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DETERMINATION OF BUBBLE SIZE DISTRIBUTION IN A LABORATORY MECHANICAL FLOTATION CELL BY A LASER DIFFRACTION TECHNIQUE

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Abstract: In this study, a laser diffraction technique (LDT) was used to measure size distribution of bubbles generated in a two-phase system in a laboratory mechanical flotation cell. In LDT, a laser light beam passed through the bubbles inside the measurement cell and the scattered light was recorded by detectors. In order to show the effectiveness of LDT, an image analysis technique (IAT) was applied in parallel to measure the size of bubbles. To determine the bubble size by IAT, around 200 images were taken in each test. In addition, the important operating parameters of the mechanical flotation cell affecting the bubble size distribution, including the impeller speed, aeration rate and frother concentration, were investigated. The response parameter in this study was $D_b(50)$ which represent the size of bubble at which there is 50% of the distribution.

The results of this study showed that the LDT and IAT techniques were in a good agreement when $D_b(50)$ was in the range of -800+400 μm and there was a discrepancy for $D_b(50)$ in the range of -400+100 μm . Furthermore, $D_b(50)$ decreased from 727 to 284 μm when impeller speed increased from 700 to 1200 rpm. Additionally, an increase in the aeration rate from 1 dm^3/min to 2.5 dm^3/min led to a rise in $D_b(50)$ from 418 to 456 μm . Finally, increasing the frother concentration from 10 to 60 ppm reduced the $D_b(50)$ from 704 to 387 μm .

Keywords: bubble size distribution, mechanical flotation cell, Laser Diffraction Technique (LDT), Image Analysis Technique (IAT), effective parameters

Introduction

Flotation is one of the most widely used methods for separation of valuable minerals particularly base metals. It is well documented that a bubble size distribution has a direct influence on the flotation performance (Grau and Heiskanen, 2005; Zhang et al., 2010). An image analysis technique (IAT), a standard technique in bubble size distribution measurement, has had significant advancements in the equipment, software and processing procedure. However, problems such as time consuming

analysis, bubbles overlap and perspective in images still exist in this method. A laser diffraction technique (LDT) was introduced in the mid 1970's but its instrumentation has only had a strong development in the past two decades. This technique is classified as a non-destructive and non-intrusive method and relies on the fact that the laser diffraction angle is inversely proportional to the particle size (Xu, 2002). LDT uses optical models to determine the particle/bubble size from measurement data. One of these models is the Fraunhofer model in which the pattern of scattered light is predicted by a disc at the time of exposure to the laser beam. The Fraunhofer model has shown satisfactory results for some particles but it has not been able to describe the light scattering accurately (Ma et al., 2000). The accepted theory for all materials and in all circumstances that accurately predicts the behavior of light scattering is the Mie theory. This theory is derived from the Maxwell equations describing electromagnetic radiation for the light scattered by a homogeneous sphere under uniform illumination. In the Mie model, information on the optical properties of particles and dispersants such as a refractive index is essential.

LDT has been applied by a laser particle size analyzer (LPSA) to measure the size of the particles/bubbles in an aqueous or aerial environment. A typical LPSA system consists of a laser light source of He-Ne, which is a red light with the wavelength of 633 μm in the axis of device, LED which is a blue light with the wavelength of 455 μm out of axis of device, wet or dry dispersing units, Fourier lens, suitable detectors and a PC for signal processing and reporting the results (Ma et al., 2000). In LDT, the focused laser light beam passes through the suspended sample and is scattered by either particles or bubbles in various angles. Light scattering is recorded by silicon detectors or light-sensitive diodes. Then, a computer calculates the experimental size distribution, E_{mes} , through an optical model (Stojanovic and Markovic, 2012) and predicts the scattering pattern, E_{cal} , using the mathematical procedures. The size distribution is then determined by comparison of E_{mes} and E_{cal} until the sum of the squared errors reaches to a minimum value. Finally, the statistics of bubble distribution are calculated from the results using the derived diameter, $D[m,n]$, given by:

$$D[m, n] = \left[\frac{\sum v_i d_i^{m-3}}{\sum v_i d_i^{n-3}} \right]^{\frac{1}{m-n}} \quad (1)$$

where v_i and d_i are the volume fraction and geometric diameter of size band i , respectively. LDT which is applicable for measuring the particle size range from 0.1 to 3000 μm according to ISO13320-1 (1999), has successfully been used to measure dry solids, colloidal particles, and emulsions. Regarding non-solid particle measurement, this method was developed in the size distribution determination of colloidal gas afrons (CGA), which is a type of foam used in a separation process (Couto et al., 2004). Additionally, some researchers have used this technique to determine the size distribution of fine bubbles in the micrometer and nanometer scales. Fan (2008) determined the size of pico bubbles produced by a Venturi tube in a plexiglass column

