Gas dispersion measurements in microbubble flotation systems

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This work summarises results of microbubble dispersion parameters in a controlled laboratory system. The effects of the temperature and frother concentration (MIBC) on the gas holdup (d), superficial area flow density (Jg), superficial air velocity (Jg), bubble Sauter diameter (d32) and air liberation efficiency were studied. The results obtained with natural water show that (a) increasing the temperature from 10 to 30 °C significantly increased the bubble Sauter diameter from 80 to 150 μm, improving the air liberation (bubble formation) and (b) increasing the superficial air velocity from 0.01 to 0.06 cm/s enhanced the air holdup from 0.4% to 1.8%, the Sauter diameter from 60 to 120 μm and the bubble surface area flux from 5 to 25 s⁻¹. The experimental results also showed that frother addition (MIBC) reduced the Sauter diameter, while increasing all other variables.

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1. Introduction

In the last few decades, the use of microbubbles that are generated by depressurisation of dissolved air in water (DAF) has attracted interest due to its possible applications. In particular, in the mineral and liquid effluent treatments, and especially for the flotation of fine particle sizes (Rubio et al., 2002, 2003; Capponi et al., 2005).

For efficient mineral flotation recovery of coarse and fine particles, there must be an optimal bubble size and bubble size distribution, whereby the number of particles “captured” by bubbles reaches a maximum (Yoon 1993, 2000; Rubio et al., 2006). For the recovery of small particles, the flotation cell should have fine bubbles or microbubbles suitable to catch these particles (Yoon, 2000; Rubio et al., 2003; Zhou et al., 1997). Unfortunately, this does not occur in practice and the flotation cells that are commercially available do not provide the required bubble size distribution.

According to some authors (Capponi et al., 2005), using microbubbles (an injection of 30–100 μm) in addition to conventional bubbles (between 600 and 2500 μm) for the flotation of fine particles (Cu and Mo sulphides) led to slight improvements in recovery (2%) and in flotation kinetics (rate constant, 3% higher) on the laboratory scale. It was claimed that by decreasing the bubble size distribution, the bubble surface area flux and the fine particle capture were both increased. However, the use of “single” microbubbles (without coarse bubbles) in ore flotation (fine and coarse particles) has proven unsuccessful. This has been attributed to problems with the low lifting power of these bubbles, especially at high solid concentrations (Solari and Gochin, 1992; Peng et al., 2005; Capponi et al., 2005).

Conversely, the use of flotation is showing great potential in effluent treatment due to the high throughput of modern equipment, low sludge generation and the high efficiency of the separation schemes already available. Examples can be found in the treatment of contaminated water (oils, pigments, heavy metals removal), in the recovery of proteins, sugar impurities, inks and resins, as well as in microorganism separation and the treatment of sewage and sludge, turbidity, colour, suspended solids, and micro-organisms (Rubio et al., 2002; Englert et al., 2009; Carissimi et al., 2007).

2. Background

The micro-bubble generation equipment is well described in several papers (Rodrigues and Rubio, 2003), so only a brief summary will be described in this section. An important step in micro-bubble formation is air dissolution. The air dissolution process obeys Henry’s law, where the kinetics are mass transfer dependent and highly influenced by the pressure and design of the saturator (3–6 atm). During the generation of the bubbles, the energy transferred is determined by the superficial tension and the pressure gradient around the constriction valve (Rodrigues and Rubio, 2003).

Bubbles are formed by a reduction in pressure of water that has been pre-saturated with air and occurs at pressures higher than atmospheric. The supersaturated water is forced through needle valves or special orifices and clouds of bubbles 0.02–0.1 mm in diameter are produced just down-stream of this constriction.
A disadvantage of the DAF process is the high cost of water saturation required for bubble formation. However, it has been shown that bubble generation is possible at working pressures lower than 3 atm by lowering the air/liquid surface tension in the saturator. It was concluded that very low concentrations of surfactants or flotation collectors are required to operate the DAF units at 2–2.5 atm (Féris et al., 2001). This process can drastically reduce the cost of energy and optimisation of the whole process (Féris and Rubio, 1999; Féris et al., 2001).

These results were explained as cavity formation occurring as a result of a minimum energy, $\Delta F$ (joules), being transferred to the liquid phase and forming bubbles, following the equation (Takahashi et al., 1979):

$$\Delta F = \frac{16 \pi \cdot \gamma^3}{(P_0 - P_a)^2}$$

where $\gamma$ is the air/water surface tension (Nm$^{-1}$), $P_a$ is atmospheric pressure (atm or Pascal units), and $P_0$ is the saturation pressure (atm or Pascal units).

