

Foaming and gas dispersion properties of non-ionic frothers in the presence of hydrophobized submicron particles



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ABSTRACT

Foams represent an important area of research due to their relevance in many industrial processes. In many systems, particulates and surfactants co-exist and can largely define the stability of the foam. A typical example is froth flotation where hydrophobic particles and frothers are essential to maintain a stable froth. Grinding operations may yield a product containing submicron-size particles, which can affect the flotation process. The present study compares the foaming and gas dispersion properties of the non-ionic surfactants 1-pentanol, tri(propylene glycol) methyl ether, and poly(propylene glycol) 425 in the presence of hydrophobic colloidal silica particles in a column. In all cases, it has been found that the gas holdup increases proportionally with the increase in the concentration of particles in the system although an initial decrease may be observed. The effect of particles on the foaming seems to be dependent upon the surfactant. With the 1-pentanol, the addition of particles systematically decreased the foaming of the solutions. This is in contrast to poly(propylene glycol) solutions, which exhibited an increase. Interestingly, low concentrations of particles appeared to improve the foaming of tri(propylene glycol) methyl ether; however, the froth became less stable with further increases in the number of particles in the column. Visual observation showed that the colloidal particles play a crucial role in defining the quality of the structure of the froth.

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1. Introduction

Dispersed fine particles are known to impact the stability of foams in the pulp and paper industry, oil distillation, water treatment, and many other industrial processes (Hunter et al., 2008). The foam stabilisation mechanism can be classified into two broad categories: particles in the continuous phase and particles at the air–liquid interface. Particles in solution can have a significant coalescence retarding effect on bubbles (Sethumadhavan et al., 2001a, 2001b, 2004; Wasan et al., 1992, 2005). For sufficiently high concentrations of particles an internal layering of the particles occurs, which is generated by an entropic excluded volume effect. This phenomenon is caused by an osmotic pressure gradient arising from the draining out of particles from the film, which are replaced by vacancies. The number of layers increases with particle concentration. The thinning of the film occurs in a step-wise manner called stratification.

Alternatively, hydrophobic particles adsorbed at the air–dispersion interface can offer substantial resistance to coalescence through various mechanisms (Hunter et al., 2008). One of the basic mechanisms is the formation of a steric barrier (Kaptay, 2004, 2006). Thus, an increased capillary pressure is required to thin the liquid film separating two

interfaces to zero. The actual value of the additional pressure is dependent on factors such as the geometrical packing, particle agglomeration, and particle hydrophobicity. Some of these characteristics also affect the attachment of particles at the interface.

The recovery of fine particles by flotation has been the subject of detailed research. Theoretically, the probability of the adhesion of fine, micron-size, particles of any hydrophobicity is extremely high (Luttrell and Yoon, 1992; Yoon and Luttrell, 1989). However, attachment can only take place upon the collision of the particle with a bubble. Particles less than 10 μm, due to their small inertial force, do not collide with larger bubbles but rather follow the streamlines around the bubbles (Yoon and Luttrell, 1989). The flotation rate of ultrafine particles can be enhanced by reducing the bubble size and/or increasing the shear rate (Jameson, 2010). Although the collision efficiency decreases with the size of the particles, there is a minimum after which a further decrease in particle size causes an increase in the collection efficiency. Deviation from hydrodynamic models as the particle size decreases is attributed to the Brownian motion of the particles. Whilst gravitational and inertial forces become negligible, a growing effect of surface forces is observed (Collins and Jameson, 1976, 1977; Nguyen et al., 2006).