using a laser particle size analyzer. The bubble size range in the pico bubbles moving from the column to LPSA was determined to be between 0.1 μm and 100 μm . In another study, Couto et al. (2008) measured the size distribution of bubbles in dissolved air flotation (DAF) using LDT. By comparing LDT with a fluid flow dynamics method, they confirmed that the results of LDT are reproducible and reliable. However, it is worth to note that the volume of dissolved air in DAF is 3-4% which is much lower than that of 20-30% in the actual flotation process. Moreover, the number of bubbles in DAF is very low in comparison to the real flotation process (Rubio et al., 2007). Ahmadi (2013) designed a nano-micro bubble producer device on a basis of hydrodynamic cavitation and used LDT to measure the nano-micro bubble size distribution. He evaluated LDT as a fast and reliable method for bubble size measurement. One advantage of LDT is that the size analysis of sample could be performed in less than one minute and therefore, it can be used in industrial operations due to the easy and fast repetition.

According to the literature review, the IAT has mostly been used to measure the size of bubbles generated in the mechanical flotation cells in the range of millimeter in size. Furthermore, the LDT has only been applied for measurement of bubbles with sub-micron sizes and hence, there is no comprehensive study to use this method in measuring the millimeter size range bubbles. Moreover, LDT and IAT have not been compared with each other in the bubble size measurement application. In this paper, LDT and IAT were simultaneously used to measure the size distribution of bubbles generated in a laboratory mechanical flotation cell. Then, LDT results were qualitatively and quantitatively compared with the IAT results. Finally, the effect of the operating variables of the laboratory mechanical flotation cell on the bubble size was investigated.

Materials and methods

In this study, the bubbles were produced in a laboratory Denver flotation cell which was constructed with special dimensions of 17×15×25 cm representing the length, width, and height, respectively, and a hole on one wall located at 2 cm above the bottom of the cell for quick transmission of generated bubbles to the bubble size measurement device. In all experiments, double-distilled water was used to prepare the aqueous solutions and methyl isobutyl carbinol (MIBC) with molecular weight of 102.17 g/mol from Aldrich Sigma was applied as a frother to generate bubbles. The aeration rate was controlled by an air flow meter with accuracy of 0.5 dm^3/min calibrated with oxygen. To prepare the aqueous solution in the flotation cell, the frother was added to water and mixed for 2 minutes. Then, the air was introduced into the cell with adjusted aeration rate.

The laser particle size analyzer (LPSA), model MS2000 manufactured by Malvern Company, UK, was applied to measure the size distribution of bubbles using LDT. Figure 1 shows the equipment used in the LDT measurements. As displayed in Figure

1, the flotation cell was placed next to LPSA and the bubbles generated inside the flotation cell were continuously transferred into the measurement cell of the LPSA using pressure difference. In LPSA, the Mie optical model was selected to calculate the bubble size distribution. To increase the accuracy of the results, each measurement was repeated four times and the median bubble diameter was calculated on a volumetric basis. The average of four measurements was shown as $D_b(50)$ which represents the size of bubble at which there is 50% of the distribution.

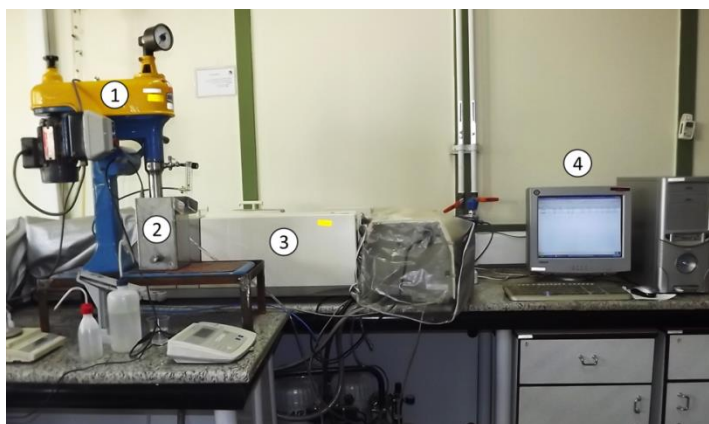


Fig. 1. Equipment used in LDT measurements:
1) flotation machine, 2) flotation cell, 3) LPSA, 4) PC

To investigate the effectiveness of the LDT results, the IAT measurements were also conducted. Figure 2 shows the equipment used in the IAT measurements. The equipment displayed in Figure 2 includes a bubble viewer, digital camera and light source. The bubble viewer consists of a sampling tube and a viewing chamber with certain dimensions created out of Plexiglas. The 15° slope of the chamber makes the bubbles to move almost as a single layer near the viewing chamber screen and decreases overlapping of the bubbles in pictures. A digital camera, Canon 5D Mark II with micro lens, was positioned in a fixed location and the imaging depth was adjusted based on the presence of the majority of bubbles. In addition, the measurement scale was placed exactly at the focal plane and the sharp and clear bubbles were selected for the processing step, whereas the blurry bubbles were removed. The light source producing cool light was perpendicular to the rear panel. To increase the accuracy of the measurements, at least 200 images were taken in each test out of which a few were randomly chosen for the processing step, in which the cross-sectional area and consequently the diameter of bubbles known as “projected area diameter” were determined. Finally, the $D_b(50)$ and size distributions of bubbles were calculated.

