

# **The Effect of Solids on Gas Holdup, Bubble Size and Water Overflow Rate in Flotation**

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# Abstract

This thesis reports the effects of solids (hydrophobic and hydrophilic) on gas holdup, bubble size and water overflow rate with different frother types in a continuous flotation column setup. Four frothers were investigated: F150, 1-pentanol, 1-heptanol and DowFroth 250. The gas holdup, bubble size and water and solids overflow rate were measured as solids were added to the gas-water system. Solids used were talc (hydrophobic) and silica (hydrophilic). Measurements were taken at fixed froth depth and gas velocity. The following was found when talc was added: F150 - gas holdup decreased while bubble size increased; 1-pentanol - gas holdup increased while bubble size remained the same; gas holdup and bubble size remained the same with 1-heptanol and DowFroth 250. When silica was used as solids, no significant changes were observed in bubble size and gas holdup with all frothers tested. The observed behaviour of gas holdup and bubble size with F150 upon addition of talc is attributed to two factors: adsorption of frother by talc (an indirect effect) and talc promoting coalescence (a direct effect). The observed behaviour of gas holdup and bubble size with 1-pentanol upon addition of talc can be attributed to talc loading and slowing the rise velocity of bubbles which unlike the case with the other frothers, have not reached terminal velocity. Water overflow in 3-phase talc systems with 1-pentanol, 1-heptanol and DowFroth 250 increased with the addition of talc but in the case for F150 it fluctuated around an approximately constant value. Test results seem to show that the water overflow rate ranking for frothers in 2 phase is reversed in 3 phase with talc. Recommendations were made for future work.

# Resumé

L'on rapporte les effets de solides (aussi bien hydrophobiques que hydrophiliques) sur la rétention de gaz, la taille de la bulle, la vitesse d'écoulement du trop plein d'eau, par usage de différents types de moussants dans une colonne de flottation continue. Quatre moussants étaient étudiés: le F150, le pentanol-1, l'heptanol-1 et le DowFroth 250. La rétention du gaz, la taille de bulle et la vitesse d'écoulement du trop plein d'eau et des solides étaient mesurées en même temps que l'on procédait à l'ajout des solides dans le système gaz-eau. Les solides utilisés étaient le talc (hydrophobique) et la silice (hydrophilique). Les mesures étaient prises à des hauteurs de la mousse et à la vitesse du gaz déterminées. Les résultats suivants étaient obtenus lorsque le talc était ajouté: pour le F150; la rétention du gaz avait diminué tandis que la taille de la bulle avait augmenté; pour le pentanol-1; la rétention du gaz avait augmenté alors que la taille de la bulle était invariable; pour l'heptanol-1 et DowFroth 250; la rétention de gaz et la taille de la bulle étaient restées inchangées. Lorsque la silice était utilisée, aucun changement majeur n'était observé, ni pour la taille de la bulle, ni pour la rétention du gaz et ce pour tous les types de moussants testés. Les changements observés pour la rétention de gaz et la taille de la bulle lors de l'utilisation du F150 comme moussant sont attribués à deux facteurs: l'adsorption du moussant par le talc (effet direct) et le talc qui fait la promotion de la coalescence (effet indirect). Le comportement observé dans la rétention du gaz et la taille de la bulle avec l'usage du pentanol-1 est dû à l'accroissement de la masse du talc qui ralentit du coup la vitesse de la bulle, laquelle à la différence des autres moussants, n'a pas encore atteint sa vitesse terminale. L'écoulement du trop plein d'eau dans le système triphasé de talc s'était accru pour le pentanol-1, l'heptanol-1 et le DowFroth 250. Il

fluctuait autour d'une valeur constante dans le cas de F150. Au final, les résultats semblent indiquer que l'ordre des vitesses du débordement d'eau se rapportant aux moussants dans un système biphasé est inversé dans un système triphasé dans le cas du talc. Les recommandations ont été proposées pour des travaux futurs.

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*'Voici mon secret. Il est très simple: on ne voit bien qu'avec le cœur. L'essentiel est invisible pour les yeux.'*

*- Antoine de St Exupéry*





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# Chapter 1 - Introduction

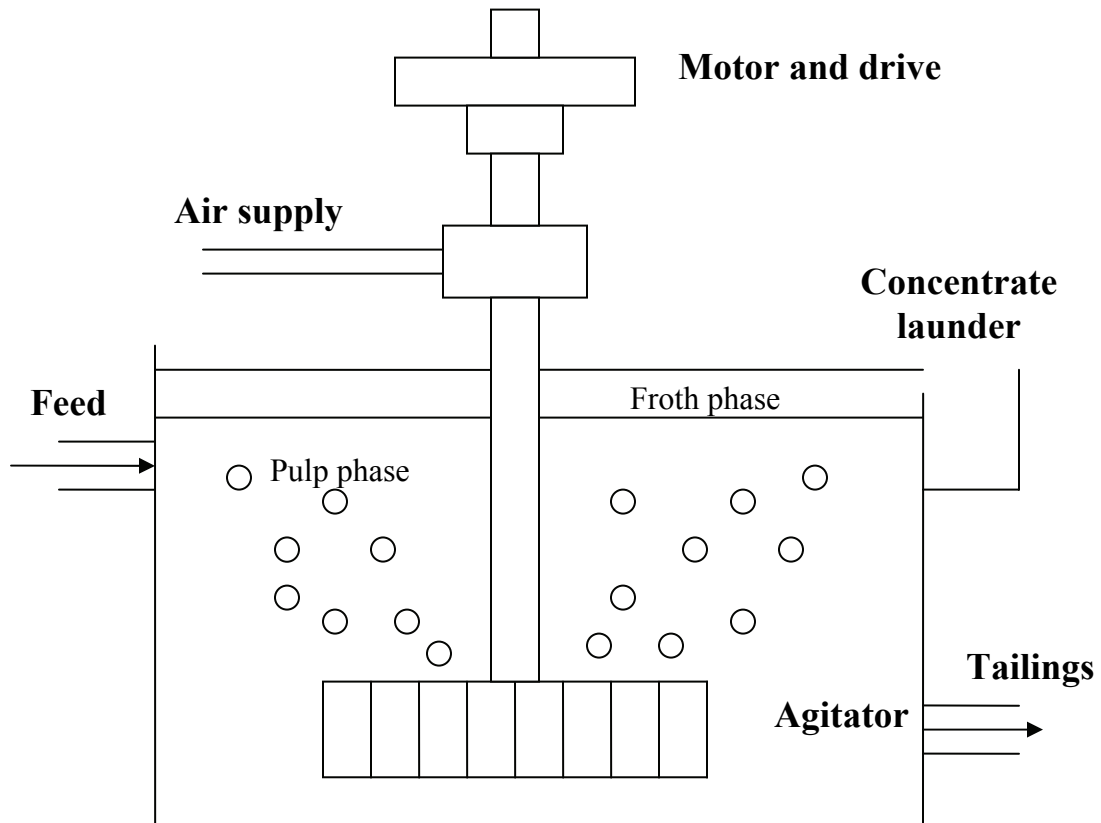
## 1.1 – General Background

Flotation is a physico-chemical separation process that utilises the difference in surface properties of the valuable minerals and the unwanted gangue minerals (Wills et al., 2006). In operation, air bubbles are dispersed into a flotation vessel (cell or column) containing pulp (solid particles suspended in an aqueous medium). Chemical reagents known as collectors are added to the pulp. Selected minerals are made hydrophobic by the collectors and they attach to the rising bubbles in the vessel. Another chemical reagent, frother, is added to stabilize small bubble formation and to create a stable froth. The froth phase is formed at the top of the vessel by the rising bubble-particle aggregates. The process of particle recovery by flotation from the pulp comprises three mechanisms (Wills et al., 2006):

- Selective attachment to air bubbles (or ‘true flotation’).
- Entrainment in the water which passes through the froth.
- Physical entrapment between particles in the froth attached to air bubbles (often referred to as ‘aggregation’).

Figure 1.1 shows the basic components of a flotation cell. The agitator disperses the air into bubbles and distributes them throughout the cell. Hydrophobic particles attach to rising bubbles and are recovered to the overflow concentrate launder via the froth phase.

This thesis focuses on aspects of air dispersion into bubbles, the measurement of properties such as gas holdup and bubble size and the impact of variables such as frother type and addition of solids.



**Figure 1.1 - Schematic of a mechanical flotation cell**

## **1.2 – Effect of Solids on Gas Holdup and Bubble Size in Flotation**

To date, much laboratory-based research has been conducted on gas-water (2 phase) systems. Since one cannot conclude that results in 2 phase systems necessarily apply to 3 phase, ‘surrogate’ solids such as talc, silica and coal have been used. Though simplified relative to natural ores, they provide important revelations about the nature of 3 phase systems in flotation. Despite extensive research on the effect of solids on gas-liquid systems, results are not conclusive and sometimes contradictory. Little is known about

the physical mechanisms underlying the observed macroscopic effects. Also, much of the data is not relevant to flotation systems, which are characterized by mixtures of hydrophilic and hydrophobic particles in the presence of surfactants, notably frothers.

Gas holdup is one of the most important parameters used to characterize the hydrodynamic state of bubble column reactors (Luo et al., 1999). It is useful because it combines the influence of both bubble size and gas rate. It provides a holistic indication of the hydrodynamic conditions because it is dependent on various factors such as frother type and concentration, cell dimensions, operating temperature and pressure, and solid phase properties and concentration.

Though the majority of literature on the topic points to a decrease in gas holdup with increasing solid concentration (Kara et al., 1982; Kelkar et al., 1984; Koide et al., 1984; Banisi et al., 1995; Lu et al., 1995; Reese et al., 1996; Swart et al., 1996; Jianping and Shonglin et al., 1998; Fan et al., 1999; Krishna et al., 1999, Zon et al., 2002), there appears to be no general conclusion regarding the effect of solids on gas holdup, both increasing and decreasing effects being reported based on major studies conducted from 1964 – 1992 as reported by Banisi et. al. (1995).

Of all the major studies, only the work of Banisi et al. (1995) was specifically focused on the flotation process. The work showed that the presence of solid particles reduced gas holdup in a column operated under conditions relevant to flotation. The extent of reduction increased with solids concentration over the range 0 – 15% v/v. Hydrophilic



(silica and calcite) and hydrophobic (coal) particles produced similar reductions in gas holdup. Banisi explored four possible mechanisms responsible for the gas holdup reduction in the presence of solids: coalescence, change in the density and viscosity of the pulp, change in radial gas holdup and flow profiles, and bubble wake effects. It was concluded that changes in gas holdup due to the addition of solids were due to a combination of the two latter mechanisms.

At the time of the work of Banisi et. al., there was no convenient method to measure bubble size. Since then advances in imaging technology have changed the situation. In this study the McGill Bubble Size Analyzer (Hernandez-Aguilar et al., 2002) was employed. In addition, Banisi et al. only used one frother type, DowFroth 250. Moyo et al. (2007) classified frothers into groups based on the relationship between water overflow rate (referred to as ‘carrying rate’) and gas holdup. The strong correlation between water overflow rate and gas holdup based on frother type encourages the opportunity to expand the study of the relationship as solids are introduced into the 2-phase system.

### **1.3 – Objectives of Thesis**

This thesis is aimed at studying and interpreting the effects of solids (hydrophobic and hydrophilic) on pulp properties (gas holdup and bubble size) and froth properties (water and solids overflow rate) with different frother types using a flotation column.

## **1.4 – Structure of Thesis**

Chapter 1 provides the overview of the flotation process, the origin of the vision as well as the objectives of the thesis. Chapter 2 is an in-depth review of related work. Chapter 3 outlines the experimental setup and procedures. The results and observations are presented in Chapter 4. Chapter 5 discusses possible causes and mechanisms of the observed results. Finally, key findings and recommendations for future work are outlined in Chapter 6.

# Chapter 2 – Literature Review

## 2.1 - Gas Dispersion Parameters

Some important hydrodynamic variables that characterize gas dispersion in a flotation cell are: superficial gas velocity ( $J_g$ ), bubble size ( $D_b$ ), gas holdup ( $\varepsilon_g$ ) and bubble surface area flux ( $S_b$ ).

The superficial gas velocity ( $J_g$ ) usually referred to as ‘gas velocity’ is the volumetric flowrate of air ( $Q_g$ ) per cross sectional area (A) of the flotation cell (Equation 1). It is usually reported in cm/s. Depending on factors such as bubble size and slurry rheology,  $J_g$  values range from 0.5 - 2.5 cm/s (Finch and Dobby, 1990).

$$J_g = \frac{Q_g}{A} \quad (1)$$

Bubble size distribution is determined using the McGill Bubble Size Analyzer (Hernandez-Aguilar et al., 2002). Two types of average bubble diameters are typically reported, the number mean,  $D_{10}$  and the Sauter mean,  $D_{32}$ , calculated as follow:

$$D_{10} = \frac{\sum D_b}{n} \quad (2a)$$

$$D_{32} = \frac{\sum D_b^3}{\sum D_b^2} \quad (2b)$$

The Sauter mean diameter is commonly considered the relevant metric in flotation systems (Gorain, et al., 1995). Typical sizes range from 0.5 – 2.5mm (Gorain et al., 1995).

Gas holdup ( $\varepsilon_g$ ) is defined as the volumetric fraction of gas phase in a cell. This is one of the most important parameters used to characterize hydrodynamics of bubble reactors in general. It combines the influence of both bubble size and gas rate, and is dependant on frother type, frother concentration, cell dimensions, operating temperature and pressure, gas distributor design and solid phase properties and concentration.

Bubble surface area flux ( $S_b$ ) is the total surface area of bubbles that passes through a cross section of cell per unit time. It is calculated using the gas velocity and Sauter mean bubble diameter,

$$S_b = \frac{6J_g}{D_{32}} \quad (3)$$

It is commonly used to link flotation rate with the hydrodynamic variables. Under conditions where first order flotation kinetics apply, the flotation rate constant is claimed to be directly proportional to  $S_b$  (Gorain et al., 1997; Hernandez et al., 2001).

## 2.2 - Frothers

Frothers reduce bubble size by retarding bubble coalescence (Harris, 1976). They also enable froth to form that remains stable to overflow into the flotation launder and then break down. Frother molecules are heteropolar, consisting of two groups, polar and non-polar. The non-polar group is a hydrocarbon chain that can either be straight, branched or cyclic. The polar group could be a hydroxyl (OH), carbonyl (-C=O-), ester (-COOR-), carboxyl (-COOH), amine (-NH<sub>2</sub>), nitrile (-CN), phosphate (=PO<sub>4</sub>), or sulphate (=SO<sub>4</sub>) (Wrobel et al., 1953, Booth et al., 1962; Laskowski et al., 1998).

At the moment of bubble generation, frother molecules start to adsorb on the bubble surface oriented with the hydrocarbon chain on the air side and the polar group on the water side. This alters the surface properties promoting the coalescence inhibition effect.

There are three principal frother groups: alcohols, alkoxy type and polyglycols. The alcohol and polyglycol groups are more commonly used in industry. The alcohol group produce relatively shallow froth of low stability and carry little water, i.e., entrain less gangue fines. The common alcohol frother is methyl isobutyl carbinol (MIBC); others sometimes used include pentanol, heptanol, hexanol, octanol, and  $\alpha$ -terpineol (pine oil). Polyglycol frothers produce deeper, more stable froths compared to alcohols, with higher water retention; i.e. tend to entrain more fines and have low sensitivity to pH changes (Riggs et al., 1986). Examples of polyglycol frothers are Dow Froth 200, Dow Froth 250 and F150. Table 2.1 shows the molecular structure and weight of some typical frothers used in industry.

