

Comparative study of the main flotation frothers using a new HYDROMESS adapted technique

Estudo comparativo dos principais espumantes com o uso de uma nova técnica HYDROMESS

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Abstract

Stability of bubble size in the pulp zone and froth control is an important parameter for a successful flotation process. The present study compares the action of the main frothers in terms of: mean bubble size in the pulp zone, froth stability, surface-tension-lowering ability and reagent participation in flotation of a naturally hydrophobic mineral. Bubble size was determined using a new technique adapted from HYDROMESS, a device designed to measure drop size. The remaining indicators were obtained by determining surface tension and conducting flotation tests with the addition of hydrophobic (graphite) and hydrophilic (calcite) particles. The results indicate that methyl isobutyl carbinol (MIBC) is the most efficient frother in controlling coalescence, with critical coalescence concentration (CCC) reached at 20 ppm. Moreover, this frother has greater potential for selective action, since it increased calcite entrainment by only 2.3% at a concentration of 30 ppm. In flotation tests with a sample of graphite, MIBC produced higher recoveries.

Keywords: flotation, frother, froth control, coalescence, surface tension, froth stability.

Resumo

A estabilidade do tamanho das bolhas na zona de polpa e o controle da zona de espuma são passos importantes para o êxito de um processo de flotação. O estudo compara a ação dos principais espumantes com relação ao tamanho médio das bolhas na zona de polpa, à estabilidade da espuma, à capacidade de abaixamento da tensão superficial e à participação do reagente na flotação de um mineral naturalmente hidrofóbico. O tamanho das bolhas foi determinado com o uso de uma nova técnica adaptada com base no HYDROMESS, equipamento projetado para a medição do tamanho de gotas. Os demais indicadores foram obtidos por meio de determinação da tensão superficial e de testes de flotação com a adição de partículas hidrofóbicas (grafita) e hidrofílicas (calcita). Os resultados indicam que o metilisobutilcarbinol (MIBC) é o espumante mais eficiente no controle da coalescência, com a concentração crítica de coalescência (CCC) sendo atingida com 20 ppm. Além disso, possui maior potencial para uma ação seletiva, já que, numa concentração de 30 ppm, aumentou o arraste da calcita em apenas 2,3%. Nos testes de flotação com uma amostra de grafita, o MIBC proporcionou as maiores recuperações.

Palavras-chaves: Flotação, espumante, controle da espuma, coalescência, tensão superficial, estabilidade da espuma.

1. Introduction

Frothing has important functions in flotation, among which are: (1) to reduce surface tension of the liquid-gas interface to enable froth formation (Baltar, 2010); (2) hinder coalescence in order to stabilize bubble size (Grau et al., 2005) and (3) facilitate hydrophobic particle adhesion to air bubbles (Laskowski et al., 2003).

Frother reagents can be separated into two groups: ionic and neutral. Reagents from the first group are characterized by strong activity on solid surfaces

and therefore unsuitable as flotation frothers. Neutral reagents, however, do not usually act on mineral surfaces, contributing to process selectivity (Baltar, 2010). Neutral frothers are characterized by a molecule in which the polar group is a hydroxyl (-OH), generally in the form of alcohol or glycol. The most widely used types in flotation are: (1) methyl isobutyl carbinol (MIBC); (2) pine oil and (3) polyglycolic ethers.

Froth stability level depends, among

other factors, on the type of frother used. Stable froths favor recovery, while unstable ones produce concentrates with higher grades. This study aims to determine the influence of the main frothers on bubble characteristics in the pulp zone and froth produced. Experimental results allow comparing the action of these frothers in terms of bubble size stability in the pulp zone, surface tension lowering, froth stability and increased recovery of naturally hydrophobic minerals.

2. Experimental

2.1 Material

Reagents

Three frother reagents were used: (a) Methyl isobutyl carbinol (MIBC),

manufactured by CYTEC; (b) Pine oil, from Hercules Yarmor F and (c)

Glycolic ether – manufactured by Flomin Inc.

Mineral Samples

Highly pure mineral samples of graphite and calcite were used for the flo-

tation tests. Vonder graphite and calcite from the Brejuí mine located in Currais

Novos (Rio Grande do Norte, Brazil) were used.

