

Bubbling properties of frothers and collectors mix system

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Abstract: This paper studies the effect of the type and concentration of selected frothers and collectors mix system on the bubble sizes (Sauter mean diameter, *SMD*) of bubbling flow produced in a micro flotation cell and the determination of bubble size distribution (*BSD*). The usage of dodecyl amine hydrochloride (*DAH*) collector on the critical coalescence concentration of commercial frothers *PPG200*, *PPG400*, and *PPG600* was investigated in detail. The results of these studies showed that the usage of *DAH* decreased the *CCC* of these frothers. Each frother + collector mixing system exhibited its unique ability in preventing coalescence of the bubbles in the order of *PPG200* < *PPG400* < *PPG600*. The factorial experiments established that the type of the frother, collector, and their concentration had a major effect on the size of the bubbles. The *BSD* in the presence of *PPG600* + *DAH* mix system resulted in a little bit wider *BSD* which indicated the effect of frother type in mixed systems.

Keywords: critical coalescence concentration (*CCC*), Sauter diameter (d_{32}), bubble size distribution (*BSD*)

1. Introduction

Flotation is a separation process providing the selective enrichment of particles based on their hydrophobicity degree and their affinity to attach to bubbles (Güven et al., 2015). In this manner, the flotation kinetics rate and the particle-bubble collision frequency strongly depend on bubble size and bubble size distribution (*BSD*). Therefore, the properties of frothers such as frother type and concentration have the strongest influence on the gas dispersion properties besides the effects of other parameters on bubble size (Batjargal et al., 2021). The effect of some commercial frothers on bubble size has been investigated recently (Grau et al., 2005; Szyszka, 2008). In these studies, it was reported that this effect can be ascribed to their ability on inhibiting bubble coalescence. In other words, when two bubbles come in and encounter each other, the liquid film between these bubbles becomes thinner and breaks which will cause the bubbles to coalesce (Leja, 1982). If the coalescence of bubbles does not occur within seconds, they rise to the surface and form a foam or froth. As it is well-known from the literature, the bubble size decreases rapidly until a transition value, referred to as the critical coalescence concentration (*CCC*) (Cho et al., 2002; Nasset et al., 2007). Although at concentrations above the *CCC*, bubble size does not materially decrease, it does lead to increased water recovery to the froth with the attendant increase in unselective particle entrainment (Zhang et al., 2010; Welsby, 2014; Zangooi et al., 2017). These effects of some frothers have already been well reported in the literature (Cho et al., 2002; Grau and Laskowski, 2006; Nasset et al., 2007; Maldonado et al., 2008; Zhang et al., 2010; Gomez et al., 2011; Kracht et al., 2013; Welsby 2014; Vazirizadeh et al., 2016; Zangooi et al., 2017; Mohagheghian and Elbing, 2018; Güven et al., 2020; Karakashev et al., 2020). The coalescence of the bubbles is important in controlling the interfacial area in equipment that comes into contact with the gaseous liquid and in the formation of bubbles in micro-flotation devices. Bubble size distribution (*BSD*) by measuring foam properties (gas retention, bubble numbers, and bubble size) is important in flotation practice. To assess

this effect on an industrial scale, the bubble surface area flux is typically estimated from the gas rate and *BSD* measurements, where complete *BSD* is compressed to an overall value, namely the Sauter mean diameter (d_{32}) (Güven et al., 2005; Vazirizadeh et al., 2016; Karakashev et al., 2020). As it is well known from the literature, the Sauter mean bubble diameter (d_{32}) and the mean number diameter of the sample are calculated along with other associated parameters (Grau and Laskowski, 2006; Kracht et al., 2013; Kowalczyk, 2016; Mohagheghian ve Elbing, 2018).

On the other hand, other factors such as air velocity, collector (and other reagents) type and concentration, pulp ionic strength, and used machine also control the *CCC*, bubble size, and *BSD* (Wills and Finch, 2016). The results of bubble–bubble coalescence can depend on the presence of frothers and surfactants in the solution as well as on the bubble size. The tendency for bubbles to coalesce influences the size distribution in bubble suspensions. For example, Narsimhan and Ruckenstein (1986) observed a decrease in the mean bubble size in a bubble column as the concentration of surfactants was increased.

To our knowledge, significant progress has not been made over the past few years on characterizing and understanding the hydrodynamic properties of frothers and collectors and their effect on bubble size and *CCC* performance. This new understanding of the relationship between chemistry and hydrodynamics has dramatic implications for how flotation circuits should be controlled and optimized. Therefore, this study aimed to determine the differences between single frothers and frother + *DAH* by adding the concentration of collectors in the presence of different commercial frothers (*PPG200*, *PPG400*, and *PPG600*) in terms of critical coalescence concentration (*CCC*). Additionally, to better understand the surface activity of the frothers, bubbling properties and bubble size distribution were evaluated for each frother and collector in the air-water phase. Therefore, the effects of only frothers, only collectors, and their mixtures on *CCC* values in the absence of particles can be shown in the light of these findings.

