Effect of some operational variables on bubble size in a pilot-scale mechanical flotation machine

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Abstract: This work aims to provide a relationship of how the key operational variables of frother type and impeller speed affect the size of bubble ($D_{32}$). The study was performed using pilot-scale equipment (0.8 m\textsuperscript{3}) that is up to two orders of magnitude larger than equipment used for studies performed to date by others, and incorporated the key process variables of frother type and impeller speed. The results show that each frother family exhibits a unique CCC95-HLB relationship dependent on $n$ (number of C-atoms in alkyl group) and $m$ (number of propylene oxide group). Empirical models were developed to predict CCC95 from HLB associated with other two parameters $\alpha$ and $\beta$. The impeller speed-bubble size tests show that $D_{32}$ is unaffected by increased impeller tip speed across the range of 4.6 to 9.2 m/s (representing the industrial operating range), although $D_{32}$ starts to increase below 4.6 m/s. The finding is valid for both coalescing and non-coalescing conditions. The results suggest that the bubble size and bubble size distribution (BSD) being created do not change with increasing impeller speed in the quiescent zone of the flotation.

Key words: flotation; bubble size; operational variables; critical coalescence concentration; hydrophile- lipophile balance; impeller speed

1 Introduction

Froth flotation is a versatile and extremely complex physico-chemical process that has been widely used in recovering valuable minerals. During the flotation process, bubbles play a fundamental role and it is well documented that the bubble size or bubble size distribution (BSD) have a direct influence on the flotation performance [1–3]. The importance of the bubble size in flotation has been investigated by a number of researchers in the 1960’s and 1970’s. REAY and RATCLIFF [4] showed that collection efficiency of particles by bubbles would strongly depend on the bubble to particle size ratio. They concluded that, within practical constraints, there was a powerful incentive to make bubble size as small as possible. More specifically, ANFRUNS and KITCHENER [5] demonstrated the strong dependence of particle collection efficiency on this ratio, and the need for small, sub-1 mm bubbles in collecting sub-40 \(\mu\)m particles. In a major review of the physical factors affecting flotation, JAMESON [6] concluded that bubble size is an important key which is almost never controlled in plants. They offered the opinion that sub-500 \(\mu\)m bubbles would be required to significantly improve the collection efficiency. Although the importance of the bubble size was established as early as 1970’s, these studies and their outcomes might be inadequate since the effects of many key variables affecting the flotation process have not been fully considered. These key variables which govern the flotation process are classified into two general groups: “ituational” variables (e.g., water quality, gas/air density, pulp viscosity, etc.) and “operational” variables (e.g., frother type and concentration, gas rate, impeller speed, etc.). The operational variables comprise of two aspects: chemical-related and machine-related. In the past, the role of operational variables has received less attention [7–8] than situational variables, even though they are a vital part of the flotation process. The review of literature has highlighted that there currently exists no generally accepted relationships and models between bubble size and operational variables, nor is the extent to which these relationships can be predicted from phenomenological or more fundamental understandings of the bubble formation processes. This research attempts to address
this shortcoming, which has eluded researchers up to now.

Frothers are heteropolar surfactants, comprising polar and non-polar groups, which are used to promote flotation performance through control of hydrodynamic and froth properties [9–14]. The most commonly used frothers fall in two main categories: Aliphatic Alcohols (C_{n}H_{2n+1}OH) and Polyglycols (C_{n}H_{2n+1}(OC_{3}H_{6})_{m}OH) [9–10]. The frother functions in some manner must depend on the frother structure [9], e.g., the relative strength of the hydrophobic to hydrophilic groups. In each frother family, the relative strength can be modified by changing the number of —CH_{2}— groups (represented by n) in the Alkyl chain in the case of Alcohols and Polyglycols, and/or number of Propylene Oxide (PO, represented by m) groups in the case of Polyglycols. Varying the relative strength of the hydrophobic to hydrophilic groups in the molecule changes its hydrophilic-lipophobe balance, or HLB. The common calculation of HLB is the Davies method where group numbers are assigned and summed [15]. There have been some attempts to link frother functions to HLB [9, 16–19].

