



Short communication

Indirect measurement of frother concentration based on *l*-CCC curvesW. Kracht<sup>a,b,\*</sup>, C. Hunt<sup>c</sup><sup>a</sup> Department of Mining Engineering, Universidad de Chile, Santiago, Chile<sup>b</sup> Advanced Mining Technology Center, AMTC, Universidad de Chile, Santiago, Chile<sup>c</sup> Department of Metallurgical and Materials Engineering, Federico Santa Maria Technical University, Valparaiso, Chile

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## ABSTRACT

The local critical coalescence concentration *l*-CCC presents a relationship between frother concentration and the gas flow rate  $Q'_g$  at which coalescence starts to occur at a capillary tube. Such a relationship is used in this work to determine frother concentration. The solution with unknown frother concentration is tested to determine  $Q'_g$  and the concentration is determined from a reference *l*-CCC curve that has been previously determined for the frother to be tested. The technique is simple, and easy to implement. The determination of the reference *l*-CCC curve takes 1 h or less, after which the measurements are very quick, taking no more than 5 min per sample. The technique was validated against the total organic carbon (TOC) method for a series of samples of unknown frother concentration, for three different frothers, with a correlation coefficient  $R^2$  equal to 0.997. The measurements are not affected by the presence of xanthate in the system, when using MIBC as a frother, which suggests the possibility of using it, combined with TOC analysis to determine both frother and xanthate concentration by mass balancing.

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## 1. Introduction

Frothers are used to stabilise froths and to reduce bubble size in flotation (Cho and Laskowski, 2002; Grau et al., 2005). Since these reagents partition between the pulp and froth zones (Zhang et al., 2013), it can be said that frother concentration varies during flotation, which may affect flotation performance through their effect on gas dispersion properties (Finch et al., 2006; Gomez and Finch, 2007). At plant, frothers are usually added without paying attention to how their concentration varies throughout the plant, nor to the effects that this may have on flotation performance. This is because the methods to measure frother concentration are not simple enough to be implemented in plant to perform measurements on a regular basis. Some methods to determine frother concentration are gas chromatography (Tsatouhas et al., 2005), total organic carbon (TOC) (Hadler et al., 2005; Zhang et al., 2010), and colorimetric techniques (Coles and Tournay, 1942; Gélinas and Finch, 2005, 2007; Zangooi et al., 2010). In the present work, an indirect technique to measure frother concentration is presented. This technique is based on the characteristic sound that bubbles emit when they form and/or coalesce (Strasberg, 1956; Manasseh et al., 2008) and the coalescence curves presented in

the work of Kracht and Finch (2009) that allowed defining the concept of local critical coalescence concentration, *l*-CCC (Kracht and Rebolledo, 2013).

## 2. Experimental

## 2.1. The technique

The technique consists of determining a reference *l*-CCC curve for the frother to be analysed. Such a curve shows the gas flow rate at which consequent bubbles start to coalesce at a capillary tube, i.e., it represents a local direct measurement of the critical coalescence concentration. That curve is characteristic of the frother, and can be written as:

$$Q'_g = f(C) \quad (1)$$

where  $Q'_g$  corresponds to gas flow rate at which coalescence occurs at the frother concentration  $C$ .

Once the reference *l*-CCC curve has been determined, it can be inverted to allow determining the frother concentration:

$$C = f^{-1}(Q'_g) \quad (2)$$

The technique is simple, and easy to implement. The determination of the reference *l*-CCC takes 1 h or less, after which the mea-

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measurements are very quick, taking no more than 5 min per sample. Should the frother or water in the system change, the reference curve ought to be repeated for calibration.

## 2.2. Experimental setup

The experimental set-up is the same that was used by Kracht and Rebolledo (2013). It comprises a 5 L acrylic tank where air bubbles are injected through a glass capillary tube of 0.254 mm inner diameter (6.4 mm external diameter). The air is provided by a compressed air cylinder, and measured/controlled with mass flow metre controller Omega FMA 5508 (1100 sccm). The acoustic emissions are measured with a hydrophone Bruel & Kjaer type 8103 and amplified with a low noise charge amplifier Bruel & Kjaer type 2635. The acoustic emissions are recorded with the freeware audio software Audacity 1.3 beta. The technique here presented may be implemented with a common hydrophone, like that used by Kracht and Finch (2009) in their work.

## 2.3. Reagents

Three frothers were used: an alcohol, MIBC (Sigma Aldrich); a polyglycol, Oreprep F549 (Cytec); and a blend corresponding to an industrial sample of MIBC and DF250 (5:1). The industrial blend was provided by a Chilean sulphide copper flotation plant, located near Santiago. All the tests were run with Santiago tap water. In order to test the effect of collector on the technique, sodium isopropyl xanthate (Cytec) was also used in a series of tests.

