Factors involving the solids-carrying flotation capacity of microbubbles

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Abstract

Dissolved air flotation (DAF) is a technique used extensively for separating fine particles in water and wastewater treatment, but, unfortunately, its use is still limited for froth flotation of minerals. This appears to be due to the very low lifting power of the microbubbles (40–70 \( \mu \)m) and low airflow rate because of the low solubility of air in water. Thus, the efficiency of DAF in treating mineral particles has shown to be poor and as a solid/liquid separation technology is limited to slurries with no more than 3% solids. This work presents results showing (measuring) the limits of DAF as a function of particle size distribution, solids content and air superficial velocity. Interestingly, the microbubbles were found to be not selective with respect to particle size, floating both fine and coarse particles, which is most likely due to the existence of several mechanisms acting on the flotation of particles by these minute bubbles.

1. Introduction

Flotation with the use of microbubbles (<100 \( \mu \)m) or DAF (dissolved air flotation) is a well-known technique for removing solids, fibers, microorganisms, and other impurities in water and wastewater treatment (Rubio et al., 2002; Li and Tsuge, 2006; Carissimi et al., 2007; Rodrigues and Rubio, 2007; Englert et al., 2009). Despite some authors have reported benefits regarding fine minerals recovery by DAF (Rubio et al., 2006; Englert et al., 2009; Rodrigues and Rubio, 2007), its application for the separation of ores is fairly poor, mainly due to the low carrying capacity of the fine bubbles, coalescence with larger bubbles (limiting the particle sizes they can float) and low Sb values (Pérez-Garibay et al., 2012).

Currently, it is believed that in the froth flotation of mineral particles, bubble size is an important factor affecting the collection efficiency and the carrying capacity of the flotation cells. Vianatos (2005) has reported that each particle size distribution requires an ideal bubble size distribution, which can be adjusted by manipulating the operational variables.

The mean bubble size encountered in conventional froth flotation usually varies from 600 to 2500 \( \mu \)m, hindering the capture of fine and ultrafine particles, which requires smaller bubbles (Rubio et al., 2006). Among other problems, the low particle mass and inertia and short residence time tend to dramatically reduce the collection probability of large bubbles with rapidly rising velocity. Waters et al. (2008) explain that finer particles have lower momentum and are unable to break through the liquid barrier surrounding a bubble. To overcome this limitation, the authors explore the particle–microbubble electrostatic attraction, but, again, the use of microbubbles has the disadvantages of low lifting forces and high water recoveries (Miettinen et al., 2010).

Within this context, the aim of this paper was to evaluate the abilities of microbubbles to float different particle sizes, studying the effect of the pulp density on the loading and carrying capacity of microbubbles and the recovery of particles to the concentrate. This paper also analyses possible mechanisms for the flotation of coarse particles with microbubbles and their particle size selectivity. Noteworthy, although carrying capacity has been experimentally studied with large bubbles (1–3 mm of diameter), this research appears to be the first focusing on microbubbles.

The carrying capacity of individual bubbles has been defined by geometrical variables as the bubble superficial area disposed for particle collection and the surface area occupied by these particles. Thus, considering that particles occupy all of the superficial area of the bubble under a square packing factor, the theoretical carrying capacity can be defined using Eq. (1) (King et al., 1974):

\[
C_a = \frac{\pi \cdot d_b \cdot \rho_p \cdot J_g}{d_b},
\]

where \( C_a \) is the carrying capacity (g/s/cm\(^2\)); \( d_b \) and \( \rho_p \) are the diameter (cm) and density of the particle (g/cm\(^3\)), respectively; \( J_g \) is the bubble diameter (cm); and \( J_g \) is the superficial velocity of the air (Finch and Dobby, 1990).

Due that the packing ratio has a large impact on the number of particles loaded onto the bubbles and consequently on the density of the bubble–particle aggregates, this ratio has to be well
estimated. Based on previous studies (Gallegos-Acevedo et al., 2006; Bourmain and Ata, 2010) it was observed, with angular and semi-rounded particles, that particles are attached on the bubble surface with cubic or hexagonal geometrical arrays, whose packing ratio is 0.78.

