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## A novel technique to investigate the bubble coalescence in the presence of surfactant (MIBC) and electrolytes (NaCl and CaCl<sub>2</sub>)

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**Abstract:** An efficiency of flotation process is strongly dependent upon the collecting ability of air bubbles. On the other hand, the liquid film formed between two fully or partially mobile air/liquid interfaces being in contact has low stability, which leads to fast liquid drainage. Therefore, when they approach to each other, they tend to coalesce. Therefore, bubble coalescence is usually controlled with frothers in flotation process. Meanwhile, it is known that dissolved ions inhibit bubble coalescence. In this study, the bubble coalescence in the presence of MIBC was determined using a novel technique with a modified bubble-particle attachment timer. Additionally, the effect of NaCl and CaCl<sub>2</sub> on bubble behavior was investigated along with surface tension and bubble coalescence time aspects. As a result of study, it is seen that the bubble coalescence time can be successfully determined with a bubble-bubble coalescence timer.

**Keywords:** Bubble coalescence time measurement, salt, MIBC, surface tension

### 1. Introduction

Flotation is a process used for separating valuable minerals from gangue minerals according to the differences in their hydrophobicity. In flotation technique, while hydrophilic particles remain in the suspension, hydrophobic particles are recovered with attaching them to air bubbles. Since flotation rate and recovery is strongly dependent on bubble size (Ahmed and Jameson, 1985; Yoon and Luttrell, 1986; Yoon and Luttrell, 1989; Cho and Laskowski, 2002; Tao, 2005; Bournival et al., 2012; Corona-Arroyo et al., 2015), the bubble behaviors are generally controlled with the use of frothers, which reduce air/liquid interface tension (Bournival et al., 2012).

Bubbles, produced in the suspension, in the absence of frothers are extremely hydrophobic, and therefore the repulsion forces between them are very weak. When two bubbles approach to each other, a thin liquid film is formed between them, and the thickness of the liquid film decreases due to the drainage of the liquid, and finally the film ruptures, resulting bubble coalescence. Meanwhile, wave motions occur during the bubble coalescence, and this causes the detachment of hydrophobic particles from bubble surfaces (Leja, 1982; Craig, 2004; Nguyen and Schulze, 2004; Ata and Jameson, 2005; Ata, 2008; Ata, 2009; Horn et al., 2011; Bournival et al., 2012; Castro et al., 2013; Bournival et al., 2014; Gungoren et al., 2015; Orvalho et al., 2015).

An alternative method to the use of frothers is employing inorganic electrolyte solutions (Foulk and Miller, 1931; Craig et al., 1993; Christenson and Yaminsky, 1995; Weissenborn and Pugh, 1995; Angarska et al., 1998; Ribeiro and Mewes, 2007; Ozdemir et al., 2009; Horn et al., 2011; Kurniawan et al., 2011; Bournival et al., 2012; Castro et al., 2013; Ozdemir, 2013). While most of inorganic electrolytes show positive coalescence inhibition, some salts do not affect it suggesting that specific effects and ion-water interactions play a role (Castro et al., 2013). Moreover, bore water, seawater or plant recycled water can be used in some flotation plants due to scarcity of fresh water sources. These water sources contain high concentration of dissolved ions, which can affect the flotation process (Barbian et al., 2005; Farrokhpay, 2011; Ang et al., 2013; Ozdemir, 2013; Cilek and Karaca, 2015; Gungoren et al., 2017; Xing

et al., 2017). Bournival et al. (2014) found that water and diluted solutions of electrolytes did not prevent bubbles from coalescing whereas increasing the concentration of the solutions appeared to decrease the probability of bubble coalescence. Bubble coalescence in aqueous solution is largely inhibited above a critical concentration range of electrolyte, the so-called transition concentration (Christenson and Yaminsky, 1995).