Based on other researchers, GORAIN et al [20] recognized that frother is the single most important variable affecting bubble size. GORAIN’s work has been seminal in that it was the first to clearly link chemical variables such as frother type as an important variable to bubble size, or more clearly, flotation recovery. As frother concentration increases, the bubble size distribution (BSD) narrows and becomes finer. The Sauter mean size vs. frother concentration (D_{32}−C) relationship shows an exponential decay to a minimum (limiting) size (D_{1}) at a concentration characteristic of the frother. This concentration is referred to as the critical coalescence concentration (CCC) [16]. Noting the difficulty in defining the end point of an exponential function, NESSET et al [18–19] refined the estimation by fitting a three-parameter model to the D_{32}−C and determining the CCC95, i.e., the concentration giving 95% size reduction in D_{32} relative to water alone (D_{0}). The CCC95 was selected as the bubble size function parameter. In this work, the frother type-bubble size tests were conducted on 36 frothers in three different families and the ambition is to provide fundamental input to the selection of frother for an optimum bubble size, such as establishing a correlation between CCC and HLB, which remains a largely empirical exercise.

Typically, the larger flotation machines have a power input of 1−2 kW/m^{3} of cell volume, with 1 kW/m^{3} being the more typical for Outokumpu, Bateman, Dorr Oliver and Metso designs. Recent reports from the South African platinum industry suggest that metallurgical recovery of small, sub-10 μm PGM particles can be improved with increased power input in scavenging applications [21]. This is achieved by increasing the impeller rotational speed: values of 5−10 kW/m^{3} have been reported. Apparently effective, it is a costly way to improve the recovery of fine particles. Plant operators often consider the impeller rotational speed of the flotation machine as an adjustment that can be used to change the pumping rate of slurry, impact particle suspension and bubble particle contact, or more importantly, change bubble size. Recent laboratory investigations by GRAU and HEISKANEN [1] on 50 and 70 L laboratory flotation machines in air-water, determined that bubble size was dependent on impeller tip speed to the power, −0.41 and −0.55 for the 50 L unit, for an impeller tip speed range of 2 to 5 m/s. However, similar test on the 70 L unit showed no significant relationship, which possibly reason for the lack of a similar dependency. It is important note that the range of impeller tip speeds in these tests is largely below those typically used in industrial units, 5 to 7 m/s [21]. The literatures reveal that the relationship between impeller speed (or power intensity) and bubble size is incomplete and has not gained acceptance as evidenced by the observation. It is therefore deemed as an important variable in this work.

Based on the above discussion, knowledge of how key operational variables affect the bubble size is seen as an essential component of process understanding and optimization. This work attempts to provide new insights into the effects of chemical (frother type) and machine (impeller speed) related operational variables on bubble size in a mechanically agitated flotation cell. The proposed testing will make use of a pilot-sized flotation machine considerably larger than a standard lab unit, approximately 0.8 to 1 m^{3} in volume, to limit dimensional effects associated with smaller laboratory cells. The ability to have a distinct separation zone above the mixing zone around the impeller is important for sampling the bubble size and gas rate exiting the cell cross-sectional area. Uniformity of bubble distribution within the larger cell is also expected to be better. Locating the McGill Bubble Size Analyzer (MBSA) [2−3, 22] will also be easier in the larger cell. Testing will initially be conducted in the two-phase water-air system and will be followed by tests using slurries in the next stage.

2 Experimental

2.1 Apparatus and methods

The same rig as previously described in Ref. [22] was used. It consists of a mechanical flotation cell (0.8 m^{3} in volume) with a McGill Bubble Size Analyzer (MBSA) (Fig. 1). The flotation cell will be fitted with a
gas-flow meter and valve on the air intake, a transmitting torque-meter (strain gauge) for measuring power input on the rotor drive shaft, automatic level control, variable speed motor, changeable rotors, and an ability to operate in either self-aerated or forced air mode. The ability to alter chemistry (frother type/concentration) and machine (impeller speed) variables in a test program that accurately establishes the effect on bubble size distribution is primary. Bubble size distributions will be measured using McGill’s bubble viewer technology [22]. Gas rate will be measured by both the installed flow meter and a \( J_g \) device adapted for the cell.

The unit was operated with 700 L water at room temperature (22–24 °C) with air dispersed through the 21 cm diameter at a superficial air rate \( J_g \) (volumetric air rate divided by cell cross-sectional area) of 0.5 cm/s (both of 0.5 and 1.0 cm/s air rates were applied in impeller speed-bubble size tests). Bubbles were sampled 20 cm below the water surface, well above the turbulence of the impeller-stator region. The images were processed using in-house software to give \( D_{32} \). Further details are given in Ref. [19].