**Table 2.1 - Molecular structures and weight of frothers**

Frother	Structure	Formula	Molecular Weight g/gmol
3 Methyl-1-butanol	Aliphatic alcohol	$\begin{array}{c} \text{CH}_3\text{CHCH}_2\text{CH}_2 \\   \quad   \\ \text{CH}_3 \quad \text{OH} \end{array}$	88.15
1-Pentanol	Aliphatic alcohol	$\begin{array}{c} \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2 \\   \\ \text{OH} \end{array}$	88.15
MIBC	Aliphatic alcohol	$\begin{array}{c} \text{CH}_3\text{CHCH}_2\text{CHCH}_3 \\   \quad   \\ \text{CH}_3 \quad \text{OH} \end{array}$	102.18
1-Hexanol	Aliphatic alcohol	$\begin{array}{c} \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2 \\   \\ \text{OH} \end{array}$	102.18

1-Heptanol	Aliphatic alcohol	$\begin{array}{c} \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2 \\   \\ \text{OH} \end{array}$	116.20
1-Octanol	Aliphatic alcohol	$\begin{array}{c} \text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2 \\   \\ \text{OH} \end{array}$	130.22
Dowfroth 250	Polyglycol ether	$\text{CH}_3(\text{PO})_4\text{OH}$	264.35
Dowfroth 200	Polyglycol ether	$\text{CH}_3(\text{PO})_3\text{OH}$	206.29
F150	Polyglycol	$\text{H}(\text{PO})_7\text{OH}$	425

PO = C<sub>3</sub>H<sub>6</sub>O

### 2.3 - Water and Solids Overflow Rate in 3-Phase Systems

Quinn (2006) studied the effects of salts commonly present in process water on gas dispersion and froth properties compared to a typical frother, methyl isobutyl carbinol (MIBC). Three sets of experiments were run: 2-phase, 3-phase with talc and 3-phase with ore. The 3-phase tests with talc results are examined.

Quinn found that the water overflow rate always exceeded solids overflow rate and increases more rapidly with gas velocity. Figure 2.1 illustrates this point. The water overflow rate exceeds the solids rate and increases more rapidly with gas velocity, to exceed the solids by 6 times (120g/min to 20g/min) at 2cm/s. As a consequence, percent solids in the overflow decreased with increasing gas velocity.

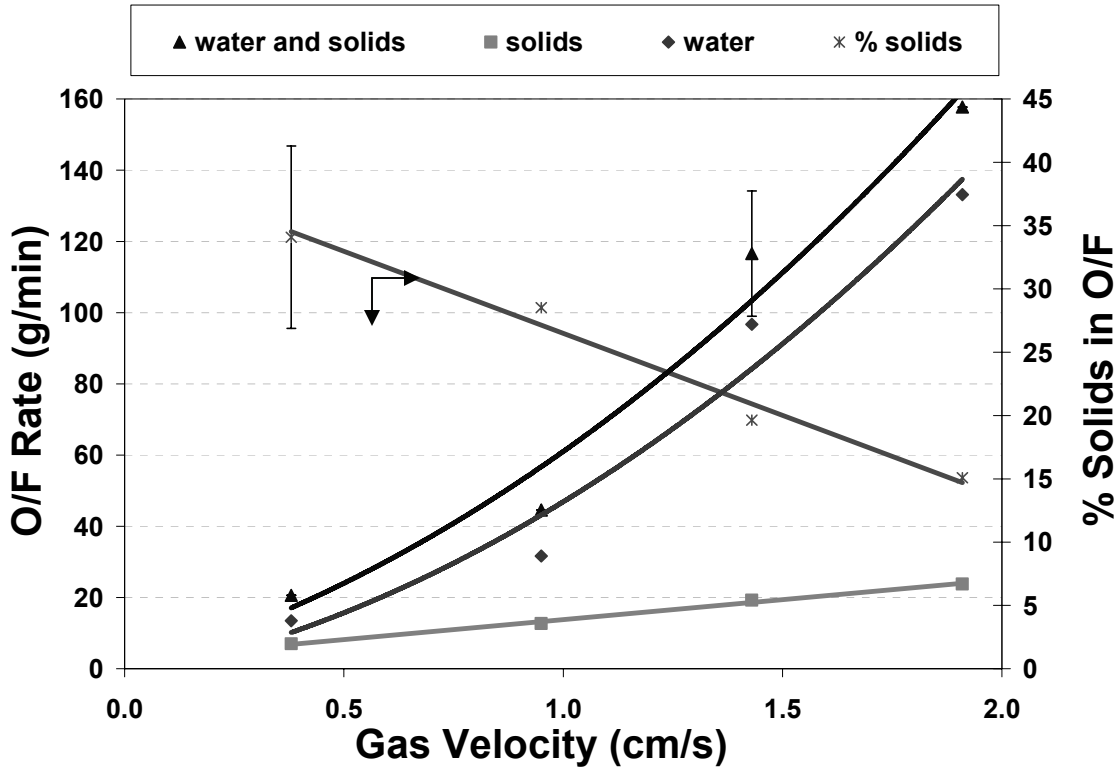


Figure 2.1 – Overflow rates and percent solids for 0.1M NaCl system (Quinn, 2006). Note: solids are talc.

Results show a similar pattern for higher salt concentration (0.4M NaCl, Figure 2.2) and frother solution (15ppm MIBC, Figure 2.3).

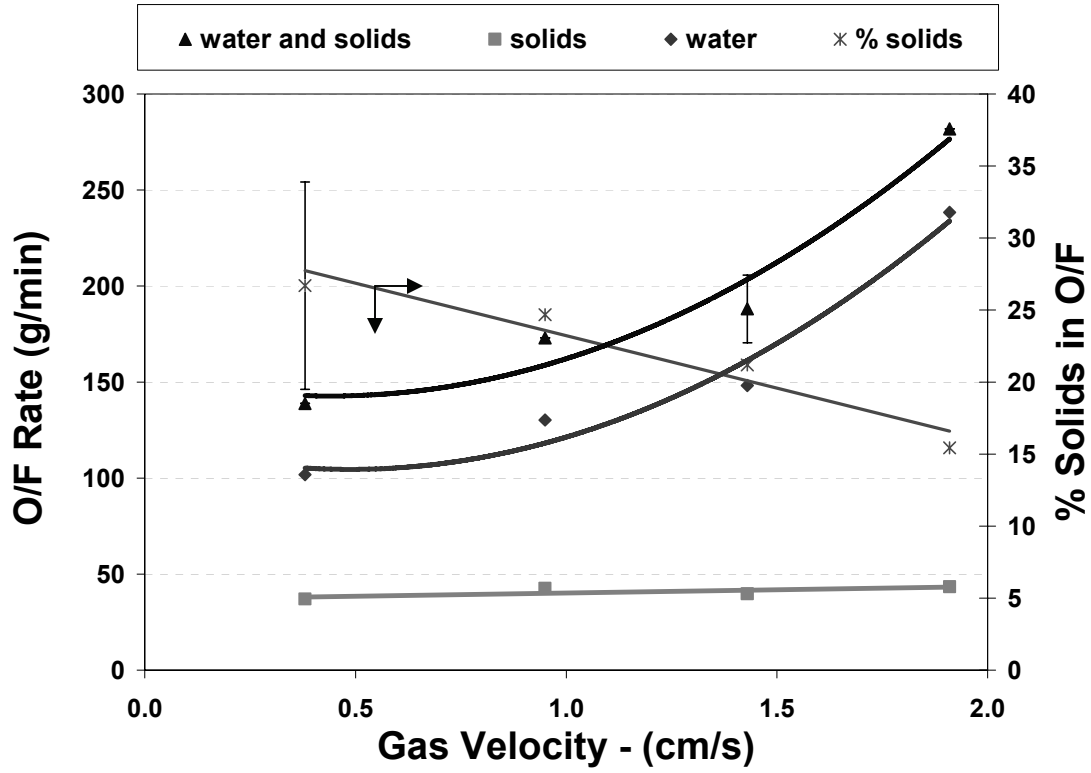


Figure 2.2 - Overflow rates and percent solids for 0.4M NaCl system (Quinn, 2006). Note: solids are talc.



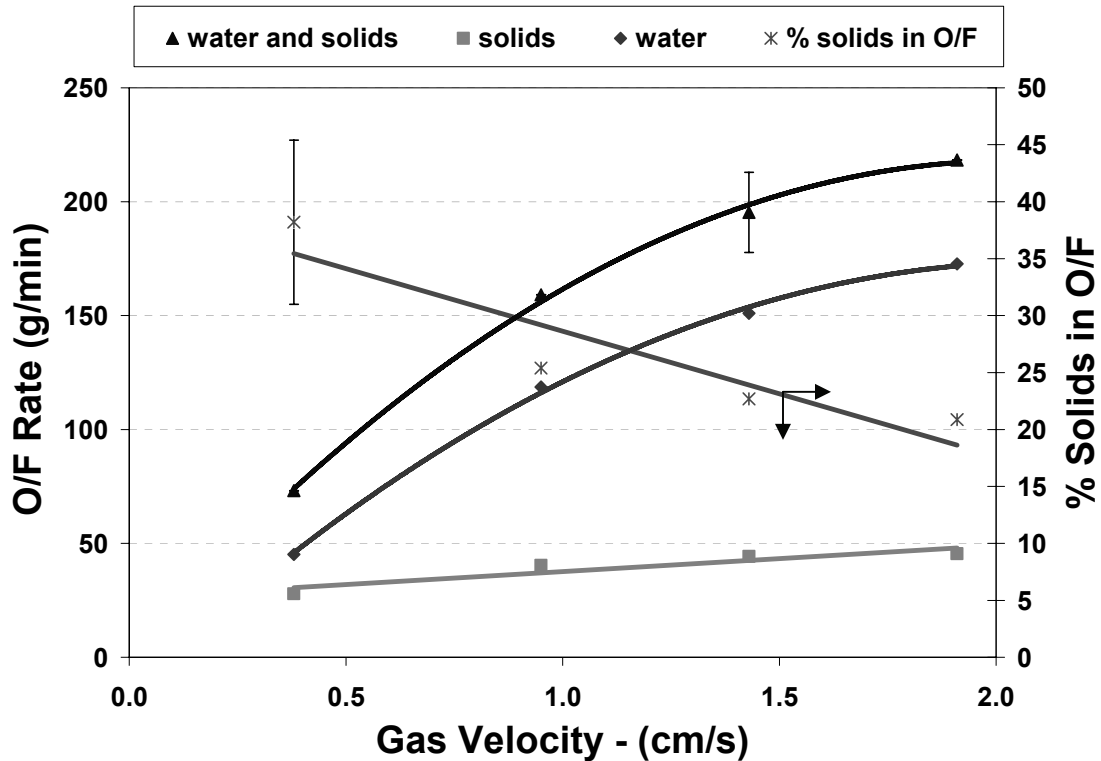


Figure 2.3 - Overflow rates and percent solids for 15ppm MIBC system (Quinn, 2006). Note: solids are talc.

## 2.4 - Effect of Solids on Water and Solids Flotation Rates

Melo and Laskowski (2006) reported that in the presence of bituminous coal, water flotation rates were different than compared to the absence of solids. These results indicated that the amount of water recovered with the froth product depends not only on the properties of the frother but also on the solid particles. Two frothers, diacetone alcohol and MIBC, which gave the lowest water recoveries in 2 phase experiments, generated the highest water flotation rate constants (and highest coal flotation rate constants) in the experiments with coal. DF-1012, the most surface active frother of the tested group, which gave the highest water recoveries in the 2 phase flotation experiments, provided very low flotation rates for water in the flotation experiments with

coal and was the worst agent for flotation of the tested bituminous coal. Melo and Laskowski also found that the presence of fine silica mixed with the coal sample in the feed increased significantly the flotation rate of water and the entrainment of silica into the froth product.

## **2.5 - Correlation of Water Carrying Rate and Gas Holdup**

Moyo et al. (2007) established gas holdup as an independent variable and classified frothers into groups based on overflow rate (referred to as 'water carrying rate'). A comparison of straight chain alcohols (n-alcohols) showed an increase in water overflow rate with carbon chain length (Figure 2.4). When the data were combined, the alcohols appear to classify into 3 groups (Figure 2.5). Comparison of polyglycols revealed that F150 gave the highest water overflow rate while the DowFroths show an increase in water overflow rate with chain length i.e., number of propoxy, PO, groups (Figure 2.6). Figure 2.7 shows all the frothers tested could be classified into four major groups. While an attempt was made to correlate water carrying rate and bubble surface area flux, a relation between the two could not be established. The problem was that at the frother concentration used, bubble size was virtually unchanged between frothers, meaning bubble surface area flux was essentially constant over the range of conditions.

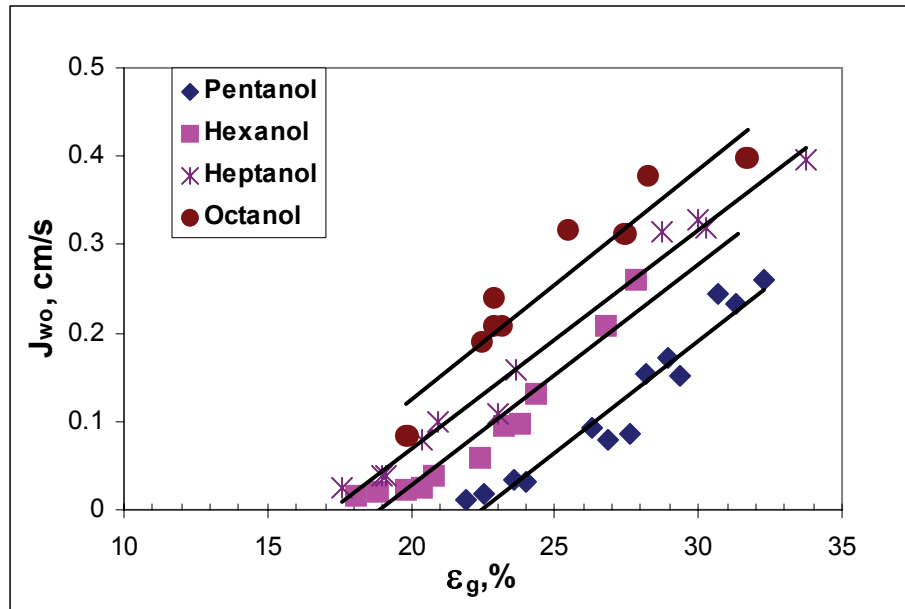


Figure 2.4 – Water overflow ( $J_{wo}$ ) – gas holdup ( $\epsilon_g$ ) relationship: comparison of n-alcohols. Conditions: Froth depth = 7cm (Moyo et al., 2007)

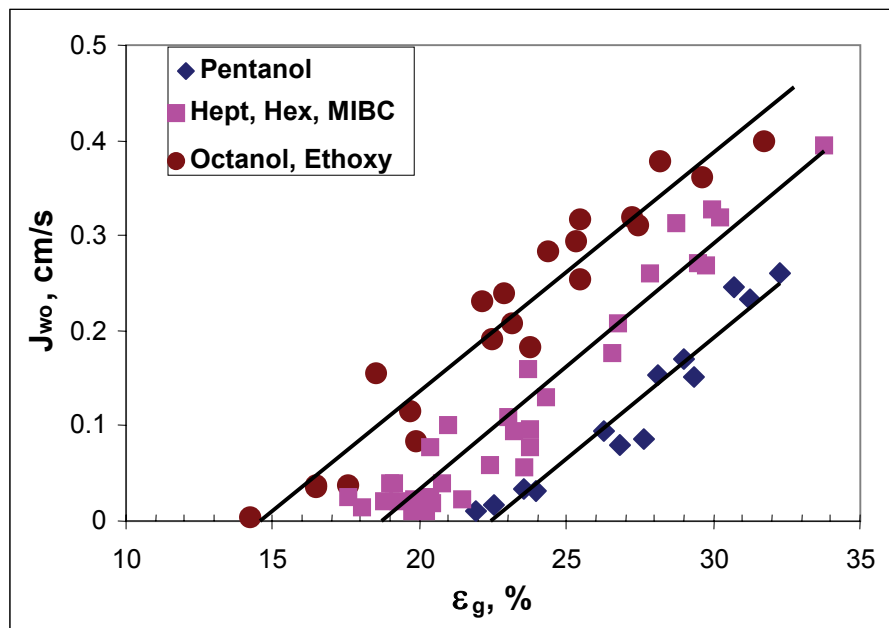


Figure 2.5 - Water overflow ( $J_{wo}$ ) – gas holdup ( $\epsilon_g$ ) relationship: classification of alcohols. Conditions: Froth depth = 7cm (Moyo et al., 2007)