The time needed for the coalescence of bubbles when they are in close proximity is defined as bubble coalescence time (Ata, 2008; Bournival et al., 2012; Owoeye and Schubring, 2017). And, generally, high-speed cameras are used in the studies on measuring of coalescence time (Ribeiro and Mewes, 2007; Kracht and Finch, 2009; Bournival et al., 2014; Orvalho et al., 2015; Karn et al., 2016; Zawala et al., 2017). However, this method is very complex because of focusing the bubble coalescence scene by the high-speed camera and investigating the high number of frames that shot in high-speed. In addition, this method is also pretty expensive due to the high prices of high-speed cameras, appropriate lenses and the experimental set-up with motion mechanisms.

In this study, a bubble-particle attachment timer was modified to a bubble-bubble coalescence timer, first time in literature, and it was investigated the effect of dissolved ions on the bubble coalescence in the absence and presence of a frother using a new experimental method. In this purpose, surface tension experiments and bubble coalescence time measurements (the time required for the bubble coalescence) were carried out in order to observe the bubble-bubble interaction in the presence of Methyl isobutyl carbinol (MIBC) as a frother, sodium chloride (NaCl) and calcium chloride (CaCl<sub>2</sub>) as electrolytes.

## 2. Materials and methods

### 2.1. Materials

In this study, analytical grade MIBC [98% of (CH<sub>3</sub>)<sub>2</sub>CHCH<sub>2</sub>CH(OH)CH<sub>3</sub>] (ABCR, Germany), NaCl (Carlo Erba, Italy) and CaCl<sub>2</sub> (Merck, Germany) were used as a frother and electrolytes, respectively. All solutions were prepared with mono-distilled water (GFL, Germany) at room temperature (23°C ±2). All surfactant and salt solutions were freshly prepared and used.

### 2.2. Methods

The surface tension experiments with salt solutions (1×10<sup>-3</sup>, 1×10<sup>-2</sup>, 1×10<sup>-1</sup>, and 1 mol/dm<sup>3</sup>) were carried out at various MIBC concentrations (1, 10, and 100 ppm). The experiments were performed with reversed (inverted) pendant drop technique using KSV Attension Theta Lite optical tensiometer (Biolin Scientific, Sweden). The procedures used for the surface tension measurements are described in a previous paper (Ozdemir et al., 2006), and were used without modification. In this technique, an air bubble was formed in distilled water or solution by a syringe with a U-shaped needle. The surface tension was obtained by the tensiometer by observing the shape of the backlighted bubble by a CCD camera (Fig. 1). The shape of a bubble is governed by the Young-Laplace equation, which relates the pressure drop across a curved interface by Eq. 1:

$$\sigma \left( \frac{1}{R_1} + \frac{1}{R_2} \right) = \Delta P \quad (1)$$

where  $\sigma$  is surface tension (mN/m),  $R_1$  and  $R_2$  are the two principal radii of the gas-liquid interface, and  $\Delta P$  is the pressure difference across the interface. Finally, the digitized bubble profiles were fitted numerically with the solution of the Young-Laplace equation to obtain the surface tension value. Ten measurements at each concentration were performed, and the average error of the measurements was obtained about 2 mN/m.

In literature, the bubble coalescence time is usually measured with high-speed cameras (Ribeiro and Mewes, 2007; Ata, 2008; Ata, 2009; Kracht and Finch, 2009; Bournival et al., 2012; Ang et al., 2013; Bournival et al., 2014; Orvalho et al., 2015; Karn et al., 2016). Alternatively, in this study, the bubble coalescence time measurements were carried out using a modified bubble-particle attachment timer (BKT-100, Bratton Engineering and Technical Associates, LLC, USA) for the first time. The images were taken using a CCD camera (Hikvision, China).

In this measurement technique, a glass container was placed under the glass capillary tube and filled with the desired electrolyte/frother solution. Then, a glass syringe with a U-shaped needle was located beneath the glass capillary tube. Two bubbles at close sizes were formed both of at the end of the glass capillary tube and syringe. Then, the upper bubble is moved towards the bottom bubble and contacts for a certain time, and moved back to its original position. Finally, the bubbles observed on the computer screen if the coalescence occurred. The coalescence time is determined if 50% of the observations results in coalescence. The experimental set-up and the steps for the bubble coalescence measurements are given in Figs. 2 and 3, respectively. As known from literature, one of the most dominant factors that affect the bubble coalescence time is bubble bouncing (Tsao and Koch, 1994; Sanada et al., 2005), and it is strongly affected by bubble approach velocity. The bouncing probability is higher in high bubble approaching velocities (Kirkpatrick and Lockett, 1974; Zawala and Malysa, 2011). In this study, the bubble approaching speed was kept constant as 100 mm/sec.