Equipment

(a) Hydromess system, composed of a photoelectric probe and glass capillary tubes with internal diameter of 0.8 mm, used to measure bubble size;(b)

Modified Hallimond tube, 1” diameter, equipped with porous plate (10 µm), was used in flotation tests; (c) K100 Krüss tensiometer, used to measure surface

tension of the solutions and (d) MALVERN Mastersizer 2000 Laser Particle Size Analyzer, used to characterize the graphite and calcite.

2.2 Methodology

Determination of Bubble Size

The Hydromess® 1.40 system was used to determine mean bubble diameter. The device is equipped with data acquisition software and a photoelectric probe, composed of two luminous cells and two phototransmitters coupled to a glass capillary tube.

Constant-speed suction, produced

at the end of the glass capillary tube, generates a collection of bubbles that pass through the probe (Figure 1). Luminous cell 1, located at a distance “a” of 2 mm from luminous cell 2, with an inclination of 90° between them, emits light beams to phototransmitters aligned with the luminous cells. They detect the begin-

ning and end of the cylindrical ellipsoid (deformed bubble) inside the glass capillary tube, based on the refraction index of the phases.

The readings of captured bubbles are sent by electrical signal based on the difference between the refraction indices of the phases present.

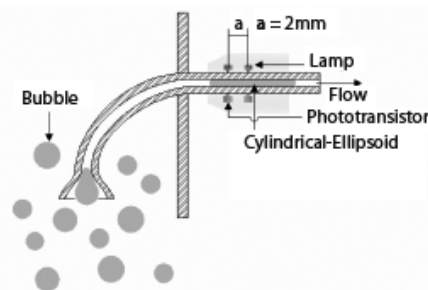


Figure 1 Photoelectric suction probe to capture air bubbles and cylindrical ellipsoid formation in the glass capillary tube (Paulo et al., 2003).

The system is composed of a glass capillary tube, photoelectric probe, flotation column, escape valve to control suction speed of the aspirated bubbles, vacuum pump coupled to a Kitasako flask to generate a vacuum and bubble suction in the glass capillary tube immersed in the solution, porous plate aerator

and rotameter to control air flow into the system, as illustrated in Figure 2.

Distilled water was used in the tests. Suction speed was adjusted to values of 1.5 m/s with the aid of a vacuum pump and air flow was maintained constant at 122 cm³/min for all assays.

The influence of frother concentration at the 0 - 50 ppm range was determined. Next, pH was varied between 2 and 10, using a constant concentration of 10 ppm for all assays. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were used to adjust pH levels.

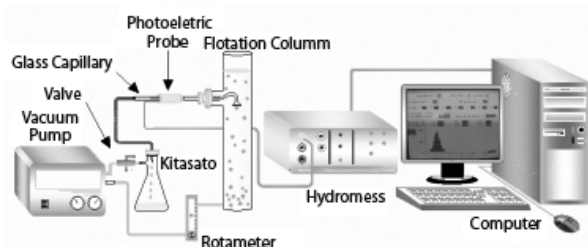


Figure 2
Apparatus to determine mean bubble diameter using the Hydromess technique in a flotation column.

Flotation Tests

Flotation tests were conducted in a modified Hallimond tube, with a vacuum pump for aeration and a rotameter to control air flow, in addition to a kitasato flask connected to the vacuum pump

with a funnel to filter the floated mass. Operational conditions were kept constant during all flotation tests, as follows: air flow of 122 cm³/min, distilled water with characteristic pH (around 5.8), condition-

ing time and flotation time fixed at 1 minute. Frother concentration ranged from 0 to 50 ppm. Floated material was filtered in a vacuum pump and then oven-dried at 85°C for later weighing.

3. Results and discussion

Surface tension lowering

The frothing ability of a surfactant can be indirectly evaluated by determining surface tension. Higher surface tension lowering indicates

greater frothing intensity (Xia and Peng, 2007). Results obtained for the surface tension of pure reagents are presented in Table 1.

Results indicate that higher surface tension lowering was obtained with MIBC, suggesting greater frothing power for this surfactant.

