



# The Effect of Particles on Surface Tension and Flotation Froth Stability

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## Abstract

It is widely accepted that particles stabilise flotation froths and that stable froths result in improved flotation performance. Predicting the effect of particle addition on froth stability is, however, challenging. Dynamic surface tension measurement using maximum bubble pressure presents an attractive technique to investigate the effect of surfactant and particles at the air-water interface. The range of bubble lifetimes that can be studied (typically 0.1 to 60 s) is analogous to variations in air rate in flotation cells, and the corresponding changes in surface tension give an indication to the diffusion and adsorption rates of particles at the interface. In this paper, we use dynamic surface tension measurements to investigate the effect of particles on bubble surfaces at the microscale and link this to bulk froth stability measurements carried out using a froth column. Using the maximum bubble pressure method, the results show that the addition of particles results in lower surface tension, both at the dynamic (i.e. short) bubble lifetimes and towards equilibrium (i.e. 60 s bubble lifetime). This corresponds with the bulk froth stability measurements, where the three-phase system yielded more stable froths than the surfactant only system. Furthermore, increased particle loading at the air-water interface, whether through higher surfactant concentrations or lower air rates (longer bubble lifetimes), gave lower surface tension and higher froth stability. This demonstrates the link between bubble loading and froth stability. It is proposed that the maximum bubble pressure technique can be used to predict froth stability for two- and three-phase systems, enabling the effect of particle loading to be accounted for and quantified. Moreover, the technique has the potential to allow rapid determination of particle and surfactant diffusion at the air-water interface and prediction of the corresponding effect on bulk froth behaviour.

**Keywords** Froth flotation · Flotation froths · Froth stability · Surface tension

## 1 Introduction

Froth flotation is a separation technique that is based on differences in the surface properties of particles and which is used at a huge scale in the minerals industry. The performance of the separation is dependent on the stability of the overflowing froth phase, where operating under conditions that yield stable froths results in improved mineral recovery [1, 2]. Froth stability can be described in a number of ways, including bubble burst rate [3], air recovery [1] and the froth stability column [4].

The Bikerman column is one of the most widely used methods of quantifying froth and foam stability, giving rise to the unit of ‘foaminess’, or dynamic froth stability,  $\Sigma$  [5]

(Eq. (1)):

$$\Sigma = \frac{V_f}{Q} = \frac{H_{\max}A}{Q} \quad (1)$$

The concept of the Bikerman column is that an equilibrium froth volume ( $V_f$ ) is obtained where the inlet air flowrate ( $Q$ ) is equal to the air leaving through bursting at the top surface. The equilibrium froth height ( $H_{\max}$ ) and the column cross-sectional area ( $A$ ) yield the froth volume. The ‘foaminess’ is typically referred to as the dynamic froth stability factor. The simplicity of the Bikerman column approach, and the use of air flowrate as a key variable, has resulted in extensive use for froth stability quantification, from industrial flotation cells [6] and laboratory flotation tests [7–9], to more fundamental studies of foam behaviour [10]. In [5], for example, the dynamic froth stability was linked directly to the flotation performance.

A flowing froth has two key properties: Bubble stability and froth mobility [11]. At the bulk scale, froth mobility is governed by operating variables such as air rate and froth properties such as water content, while bubble stability is

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linked strongly to particle attachment [12]. At the microscale, particles and surfactant molecules act at the air-solution interface, affecting surface tension and surface viscoelasticity and therefore film stability [10], while at the macroscale, the stability of foams and froths is governed by coarsening, coalescence and drainage [13].

The role of particles in stabilising (and destabilising) foam films has been the focus of much research, reviewed comprehensively by Wang et al. [10] and Hunter et al. [14]. Surfactants stabilise foams by reducing the interfacial surface tension [14], however the effect of particles on interfacial surface tension has received comparatively little attention, particularly in terms of particle loading at the interface. In [15], for example, it was shown that particles adsorbed at an air-water interface could inhibit bubble coarsening by increasing the surface dilational elasticity of the interface, which is a function of surface tension and interfacial area. They showed that, for silica nanoparticles adsorbed at the air-water interface, an increasing concentration of particles resulted in a lower surface tension and increased dilational elasticity. Another study [16] measured the surface tension of a titania particle ( $< 1.4 \mu\text{m}$ ) suspension using a DeNouy ring. The surface tension decreased when increasing the mass loading of particles from 0 to 5% and increased to above the surface tension of pure water at higher concentration.