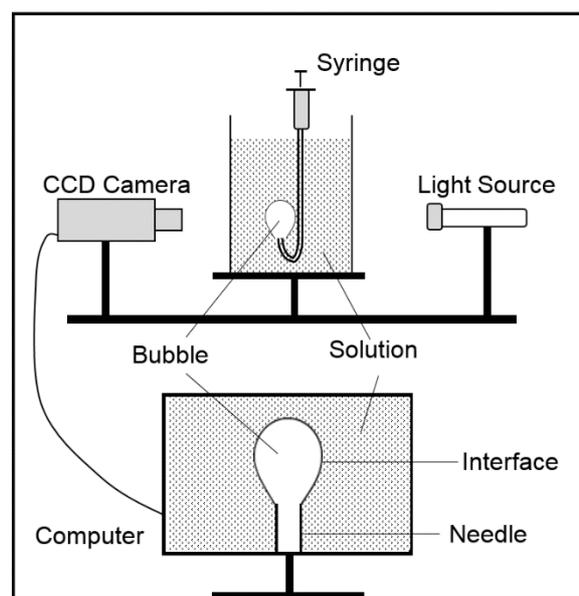


Fig. 1. Surface tension measurement setup

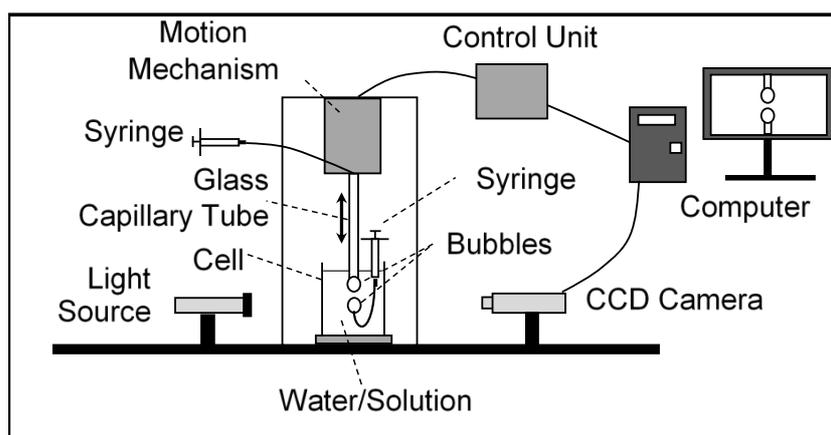


Fig. 2. Experimental set up of bubble coalescence time measurements

The bubble coalescence time measurements were carried out at 1 ppm MIBC as a function of contact time. Then, the measurements were performed in respect to MIBC concentration at 100 ms constant contact time with 40% bubble coalescence percentage, which was decided to be appropriate for decreasing the bubble coalescence percentage down to 0% with the increasing MIBC dosage and the addition of electrolytes. Additionally, the effect of NaCl and CaCl<sub>2</sub> ( $1 \times 10^{-3}$  mol/dm<sup>3</sup>) on the bubble

coalescence were investigated in the presence of 1 ppm MIBC. Furthermore, the bubble coalescence time measurements were also carried out as a function of salt concentration in the absence of MIBC in order to determine the salt transition concentration.

