



Effect of surfactant type on the entrainment factor and selectivity of flotation at laboratory scale



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ABSTRACT

In froth flotation, minerals report to the concentrate either by true flotation or entrainment. Previous research reported that flotation by entrainment is related to the amount of water that is transported to the concentrate (water recovery). On the other hand, plant operational experience indicates that frother type can be used to control the amount of water in the concentrate. In this work, a relationship between surfactant type and flotation by entrainment was obtained at laboratory scale using a batch flotation tests. The results indicate that the structure and molecular weight of surfactant influences the water reported to the concentrate, which is also related to the recovery of hydrophilic particles. The relationship between entrainment and water recovery is not unique, but depends on surfactant type. When comparing grade-recovery curves generated with different surfactants, the results show that there is an important effect of the surfactant type on the selectivity of the process.

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1. Introduction

Frothers, also called surfactants, are surface active reagents used in flotation to generate small bubbles and produce stable froths (Finch et al., 2006, 2008; Grau et al., 2005; Kracht and Finch, 2009). They can be grouped, according to their structure, into four families: aromatic alcohols; alkoxy types; aliphatic alcohols; and polyglycols, which may be represented by the general formula $R(X)_nOH$ where R may be H or C_nH_{2n+1} and X corresponds to CH_2CH_2O , $CH_2(CH_3)CHO$, or $CH_2CH_2(CH_3)CHO$, for polyethylene, polypropylene, and polybutylene respectively (Tan et al., 2005b).

Frother structure affects both bubble and froth behaviour, and also flotation performance (Pugh, 2000; Cho and Laskowski, 2002; Finch and Zhang, 2014; Tan et al., 2004, 2005a). Zhang et al. (2012), for instance, presented a link between frothers' ability to reduce bubble size, expressed in terms of critical coalescence concentration, CCC, and frother structure, characterised by hydrophile-lipophile balance (HLB) numbers. They found a correlation between CCC_{95} , i.e., the frother concentration producing a 95% reduction in bubble size from water alone, and the HLB number for different molecular structures. Nasset et al. (2012) had previously

showed that CCC_{95} values correlate against HLB/M_w , where M_w is the frother molecular weight.

Laskowski et al. (2003), showed an effect of frother structure on froth behaviour, expressed as changes in the dynamic foamability index (DFI) for frothers of different molecular structure or molecular weight. The DFI, on the other hand, can be related to the water flotation rate constant (Melo and Laskowski, 2006), which translates into an effect of frother type on water transport to the froth (Melo and Laskowski, 2007). Moyo et al. (2007) found, at laboratory scale, that frothers have an effect on the water carrying capacity, i.e., on water recovery. Their results show that for a given gas holdup in the collection zone, different water carrying capacities can be obtained by changing frother type in the system.

By using sulphur containing frothers, analogous to the common oxygen containing frothers, Harris and Jia (2000) showed that changes in the HLB number, even for the same frother molecular structure, has an effect on concentrate grade and recovery.

1.1. Frothers and entrainment

In flotation there are two mechanisms by which particles report to the concentrate: true flotation, which corresponds to particles that float attached to bubbles; and entrainment, where particles are drawn to the froth and concentrate along with the water that accompanies the bubbles. While true flotation is a selective process, entrainment is not, and has a negative effect on the selectivity

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of the process, lowering the concentrate quality (Smith and Warren, 1989; Ross, 1990). The most important factors that affect entrainment are: recovery of water, particle size, solid content in the pulp, froth structure, froth residence time, and particle specific gravity (Savassi et al., 1998; Wang et al., 2015). Froth structure is affected by frother type (Savassi et al., 1998), which may explain the effect of frother type (structure) on flotation selectivity that has been reported in the literature (Klimpel and Isherwood, 1991; Gupta et al., 2007, 2009).

Trahar (1981) studied entrainment and found a relationship between entrainment and water recovery. The correlation can be described by a convex curve that tends to linearity in the case of fines (Zheng et al., 2006), which can be expressed as:

$$R_{ENT} = ENT \cdot R_W \quad (1)$$

where ENT corresponds to the entrainment factor.