In this context, the critical coalescence concentration and bubble size distribution for the different commercial frothers of *PPG200*, *PPG400*, and *PPG600* in the absence of the presence of *DAH* collector without particles were determined by measuring the variation of solution turbidity as a function of light intensity.

2. Materials and methods

2.1. Materials

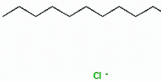
In this study, all the frothers (polypropylene glycols (*PPG200*, *PPG400*, and *PPG600*) were provided by BASF Company (Germany). Dodecyl amine hydrochloride (*DAH*) with 97% purity obtained from Sigma Aldrich was used as a collector. The chemical properties of the frothers and collector are shown in Tables 1 and 2, respectively.

All measurements were carried out at a constant room temperature of $23\pm 1^\circ\text{C}$. Before each test, the glassware was rinsed in ethanol (99% purity, MERCK) and washed with distilled water, followed by steam cleaning, and drying in a clean oven. Each frother solution was mixed at 500 rpm for 5 min to allow the frother to solve in the aqueous phase.

Table 1. Chemical properties of the frothers

| Chemical | Hydroxyl Value (mgKOH/g) | Molecular Weight (g/mol) | Formula | HLB | pH (Solution of 1%) | Molecular Structure |
|----------------------------------|--------------------------|--------------------------|---|-------|---------------------|---------------------|
| <i>PPG200</i> ($n\sim 3.5$) | 510~623 | 180~220 | $\text{OH}(\text{C}_3\text{H}_6\text{O})_n\text{H}$ | 10.55 | 5.0~7.0 | |
| <i>PPG400</i> ($n\sim 6.5$) | 255~312 | 360~440 | $\text{OH}(\text{C}_3\text{H}_6\text{O})_n\text{H}$ | 8.69 | 5.0~7.0 | |
| <i>PPG600</i> ($n\sim 10$) | 170~208 | 540~660 | $\text{OH}(\text{C}_3\text{H}_6\text{O})_n\text{H}$ | 8.25 | 5.0~7.0 | |

Table 2. Chemical properties of the collector

| Chemical | Form | Purity (%) | Molecular Weight (g/mol) | Formula | CMC (mol/dm ³) | Density (g/cm ³) | Molecular Structure |
|----------|-------|------------|--------------------------|-------------------------------------|-----------------------------|------------------------------|---|
| DAH | Solid | ≥ 99% | 221.81 | C ₁₂ H ₂₈ ClN | 1.5×10 ⁻² (25°C) | 1.0 (20°C) |  |

2.2. Methods

2.2.1. Bubble coalescence measurements of frothers and collectors mix system

The captured images during bubble size measurements and the schematic representation of the unit are shown in Figs. 1a and 1b, respectively. Two different parts of the test device, mechanical and optoelectronic, can be distinguished. A mechanical part is used to produce bubbles at a known volume and speed. The optoelectronic part consists of a light source and electronic equipment for signal conversion, data acquisition, and data processing. In this setup, a 30×220 mm micro flotation cell with a volume of 155 cm³ was used. By using a light source in the measurements, the light is sent to the bubbling solutions prepared at various concentrations. Some of the light passing through the cell is absorbed by the solution, while the light intensity of the part leaving the cell (not adsorbed) is measured with an adapter (Thorlabs, USA). The procedure for the definition of CCC was extensively explained in a previous paper by Guven et al. (2020). Briefly, the critical coalescence concentration for the frothers was determined at 50% values of the bubble coalescence (Nguyen et al., 2012). All the experiments were repeated 3 times, and the average error for the measurements was about ±2 mW.

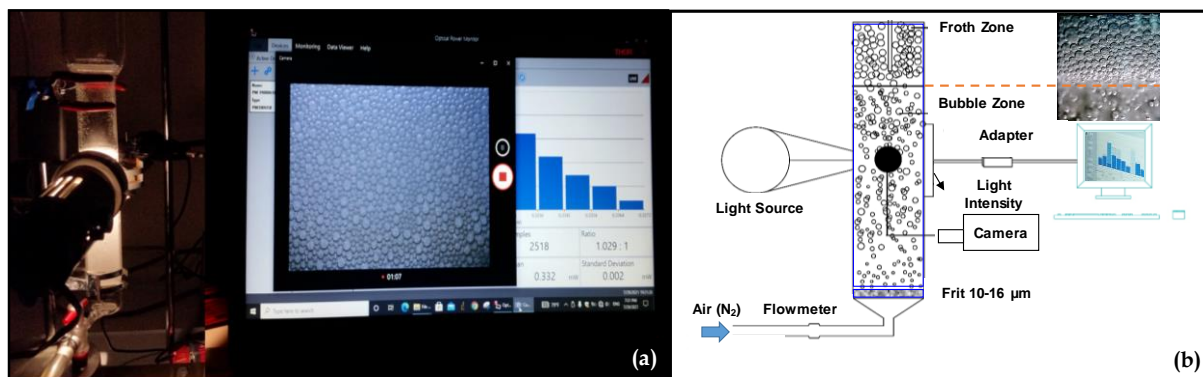


Fig. 1. (a) Experimental setup for bubble coalescence measurements (b) Schematic representation of bubble coalescence measurements

