



# The influence of submicron particles and salt on the recovery of coarse particles



G. Bournival<sup>a</sup>, S. Ata<sup>b,\*</sup>, G.J. Jameson<sup>a</sup>

<sup>a</sup>Centre for Multiphase Processes, The University of Newcastle, Callaghan, NSW 2308, Australia

<sup>b</sup>The School of Mining Engineering, The University of New South Wales, Sydney, NSW 2052, Australia

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## ABSTRACT

Coarse particles are more difficult to float. One of the factors that contributes to poor floatability is the stability of froth. The froth formed in industrial flotation cells is typically not strong enough to provide adequate support for coarse and dense particles. The present study investigates how the presence of hydrophobic submicron particles at low concentration increases the recovery of relatively coarse particles through improvement in the froth stability. Silica particles with  $d_{50}$  of approximately 230  $\mu\text{m}$  were floated in a laboratory mechanical flotation cell in a collector-free environment in the presence of poly(propylene glycol) 425 as a frothing agent. The hydrophobicity of the feed particles was modified through an esterification process with different alcohols ranging from 3 to 8 hydrocarbon groups to form a coating of intermediate hydrophobicity. Hydrophobised silica submicron particles of 300 nm in size were added to the flotation cell at 0.01 and 0.1 wt% concentration. The effect of electrolyte, sodium chloride, in the concentration range  $10^{-5}$ – $10^{-1}$  M on the recovery of coarse particles was also investigated. For the feed employed, 1-butanol was found to provide relatively good flotation properties with a possibility for improvement by stabilising the froth phase. Both additives slightly stabilised the froth phase, which resulted in an increase in the maximum recovery of up to approximately 8%. It appeared that the additives had no significant effect on the first-order flotation rate constant.

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## 1. Introduction

Flotation is used to selectively separate particles based on their surface properties. Bubbles, capturing particles, segregate to the surface in the froth phase. The first function of the froth phase is to transfer the mineral-covered bubbles to the concentrate launder. The second function is to provide a secondary concentration process by gravitational drainage of hydrophilic particles back to the pulp zone (Nguyen and Schulze, 2004). The particle laden bubble rises to the interface and pushes the bottom layer resulting in the formation of a thin film. Water drains through gravitational forces and causes the formation of lamellae. These lamellae can be either stabilised or destabilised by the presence of particles. This is significant since generally particles play a larger role than the frother to determine the stability of the froth phase (Farrokhpay, 2011).

There are numerous accounts on the effect of particles on foam and froth stability. The increased or decreased stability is attributed to a range of factors such as hydrophobicity, size, shape,

and concentration (Dippenaar, 1982; Pugh, 1996; Ata et al., 2003; Schwarz and Grano, 2005). Morris et al. (2008) theoretically investigated the effect of particle separation distance and hydrophobicity on the stability of froth films. The capillary pressure required to reduce the separation distance to zero between the film elements was found to decrease with increasing contact angle and increasing separation distance between the particles. However, the attachment probability is low for lower particle contact angles. This work agrees with the experimental observations that the froth becomes less stable and more mixed in behaviour as the pulp density decreases (Goodall and O'Connor, 1991). It is also known that, although reducing the concentrate grade, naturally floatable gangue increases the concentration of particles in the froth, which can improve its stability (Corin et al., 2011). An alternative to the monolayer steric barrier is the streaming of particles to the Plateau border, which increases the rigidity of the interface and, successively, the stability of the whole froth (Johansson and Pugh, 1992). As such, the presence of fine particles was observed to affect the structure of the froth and promote the recovery of coarser particles (Vieira and Peres, 2007; Rahman et al., 2012; Ata and Jameson, 2013). It has been shown that the froth phase acts as a barrier when it comes to the recovery of coarse particles

\* Corresponding author.

E-mail address: [s.ata@unsw.edu.au](mailto:s.ata@unsw.edu.au) (S. Ata).

(Rahman et al., 2012). The presence of an increasing amount of finer particles increased the recovery of coarser particles. The basic operative mode of finer particles to improve coarse particle recovery was by preventing bubbles from coalescing and providing a more structurally sound froth. Pugh (1996) noted that from a mixture of particles, only a few particles are required to destabilise a foam given the appropriate properties. However, for stabilising a foam, a relatively large concentration of particles with stabilising properties is required. It follows that in flotation, these particles are recovered and purposely depleted from the system (Zanin et al., 2009). As the number of particles is depleted, the stability of the froth phase becomes more reliably dependent on other variables such as the amount of frothing agent for example. It has been proposed that the addition of colloidal particles could be used to increase froth stability due to their success in foams (Ata, 2012).

The foam stability induced by surfactant can be improved by the addition of hydrophobised silica submicron particles of approximately 300 nm or salt as previously established in a column (Bournival et al., 2014a). At a low surfactant concentration, the inorganic electrolyte was believed to dampen the attractive hydrophobic force, which destabilises foams. The addition of submicron particles into some non-ionic surfactants solutions was found to increase the foaminess of the solutions. These authors also showed that the structure of the foam phase could be improved in the presence of submicron particles presumably due to the reduction in bubble coalescence within the foam phase. The aim of this study is to investigate the effects of low concentrations of colloidal particles of intermediate hydrophobicity and NaCl on the batch flotation of relatively coarse silica, which were expected to be inherently more difficult to recover. Since improvements were observed in gas holdup and foaminess in our previous study, the results were analysed in terms of both the first-order flotation rate constant and the maximum recovery. It is theorised that increasing the gas holdup would increase the residence time of the bubbles in the pulp which in turn could increase the flotation kinetics. The depletion of particles, characteristic to batch experiment, offers a system in which an increased foaminess of the solution, in the absence of floatable particles, could result in an increase in recovery. However, other parameters, such as the froth depth and the hydrophobicity of the feed particles need to be tested to make the possible gain in froth stability meaningful.