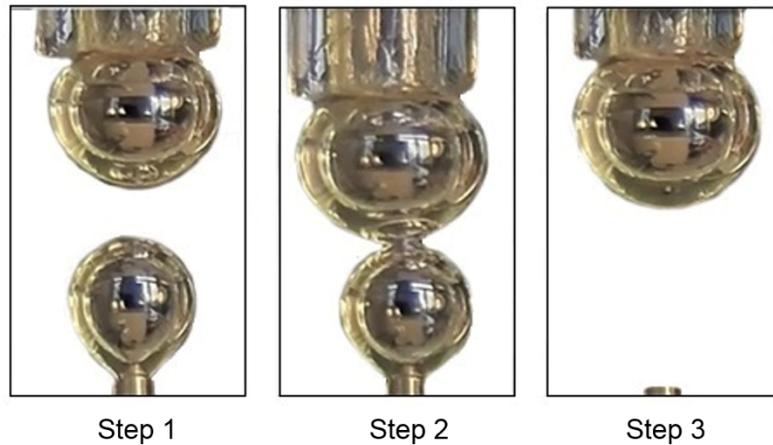


Fig. 3. Steps of a bubble coalescence time measurement

(Step 1: Two bubbles at close sizes were formed both of at the end of the glass capillary tube and syringe, Step 2: Upper bubble is moved towards the bottom bubble and contacts for a certain time. Step 3: Upper bubble is moved back to its original position resulting in coalescence.)

### 3. Results and discussion

The comparison of the surface tensions of MIBC at various concentrations in the presence of NaCl and CaCl<sub>2</sub> is shown in Fig. 4(a-d). As seen from Fig. 4(a) that surface tension of the solution was obtained as 71.5 mN/m at 1 ppm MIBC in the absence of salts. Moreover, it decreased with MIBC concentration according to salt type and concentration. It is clearly seen from Fig. 4(a-d) that CaCl<sub>2</sub> decreased the surface tension more than NaCl. While the surface tension was 62.5 mN/m at 100 ppm MIBC concentration, it decreased to 52.8 mN/m and 49.5 mN/m with the addition of 1 mol/dm<sup>3</sup> of NaCl and CaCl<sub>2</sub>, respectively.

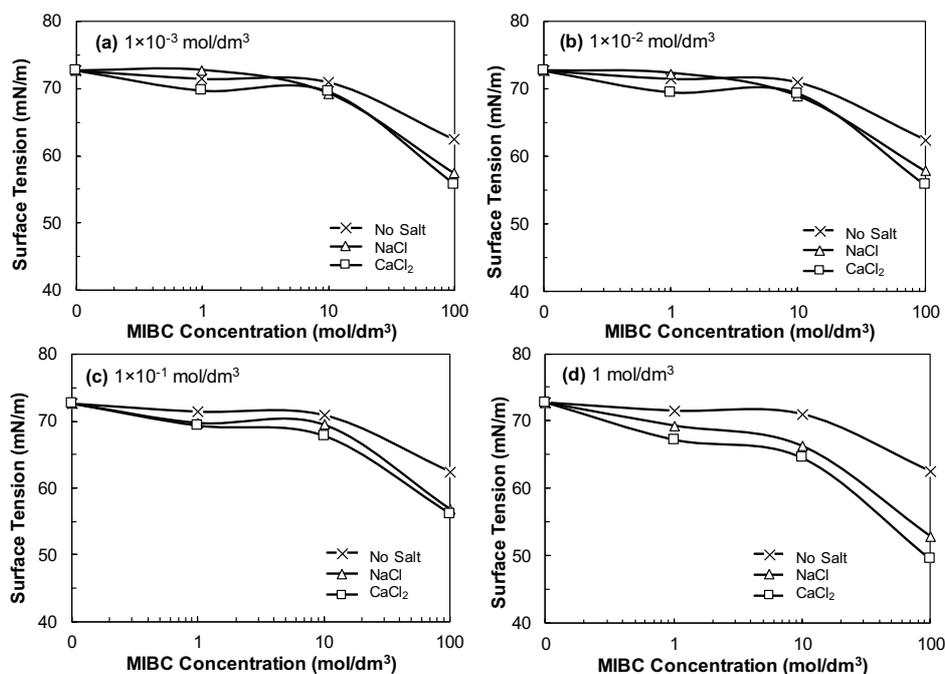


Fig. 4. Surface tension results of MIBC at NaCl and CaCl<sub>2</sub> salt concentrations (a) 1 × 10<sup>-3</sup> mol/dm<sup>3</sup> (b) 1 × 10<sup>-2</sup> mol/dm<sup>3</sup> (c) 1 × 10<sup>-1</sup> mol/dm<sup>3</sup> (d) 1 mol/dm<sup>3</sup>

Bubble coalescence measurements were first carried out at 1 ppm MIBC as a function of contact time (Fig. 5). As seen in Fig. 5 that 50% of the experiments were resulted in coalescence at 700 ms. Then, the effect of MIBC concentration on the bubble coalescence was investigated at 100 ms contact time. Fig. 5(b) showed that no coalescence was observed at 10 and 100 ppm MIBC.

