

Article

Estimation of Bubble Size and Gas Dispersion Property in Column Flotation

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Abstract: This study investigates bubble size measurements, bubble characteristics, and the relationship between key operating variables and gas dispersion properties in column flotation. As the frother concentration increased to 120 ppm, the bubble size distribution (BSD) transformed from bimodal to unimodal and achieved a minimum bubble size of 0.62 mm. The critical coalescence concentration (CCC) was identified as 120 ppm. Gas velocity and wash water velocity significantly influenced bubble size, with gas holdup peaking at 27% at 1.08 cm/s a gas velocity. The bubble-rising velocity increased as the bubble size increased, indicating that the bubble size and bubble-rising velocity were proportional. The bubble surface area flux decreased linearly with increasing bubble size and was significantly affected by the gas velocity. A strong correlation ($R^2 = 0.86$) between measured and calculated bubble sizes was achieved, with an average size of 0.64 mm and an estimation error of $\pm 13\%$. The study demonstrated that bubble size and distribution could be effectively controlled under specific operational conditions ($J_g = 0.65\text{--}1.3$ cm/s, $J_w = 0.13\text{--}0.52$ cm/s, frother = 30–120 ppm). These findings highlight the importance of optimizing key variables to enhance column stability, regime maintenance, and flotation performance.

Keywords: bubble size; column flotation; critical coalescence concentration; gas dispersion



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1. Introduction

Flotation is a separation method that uses bubbles to separate particles based on differences in their physicochemical properties. Hydrophobic particles float and concentrate through collisions and attachment to rising bubbles, whereas hydrophilic particles do not attach to bubbles or sink [1,2]. These properties are used in diverse fields such as mineral processing and water treatment [3].

Bubbles used to recover valuable minerals play an important role in flotation because they attach and transport hydrophobic mineral particles. Bubble size and bubble distribution significantly affect the recovery rate of the target minerals during flotation [4]. Large bubbles can quickly recover coarse particles, but fine particles move along the water streamline, reducing the particle–bubble collision probability. Therefore, fine bubbles have a higher particle–bubble collision efficiency than large bubbles and thus have an advantage in terms of flotation efficiency [5,6]. According to Ahmed and Jamson (1985), when the bubble size is reduced from 0.66 to 0.075 mm, the flotation rate of quartz increases by approximately 100 times [7]. Gorain et al. (1997). reported that the first-order flotation rate constant and bubble surface area flux are linearly related. Moreover, because the bubble surface area flux, which is proportional to the gas velocity and inversely proportional to the Sauter mean bubble diameter, is a parameter that affects the flotation recovery, the importance of measuring the bubble size for predicting the flotation performance was emphasized [8]. Therefore, bubble size measurements are crucial for controlling the flotation process.

Conventional methods for measuring the bubble size in flotation include the flow dynamics “Stokes law” based on empirical and semi-empirical experiments, electrical

resistance measurements, and ultrasonic methods [9–11]. However, these methods have the disadvantages of requiring a long measurement time, low accuracy, and large deviations from the actual bubble size [12]. Recently, many researchers have studied the application of image analysis methods to measure bubble size. Ksentini et al. (2013) determined the bubble diameter, rising velocity, and gas holdup in a column using video recordings and image-treatment methods [13]. Image analysis methods were used by Nasset et al. (2006) to measure bubble size in a mechanical flotation cell [14]. Seger et al. (2019) applied sampling image technology to measure bubble size in a flotation column. Bubble size was measured by image analysis using a bubble size analyzer at the column bottom [15]. Mazahernasab et al. (2021) estimated bubble size using image analysis and laser diffraction methods [16]. Eskinlou et al. (2018) and Riquelme et al. (2015) analyzed images captured using a camera and processed bubble size using MATLAB software [16,17].

Bubble size control is an important factor for stabilizing flotation process performance. Surfactants, generally known as frothers, determine the characteristics of bubbles during flotation. The frother generates bubbles and prevents the coalescence of the generated bubbles, thereby improving gas dispersion [18,19]. In addition, the bubble size distribution directly affects the selectivity of the bubbles, their ability to carry valuable minerals, and their flotation efficiency. Factors affecting the bubble size include the type of frother concentration and gas velocity [20,21]. The bubble size decreases as the frother concentration increases and remains uniform above a certain concentration. This concentration is called the critical coalescence concentration (CCC). This phenomenon occurs because the coalescence of bubbles decreases as the frother concentration increases because the coalescence of bubbles is prevented at the CCC [22]. Therefore, even when the frother concentration exceeds the CCC value, the bubble size is unaffected. As the gas velocity increases, the bubble size increases because of the collision and coalescence of bubbles, and the measurement of the bubble size as a function of the gas velocity is considered an essential factor for controlling the stability and efficiency of column flotation. The bubble surface area flux has an important impact on the frequency of bubble–particle collisions and flotation performance, which is affected by gas velocity and bubble size. A study by Capponi et al., 2005 confirmed that recovery increased by approximately 2% and flotation kinetics (rate constant) improved by approximately 3% as the bubble size decreased in laboratory-scale sulfide mineral flotation. They argued that this was because the bubble surface flux and capture of fine sulfide mineral particles increased as the bubble size decreased [23]. Angelica et al. (2019) evaluated the recovery characteristics of apatite for three different bubble size distribution ranges. As a result, the highest grade of apatite was obtained when the bubble size range was less than 0.3 mm, and the highest recovery was obtained when the bubble size range was more than 1 mm. Considering the grade and recovery, the best flotation performance was obtained in the bubble size distribution range of 0.3–1 mm [24]. Therefore, because the bubble size in column flotation directly affects the flotation efficiency, it is essential to evaluate the correlation with various factors through bubble measurements for flotation performance evaluation.

Therefore, in this study, we calculated the size of the bubbles directly in a flotation column using a high-speed camera and an image analysis program. We aimed to evaluate the relationship between bubble size as a function of the frother concentration and the CCC, gas velocity, bubble surface area flux, and wash water velocity, which can maintain the column flotation regimen and control flotation efficiency.

2. Theoretical Aspects

Bubble size in flotation columns is an important basic factor in studying gas dispersion properties, calculating carrying rates, optimizing operation variables, and securing mixing models and scaled-up data. In particular, gas dispersion properties such as superficial gas velocity, bubble size, gas holdup, and bubble surface area flux are significant parameters that affect the maintenance of the column system and the efficiency of column

flotation. These factors can be used to evaluate the basic characteristics and understand the phenomena occurring in a column.

The gas and liquid velocities inside the column are related to the flow velocity of the fluid. In other words, the superficial gas velocity is expressed by dividing the flow rate of the gas injected into the column by the cross-sectional area of the column, and the superficial wash water velocity can also be expressed by dividing the flow rate of the incoming wash water by the cross-sectional area of the column. The gas (J_g) and liquid velocities (J_l) can be expressed using the following equations [25]:

$$J_g = \frac{Q_g}{A_c} \quad (1)$$

$$J_l = \frac{Q_l}{A_c} \quad (2)$$

where J_g is the gas velocity, J_l is the liquid (slurry) velocity, Q_g is the gas flow rate, Q_l is the liquid (slurry) flow rate, and A_c is the cross-sectional area of the column.