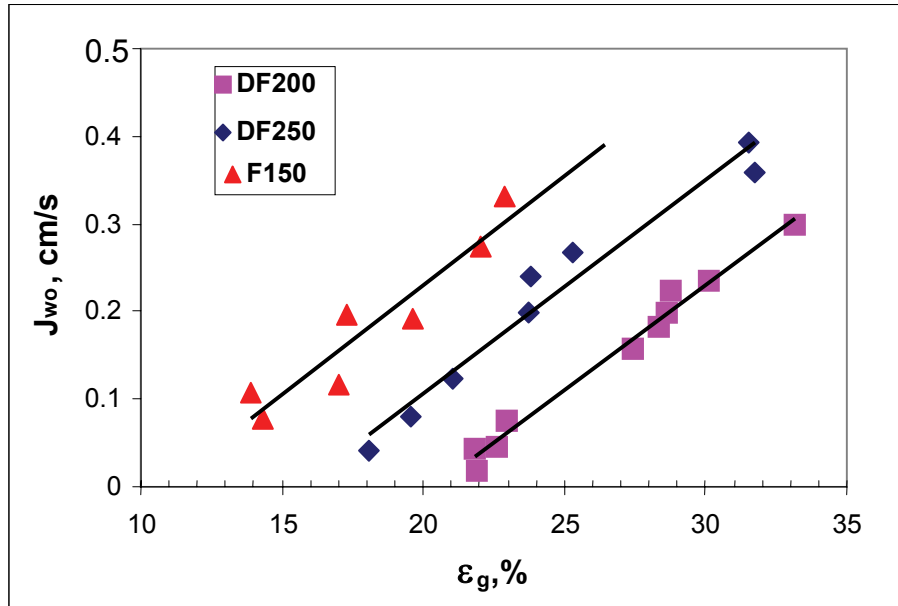


Figure 2.6 - Water overflow ( $J_{wo}$ ) – gas holdup ( $\epsilon_g$ ) relationship: comparison of polyglycol type frothers. Conditions: Froth depth=7cm (Moyo et al., 2007)

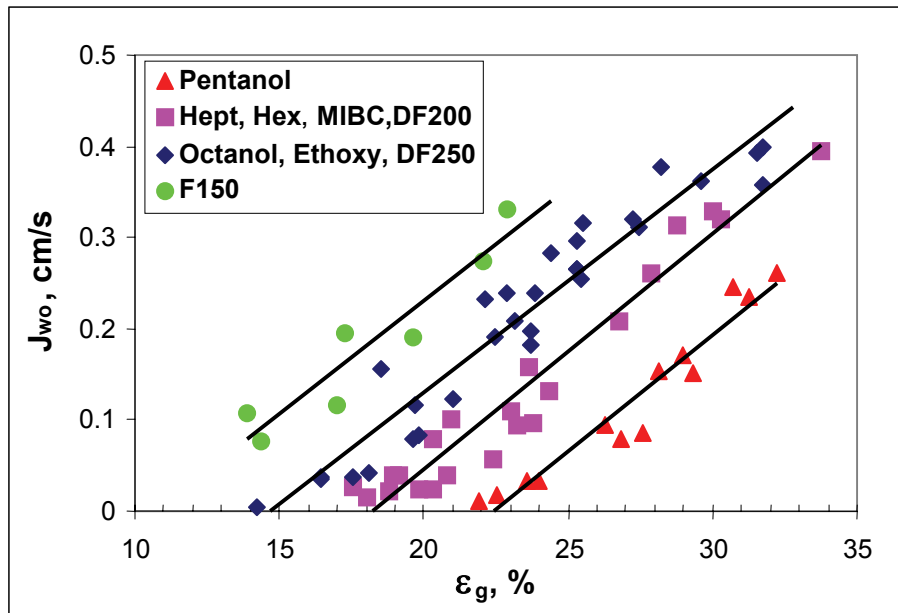


Figure 2.7 - Water overflow ( $J_{wo}$ ) – gas holdup ( $\epsilon_g$ ) relationship: results for and classification of all frothers tested. Conditions: Froth depth =7cm (Moyo et al., 2007)

## 2.6 - Characterizing Frothers Using Gas Holdup

Azgomi (2007) explored a possible way to classify frothers using gas holdup as a surrogate for bubble size. A correlation between frother type and gas holdup was noted: for alcohols, gas holdup increased with hydrocarbon chain length (the effect was the same whether branched or straight chained); for polyglycols, gas holdup increased with number of propoxy groups.

Figure 2.8 shows that gas holdup is related to frother type. The ranking according to gas holdup is essentially the same as that given in terms of bubble size (Sweet et al., 1997; Laskowski et al., 2003), dynamic foamability index (DFI) (Sweet et al., 1997; Laskowski et al. 2003) and water carrying rate (Moyo et al., 2007) as Table 2.2 demonstrates.

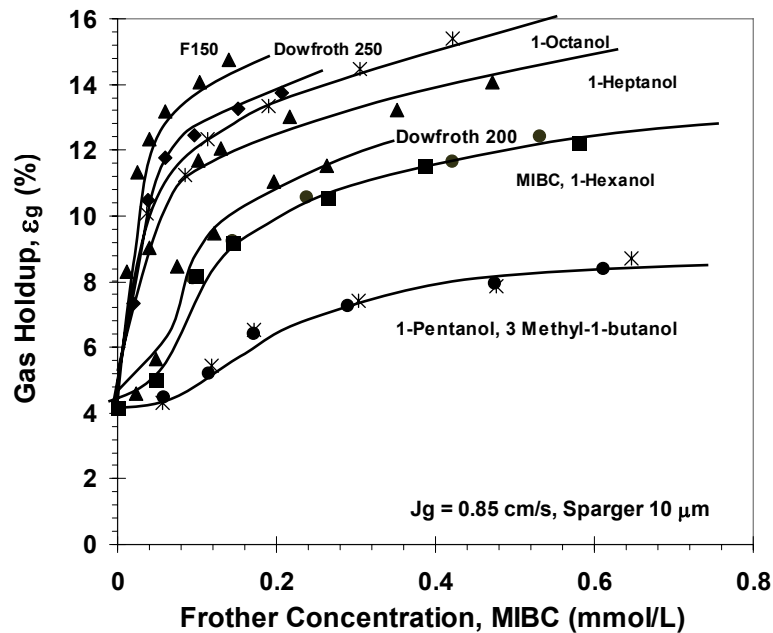


Figure 2.8 - Gas holdup as a function of frother concentration (Azgomi, et al., 2007)

Table 2.2 - Summary of frother rankings

Gas Holdup	Bubble Size <sup>1,2</sup>	Dynamic Foamability Index (DFI) <sup>1,2</sup>	Water Carrying Rate <sup>3</sup>
3 Methyl-1-butanol / 1-Pentanol	1-Butanol	1-Butanol	-
	1-Pentanol	1-Pentanol	1-Pentanol
MIBC / 1-Hexanol	MIBC	MIBC	MIBC
	1-Hexanol	1-Hexanol	1-Hexanol
Dowfroth 200	Dowfroth 200	Dowfroth 200	Dowfroth 200
1-Heptanol	1-Heptanol	1-Heptanol	1-Heptanol
1-Octanol	1-Octanol	1-Octanol	1-Octanol
Dowfroth 250	Dowfroth 250	Dowfroth 250	Dowfroth 250
F150	-	-	F150

<sup>1</sup>Sweet et. al. (1997)

<sup>2</sup>Laskowski et. al. (2003)

<sup>3</sup>Moyo et. al. (2007)

Azgomi examined the correspondence of gas holdup with bubble size. The frother selected was MIBC and the results are in Figure 2.9.

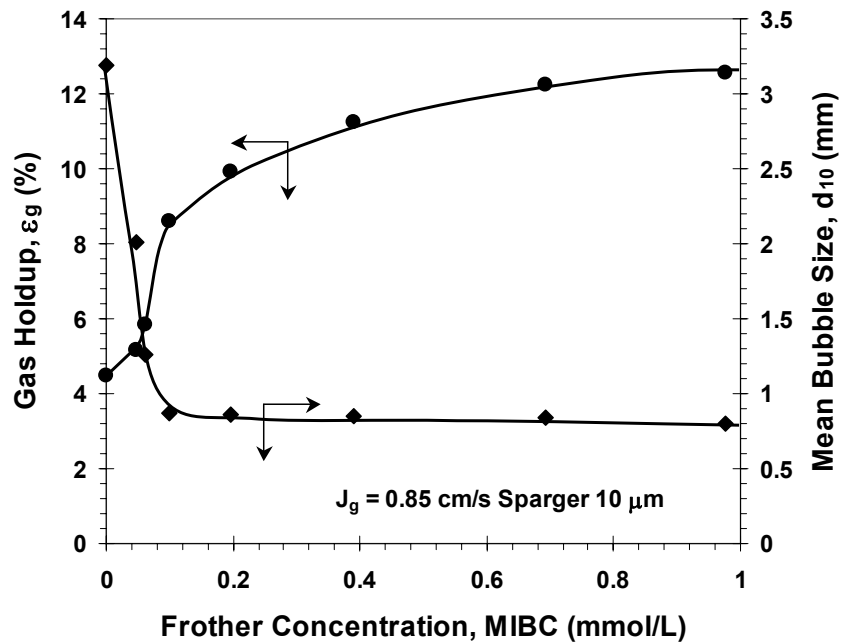


Figure 2.9 - Gas holdup and bubble size vs. frother (MIBC) concentration (Azgomi et al., 2007)

The relatively slow increase in gas holdup at low concentration appears to result from bubbles being greater than ca. 1.5mm where bubble rise velocity is insensitive to size. Above a certain concentration bubble size no longer decreases, called the critical coalescence concentration, CCC (Laskowski, 2003); in the present case  $CCC \approx 0.15$  mmol/L. This is reflected in a slowing in the increase in gas holdup. The continued increase in gas holdup at high concentration appears to result from a continued (if slow) increase in the population of very fine bubbles that is not well reflected by taking a mean value.

## **2.7 - Effect of Solid Particles on Gas Holdup in Flotation**

There are numerous ways that solid particles may affect a gas-liquid system (Mena et al., 2005):

- bubble formation
- bubble rise
- axial and radial profiles
- mixing and dispersion
- mass transfer
- flow regimes

To date, despite extensive research on the effect of solids on gas-liquid systems, results are not conclusive and sometimes contradictory. Little is known about the physical mechanisms underlying the observed macroscopic effects. The majority of literature on the topic points to a decrease in gas holdup with increasing solid concentration (Kara et

al., 1982; Kelkar et al., 1984; Koide et al., 1984; Banisi et al., 1995; Lu et al., 1995; Reese et al., 1996; Swart et al., 1996; Jianping and Shonglin et al., 1998; Fan et al., 1999; Krishna et al., Zon et al., 2002). Decrease of gas holdup infers an increase in the mean bubble rise velocity in the vessel. The faster the bubbles rise, the less accumulation of bubbles in a given section in the vessel and hence, the less volumetric fraction of gas (gas holdup) in the vessel. For this to happen, the existing literature points to several possible causes:

- increase in bubble coalescence caused by the solids which results in bigger, faster moving bubbles (Kato et al., 1972; Banisi et al., 1995).
- reduction of bubble breakup (Gandhi et al., 1999)
- change in mixture density and viscosity (Kara et al., 1982; Banisi et al., 1995)
- radial profiles (Banisi et al., 1995)
- wake effects (Banisi et al., 1995)

Jamialahmadi and Muller-Steinhagen (1991) reported a profound difference between hydrophilic (Styrocel) and hydrophobic (Nylon, Diakon) particles less than 1mm where the former increased gas holdup while the latter decreased gas holdup. Banisi et al. (1995) found no difference in gas holdup between hydrophilic and hydrophobic particles provided frother concentration in solution was maintained the same.

Table 2.3 summarizes that there appears to be no specific trend in the effect of solids on gas holdup; for example, both increasing and decreasing effects were reported based on studies conducted from 1964 – 1992 (Banisi et al., 1995).



Table 2.3 - Major studies on the effect of solids on gas holdup in three phase reactors (Banisi et al., 1995)

Gas holdup in solid-gas-water compared to gas-water	Particle size ( $\mu\text{m}$ )	% Solids (v/v)	Density (g/ml)	Column dimensions ( $\phi \times H$ ) (cm)	Gas flow rate (cm/s)	Investigators and comments
Lower	< 5	2.6-7.7	5.1	50 x 300	2-12	Bukur <i>et al.</i> (1990)
Lower	10	3	3.18	9.5 x 100	0-8	Quiker <i>et al.</i> (1984)
						higher the % solids lower the gas holdup
Lower	20-44	4.6-2.7	2.7	5 x 300	2-12	Bukur <i>et al.</i> (1990)
Lower	45-63	2.2	2.5	7.5 x 300	1-6	Clark (1990)
						continuous operation
Lower	53	20-40	2.5	12.5 x 12.5	2.5	Joosten <i>et al.</i> (1977)
						higher the % solids lower the gas holdup
Lower	63-125	4	2.52	12.2 x 196	2-6	Kato <i>et al.</i> (1972)
Lower	75-250	1-8	2.5	10 x 230	2-12	Koide <i>et al.</i> (1984)
Lower	< 140	6.6	2.7	12.5 x 152	0-6	Ying <i>et al.</i> (1980)
						no effect of further increase in % solids
Lower	100-150	Small	2.55	10 x 200	>4	Imafuku <i>et al.</i> (1968)
						no effect of further increase in % solids
Lower (slightly)	44-265	1.6-6	2.6	7.5 x 265	0.06-2	Shah <i>et al.</i> (1983)
						little effect of % solids
Lower	118-1300	10-30	2.5	7 x 42.5	2-9	Matsumoto <i>et al.</i> (1989)
						dependent on % solids not particle size
Lower	1000-2000	1-5	1.2	7.7 x 128	0-4	Tang <i>et al.</i> (1989)
Lower	2500	0.9-2.7	1.15	15.2 x 122	0-5	Fan <i>et al.</i> (1984)
						higher the % solids lower the gas holdup
Lower	280	2.8-8.6	2.96	10 x 300	0-2	Ostergaard <i>et al.</i> (1966)
Higher	10 <	0.4	2.24	7.8 x 150	2-15	Sada <i>et al.</i> (1986)
						dependent on particle size
Higher	100 <	0.6	4.0	20 x 280	2-22	Khare <i>et al.</i> (1990)
						dependent on particle size and solids
Higher	813	0.08-0.2	1.2	15.2 x 200	1-15	Jamilalahmadi <i>et al.</i> (1991)
						opposite effect for non-wett-able particles
Higher	928	14	2.7	10 x 150	0.5-2	Viswanathan <i>et al.</i> (1964)
Higher	6000	40-55	2.5	65 x 2.5 x 210	0-11	Armstrong <i>et al.</i> (1975)
						opposite effect for non-wett-

One consensus does appear. The trend appears to be an increase in the gas holdup for very fine particles (<10  $\mu\text{m}$ ) at low solids concentration (< 0.6 % v/v) and for very large particles (>2000  $\mu\text{m}$ ) at high solids concentration (> 10% v/v). For intermediate particles

size range (10 – 2000  $\mu$  m) and moderate solids concentration ( $>3\%$  v/v), the effect is a decrease in gas holdup.

Of all the work highlighted in the table, none of the experimental conditions are close to the operating parameters of flotation. Some of those characteristics are:

- Small bubbles ( $< 3\text{mm}$ ) in the presence of frother
- Low gas velocities ( $< 4\text{cm/s}$ )
- Intermediate solids concentration (10 – 15% v/v)
- Small particle size ( $< 100 \mu$  m)
- Mixture of hydrophilic and hydrophobic particles

Banisi approached these conditions using the following:

- 0 -15% v/v solids
- Particle size range 74%  $> 53 \mu$  m to 95%  $< 53 \mu$  m.
- Gas velocity 0 – 2.5cm/s

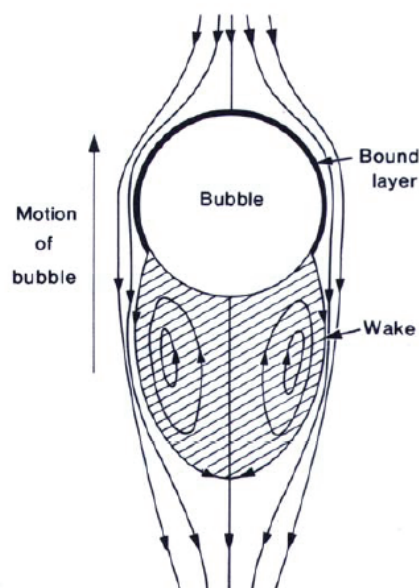
They showed that the presence of solid particles reduced gas holdup in a column operated under these conditions. The extent of reduction increased with solids concentration over the range 0 – 15% v/v. Hydrophilic (silica and calcite) and hydrophobic (coal) particles produced similar reductions in gas holdup.