For particle sizes more typical of flotation systems, Brian and Chen [17] suspended iron and silicon oxide particles in three size range up to  $106 \mu\text{m}$  at solids concentrations as high as 45%. They employed the maximum bubble pressure method and agitated the solution to suspend the particles, which apparently increased the surface tension. No surfactant was present, and the authors concluded that ‘for practical purposes the surface tension of the liquid was unaffected by the addition of solid particles’. Additionally, Kihm and Deignan [18] measured the dynamic surface tension of the  $55 \mu\text{m}$  coal suspensions in the presence of various surfactants. Solids concentrations of 40% and 50% were tested in combination with a number of surfactant concentrations. Particles were suspended by the addition of a xanthan gum stabiliser. Tests were performed using the maximum bubble pressure tensiometer, with bubble rates of  $10 \text{ bubbles s}^{-1}$ . Without any surfactant, the surface tension was higher than that of pure water, as found by [16], and also higher for the higher solids concentration. As the surfactant concentration was increased, the surface tension decreased, but remained higher for the higher solids concentration.

The maximum bubble pressure method is particularly attractive for studying foam and froth systems as it replicates the dynamic process of bubble formation and the diffusion of surfactant molecules and/or hydrophobic particles to the air-solution interface. A thorough analysis of the operation and analytical shortcomings of the maximum bubble pressure method is given in [19, 20]. One further benefit is that it allows

variation of bubble lifetime, therefore dynamic surface tension and diffusion effects can be explored. Bubble-particle attachment times in flotation systems have been shown to be short; static induction time measurements are of the order of  $< 250 \text{ ms}$  [21, 22].

Although previous studies have investigated the effect of particles on interfacial surface tension, and it is accepted that reduced surface tension results in more stable foams, there is no study that explicitly links particle attachment, surface tension and froth stability. Furthermore, there is currently no method for predicting the froth stability for a given particle and surfactant combination. In this paper, the effect of surfactant concentration and particle addition on surface tension and froth stability is measured. It is shown that the maximum bubble pressure tensiometer results can be correlated to froth stability trends for different combinations and concentrations of particles and surfactant.

#### Materials and Methodology

All experiments were carried out using spherical glass beads (ballotini) and the cationic surfactant trimethyltetradecylammonium bromide (TTAB, Sigma Aldrich). All experiments were carried out at concentrations lower than the critical micelle concentration (CMC) which has variously been reported as  $1.6 \text{ mM}$  [23] and  $3.70 \text{ mM}$  [24]. Deionised water was used throughout the experiments. The ballotini had a nominal size range of  $40\text{--}70 \mu\text{m}$ , with a measured  $d_{50}$  of  $65.7 \mu\text{m}$  and a surface area of  $0.09 \text{ m}^2 \text{ mg}^{-1}$ , determined by Malvern Mastersizer. SEM images of the particles showed they were spherical. The  $\text{pH}_{\text{PZC}}$  and  $\text{pH}_{\text{IEP}}$  for silica particles have been reported [25] as 4.7 and 2.7, respectively, therefore the surfaces can be assumed to be negatively charged in deionised water.

### 1.1 Surface Tension Measurements

Dynamic surface tension was measured using an online SITA Scienceline t60 maximum bubble pressure tensiometer. For three-phase experiments (i.e. with particles), a magnetic stirrer was used to maintain the solids in suspension. The measuring vessel was fitted with baffles to stop vortex formation. It was confirmed that the distance of the capillary tube from the stirrer did not affect the tensiometer readings. A water bath was used to maintain a steady temperature. Before each test, the system was calibrated using deionised water. Preliminary tests were also carried out to ensure the volume of liquid in the tensiometer did not affect the readings, and this was maintained at  $50 \text{ ml}$  throughout all experiments.