The average Sauter bubble diameter (d_{32}) is generally used to evaluate the value of the average bubble and is expressed as follows [26]:

$$d_{32} = \frac{\sum_{i=1}^n d_i^3}{\sum_{i=1}^n d_i^2} \quad (3)$$

where d_i is the bubble size and n is the number of bubbles. A minimum of 100 bubble images were processed to obtain an appropriate Sauter mean bubble diameter estimate.

The bubble size estimation can be obtained using Wallis' drift flux (Equation (4)) and Masliyah's hindered settling (Equation (5)) equations. The bubble size can be calculated by repeatedly substituting the given variables until the relative velocities, U_s , of the two equations correspond to [27,28].

$$U_s = \frac{J_g}{\epsilon_g} \pm \frac{J_l}{(1 - \epsilon_g)} \quad (4)$$

where J_g and J_l are the gas and liquid velocities, respectively, and ϵ_g is the gas holdup. The drift flux equation applies to co-current and counter-current gas and liquid flows and can be obtained by measuring the gas and liquid velocities and gas holdup.

$$U_s = \frac{g d_b^2 \rho_l (1 - \epsilon_g)^{m-1}}{18 \mu_l (1 - 0.15 Re_{bs}^{0.687})} \quad (5)$$

Masliyah modified the Schiller–Neumann formula for the drag coefficient of spherical particles to incorporate the Reynolds number for bubbles rising in the same swarm as the hindered settling conditions for particles [28]. The interference settling equation can be obtained using the following Equations (6)–(10) [25,29,30]:

$$Re_{bs} = \frac{d_b U_{sg} \rho_{sl} (1 - \epsilon_g)}{\mu_{sl}} \quad (6)$$

where Re_{bs} is the Reynolds number of a bubble in a swarm, d_b is the bubble diameter, U_{sg} is the relative velocity, ρ_{sl} is the liquid density, and μ_{sl} is the liquid viscosity.

Richardson (1954) also suggested that m may be calculated using the following Equations (7)–(8) [30]:

$$m = \left[4.45 + 18(d_b/d_c) Re_b^{-0.1} \right] (1 < Re_b < 200) \quad (7)$$

$$m = Re_b^{-0.1} (200 < Re_b < 500) \quad (8)$$

where d_c is the column diameter and Re_b is the Reynolds number of a single bubble rising in slurry following Equation (9).

$$Re_b = \frac{d_b U_b \rho_{sl}}{\mu_{sl}} \quad (9)$$

U_b can be calculated by assuming that there is one bubble inside the entire column as the maximum rising velocity of a single bubble, and ϵ_g can be set to 0 in the interference-settling equation. Another method uses the relationship between the gas velocity and gas holdup, which can be expressed by the following equation [25]:

$$U_b = \frac{dJ_g}{d\epsilon_g} \epsilon_g \rightarrow 0 \quad (10)$$

Therefore, this study estimated the bubble size using measured gas holdup and Equations (4)–(9). The iteration approach is as follows: (a) assume an arbitrary value for d_b ; (b) estimate the slip velocity using drift flux theory (Equation (4)); (c) estimate the slip velocity using Equations (5)–(9); (d) since the difference between the slip velocities calculated in (b) and (c) decreases monotonically as d_b increases, the assumed value of d_b can be continuously refined until the two values are within a specified error. The d_b value that satisfies this condition is considered the optimum estimate of d_b .

Gas holdup (ϵ_g) indicates the gas volume ratio within the column and plays an important role in the interaction between variables in gas dispersion characteristics and selection efficiency. There are several methods for measuring the gas holdup in a floating column, such as head difference, pressure difference, and sensors. The method using the head difference can measure the gas holdup for the entire column area, whereas the method using the pressure difference and a sensor can measure the gas holdup for a certain area of the column. In this study, the gas holdup was measured using a manometer and the pressure-difference method. Figure 1 illustrates the gas-holdup measurement method using a manometer. Using the pressure difference between the two manometers, the gas holdup between two points can be expressed as follows [25]:

$$\epsilon_g = 1 - \frac{\rho_w}{\rho_{sl}} \left(1 - \frac{\Delta h}{\Delta L} \right) \quad (11)$$

where ρ_w is the density of the liquid, ρ_{sl} is the density of the slurry, L is the distance between points A and B, and h is the head difference between the two manometers. In two phases, the gas holdup can be expressed by a simplified equation [25].

$$\epsilon_g = \frac{\Delta h}{\Delta L} \quad (12)$$

The total surface area of the bubbles passing through a cross-section of the cell per unit of time defines the bubble surface area flux (S_b). The bubble surface area flux was calculated as follows [25]:

$$S_b = \frac{6J_g}{d_b} \quad (13)$$

where J_g is the gas velocity and d_b is the Sauter mean bubble diameter.

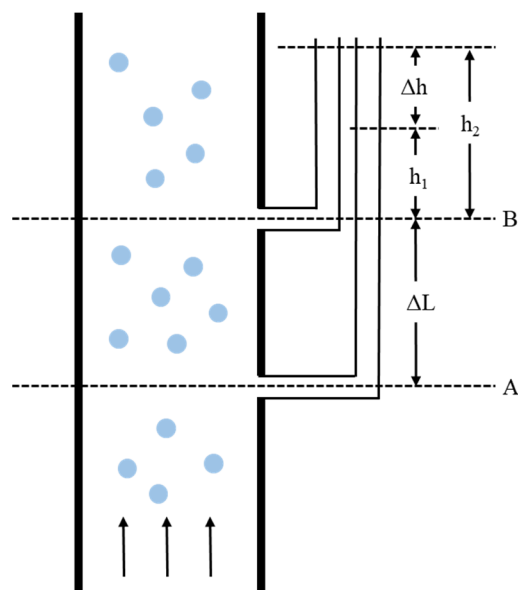


Figure 1. Measurement of gas holdup by manometer method.

3. Experimental

3.1. Column Flotation System

Figure 2 shows a schematic diagram of the column flotation system used in this study. The flotation column measured 300 cm in height and 7 cm in diameter and had a cross-sectional area of 38.48 cm². We placed the feeder at the top 1/3 of the column and injected air through a porous sparger (pore size 0.5 μm) at the column bottom. We controlled the flow rate using an airflow meter. The porous sparger generated bubbles as compressed air passed through the micropores, and the size and distribution of the bubbles varied depending on the pore size and air pressure. This type has the characteristics of a simple structure and easy manufacturing; therefore, it is primarily used in experiments. Wash water was placed at the top of the column and its flow rate was controlled using a peristaltic pump. Two manometers were installed at 35 cm intervals to measure the gas holdup in the column. The high-speed camera (Mach-F340, Comart system, Seoul, Republic of Korea) is 20 cm from the column and equipped with a CMOS image sensor (2048 × 1088 pixels), enabling shooting from 340 fps (Frames per second) to 2650 fps at a resolution of 920 × 1080 p. Additionally, a flash device was installed to increase the contrast of the bubbles while shooting using a high-speed camera to obtain a clear image.

