



Frother analysis in flotation circuits: Refinement of a colorimetric technique



A. Zangooi*, C.O. Gomez, J.A. Finch

Department of Mining and Materials Engineering, McGill University, Montreal, QC H3A 2B2, Canada

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ABSTRACT

Mineral flotation is a particle separation process based on selective collection on the surface of bubbles leading to formation of bubble-particle aggregates. The separation is driven by natural or induced hydrophobicity of exposed mineral areas on the particle surface. Performance depends on the size range of the bubble population, which in most cases is controlled by addition of frothers. Measurement of frother concentration would help achieve this control objective. A previously developed colorimetric technique suitable for on-site use has been refined to increase analysis rate and reproducibility, particularly for low-solubility frothers. The refinements include the sample preparation procedure, wavelength range for spectra collection, and criteria for selecting calibration standard concentrations and wavelengths. The refined technique is demonstrated to be robust, reliable, and accurate, with a daily analysis rate up to 20 samples while remaining suitable for on-site analyses.

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1. Introduction

Flotation is a separation process based on the selective collection of particles by bubbles. Measurement of properties of the bubble population, so-called gas dispersion parameters (gas velocity, gas holdup and bubble size), has become a tool to characterize industrial flotation cells (Gomez and Finch, 2002, 2007). Cell characterization measurements have shown the important role played by frothers in controlling bubble size and gas holdup (Gomez et al., 2011). Frothers also play a role in stabilization of the froth layer, controlled by transport of water into the froth (Rao and Leja, 2004; Moyo et al., 2007). Recent plant work has shown that knowing frother concentration can help improve cell operation and circuit performance (Gelinis and Finch, 2007; Cappuccitti and Nasset, 2009).

The action of frother in reducing bubble size is commonly attributed to inhibiting coalescence (Harris, 1976). Basic studies bringing two bubbles into contact support this contention showing that the time to coalesce increases in the presence of frother (Cho and Laskowski, 2002; Ata, 2008). These observations have introduced the hypothesis that the flotation machine produces small bubbles, and frother preserves them. As frother dosage increases, a certain concentration (critical coalescence concentration) is reached above which bubble size is approximately constant

(Finch and Dobby, 1990; Cho and Laskowski, 2002). Plant experience, backed by laboratory experiments, has demonstrated that water flow rate to the froth product is also a strong function of frother concentration (Nguyen et al., 2003; Moyo et al., 2007; Zhang et al., 2010), as well as the loading of hydrophobic particles on the bubbles (Hunter et al., 2008).

Although frothers are commonly added (dosed) on the basis of solids feed rate (e.g., g frother/tonne ore), the frother concentration required is that in solution. Calculation of solution concentration (e.g., ppm) is straightforward if the pulp density (% solids) is known. However, possibilities such as incomplete frother dissolution, unknown frother content in recycled process water, and uptake (adsorption) of frother by solids (carbonaceous materials are particularly prone to do this) render the calculation uncertain. Other difficulties in estimating concentration include the use of frother blends, and the contribution of reagents with frothing properties, such as collectors like fatty acids and diluents like alcohols in xanthate collectors. The calculation of the concentration in the froth water is even more complicated as the amount of frother adsorbed on bubbles and released to the water upon coalescence and bursting is not known. A literature search indicated that several techniques have been used to measure frother concentration in flotation systems: gas chromatography (Huang et al., 2002), color intensity (Gelinis and Finch, 2005), total organic carbon (Hadler et al., 2005), high performance liquid chromatography (HPLC), and nuclear magnetic resonance (NMR) (Zhang et al., 2013).

* Corresponding author.

E-mail address: azin.zangooi@mail.mcgill.ca (A. Zangooi).

The colorimetric technique is considered the most appropriate for industrial settings. The technique is based on the Komarowsky reaction for the analysis of aliphatic alcohols (Coles and Tournay, 1942) and was first used for a frother, MIBC (methyl-iso-butyl-carbinol), by Parkhomovski et al. (1976). The Komarowsky reaction involves the formation of a colored solution as a consequence of the dehydration of the frother molecule by concentrated sulfuric acid and its subsequent reaction with aldehyde. The reaction occurs in aliphatic alcohols except ethanol and methanol (Ekkert, 1928, quoted by Gelinas and Finch, 2005), aromatic alcohols and phenols (Fellenberg, 1910, quoted by Gelinas and Finch, 2005), and polyglycols (Gelinas and Finch, 2005), which covers most of the frother chemistries used in mineral flotation. The potential for process diagnosis and optimization was demonstrated by Gelinas and Finch (2007), for example, identifying the presence of remnant frother in recycle water. Subsequent use, however, showed refinements were necessary, notably to increase the analysis rate and to extend application to low solubility frothers (Zangooi et al., 2010). Application requires a flexible procedure capable of adjusting to reagents with different specifications and to waters containing diverse ions, sometimes at very high concentrations.