Thus, less energy is required to generate micro-bubbles with a lower air/liquid interfacial tension, or with greater pressure differences between the liquid phase and atmosphere. Accordingly, in reducing $\gamma$, the liquid/solid attrition will be reduced and the flow fluid velocity and the bubble formation will become faster.

Under light turbulent conditions, Libra (1993) observed that oxygen dissolution in water decreases with anionic surfactants. This effect is reduced in turbulent conditions when the bubble surface area generation increases after improvement of mass transfer.

Another effect of the surfactant is the diminution of coalescence and the increase in the superficial area. However, not all surfactants prevent coalescence. An example is the use of non-ionic surfactants (commonly used as deformers), which are used to avoid bubble surface area generation (Zlokarnik, 1979).

The nucleation of air may be homogeneous or heterogeneous. In the case of homogeneous nucleation, the air nucleation around the molecular cavities precedes micro-bubble formation, and may be favoured by the presence of high concentrations of gas close to this cavity. Commonly, heterogeneous nucleation is present on the surface of the constrictor because the surface of the needle valve serves as a site for air nucleation. Additionally, the presence of particles favours air nucleation.

In the last decade, the froth flotation industry has used a variety of sensors to evaluate the aeration quality of their processes. Conventional bubble sizes are used (500–2500 m). Several prototypes of sensors and techniques have been developed for evaluating the bubble diameter in conventional flotation cells; although the most commonly used methods involve image analysis. However, measuring the Sauter diameter of a micro-bubble dispersion has it owns difficulties and requires special equipment.

Rodrigues and Rubio (2003) developed a device for measuring the bubble diameter of micro-bubbles in a two-phase system. Their system included a bubble capture cell, a microscope and a digital camera. One of the more important details of this system is that turbulent movements must be avoided, as they make it difficult to obtain a clear picture.

Gas holdup is the volume percentage or volume fraction of air within the collection zone. Ahmed and Johnson (1989) found that gas holdup favours flotation kinetics because it increases the number of bubbles, and therefore the superficial area available for particle collection. Frequently, this variable is estimated using a simplified mathematical model of Maxwell (Eq. (2)), which requires the measurement of the electrical conductivities of the pulp ($k_{ps}$, liquid–solid dispersion) and the dispersed phase ($k_{gse}$, liquid–gas–solid). Tavera et al. (1995) developed an industrial air holdup sensor, which has been used in off-line industrial measurements.

$$e_g = \frac{1 - k_{ps}/k_{gse}}{1 + 0.5 \cdot k_{ps}/k_{gse}}$$

(2)

The rate of bubble surface area passing through a cross section of the flotation cell is also known as the bubble surface area flux (Gorain et al., 1997). The importance of this variable is its relationship to the flotation efficiency. It can be estimated from the Sauter diameter and the superficial gas velocity, as:

$$S_b = \frac{6 \cdot J_s}{d_{32}}$$

(3)

To evaluate the aeration state in conventional flotation cells, Gomez and Finch (2007) proposed a gas velocity sensor, which was constructed with a plastic tube large enough to be introduced in the collection zone and to reduce the bubble sampling biases. The authors explained that the collected bubbles reach the liquid surface in the tube and burst. If the tube is closed, the pressure increases; this pressure is registered by a data acquisition system and serves to compute the superficial air velocity ($U_j$). Thus, the air flow ($Q_g$) is commonly estimated measuring $J_s$ and multiplying by the cross-sectional area of the flotation cell ($A$).

$$Q_g = J_s \cdot A$$

(4)

Matiolo et al. (2011) have characterised the aeration variables ($J_g$, $S_b$, $d_{32}$ and $e_g$) with fine bubble sizes (470–1000 m) in a water/air system. The gas holdup and bubble size (and their distributions) were found to be strongly dependent on the concentration of Dowfroth 250 and the superficial gas velocity. A fairly linear relationship between the experimental $e_g$ and the bubble superficial area flux ($S_b$) was established, the results of which were compared to those calculated using drift flux analysis. No similar studies appear to be reported with micro-bubble size dispersions, (10–100 m) and accordingly, the main goal of this work is to contribute to the knowledge of this subject.

3. Experimental

The experimental setup was composed of two principal parts: the air-saturation water reactor and the flotation column. The saturation reactor was made of polyvinyl chloride (PVC; 80; 30 cm internal diameter; 140 cm high). This reactor was designed for saturated-water level control and internal pressure control; the internal pressure was kept constant at 54 psig ($\pm$0.2). To visualise the bubble dispersion, the flotation column was made with a

![Fig. 1. Experimental setup.](image-url)
translucent acrylic tube (2 m high and 0.095 m in diameter). Fig. 1 shows the experimental setup.