3.2. Bubble Size Measurements

Pine oil is used as a frother for bubble generation and is primarily used in flotation. The general characteristics of pine oil are that it contains approximately 44% (weight ratio) of alcohol, distills at 170 to 220 °C, and has a solubility in water of approximately 2.5 g/L. The concentration of the frother (pine oil) in the conditioner was adjusted, and the frother, feed, wash water, and gas were supplied under the operating conditions. Simultaneously, the concentrate and tailings were discharged. Subsequently, a stabilization period was provided to maintain the column system. High-speed photography was performed on the bubbles in the stabilized collection zone. The bubble size in the photographed images was measured for more than 100 bubbles using the ZEISS image analysis program (AxioVision 4.8), and the average Sauter mean diameter was obtained. The experimental conditions were a gas velocity of 0.65–1.30 cm/s, wash water velocity of 0.13–0.52 cm/s, and a 5–250 ppm frother concentration. All mass balances, that is, supply and discharge, were measured using a flow meter and expressed as the velocity according to the cross-sectional area of the column and time.

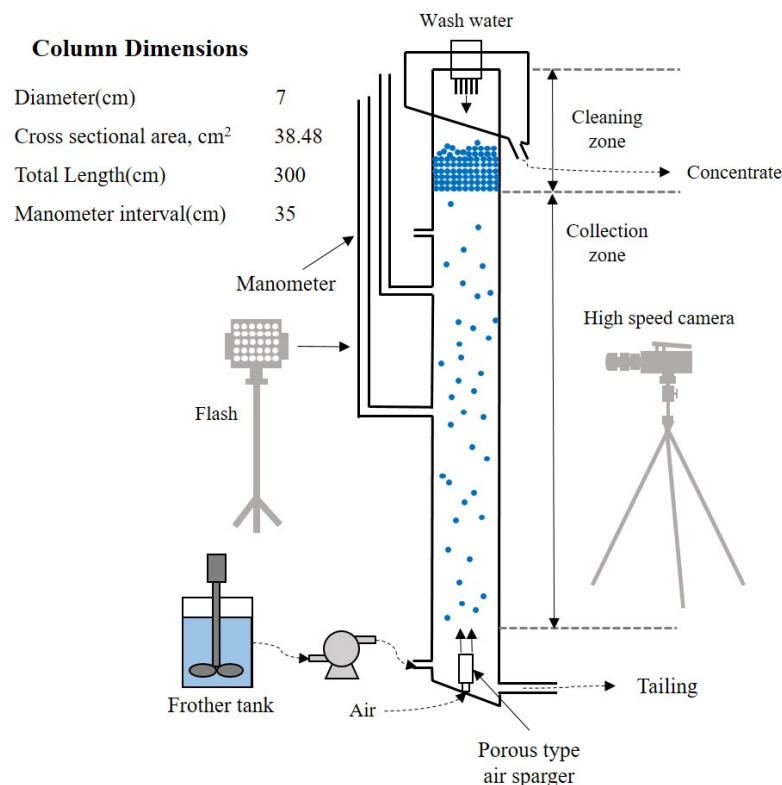


Figure 2. Flotation column and bubble size measurement system.

4. Results and Discussion

4.1. Frother Concentration

A frother is essential for lowering the surface tension of water and for forming, distributing, and maintaining bubbles. In this study, surface tension was measured using an extension device (unit of measurement, mN/m) that applied the Dunouy method. This device measures the surface tension by measuring the tension generated when lifting a Dunouy ring made of platinum from the surface of the solution. Average values were obtained from three repeated measurements. Figure 3 shows the changes in the surface tension as a function of the frother concentration. The measurement resulted in the initial surface tension of water of 71.19 mN/m, which was a reasonably significant value, but decreased as the frother concentration increased. The surface tension at a frother concentration of 120 ppm was 46.24 mN/m, and when the frother concentration was above 120 ppm, there was no significant variation in surface tension. It is believed that above a specific frother concentration, the role of the frother and the consumption of reagents are limited.

Figure 4 shows the change in bubble size (d_b) as a function of gas velocity (J_g) at different frother concentrations. CCC is generally determined by observing the behavior of bubbles (constant bubble size) when increasing frother concentration in a flotation column. The bubble size decreased as the frother concentration increased, and the average bubble size was distributed in the range of approximately 0.62–0.89 mm. This is because the mutual attraction of the liquid with a lower surface tension decreased, forming smaller bubbles [31]. As the initial frother concentration increased, the bubble size decreased rapidly and became almost constant above a certain concentration, which was 120 ppm [22]. The effect of these frother concentrations on bubble size reduction corresponds to the similar behavior on surface tension [32]. This is because the coalescence of air bubbles decreases as the concentration of the frother increases, and above the critical coalescence concentration, the coalescence of air bubbles is completely prevented [22,33]. In other words, bubble coalescence is affected at concentrations below the critical coalescence concentration, whereas coalescence does not occur at higher concentrations [34]. The bubble size tends to increase as the gas velocity increases. The maximum bubble size was measured to be

0.87 mm at a gas velocity of 1.29 cm/s and a frother concentration of 5 ppm, and the minimum bubble size was confirmed to be 0.62 mm at a gas velocity of 0.65 cm/s and a frother concentration of 120 ppm. The bubble size increase with increasing gas velocity was mainly due to increased kinetic energy, a higher collision frequency, and enhanced bubble coalescence due to shear forces and turbulence [35]. Therefore, gas velocity is an important parameter in column flotation to balance the bubble size and ensure optimal separation efficiency. It was noted that the bubble size increased owing to the collision and coalescence of bubbles as the gas velocity increased, and the measurement of bubble size with gas velocity is considered an important parameter for adjusting the efficiency and stability of column flotation.

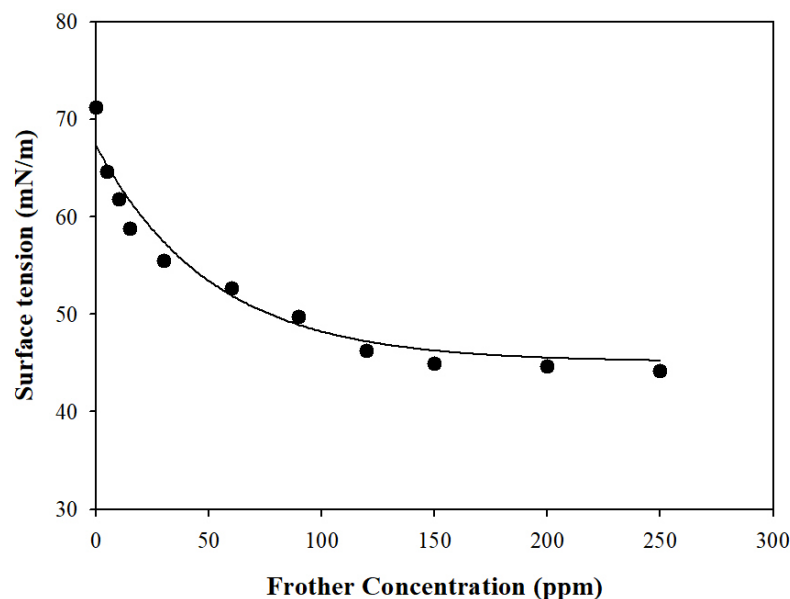


Figure 3. Surface tension as a function of frother concentration.