In a recent study, McFadzean et al. (2016) reported changes in the entrainment factor for different frothers, with a ratio of about 3:1 between the highest and lowest entrainment factors, determined for a polypropylene glycol and MIBC. The authors suggest that the behaviour is explained by changes in the froth structure and its capacity to hold small, hydrophilic particles. The current work supports their finding by showing that the entrainment factor is not unique, but depends on surfactant type. The frother types studied in this case correspond to aliphatic alcohols and polyethylene glycols.

2. Experimental

A series of flotation tests was carried out in a 5.1 L Labtech-Essa flotation cell. The amount of water reported to the concentrate was recorded for each flotation time interval in order to compute water recovery. Two kind of kinetics tests were performed: first with hydrophilic particles (quartz), and later with a synthetic ore, composed of quartz and a copper concentrate.

The quartz was crushed and ground down to 90% -400#. The particle size was then determined using laser diffraction analysis, giving an average value of d_{50} equal to 10.3 μm , with a standard deviation of 3.2%. The copper concentrate, on the other hand corresponded to a final concentrate provided by a Chilean mining company. The XRD showed that the concentrate was composed mainly by chalcopyrite, with some pyrite and little amounts of silicate. The copper content of the concentrate was 29.3%. Since the samples corresponded to a final concentrate, the particle size d_{50} was also fine, under 10 μm .

2.1. Reagents

The surfactants were selected in order to represent two families of different molecular structure: aliphatic alcohols and polyethylene glycols, PEG, with the following generic formula, $\text{H}(\text{OCH}_2\text{CH}_2)_n\text{OH}$. Table 1 shows the reagents used, all of them from Sigma Aldrich. HLB numbers are included as a scale of surfactant solubility (Rao and Leja, 2004). The higher the HLB the more

water-soluble (hydrophilic) the reagent. The HLB numbers in Table 1 were calculated using the Davies method (Davies, 1957).

In the case of the kinetics tests with quartz and concentrate (synthetic ore), the collector used was Aeron 343 Xanthate, from Cytec, which corresponds to sodium isopropyl xanthate. The pH was adjusted with lime (CaOH_2).

2.2. Procedure

A first series of tests was performed only with quartz, at 17.8% solids, natural pH and 0.5 mmol/L of surfactant. The concentration was chosen so that all the surfactants produced enough froth in the system to perform the measurements. The impeller speed was set at 450 RPM, and a superficial gas velocity, J_g , of 0.56 cm/s was used. The cell was scrapped every 15 s to collect froth at the following time intervals: 0–1, 1–2, 2–4, and 8–12 min. In order to avoid any interference with the froth when adding reposition water, this was done through an orifice below the slurry-froth interface, using a peristaltic pump. The conductivity of the aerated slurry was measured to determine the gas holdup in the cell (Gomez and Finch, 2007).

After 12 min of flotation, the product, collected in trays, was weighted, filter, dried and weighted again to obtain both water, and mineral recovery. The water recovery was calculated dividing the mass of water collected in each tray by the mass of water in the cell at the beginning of the test.

In the case of the tests performed with synthetic ore, the procedure was the same, except that 85 g of concentrate were added, and a collector dose of 30 g/ton. The pH was adjusted with lime to 10.5. The dried samples were analysed in order to determine the content of copper and quartz, and to calculate the valuable (copper) and gangue (quartz) recovery. The surfactants used in these tests were Octanol and PEG300.

For those two reagents, images of 2D foams (water-air) were generated to compare their foamability. The foam was produced in an acrylic cell of 20 cm height, 15 cm wide and 1 cm depth equipped with a slot sparger for air dispersion, at a J_g of 2.5 cm/s. Note that since the system did not have solids, the gas flow rate had to be increased to generate foam compared to the flotation cell.