To facilitate bubble visualisation, the experimental program was conducted with a biphasic system (air–water), and eventually with an aqueous solution with MIBC surfactant. This solution was agitated for 5 min to stabilise the temperature and to homogenise the frother concentration. Afterward, the solution was introduced into the saturator with the aid of a hydro-pneumatic pump. The flow of water containing micro-bubbles was fed into the column at 1.75 m under the lip of the concentrate overflow. Measurements of the bubble diameters were made with ImageJ® software, which was programmed with a macro-program to handle and process the images. ImageJ is a free and versatile program package for image processing and manipulation that runs under Java. To obtain reliable results, several steps must be followed: (1) calibration of the image magnification, (2) finding a threshold for the image, and (3) analysis of the particles. To calibrate the software, a thin wire made of stainless steel (0.3 mm) was used as a reference and placed close to the photographic plane.

The software reports a list of computed objects (bubbles) and their corresponding diameters. This software makes it possible to select the bubbles that will be measured and that satisfy the restrictions defined by the user (threshold sphericity, minimum and maximum size, etc.). The software also shows a mask of the photograph, indicating the computed bubbles with filled circles. Once each arithmetical bubble diameter ($d_b$) was measured, Eq. (5) was used to estimate the Sauter diameter ($d_{32}$). Also, to obtain a representative Sauter diameter, the number of bubbles that needed to be counted was also evaluated.

$$d_{32} = \frac{\sum d_b^3}{\sum d_b}$$  \hspace{1cm} (5)

One of the goals of this work was to evaluate the air liberation efficiency. Unfortunately, it was not possible to measure the air concentration ($N_2$ and $O_2$) around the constriction valve. Instead, the dissolved $O_2$ was measured, and the assumption was made that the air liberation efficiency was approximately proportional to the $O_2$ liberation efficiency. This is strictly an approximation because the solubility of $N_2$ and $O_2$ are different. Thus, the $O_2$ liberation efficiency was estimated with Eq. (6). In order to estimate the nucleation percentage, the dissolved oxygen was measured before and after the nucleation process. These measurements were conducted at 10 cm preceding the constriction valve and 30 cm below the overflow lip of the flotation column. Fig. 1 indicates the position of the dissolved $O_2$ electrodes.

$$O_2_{liberation\%} = \frac{(O_2_{before} - O_2_{after}) \times 100}{O_2_{before}}$$  \hspace{1cm} (6)

where $O_2_{liberation\%}$ is the percentage of $O_2$ liberated on the constriction valve, $O_2_{before}$ is the ppm of $O_2$ before the valve, and $O_2_{after}$ is the ppm of $O_2$ after the valve.

### 4. Results and discussion

Fig. 3 (a) shows a typical image of a microbubble dispersion and its corresponding mask (b) indicating the bubbles to be computed. It was observed that the typical Sauter diameter was smaller than 150 μm, confirming that this work was conducted with microbubbles.
Fig. 4 shows that after counting 450 micro-bubbles, the standard deviation becomes relatively constant, which implies that this number is sufficient. Notably, in the case of conventional flotation processes, the relatively large bubbles can be deformed when rising in a turbulent regime, forcing the operator to compute between 5 and 10,000 bubbles to obtain a good Sauter diameter (Gomez and Finch, 2007). In contrast, the micro-bubbles are highly spherical, resulting in fewer being required to obtain a representative Sauter diameter.

4.1. Effect of temperature

Fig. 5a shows the effect of the superficial air velocity on the air holdup. These experiments were conducted without the addition of a frother. These results indicate that the air holdup increased as the superficial air velocity increased, reaching values close to 1.8%. This effect appears to be due to the greater accumulation of air in the collection zone as more air is fed into the column. In addition, the air holdup decreased as the temperature increased.
because the bubble diameter increases jointly with the rising velocity. Similar to conventional flotation processes, these results show that when the temperature was kept constant, a linear relationship between the air holdup and the superficial air velocity is attained (Finch et al., 2000; Massinaei et al., 2009).

The temperature and superficial air velocity increased the bubble size, as shown in Fig. 5b. The temperature effect on the bubble diameter is well known because the volume of a gas increases with temperature. In the case of the superficial air velocity, the aperture of the needle valve generated turbulent conditions, which facilitated bubble coalescence and increased the Sauter diameter. The experimental data was fitted with a power-law curve and a similar relationship was observed by Nesset et al. (2006) with mechanical flotation machines.