Banisi discussed the mechanisms of gas holdup reduction in the presence of solids. Four possible mechanisms were explored namely:

- Coalescence
- Change in the density and viscosity of the slurry

- Change in radial gas holdup and flow profiles
- Bubble wake effects

It was shown that the first two cannot explain the holdup decrease but the second two may. It was shown experimentally that the solids used did not cause bubbles to coalesce. Using drift flux analysis, changes in density and viscosity of the slurry due to the presence of solids could not account for the observed reduction in gas holdup. Drift flux analysis did support that bubbles rose more rapidly in the presence of solids if there were changes in radial gas holdup and flow profiles. The bubble wake mechanism proposed that the presence of solids stabilized the wake structure of a bubble due to increased viscosity. This stabilizing effect of the wake increased the velocity of trailing bubbles due to in-line bubble-bubble interaction. This results in the bubbles rising more quickly and thus the gas holdup is reduced. It was proposed therefore that the reduced gas holdup with the introduction of solids is due to a combination of the latter two mechanisms. The following figure from literature shows the wake structure of a rising bubble:



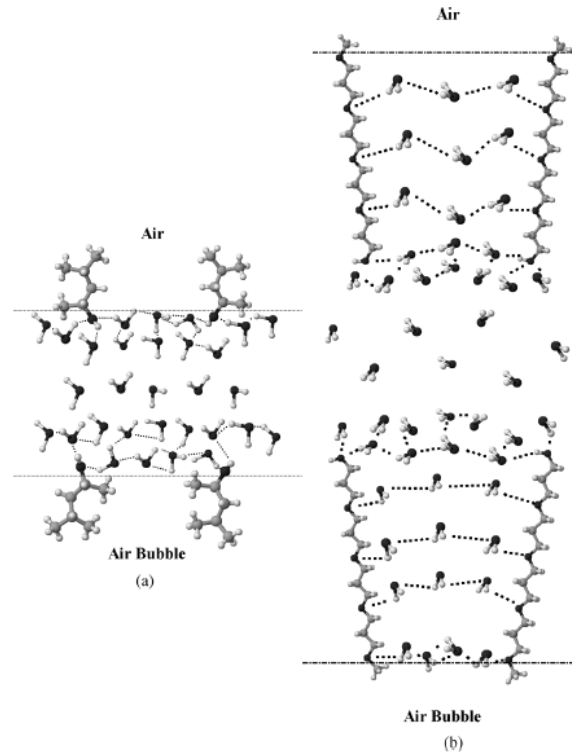
**Figure 2.10 - Ascending bubble with bound water layer and an attached wake (Smith and Warren, 1989)**

## **2.8 - Frother Bubble Thin Films**

Methylisobutyl carbinol (MIBC) and DowFroth 250 bubble thin films were characterized by composition and thickness determinations using FT-IR spectroscopy and UV-visible spectrophotometry (Gelinias et al., 2005). The investigation suggested that the role of frother molecules in creating bubble thin films is based on their effectiveness in inducing H-bonding reorganization of the surrounding free water molecules into a coherent network. The higher stability of a MIBC bubble over a DowFroth 250 bubble was found to be related to the different polarization and orientation of the molecules sustaining the boundary surface water network.

Figure 2.11 shows the hydrocarbon chain of MIBC situated in the air bubble which results in inter-chain van der Waals attraction, strengthening the interfacial film and

increasing the stability of the bubble. A DowFroth 250 molecule is shown (Figure 2.11) situated at the interface with methyl ( $CH_3$ ) group on the air side, the OH group and the polyoxypropylene chains in the water phase.



**Figure 2.11 – Proposed molecular structures of MIBC (a) and DowFroth 250 (b) bubble films (Gelinas et al., 2005).**

Comparable mass concentration results show that fewer DowFroth 250 than MIBC molecules are necessary to create bubble thin films. The free water layer thickness of DowFroth 250 was found to be slightly higher than that of MIBC and this suggests that the bubble carries more water than a MIBC bubble. DowFroth 250 does produce wetter froths and a possible explanation is that DowFroth 250 bubbles have a lower stability than MIBC bubbles and water is released into the froth from the boundary and free water layers as they coalesce.

## **2.9 – Adsorption of Frother on Carbonaceous Material**

Fuerstenau and Pradip (1982) studied the adsorption of three commonly used frothers, namely, terpineol, MIBC and cresol oil on coal surfaces through UV spectrophotometric and gas chromatographic techniques. It was found that adsorption occurs through hydrophobic interactions between the frother molecules and coal surface. All frothers except MIBC exhibited typical Langmuir-type adsorption isotherms. For MIBC, the adsorption isotherm was found to rise linearly with the equilibrium concentration in solution. The adsorption can occur in two ways: displacement of water molecules by the non-polar portion of the adsorbing molecules interacting with the nonpolar surface sites of coal and the adsorption of frother molecules into the pores of coal.

Gredelj et. al. (2008) addressed issues at the Zinifex Century Mine (Australia) such as loss of carbon from preflotation circuit, very high frother (MIBC) consumption and poor selectivity against zinc. They conducted detailed plant and laboratory studies. The work revealed that most of the MIBC added during flotation disappears from solution due to adsorption onto ore particles and especially on fine carbon enriched particles which have high surface area. Frother deportment was determined through measurements of residual frother concentration in different streams. It was found that the amount of MIBC remaining in solution corresponded well with reported measured values in other sulphide plants such as Vale Inco's Clarabelle mill and Rio Tinto's Copperton concentrator of  $\approx$  10ppm and was also close to the MIBC critical coalescence concentration reported by Melo and Laskowski (2006) of 11.2 ppm (indicating that the amount of MIBC remaining in solution is sufficient for formation of small bubbles). Nevertheless, the residual frother

concentrations are extremely low compared to the dosage added ( $\approx 160\text{ppm}$ ). It was confirmed that most of the MIBC added during flotation was removed from solution and adsorption onto carbonaceous material was believed to be the cause. Gredelj et. al. (2008) also found that among the polypropylene glycol frothers (PPG 192<sup>1</sup>, PPG 425 and PPG 1000), PPG of the lowest molecular weight and highest ratio of hydrophilic to hydrophobic groups (i.e., PPG 192) adsorbed at the solid/liquid interface the least and produced the most stable froth. This finding is in contrast with data in the air-water only case reported by Tan et. al. (2005) where both PPG 400 and PPG 1000 produced more stable foams compared to PPG 192. Gredelj et. al. (2008) attributed this to the fact that Tan et. al. (2005) studied frother in solution only, in the absence of particles. Gredelj et. al. (2008) speculated that further decreases in hydrophobicity should further decrease adsorption of frother onto the carbonaceous material and may be sought by decreasing the chain length of the polymer backbone.

## **2.10 – Prediction of Concentrate Mass Flow Rate of Solids**

Gallagos-Acevedo et. al. (2007) modeled the concentrate mass flow rate of solids from a flotation column based on geometrical principles considering first, complete recovery to the concentrate and second, a fraction of the air being recovered due to bursting of a fraction of the bubbles that reach the top of the froth. The experimentally measured concentrate mass flow rate of solids was smaller than the one estimated with the geometrical model assuming full air recovery. To account, bursting of a 0.45 fraction of the bubbles reaching the top of the froth gave improved results. This bursting means

<sup>1</sup>Number refers to molecular weight.

some particles return to the collection zone (i.e., drop back). Solids drop-back was measured as a function of froth zone height, air flow rate and frother addition and results show that drop-back increases with the froth depth and decreases when air flow rate increases. The authors also made an alternative speculation for the difference between the experimental and estimated mass flow rates of concentrate as being due to the equivalent bubble diameter to calculate the bubble surface area flux. According to Bennett et. al. (2002) there exist a segregation of smaller bubbles toward the centre of the column due to the nature of the radial profile of water holdup in a froth. As well, other reasons for the difference considered were that bubbles are not fully loaded and that particles are shared by two bubbles.



## Chapter 3 – Experimental

A 250 cm x 10 cm diameter bubble column was used for the tests (Figure 3.1). A cylindrical porous sparger with a  $40\ \mu\text{m}$  nominal porosity was positioned vertically at the base of the column to disperse air. Three Bailey differential pressure transmitters ( $P_1$ ,  $P_2$  and  $P_3$ ) were used. Two ( $P_2$  and  $P_3$ ) were used for level control and one ( $P_1$ ) was used for gas holdup measurements. They were each positioned at respective heights ( $h_1$ ,  $h_2$  and  $h_3$ ) from the top of the column. The McGill Bubble Size Analyzer (Figure 3.2) was used to capture bubble images (Hernandez-Aguilar et al., 2002). The device was placed on top of the column with the sampling tube in the centre. Bubble images were analyzed and dimensions calculated using available software. Slurry was prepared in the conditioning tank. Solids were added to water and frother was added according to the planned dosages and the slurry conditioned using a mechanical stirrer. The column was operated in continuous mode. The feed pump delivers the feed from the conditioning tank to the column and the underflow pump returns the underflow to the conditioning tank. Water and solids overflow samples were collected at the top of the column with a pail. Timed samples were used to calculate the flow rate. In the 3 phase tests, the slurry in the pail was weighed and filtered using a pressure filter and the cake dried in an oven at  $100^\circ\text{C}$  for about 2 hours. The dry solids were weighed to obtain the solids overflow rate, and by difference, the water overflow rate.

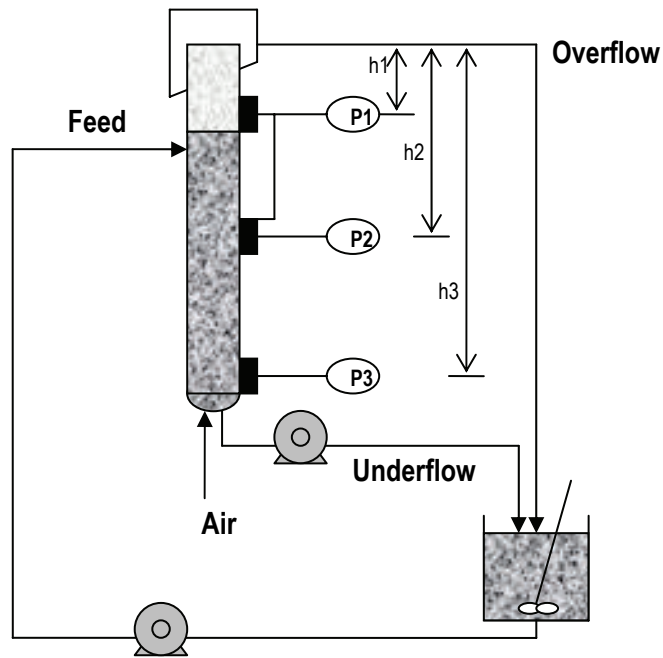


Figure 3.1 - Experimental setup of bubble flotation column (continuous operation)

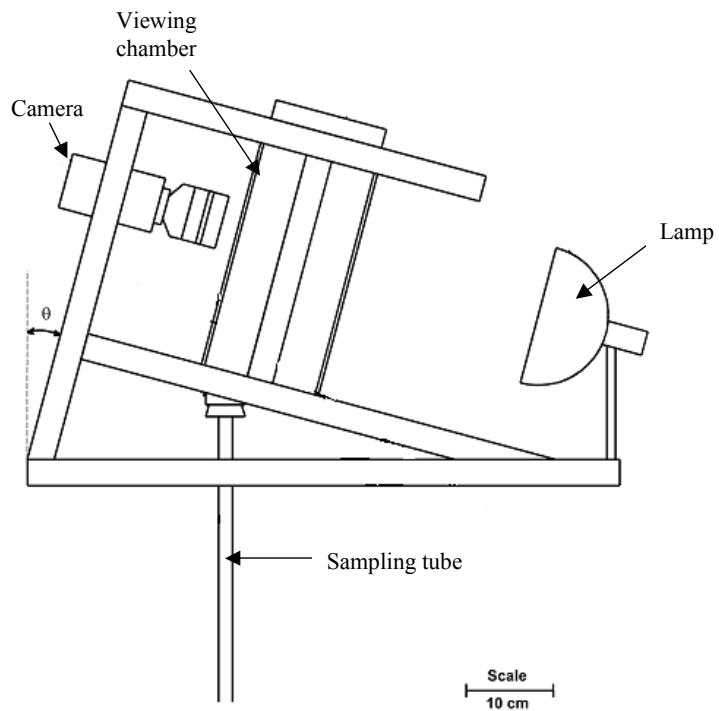


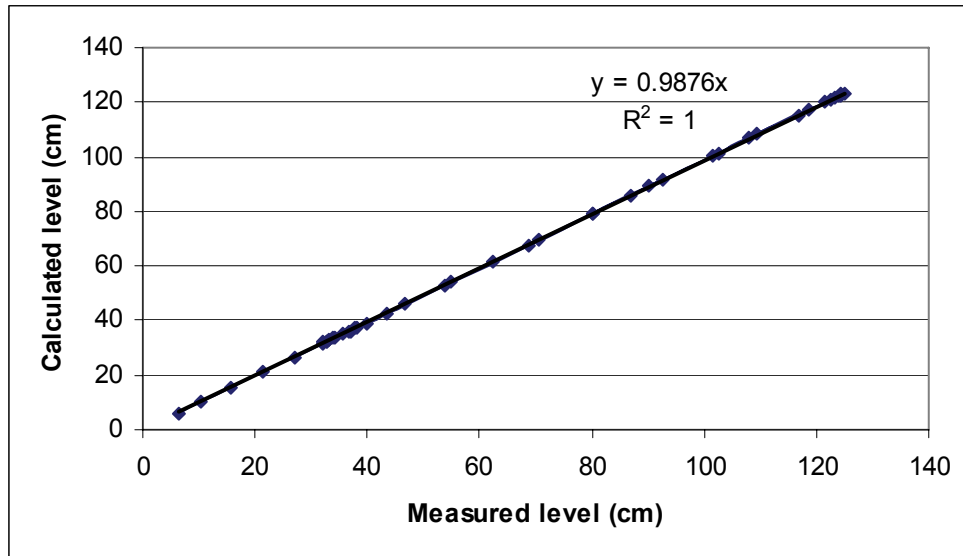
Figure 3.2 - McGill bubble size analyzer.  $\theta$  is the angle of inclination of the viewing chamber,  $15^\circ$  in this case (Hernandez-Aguilar, et al., 2002).

The equation used to calculate level (froth depth) is derived from static pressure considerations (Fernandez, 1995), as follows:

$$Level = \frac{h_2 \left( \frac{P_3 - P_2}{h_3 - h_2} \right) - P_2}{\left( \frac{P_3 - P_2}{h_3 - h_2} \right) - \rho_f} \quad (4)$$

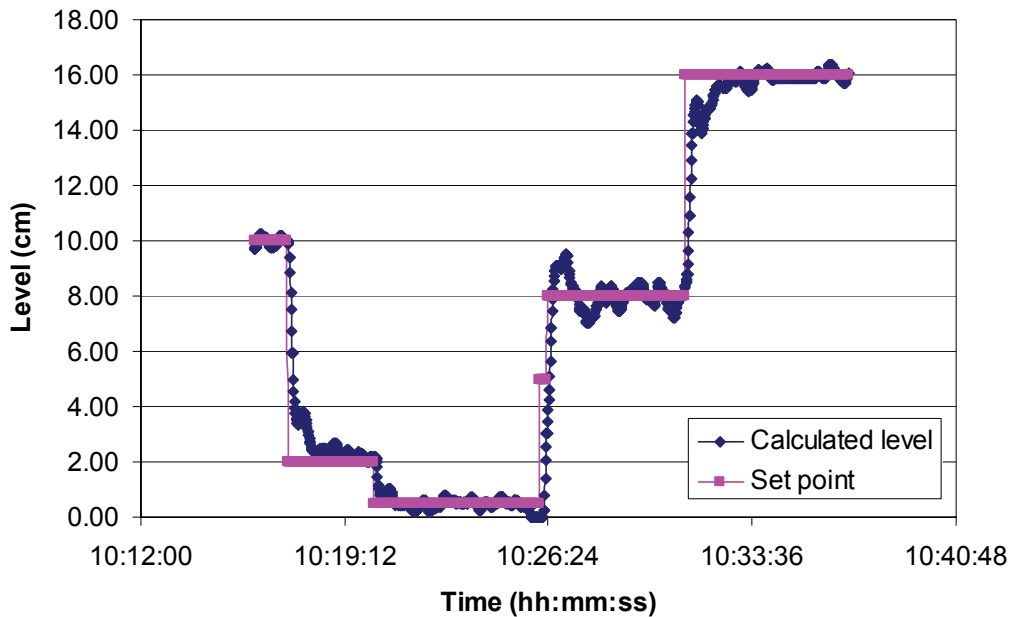
where  $h_1$ ,  $h_2$  and  $h_3$  are the distance of each pressure transmitter to the top of the column,  $P_2$  and  $P_3$  are absolute pressure readings and  $\rho_f$  is the density of the froth which changes according to frother type, frother concentration, percent solids, gas velocity and other factors that affect the nature of the froth. It is assumed as zero for reasons explained later in this section.