Surface tension was measured over a range of bubble lifetimes, from  $0.03$  to  $60 \text{ s}$ , and  $60 \text{ s}$  was assumed to be the equilibrium surface tension. Each bubble lifetime was repeated three times, allowing confidence intervals to be determined using Student’s  $t$  test. In experiments where no solids were present, the surface tension was measured both with and without stirring. The TTAB concentrations used were  $0 \text{ mM}$ ,

0.2 mM, 0.3 mM and 0.4 mM. An additional dynamic surface tension test was carried out at a TTAB concentration of 1 mM. Whenever solids were added to the system, it was at a concentration of 4% (w/v). The maximum bubble pressure method determines surface tension by assuming a bubble radius; given the small size of particles relative to the bubble, it has been assumed that the effective radius is still that of the capillary tip.

## 1.2 Dynamic Froth Stability Experiments

For the froth stability experiments, an open-ended, non-overflowing froth stability column, adapted from [5], was constructed using a 1.4 m transparent Perspex tube (internal diameter 33.6 mm) and a porous sintered glass frit (pore size 100–160  $\mu\text{m}$ ). A tube connected an air flow meter and air supply to the bottom of the column, below the frit, as shown in Fig. 1.

Dynamic froth stability measurements were carried out using TTAB concentrations of 0 mM, 0.2 mM, 0.3 mM and 0.4 mM and with air flowrates from 0.2 to 0.6  $\text{L min}^{-1}$  (3.33 to 10  $\text{cm}^3 \text{s}^{-1}$ ). In tests where solids were added, this was at a concentration of 4% (w/v).

For two-phase experiments, 100 mL of solution was added to the column and the air supply turned on immediately. For three-phase experiments, the dry ballotini were added to the column before the surfactant solution was added. The equilibrium froth height ( $H_{\text{max}}$ ), the height at which the volume of air

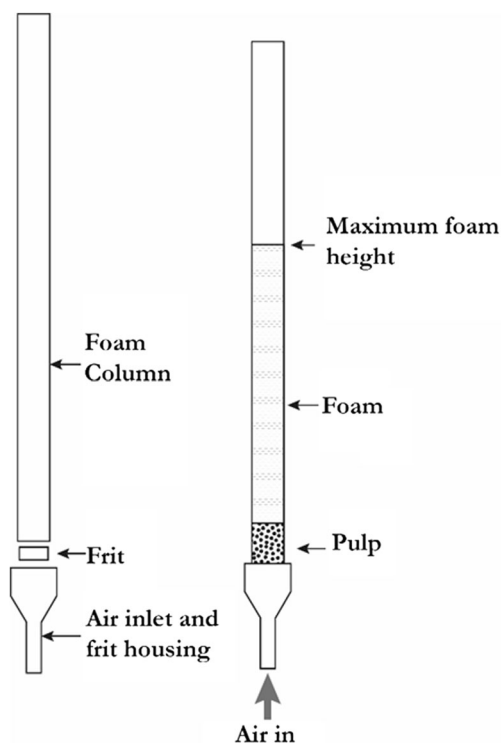


Fig. 1 Diagram showing the froth stability column experimental system

forming bubbles per unit time is equal to the volume of air escaping from burst bubbles, was measured and the dynamic froth stability calculated using Eq. (1). Each experiment was repeated five times, with confidence intervals calculated using Student's *t* test.

## 1.3 Comparison Between Maximum Bubble Pressure Method and Dynamic Froth Stability

For the maximum bubble pressure method, the varying bubble lifetime is analogous to variation in air flowrate [20], as depicted in Fig. 2. An increase in bubble lifetime using the tensiometer is equivalent to a decrease in air flowrate.

The estimated bubble lifetime at the base of the froth stability column is several orders of magnitude shorter than the equilibrium surface tension lifetime of 60 s, suggesting that the behaviour of the particulate system is better represented by the dynamic range of bubble lifetimes. Over the height of the froth, however, total bubble lifetimes are longer than those used in the equilibrium surface tension measurement.

In this paper, surface tension at various bubble lifetimes in the maximum bubble pressure method is compared directly to froth stability at a range of air flowrates in the froth stability column. While the exact magnitudes of air rate and bubble lifetime are not comparable, the trends are comparable in terms of the degree of particle attachment at the bubble surface.

