Evaluation of Micro-bubble Flotation for Treating Ultra-fine Particles and Improvement by Applying Ultrasonic Irradiation

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**Background**

**Further Treatment of Mineral Resources**
Lack of high grade ores $\rightarrow$ Low grade and complex ore treatment and/or Reuse of old (rich) tailings $\rightarrow$ (Ultra-) Fine grinding $\rightarrow$ Fine (nano) particles treatment

**Wastewater Treatment**
Solid/liquid separation and/or mutual separation of precipitates to remove or recover specific components

**Management of Hazardous Elements**
Hazardous elements are often concentrated into fine (nano) fractions and necessary to be removed
Flotation probability

Total Flotation Probability: \[ P_{\text{flot}} = P_c * P_a * (1 - P_d) \]

- \( P_c \): Collision of particles and bubbles
- \( P_a \): Adhesion of particles to bubbles
- \( (1 - P_d) \): Non-detachment of particles from bubbles

Assumptions
1. Homogeneity of particles
2. No interaction of particles
3. No interaction of various flotation phenomena.
   * Argument: Flotation follows 1st order kinetics.
   * Weight floated \( dw \) in the time \( dt \) is proportional to the product of residual particle weight and \( dw \).
Necessity of Micro-bubble Flotation

LOW Collision Probability

HIGH Collision Probability

20 times Sectional Area and Higher collision probability

Relation between particle size and flotation probability

ti: Induction time (s)

Particle size (μm)

Flotation Probability (-)

50 μm bubble

1 mm bubbles
Problems in Micro-bubble Flotation

1. Rising velocity is low because of small bubble size, then, flotation capacity is low.

2. Although the effectiveness of micro-bubble flotation to improve collision probability is clear but NOT adhesion and non-detachment probabilities, and little experimental validation.

3. Air-pressure and/or pulp-circulation types of micro-bubble generators are difficult to control bubble size and flow rate.

→ Detailed theoretical and experimental comparison of Micro- and Milli-bubble flotation

→ “Ultrasonic Irradiation” to air bubbles in order to solve the above problems and improve the effectiveness
Detailed comparison of Micro- and Milli-Bubble Flotation
Experimental

Pulp of hematite fines (0.45 wt% PD, 220 mL)

- 0.002 M with KNO₃
- pH adjustment with HNO₃ or KOH
- SDS addition (1.0 × 10⁻⁴ M)
- pH adjustment with HNO₃ or KOH

10 min conditioning

- 33 µL of MIBC
- Air bubble introduction

10 min Flotation

Froth Tailin g

Micro-bubble 52 µm of 50% size Milli-bubble 708 µm of 50% size

Bubble size distribution at pH6, SDS 1.0 × 10⁻⁴ M

Micro-bubble
Milli-bubble
Cumulative undersize (wt%)

Bubble size

52 µm of 50% size
708 µm of 50% size

Hematite fines Synthesized by gel-sol method

pH 8.0 of IEP

ζ-potential of hematite

μm
Flotation results for Hematite fines

Relation between particle size and flotation recovery / flotation rate constant

- It was confirmed that micro-bubble flotation has an advantage to milli-bubble flotation in flotation recovery and the rate constant, especially in finer size ranges.
- It is also demonstrated that flotation recovery and the rate constant becomes lower in finer size ranges with both flotation methods.
Collision & Detachment Probability

Heindel Collision Model, $P_c$

$$P_c = \frac{1}{1 + |G|} \left[ \frac{1}{2(R_p + R_b)^3} \left\{ 2R_p^3 \right. \right.$$ 
$$\left. + 3R_p^2R_b \right\} + \frac{2R_b^{0.72}}{15(R_p + R_b)} \left\{ R_p^3 \right. \right.$$ 
$$\left. + 2R_b^2R_p^2 \right\} + \frac{|G|}{1 + |G|} \right]$$

* Considering particle size and velocity

Drzymala Detachment Model, $P_d$

$$P_d = \frac{1}{1 + F_{at}/F_{de}}$$

Detachment force: $F_{de} = F_w + F_d$

Attachment force: $F_{at} = F_c + F_e$

Apparent gravity of particle

Drag of particle

Vertical comp. interface force

Excess force
Adhesion Probability

Yoon Adhesion Model

\[ P = P_c \times \left[ \frac{P_a}{\bar{r}} \right] \times (1 - P_d) \]

**Induction time**

\[ P_a = \sin^2 \left[ 2 \arctan \exp \left\{ \frac{- \left( 45 + 8 \text{Re}^{0.72} \right) \mu_b t_i}{30 R_b \left( R_b / R_p + 1 \right)} \right\} \right] \]

*Induction time*: must be shorter than *slipping time* for adhesion
Comparison of Micro-bubble Flotation with Milli-bubble Flotation

Property of Micro-bubble flotation compared with Milli-bubble flotation

<table>
<thead>
<tr>
<th>Probability</th>
<th>Comparison</th>
</tr>
</thead>
<tbody>
<tr>
<td>Collision</td>
<td>Much higher</td>
</tr>
<tr>
<td>Adhesion</td>
<td>Lower</td>
</tr>
<tr>
<td>Non-detachment</td>
<td>Fair</td>
</tr>
<tr>
<td><strong>Total flotation</strong></td>
<td><strong>Higher</strong></td>
</tr>
</tbody>
</table>

- $P_c$: Collision probability
- $P_a$: Adhesion probability
- $P_{1-d}$: Non-detachment probability