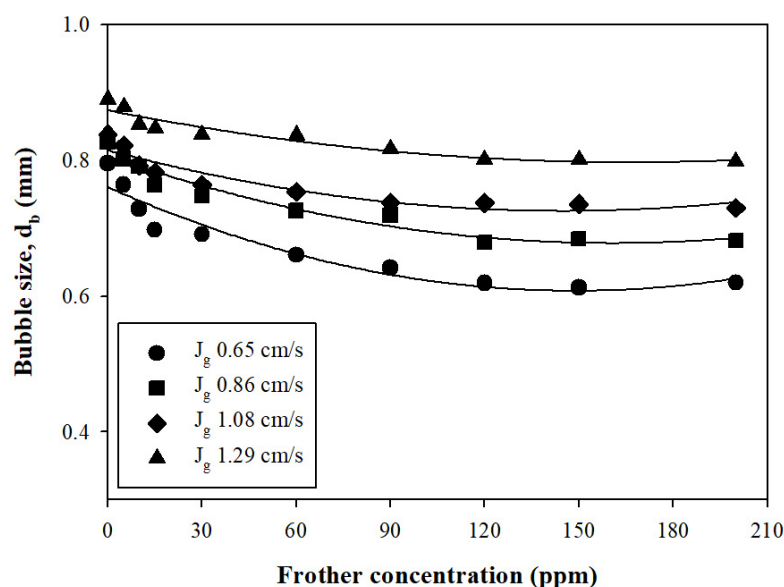


Figure 4. The variation in bubble size as a function of frother (pine oil) concentration.

Figure 5 shows the bubble size distribution (BSD) as a function of the frother concentration. As the frother concentration increased from 30 to 120 ppm, the bubble size distribution, which was bimodal at 30 ppm, gradually became unimodal, finer, and narrower at 120 ppm. This phenomenon may be because, with an increase in small bubbles,

the bubble size distribution range is narrowed and bubble coalescence is prevented at the CCC. In this regard, a study by Rodrigues et al. (2003) and Nasset et al. (2006) showed similar results, in which a narrower range of small bubbles was formed as the frother concentration increased [14,36]. In addition, because the minimum value of the bubble size did not change significantly even when the frother concentration increased, the average bubble size that could be reduced using the frother under the conditions of this study was 0.5 mm.

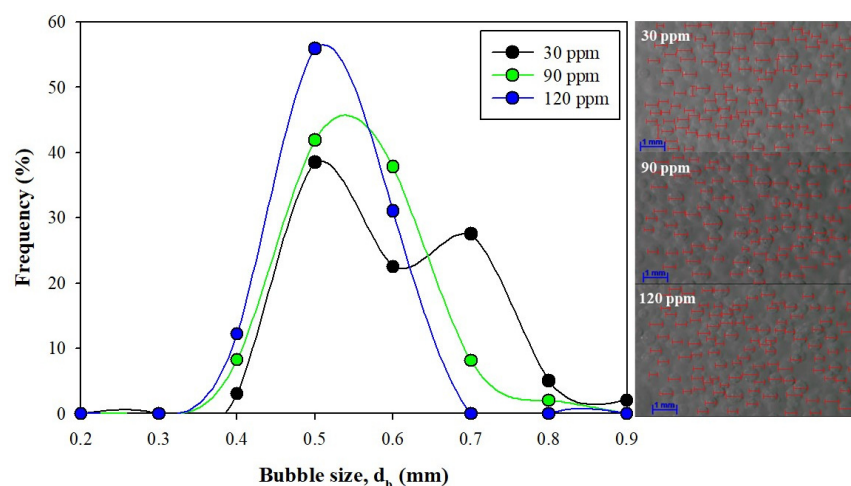


Figure 5. Bubble size distribution as a function of different concentrations of pine oil.

4.2. Gas Velocity

Gas velocity (J_g) is an important operating variable in column flotation and has a significant influence on bubble generation/distribution, maintenance of the system (upstream), mixing, and transport within the column. Wash water is a significant factor in maintaining and mixing the column regime, which suppresses entrained and entrapped gangue minerals within the froth zone and affects the downstream flow along with the feed. The gas and wash water velocities can control the bubble size in the column and affect the gas holdup and bubble rise velocities. The gas and wash water velocities and bubble size were calculated using Equations (1)–(3). Figure 6 shows the change in bubble size as a function of gas and wash water velocities. The bubble size tends to increase as the gas velocity increases. The maximum bubble size was 0.97 mm at 1.30 cm/s J_g and 0.13 cm/s J_w and the minimum was 0.62 mm at 0.65 cm/s J_g and 0.52 cm/s J_w . An increase in gas velocity forms larger bubbles because the amount of gas flowing into the column increases, increasing the bubble size by coalescence between bubbles [37,38]. If the gas velocity inside the column becomes very high, turbulent flow is formed, resulting in a nonuniform bubble size. However, if the gas velocity decreases, bubbles of uniform size are formed with laminar flow [39]. The increase in the wash water velocity from 0.13 cm/s to 0.52 cm/s resulted in a reduction in the bubble size. It seems that the expansion of the bubble size was suppressed by increasing the pressure of the liquid flowing into the column. The decrease in bubble size with increased wash water is due to the shear stress on rising bubbles. This shear stress by the wash water tends to decompose or break up the rising bubbles, resulting in the formation of a small gas bubble. The higher gas velocity would increase the bubble coalescence; however, this coalescence tendency decreases with increased wash water flow [40]. Hence, the bubble size as a function of gas and wash water velocity is important for adjusting column flotation's stability, regime, and efficiency.

Figure 7 shows the change in gas holdup (ϵ_g) as a function of the gas velocity (J_g) at different wash water velocity values (J_w). The gas holdup was calculated using Equation (12). The gas holdup increased with the gas velocity. The results show a similar trend to the change in bubble size as a function of the gas velocity, as shown in Figure 6. This was attributed to the increase in the gas volume in the column. The gas holdup is measured

to be less than <25% in conditions of gas velocity of 0.87 cm/s. However, it is more than approximately 40% in conditions of gas velocity of 1.08 cm/s. In addition, the relationship between the gas velocity and gas holdup was affected by the bubble size, frother concentration, and wash water velocity [41]. Therefore, the bubble size and gas holdup increased as the gas velocity increased, and the gas holdup increased as the wash water increased at the same gas velocity. It seems that the increase in gas holdup with an increase in wash water velocity may be due to an increase in the wash water and variation in bubble-rising velocity reported in the collection zone in the column [42]. The wash water velocity reduces both the relative gas velocity and the turbulence intensity. Also, as the wash water velocity increases, its downstream flow contributes resistance to the bubble rising. This reduces the bubble size and the bubble residence time, thereby decreasing the gas holdup [43]. In addition, an increase in the frother concentration causes a decrease in the bubble size, as discussed in Section 4.1. It is shown that the gas holdup is dependent on the gas velocity, wash water velocity, and frother concentration related to the bubble size and is significantly affected by adjusting the correlation between these variables.