To confirm the reproducibility of bubble size measurements, four measurements were performed under the same conditions in each test. The coefficient of variation of measurements was 1.64 % which was acceptable according to BS ISO 13320-1(1999).

Moreover, the weighted residual values of tests, which represent the fit of the calculated data obtained by model with the measured data, were in the range of 0.46-0.75%. The weighted residual value less than 1% indicates a good and acceptable fit and greater than 1% indicates the wrong choice of refractive index values.

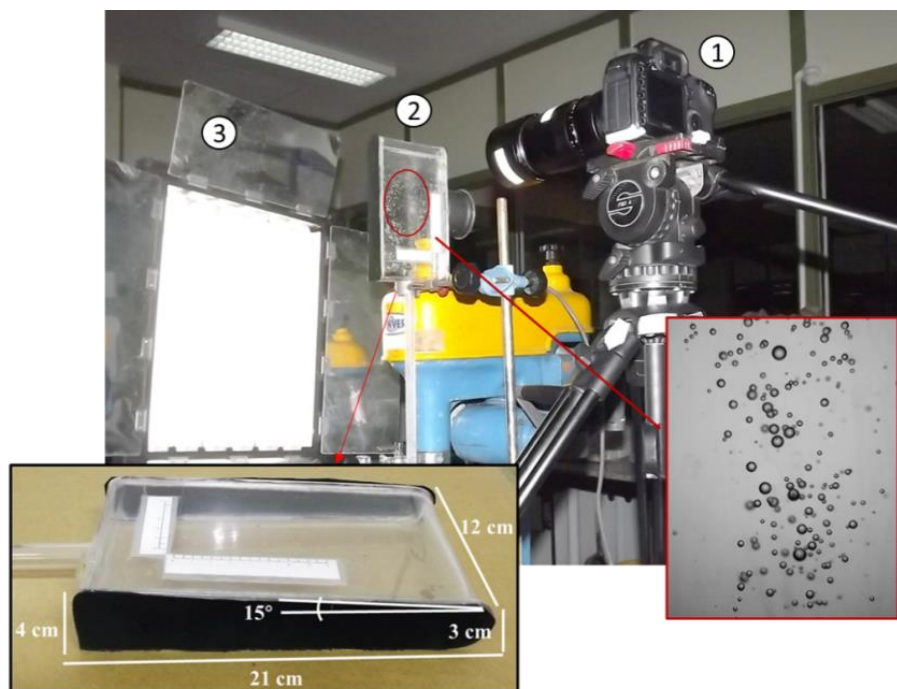


Fig. 2. Equipment used in IAT measurements:
1) digital camera, 2) bubble viewer, 3) light source

Results and discussion

Effect of impeller speed

The impeller speed of the mechanical flotation cell is one of the most effective parameters on the bubble size generated inside the cell. Figure 3 shows the effect of impeller speed on the bubble size distribution at aeration rate of $2 \text{ dm}^3/\text{min}$ and MIBC concentration of 30 ppm. As shown in Fig. 3, increasing the impeller speed from 700 to 1200 rpm shifted the bubble size distribution to finer sizes and decreased the $D_b(50)$ of bubbles from 727 to 284 μm . Figure 4 displays the image of bubbles at different impeller speeds. It is clear from Figure 4 that the smallest bubbles were generated when the impeller speed was at its highest value of 1200 rpm.

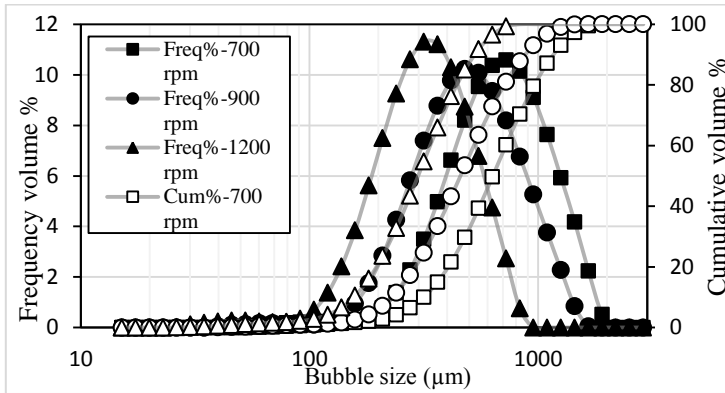


Fig. 3. Effect of impeller speed on bubble size distribution at aeration rate of 2 dm³/min and MIBC concentration of 30 ppm

