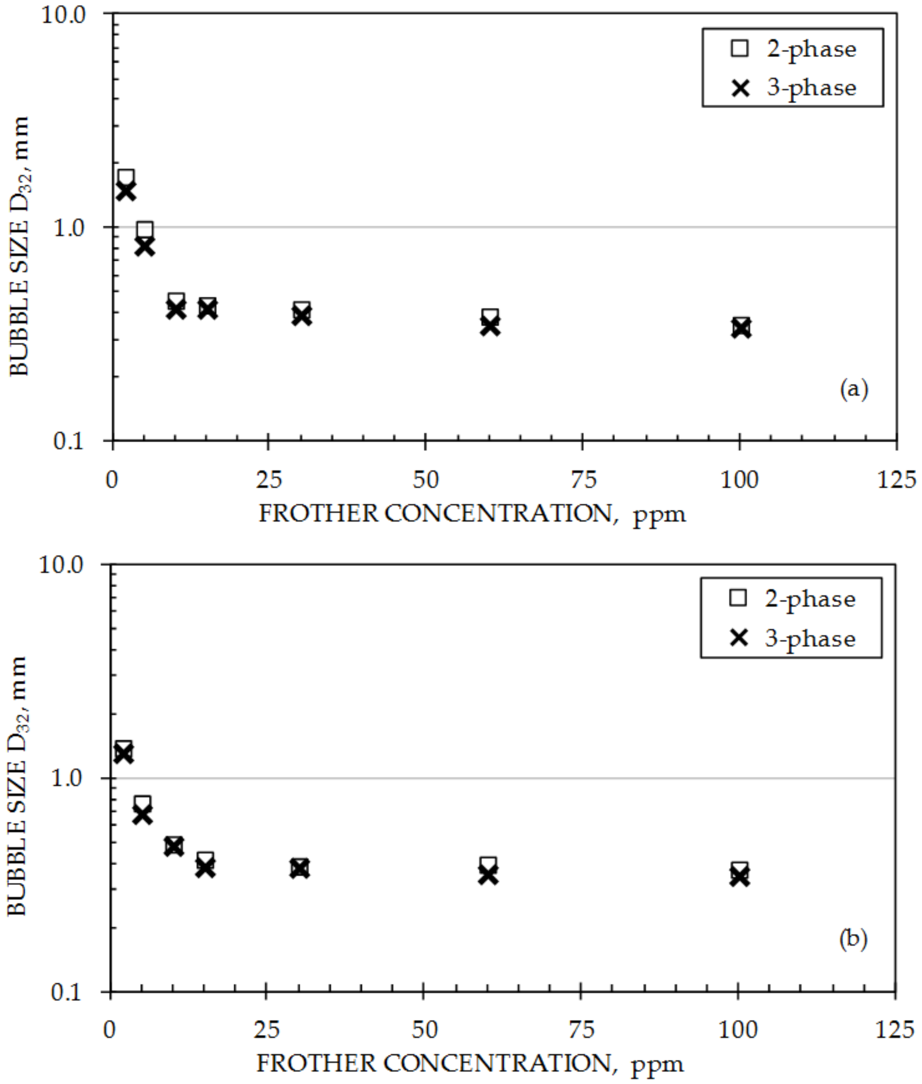

Figure 4 Comparison of bubble sizes measured in 2- and 3-phases as a function of frother concentration for frothers: (a) MIBC, (b) Flomin500

The bubble size variation as a function of frother concentration was modelled considering two contributions (Gomez, Castillo & Alvarez, 2014): i) one characterizing the effect of surface tension, determined by fitting an exponential function to the results for high concentrations (30, 60 and 100 ppm); and ii), one characterizing coalescence control obtained by fitting a second exponential function to the low concentrations results (2, 5 and 10 ppm) with surface tension effects subtracted. The equation to calculate bubble size as a function of frother concentration was obtained by summation of these contributions:

$$D_{32} = a_{st} e^{b_{st} C_f} + a_{cc} e^{b_{cc} C_f} \tag{1}$$

Where D32 is bubble size (mm), a and b are fitting parameters for the surface tension (subscript st) and coalescence control (subscript cc) contributions, and Cf is frother concentration (ppm). Critical coalescence concentration values were calculated as the concentration at which coalescence increased bubble size by 0.1 mm over the value predicted considering only surface tension effects.