3. Results and discussion

Fig. 1 shows the recovery of hydrophilic particles (quartz) by entrainment against water recovery for the series of tests per-

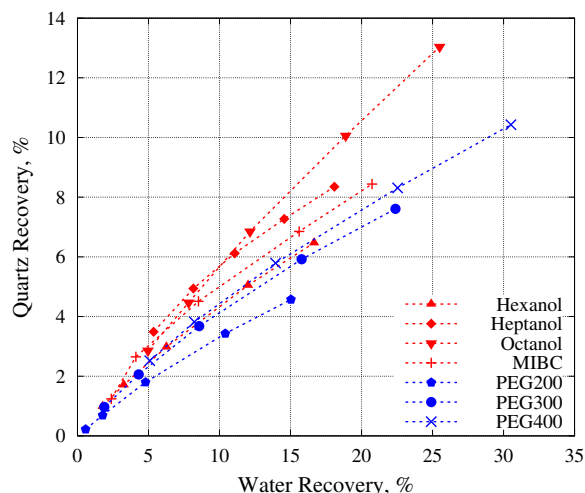


Fig. 1. Quartz recovery vs. water recovery for all surfactants.

Table 1
Surfactants.

Surfactant	Molecular weight	HLB number
Hexanol	102	6.1
MIBC	102	6.1
Heptanol	116	5.6
Octanol	130	5.1
PEG200	200	10.9
PEG300	300	11.7
PEG400	400	12.5

formed only with quartz. It can be observed that the relationship between recovery by entrainment and water recovery is not unique.

For both families of surfactants, alcohols and polyethylene glycols, the recovery by entrainment increases with the molecular weight of the reagent. If one observes the curve corresponding to MIBC, it shows a higher recovery of quartz than Hexanol, despite de fact of having the same molecular weight, formula and HLB number. This may be due to the branched molecular structure of MIBC, compared to the linear structure of Hexanol. The results show that alcohols tend to report more hydrophilic particles to the concentrate than polyethylene glycols. For each surfactant, the entrainment factor, ENT, was determined. The results are summarised in Table 2, along with the values of gas holdup determined for each reagent.

The entrainment factors were plotted against HLB numbers as shown in Fig. 2. The data points in Fig. 2 appear clustered according to reagent family, showing that polyethylene glycols are more soluble (higher HLB numbers) and more selective than alcohols.

Since HLB numbers do not account for molecular weight (Mw), which means that two reagents with different molecular weight could have the same HLB number, it is of interest to compare the results against HLB/Mw (Fig. 3), as it has been done by other authors (Nesset et al., 2012; Zhang et al., 2012).

Although in Fig. 3 the two reagent families cover a similar range of HLB/Mw, the data does not show a consistent trend. Nevertheless, alcohols and polyethylene glycols can be still separated into two different groups.

One reagent of each family was selected for comparison: Octanol and PEG300 (Fig. 4). The selection was based on the fact that both have the same value of HLB/Mw (0.039), and the gas holdup obtained with both surfactants was very similar, 10.3 and 10.2% respectively. The data presented in Fig. 4 corresponds to quadruplicates for each reagent.

The slopes of the curves presented in Fig. 4 correspond to the entrainment factors of Octanol (0.49) and PEG300 (0.33). The results show that the entrainment factor is not unique, but depends on the surfactant type. This implies that for a given amount of water reported to the concentrate, Octanol reports a higher quantity of hydrophilic particles to the concentrate than PEG300, which should have an effect on flotation selectivity.

In order to complement the results and to contribute to the analysis, 2D foams (water-air) were generated for Octanol and PEG300 at 0.5 mmol/L. The 2D foams are presented in Fig. 5. The actual height of the area shown in the images is 10 cm. It can be seen that PEG300 generates twice as much foam as Octanol, which means that there is a marked difference in foamability between both reagents at the tested concentration.

It is interesting that though PEG300 shows a higher foamability than Octanol, it reports roughly the same amount of water than Octanol to the concentrate (Fig. 4). This suggests that the difference in foamability is not directly related to the amount of water in the foam, but to a combination of bubble properties, foam/froth structure and behaviour.

Table 2
Entrainment factor and gas holdup for each surfactant.