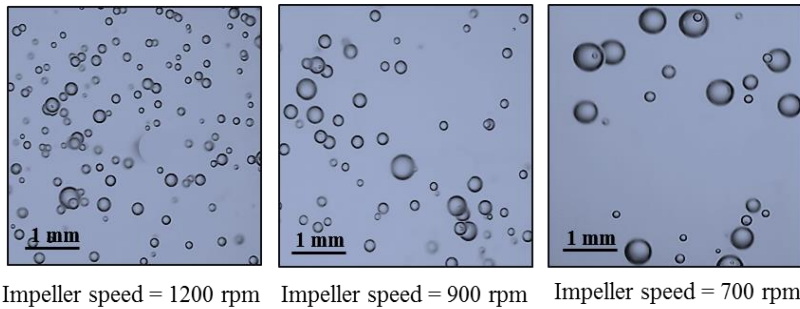


Fig. 4. Images of bubbles at different impeller speeds

Other studies have also shown that increasing the impeller speed decreased the bubble size distribution (Gorain et al., 1994; Yang and Aldrich, 2005; Miskovic, 2011). It is well understood that the size of bubbles in a flotation system is a function of three hydrodynamic processes, including bubble production in the gas generator, bubble breakage and bubble coalescence. The last two mechanisms are controlled by the environment turbulence which is restrained with the impeller speed (Miskovic, 2011). The role of the impeller is to dissolve air in water, produce bubbles and thus, more air is dispersed in water when impeller speed is higher. Subsequently, the bubble breakage in higher impeller speeds leads to smaller bubble size.

Gorain et al. (1998) carried out extensive experiments on three types of impellers in a flotation cell with a volume of 3 m³ in Tasmania and Western Australia. They introduced two empirical models to estimate the bubble surface flux S_b , and bubbles Sauter mean diameter (μm) d_{32} . The first model is:

$$S_b = 123 N_s^{0.44} \left(\frac{Q}{A}\right)^{0.75} A_s^{-0.10} P_{80}^{-0.42} \tag{2}$$

where N_s is the peripheral impeller speed (m/s), Q aeration rate (cm^3), A section area (cm^2), A_s aspect ratio of impeller and P_{80} is the size of sieve through which 80% of particles pass (μm). The second model is:

$$d_{32} = \frac{6 J_g}{S_b} \quad (3)$$

where, J_g is the superficial gas velocity (cm/s). According to the Gorain models (Eqs. 2 and 3), increasing the impeller speed increases the bubble surface flux, which consequently reduces the Sauter mean diameter of bubbles.

Effect of aeration rate

Figure 5 shows the effect of aeration rate on bubble size distribution at impeller speed of 900 rpm and MIBC concentration of 30 ppm. As exhibited in Fig. 5, increasing the aeration rate from 1 to 2.5 dm^3/min increases $D_b(50)$ from 418 to 456 μm . It means that the size of the bubbles shifts toward larger sizes and wider distributions. Figure 6 demonstrates the image of bubbles at different aeration rates. It can be seen from that the size of bubbles was larger at higher aeration rates (Figure 6).

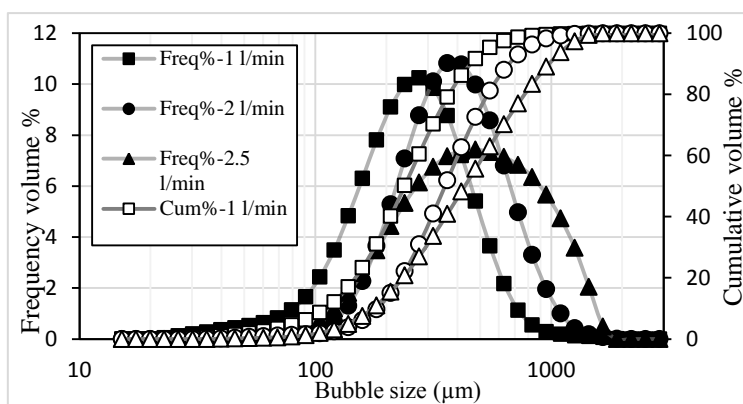


Fig. 5. Effect of aeration rate on bubble size distribution at impeller speed of 900 rpm and MIBC concentration of 30 ppm

Many studies have indicated that increasing the aeration rate in a mechanical flotation cell results in generating larger bubbles (Gorain et al., 1994; Bai and Thomas, 2001; Yang and Aldrich, 2005; Gomez and Finch, 2007; Zhang et al., 2014). Grau and Heiskanen (2005) reported that the effect of aeration rate on the bubble size is related to the air cavities behind the impeller blades, which become larger when the aeration rate increases. Therefore, energy consumption decreases which leads to rise in the maximum size of stable bubble. O'Connor et al. (1989) introduced a model, which predicts the bubble diameter d_b , in two-phase and three-phase systems:

$$d_b = kv_g^x \quad (4)$$

where k is a constant, v_g superficial gas velocity, and x is 0.40 for two-phase systems.