2.2 Reagents

The surfactants from the three frother families are identified in Table 1 which shows the range in \( n \) and \( m \) and corresponding range in HLB (note, Polypropylene Glycol Ethyl Ethers (i.e., \( n=2 \)) and Monopropylene Glycol (\( m=1 \)) are not available). All were reagent grade from Aldrich-Sigma (98%–99.9% purity). Several commercial frothers were included and are listed in Table 2.

3 Results and discussion

3.1 Reliability

Figure 2 shows Sauter mean bubble size (\( D_{32} \)) as a function of concentration (\( C \)) for three repeats for the commercial frother DowFroth 250 (DF250). Replicate tests (i.e., starting from solution preparation) were conducted by two different operators (inter-operator)
at three different times (intra-operator). The $D_{32}$–$C$ curves were consistent and the 95% confidence interval on the calculated CCC95 was $0.6 \times 10^{-6}$ or 0.0024 mmol/L, which is too small to indicate on subsequent plots. The results (Fig. 2) show high precision.

3.2 Effect of frother type and concentration (CCC95 vs. HLB)

In many flotation systems, frothers have the key function of controlling bubble size. Consequently, understanding and predicting their action are of interest to modellers and plant operators alike. The approach here was to explore a structure-function relationship. To quantify structure, HLB was used as it encompasses the hydrophilic-hydrophobic (amphipathic) character that controls adsorption at the air-water interface, which arguably is the basis for frother action. The function, bubble size reduction, was quantified through the CCC concept derived from the plot of Sauter mean diameter ($D_{32}$) versus concentration ($C$). $D_{32}$ was calculated from bubble size distribution using a sampling-for-imaging technique and was validated in Fig. 2. The trend in Fig. 2 was seen for all frothers, illustrated in Fig. 3 for three surfactants. The estimation of CCC95 from the three-parameter model fit to the $D_{32}$ vs. $C$ data proved the reliability based on replicated tests (Fig. 2).

Efforts along this structure-function approach by LASKOWSKI [16] and NESSET et al [18–19] laid a foundation. The pursuit revealed a family-based CCC-HLB pattern, confirming the possibility entertained by PUGH [9]. Figure 4(a) shows the CCC95-HLB relationship for the Aliphatic Alcohols. Starting with Propanol there is a sharp decrease in CCC95 as HLB decreases (i.e., $n$ increases), which levels off above 6 carbons ($n=6$ or C-6). Previous studies on inhibition of bubble coalescence by Alcohols reveal a similar trend. The concentration required decreases with increasing $n$ to approach a limiting value for $n>6$ [23−24]. For $n=6$, there is an increasing isomer effect, i.e., effect of position of the OH group, which is illustrated by comparing Hexanol and Pentanol in Fig. 4(b). For practical purposes, however, since such short chain Alcohols are not employed as frothers, the isomer effect is later ignored. The commercial frother FX120-01 is seen to fit the trend (Fig. 4(a)).

Figure 5 shows the relationship of CCC95 vs. HLB for the two Polyglycol families, in this case, as a function

![Fig. 2 Reliability: inter-operator and intra-operator replicated experiments at same conditions for DF250](image)

![Fig. 3 Effect of frother addition on $D_{32}$ for 3 frother types (CCC95 is noted by the vertical dashed line)](image)

![Fig. 4 CCC95 versus HLB for aliphatic alcohols and their isomers (1-alcohol, 2-alcohol and 3-alcohol) (a) and effect of $-OH$ group position on CCC95 for pentanol and hexanol isomers (b)](image)
of $m$ for a given $n$. There is a pattern: CCC95 decreases with increasing $m$ in a series of parallel or self-similar plots which trend to lower HLB with increasing $n$. Although the PPGAEs and PPGs are usually considered as separate families, the pattern suggests that they can be treated as one. The commercial frothers are shown to fit the pattern.

The trends in Figs. 4(a) and 5 show consistent patterns that can be fitted to the following exponential equation:

$$
\text{CCC95} = \alpha \cdot \exp(\beta \cdot \text{HLB})
$$

(1)

where $\alpha$ and $\beta$ are constants that depend on the family (i.e., $n$ and $m$). Table 2 gives the values for the polyglycols and 1-alcohols.