**Graphs:**
- Collision probability vs. Particle size (μm) for Micro-bubble and Milli-bubble.
- Adhesion probability vs. Particle size (μm) for Micro-bubble and Milli-bubble.
- Non-detachment probability vs. Particle size (μm) for Micro-bubble and Milli-bubble.
Improvement of Micro-bubble Flotation by Applying Ultrasonic Irradiation
Ultrasonic Irradiation to Air Bubbles

**Primary Bjerknes Force:**
Change in bubble volume in the stationary wave

**Secondary Bjerknes Force:**
Interaction of bubbles

Secondary ultrasonic vibration is generated from the bubbles

$$F_{B2} = \frac{2\pi |A|^2 \omega^2 R_{10} R_{20}}{\rho L^2 (\omega_1^2 - \omega^2)(\omega_2^2 - \omega^2)}$$

- $A$: Amplitude of stationary wave
- $\omega$: Frequency, $R_{10}$ & $R_{20}$: Bubble size, $\rho$: Liquid density
- $L$: Distance between bubbles, $\omega_1$ & $\omega_2$: Resonance frequency of bubbles

F > 0: Attractive $\Rightarrow$ Coagulation and incorporation of bubbles
F < 0: Repulsive $\Rightarrow$ Dispersion of bubbles
Experimental of Flotation with Ultrasonic Irradiation

- Compressed air
- Ultrasonic generator
- Flow meter
- Impeller rotator
- Precision regulator
- Impeller
- Lens
- Light source
- Flotation cell
- High-speed Camera system
- Ultrasonic irradiator
- SPG filter
- Compressed air
- Ultrasonic generator
Experimental of Bubbles Observation

Measurement of bubble size distribution by high-speed camera

Conditions:
SDS conc.: $1.0 \times 10^{-3}$, $1.0 \times 10^{-4}$, $1.0 \times 10^{-5}$ M
MIBC: 150 $\mu$L/L
pH: 3.0, 7.0, 11.0
Ionic strength: $4.0 \times 10^{-3}$ M

No. bubbles counted with high speed camera:
200 for single bubbles
50 for coagulated bubbles
- **Coalescence of bubbles** could lead to the higher selectivity of mixed components.
- **Increase in bubble size** could lead to the increase in flotation rate.
Nobel Micro-bubble Flotation System

For Higher capacity & Higher selectivity

Micro-bubble generator

Plastics

Feed

Froth

Tailing

Ultrasonic irradiation

Ultrasonic irradiation

Froth

Tailing
Dramatic decrease of apparent CMC with ultrasonic irradiation
⇒ It might be suggested that the increase in hydrophobic interaction among surfactant molecules can be achieved.
Change in the SDS Concentration of 50% flotation and Bubble-Particle Attractive Force

⇒ Flotation recovery can be achieved with much lower SDS concentration in case of higher ultra-sonic frequency.
Conclusion

1. Flotation recovery, rate constant, and the probability were higher in Micro-bubble flotation than in Milli-bubble flotation.

2. We modified Yoon flotation model and Heindel collision model by considering the bubbles and particles rising spaces and particle size.

3. Adhesion probability of particles to bubbles, which was obtained by combining experimental flotation probability, Heindel model and Drzymala model, were lower in Micro-bubble flotation than in Milli-bubble flotation.

4. Induction time, calculated from the adhesion probability, was increased by decreasing particle size both in Micro- and Milli-bubble flotation, which indicated that the energy to break bubble surface by particle attachment was smaller in the case of finer particles.

5. Bubbles were coalesced and/or coagulated by the secondary Bjerkness force generated by ultrasonic irradiation. Ultrasonic wave whose frequency is closer to the resonance frequency had a higher effect.

6. We demonstrated two kinds of Micro-bubble flotation systems with ultrasonic irradiation in order to increase the capacity and selectivity.

7. It might be suggested that ultrasonic irradiation of higher frequency increased the flotation recovery with much lower collector concentration.
Problem in Milli-bubble Flotation: Low Collision Probability

Collision probability is very low in mm-size bubbles.

\[ P = P_c \times P_a \times (1 - P_d) \]

Difficult in fines flotation
Effect of Ultrasonic Irradiation to Bubbles Behavior

Bubbles coalescence ratio

<table>
<thead>
<tr>
<th>Frequency</th>
<th>Ratio</th>
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<tbody>
<tr>
<td>430kHz</td>
<td>9.63</td>
</tr>
<tr>
<td>38kHz</td>
<td>14.6</td>
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Decrease in Specific Surface Area

<table>
<thead>
<tr>
<th>Irradiation</th>
<th>Area (m⁻¹)</th>
</tr>
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<tbody>
<tr>
<td>Non irradiation</td>
<td>81 m⁻¹</td>
</tr>
<tr>
<td>430kHz irradiation</td>
<td>15 m⁻¹</td>
</tr>
<tr>
<td>38kHz irradiation</td>
<td>3.3 m⁻¹</td>
</tr>
</tbody>
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Effect of ultrasonic frequency to flotation behavior

<table>
<thead>
<tr>
<th>Frequency</th>
<th>Flotation rate</th>
<th>Grade</th>
<th>Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>430kHz</td>
<td>Fair</td>
<td>Fair</td>
<td>Fair</td>
</tr>
<tr>
<td>38kHz</td>
<td>Excellent</td>
<td>Excellent</td>
<td>Low</td>
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