The effect of NaCl and CaCl<sub>2</sub> ( $1 \times 10^{-3}$  mol/dm<sup>3</sup>) on the bubble coalescence in the presence of 1 ppm MIBC is shown in Fig. 6. As seen in Fig. 6, while the coalescence was 40% (at 100 ms contact time) in the presence of 1 ppm MIBC, it decreased to 10% with  $1 \times 10^{-3}$  mol/dm<sup>3</sup> NaCl addition, and no coalescence was occurred in the presence of 1 ppm MIBC and  $1 \times 10^{-3}$  M CaCl<sub>2</sub>.

The bubble coalescence in salt solutions as a function of salt concentration at 100 ms contact time is seen in Fig. 7. As seen in Fig. 7, the bubble coalescence for each salt inhibited with the increasing ion concentration. The transition concentration is defined as the concentration resulting in 50% coalescence percentage (Kim et al., 1990). As obtained from the results in Fig. 7, the salt transition concentration was determined as  $6 \times 10^{-2}$  mol/dm<sup>3</sup> and  $1.5 \times 10^{-1}$  mol/dm<sup>3</sup> for CaCl<sub>2</sub> and NaCl, respectively, which is agreed with literature that the values were  $3 \times 10^{-2}$  and  $8 \times 10^{-2}$  mol/dm<sup>3</sup> for CaCl<sub>2</sub> and NaCl, respectively (Marrucci and Nicodemo, 1967).

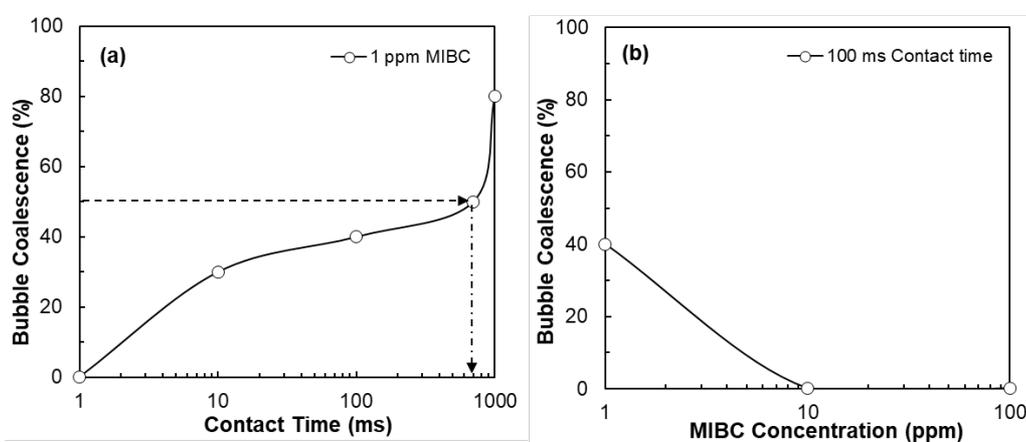


Fig. 5. Bubble coalescence percentage of MIBC (a) in respect to contact time and (b) concentration

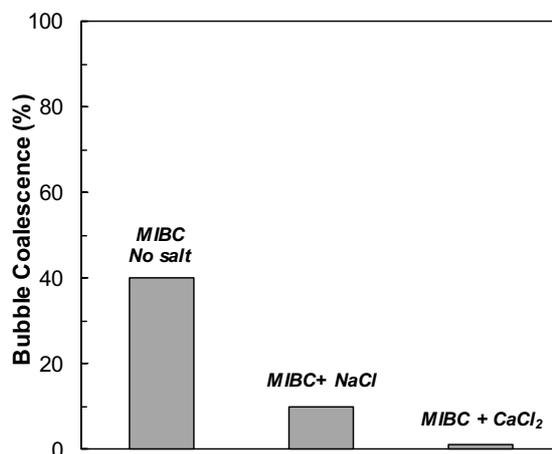


Fig. 6. Bubble coalescence in the presence of 1 ppm MIBC with  $1 \times 10^{-3}$  M of NaCl and CaCl<sub>2</sub> at 100 ms contact time