For polyglycols, $\alpha$ and $\beta$ can be linked to $n$ as

$$
\alpha = 4.74 \times 10^{-17} \cdot \exp(-3.497 \cdot n) + 1.956 \times 10^{-19} \cdot \exp(-0.001452 \cdot n)
$$

(2)

$$
\beta = \frac{6.985 \cdot n + 4.814}{1.455 + n}
$$

(3)

The large database permitted development of empirical models, which well describe the results in Eq. (1) and Table 2 for polyglycols and 1-alcohols. It is also possible to deduce CCC95 knowing $n$ and $m$, from HLB via Eqs. (2) and (3). This approach represents a significant step towards a structure-based prediction of the impact of frother on bubble size in flotation machines which was illustrated for the commercial frothers.

At present, the prediction relates directly to mechanical flotation cells but the trends are likely applicable to all machines. Future work may see a relationship between CCC and machine type enabling the present results to be generalized.

### 3.3 Effect of impeller speed (power intensity)

Of particular interest to the industry is the extent to which bubble size can be manipulated by either chemical means (e.g., frother change) or machine means (e.g., higher power intensity via rotor design or a change in impeller rotational speed). Therefore, one goal of this work is to investigate the effectiveness of machine means such as increasing impeller speed in order to reduce the bubble size.

There have been two opportunities to date to examine the effect of increased power intensity on bubble size. Figure 6 shows the $J_g$-$D_{32}$ comparison between the Lac des Iles 130 m$^3$ Outokumpu tank cells having 1 kW/m$^3$, and 75 L Denver pilot cells having approximately 3 kW/m$^3$ of input power. The chemistry of both systems was identical so the majority of the difference is likely due to the difference in power intensity. An opportunity also presents itself at the South African Impala plant where new Metso 30 m$^3$ RCS tank cells have just been installed on a tailings re-treatment duty using oversized (70 m$^3$) mechanisms. A good comparison on similar feed with upstream Bateman 50 m$^3$ tank cells and no additional frother added is therefore possible during downtime of half the plant. Figure 7 shows the comparison of bubble size versus gas rate for the Metso unit at 3 kW/m$^3$ versus the Bateman cell at 1 kW/m$^3$. In both Figs. 6 and 7, there is a clear reduction in bubble size, seemingly due to the higher power intensity (impeller speed). Some assumptions

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**Table 2 Constants in Eq. (1) for range of $n$ and $m$ and goodness-of-fit (precision) statistics**

<table>
<thead>
<tr>
<th>Family</th>
<th>$n$</th>
<th>$m$</th>
<th>$\alpha$</th>
<th>$\beta$</th>
<th>Precision</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Data points, $N$</td>
</tr>
<tr>
<td>1-alcohol</td>
<td>3−8</td>
<td>0</td>
<td>$1.52 \times 10^{-10}$</td>
<td>3.207</td>
<td>6</td>
</tr>
<tr>
<td>Polypropylene glycol</td>
<td>0</td>
<td>2−17</td>
<td>$4.76 \times 10^{-17}$</td>
<td>3.951</td>
<td>0.9815</td>
</tr>
<tr>
<td>Polypropylene glycol methyl ether</td>
<td>1</td>
<td>1−7</td>
<td>$1.61 \times 10^{-18}$</td>
<td>4.855</td>
<td>0.9615</td>
</tr>
<tr>
<td>Polypropylene glycol propyl ether</td>
<td>3</td>
<td>1−3</td>
<td>$3.15 \times 10^{-19}$</td>
<td>5.624</td>
<td>0.9745</td>
</tr>
<tr>
<td>Polypropylene glycol butyl ether</td>
<td>4</td>
<td>1−3</td>
<td>$9.58 \times 10^{-20}$</td>
<td>6.125</td>
<td>0.9937</td>
</tr>
</tbody>
</table>

**Precision**

$R^2$, $R^2_{\text{Adjusted}}$, SSE, RMSE
have been made in these comparisons [17], and clearly, the conclusions would be strengthened by a more controlled set of laboratory experiments measuring power intensity at the shaft for various combinations of impeller speed and size.