$$CCC = \frac{\ln\left[\dfrac{0.1}{a_{cc}}\right]}{b_{cc}} \tag{2}$$

The value 0.1 mm, which was determined for our operator and equipment available by running repeat measurements, was used because it was larger than the error associated with bubble size measurement at a 95 % confidence interval. CCC determination results for the analytical grade sample of MIBC are illustrated in Figure 5, which includes measurements in 2- and 3- phases, the corresponding model fitting, and the calculated CCC values. The CCC difference between the 2- and 3-phase cases was minor in spite of the lower bubble sizes measured when solids were present. Calculations for all the frothers tested in this work, summarized in Table 3, showed as in the case of the analytical grade MIBC, minor differences between measurements in 2- and 3- phases.

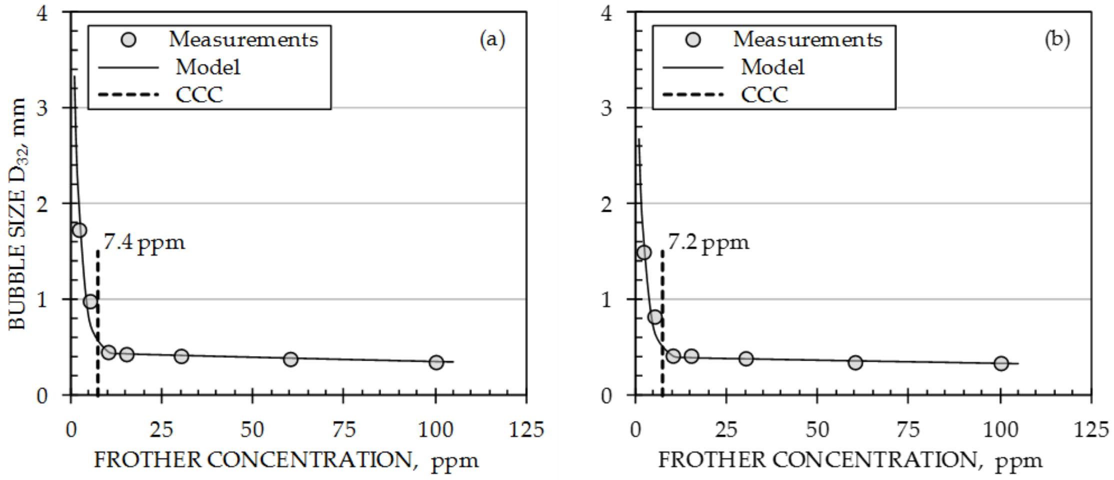

Figure 5 CCC determination for MIBC in: (a) 2-phases; and (b), 3-phases

Table 3 Critical coalescence concentrations (CCC) measured

Frother	CCC (ppm)	
	2-phases	3-phases
1-heptanol	6.7	6.4
MIBC (analytical grade)	7.4	7.2
MIBC (commercial)	6.9	7.0
Flomin 500	9.7	9.7

Bubble surface area flux (Sb) is considered the variable that characterizes machine contributions to flotation kinetics. It is calculated from:

$$S_b = \frac{6 J_g}{D_{32}} \tag{3}$$

Where Jg is the superficial gas velocity (volumetric flow rate / cross sectional area of the cell).