## 2 Results and Discussion

### 2.1 Turbulence and Diffusion Effects on Measured Surface Tension

The effect of surfactant concentration and particle addition on dynamic surface tension was investigated using the maximum bubble pressure method. The bubble lifetime is automatically increased from the minimum to the maximum and the surface tension measured for each bubble. To maintain the particles in

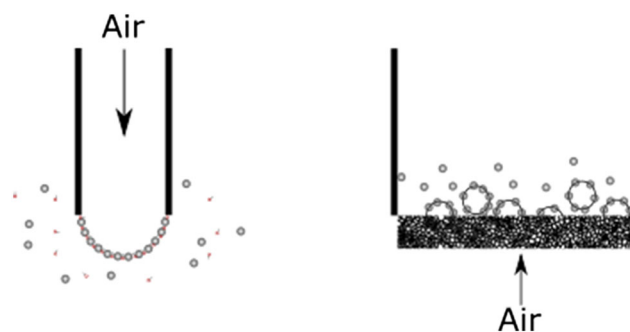


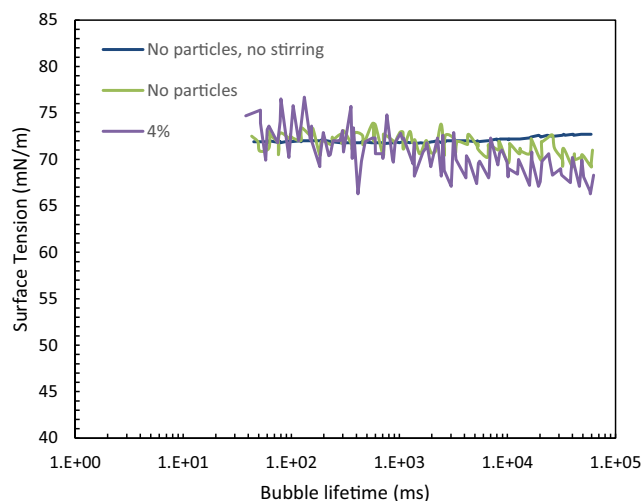
Fig. 2 Diagram depicting the diffusion of particles and TTAB molecules to the air-solution interface in the maximum bubble pressure tensiometer (left) and in the froth stability column (right)

suspension, it was necessary to stir the mixture. Baffles were placed in the measurement cell to avoid the formation of a vortex causing a surface tension gradient across the cell [19]. Conversely, the introduction of baffles and stirring introduced turbulence into the system. The effect and magnitude of the turbulence on the measured value of dynamic surface tension was determined through an investigation into stirring using deionised water only (Fig. 3).

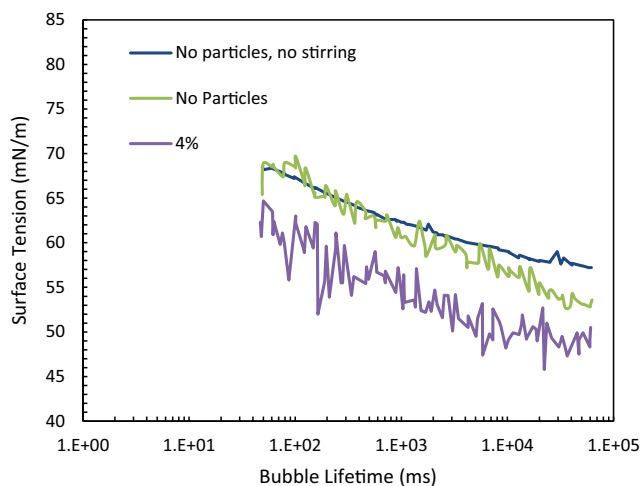
For pure water, no surface tension gradients with variation in agitation or bubble lifetime are expected. With stirring, however, the dynamic surface tension exhibits random variation and decreases below that of the unstirred system for long bubble lifetimes ( $> \sim 1$  s), suggesting that the measured surface tension is affected by the turbulence. One explanation is that the disturbance of the bubble formed at the capillary tip results in a change in interfacial area or effective bubble radius that affects the measured pressure drop, but which is not accounted for in the calculation of the surface tension by the instrument. Alternative measurements, such as those using the oscillating drop method, can account for fluctuations in the interfacial area and surface elasticity [10], however they are less comparable directly to the flotation system.