Surfactant	ENT	Gas holdup, %
Hexanol	0.41	9.4
MIBC	0.43	9.6
Heptanol	0.51	11.2
Octanol	0.49	10.3
PEG200	0.32	8.5
PEG300	0.33	10.2
PEG400	0.39	10.5

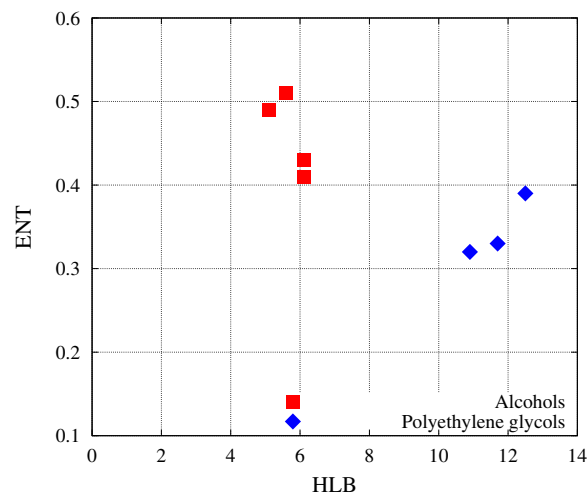


Fig. 2. Entrainment factor vs. HLB for alcohols and polyethylene glycols.

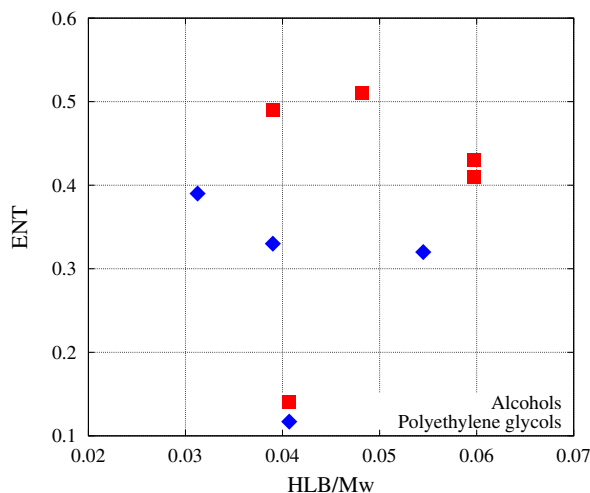


Fig. 3. Entrainment factor vs. HLB/Mw for alcohols and polyethylene glycols.

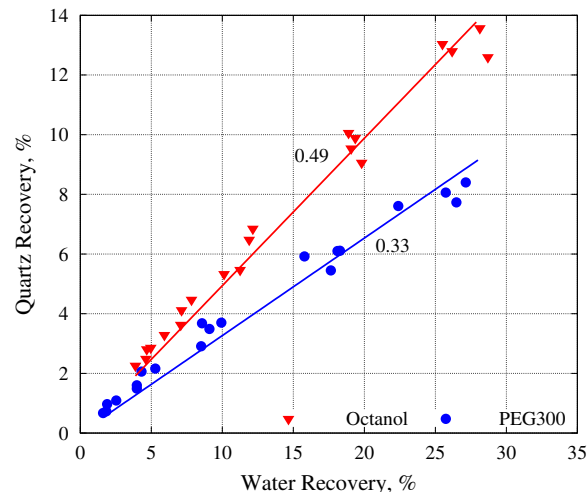


Fig. 4. Quartz recovery vs. water recovery, for Octanol and PEG300 (entrainment factors are shown).

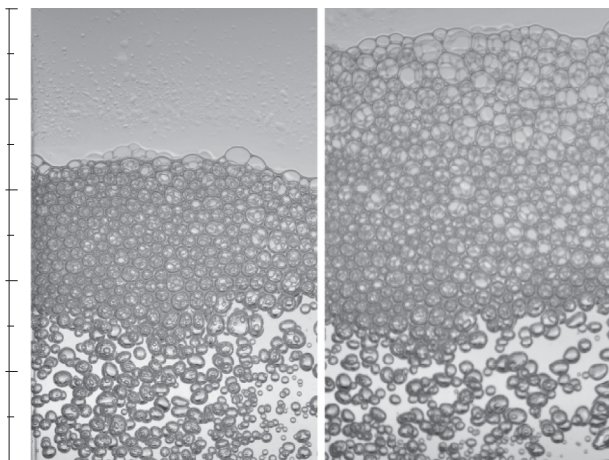


Fig. 5. 2D foams for Octanol (left) and PEG300 (right).