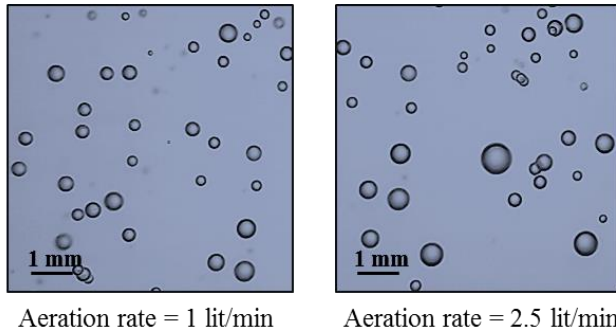


Fig. 6. Images of bubbles at different aeration rates

The O'Connor model (Eq. 4) shows that the aeration rate and bubble diameter have a direct relationship. Sada et al. (1978) suggested that the direct relationship between aeration rate and bubble size is due to higher bubble coalescence at high aeration rates.

Effect of frother concentration

Figure 7 displays the effect of MIBC concentration on bubble size distribution at impeller speed of 900 rpm and aeration rate of 2 dm³/min. As shown in Fig. 7, increasing the frother concentration from 10 to 60 ppm reduces the $D_b(50)$ from 704 to 387 μ m and moves the bubble size distribution toward finer sizes. Figure 8 shows the image of bubbles at different MIBC concentrations. It is evident from Fig. 8 that bubble size decreases at higher concentration of MIBC.

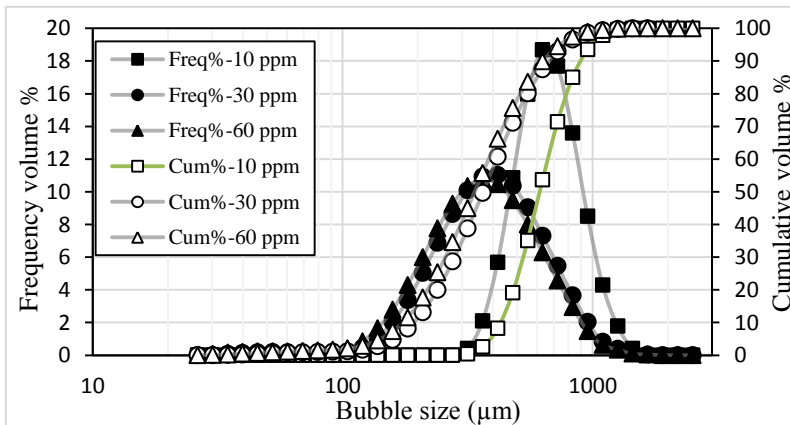
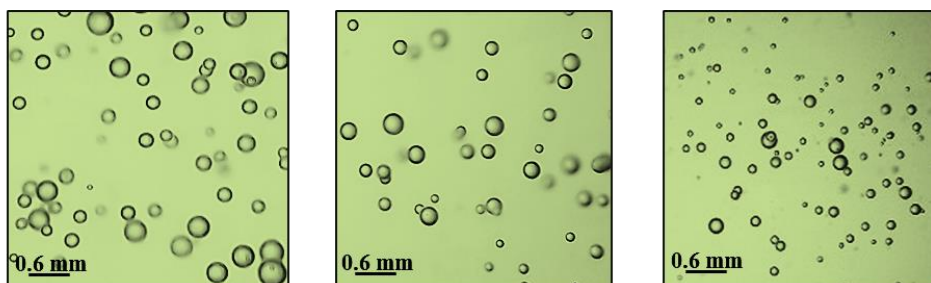


Fig. 7. Effect of MIBC concentration on bubble size distribution at impeller speed of 900 rpm and aeration rate of 2 dm³/min



MIBC concentration = 10 ppm MIBC concentration = 30 ppm MIBC concentration = 60 ppm