In these experiments, the effect of the collectors on the bubble coalescence of the frother was investigated. As given in Fig. 3 that the CCC values of PPG600, PPG400, and PPG200 were determined as 3, 4, and 11 ppm, respectively. The idea was to obtain the effect of collectors on the bubble coalescence for the selected frothers, therefore it was decided to choose the 50% of the CCC concentrations of the frothers; 1, 2, and 6 ppm for PPG600, PPG400, and PPG200, respectively as presented in Table 3. And, all experiments were carried out at these concentrations. The solutions in all experiments were mixed for 10 min at constant room temperature (23±1°C), 500 rpm mixing speed, and prepared as 100 cm³.

Bubble incorporation measurements using the collector and frothers were performed with the pre-prepared solutions using the frit pore size of the micro-flotation cell with 10-16 μm and the setup shown in Fig. 1a. This was achieved by measuring the light intensity values every 60 sec following the addition of N₂ gas to the system with a 50 cm³/min air rate.

2.2.2. Sauter mean diameter (SMD) measurements

These measurements were also made with the system shown in Fig. 1. The procedure for these measurements can be defined as follows; after filling the column cell with aqueous solutions of tested frothers and a mixture of frothers and collectors, N₂ gas was sparged through the system at a 50 cm³/

Table 3. The concentration of DAH collectors and frothers

| PPG200 | | DAH | | PPG400 | | DAH | | PPG600 | | DAH | |
|--------|------------------------|-----|------------------------|--------|------------------------|-----|------------------------|--------|------------------------|-----|------------------------|
| ppm | (mol/dm ³) | ppm | (mol/dm ³) | ppm | (mol/dm ³) | ppm | (mol/dm ³) | ppm | (mol/dm ³) | ppm | (mol/dm ³) |
| 6 | 3×10 ⁻⁵ | 1 | 4.51×10 ⁻⁶ | 2 | 5×10 ⁻⁶ | 1 | 4.51×10 ⁻⁶ | 1 | 1.66×10 ⁻⁶ | 1 | 4.51×10 ⁻⁶ |
| 6 | | 3 | 1.35×10 ⁻⁵ | 2 | | 3 | 1.35×10 ⁻⁵ | 1 | | 3 | 1.35×10 ⁻⁵ |
| 6 | | 5 | 2.25×10 ⁻⁵ | 2 | | 5 | 2.25×10 ⁻⁵ | 1 | | 5 | 2.25×10 ⁻⁵ |
| 6 | | 7 | 3.16×10 ⁻⁵ | 2 | | 7 | 3.16×10 ⁻⁵ | 1 | | 7 | 3.16×10 ⁻⁵ |
| 6 | | 10 | 4.51×10 ⁻⁵ | 2 | | 10 | 4.51×10 ⁻⁵ | 1 | | 10 | 4.51×10 ⁻⁵ |
| 6 | | 100 | 4.51×10 ⁻⁴ | 2 | | 100 | 4.51×10 ⁻⁴ | 1 | | 100 | 4.51×10 ⁻⁴ |

min rate. After 60 sec of conditioning, the representative images and video recordings of bubbles were captured by a digital camera (Bushman MOS 8 Led Microscope, Bushman Equipment Inc., Menomonee Falls, WI, USA) with a 50X magnification rate while the light was sent through this column from a constant location of the cell (around 10 cm above the plane of the frit). This location was selected based on the volume of the solution and the sizes of the cell as well. Based on previous measurements of CCC values of frothers, at least 20 images were captured for each concentration of frothers as 1, 2, and 6 ppm for PPG600, PPG400, and PPG200 respectively in different collector concentrations (1, 3, 5, 7, 10, and 100 ppm). Thus, the bubble diameters in terms of Sauter Mean Diameter (d_{32}) (SMD) were calculated using free *ImageJ* software (Version 1.8.0) by using Eqs. 1 and 2 with an accuracy of 0.05 mm.

$$\frac{V_b}{A_b} = \frac{\frac{4}{3}\pi \cdot (d_v/2)^3}{4\pi \cdot (d_s/2)^2} = \frac{(d_v/2)^3}{3(d_s/2)^2} = \frac{d_{32}}{6} \quad (1)$$

$$d_{32} = 6 \frac{V_p}{A_p} \quad (2)$$

where d_s and d_v are the surface diameter and the surface volume of the bubbles, respectively, and V_b and A_b are the volume and area of the bubbles, respectively.