The bubble surface area flux is the bubble interfacial area generated and rising through the cell per unit time. It defines the bubble-particle collision frequency, and therefore, it is expected to correlate with metallurgical performance. To establish whether this correlation occurs, samples of the tailings and concentrate fractions obtained at the different frother concentrations, for the analytical grade MIBC tests, were analyzed for copper to calculate its recovery. The bubble surface area flux was calculated from the gas flow rate delivered to the unit (10 L/min), the cross sectional area of the cell (240.25 cm2), and the bubble diameter obtained at each concentration. The recovery vs. bubble surface area flux plot (Figure 6) demonstrated a close correlation; ignoring the effect of frother concentration on performance may have major consequences on flotation kinetics measurements and on the interpretation of new reagents testing results.

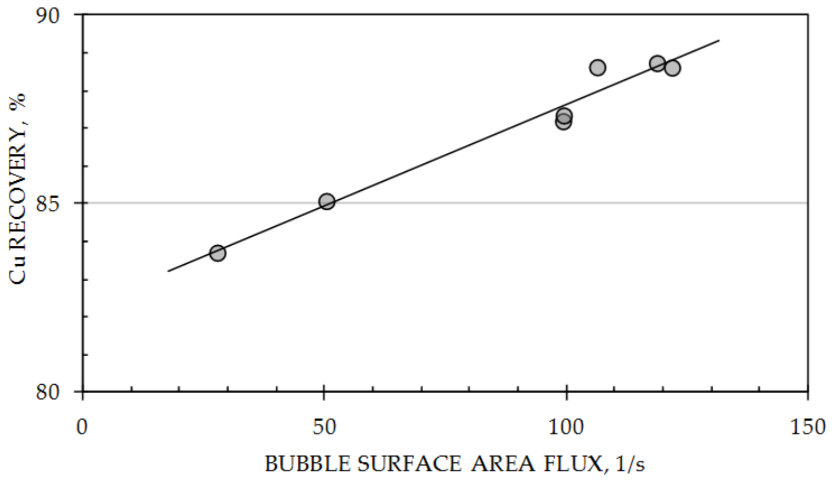

Figure 6 Recovery vs. bubble surface area flux correlation for the analytical grade MIBC tests

CONCLUSIONS AND FUTURE WORK

The following conclusions can be drawn from the results obtained in this work. Some suggestions for future work are also included:

Bubble size measurements in 2- and 3-phases, at the same cell operating conditions and frother concentrations, consistently showed lower sizes when solids were present. It is important to establish whether these smaller values were the consequence of a more effective preservation of bubble size and not just the effect of bubbles missing the imaging area when loaded with mineral particles. Similar tests run with hydrophilic particles, which will not affect bubble buoyancy, will provide evidence to settle this issue.

CCC values determined for the four frothers tested in this work showed practically no differences for bubble size measurements in frother solutions (2-phases) and in mineral pulps (3- phases).

There was a close correlation for copper recovery vs. bubble surface area flux, which was varied by changes in frother concentration. It is important to demonstrate that this correlation also stands when bubble surface area flux is varied by changes in gas flow rate.

REFERENCES

Ahmed, N. & Jameson, G. (1985) The effect of bubble size on the rate of flotation of fine particles, International Journal of Mineral Processing, vol. 14, pp. 195–215.

Azgomi, F., Gomez, C.O. & Finch, J.A. (2007) Correspondence of gas holdup and bubble size in presence of different frothers, International Journal of Mineral Processing, vol. 83, pp. 1–11.

Alvarez, J., Gomez, C.O. & Wen L. (2014) *Gas dispersion characterization of a laboratory mechanical* flotation *cell*, in Proceedings of XXVII International Mineral Processing Congress IMPC 2014, IMPC 2014 Organization, Santiago, Chile, Volume 1, Chapter 5, Paper 188.

Cho, Y.S. & Laskowski, J.S. (2002) *Effect of flotation frothers on bubble size and foam stability*, International Journal of Mineral Processing, vol. 64, pp. 69–80.

Finch, J.A., Gomez, C.O., Hardie, C., Leichtle, G., Filippone, R, & D. Leroux, (1999) *Bubble surface area flux: A* parameter *to characterize flotation cell*', Proceedings 31st Annual Meeting of the Canadian Mineral Processors, Canadian Institute of Mining, Metallurgy and Petroleum, Montreal, Canada, pp. 199–209.