The column flotation carrying capacity ($C_0$) has been defined as the maximum mass flow rate of solids in the concentrate per unit of cross-sectional area of the column. In general, the literature suggests only that some of the bubble surface is covered with particles, although other authors suggest that under controlled conditions, the bubbles might be completely covered by particles (Gallegos-Acevedo et al., 2006). Some authors (Finch and Dobby, 1990) have studied the carrying capacity using statistical correlations and semi-empirical models based on physical and practical relationships. In this case, carrying capacity is defined as the mass flow rate of solids in the concentrate per unit area of the cross-section of the column, which can be estimated using the following equation:

$$C_0 = 0.068 \cdot d_{60} \cdot \rho_p,$$

where $d_{60}$ and $\rho_p$ are the average diameter (cm) and particle density (g/cm$^3$), respectively. In this work, the carrying capacity was assumed to be independent of column diameter.

Gallegos-Acevedo et al. (2006) have proposed geometrical models to predict the maximum mass flow rate of solids that a flotation column is capable of transporting when operating at its maximal carrying capacity. Further, they have reported that rounded particles accommodate on the bubble surface according to a cubic arrangement with a bubble surface coverage of approximately 0.78, suggesting that these models can be used to estimate the density of the bubble–particle units. Thus, the bubble diameter, particle density, particle diameter, particle shape, and the geometrical particle arrangement are important variables for bubble load estimation. In this paper, these models have been used to estimate the theoretical particle coverage density of a microbubble surface.

2. Experimental

2.1. Materials

The flotation work employed high-purity micronized silica sand, as particles model five samples of different size distributions were sieved and classified as fine, medium, semi-coarse, coarse, and very coarse. Fig. 1 shows the particle size distribution within each class and their average particle diameter ($d_{10}$), which was measured by laser diffraction.

The pulp was conditioned at 30 °C with 0.057 g of dodecylamine/kg of mineral (C$_{12}$H$_{25}$NH$_2$, Aldrich, 98%) and 15 ppm Dowfroth 250 at pH 9.5 (adjusted with NaOH). This value of pH was selected because the superficial charge of the quartz is highly negative and the more predominant species (cationic) of the dodecylamine is the RNH$_3^+$, favouring its adsorption at the silica/water interface.

2.2. Methods

The experiments were carried out in a laboratory flotation column endowed with a transparent acrylic tube (1.8 m in height and 0.095 m in diameter). The slurry and the micro-bubbles were fed into the column, at 50 cm under the lip of the concentrate overflow.

Fig. 2 schematically shows the experimental setup, which was configured to work in an open circuit to avoid recirculating traces of pre-charged microbubbles. After 40 min of conditioning, the flotation began and operates for at least 15 min to achieve a steady state. Once this period elapsed, timed samples of feed, concentrate, and tailings were collected over approximately 40 s to be filtered, dried, and weighted. After the first sample was collected, the slurry density was increased, adding a volume of preconditioned solids. The same procedure was repeated until the solids percentage was approximately 20%. Next, samples were prepared to evaluate the solids content and the liquid and solids flow rates. It should be noted that a similar practice was followed for each particle size class.

The column was operated under two different constant gas rates ($5.9 \times 10^{-3}$ and $1.59 \times 10^{-2}$ cm$^3$/s) and an approximately constant feed flow rate at 0.3 cm/s. The data were used to carry out a mass balance around the column and obtain the solids recovery, carrying capacity, and all other results reported herein.

Each test consists of varying the mass flow rate of solids in the feed stream until the mass flow rate of the concentrate peaks. This maximum is the result of the bubble surface becoming completely covered or the density of the aggregate particle–bubble exceeding that of the pulp density, causing the aggregate to sink instead of float. This phenomenon may determine the maximum processing capacity of the column and may give rise to the limiting carrying capacity itself.

An important part of the experimental setup is the image acquisition system, composed of a digital camera (Canon Digital Revel XTi EOS) equipped with a Canon macro lens MP-E65 mm (1-5×) focused to the plane of the peephole. This system was placed at the centre of the collection zone to visualise better the microbubbles.