3. Results and discussion

3.1. Effects of frothers and collector on critical coalescence concentration

First, the effect of three different frothers on bubble size under varying concentration conditions was investigated. During these measurements, 50 cm³/min N₂ gas was sparged through the pure water, and then the light intensity values were measured as a function of time, to determine the optimum time value for the light intensity. As seen in Fig. 2, while it was not stable up to 60 sec, it became almost stable after this time. For this reason, the optimum time was determined as 60 sec.

Under these conditions, the coalescence rates of the bubbles as a function of concentration were found under a constant gas flow rate, and the results are shown in Fig. 3. As can be seen from the results, the bubble incorporation rate was at lower concentrations in the presence of PPG600, while in the presence of PPG200, this occurred at higher concentrations.

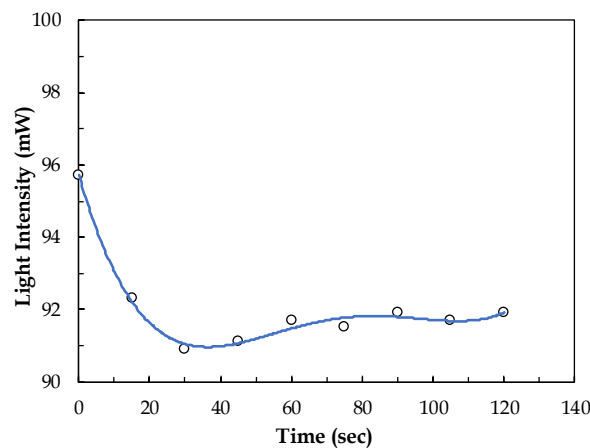


Fig. 2. Light intensity values of distilled water at 50 cm³/min air flow rate according to time

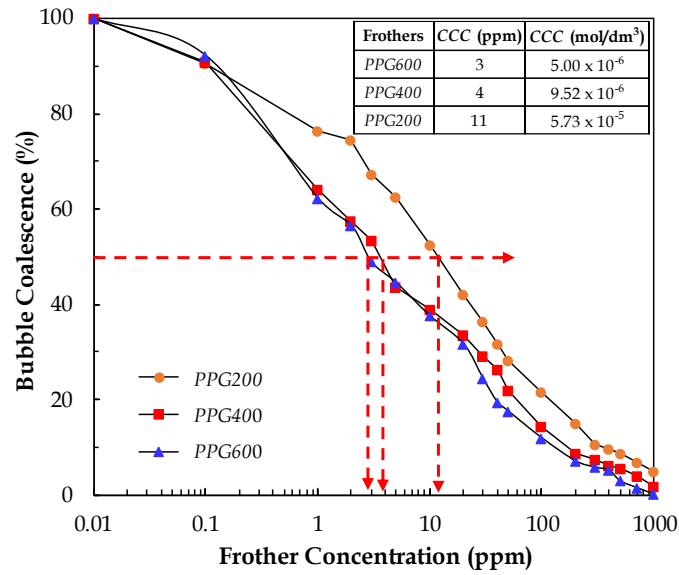


Fig. 3. Variation of % bubble coalescence against the concentration of different frothers

It could be concluded from the results as shown in Fig. 3 that generally *PPG600* and *PPG400* achieve CCC values at low concentrations when comparing frothers of the poly-glycol family with each other. It was stated that it could be related both to the increase in the number of poly-glycol and to the molecular weight. The results of the bubble association measurement in the presence of frothers depending on the *DAH* concentration are shown in Fig. 4. As seen in Fig. 4a, the CCC of *PPG200* is 11 ppm without *DAH*, while the CCC of *PPG200* is 6 ppm in the presence of *DAH* (6 ppm *PPG200* + 5.3 ppm *DAH*). The CCC of other frothers was decreased in the presence of *DAH*. The results are presented in Table 5.