The PI control loop function in the software (iFix) was used to control the level (froth depth). The two pressure transmitters ( $P_2$  and  $P_3$ ) send signals to the software which calculates level (Equation 4). As soon as the user keys in the level set point, the control loop manipulates the speed of the underflow pump (i.e., changes the flow rate) so that the calculated froth level nears the set point. Figure 3.3 shows the correlation between the calculated level and the measured level (using a measurement tape) in the air/water system.



**Figure 3.3 – Correlation between calculated level and measured level**

As noted, froth density changes with conditions, e.g., frother type, frother concentration, percent solids and gas velocity. When these parameters change, this introduces an error between the calculated level (Equation 4) and the measured level. Because the conditions do not change within one test, the froth density does not change within that test. It was decided that because of difficulties in measuring froth density, the assumption of zero would be retained. The control loop serves to maintain the level at a constant value. The actual froth level is recorded in every test by measurement with the tape. If the actual level does not reach the desired level, the set point is changed until this is achieved. Figure 3.4 shows the response of the calculated level to changes in the set point.



**Figure 3.4 – Response of calculated level to changes in set point. Average standard deviation is  $\pm 0.13$  cm (allowing a 2 min stabilizing period after every change)**

Air flow rate was set at 4 LPM ( $J_g = 0.82$  cm/s at injection point) and froth level (depth) controlled at 1 cm. Four frothers were chosen for the testwork, two alcohols and two polyglycols: 1-pentanol, 1-heptanol, DowFroth 250 and F150. Table 3.1 show a summary of the properties of the frothers.

**Table 3.1 - Summary of frother properties**

Frother	Structure	Formula	Molecular Weight g/gmol
1-Pentanol	Aliphatic alcohol	$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$	88.15
1-Heptanol	Aliphatic alcohol	$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{OH}$	116.20
Dowfroth 250	Polyglycol ether	$\text{CH}_3(\text{PO})_4\text{OH}$	264.35
F150	Polyglycol	$\text{H}(\text{PO})_7\text{OH}$	425

PO = C<sub>3</sub>H<sub>6</sub>O

The froth depth of 1 cm was chosen to try to minimize the effect of water drainage (from the froth zone to the pulp zone) on the water overflow rate. This provides the opportunity to correlate water overflow rate to bubble size and gas holdup measured in the pulp zone.

Talc and silica was chosen as the solids to be used in this work, as ‘model’ hydrophobic and hydrophilic solids, respectively. Table 3.2 shows properties of the solids used:

**Table 3.2 – Properties of talc and silica**

<b>Solid</b>	Talc	Silica
<b>Synonym</b>	Magnesium silicate hydrate	Silicon dioxide
<b>Chemical formula</b>	$3MgO_4SiO_2H_2O$	$SiO_2$
<b>Molecular weight (g/mol)</b>	166.4	60.08
<b>Specific gravity (g/cm<sup>3</sup>)</b>	2.5 – 2.8	2.2
<b>Average particle size (μ m)</b>	50	3.5

Tables 3.3 and 3.4 summarize the test conditions. They include solids type, frother type, frother concentration and percent solids.

**Table 3.3 - Test conditions with talc as solids**

<b>Solids type</b>	<b>Frother type</b>	<b>Frother conc (ppm)</b>	<b>Frother conc (mmol/L)</b>	<b>% solids</b>
Talc	F150	2.5	0.006	0
				0.2
				0.5
				1
				3
				5
		15	0.035	0
				0.2
				0.5
				1
				3
				5
		25	0.059	0

				0.2
				0.5
				1
				3
				5
	1-pentanol	21	0.238	0
				0.2
				0.5
				1
				3
				5
		35	0.397	0
				0.2
				0.5
				1
				3
				4.2
	1-heptanol	30	0.258	0
				1
				3
				5
	DF250	28	0.106	0
				1
				3
				4

**Table 3.4 - Test conditions with silica as solids**

<b>Solids type</b>	<b>Frother type</b>	<b>Frother conc (ppm)</b>	<b>Frother conc (mmol/L)</b>	<b>% solids</b>
Silica	F150	25	0.059	0
				0.5
				1
				3
				5
	1-pentanol	35	0.397	0
				0.5
				1
				3
				5
	1-heptanol	30	0.258	0
				1
				3
				5
				DF250
	1			
	3			
	5			

Previous tests with talc had shown indication of frother adsorption (Quinn, 2006). Frother adsorption by talc in this thesis was determined using a TOC (Total Organic Carbon) analyzer. Overflow tests were conducted with addition of talc using F150 (2ppm and 25ppm) and 1-pentanol (8ppm and 35ppm). Samples were extracted from the froth zone and pulp zone using a pipette. Frother concentration in each sample was then determined using the TOC analyzer. This was repeated for increasing additions of talc.



# Chapter 4 –Results

## 4.1 - Reproducibility

The sample standard deviation ( $s$ ) was calculated with the following:

$$s = \sqrt{\frac{\sum (x - \bar{x})^2}{(n - 1)}} \quad (5)$$

where  $x$ ,  $\bar{x}$  and  $n$  are the individual measurements, the average and the number of measurements, respectively.

Standard deviations of individual data sets were pooled to represent the standard deviation of the analysis method. The pooled standard deviation ( $s_p$ ) was calculated as follows:

$$s_p^2 = \frac{\sum_{i=1}^k ((n_i - 1)s_i^2)}{\sum_{i=1}^k (n_i - 1)} \quad (6)$$

Pooled standard deviations were calculated for data sets according to frother type at its respective concentration. The 95% confidence interval was then computed as follows:

$$95\% \text{ confidence interval} = \pm 1.96 \frac{s_p}{\sqrt{n}} \quad (7)$$

The error bars shown in this thesis correspond to the 95% confidence interval. Table 4.1 shows the 95% confidence intervals for all the data sets.

**Table 4.1 - 95% confidence levels (all tests)**

<b>Test</b>	<b>Gas holdup, <math>\varepsilon_g</math> (%)</b>	<b>Bubble size, <math>D_{32}</math> (mm)</b>	<b>Water overflow rate (g/min)</b>	<b>Solids overflow rate (g/min)</b>
25ppm F150 with silica	$\pm 0.28$	$\pm 0.01$	-	-
35ppm 1-pentanol with silica	$\pm 1.04$	$\pm 0.07$	-	-
30ppm 1-heptanol with silica	$\pm 0.24$	$\pm 0.06$	-	-
28ppm DF250 with silica	$\pm 0.14$	$\pm 0.01$	-	-
2.5ppm F150 with talc	$\pm 0.13$	$\pm 0.09$	-	-
15ppm F150 with talc	$\pm 0.01$	$\pm 0.06$	-	-
25ppm F150 with talc	$\pm 0.18$	$\pm 0.03$	$\pm 6.80$	$\pm 1.50$
21ppm 1-pentanol with talc	$\pm 0.08$	$\pm 0.02$	-	-
35ppm 1-pentanol with talc	$\pm 0.37$	$\pm 0.04$	$\pm 36.0$	$\pm 3.40$
30ppm 1-heptanol with talc	$\pm 0.43$	$\pm 0.07$	$\pm 39.6$	$\pm 4.60$
28pm DF250 with talc	$\pm 0.32$	$\pm 0.04$	$\pm 18.9$	$\pm 3.00$

#### **4.2 – Effect of Silica on Gas Holdup and Bubble Size**

Gas holdup and bubble size did not show significant change for all frothers as silica was added from 0 to 5% w/w (Figures 4.1 and 4.2). A consistent but slight decrease in gas holdup was observed with F150 at 25ppm but this was not considered significant enough to warrant further investigation.

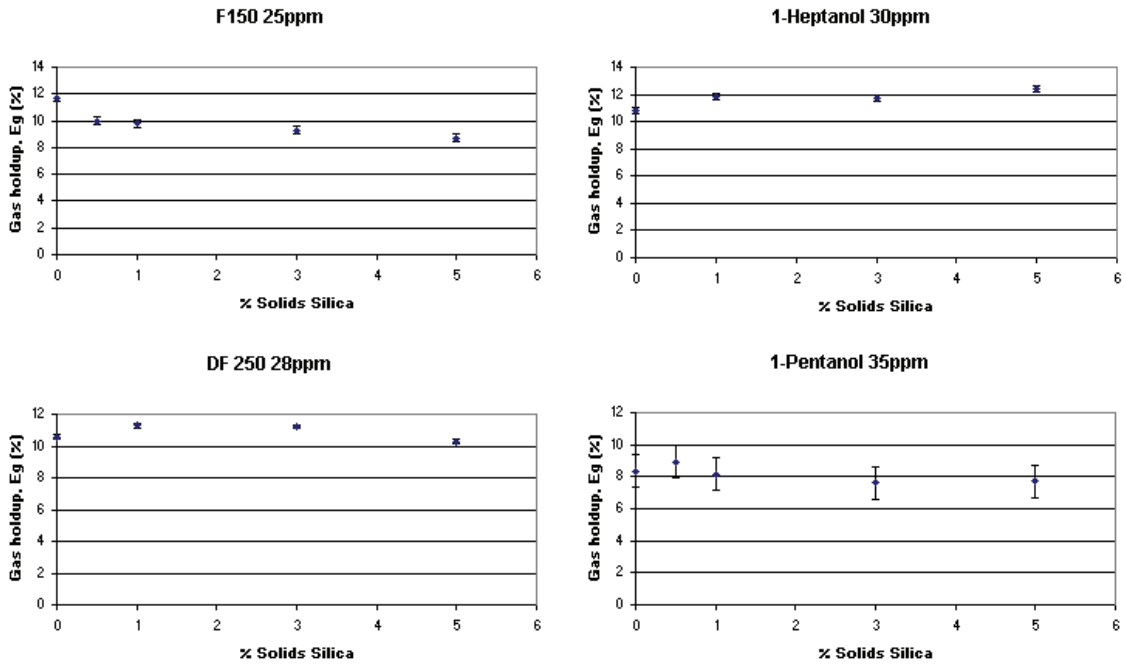


Figure 4.1 - Effect of silica on gas holdup with different frothers

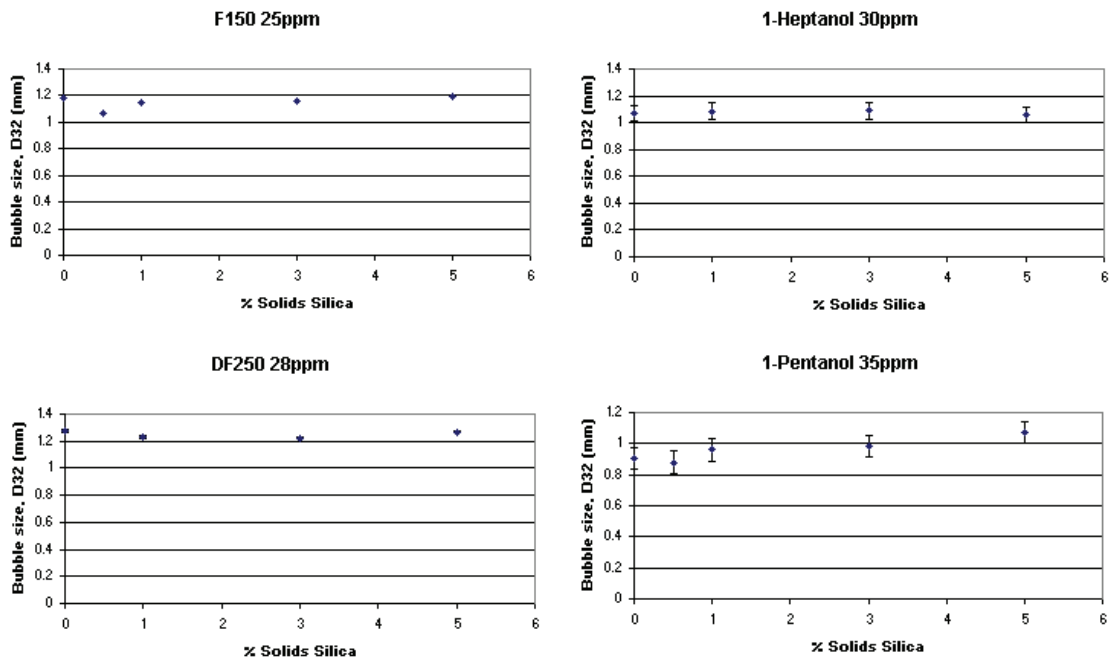


Figure 4.2 - Effect of silica on Sauter mean bubble size with different frothers

Poorer repeatability was found in gas holdup and bubble size measurements when 1-pentanol and 1-heptanol were used compared to the polyglycol frothers. Alcohol frothers are known to be less stable and persistent compared to polyglycol frothers (Rao and Leja, 2004).

### 4.3 - Effect of Talc on Gas Holdup and Bubble Size

It was observed that as talc was added to the gas-water system when F150 was used as frother, gas holdup decreased (Figure 4.3) while bubble size increased (Figure 4.4). This held true across the range of concentration used, 2.5 to 25ppm.

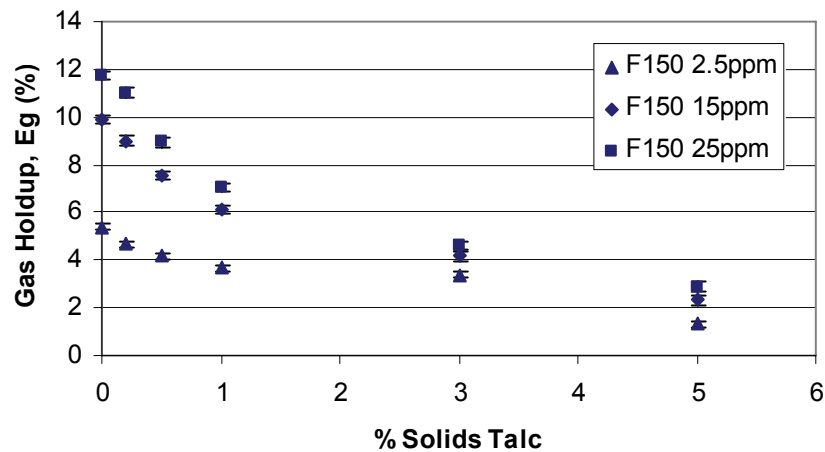


Figure 4.3 – Effect of talc on gas holdup with different concentrations of F150

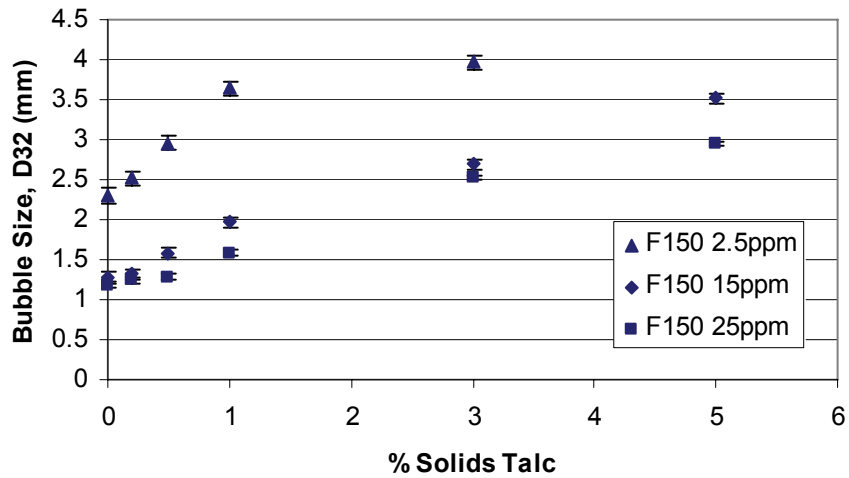


Figure 4.4 – Effect of talc on bubble size with different concentrations of F150

Considering data at 0 % solids (i.e., the air-water system) the results are consistent with the literature: as frother concentration increases, bubble size decreases and gas holdup increases. The impact of solids is notable. For example, at 2.5 ppm F150 the addition of 3% w/w talc increases bubble size to about 4mm (the bubble size in water alone is ca. 4.3 mm in this setup; Quinn, 2007), while at 5% w/w talc the bubbles were now too large and the numbers too few to give a consistent result and so bubble size was not recorded. The difference of bubble size between 2.5ppm and 15ppm F150 is significantly larger than the difference of bubble size between 15ppm and 25ppm F150. This suggests a critical coalescence concentration (CCC) between 15 and 25ppm F150 in the presence of talc.