![Fig. 1. Mass density functions of the particle size distributions of the silica sand classified as fine, medium, semi-coarse, coarse, and very coarse particles.](image-url)
Fig. 2 shows a schematic of this peephole made of acrylic and circular plaques: (1) the plaque closest to the camera is transparent and 1-cm thick, (2) next is a sampling window (rectangular empty volume; 1.5-cm wide, 3-mm high, 3.8-cm deep), (3) a semi-opaque white plaque is used to diffuse the light, (4) a small LED energised by a 3-V battery is attached to a thick transparent plaque, and (5) an opaque plaque was placed at the end. The withdraw of bubbles was taken close to the wall of the flotation column, assuming that there is a minimal segregation due to the small column diameter (0.095 m). Xu et al. (1992) observed that in a flotation column with 0.91 m of diameter, the maximum absolute variation of the radial gas holdup (and possibly the bubble diameter) was about 4% under all conditions; and that for practical purposes, it could be considered uniform distribution.

Measurements of the bubble diameters were made with ImageJ software, which was supplemented with a macro-progam to handle and process the images. To obtain reliable results, several steps must be followed: (1) calibration of the image magnification, (2) identification of a threshold for the image, and (3) analysis of the bubbles. To calibrate the software, a thin stainless steel wire (0.3 mm) was used as a reference and placed close to the photographic plane. In order to eliminate problems with clusters of bubbles, the ImageJ software was configured to measure bubbles with a maximal bubble diameter of 1 mm and circularity between 0.8 and 1. To estimate a minimal standard deviation of the Sauter diameter, more than 450 micro-bubbles were counted; this has been already proposed by Pérez-Garibay et al. (2012).

The software reports a list of computed objects (bubbles) and their corresponding diameters. This software makes it possible to select the bubbles for measurements 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. (3) was used to estimate the Sauter diameter ($d_{32}$). Additionally, to obtain a representative Sauter diameter, the number of bubbles that needed to be counted was also evaluated.

$$d_{32} = \frac{\sum d_b^2}{\sum d_b}$$

(3)
3. Results

3.1. Air superficial velocity and bubble size distribution

Fig. 4 shows that different bubble size distributions are generated at different air superficial velocities \( \frac{1.59 \times 10^{-2}}{10^{-2}} \) and \( \frac{5.9 \times 10^{-3}}{10^{-3}} \) cm/s, with the bubble size increasing with the air superficial velocity. Thus, the Sauter diameter of the microbubbles increased from 60.5 to 70.7 \( \mu \)m when the air superficial velocity increased from \( \frac{5.9 \times 10^{-3}}{10^{-3}} \) to \( \frac{1.59 \times 10^{-2}}{10^{-2}} \) cm/s. According to Pérez-Garibay et al. (2012) the aperture of the needle valve generates turbulent conditions which facilitate bubble coalescence and the increase of the averaged diameter.

3.2. Carrying capacity

Fig. 5 shows the carrying capacity of the laboratory column operating with each particle size distribution at two air superficial velocities \( \frac{1.59 \times 10^{-2}}{10^{-2}} \) and \( \frac{5.9 \times 10^{-3}}{10^{-3}} \) cm/s. The results show that this parameter improves the carrying capacity by increasing the number of microbubbles and the bubble surface area occupied by particles. It was also observed that the microbubbles have a low carrying capacity for fine particles (Fig. 5a), as these particles easily cover the entire bubble surface at low solids content. Conversely, the carrying capacity is higher for coarse particles because a few large particles may have a larger mass than many.

**Fig. 5.** Carrying capacity for fine, medium, semi-coarse, and coarse particles \( d_{10} = 8.9, 22.4, 28.7, 72.4 \) \( \mu \)m; \( 3.37 \times 10^{-7} \) M of dodecylamine, pH 9.5.