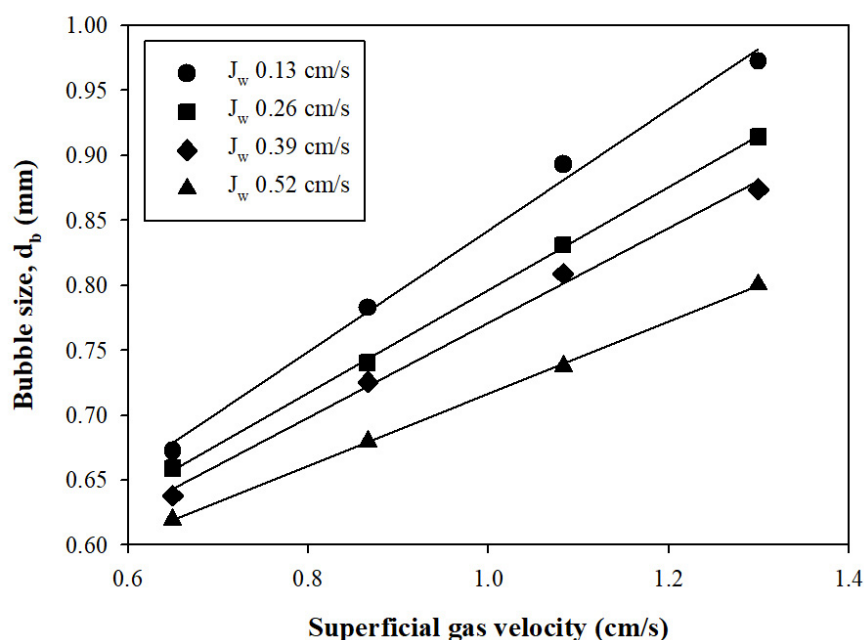


Figure 6. The variation in bubble size as a function of superficial gas velocity.

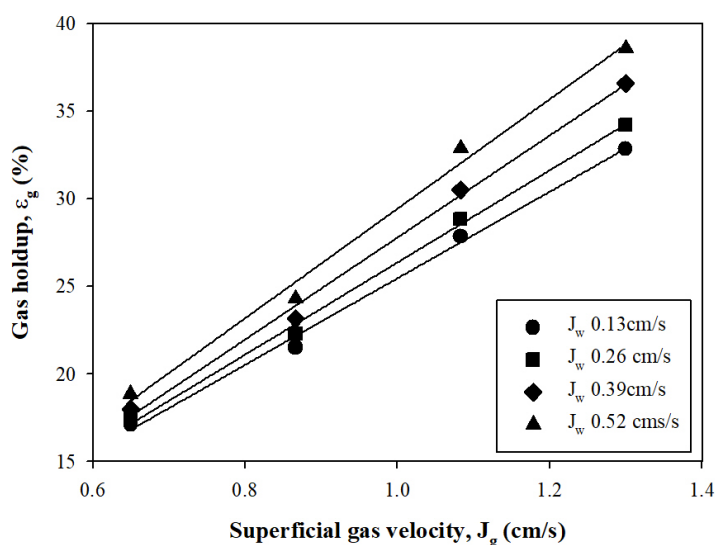


Figure 7. The variation in gas holdup as a function of superficial gas velocity.

4.3. Bubble-Rising Velocity

Figure 8 shows the bubble-rising velocity (U_b) as a function of the bubble size (d_b). The bubble-rising velocity was calculated using Equation (10). As shown in the figure, the rising velocity increased as the bubble size increased, indicating that the bubble size and bubble-rising velocity were proportional. Tan et al. (2013) reported that when the frother concentration increases, the bubble size decreases and the rising velocity decreases at this time [44]. In this study, the bubble rising velocity was 6.48 cm/s at a bubble size of 0.51 mm and 18.03 cm/s at a bubble size of 0.83 mm. This is because the buoyancy increases as the bubble size increases, which in turn increases the rising velocity. Leiva et al. (2010) compared the bubble rise velocity with the frother concentration and gas velocity and reported that the bubble rise velocity increased as the bubble size and gas velocity increased. They also demonstrated that the error in the bubble size for larger bubble sizes and gas velocities was more extensive than that for smaller bubble sizes and gas velocities, owing to the sensitivity of the measurement mechanism [45]. Leonard et al. (2021) reported that the bubble-rising velocity affected the pressure, temperature, wash water velocity, gas velocity, etc. [46]. The bubble-rising velocity is a critical parameter for optimizing bubble residence time and gas dispersion. When the rise velocity is excessive, bubbles may not adequately capture mineral particles, and gangue particles could be entrained, resulting in a low grade. Conversely, an insufficient bubble-rising velocity reduces the probability of particle–bubble interactions. Therefore, an optimal bubble-rising velocity is essential to maximize valuable mineral capture and enhance recovery efficiency. Hence, it is possible to improve flotation efficiency, including the stability and maintenance of the column regime, by adjusting the bubble-rising velocity and bubble size.

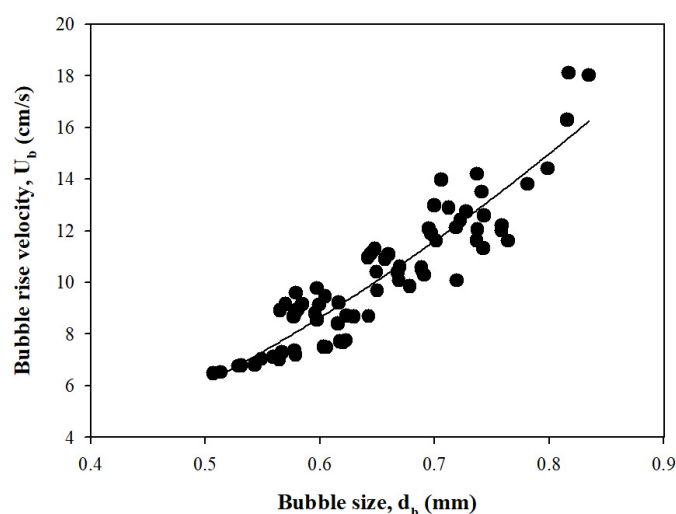


Figure 8. Relationship of bubble size versus bubble-rising velocity.