Table 1
Surface tension of each of the frother samples.

FOAMING	SURFACE TENSION, MN/M
MIBC	22.7
Pine oil	30.2
Polyglycolic ether	29.6

Bubble size in the pulp zone

The ability to avoid bubble coalescence was determined by mea-

suring mean bubble size as a function of the concentration of each of

the frothers. Results are displayed in Figure 3.

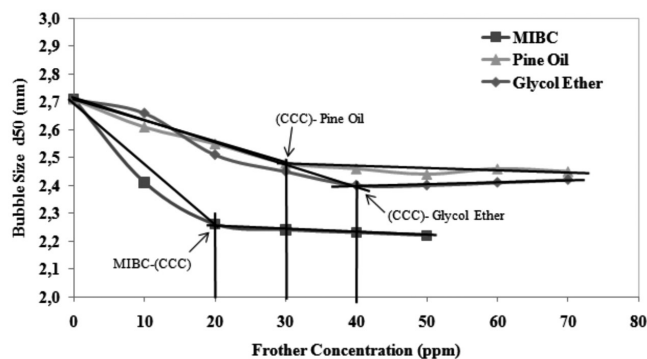


Figure 3
Influence of frother concentration on mean bubble diameter.

Results (Figure 3) show that the smallest bubble sizes were obtained with MIBC, indicating that this reagent is the most efficient in preventing coalescence. Critical coalescence concentration (CCC) for MIBC was found at a concentration of 20 ppm. At this concentration, minimum bubble size was stabilized at approximately 2.2 mm. The reduction in mean bubble size is around 18% when compared to mean diameter in the absence of frother. Cho and Laskowski (2002) observed bubbles with a mean size of 2.4mm in the absence of frother. The CCC of MIBC was observed with approximately 8.0×10^{-5} mol.L⁻¹, promoting a reduction in mean bubble size of around 28%. The CCC of polyglycolic

ether was observed at a concentration of around 40 ppm, with a minimum bubble size of 2.4 mm, representing a reduction of 11% compared to mean bubble size in the absence of frother. Pine oil was the least able to prevent coalescence. The CCC was obtained at 30 ppm of surfactant, with minimum bubble size of around 2.5 mm, a 10% reduction in mean size compared to that obtained without frother. Szyszka et al. (2006) found CCC for pine oil at a concentration of 0.16 mmol/dm³, with bubble diameter corresponding to CCC of approximately 3.3 mm, while Gupta et al. (2007) observed CCC of pine oil at around 10 ppm. Results are summarized in Table 2.

The results of the influence of pH on

mean bubble size in the presence of each of the frothers are shown in Figure 4.

Results presented in Figure 4 confirm the greater ability of MIBC in preventing coalescence, given that bubbles in the presence of this frother exhibited the lowest sizes over the entire pH range tested. Mean sizes varied between 2.2 and 2.4 mm, with the largest size observed near natural pH.

Pine oil demonstrated the greatest sensitivity in terms of pH variation; it seems to be more efficient in preventing coalescence in acid medium. By contrast, glycol ether was the most resistant to pH variation, with a slight tendency to being more efficient in preventing coalescence in alkaline medium.

FROTH	CCC (ppm)	MINIMUM BUBBLE SIZE (mm)
MIBC	20	2.2
Polyglycolic Ether	40	2.4
Pine Oil	30	2.5

Mean bubble size in the absence of frother: 2.7 mm.

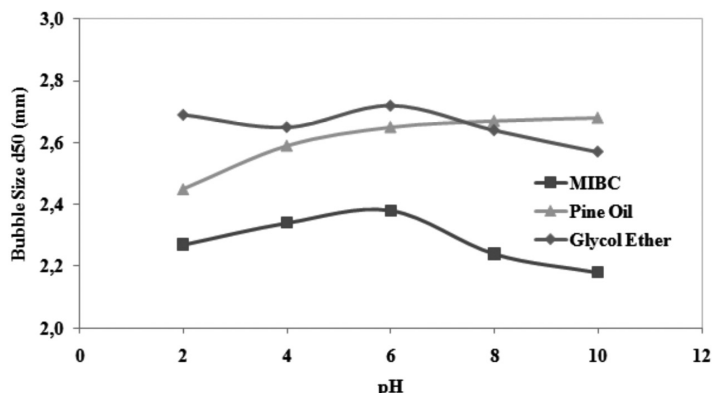


Table 2 Ability of frothers to prevent bubble coalescence.