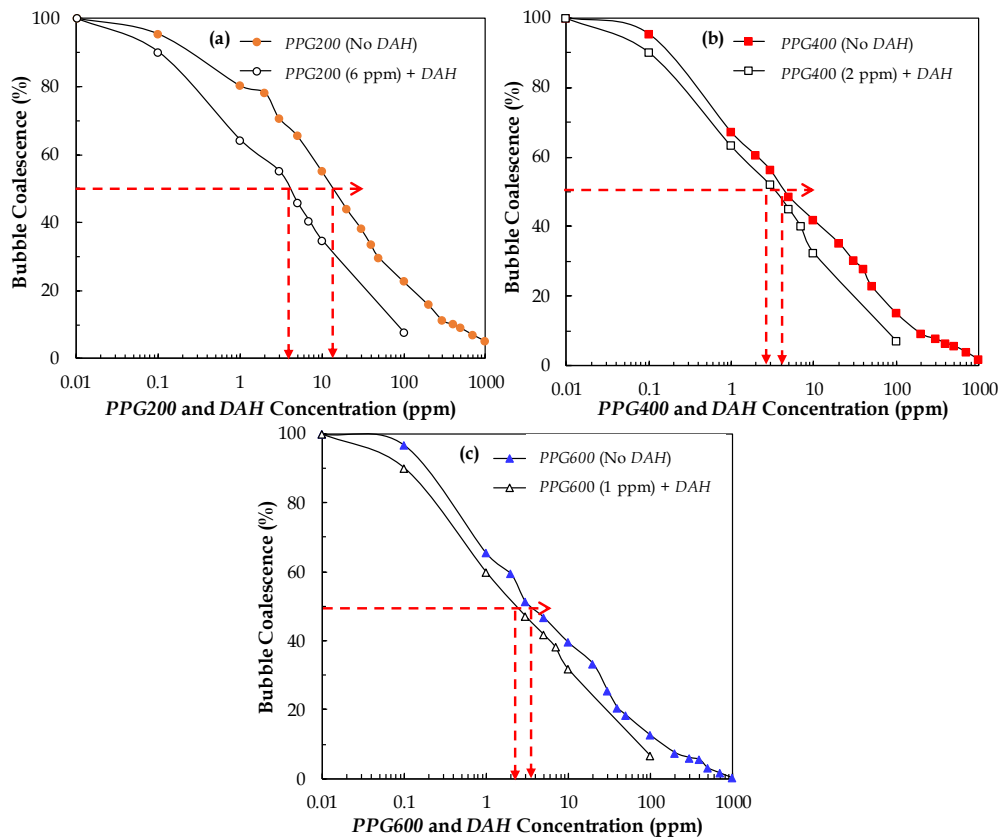


Fig. 4. Bubble coalescence results for frothers as a function of *DAH* concentration (a) *PPG200*, (b) *PPG400*, (c) *PPG600*

Appropriate interaction of collector and frother was observed between *DAH* and Polypropylene glycols. This can be ascribed to the chemical structures of these reagents. In other words, both *DAH* and *PPG* have an oxygen atom in their structure; therefore, they can form a hydrogen bond, which ensures a good steady-state. Thus, the *DAH* collector also presents frother characteristics that in a mixed system of *DAH* and other types of frothers, it will be possible to reach the CCC point in a lower concentration. In addition, as mentioned in a previous study, the usage of a commercial anionic collector (BD-15, Da-Cemex Company) consisting of mainly oleic acid and frother in the same system would not only change the surface tension values but also vary the zeta potential of bubbles which in turn affects the critical coalescence concentration of bubbles (Karaguzel et al., 2010). Accordingly, as shown in Fig. 4, CCC values of *DAH* collector (*PPG200*, *PPG400*, and *PPG600*) mixed with frothers were determined as 5.3 ppm for *PPG200*, 3.2 ppm for *PPG400*, and 2.7 ppm for *PPG600* in air bubble coalescence measurements.

Among the CCC values of *PPG* (200, 400, 600) belonging to the polypropylglycol family, the *PPG200* + *DAH* system showed more activity. According to these results, when the frothers (*PPG200*, *PPG400*, and *PPG600*) are mixed with the *DAH* collector, the CCC values were determined as 11.3 ppm for *PPG200*, 4.29 ppm for *PPG400*, and 2.70 ppm for *PPG600*.

Table 5. CCC values of the frother in the presence of *DAH*

| | CCC (ppm) | | |
|-----------------|--|--|---|
| | <i>PPG200</i> | <i>PPG400</i> | <i>PPG600</i> |
| No <i>DAH</i> | 11 | 4 | 3 |
| With <i>DAH</i> | 5.3* + 6 ppm <i>PPG200</i> | 3.2** + 2 ppm <i>PPG400</i> | 2.7*** + 1 ppm <i>PPG600</i> |
| Total | 11.3 | 4.29 | 2.90 |
| | * 2.4×10^{-5} mol/dm ³ | ** 1.44×10^{-5} mol/dm ³ | *** 1.23×10^{-5} mol/dm ³ |

3.2. Effect of frothers and collector (mix system) on SMD

In the mix test series, the effect of *PPG* (200, 400, and 600) different frothers and collectors on bubble size under varying concentration conditions was investigated. First, 50 cm³/min N₂ gas was sparged through pure water, and then the light intensity values are measured every 60 sec. Under the same flow rate, the effect of frother + *DAH* was also investigated on SMD (d_{32}) by processing bubble images captured on the micro flotation cell from a single viewing point with *ImageJ* software, and the results are shown in Fig. 5 and Table 6. Additionally, Figs. 7 and 8 present the images of these bubbles of these frothers obtained according to the frother concentration and *DAH* + frothers mix system.