## 2. Methodology

### 2.1. Materials

The submicron particles used were SP-0.3B silica synthesised using the Stöber method. The particles were supplied as a dry powder by FUSO Chemicals (Japan). The average number and volume size distribution were determined by dynamic light scattering (Malvern Zetasizer Nano ZS). The number size distribution ranged 260–272 nm whereas the particle average volume size distribution was 299–310 nm. The chemical reagent used in modifying the hydrophobicity of the particles is 1-octanol (Merck, for synthesis), which was removed with acetone (Chem-Supply,  $\geq 99.8\%$ ) and ethanol (Chem-Supply, absolute ( $\geq 99.8\%$ )).

The flotation feed was composed of a graded silica 50 N provided by Unimin Australia Ltd (Victoria). A key reason for selecting a grade of 50 N is the relatively narrow size distribution of the particles and the absence of fine particles, which tend to be collected through entrainment. In addition, the absence of fine particles will not interfere with the effect of submicron particles. The Sauter mean diameter of the particles is 235  $\mu\text{m}$  and the 80% passing size is 230  $\mu\text{m}$  as determined using a Mastersizer 2000 apparatus (Malvern Instruments). As mentioned previously, such large

particles produce less stable froth than their finer counterpart. Therefore, it was investigated if the presence of submicron particles can help to recover particles which could tend to be more complex to recover. Similarly to the submicron particles, the hydrophobicity of the flotation feed was modified by esterification with different alcohols, which includes 1-propanol (Sigma–Aldrich, ACS reagent  $\geq 99.5\%$ ), 1-butanol (Chem-Supply, analytical reagent  $\geq 99.0\%$ ), 1-hexanol (Sigma–Aldrich, reagent grade  $\geq 98\%$ ), and 1-octanol after washing with sodium hexametaphosphate (Sigma–Aldrich, 96%). The contact angle given by the different alcohols were measured on silicon wafers (Silicon Valley Microelectronics, U.S.A.), which had a 115 nm thermal oxide layer. The wafers were cleaned using ammonia (Merck, 25 v/v%) and hydrogen peroxide (Chem-Supply, 30 v/v%). In addition to the alcohols previously mentioned, 1-pentanol (Sigma–Aldrich, ACS reagent,  $\geq 99\%$ ) and 1-dodecanol (Sigma–Aldrich, ACS reagent  $\geq 98\%$ ) were used.

The pH of the dispersions were maintained at 9 by using a buffer solution of boric acid ( $\text{H}_3\text{BO}_3$ ) (Analar Merck Pty Ltd., analytical grade) and sodium hydroxide (NaOH) (Ajax Finechem Pty Ltd., analytical grade). The ionic strength of the solutions was adjusted using sodium chloride (NaCl) (Merck, analytical grade) whereas poly(propylene glycol) (PPG) with a number average molecular weight of approximately 425  $\text{g mol}^{-1}$  (Sigma–Aldrich,  $\geq 99\%$ ) was used as a frothing agent.

All glassware and labware were cleaned as previously reported (Bournival et al., 2014b). All water used in the experiments, including washing glassware, was dispensed from a Milli-Q Pure water system and had a resistivity of 18.2  $\text{M}\Omega \text{ m}^{-1}$ . Experiments were carried out in a temperature controlled room at  $20 \pm 2^\circ \text{C}$ .

## 3. Experimental procedure

### 3.1. Surface modification of silica

The particles (1 kg) were cleaned in a solution of 25 g of sodium hexametaphosphate (Sigma–Aldrich, 96%) in 1 L of water at pH 8–9 adjusted with a solution of NaOH. Particles were stirred for 30 min before being rinsed and decanted in water for several times. Particles were then dried in an oven at a temperature of 60  $^\circ\text{C}$ .

In order to observe the effect of frother and submicron particles only on the recovery and flotation kinetics, no surfactant (i.e. collector) was added. Instead the particles were artificially hydrophobised using an esterification process. The procedure for the treatment of submicron particles with 1-octanol can be found elsewhere (Bournival et al., 2014a). Briefly, modification was carried out by reacting 50 g of silica nanoparticles with 130 g of 1-octanol (Merck, for synthesis). The alcohol was refluxed in a 500 ml insulated conical flask for a period of 7 h. The particles were then centrifuged (Centurion Scientific C2) to remove the alcohol and the particles were dispersed twice in acetone (Chem-Supply,  $\geq 99.8\%$ ) and in ethanol (Chem-Supply, absolute ( $\geq 99.8\%$ )), both solvent being removed by centrifugation. The particles were dried in a dust-free environment (i.e. laminar flow cabinet) and stored in a vacuum desiccator. For purposes of discussion, the esterified silica nanoparticles are designated  $\text{Si-OC}_8\text{H}_{17}$ .

The esterification process for the feed particles was similar to that used for the nanoparticles employed to make the particles hydrophobic. For each esterification treatment, 150 g of silica was placed in the reaction vessel with 390 g of alcohol. The different alcohols used were 1-propanol, 1-butanol, 1-hexanol, and 1-octanol. The vessel was placed in a heating mantle to boil and condense the alcohol for 7 h. The particle/alcohol mixture was stirred by a laboratory impeller during that period. After the alcohol had cooled down, it was decanted. The particles were washed and decanted four times in acetone and twice in ethanol.

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