This purpose of the paper is to provide a critical assessment of the steps in the experimental procedure, to describe the refinements, and to demonstrate that the refined technique is reproducible and accurate.

2. Frother analysis by colorimetric technique: Established technique

The analytical procedure involves the following steps: frother extraction, the formation of colored solution, collection of UV–VIS spectrum, establishment of calibration line and determination of the frother concentration. These steps are described to establish where and why refinements were necessary to improve analysis reliability.

1. *Frother extraction from the sample into concentrated sulfuric acid* (Fig. 1). This is done in two stages: first, by partitioning the frother between two immiscible liquids (100 mL sample, 10 mL of chloroform and 15 g of solid sodium chloride to facilitate frother transfer by reducing foaming at the interface) in a 250 mL separatory funnel (although the equilibrium partition coefficient favours transfer to the organic phase, two extractions were done to ensure frother removal close to 100%); and second, by dehydrating the frother molecule by mixing 20 mL of loaded chloroform with 10 mL of concentrated sulfuric acid in a 100 mL separatory funnel. The reaction rapidly goes to completion with the dehydrated frother dissolving in the acid phase. The loaded acid is separated and passed to the next step: the generation of a colored solution.

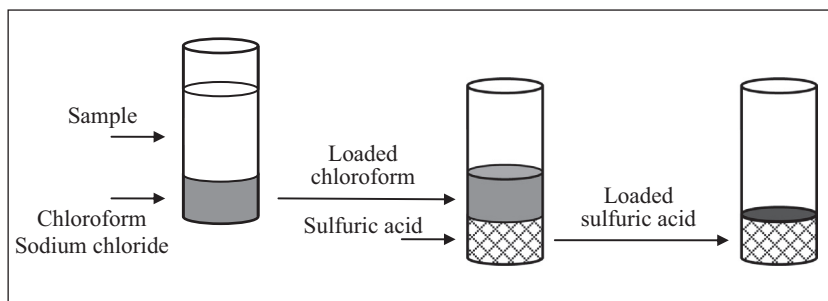


Fig. 1. Frother analysis: frother extraction stage.

2. *Formation of a colored solution via the Komarowsky reaction* (Fig. 2). This is accomplished by adding an indicator (0.1 mL of 5% salicyl-aldehyde dissolved in a 1:1 solution of acetic acid in water) to the vial containing the loaded sulfuric acid, and maintaining the mixture in boiling water for 15 min. The reaction is stopped by immersion of the vial in an ice bath.
3. *Collection of UV–VIS absorbance spectrum*. The colored solution is transferred to a rectangular quartz cell and placed in the UV–VIS instrument. Some precautions were necessary as the maximum absorbance measurable in the UV–VIS spectrophotometer in use is 3 (i.e., the frother concentration in the sample must result in a solution with a color intensity giving an absorbance below 3). Absorbance spectra are collected for wavelength ranges between 490 and 560 nm.
4. *Calibration and calculation of frother concentration*. The calibration curve, concentration vs. absorbance, is constructed with absorbance values obtained on standard solutions (known concentrations) at the selected wavelength in the 490–560 nm region. By ensuring that concentration levels are not too high the calibration is linear. Measuring the absorbance of the unknown sample, at the same selected wavelength, the unknown concentration is estimated from the calibration equation. As wavelength selection affects the precision of the analysis, the recommended wavelength is that giving the maximum (peak) absorbance.

3. Frother analysis by colorimetric technique: Refinements to procedure

Refinements were initially driven by the need to increase reproducibility for low solubility frothers and to speed up analysis while retaining reliability. As the work progressed, however, it became clear that scrutinizing every aspect of the procedure had the potential to increase accuracy (validity) and precision (reliability). The results of this examination are described.

3.1. Preparation of reagents and calibration standards

There are two reagents that need to be prepared: concentrated sulfuric acid, and the Komarowsky indicator. The concentration of the sulfuric acid (75% by volume which corresponds to 84.6% by weight) was increased to 85% by volume to develop a more intense color. The Komarowsky indicator was prepared as before (diluting 5 mL of salicyl-aldehyde in 50 mL of glacial acetic acid and 45 mL of water).

Following Gelinas and Finch (2007), calibration standards (100 mL solutions of known concentrations) were initially prepared by dilution from a 50-ppm solution made from a 500-ppm stock solution, produced by dissolving 0.5 g of frother in 1 L of water. The procedure was changed to preparation of 2 L of a more dilute stock solution (25 ppm) to ensure that this concentration

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