Examining the individual bubble size distributions, at 25ppm F150 the bubble size distribution was unimodal with a mode at about 0.8mm (Figure 4.5). At 0.2% w/w talc, a mode at a smaller size (0.3 mm) appeared although the Sauter mean bubble size ( $D_{32}$ )

remained almost the same. As more talc was added, this bimodality increased with the mode of the larger bubbles moving to a larger size while the mode of the smaller bubbles remained unchanged from 0.2% w/w talc to 5% w/w talc. At 5% w/w talc, the upper mode increased from ca. 1mm to ca. 3mm and the distribution was more spread. The overall  $D_{32}$  progressively increases as the upper mode increases.

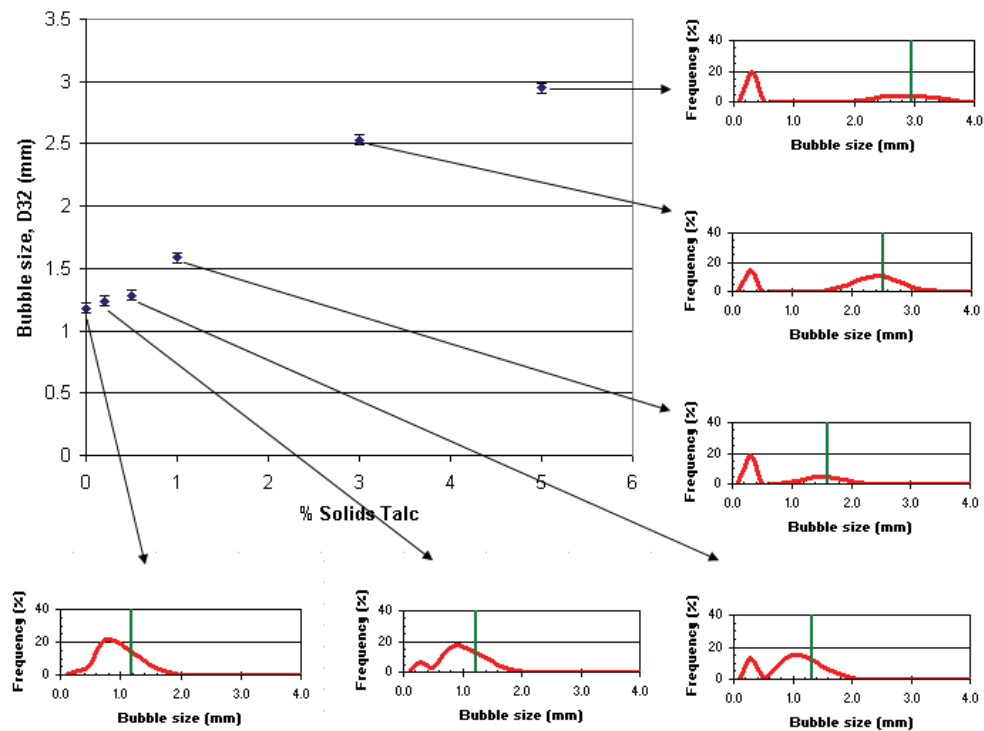


Figure 4.5 - Bubble size distribution as a function of talc addition for F150 25ppm (the vertical line on the distribution is the  $D_{32}$ )

When 1-pentanol was used, different patterns emerged: As talc was added, gas holdup after an initial decrease, increased while bubble size remained essentially the same (Figures 4.6 and 4.7).

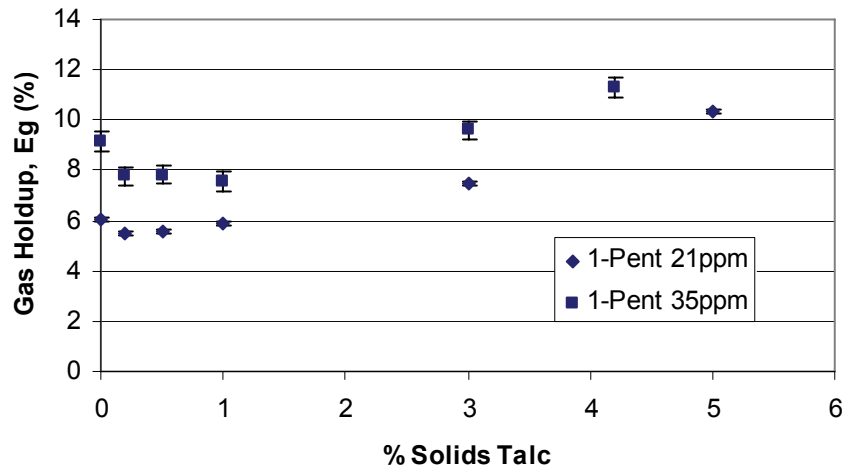


Figure 4.6 – Effect of talc on gas holdup with different concentrations of 1-pentanol

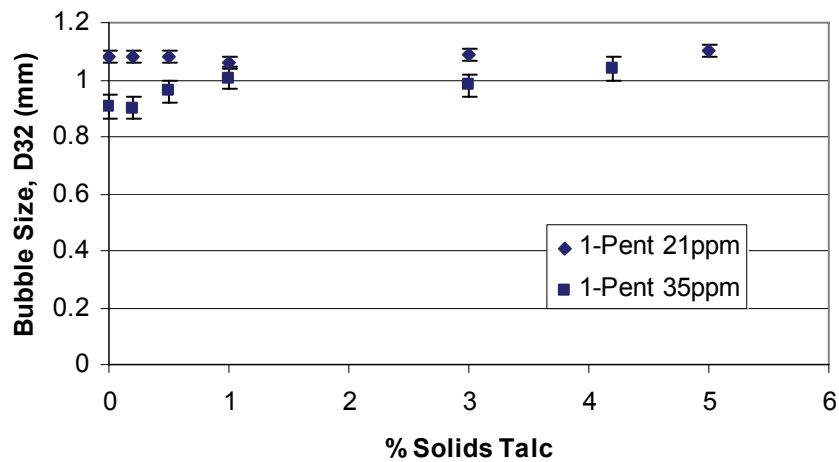


Figure 4.7 – Effect of talc on bubble size with different concentrations of 1-pentanol

At both frother concentrations, the initial gas holdup decrease was about 1% (absolute) with the introduction of up to 1% w/w talc and was followed by a steady increase to ca. 10% as talc was added to 5% w/w. Bubble sizes at both concentrations were close, suggesting a CCC between 21 and 35ppm 1-pentanol.

The lack of change in bubble size also shows in the bubble size distribution (Figure 4.8) with 35ppm 1-pentanol. At 0% w/w talc, the distribution is unimodal and remained so as talc content was increased to 5% w/w.

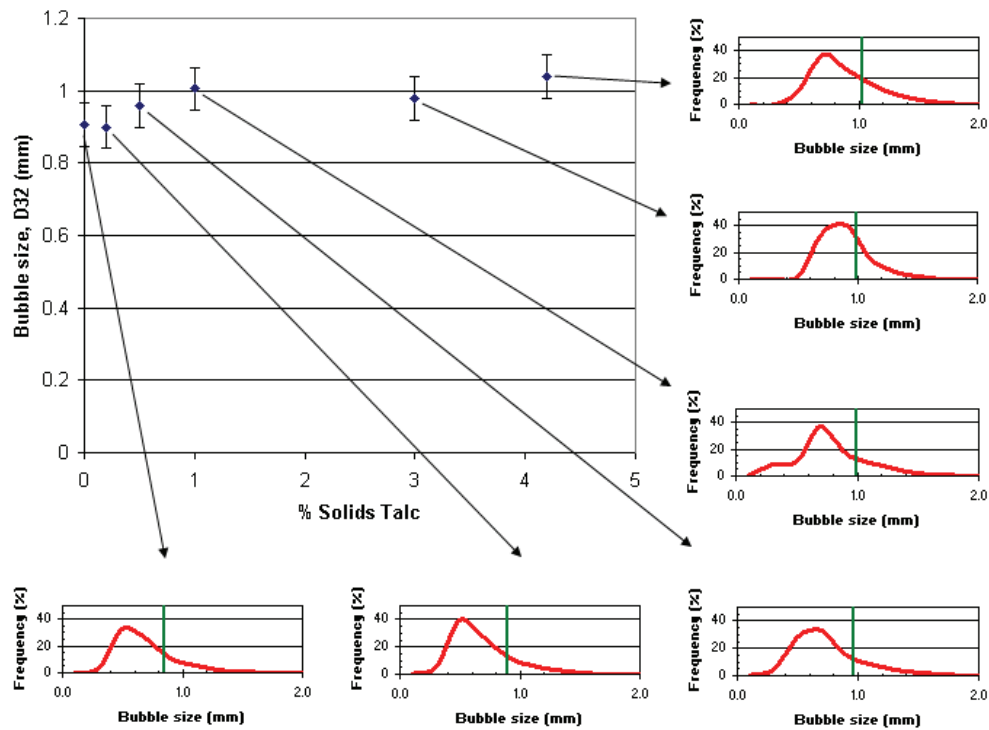
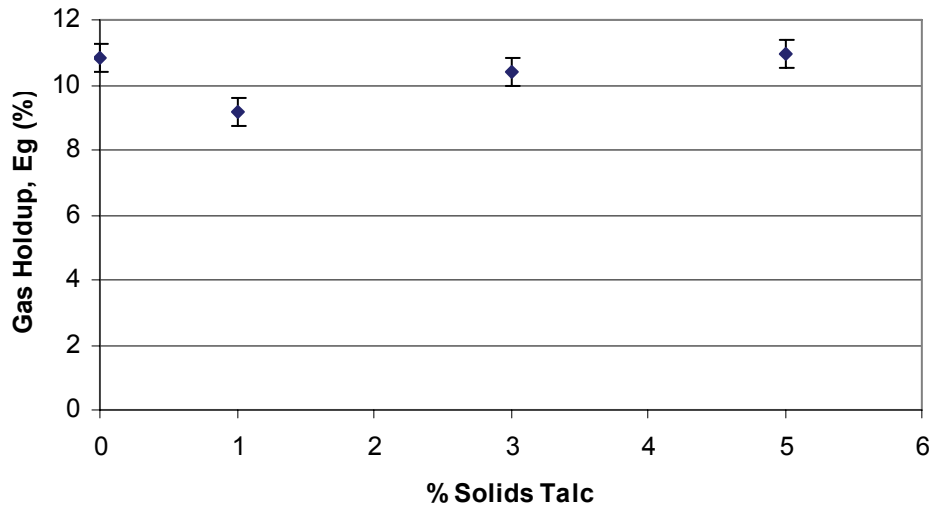


Figure 4.8 - Bubble size distribution as a function of talc addition for 1-pentanol 35ppm (the vertical line on the distribution is the  $D_{32}$ )



The results for the two other frothers, 1-heptanol and DowFroth 250), showed no significant change in gas holdup and bubble size as talc was added (Figures 4.9, 4.10, 4.11 and 4.12). Interestingly, the slight variations in bubble size are mirrored in the gas holdup: for example in 1-heptanol at 1% w/w talc the bubble size increases (compared to 0% w/w) and gas holdup decreases; and for DowFroth 250 at 3 % w/w talc bubble size increases (compared to 1% w/w) and gas holdup decreases. There appear to be some subtle effects but are considered too small to warrant further probing.



**Figure 4.9 – Effect of talc on gas holdup with 30ppm 1-heptanol**

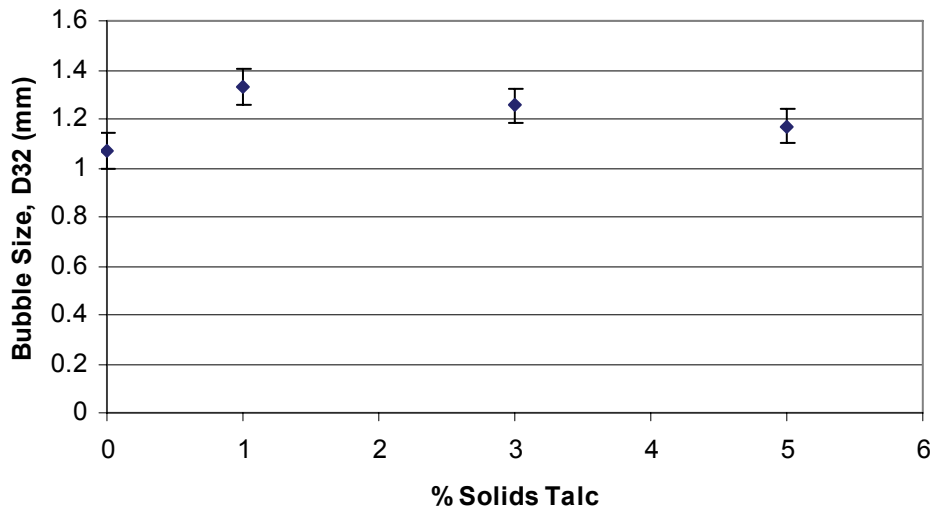


Figure 4.10 – Effect of talc on bubble size with 30ppm 1-heptanol

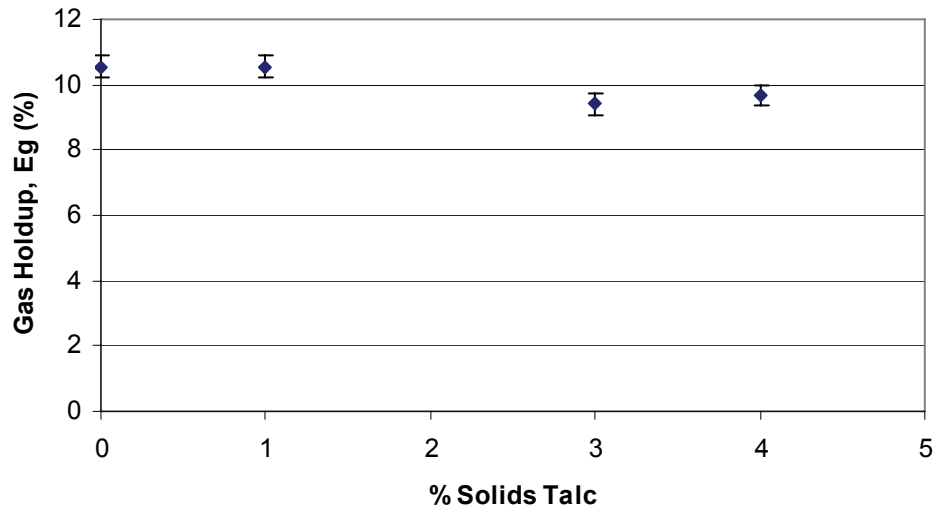
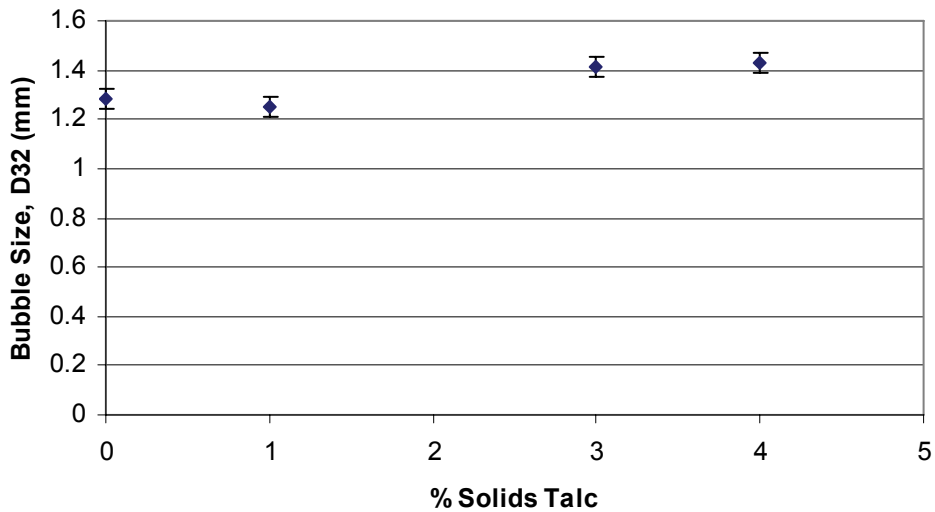


Figure 4.11 – Effect of talc on gas holdup with 28ppm DowFroth 250



**Figure 4.12 – Effect of talc on bubble size with 28ppm DowFroth 250**

As with 1-pentanol, 1-heptanol (30ppm) and DowFroth 250 (28ppm) systems showed no significant changes in bubble size distribution as talc was added up to 5% w/w, remaining unimodal throughout. Their bubble size distributions can be found in Appendix 4.