4.4. Bubble Surface Area Flux

Figure 9a shows the bubble surface area flux (S_b) as a function of the bubble size at different gas velocities (J_g). The bubble surface area flux was calculated using Equation (13). At each gas velocity, the bubble surface area flux decreases linearly as the bubble size increases. The bubble surface area flux showed a minimum value of 62.58 cm²/s/cm² at a gas velocity of 0.65 cm/s and a maximum value of 111.86 cm²/s/cm² at a gas velocity of 1.29 cm/s. The bubble surface area flux values exhibited a sharper curve depending on the gas velocity rather than the bubble size. This trend is similar to the experimental results obtained by Nesset (2011) [47]. Increasing the gas velocity can increase the number of bubbles and decrease bubble size owing to stronger bubble breakup [40,45]. As the gas holdup is also a function of the gas holdup and bubble size, the bubble surface area flux was plotted against the measured gas holdup, as shown in Figure 9b. The resulting relationship is $S_b = 2.67 \epsilon_g$. This compares well with Finch et al.'s (2000) result of $S_b = 5.5 \epsilon_g$ [48]. The

correlation between the bubble surface area flux and the gas holdup was proportional. As shown in Figure 7, the gas holdup depends on the gas velocity, wash water velocity, and frother concentration, which are related to the bubble size. As the gas holdup increased, more bubbles were generated in the column. In addition, smaller bubbles tend to increase both the gas holdup and bubble surface area flux, which have a higher surface area of rising bubbles per unit of cross-sectional area of the column per unit time, leading to a longer residence time and lower rise in the column [8,47]. In addition, the bubble surface area flux is too low and the collision opportunities between bubbles and particles decrease, leading to lower recovery. Conversely, excessively high bubble surface area flux can cause turbulence, resulting in the entrainment of gangue particles and a reduced grade. Effectively regulating the bubble surface area flux optimizes bubble–particle interactions, improving flotation efficiency. Therefore, the bubble surface area flux can be estimated by the measurement and/or relationship of gas velocity, gas holdup, and bubble size and is considered a critical variable that characterizes gas dispersion in a flotation column.

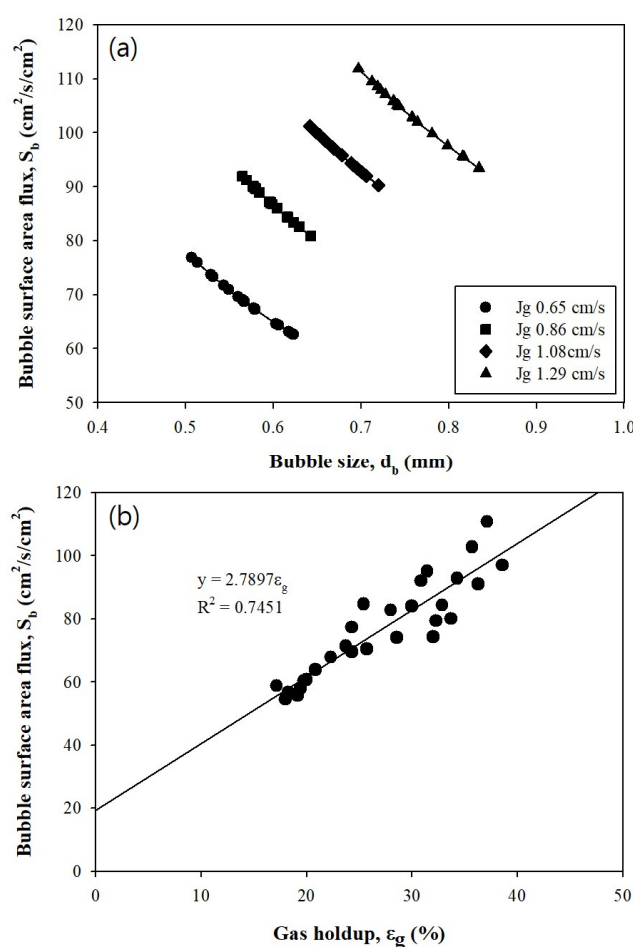


Figure 9. (a) Bubble surface area flux as a function of bubble size; (b) plot of bubble surface area flux and gas holdup.

4.5. Bubble Size Estimation

Figure 10 shows the relationship between the measured and calculated bubble sizes (d_b) in the porous-type column flotation. The actual bubble size was measured using a bubble size measuring system and the ZEISS image analysis program. The bubble size was estimated using Equations (4) and (5), and the linear model was successfully obtained from the relationship between the estimated d_b values and measured d_b values. The model obtained was presented at a 95% confidence level and under 0.05 p -values. The regression analysis of the relationship between the bubble sizes showed an R^2 of 0.86, and the average

bubble size was evaluated to be 0.64 mm. Errors in bubble size estimation may be caused by various operating conditions that affect bubble formation. The error range of the bubble size was $\pm 13\%$, which was similar to the error range in the studies of Yianatos et al. (1988) and Luis Vinnett et al. (2022) [49,50]. As a result of this study, it was noted that the bubble size and bubble distribution could be controlled under the current operating conditions ($J_g = 0.65\text{--}1.3$ cm/s, $J_w = 0.13\text{--}0.52$ cm/s, frother = 30–120 ppm), leading to the stability and maintenance of the column regime. A more systematic study on column flotation, including the gas dispersion properties between various operating variables and sparger types, is required.

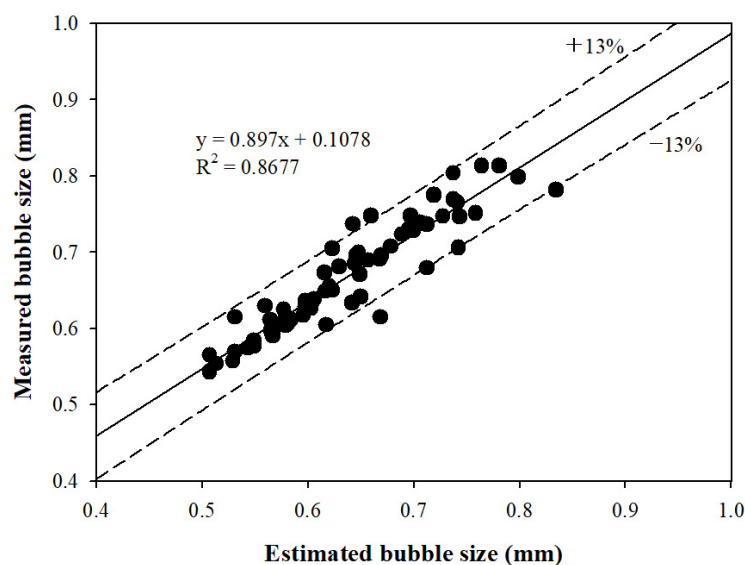


Figure 10. Relationship between measured and estimated bubble size in flotation column ($J_g = 0.65\text{--}1.3$ cm/s, $J_w = 0.13\text{--}0.52$ cm/s, frother = 30–120 ppm).