**Fig. 6.** (a) Carrying capacity as a function of particle size \( d_{10} \). (b) Photo of the particle–microbubble aggregates.
fine particles (Fig. 5d), even if the entire bubble surface area is not occupied. The results show that the maximal carrying capacities of the column at \( J_g = 1.59 \, \text{cm}^2/\text{s} \) for fine, medium, coarse, and very coarse particles are 0.04, 0.15, 0.16, and 0.19 g/min/cm\(^2\), respectively (Fig. 5(a–d)). By comparison, for \( J_g = 5.9 \, \text{cm}^2/\text{s} \), the carrying capacities were 0.028, 0.043, 0.060, and 0.058 g/min/cm\(^2\) for fine, medium, coarse, and very coarse particles, respectively.

The maximal carrying capacity estimated using the statistical Eq. (2), proposed to predict the results of a flotation column operating under conventional conditions, is \( C_{\text{amax}} = 0.00041 \, \text{g/min/cm}^2 \) when \( d_{80} = 190 \, \mu\text{m} \) and \( \rho_b = 2.7 \, \text{g/ml} \) for silica. This result is very far from the maximum (break point) observed experimentally for the two air superficial velocities with coarse particles (Fig. 5d). Evidently, the discrepancy between the predicted and the experimental results is because the statistical equation is only valid for bubbles and particles of conventional size in froth flotation. Therefore, a new equation for microbubbles generated after depressurization of dissolved air in water is required.

Fig. 6 shows the maximal carrying capacities as a function of particle size and air superficial velocity. For both air velocities, the carrying capacity reaches a maximum but then decrease when the particles are much larger than the bubble size. Evidently, larger carrying capacities are obtained for higher air superficial velocities when the number of bubbles is high.

Figs. 5 and 6, show that fine particles float readily with microbubbles, but it is not clear why coarse (\( d_{10} = 72.4 \)) and very coarse particles (\( d_{10} = 192 \, \mu\text{m} \)) can also float with these minute bubbles with diameters of 60–71 \( \mu\text{m} \). Some photographic evidences indicate that the formation of particle–bubbles clusters is the responsible mechanism that makes possible this last phenomenon (Fig. 6b). It may appear that the microbubbles are more selective for finer particles, but the results presented in the next section suggest other mechanisms.

Fig. 7 shows that bubble surface area flux increase linearly with the superficial air velocity. In this case, it is observed that a low superficial air velocity can produce a high bubble surface area flux due to the fact that microbubbles have a great superficial area.
3.3. Selectivity of microbubbles for different particle size distributions

Fig. 8 shows the density function of the feed and concentrates obtained when the flotation is operated with a \(1.59 \times 10^{-2}\) cm/s air superficial velocity. Notably, the results of the concentrate (pointed lines) correspond to operation conditions with feeds containing low and high solids percentages \(C_{\text{max}}\). The results shown in Fig. 8 do not show evident selectivity by particle size, even if, surprisingly, it seems that microbubbles are slightly selective for the coarse particles of each distribution. One hypothesis that explains these results is that the bubbles attach to the particles rather than the particles to the bubbles, as, in this case, the fine and coarse particles have similar flotation abilities. This first-hand mechanism allows the possibility of using microbubbles to float wide particle size distributions, although its disadvantages should be evaluated before any application. To study this hypothesis, the density that a theoretical bubble–particle aggregate can reach when a monolayer of particles occupies its surface was estimated. For this estimation, it was supposed that the microbubble \((d_{80} = 7.0 \mu m)\) can be covered with a monolayer of particles with similar sizes as those used during the tests (fines \((d_{80} = 8.9 \mu m)\), medium \((d_{80} = 32 \mu m)\), and coarse \((d_{80} = 101 \mu m)\)) (see Fig. 9a). The density that each particle size achieves when a monolayer of microbubbles occupies the particle surface was also estimated (see Fig. 9b).