As well known, frothers decrease the surface tension of water according to its concentration. On contrary, the surface tension of water increases with most of the electrolytes including NaCl as a function of ion concentration (Weissenborn and Pugh, 1995; Ozdemir et al., 2009; Ozdemir et al., 2011; Bournival et al., 2012).

The effect of salts on bubble coalescence is strongly dependent upon to their concentration. Salts should promote bubble coalescence because of the effect of increasing the surface tension and suppressing the electrostatic repulsion between the bubbles. However, most inorganic electrolytes

stabilize bubbles above a certain concentration (Craig et al., 1993; Christenson and Yaminsky, 1995; Deschenes et al., 1998; Tsang et al., 2004; Henry et al., 2007).

The ion type is also another important factor in bubble coalescence while some salts such as sodium acetate ( $C_2H_3NaO_2$ ) or HCl do not have an effect on the bubble coalescence at any concentration, some others including NaCl or  $KNO_3$  inhibit bubble coalescence (Ozdemir et al., 2011). The reduction of bubble coalescence depends upon the valence of the electrolytes as well as the magnitude of the surface tension (Marucci and Nicodemo, 1967).

Bournival et al. (2012) reported that NaCl at higher concentrations as effective as MIBC in preventing bubble coalescence in a dynamic environment. That is to say, a relatively small amount is required to prevent coalescence when contact times are short. However, very concentrated solutions must be used in order to observe extended stability.

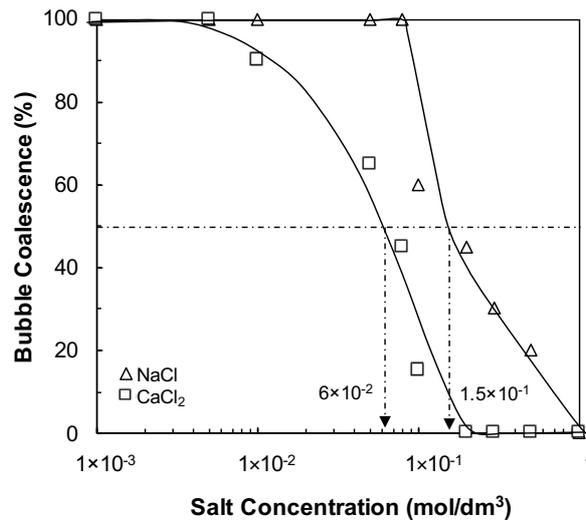


Fig. 7. Bubble coalescence in NaCl and  $CaCl_2$  salt solutions in respect to salt concentration at 100 ms contact time

#### 4. Conclusions

In flotation system, the interaction between bubble and particle as well as bubble and bubble (coalescence) play an important role on flotation recovery is higher when the bubble-particle attachment time is shorter, and the bubble coalescence time is longer. This study focused on determining the bubble coalescence time using the classical device used for measuring the bubble-particle attachment time after modification. The results of this study showed that the presence of  $CaCl_2$  on the surface tension of MIBC was more effective than that of NaCl. Using this new method, the contact time required for bubble coalescence was measured 700 ms in the presence of 1 ppm MIBC, and it was seen that no coalescence was occurred in the presence of 10 ppm MIBC at 100 ms. Furthermore, salt addition to MIBC solution decreased the coalescence probability. Salt transition concentration for  $CaCl_2$  and NaCl was determined as  $6 \times 10^{-2}$  mol/dm<sup>3</sup> and  $1.5 \times 10^{-1}$  mol/dm<sup>3</sup>, respectively. The result obtained from this study clearly indicated that the bubble coalescence time can be successfully determined with a new measurement system which will help us improving the flotation technology in the mineral processing industry.

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