Fig. 8. Images of bubbles at different MIBC concentrations

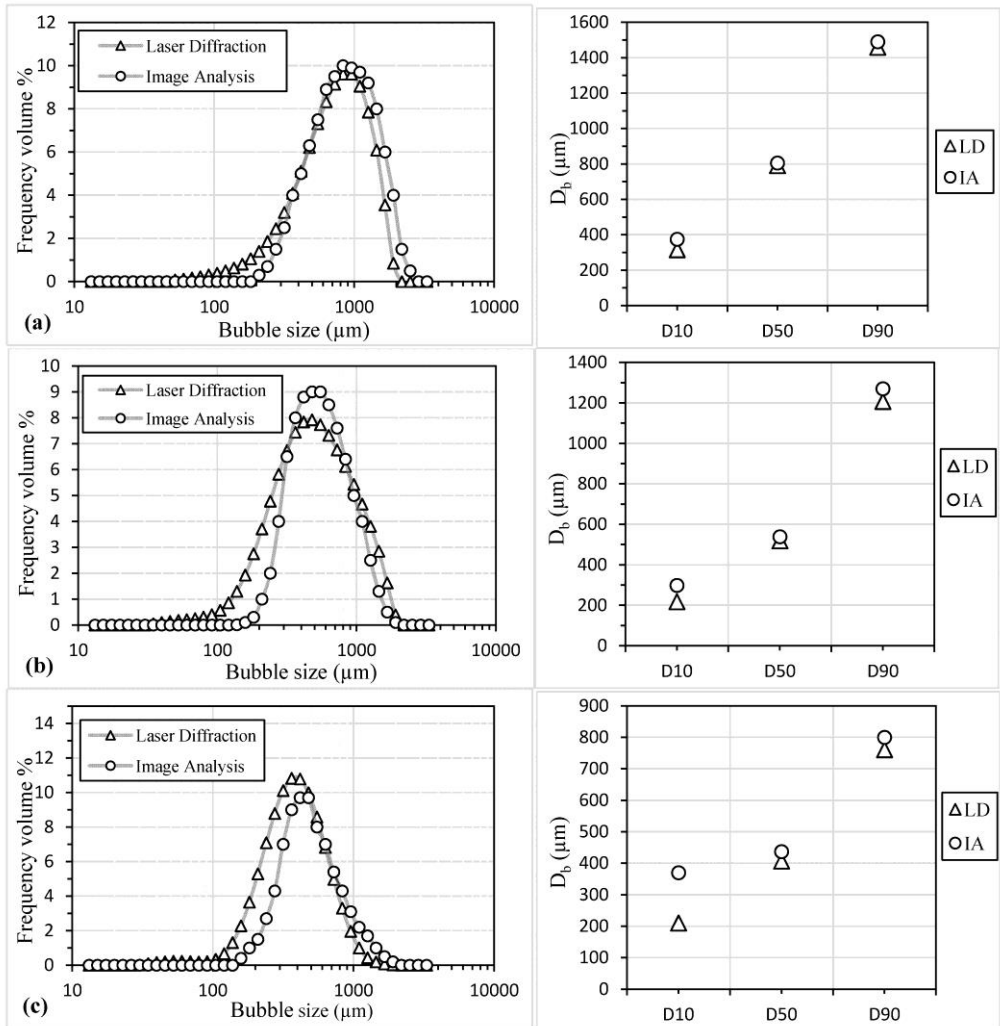
Increasing the frother concentration could reduce the surface tension and prevent bubble coalescence which consequently result in producing finer bubbles (O'Connor et al., 1989; Comely et al., 2002). However, there are some studies suggesting that the bubble size is not solely controlled by the surface tension. Gupta et al. (2007) studied the relationship between the bubble size and surface tension in the presence of two frothers including MIBC and DF-1012. They demonstrated that when DF-1012 is used as frother, although the surface tension is lower, the bubble size is larger in comparison to applying MIBC with the same concentration. Moreover, Moyo (2005) showed that adding some salts to the aqueous solution makes the bubbles finer, while increases the surface tension. Azgomi (2006) explained the effect of frother on the bubble size using the bubble coalescence prevention mechanism. He indicated that the frother molecules at the air/liquid interface create hydrogen bonds with water and make the liquid film on the bubble surface more stable. Bubble coalescence prevention is expressed based on the frother critical coalescence concentration (CCC) concept which means that the bubbles are produced in small sizes and frother prevents them from coalescence. Furthermore, Azgomi (2006) suggested that only a part of bubbles coalescence prevention takes place at CCC and there is another factor having a direct impact on the bubbles size, such as the surface tension.

Comparison of LDT and IAT

In order to investigate the effectiveness of LDT in measuring bubble size, the results of LDT and IAT experiments carried out in this study were compared together. Figures 9 compares the results of bubble size measurements using LDT and IAT. As shown in Figure 9, the results of LDT and IAT are in a good correlation when $D_b(50)$ is in the range of -800+400 μm (Figures 9a, 9b and 9c). However, as seen in Figure 8, the exact value of bubble size obtained by IAT is slightly larger than that measured by LDT. The slightly larger bubble size in IAT could be explained by the fact that in IAT measurements, bubbles move vertically and in a longer distance during sampling and before imaging. Therefore, the bubbles have a higher possibility to coalesce in IAT in comparison to LDT measurements, in which bubbles move in a horizontal and shorter

path toward the measurement device. Additionally, longer measurement time in IAT increases the possibility of bubble coalescence leading to larger bubble size measurements.

As shown in Figures 9d and 9e, there is a significant difference between the LDT and IAT results when $D_b(50)$ is in the range of $-400+100 \mu\text{m}$. It is clear from Figure 10b that the IAT measurements are larger than the LDT measurements. It could be due to the ineffectiveness of IAT in detecting and measuring the bubbles finer than $200 \mu\text{m}$ since IAT has mostly been used for determination of the bubbles larger than $200 \mu\text{m}$ (Azgomi et al., 2007; Gupta et al., 2007; Finch et al., 2008; Miskovic, 2011).