5. Conclusions

This study investigated bubble size measurement methods and the correlation between operating variables and gas dispersion properties in column flotation. The main results are summarized as follows:

1. Increasing the frother concentration significantly reduces bubble size, with a CCC identified at 120 ppm, above which bubble coalescence is effectively prevented. Increasing the frother concentration from 30 to 120 ppm changed the BSD from bimodal to unimodal, resulting in finer bubbles with minimum sizes of approximately 0.62 mm.
2. Gas and wash water velocities substantially influenced bubble size, with gas holdup peaking at 27% at 1.08 cm/s a gas velocity. A higher gas velocity increases the bubble size through coalescence, whereas a higher wash water velocity reduces it, owing to the increased pressure. Optimizing these variables, along with the frother concentration, can enhance column stability and flotation performance.
3. Bubble surface area flux decreases linearly with increasing bubble size and is significantly affected by gas velocity, ranging from 62.58 to 111.86 cm²/s/cm² at gas velocities of 0.65 and 1.29 cm/s, respectively. The relationship between the bubble surface area flux and gas holdup is proportional, and both are influenced by the gas velocity, wash water velocity, and frother concentration.
4. A strong correlation ($R^2 = 0.86$) between measured and calculated bubble sizes was achieved, with an average bubble size of 0.64 mm and an estimation error of $\pm 13\%$. The study demonstrates that bubble size and distribution can be effectively controlled under specific operational conditions ($J_g = 0.65\text{--}1.3$ cm/s, $J_w = 0.13\text{--}0.52$ cm/s, frother = 30–120 ppm). Further research on gas dispersion properties and sparger types is recommended to optimize flotation column performance.

Currently, Samyang Mining's molybdenum flotation plant in Korea is in demand for high-grade concentrates for lubricant applications. However, a low-grade concentrate with 88% MoS₂ is being produced. Therefore, it is predicted that the application of column flotation processes, along with the bubble size and gas dispersion characteristics evaluated in this study, could improve the flotation efficiency of high-grade concentrates to 98% or higher.

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Data Availability Statement: All data generated or analyzed during this study are included in this published article.

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References

1. Trass, O. Characterization and preparation of biomass, oil shale and coal-based feedstocks. In *Advances in Clean Hydrocarbon Fuel Processing*; Elsevier: Amsterdam, The Netherlands, 2011; pp. 3–53.
2. Mathimani, T.; Mallick, N. A comprehensive review on harvesting of microalgae for biodiesel—key challenges and future directions. *Renew. Sustain. Energy Rev.* **2018**, *91*, 1103–1120. [[CrossRef](#)]
3. Sakr, M.; Mohamed, M.M.; Maraqa, M.A.; Hamouda, M.A.; Hassan, A.A.; Ali, J.; Jung, J. A critical review of the recent developments in micro–nano bubbles applications for domestic and industrial wastewater treatment. *Alex. Eng. J.* **2022**, *61*, 6591–6612. [[CrossRef](#)]
4. Jameson, G.J.; Emer, C. Effect of bubble loading on the recovery of coarse mineral particles by flotation. *Miner. Eng.* **2024**, *215*, 108788. [[CrossRef](#)]
5. Miettinen, T.; Ralston, J.; Fornasiero, D. The limits of fine particle flotation. *Miner. Eng.* **2010**, *23*, 420–437. [[CrossRef](#)]
6. Reis, A.S.; Filho, A.; Carvalho, G.; Barrozo, M.A. Effect of surfactant on bubble size and air holdup on column flotation. *Mater. Sci. Forum* **2017**, *899*, 71–76. [[CrossRef](#)]
7. Ahmed, N.; Jameson, G. The effect of bubble size on the rate of flotation of fine particles. *Int. J. Miner. Process.* **1985**, *14*, 195–215. [[CrossRef](#)]
8. Gorain, B.K. The Effect of Bubble Surface Area Flux on the Kinetics of Flotation and Its Relevance to Scale-Up. Ph.D. Thesis, The University of Queensland, Brisbane, Australia, 1997.
9. Thomas, B.G.; Denisov, A.; Bai, H. Behavior of argon bubbles during continuous casting of steel. In *Proceedings of the Steelmaking Conference Proceedings*, Chicago, IL, USA, 13–16 April 1997; pp. 375–384.
10. Pandit, A.; Varley, J.; Thorpe, R.; Davidson, J. Measurement of bubble size distribution: An acoustic technique. *Chem. Eng. Sci.* **1992**, *47*, 1079–1089. [[CrossRef](#)]
11. Nikhita, B.; Deekshith, P.; Guguluto, S. A Review of Methodologies to Determine Bubble Diameter and Bubble Velocity. *Int. J. Sci. Res. Publ.* **2012**, *2*, 11–20.
12. Mazahernasab, R.; Ahmadi, R.; Ravanasa, E. Direct bubble size measurement in a mechanical flotation cell by image analysis and laser diffraction technique-A comparative study. *Iran. J. Chem. Chem. Eng.* **2021**, *40*, 1653–1664.
13. Ksentini, I.; Kotti, M.; Ben Mansour, L. Effect of liquid phase physicochemical characteristics on hydrodynamics of an electroflotation column. *Desalination Water Treat.* **2014**, *52*, 3347–3354. [[CrossRef](#)]
14. Nasset, J.E.; Hernandez-Aguilar, J.R.; Acuna, C.; Gomez, C.O.; Finch, J.A. Some gas dispersion characteristics of mechanical flotation machines. *Miner. Eng.* **2006**, *19*, 807–815. [[CrossRef](#)]
15. Seger, M.; Oliveira, C.; Rodrigues, R. Development of a laboratory-scale flotation column with inlet bubble size measurement. *Miner. Eng.* **2019**, *142*, 105936. [[CrossRef](#)]
16. Ekanlou, A.; Khalesi, M.; Abdollahy, M.; Hemmati Chegeni, M. Interactional effects of bubble size, particle size, and collector dosage on bubble loading in column flotation. *J. Min. Environ.* **2018**, *9*, 107–116.
17. Riquelme, A.; Desbiens, A.; Del Villar, R.; Maldonado, M. Identification of a non-linear dynamic model of the bubble size distribution in a pilot flotation column. *Int. J. Miner. Process.* **2015**, *145*, 7–16. [[CrossRef](#)]
18. Grau, R.; Heiskanen, K. Visual technique for measuring bubble size in flotation machines. *Miner. Eng.* **2002**, *15*, 507–513. [[CrossRef](#)]
19. Kracht, W.; Finch, J. Bubble break-up and the role of frother and salt. *Int. J. Miner. Process.* **2009**, *92*, 153–161. [[CrossRef](#)]