The adsorption of negatively charged particles at an air–water interface is rather complex even in the case of a stagnant interface, which simplifies the hydrodynamic conditions. The standard DLVO theory predicts an infinite energy barrier since the van der Waals potential energy

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is repulsive for the interaction of a particle (e.g. negatively charged silica) and the air–water interface. Although, the adsorption of hydrophobic colloidal particles is thermodynamically favourable, a non-DLVO potential energy must be accounted for to explain the kinetic instability (i.e. adsorption). The attractive potential energy arises from the solvation zone. The solvation zone (i.e. sphere/plane of influence) is a zone of structural order (or disorder) surrounding an interface due to its affinity with the medium molecules. The perturbation of the solvent molecules gives rise to attractive forces between the two surfaces and explains the instability of the particles with the air–water interface. The energy barrier for the adsorption of particles onto the air–water interface can be reduced by the addition of electrolytes (Abdel-Fattah and El-Genk, 1998a, 1998b; Nguyen et al., 1998) and increasing the hydrophobicity of the particles (Mao and Yoon, 1997; Nguyen et al., 1997). However, salts also affect the particle–particle interaction. Many studies found improved foam stability for conditions under which particles are coagulated by changing the ionic strength (Dickinson et al., 2004; Kostakis et al., 2006); or the pH (Blute et al., 2007; Pugh, 2007) or adding surfactant (Carn et al., 2009).

In froth flotation, particles can be responsible for stabilising the froth phase. The stabilising and destabilising effects are dependent on a number of parameters including particle size, shape, hydrophobicity, and concentration (Ata, 2008; Dippenaar, 1982; Johansson and Pugh, 1992; Morris et al., 2008, 2011; Subrahmanyam and Forssberg, 1988). However, in an industrial flotation plant where the feed is passed through a bank consisting of a series of cells, the most floatable particles are removed in the first few cells. Separating and recovering particles, therefore imply a depletion of stabilising particles along the bank, which weakens the froth phase (Zanin et al., 2009). One way to overcome the problem is to add submicron particles into the flotation cell to compensate for the depletion of hydrophobic particles (Ata, 2012).

This study investigates the effect of the addition of submicron silica particles of intermediate hydrophobicity on the foaming and gas dispersive properties of non-ionic surfactants. The foaming of dispersions was evaluated using a standard Bikerman column technique. The gas dispersion characteristics were quantified by measuring the gas holdup by the differential pressure method and the sizing of bubbles. In the minerals industry, *froth* is used to describe the bubble-rich mixture that forms on top of the liquid in a flotation cell. In the wider literature, such mixtures are referred to as *foams*. In this paper, *foam* is sometime used, although it is understood that *froth* and *foam* are interchangeable.

2. Methodology

2.1. Materials

The 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 size distribution, determined by dynamic light scattering (Malvern Zetasizer Nano ZS), ranges 260 to 272 nm, whereas the particle average volume size distribution is 299 to 310 nm, which is in agreement with the nominal size of 300 nm provided by the manufacturer. The chemical reagents used for modifying the hydrophobicity of the particles include 1-octanol (Merck, for synthesis), acetone (Chem-Supply, $\geq 99.8\%$), and ethanol (Chem-Supply, absolute ($\geq 99.8\%$)).

The pH of the dispersions was maintained at 9 using a buffer solution of boric acid (H_3BO_3) (Analar Merck Pty Ltd., analytical grade) and sodium hydroxide (NaOH) (Ajax Finechem Pty Ltd., analytical grade).

The non-ionic surfactants tested covered a range of properties. The alcohol surfactant tested was 1-pentanol ($\text{CH}_3(\text{CH}_2)_3\text{CH}_2\text{OH}$, MW = 88.15 g mol⁻¹; Sigma-Aldrich, $\geq 99\%$) and represents a typical short-chain, weak surfactant. Tri(propylene glycol) methyl ether (abbreviated TPM) ($\text{CH}_3(\text{OC}_3\text{H}_6)_3\text{OH}$, MW = 206.28 g mol⁻¹; Sigma-Aldrich, $\geq 97.5\%$); and poly(propylene glycol) (abbreviated PPG)

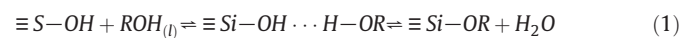
($\text{H}[\text{OCH}(\text{CH}_3)\text{CH}_2]_7\text{OH}$, $M_n \approx 425$ g mol⁻¹; Sigma-Aldrich, $\geq 99\%$) were the polyglycol surfactants. TPM and PPG were selected due to their relevance to the flotation industry being the main constituents of Dowfroth 200 and Dowfroth 400.