As seen in Fig. 5, the SMD values of bubbles of the mixing system of *PPG* (200, 400, and 600) with

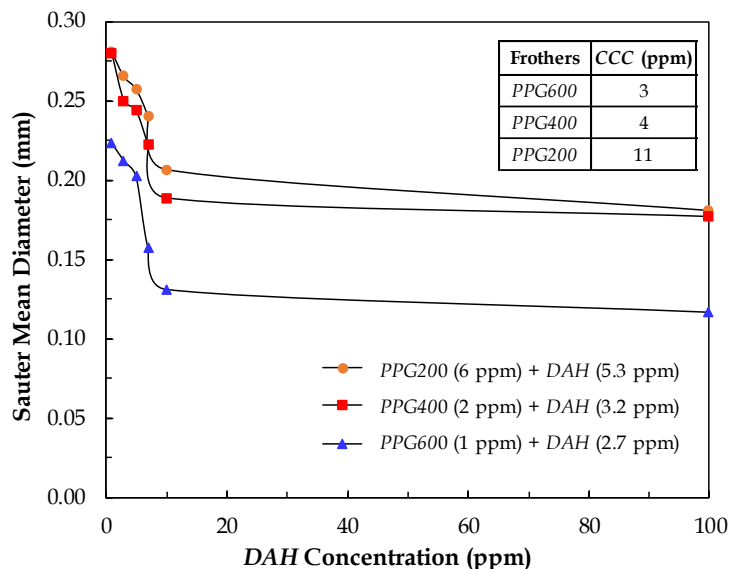
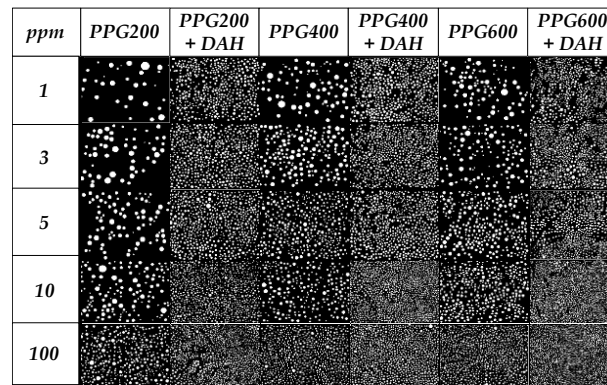
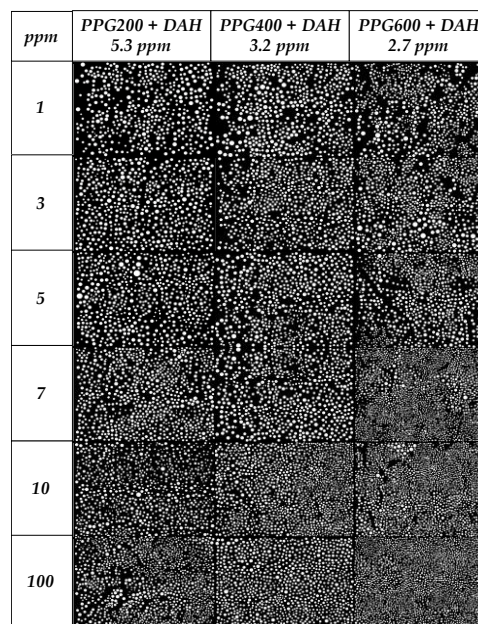


Fig. 5. Effect of frothers + *DAH* on the SMD of bubbles (frit diameter: 10-16 μ m)

Table 6. Effect of frothers and frothers + collector's concentration on SMD of bubbles

| Frother Type | SMD (mm) |
|--------------------|-------------|
| PPG200 10 ppm | 0.408 |
| PPG200 6 ppm + DAH | 0.207 |
| PPG400 10 ppm | 0.365 |
| PPG400 2 ppm + DAH | 0.188 |
| PPG600 10 ppm | 0.309 |
| PPG600 1 ppm + DAH | 0.131 |

the DAH collector at 1, 2, and 6 ppm concentrations were observed to decrease more, then reached a minimum at 10 ppm DAH and no changed up to 100 ppm. At higher concentrations from 1 ppm to 100 ppm, a gradual and significant reduction was found in these frother types, in line with previously reported critical coupling concentration results (Güven et al., 2020). According to the data at 10 ppm here and considering the SMD of each collector + frother mixture, the concentration of 10 ppm can be accepted as a critical point for most mixed systems. These results can be seen in the *ImageJ* pictures in Figs. 6 and 7 which present the images of the bubbles of these frothers using the *ImageJ* program, obtained according to the collector concentration, in addition to SMD as a function of frother + DAH concentration.