Figure 4 Variation in mean bubble size as a function of pH for the frothers studied.

Froth Stability

Froth stability is an important parameter for flotation. Stable froths favor recovery, but hinder selectivity owing to higher mechanical entrainment of gangue particles.

Considering that calcite is a mineral with a hydrophilic surface, in the absence of collector it could only be present in froth through mechanical entrainment. These tests were conducted to quantify the entrainment of hydrophilic particles in froth.

Froth Stability

Flotation of hydrophobic minerals is usually conducted with the simple addition of frother. Flotation tests were car-

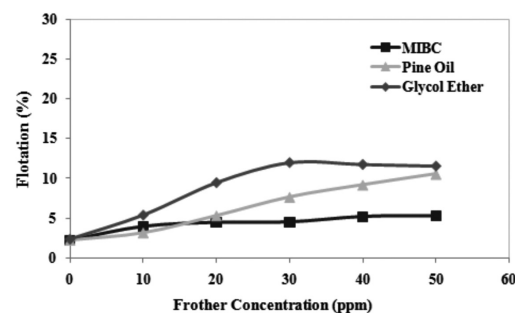
ried out with a graphite sample in order to observe the collector action of the frothers on the flotation of naturally hydrophobic

minerals. The results are presented in Figure 6. Around 65% of graphite floated without the addition of any surfactant.

There is a direct relationship between froth stability and amount of fine particles entrained (Hu et al., 2009). The more stable the froth, the greater the amount of hydrophilic particles trapped between the bubbles and, consequently, the less selective the frother. Figure 5 shows the influence of frother concentration on calcite entrainment. Results suggest that the most stable froth is produced by EPG. As expected, entrainment

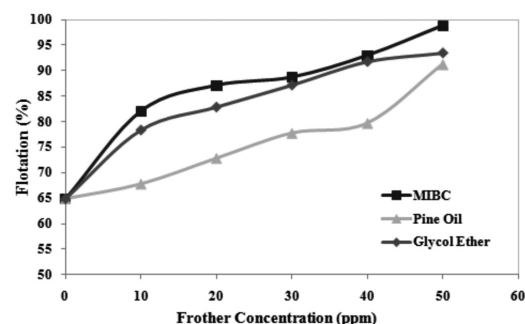
The addition of frother increased the floatability. The best results were obtained with MIBC, achieving nearly 100% recovery

Figure 5
Influence of frother concentration on mechanical entrainment of calcite particles trapped in froth.



with the addition of 50 ppm of frother. EPG and MIBC obtained similar results, while the lowest recovery was observed for pine oil.

Figure 6
Influence of frother concentration on graphite flotation.



4. Conclusions

The results showed the performance of three frothers in terms of avoiding coalescence between bubbles, frothing ability, froth stability and flotation of naturally hydrophobic minerals.

MIBC has greater frothing power. Hydromess system technique can be used to measure bubble size in a two-phase system. The equipment proved to be efficient with respect to reliability of data reading and result reproducibility. MIBC was the most efficient in preventing coalescence between

bubbles over the entire pH range tested. Critical coalescence concentration (CCC) was reached at a concentration of 20 ppm, with bubble diameter stabilizing at around 2.2 mm, representing an 18% reduction compared to bubble sizes produced in a system with no additive. No significant differences were observed between polyglycolic ether and pine oil efficiency in preventing coalescence. However, pine oil is more sensitive to pH variation, functioning better in acid medium.

MIBC showed the highest selectivity potential. Around 5% of hydrophilic calcite particles were entrained with a concentration of 50 ppm. On the other hand, glycol ether promoted the highest entrainment, suggesting that this frother produce more stable froths. Flotation of naturally hydrophobic minerals showed greater efficiency than MIBC. The addition of 50 ppm increased graphite recovery from 65% to approximately 99%, while pine oil promoted the lowest increase in graphite recovery.

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