All glassware was soaked in a mixture of 30%v/v of HNO_3 (analytical grade, Chem-Supply) in H_2SO_4 (analytical grade, Ajax Finechem Pty Ltd.) for 5 to 10 min and then thoroughly rinsed with water (resistivity 18.2 M Ω m⁻¹) dispensed from a Milli-Q Pure water system. The column was cleaned by first adding ethanol and brushing the wall of the column to remove particles that could be stuck on the glass. The column was thoroughly rinsed with water followed by a chemical cleaning using an alkaline solution, which was prepared by dissolving 120 g of sodium hydroxide in 120 g of water followed by the addition of 1 L of ethanol. The solution was poured in the column and left for 10 to 15 min to ensure that the surface was free of contaminants. The column was then rinsed with water before neutralising any residual from the alkaline solution with a solution of 10⁻³ M of hydrochloric acid (HCl) (Ajax Finechem, Pty Ltd., analytical reagent). Subsequently, the column was thoroughly rinsed with water. Experiments were carried out in a temperature controlled room at 20 \pm 2 °C.

2.2. Experimental procedure

2.2.1. Esterification of silica particles

Surface modification, not involving surfactants, is often carried out by silanization (Flinn et al., 1994). However, the reaction is subjected to side reactions, such as polymerisation, and is influenced by the water/moisture content, which may lead to unpredictable, inhomogeneous coatings if not properly controlled (Biggs and Grieser, 1994; Flinn et al., 1994; Tolnai et al., 2001; Trau et al., 1992). An alternative to the use of alkoxy silane agents in the surface modification of silica is found through esterification. The coating, which is considered to form a uniform monolayer (Biggs and Grieser, 1994), can be made using alkyl alcohols with the extent of hydrophobicity dictated by the length of the alkyl chain (Kimura et al., 1998). Unlike silanization, esterification is unaffected by side reactions (Ossenkamp et al., 2001, 2002; Trau et al., 1992). A general reaction (i.e. alkoxylation) starts with the physisorption of the alcohol molecule with the silanol group. This is followed by the chemisorption of the alcohol molecule forming an alkane through a condensation reaction. The reactions, which are reversible, follow (Ossenkamp et al., 2001):



A possible drawback of esterification is the hydrolysis of the silica surface as implied by Eq. (1). However, several alcohols were shown to be sufficiently resistant to hydrolysis (Hunter, 2008; Ossenkamp et al., 2001).

Following the work of Hunter (2008), the alcohol used for this study was 1-octanol. This alcohol produced particles of intermediate hydrophobicity, which is ideal for the stabilisation of foams with an advancing contact angle of 73.5° and a receding contact angle of 67.5° (Hunter, 2008; Hunter et al., 2009a). A contact angle hysteresis (i.e. the difference between the advancing and receding contact angles) of less than 10° is generally associated with a uniform coating (Flinn et al., 1994).

The modification of colloidal silica particles was carried out by reacting 50 g of silica with 130 g of 1-octanol. The alcohol was boiled/condensed and refluxed in a 500 ml insulated conical flask for a period of 7 h. The dispersion was magnetically stirred to avoid the coagulation of the particles. A drying tube, filled with a desiccant (i.e. CaCl_2), was employed on the top of the extractor to limit moisture uptake from the surrounding.

After cooling to room temperature, the particles were centrifuged (Centurion Scientific C2) in test tubes of 50 ml at 2300 rpm for 3 h. The supernatant was removed, and the particles were dispersed in acetone and centrifuged at 1900 rpm for a period of 50 min. The

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