When silica particles are added to the system with no TTAB present (Fig. 3), no significant change in measured surface tension is expected, and no significant change is observed. Silica carries a negative surface charge at natural pH [25], and the air-water interface also appears anionic [15], leading to an electrostatic barrier to adsorption of the particles on the bubble. The turbulence produces a large random variation in the measured surface tension, as was seen for the particle-free, stirred system.

Figure 4 shows the dynamic surface tension for a 1 mM TTAB concentration, and a significant decrease in surface tension is observed compared to Fig. 3.



**Fig. 3** Dynamic surface tension measurements for water comparing stirred and unstirred system and with particles added



**Fig. 4** Dynamic surface tension measurements for 1 mM TTAB comparing stirred and unstirred system and with particles added

It is not possible, using the maximum bubble pressure technique, to separate the effects of surfactant diffusion and turbulence. When particles are introduced into the system with TTAB present, TTAB adsorbs both at the air-solution interface and at the surface of the silica particles due to the opposite charge between the silica surface and the ammonium cation [26]. This renders the silica particles hydrophobic and affects their attachment to the bubble surface, in addition to affecting the apparent surface tension of the air-water interface. As bubble lifetime increases, there is a decrease in surface tension that may be due to the increased diffusion of TTAB molecules at the interface, or to the increased attachment of particles carrying adsorbed surfactant at the interface (whether through diffusion or increased hydrophobicity resulting from increased adsorption of TTAB), or to a combination of both of these effects. Hunter et al. [14] suggested that there may be three synergistic effects of surfactant on particle-interface stabilisation: Surfactant-particle mixtures may decrease the interfacial tension, surfactant adsorption changes particle contact angle and finally surfactant may affect the particle packing at the interface.

The effect of particle addition is more obvious at the higher TTAB concentration of 1 mM (Fig. 4), where the higher availability of TTAB molecules results in lower surface tension both with and without particles, however the addition of particles leads to a clear additional decrease in the measured surface tension values at all bubble lifetimes.

## 2.2 Effect of Particles and Surfactant on Surface Tension

The effect of particle addition on surface tension was measured for a range of TTAB concentrations from 0 to 0.4 mM. In these experiments, six bubble lifetimes were selected between 0.03 and 60 s, and each measurement repeated

five times to allow estimation of confidence intervals. The surface tension results for the static and stirred systems and the system with particles added (at 4% w/v) are shown in Fig. 5. These figures show clearly that surface tension is reduced further with increasing concentration of TTAB, both with and without particles. For all TTAB concentrations, there is a statistically significant difference in the surface tension between the surfactant only and particulate system.

At the shortest bubble lifetimes, there is little effect of particle addition on the measured surface tension. This suggests that particle diffusion to and attachment at the interface is slower than that of the TTAB molecules. For the longest bubble lifetimes, however, the addition of particles clearly results in a decrease in surface tension. As TTAB concentration increases, the effect on surface tension of particle addition becomes greater, suggesting an increased loading of particles at the bubble surface.