Fig. 6a shows the relationship between the air holdup ($\varepsilon_g$, %) and the bubble surface area flux ($S_b$, s$^{-1}$). Both variables are favoured by the superficial air velocity, as illustrated in Fig. 6b. It was observed that at 10°C with a superficial air velocity of 0.06 cm/s, the bubble surface area flux reached 32 s$^{-1}$. Notably, for the equivalent superficial air velocity, these values are proportionally higher than those obtained with conventional bubble sizes. This was concluded by observing the experimental data obtained from Nesset et al. (2006), where with industrial mechanical cells and conventional bubble sizes and a more elevated $J_g$ (0.1 cm/s), the bubble surface area flux was close to 10 s$^{-1}$.

Fig. 7 shows that the temperature and superficial air velocity had a significant effect on the liberation efficiency. The temperature enhances the liberation efficiency, while the superficial air velocity decreases it. The effect of the temperature may be explained in terms of decreased solubility, i.e., the increase in the percentage of oxygen nucleated. For example, Ramalho (1996) showed an air dissolution decrease from 29.2 to 15.7 cm$^3$/L when temperature increased from 0 to 30°C at 1 atm. The effect of the superficial air velocity on the liberation efficiency can be explained by considering the turbulent conditions around the constriction valve. Takahashi et al. (1979) explained that the energy transferred to nucleate air is proportional to the pressure gradient around the constriction valve. In this case, decreasing the constriction valve aperture improves the conditions for air liberation or microbubble formation.

![Fig. 7](image_url)

**Fig. 7.** The percentage of oxygen liberation as a function of temperature and superficial air velocity (without frother).

![Fig. 8](image_url)

**Fig. 8.** The effect of superficial air velocity and frother addition on the air holdup (a and c) and the Sauter diameter (b and d). At 10°C and 30°C.
4.2. Effect of the frother (MIBC)

Fig. 8a and b shows that the air holdup increased and the Sauter diameter decreased with the addition of frother, and both parameters increased with the superficial air velocity. Thus, when the superficial air velocity changed from 0.005 to 0.06 cm/s, the range of the air holdup fluctuated between 0.75% and 2.25% and the Sauter diameters ranged between 50 and 120 m. This effect is known in conventional bubble size dispersions, i.e., decreasing the water superficial tension makes it easier to generate smaller bubbles. Recently Finch et al. (2008) and Grau et al. (2005) suggested that the frother also retards the bubble coalescence and this may be another important factor for small bubble production. From the results obtained at 10 and 30°C (Fig. 8a and c), it is observed that the temperature decreases the air hold-up. As said, this is because the bubble size increases conjointly with the rising velocity. Also, this effect of the temperature on the bubble size is evident from the results of the Fig. 8b and d.

As shown in Fig. 9a and c, the percentage of oxygen nucleation was affected by the superficial air velocity and lightly enhanced by the frother addition. This effect is probably due to the fact that frother addition decreased the bubble diameter, thus enhancing the bubble surface area flux and promoting gas diffusion from the liquid phase to the gas phase.

Also, it is observed that the effect of the frother is more significant at minor temperature. Thus, when the temperature was 10°C, the percentage of oxygen nucleation oscillated from 40% to 60%, but when the temperature was kept at 30°C, this variable changed from 60% to 71%.

Fig. 9b and d indicates that the superficial air velocity and the frother addition enhanced the bubble surface area flux. This relationship is in accord with that obtained using conventional bubble sizes. From these figures note also that temperature decreases the $S_b$, as explained in Section 4.1.

Summarising, gas dispersion parameters were measured in a system with microbubbles in water only, as a part of a series on flotation of fine minerals with small bubbles (currently undergoing in our laboratory. It is believed that with the knowledge of these values, especially by enhancing the $S_b$ values, improved capture of fine particles by small bubbles is highly probable (more residence time).

5. Conclusions

Microbubble dispersion parameters were measured in a well controlled laboratory system. Increasing the superficial air velocity from 0.01 to 0.06 cm/s increased the air holdup from 0.4% to 1.8%, enhanced the Sauter diameter from 70 to 135 m, and increased the bubble surface area flux from 5 to 30 s$^{-1}$. An increase in temperature from 10 to 30°C resulted in decreases in the air holdup and the bubble surface area flux, but increased the Sauter diameter and the percentage of nucleated oxygen. Increasing the frother addition from 0 to 20 ppm (MIBC) increased the air holdup, the percentage of dissolved oxygen, and the bubble surface area flux, but decreased the Sauter diameter.

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References