Fig. 6. Effect of frother and frother + DAH concentration on SMD of bubbles (Frit Diameter: 10-16 μm)Fig. 7. *ImageJ* picture of the size of the frothers obtained by different frothers + DAH

3.3. Bubble size distribution (BSD)

As mentioned before, bubble size distribution (BSD) is one of the most important factors in flotation and depends on frother type and concentration, air flow rate, and frit pore size (Batjargal et al., 2021). Figure 8 shows the bubble size distribution with respect to frother type at constant concentration of frothers + concentration of collectors (1, 3, 5, 7, 10, and 100 ppm), air flowrate (50 cm³/min), and frit pore size (10–16 μm). As it can be seen from Fig. 8 that it was observed that the bubble sizes of the frothers were reduced more when PPG (200, 400, and 600) at concentrations of 1, 2, and 6 ppm was mixed with the DAH collector.

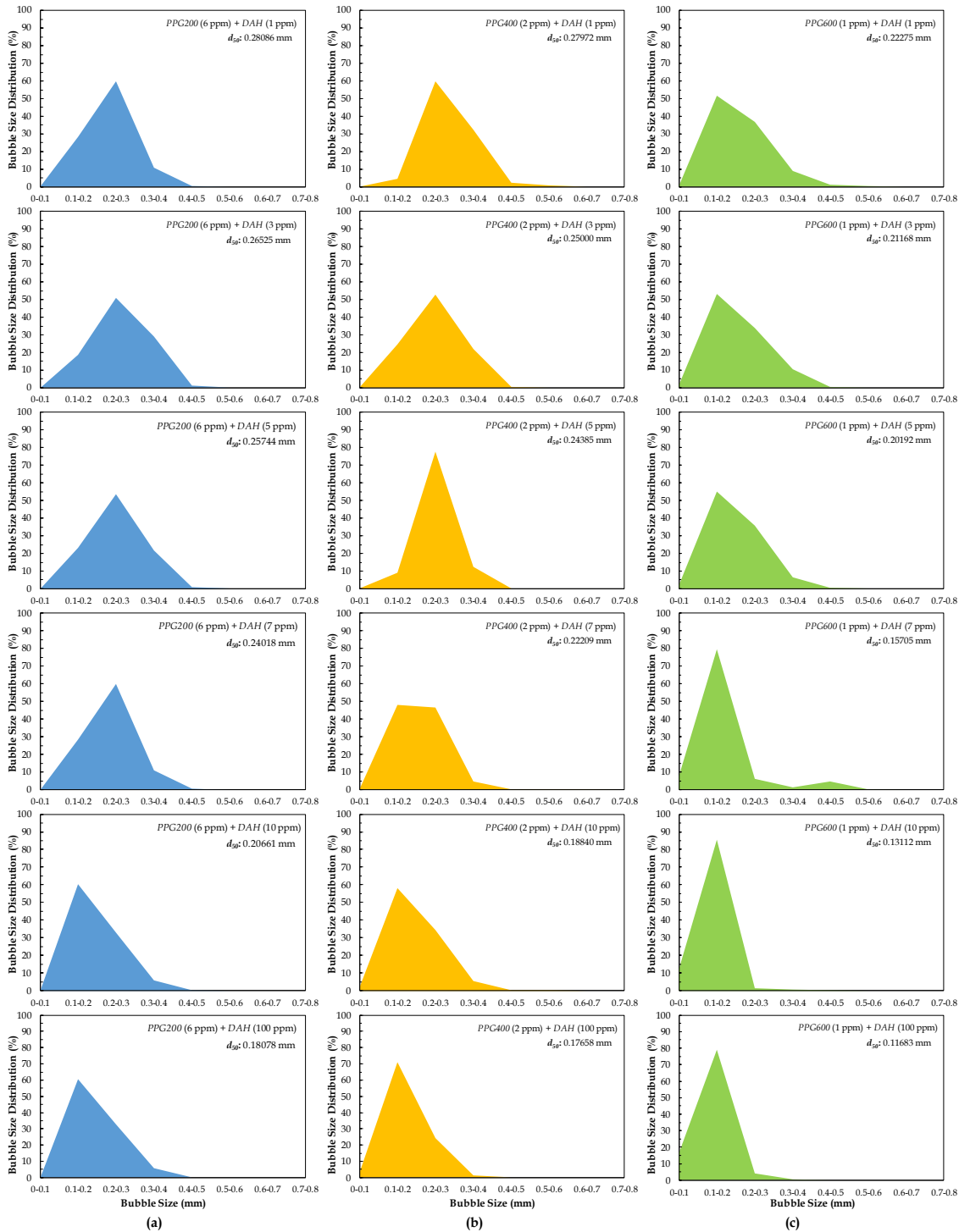


Fig. 8. Effect of frother type + collector type mixing system on BSD at different concentrations of the collector (50 cm³/min air flow rate and frit pore size of 10–16 μm: PPG200 + DAH, PPG400 + DAH, and PPG600 + DAH)