Finch, J.A., Xiao, J., Hardie, C. & Gomez, C.O. (2000) *Gas dispersion properties: Bubble surface area flux and gas holdup*, Minerals Engineering, vol. 13, no. 4, pp. 365–372.

Girgin, E.H., Do S., Gomez C.O. & Finch J.A. (2006) *Bubble size as a function of impeller speed in a self-aeration laboratory flotation cell*, Minerals Engineering, vol. 19, pp. 201–203.

Gomez, C.O. & Finch J.A. (2002) *Gas dispersion measurements in flotation machines*, CIM Bulletin, vol. 95, no. 1066, pp. 73–78.

Gomez, C.O. & Finch J.A. (2007) *Gas dispersion measurements in flotation cells*, International Journal of Mineral Processing, vol. 84, pp. 51–58.

Gomez, C.O., Castillo, P., Rebolledo M. & Alvarez J. (2013) *Frother characterization in a flotation cell*, in Proceedings of Copper 2013, The Chilean Institute of Mining Engineers, Santiago, Chile, Volume II, pp. 49–60.

Gomez, C.O., Castillo, P. & Alvarez J. (2014) *A frother characterization technique using a lab mechanical flotation cell*, in Proceedings of XXVII International Mineral Processing Congress IMPC 2014, IMPC 2014 Organization, Santiago, Chile, Volume 1, Chapter 2, Paper 71.

Gorain, B.K., Franzidis, J-P. & Manlapig, E.V. (1997) *Studies on impeller type, impeller speed and air flow rate in an industrial scale flotation cell. Part 4: Effect of bubble surface area flux on flotation performance*, Minerals Engineering, vol. 10, no. 4, pp. 367–379.

Gorain, B. K., Napier-Munn, T. J., Franzidis, J-P. & Manlapig, E. V. (1998) *Studies on impeller type, impeller* speed *and air flow rate in an industrial scale flotation cell. Part 5: Validation of the k-Sb relationship and effect of froth depth*, Minerals Engineering, vol. 11, no. 7, pp. 615–626.

Grau, R.A. & Heiskanen, K. (2002) *Visual technique for measuring bubble size in flotation machines*, Minerals Engineering, vol. 15, pp. 507-513.

Hernandez, H., Gomez, C.O. & Finch, J.A. (2003) *Gas dispersion and de-inking in a flotation column*, Minerals Engineering, vol. 16, pp. 739–744.

Jameson, G. J. & Allum, P. (1984) *A survey of bubble sizes in industrial flotation cells*, report to Australian Mineral Industries Research Association (AMIRA), pp. 32–37.

Moyo, P., Gomez, C.O. & J.A. Finch (2007) *Characterising frothers by water carrying rate*, Canadian Metallurgical Quarterly, vol. 46, pp. 215–220.

Nesset, J.E., Finch, J.A. & Gomez, C.O. (2007) *Operating variables affecting the bubble size in forced-air mechanical flotation machines*, Ninth Mill Operators' Conference 2007, The Australasian Institute of Mining and Metallurgy, Victoria, Australia, pp. 55–65.

O´Connor, C.T., Randall, E.W. & Goodall, C.M., (1990) *Measurement of the effect of physical and chemical* variables *on bubble size*, International Journal of Mineral Processing, vol. 28, pp. 139-149.

Randall, E.W., Goodall, C.M., Fairlamb, P.M., Dold, P.L. & O´Connor, C.T. (1989) *A method of* determining *the sizes of bubbles in two-and three-phase systems*, J. Phys. E. Sci. Instrum., vol. 22, pp. 827–833.

Yianatos, J.B., Bergh L.G., Condori P. & Aguilera, J. (2001) *Hydrodynamic and metallurgical characterization of industrial flotation banks for control purposes*, Minerals Engineering, vol. 14, no. 9, pp. 1033–1046.

View publication stats