#### **4.4 - Effect of Talc on Water and Solids Overflow**

Water and solids overflow were measured when 25ppm F150 was used. A fluctuating water overflow accompanied by an initial increase in solids rate to overflow is observed as talc was added. Water overflow fluctuated around 400g/min while solids overflow increases up to 3% w/w talc and then levels off (Figure 4.13):

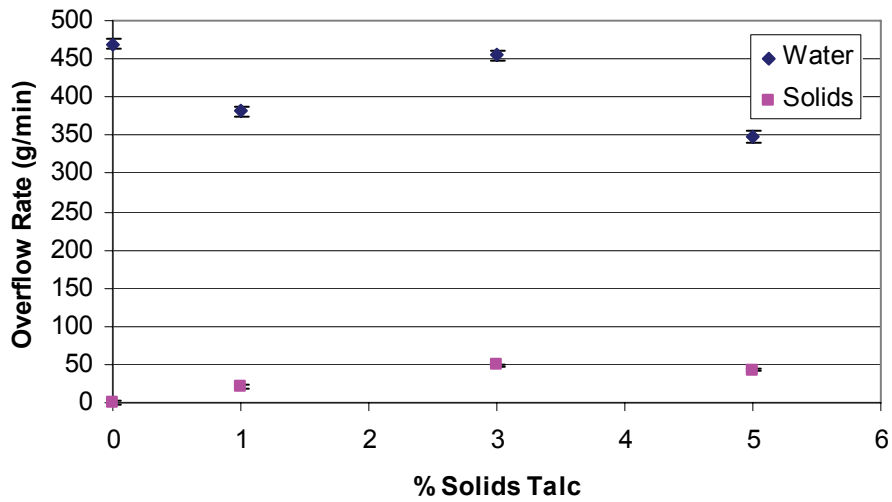


Figure 4.13 – Effect of talc on water and solids overflow rate with 25ppm F150

In contrast, tests with 1-pentanol at 35ppm showed significant increase in water and solids overflow. Water overflow increased to about 7 times its initial value as talc is added up to 3% w/w then decreased slightly (Figure 4.14)

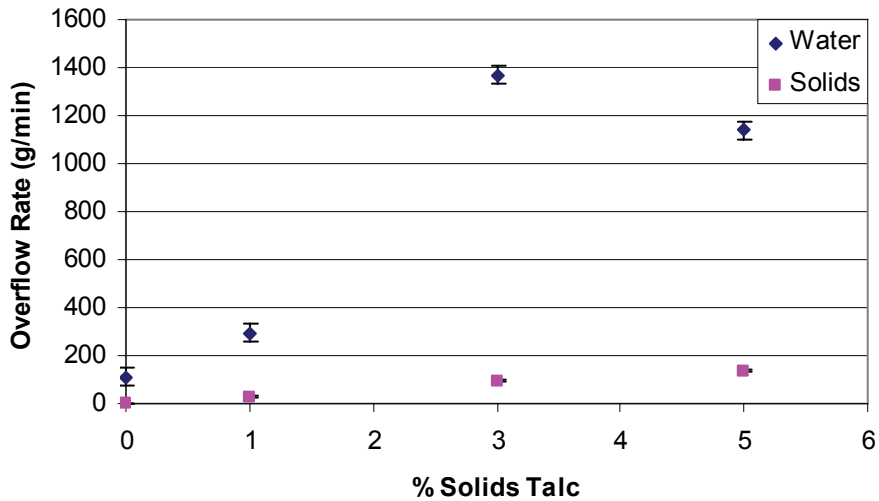


Figure 4.14 – Effect of talc on water and solids overflow rate with 35ppm 1-pentanol

Both 1-heptanol and DowFroth 250 also showed increasing water and solids overflow rates as talc is added (Figures 4.15 and 4.16, respectively).

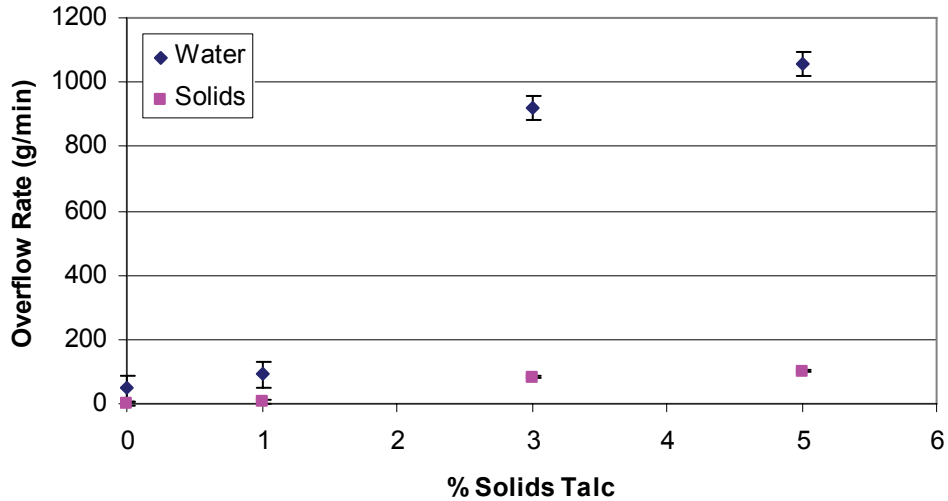


Figure 4.15 – Effect of talc on water and solids overflow rate with 30ppm 1-heptanol

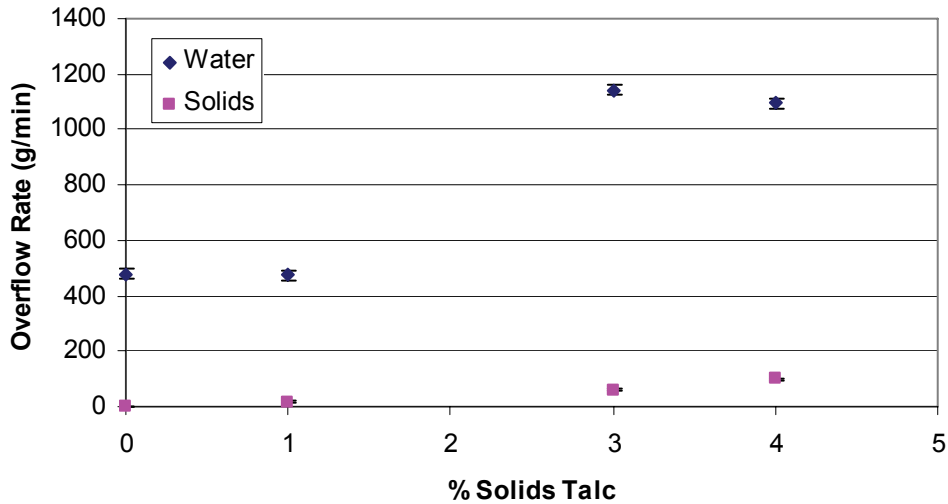


Figure 4.16 – Effect of talc on water and solids overflow rate with 28ppm DowFroth 250

Figure 4.17 compares water overflow rate for 1-pentanol and F150. The water overflow rate in 35ppm 1-pentanol started lower than that in 25ppm F150 at 0% solids but then

increased to almost seven times its initial value at 3% w/w talc to well exceed that with F150 while water overflow in 25ppm F150 did not show any significant change.

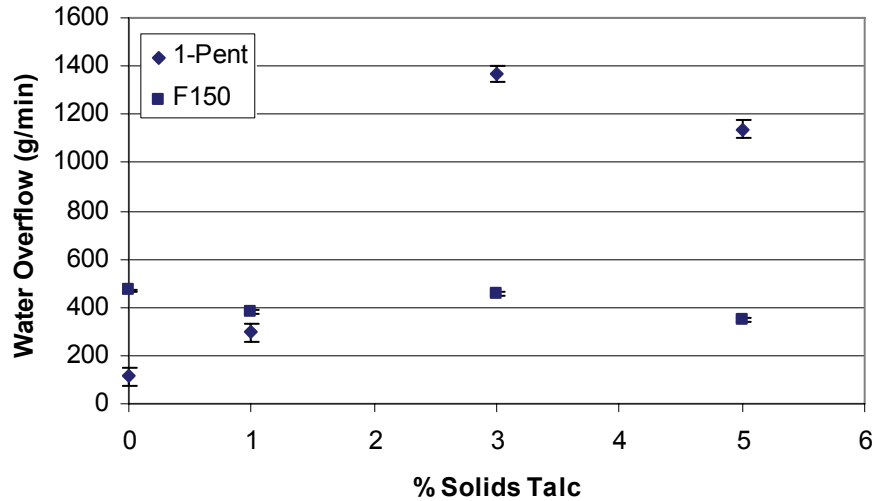


Figure 4.17 – Comparison of water overflow rates in 35ppm 1-pentanol and 25ppm F150

Referring to Figure 4.18, the water overflow rate ranking for frothers in 2 phase is progressively reversed as talc is added up to 5% w/w.

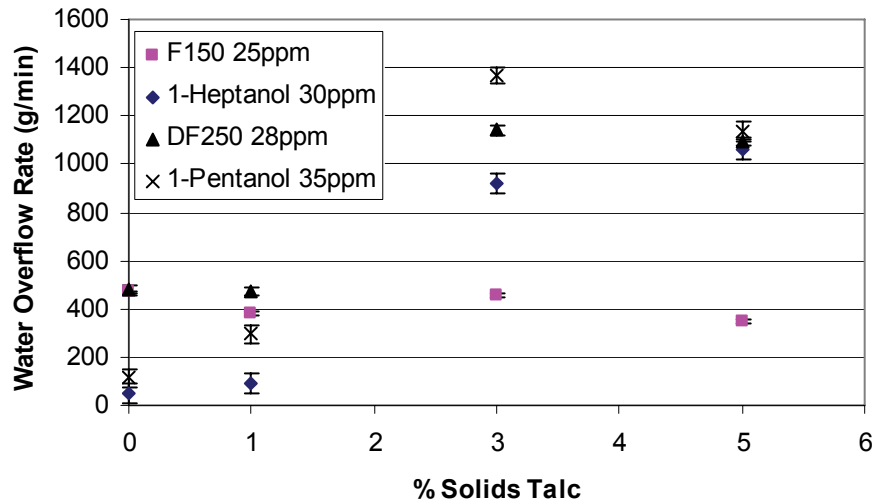


Figure 4.18 – Water overflow rate vs percent solids talc for all frothers

Similarly, the solids overflow rate ranking started off with F150 having one of the higher values at 1% w/w talc but while it leveled off as more talc was added the other frothers steadily increased overtaking F150 at 3% w/w talc. 1-Pentanol emerges with the highest solids flowrate for all the frothers tested, even at the lowest talc addition.

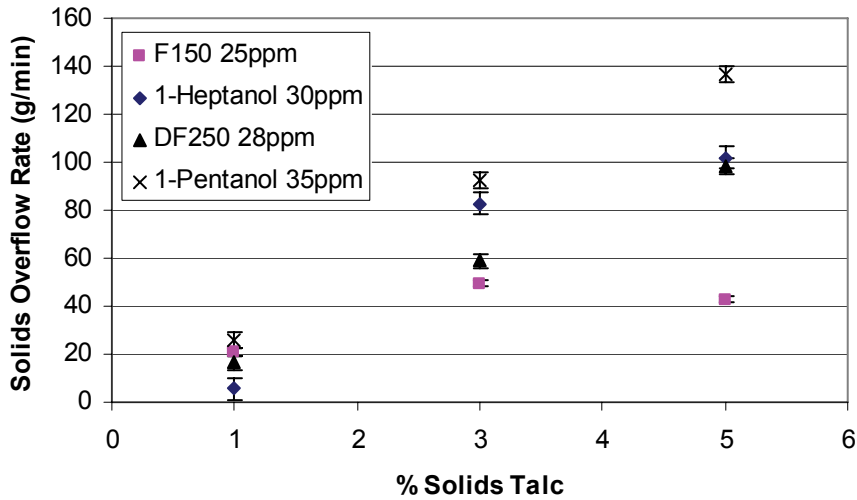


Figure 4.19 – Solids overflow rate vs percent solids talc for all frothers

#### 4.5 – Frother Concentration in Presence of Talc

Figure 4.20 shows the F150 concentrations in the froth and pulp as talc is added to the 2ppm F150 system. A sharp drop can be seen in frother concentration in the froth at 0.5% w/w talc, suggesting frother adsorption by talc although frother concentration in the pulp appears to remain unchanged.

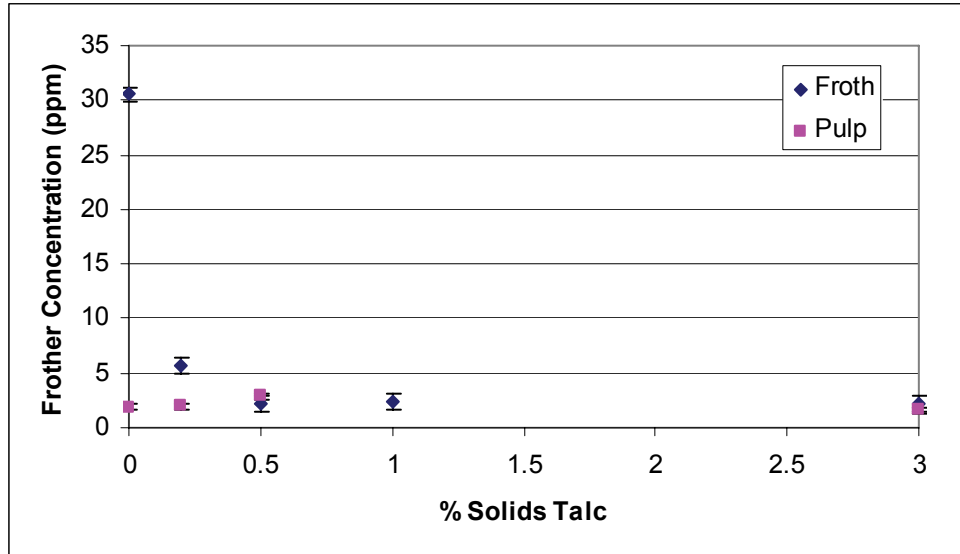


Figure 4.20 – Frother concentrations in froth and pulp with the addition of talc in 2ppm F150

The situation is clearer at 25ppm F150. Figure 4.21 shows the F150 concentration in both froth and pulp decreased consistently with the addition of talc to ca. 7 – 9 ppm at 3% w/w, confirming that F150 is adsorbed by talc.

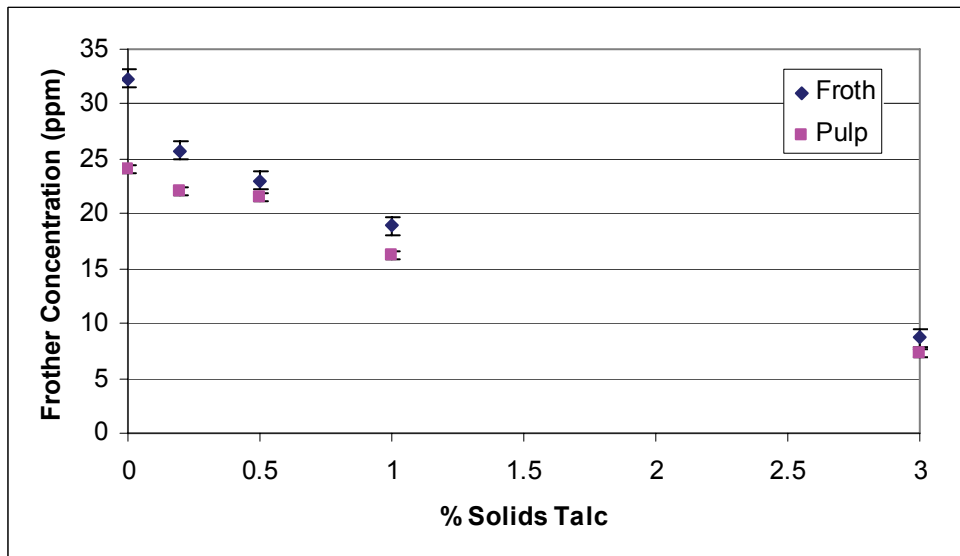


Figure 4.21 – Frother concentration in froth and pulp with the addition of talc in 25ppm F150



In contrast, Figure 4.22 shows the 1-pentanol concentration in the froth and pulp does not decrease as talc is added. In the froth, the values appear to oscillate around 10ppm but in the pulp, values appear to be unchanged ca. 9ppm.