## 3. Results and discussion

### 3.1. Reference *l*-CCC curves

Frother solutions were prepared with concentrations ranging from 0 to 25 ppm, in tap water. With these solutions, the *l*-CCC curves were determined, and inverted as shown in Fig. 1 for MIBC, Oreprep F549, and industrial blend. Each data point corresponds to the average of three consecutive measurements, and the standard deviation was always lower than 1%.

The difference observed at the starting point of the curves for the industrial blend and the other frothers (MIBC, and Oreprep F549) is explained because the measurements were performed with different capillary tubes (the capillary broke between experiments). This confirms that the reference *l*-CCC curves depend not only on the solutions, but also on the apparatus characteristics. Changes in the dimensions of the components, especially the capillary, would affect the measurements.

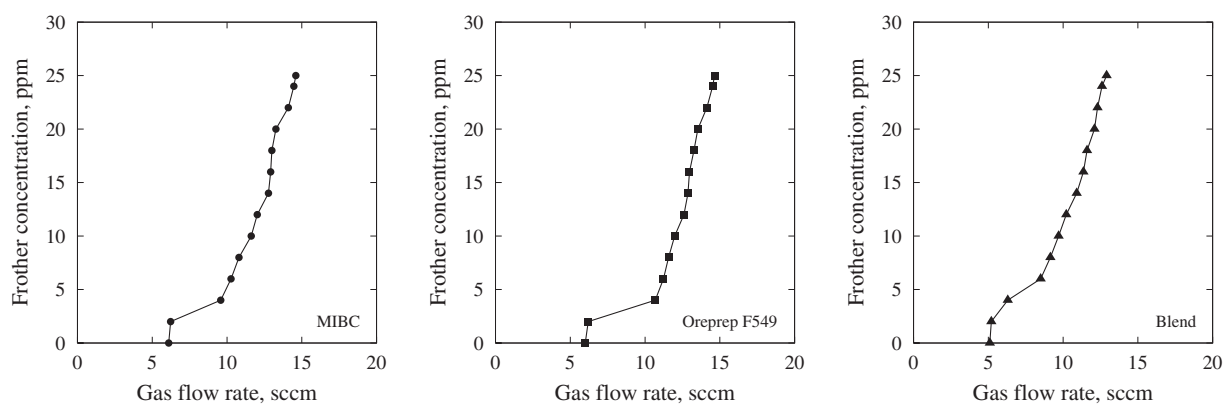


Fig. 1. Curves of frother concentration versus gas flow rate for MIBC (left), Oreprep F549 (centre), and Blend (right).

### 3.2. Total organic carbon

For the same range of concentrations, 0–25 ppm, samples were analysed by total organic carbon (TOC) for comparison. The results are shown in Fig. 2 for MIBC, Oreprep F549, and industrial blend.

It can be observed that there is a relationship between the total organic carbon (TOC) and the frother concentration. For each reagent a linear curve was fitted, which allows determining the frother concentration from TOC data.

### 3.3. Validation of the technique

In order to validate the technique with samples of unknown concentration, a series of 30 tests were run in a laboratory flotation column of 10 feet height and 3 in inner diameter. For each frother, 10 tests were run in the column, varying the reagent concentration between 5 and 25 ppm, and the superficial gas velocity ( $J_g$ ) in the column between 0.5 and 1.5 cm/s. The column was operated in batch mode, without foam overflowing, and without solids. At each condition, the column was operated for 10 min in order to allow frother partition between the collection zone and the foam (Zhang et al., 2013). After 10 min of operation, a sample of the collection zone was taken and was later analysed by both the TOC and the *l*-CCC techniques. The comparison between the frother concentrations determined by both techniques is presented in Fig. 3.

Fig. 3 shows the results of frother concentration for the 30 samples, and validates the technique of indirect measurement of frother concentration based on *l*-CCC curves. The frother concentration determined from *l*-CCC curves compares well to the frother concentration determined using the total organic carbon analysis, with a correlation coefficient  $R^2$  equal to 0.997.

### 3.4. Effect of xanthate

Since frothers are not the only reagents in flotation, it is of interest to test how a common collector, such as xanthate, affects the results obtained with frother alone. In order to do this, a series of measurements were performed with solutions containing MIBC and different concentrations of sodium isopropyl xanthate (0–15 ppm). The results are presented in Table 1.