20. Besagni, G.; Inzoli, F. The effect of liquid phase properties on bubble column fluid dynamics: Gas holdup, flow regime transition, bubble size distributions and shapes, interfacial areas and foaming phenomena. *Chem. Eng. Sci.* **2017**, *170*, 270–296. [\[CrossRef\]](#)
21. Alam, D.; Lee, S.; Hong, J.; Fletcher, D.F.; Liu, X.; McClure, D.; Cook, D.; de Carfort, J.I.N.; Krühne, U.; Cullen, P. Influence of bubble size on perfluorooctanesulfonic acid degradation in a pilot scale non-thermal plasma treatment reactor. *Chem. Eng. J.* **2024**, *489*, 151349. [\[CrossRef\]](#)
22. Grau, R.A.; Laskowski, J.S.; Heiskanen, K. Effect of frothers on bubble size. *Int. J. Miner. Process.* **2005**, *76*, 225–233. [\[CrossRef\]](#)
23. Capponi, F.; Matiolo, E.; Rodrigues, R.; Rubio, J. Flotação extensora de finos de minérios de cobre e molibdênio. *An. Do* **2005**, *21*, 235–242.
24. Reis, A.S.; Reis Filho, A.M.; Demuner, L.R.; Barrozo, M.A. Effect of bubble size on the performance flotation of fine particles of a low-grade Brazilian apatite ore. *Powder Technol.* **2019**, *356*, 884–891. [\[CrossRef\]](#)
25. Finch, J.A.; Dobby, G. Column flotation. In *Flotation Science and Engineering*; CRC Press: Boca Raton, FL, USA, 1990; pp. 291–329.
26. Riquelme, A.; Desbiens, A.; Del Villar, R.; Maldonado, M. Predictive control of the bubble size distribution in a two-phase pilot flotation column. *Miner. Eng.* **2016**, *89*, 71–76. [\[CrossRef\]](#)
27. Wallis, G.B. *One-Dimensional Two-Phase Flow*; Courier Dover Publications: New York, NY, USA, 2020.
28. Masliyah, J.H. Hindered settling in a multi-species particle system. *Chem. Eng. Sci.* **1979**, *34*, 1166–1168. [\[CrossRef\]](#)
29. Yianatos, J.; Finch, J.; Laplante, A. Selectivity in column flotation froths. *Int. J. Miner. Process.* **1988**, *23*, 279–292. [\[CrossRef\]](#)
30. Richardson, J.T. Sedimentation and fluidization: Part-1. *Trans. Inst. Chem. Eng.* **1954**, *32*, 35–53.
31. Laskowski, J. Fundamental properties of flotation frothers. In Proceedings of the 22nd International Mineral Processing Congress, Cape Town, South Africa, 29 September–3 October 2003; pp. 788–797.
32. Mendes, T.F.; Reis, A.S.; Silva, A.C.; Barrozo, M.A. Analysis of the Effect of Surfactants on the Performance of Apatite Column Flotation. *Minerals* **2024**, *14*, 840. [\[CrossRef\]](#)
33. Han, Y.; Zhu, J.; Shen, L.; Zhou, W.; Ling, Y.; Yang, X.; Wang, S.; Dong, Q. Bubble size distribution characteristics of a jet-stirring coupling flotation device. *Minerals* **2019**, *9*, 369. [\[CrossRef\]](#)
34. Cho, Y.-S.; Laskowski, J. Effect of flotation frothers on bubble size and foam stability. *Int. J. Miner. Process.* **2002**, *64*, 69–80. [\[CrossRef\]](#)
35. Wang, H.; Yang, W.; Yan, X.; Wang, L.; Wang, Y.; Zhang, H. Regulation of bubble size in flotation: A review. *J. Environ. Chem. Eng.* **2020**, *8*, 104070. [\[CrossRef\]](#)
36. Rodrigues, R.; Rubio, J. New basis for measuring the size distribution of bubbles. *Miner. Eng.* **2003**, *16*, 757–765. [\[CrossRef\]](#)
37. Krishna, R.; De Swart, J.W.; Ellenberger, J.; Martina, G.B.; Maretto, C. Gas holdup in slurry bubble columns: Effect of column diameter and slurry concentrations. *AIChE J.* **1997**, *43*, 311–316. [\[CrossRef\]](#)
38. Pandit, A.; Joshi, J. Three phase sparged reactors—Some design aspects. *Rev. Chem. Eng.* **1984**, *2*, 1–84. [\[CrossRef\]](#)
39. McMahon, A.M. Modelling the Flow Behaviour of Gas Bubbles in a Bubble Column. 2009. Available online: <https://open.uct.ac.za/items/c038e9c0-fcde-47b6-b6c1-06dce28d230c> (accessed on 1 August 2024).
40. Prakash, R.; Sangtam, B.T.; Hembrom, K.; Majumder, S.K. Bubble size analysis in a two-phase countercurrent flow in the narrow rectangular column. *Phys. Fluids* **2022**, *34*, 043305. [\[CrossRef\]](#)
41. Panjipour, R.; Karamoozian, M.; Albijanic, B. Investigations of gas holdup, interfacial area of bubbles and bubble size distributions in a pilot plant flotation column. *Miner. Eng.* **2021**, *164*, 106819. [\[CrossRef\]](#)
42. Mwandawande, I.; Akdogan, G.; Bradshaw, S.; Karimi, M.; Snyders, N. Prediction of gas holdup in a column flotation cell using computational fluid dynamics (CFD). *J. South. Afr. Inst. Min. Metall.* **2019**, *119*, 81–95. [\[CrossRef\]](#)
43. Han, J.; Li, Y.; Chen, P. A study of bubble size/shape evolution in microbubble countercurrent contacting flotation column. *Asia-Pac. J. Chem. Eng.* **2023**, *18*, e2865. [\[CrossRef\]](#)
44. Tan, Y.; Rafiei, A.; Elmahdy, A.; Finch, J. Bubble size, gas holdup and bubble velocity profile of some alcohols and commercial frothers. *Int. J. Miner. Process.* **2013**, *119*, 1–5. [\[CrossRef\]](#)
45. Leiva, J.; Vinnett, L.; Contreras, F.; Yianatos, J. Estimation of the actual bubble surface area flux in flotation. *Miner. Eng.* **2010**, *23*, 888–894. [\[CrossRef\]](#)
46. Leonard, C.; Ferrasse, J.-H.; Lefevre, S.; Viand, A.; Boutin, O. Bubble rising velocity and bubble size distribution in columns at high pressure and temperature: From lab scale experiments to design parameters. *Chem. Eng. Res. Des.* **2021**, *173*, 108–118. [\[CrossRef\]](#)
47. Nesset, J.E. Modeling the Sauter Mean Bubble Diameter in Mechanical Forced-Air Flotation Machines. 2011. Available online: <https://escholarship.mcgill.ca/concern/theses/1831ck330> (accessed on 1 September 2024).
48. Hernandez, H.; Gomez, C.; Finch, J. Gas dispersion and de-inking in a flotation column. *Miner. Eng.* **2003**, *16*, 739–744. [\[CrossRef\]](#)
49. Yianatos, J.; Finch, J.; Dobby, G.; Xu, M. Bubble size estimation in a bubble swarm. *J. Colloid Interface Sci.* **1988**, *126*, 37–44. [\[CrossRef\]](#)
50. Vinnett, L.; Urriola, B.; Orellana, F.; Guajardo, C.; Esteban, A. Reducing the presence of clusters in bubble size measurements for gas dispersion characterizations. *Minerals* **2022**, *12*, 1148. [\[CrossRef\]](#)

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