### 2.3 Effect of Particles on Froth Stability

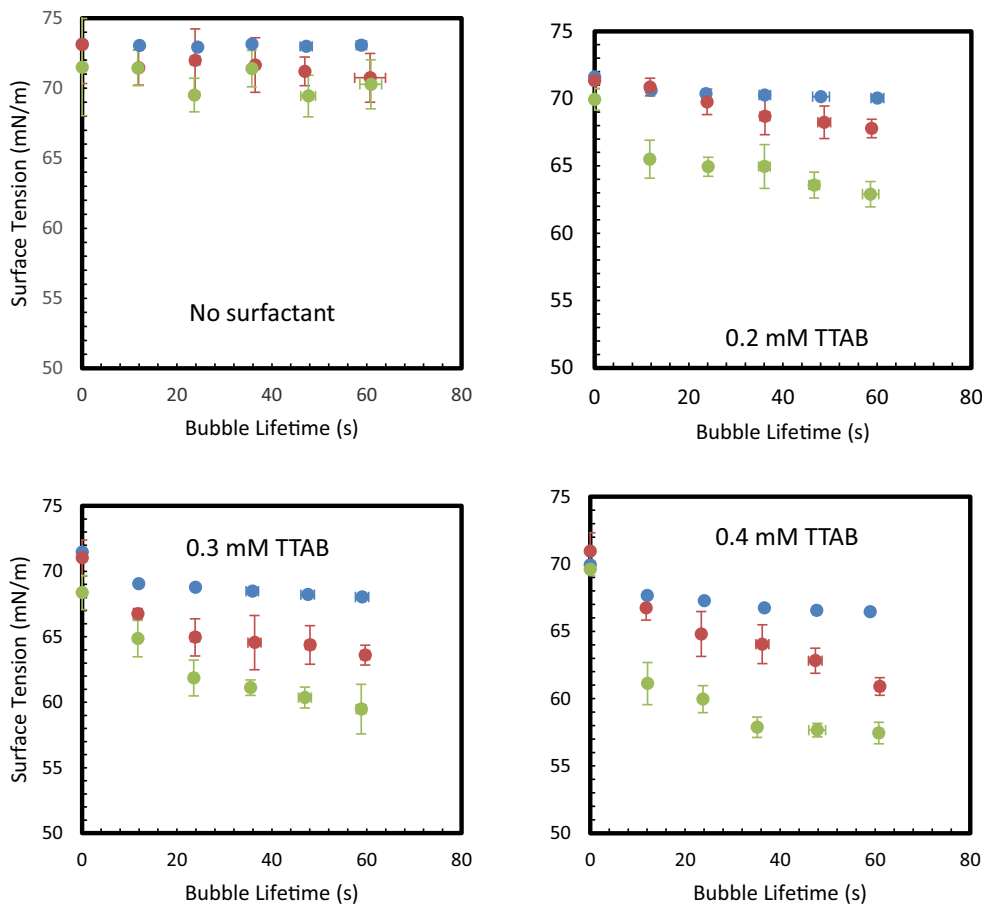
In the previous sections, it was shown that the addition of particles reduces surface tension and that the effect is greater for long bubble lifetimes and with higher TTAB concentration. Dynamic froth stability, as defined in Eq. (1), is determined

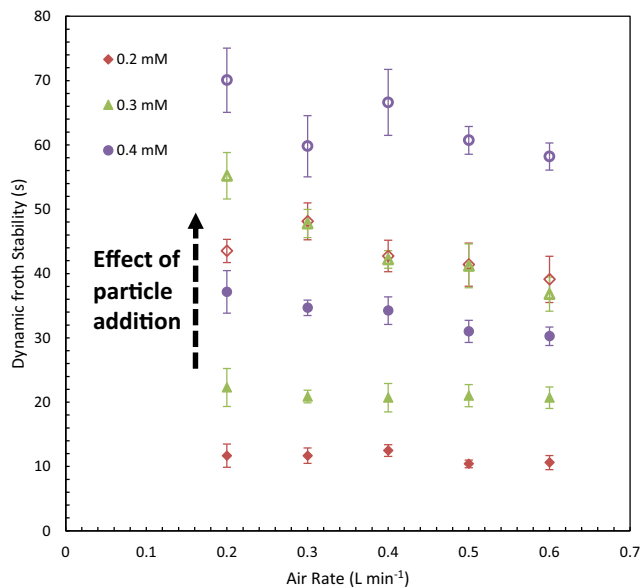
using the maximum froth height, accounting for the inlet air rate. It is affected by air rate, TTAB addition and the presence of particles. This is clear from Fig. 6, where higher TTAB concentrations yield froths of increased stability, both with and without particles in the system. The presence of particles stabilises the froth further, for all experiments.

For the two-phase system, TTAB concentration has a greater impact on the dynamic froth stability than the air rate. This indicates that the diffusion of TTAB molecules to the interface is relatively fast. In contrast, when particles are added, the air rate has a greater effect on the froth stability, which is due to the slow diffusion and attachment of particles to the bubble surfaces and particle loading. It cannot be deduced definitively whether this is the effect of particles at the interface, however this does demonstrate that the presence of hydrophobic particles results in a significant increase in froth stability.

With increasing air flowrate, the attachment of particles at the bubble surface decreases as a result of shorter contact times with the particles in the mixed zone and an increased bubble surface area for attachment. This has also been shown for industrial flotation cells [12]. The results presented in Fig. 6 suggest that increased particle loading, whether through lower air rates or higher TTAB concentration, increases and ultimately determines the dynamic froth stability.