According to Table 1, xanthate does not affect the *l*-CCC curve of MIBC, with relative standard deviations lower than 1% for all the concentrations of MIBC tested. This was as expected because xanthate does not have a frothing effect, but cannot be extrapolated to other collectors without further testing.

The results suggest the possibility of using the two techniques, *l*-CCC and TOC analysis, combined to determine both the frother and xanthate concentration by mass balancing. The TOC analysis

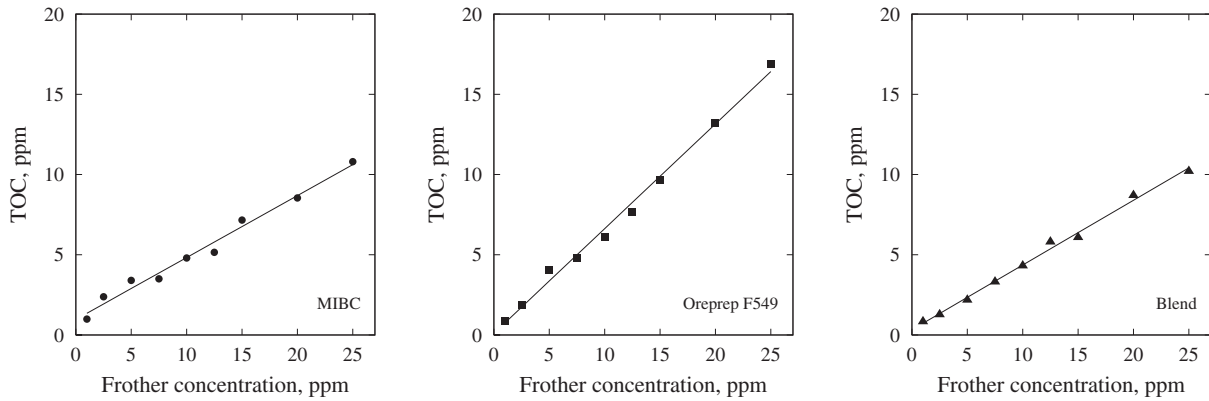


Fig. 2. Total organic carbon (TOC) vs frother concentration for MIBC (left), Oreprep F549 (centre), and Blend (right).

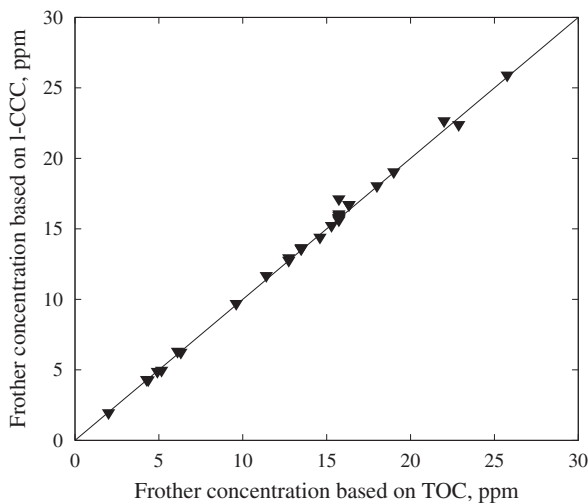


Fig. 3. Frother concentration determined with *l*-CCC method versus measurement based on TOC.

Table 1  
Effect of xanthate on the *l*-CCC curve of MIBC.

MIBC ppm	$Q'_g$ at different xanthate concentrations				Std dev %
	0 ppm	5 ppm	10 ppm	15 ppm	
0	5.88	5.90	5.83	5.87	0.50
5	11.97	11.93	11.90	11.90	0.28
10	12.90	12.87	12.68	12.80	0.76
15	13.55	13.50	13.50	13.67	0.59
20	14.77	14.70	14.73	14.87	0.50
25	16.30	16.57	16.23	16.30	0.92

gives the total amount of organic carbon, which is the sum of frother and xanthate, whereas the *l*-CCC technique allows determining the frother concentration, which can be subtracted from the TOC measurement to account for the xanthate concentration.

#### 4. Conclusions

An indirect technique to measure frother concentration was presented. The technique is simple, and easy to implement. It is based on local critical coalescence concentration (*l*-CCC) curves that serve as a reference to determine frother concentration. The technique was validated against the total organic carbon (TOC) method for a series of samples of unknown frother concentration.

The results correlate well, with a correlation coefficient  $R^2$  equal to 0.997. The measurements were not affected by the presence of xanthate in the system, when using MIBC as frother, which suggests the possibility of using the indirect technique presented in this work combined with the TOC analysis to determine both the frother and xanthate concentration by mass balancing.

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#### Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at <http://dx.doi.org/10.1016/j.mineng.2016.03.010>.

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