The assumptions of the algorithm used to estimate the density of the theoretical bubble–particles or particle–bubbles aggregates are the follows:

1. The bubbles are spherical, and their surface area may be calculated by the next equation.
   \[S_b = \pi \cdot d_b^2\] (4)
2. The number of particles that the surface of the bubble may accommodate may be estimated assuming a homogeneous particle size, a square packing arrangement \((e = 0.78)\), and a monolayer of particles using the follow equation. As mentioned in Section 1, this assumption is supported by preceding studies (Gallegos-Acevedo et al., 2006; Bournival and Ata, 2010).

\[
\eta_p = \frac{S_b}{d_p^2} \quad (5)
\]

The bubble load, or the weight of one particle \((M_p)\), is then calculated using the equation

\[
M_p = \frac{V_p}{\rho_p}, \quad (6)
\]

where \(V_p = \frac{\pi}{6} \cdot d_{cno}^3\) and \(\rho_p\) are the volume and the particle density, respectively.

To estimate the density of the bubble–particles and particle–bubbles aggregates, Eqs. (7) and (8) were used, respectively. Note-worthy that the mass of the bubble was considered negligible and this is the reason not to appear in these equations. More, for the case where the bubbles are attached on one particle, it was also assumed that bubbles are adhered on a spherical particle with a similar contact \((0^\circ)\) angle and packing factor that when particles are attached on a bubble.

\[
\rho_{dp} = \frac{\eta_p M_p}{\frac{\pi}{6} (n_p \cdot d_{cno}^3 + d_{cno}^3)}, \quad (7)
\]

\[
\rho_{dp} = \frac{M_p}{\frac{\pi}{6} (d_{cno}^3 + n_b \cdot d_{cno}^3)}, \quad (8)
\]

Fig. 9a indicates that in the case where the particles are attached to the bubble, the fine particles can cover the entire bubble surface, producing an aggregate with a density lower than that of the water, allowing flotation. However, if coarse particles are used instead, the aggregate density exceeds the water density, preventing flotation. In contrast, Fig. 9b shows that when the microbubbles are attached to particles, fine and medium particles float readily, and coarse particles float when five microbubbles are attached to a particle. This simplified theoretical analysis agrees with the experimental results, showing that very coarse particles can be floated by several microbubbles. The physical aggregates likely suffer from friction (drag) forces, which may lead to a poor flotation recovery.

3.4. Effect of the particle size distribution and solids percentage on concentrate recovery

Fig. 10 shows the concentrate recovery as a function of the percentage of solids in the feed when the column was operated with each particle size distribution at air superficial velocities of \(1.59 \times 10^{-2}\) and \(5.9 \times 10^{-3}\) cm/s. In general, higher recoveries were obtained with finer particles than with coarse particles. Notably, even for the higher recovery achieved with fine particles, the maximal percent of solids in the feeds is 13%.

Here, it is interesting to discuss the results of carrying capacity and recovery. The limited carrying capacity with the fine particles is attributed to the complete coverage of the bubble surface area by a low overall particle mass. However, why does the maximum carrying capacity increase with the particle size and the maximum recovery decrease with the same variable? In the first instance, this phenomenon reflects the fact that the reduced carrying capacity with coarse particles is a result of too few bubbles rather than a low bubble surface area. In this case, several microbubbles may be attached to the surface of the coarse particles until a point where the limited number of microbubbles reduces its carrying capacity and the recovery of coarse bubbles. This could to explain why the maximum recovery decreases gradually as the percentage of solids on the feeds increases. Rubio et al. (2002) and Rodrigues and Rubio (2007), have already reported several mechanisms involving adhesion, entrapment and entrainment of bubbles onto or inside the particles.

4. Conclusions

Results showed that the carrying capacity of the microbubbles (<100 µm) is higher for the fines than the coarser fractions, because of the reduced mass and high bubble packing degree as they are able to suit better the available bubble surface area. Conversely coarse particles appear to create clusters of aggregates bubbles–particles, which are denser than with small particles. Yet, the microbubbles were found to be not selective with respect to particle size, floating both fine and coarse particles. Thus, coarse particles \((d_{cno} = 190 \text{ µm})\) float with microbubbles \((d_{cno} = 71 \text{ µm})\), which suggests that unconventional flotation mechanisms are present in which several bubbles are attached or entrapped within particles aggregates, allowing its flotation.

Acknowledgments

The authors are grateful to CONACYT (Mexico) for a scholarship and other financial support.

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