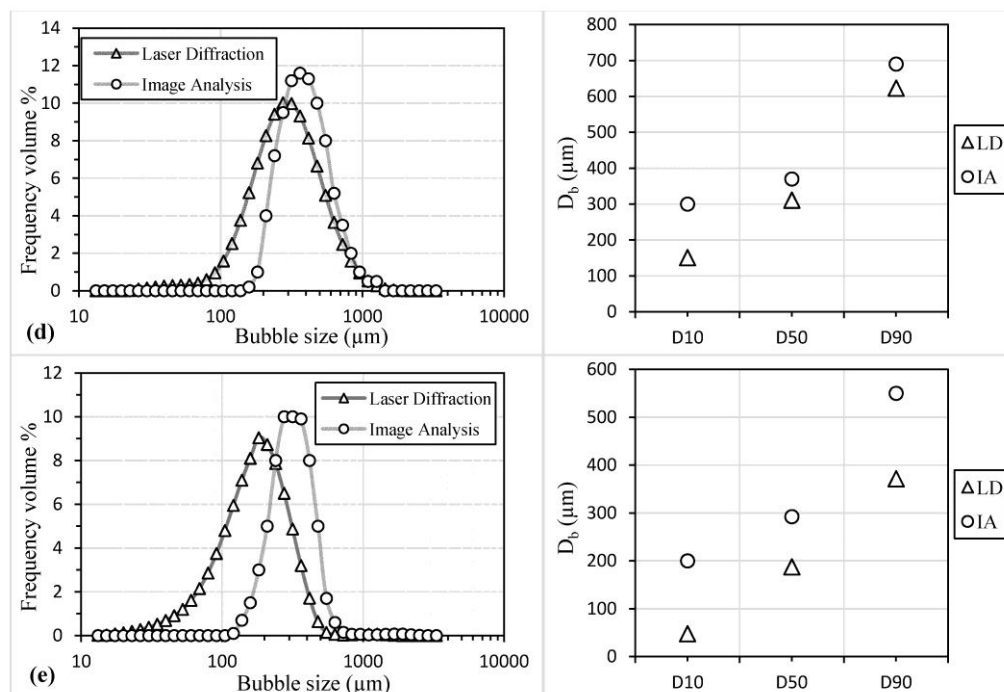


Fig. 9. Results of bubble size measurements using LDT and IAT

In mineral flotation systems, generating bubbles finer than 500 μm is instrumental in order to improve the collection efficiency of flotation (Jameson, 2010). IAT is well established and accurate method that could provide reliable results but it is a time consuming technique which requires collection and processing of large numbers of images because of unanticipated pitfalls. Therefore, LDT could be an effective technique to measure the size of bubbles in a flotation system. Due to the high speed and automation of LDT, the results are based on measuring a large number of bubbles, which increases the statistical significance of the measurements.

Conclusions

In this paper, the laser diffraction technique (LDT) was used to measure the size distribution of bubbles generated in a laboratory mechanical flotation cell. In order to increase the reliability of results, each measurement was repeated four times and the average of four measurements was reported. The coefficient of variation of the measurements was 1.64% and the weighted residual of the experiments was in the range of 0.46-0.75%. Moreover, the effects of floatation cell impeller speed, aeration rate, and frother concentration on the bubble size were investigated. The results showed that increasing the impeller speed from 700 to 1200 rpm decreased the $D_b(50)$

from 727 to 284 μm due to intensifying the cell turbulence. Moreover, increasing the aeration rate from 1 to 2.5 dm^3/min led to an increase in $D_b(50)$ from 418 to 456 μm . It was a result of growth in bubble coalescence. Finally, increasing the frother concentration from 10 to 60 ppm reduced the $D_b(50)$ from 704 to 387 μm .

To examine the reliability of the LDT results, the image analysis technique (IAT) was simultaneously applied to measure the bubble size. The comparing results showed that the LDT and IAT are in a good agreement for measuring the bubble size in the range of -800+400 μm . However, there was a discrepancy between LDT and IAT when $D_b(50)$ was in the range of -400+100 μm . In overall, the similarity between the measured size distributions by two techniques was satisfactory specifically for bubbles larger than 200 μm . This study demonstrated that the LDT could provide the bubble size distribution close to that obtained from IAT. The advantages of LDT including the ease, high speed and wide range of bubble size measurement introduce this technique as an attractive alternative to IAT for rapid measurement of bubble size in the mechanical flotation cells.