However, the above assumption cannot be conformed by the impeller speed-bubble size tests in pilot-scale Metso flotation cell. On the Metso unit, impeller rotational speed is controlled via the frequency setting (Hz) of the motor’s silicon controlled rectifier (SCR). The specific power is determined by dividing net power by 0.7 m$^3$ of water in the cell. To calculate the impeller tip speed from the motor speed, one needs the ratio of motor sheave (0.125 m in diameter) to mechanism sheave (0.356 m in diameter) and the impeller diameter (0.21), as shown in Eq. (4). The range of the SCR frequency on the unit is 30 to 80 Hz, providing a range of impeller rotation speed from 313 to 834, equivalent to tip speed variation from 3.44 to 9.17 m/s. This range extends well-beyond the typical industry range of 5 to 7 m/s [21]. The testing was done at 0, 2.5, 5 and 10×10$^{-3}$ DF250 at $J_g$ levels of 0.5 and 1 cm/s. These concentrations represent CCCx levels of 0, 52%, 77% and 95%. The range was selected so as to maintain reasonable sensitivity to bubble size change (i.e., avoiding very high levels of frother addition). The purpose of this test was to explore the effect of impeller speed on bubble size by using different concentrations DF250 frothers.

$$v_{IT} = \frac{v_M}{60} \left( \frac{d_{MS}}{D_{MS}} \right) \cdot \pi \cdot d_l$$

where $v_{IT}$ is the impeller speed, $v_M$ is the motor speed, $d_{MS}$ is the motor sheave diameter, $D_{MS}$ is the mechanism sheave diameter, $d_l$ is the impeller diameter.

The curves which are shown in Fig. 8 are $D_{32}$ versus impeller tip speed (i.e., power intensity) for $J_g=0.5$ and 1 cm/s and different frother concentrations. First, as stated in Ref. [8], the bubble size decrease markedly followed by increasing of the frother concentration or decreasing of superficial gas velocity ($J_g$). Second, it is evident that the conditions in the impeller region are highly turbulent, and bubble size and BSD are unaffected by significant changes in impeller speed (and resulting power intensity) within the full operating region of 4.6 to 9.2 m/s. Below this, as the cell approaches its lower operating limit, bubble size begins to increase. However, for practical considerations, the bubble size remains effectively constant with increasing impeller tip speed. This suggests that the system is dominated by coalescence issues and that there is no “breakage” of bubbles occurring. It also means that in plants where speeding up mechanisms (South Africa-Pt fines recovery) has proved beneficial, the effects must be due to particle-collection efficiency related to increase pumping rates and kinetic energy and not bubble size or increased bubble surface area flux ($S_b$).

The results may seem at odds with the researches by GRAU and HEISKANEN [1] who have claimed that
increased power intensity through increasing impeller speed results in decreased bubble size. Since the relatively smaller volume cell and no baffles were applied in their experiments, the turbulent and quiescent zones were not distinctly separated and the measurements could include a portion of the bubble population not exiting the pulp zone. In the turbulent region, the bubble population is distinctly different and finer compared to the quiescent region [17−18]. It is because increasing the impeller speed increases the local fluid velocity (i.e., the pumping rate through the impeller) tending to trap a higher proportion of smaller bubbles in the streamlines. This explanation offered helps sort out why GRAU and HEISKANEN [1] found impeller speed-bubble size dependence while we did not. Subsequent communication with the manufacturer, Outotec, found that they now regard that there is no significant effect of impeller speed on $D_{32}$ across the operating range [25]. The emphasis was that the bubble size in the quiescent zone does not change, while it does in the turbulent region.

4 Conclusions

1) In order to explore the relationship between frother type and concentration to bubble size, a structure-function approach to characterize frothers is established using hydrophile-lipophile balance (HLB) to represent chemical structure and critical coalescence concentration (CCC95) to represent the bubble size reduction function. The result is a series of self-similar CCC95-HLB trends dependent on $n$ (number of C-atoms in alkyl group) and $m$ (number of Propylene Oxide group). Commercial frothers of known family are shown to fit the relationships. Empirical models are developed for the polyglycols and 1-alcohols showing that CCC95 could be predicted knowing HLB, i.e., knowing frother structure.

2) Impeller speed over the range from 4.6 to 9.2 m/s, covering the normal operating range, has no effect on $D_{32}$. This conclusion applies across the spectrum of coalescing (0% frother) to non-coalescing (approaching the CCC) conditions. It reveals that increasing the impeller speed in the typical operating range only affects the particle-collection efficiency, not for bubble size. The discrepancy is explained by noting that some studies have been conducted on small-volume cells or with bubble size measured in the highly turbulent region close to the impeller. The Metso RCS™ cell used in this work is of a design, and hence afforded $D_{32}$ measurement, with distinct separation of turbulent and quiescent zones.

References


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