It is worth noting that these results are not unexpected considering the frother feature of *DAH* in addition to its collector role in flotation. Similar results were also reported in Corona-Arroyo et al. (2015) for the dodecylamine (*DA*)-*MIBC* blend. It was reported that a significant decrease was obtained in terms of the *CCC* for *MIBC* in the presence of 4 mg/dm³ *DA* while the lower concentrations of amine were not found effective on bubble size distributions, especially for *MIBC*. Thus, in this series of tests, polyglycol type frothers reported to be more powerful compared to *MIBC* were used (Elmahdy and Finch, 2013). Considering that knowledge in mind, six concentrations of the *DAH* as 1, 3, 5, 7, 10, and 100 ppm were examined to clearly show the contribution of the amine-frother blend in flotation systems. In Fig. 8, depending on the type of frother and collector, the bubbles produced in the presence of frothers + collector mixing systems were revealed at lower dimensions with the frother. For example, frothers + *DAH* (1 ppm) mixing system while the *BSD* in the presence of *PPG600* (1 ppm) accumulated between 0–0.4 mm and the d_{50} of *BSD* was determined at 0.22 mm, *PPG200* (6 ppm) gave the highest *BSD* variation between 0–0.6 mm, and the d_{50} of *BSD* was 0.2808 mm.

Additionally, the maximum *BDS* values for each frother were plotted as a function of *DAH* concentration. As shown in Fig. 9, while *PPG600* gave the highest *BSD* values with the increasing *DAH* concentration, *PPG200* and *PPG400* almost gave the same result as a function of *DAH* concentration.

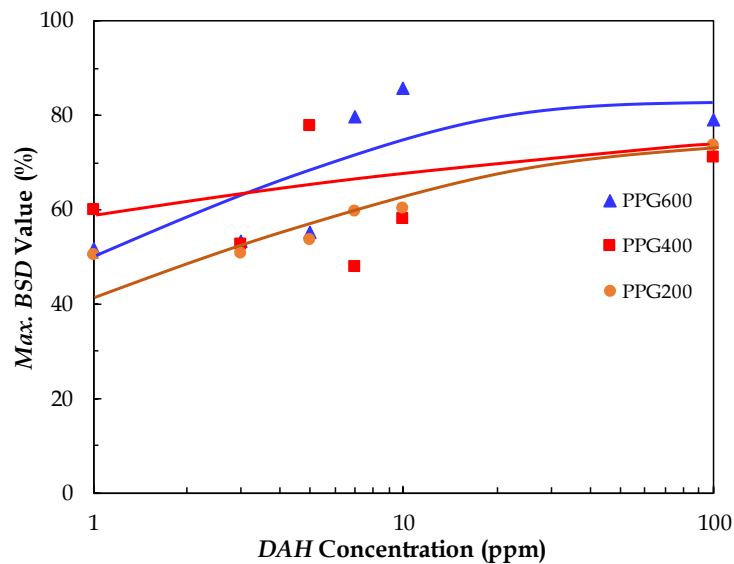


Fig. 9. Max. *BSD* values for each frother as a function of *DAH* concentration

4. Conclusions

The results of this study showed that upon the addition of *DAH* to the solutions of different frothers such as *PPG200*, *PPG400*, and *PPG600*, the *CCC* values of these frothers decreased to 11.3 ppm, 4.29 ppm, and 2.90 ppm in the same order which is quite important for flotation systems. And, accordingly, the highest *SMD* value for *PPG200* 6 ppm + 1 ppm *DAH* was 0.28 mm, while the lowest *SMD* value for *PPG600* was 1 ppm + 100 ppm *DAH* 0.1311. Even in all frothers + *DAH*, 3 ppm was immediately found at a very sufficient critical break concentration point. This can be explained by the fact that *DAH* is a significantly stronger surfactant than these frothers.

Additionally, when 10 ppm concentration was compared in all frothers and frothers + collector mix systems, the *SMD* value for *PPG200* was 0.408 mm, while it decreased to 0.207 mm for *PPG200* 6 ppm + *DAH* mix system. Therefore, it was found that the *SMD* of all frother + collector mix systems decreased by about 2 times. Moreover, when 10 ppm concentrations were compared in all frothers + collector mix systems were more concentrations.

The *BSD* of frothers + collectors mix systems also followed the same order obtained for *SMD* values; the finest distribution gradually exhibited a little larger distribution for *PPG200* + *DAH* mix system. It was shown that *SMD* and *BSD* of all mixing systems were found to be suitable even at the lowest concentration.

In sum, these results showed that tuning the flotation conditions such as the type of frother and collector mix systems, their molecular weight, their molecule's functional groups, and their concentrations would be very effective in adjusting bubble size which in turn affects the flotation recovery.

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