**Fig. 5** Surface tension as a function of bubble lifetime and TTAB concentration where the blue dot is the static system with no particles, the red dot is the stirred system with no particles and the green dot is the stirred system with 4% w/w silica added



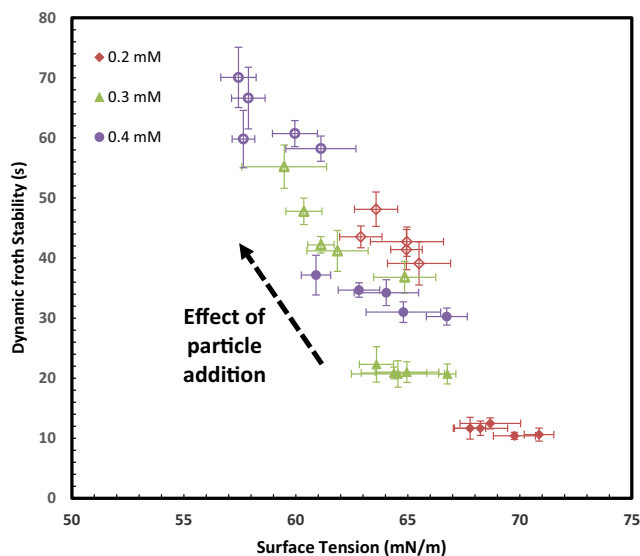


**Fig. 6** Dynamic froth stability ( $\Sigma$ ) as a function of air rate and TTAB concentration; solid markers represent two-phase system, hollow markers represent the three-phase system

## 2.4 Relationship Between Surface Tension and Froth Stability

The data have shown that addition of particles yields lower surface tension and increases froth stability. To demonstrate a direct link between surface tension and froth stability, the effect of air rate must be taken into account. This is measured directly in the froth stability column and from the bubble lifetimes in the surface tension measurements.

Figure 7 shows this comparison, where the five bubble lifetimes (excluding the very shortest), displayed in Fig. 5,



**Fig. 7** Dynamic froth stability as a function of surface tension, where the values are nominal for the surface tension; solid markers represent two-phase system, and hollow markers represent the three-phase system

are plotted against the five air rates shown in Fig. 6. The shorter bubble lifetime ( $\sim 10$  s) is plotted against the highest froth column air rate ( $0.6 \text{ L min}^{-1}$ ). As mentioned previously, the trends are indicative as the exact magnitudes of air rate and bubble lifetime are not comparable.

It is shown clearly that conditions that give lower surface tension generally result in more stable froths. The linear trend is a strong indicator of expected froth stability for a given combination of particles and surfactant concentration and the corresponding effect of air rate variation.

## 3 Conclusions

Flotation froths are stabilised by the attachment of particles to the bubble films. To predict the froth stability for a given combination of particles and surfactants, froth stability experiments can be carried out. In this paper, it has been shown that surface tension, determined using the maximum bubble pressure method, can be applied to three-phase systems and that this can be linked to dynamic froth stability for a given combination of particles and surfactant.

Experiments were carried out using negatively charged silica particles and the cationic surfactant TTAB. It was shown that the addition of particles resulted in a decrease in dynamic surface tension and that this effect was greater at higher surfactant concentrations. One of the benefits of the maximum bubble pressure method is that bubble lifetime can be varied, and this is analogous to air flowrate in a flotation system, such as in the froth stability column. As bubble lifetime increased, the surface tension decreased both with and without particles. Higher TTAB concentrations resulted in a greater difference in surface tension between short and longer bubble lifetimes and between systems with and without particles.

Froth stability experiments were carried out using a column based on the Bickerman design, highlighting the froth stabilising effect of particle addition. The results show clearly that conditions that give lower surface tension result in more stable froths.

The results show that the maximum bubble pressure method for measuring surface tension is unique amongst the available techniques as it allows the effect of bubble lifetime (air flowrate) and therefore attachment of particles at the interface to be investigated. Froth stability can be inferred through surface tension measurements carried out using this method. Furthermore, it has been shown explicitly that reduced surface tension is directly linked to higher froth stability and that it is this mechanism, and the hindering of film drainage, that leads to the froth stabilising effect of particles in systems such as flotation.

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## Compliance with Ethical Standards

**Conflict of Interest** The authors declare that they have no conflict of interest.

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