References

- AHMADI R., 2013. *Flotation of fine particles from mine tailings by coalescent of nano-microbubbles*, Doctoral Dissertation in Mineral Processing, Faculty of Engineering, Tarbiat Modarres University, Tehran, Iran.
- AZGOMI F., 2006. *Characterizing frothers by their bubble size control properties*, Master Dissertation in Metals and Materials Engineering, McGill University, Montreal, Canada.
- AZGOMI F., GOMEZ C.O., FINCH J.A., 2007. *Correspondence of gas holdup and bubble size in presence of different frothers*, Int. J. Miner. Process., 83, 1–11.
- BAI H., THOMAS B., 2001. *Bubble formation during horizontal gas injection into downward flowing liquid*, Metallurgical and Materials Transactions B, 32, 1143-1159.
- COMELY B.A., HARRIS P.J., BRADSHAW D.J., HARRIS M.C., 2002. *Frother characterization using dynamic surface tension measurements*, International Journal of Mineral Processing, 64, 81-100.
- COUTO H.J.B., MELO M.V., MASSARANI G., 2004. *Treatment of milk industry effluent by dissolved air flotation*, Brazilian Journal of Chemical Engineering, 21, 83-91.
- COUTO H.J.B., DANIAL G. Nunes, REINER N., SILVIA C.A. França, 2008. *Micro-bubble size distribution measurements by laser diffraction technique*, Minerals Engineering, 22, 330-335
- FAN M., 2008. *Picobubble enhanced flotation of coarse phosphate particles*, Doctoral Dissertation in Mineral Processing, College of Engineering, The University of Kentucky, China.
- FINCH J.A., NESSET J., ACUNA C., 2008. *Role of frother on bubble production and behaviour in flotation*, Miner. Eng., 21, 949–957.
- GOMEZ C.O., FINCH J.A., 2007. *Gas dispersion measurements in flotation cells*, International Journal of Mineral Processing, 84, 51-58.
- GORAIN B.K., FRANZIDIS J.P., MANLAPIG E.V., 1994. *Studies on impeller type, impeller speed and air flow rate in an industrial scale flotation cell- part1: effects on bubble size distribution*, Minerals Engineering, 8, 615-635.
- GORAIN B.K., FRANZIDIS J.P., MANLAPIG E.V., 1998. *The empirical prediction of bubble surface area flux in mechanical flotation cells from cell design and operation data*, Minerals Engineering, 12, 309-322.

- GRAU R.A., HEISKANEN, K., 2005. *Bubble size distribution in laboratory scale flotation cells*, Minerals Engineering, 18, 1164–1172.
- GUPTA A.K., BANERJEE P.K., MISHRA A., SATISH P., 2007. *Effect of alcohol and polyglycol ether frothers on foam stability, bubble size and coal flotation*, Int. J. Miner. Process., 82, 126–37.
- ISO 13320-1, 1999(E). *Particle Size Analysis- Laser diffraction methods*, Part 1, General Principles.
- JAMESON G. J., 2010. *New directions in flotation machine design*, Minerals Engineering, 23, 835-841.
- MA Z., MERKUS H.G., JAN G.A.E. de Smet, HEFFELS C., SCARLETT B., 2000. *New developments in particle characterization by laser diffraction: size and shape*, Powder Technology, 111, 66–78.
- MISKOVIC S., 2011. *An investigation of the gas dispersion properties of mechanical flotation cells: An in-situ approach*, Doctoral Dissertation in Mining and Minerals Engineering, Virginia Polytechnic Institute and State University, Blacksburg, Virginia.
- MOYO P., 2005. *Characterization of frothers by water carrying rate*, Doctoral Dissertation in Metals and Materials Engineering, McGill University, Montreal, Canada
- O'CONNOR C.T., RANDALL E.W., GOODALL C.M., 1989. *Measurement of the effects of physical and chemical variables on bubble size*, International Journal of Mineral Processing, 28, 139-149.
- RUBIO L., CARISSIMI E., ROSA J.J., 2007. *Flotation in water and wastewater treatment and reuse: recent trends in Brazil*, Int. J. Environment and Pollution, 30, 193.
- SADA E., YASUNZSHZ A., KATOH S., NZSHIOKA M., 1978. *Bubble formation in flowing liquid*, The Canadian Journal of Chemical Engineering, 56, 669-672.
- STOJANOVIC Z., MARKOVIC S., 2012. *Determination of particle size distribution by laser diffraction*, Technics-New Materials, 21, 11-20.
- XU R., 2002. *particle characterization: Light scattering methods*. Particle Technology Series, Chap. 3.
- YANG X., ALDRICH Ch., 2005. *Effects of impeller speed and aeration rate on flotation Performance of sulphide ore*, Trans. Nonferrous Met. SOC.China, 16, 185-190.
- ZHANG W., NESSET J.E., FINCH J.A., 2010. *Water recovery and bubble surface area flux in flotation*, Canadian Metallurgical Quarterly, 49, 353–362.
- ZHANG W., NESSET J.E., FINCH J.A., 2014. *Bubble size as a function of some situational variables in mechanical flotation machines*, J. Cent. South Univ., 21, 720–727.