To the naked eye, the first 3 cm of foam generated with PEG300 in Fig. 5 look very similar to the foam generated with Octanol; however, the foam generated with PEG300 reaches more than 6 cm height whereas with Octanol the foam height is only 3–4 cm. On the other hand, the top layers of both foams are very different. The one corresponding to PEG300 looks drier, and composed by bigger bubbles, than the top layer of foam generated with Octanol. This means that bubbles generated with PEG300 (at 0.5 mmol/L) take longer to burst, which implies that their surface has different properties than bubbles generated with Octanol. This may be due to the difference in the structure of alcohols and polyethylene glycols. As suggested by Harris and Jia (2000) when comparing MIBC and Dowfroth250, alcohols have only one OH group that interacts with water molecules to form an oriented monolayer at the bubble surface, while polyglycols have two OH groups and several oxygenated units that would make them tend to lie flat at the surface, with a possible increase in the surface viscosity. The results allow entertaining the idea that the increase in surface viscosity takes place in a very thin water layer bounded to the bubbles, but without increasing the viscosity of the water in the Plateau borders, otherwise it would be more difficult for the particles to return from the froth to the collection zone, increasing the entrainment factor.

In order to confirm the effect of surfactants on selectivity, flotation kinetics tests were carried out with both reagents, Octanol and PEG300, with a synthetic mineral composed of quartz and copper concentrate. The results are presented in Fig. 6 in the form of grade-recovery curves.

The results clearly show that the grade-recovery curve is shifted to the right when changing the reagent from Octanol to PEG300. This means that the flotation test performed using PEG300 was more selective than with Octanol, which confirms the observation made when analysing the results of entrainment factors. Since the concentrate used to prepare the synthetic mineral was received dry and to perform the tests it was resuspended, without any further treatment to remove eventual particle oxidation, the maximum recoveries differ from 100%. The difference in the maximum recovery of copper for both reagents is related to the flotation kinetics, which was also affected by the change in surfactant, presumably due to the difference in foamability between Octanol and PEG300.

The effect that surfactant has on the selectivity of the flotation process, through the modification of the entrainment factor, implies that one could have a system with a stable froth, and a high water recovery due to large amounts of water reported to the froth and concentrate, and yet produce a concentrate of good quality,

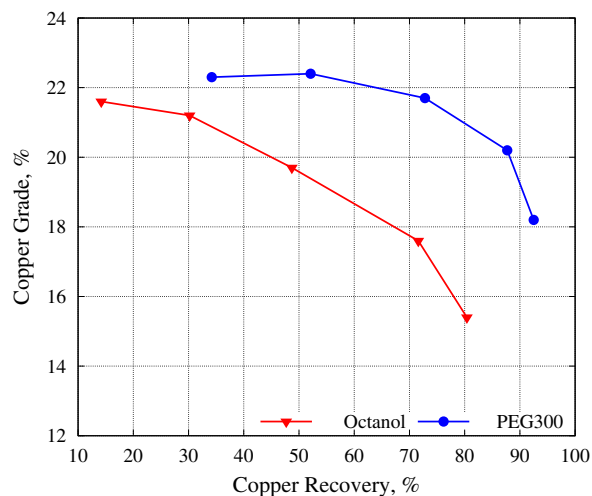


Fig. 6. Grade-recovery curves for Octanol and PEG300.

depending on the frother being used. Based on these results, the recommendation is to consider frother selection as an alternative to improve not only gas dispersion and froth stability in the cell, but also selectivity and, therefore, concentrate quality.

4. Conclusions

The results show that the entrainment factor, ENT, calculated as the slope of the curve of recovery of hydrophilic particles vs. water recovery, is not unique, but depends on the surfactant type. In the case of Octanol and PEG300, it was observed that the latter has a lower entrainment factor than Octanol (0.33 vs. 0.49) for the conditions studied, e.g., reagent dosage and particle size distribution. This means that for a given amount of water recovered, different amounts of hydrophilic particles get to the concentrate, affecting the quality of the product. When running tests with a synthetic mineral, composed by quartz and copper concentrate, the different entrainment factors observed for Octanol and PEG300 translate into a shift of the grade-recovery curve, showing clearly an effect of surfactant type on flotation selectivity. Although further testing at industrial scale is still needed, one can speculate that the right selection of frothers may improve not only the gas dispersion and froth stability in the cell, but also improve the grade-recovery curve, giving a new role to the frothers.

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