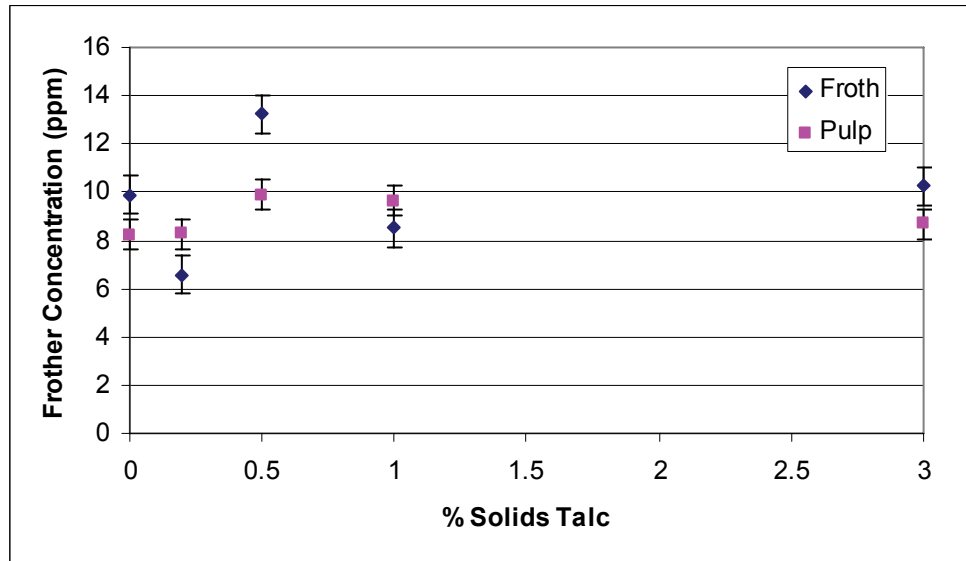


Figure 4.22 – Frother concentration in froth and pulp with the addition of talc in 8ppm 1-pentanol

Figure 4.23 confirms this lack of effect as talc is added to the 35ppm 1-pentanol system; there is variation but no evidence of consistent decrease.

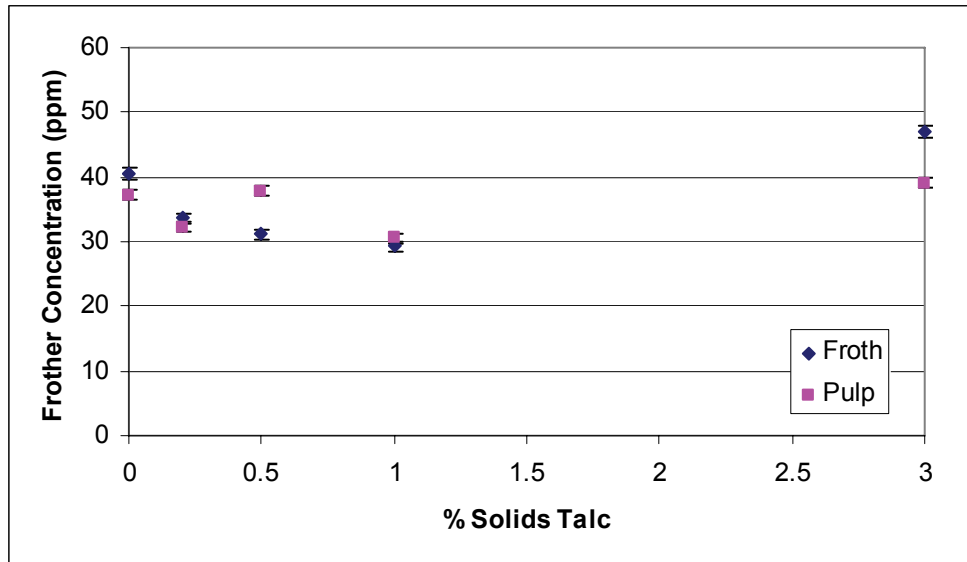


Figure 4.23 – Frother concentration in froth and pulp with the addition of talc in 35ppm 1-pentanol

# Chapter 5 – Discussion

## 5.1 – Pulp Zone

### 5.1.1 - F150 and Talc

Of all four frothers, when talc (a naturally hydrophobic solid) was added, only F150 showed a significant change in both bubble size, which increased, and gas holdup, which decreased. For 1-pentanol, the bubble size remained the same while gas holdup increased upon addition of talc, while for 1-heptanol and DowFroth 250 there was no impact on either property.

Larger bubbles rise faster in the column; the higher average rise velocity, the less the accumulation of bubbles in the column and thus, a lower volumetric fraction of gas; i.e., a lower gas holdup. This correlates with the results using F150 where a progressive decrease in gas holdup correlates with a progressive increase in bubble size upon addition of talc (Figures 4.3 and 4.4). Observing the bubble size distributions, in the presence of 25ppm F150 without talc the distribution was unimodal with mode ca. 0.8mm (Figure 4.5). As talc is added, a second smaller mode appears at ca. 0.3mm while the original mode increases. The question is, how does talc increase the bubble size and alter the distribution when F150 was used as frother?

The apparent answer is that, alone of the frothers, F150 is adsorbed to some extent by the talc (Figures 4.20 and 4.21). This would tend to return the bubble size distribution towards that in water alone, which is typically bimodal with  $D_{32} \approx 4 - 5\text{mm}$  in this setup (e.g. Quinn, 2007). But, Figure 4.21 indicates there is still about 8-10ppm of F150 left in

solution after adsorption by talc at 3% w/w. This concentration is above the CCC of F150 and in water alone, through current and previous experience, is enough to produce small bubbles. Consequently, the presence of large bubbles and a bimodal distribution indicates that in addition to frother adsorption something else is at play. It suggests that there is a coalescence promoting effect by talc.

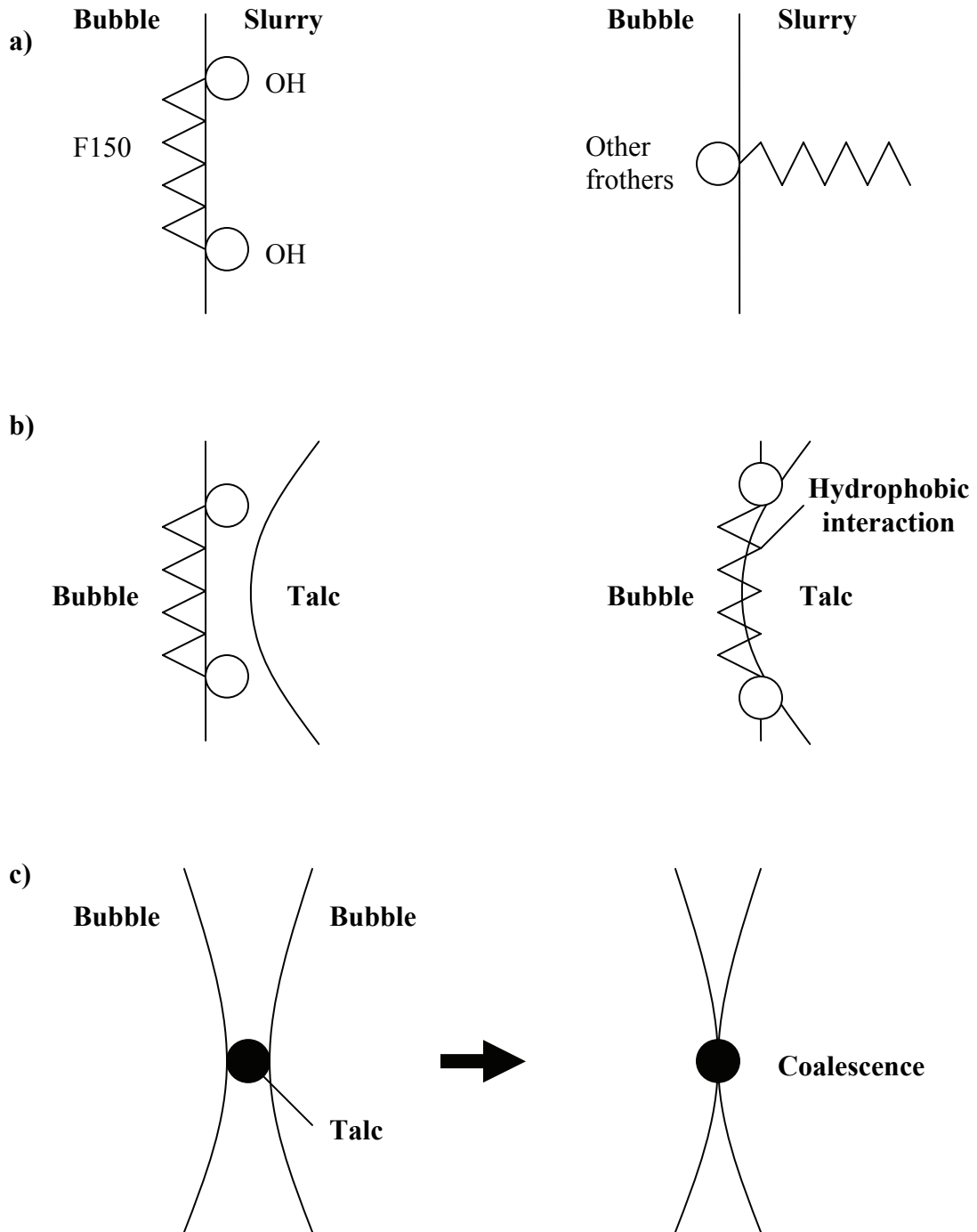
From the molecular structure of the frothers, it is noticed that two features distinguish F150 from the rest: a significantly longer hydrocarbon chain and the existence of two end OH groups. From Table 3.1, F150 has a 21 carbon atom chain compared to 1-pentanol (5 carbon atoms), 1-heptanol (7 carbon atoms) and DowFroth 250 (13 carbon atoms). This tends to make the F150 molecule more hydrophobic than the other frothers. This higher hydrophobicity may lead to stronger interaction with the hydrophobic talc particles compared to the other frothers. Adsorption of frother by carbonaceous material (e.g. coal) is well known; Fuerstenau and Pradip (1982) suggested the adsorption of frother on coal surfaces occurs through hydrophobic interactions. Though coal and talc differ in many aspects, both are naturally hydrophobic solids.

As noted, however, adsorption alone does not seem sufficient to account for the action of F150 and talc. Attention thus turns to the coalescence promoting hypothesis. Since there are two end OH groups in the F150 molecule compared to only one OH group for the other three frothers, this could mean that instead of having the hydrocarbon chain oriented into the bubble and the OH on the water-side of the bubble surface (as in 1-

pentanol, 1-heptanol and DowFroth 250), the F150 molecule lies flat at the bubble-water interface, as depicted in Figure 5.1.

The following is a speculation: F150 molecules lying on the bubble (Figure 5.1a) maximize the interaction of the hydrocarbon part of the molecule when a talc particle attaches (Figure 5.1b). The talc, as a result of hydrophobic interaction, removes F150 molecules from the bubble surface. Two bubbles coming together with a common talc particle acting as a 'bridge' (Figure 5.1c) are in a coalescence-inducing condition but now the coalescence inhibition action of the F150 has been reduced. That coalescence occurs is evident by the increasing original mode size even and, more strikingly, by the presence of the finer, secondary mode (ca. 0.3mm) attributed to coalescence mediated bubble break-up (Tse et. al., 2002).

This talc bridging notion is not restricted to the talc/F150 system but will occur with the other frother systems too. However, in those cases the frother molecule orientation and shorter hydrocarbon does not favour hydrophobic interaction and the coalescence prevention role of the frother is preserved. The role of isolated hydrophobic particles inducing coalescence is well understood, for example described by the model of Dippenaar (1982).



**Figure 5.1 – Speculated mechanism of bubble size increase in F150-talc system. a) F150 orientation at bubble/slurry interface vs. other frothers tested. b) effect of talc particle collected on bubble: F150 molecule is ‘anchored’ onto talc, preventing its coalescence inhibition action. c) talc bridging effect which combined with action depicted in b) leads to bubble coalescence.**

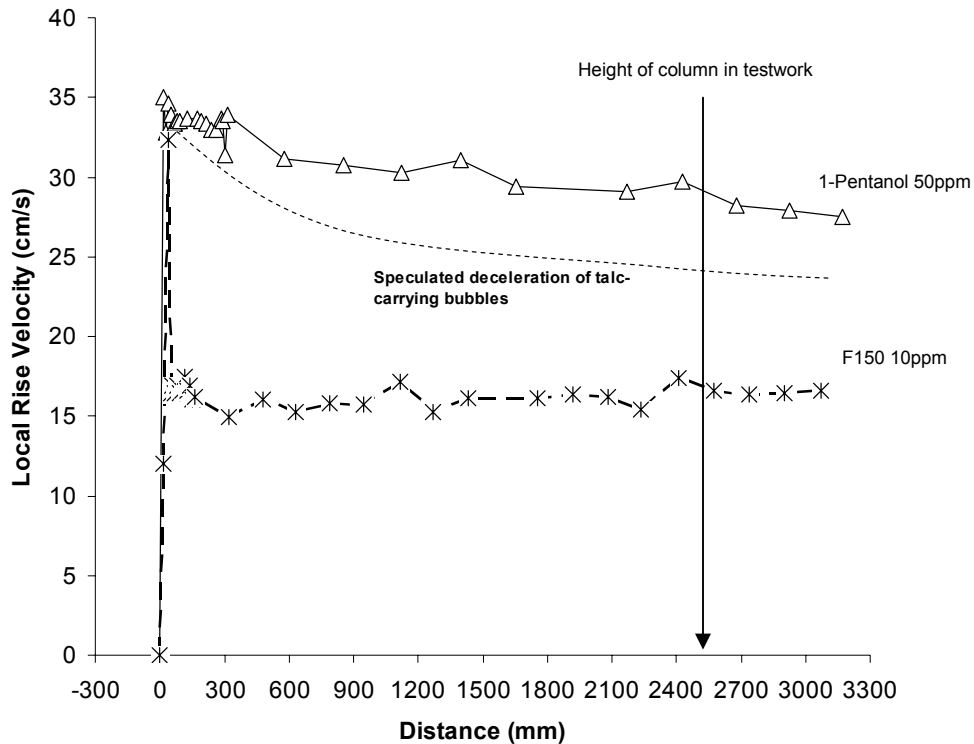
In summary, the observed behaviour of gas holdup and bubble size with F150 upon addition of talc is tentatively attributed to two factors:

- Adsorption of frother (an indirect effect)
- Talc promoting coalescence (a direct effect)

### **5.1.2 – 1-Pentanol and Talc**

The other frother that showed some impact of talc addition on pulp properties is 1-pentanol. With 1-pentanol, bubble size remained the same while gas holdup increased with addition of talc. The change is significant, in the case of 21ppm the gas holdup nearly doubling (Figure 4.6). The change in gas holdup is not due to changes in bubble size, which would have to decrease in this case, but can be attributed to bubble rise velocity. Bubble rise velocity controls gas holdup, and for gas holdup to increase, the velocity must decrease. The question then becomes: how does the talc decrease the bubble rise velocity with 1-pentanol as frother?

Rafiei (2009) have shown that bubbles in 1-pentanol, compared to F150 at concentrations similar to those here, do not reach terminal velocity over the distance available in the current test column (Figure 5.2). We can speculate that bubbles carrying talc will decelerate more than bubbles free of talc, as represented in Figure 5.2; i.e., they will have slowed more due to the attached talc and gas holdup duly increases. This does imply that the talc loading is not sufficient to reduce the terminal velocity, which bubbles do reach in F150, 1-heptanol, and DowFroth 250 (Rafiei, 2009), as there is no increase in gas holdup in those cases.



**Figure 5.2 – Single bubble velocity profile of 1.45mm diameter bubble in 1-pentanol and F150 (Rafiei, 2009)**

### 5.1.3 – Comparison of Silica and Talc

There was no significant change in gas holdup and bubble size for all frothers when silica was used as solids (Figures 4.1 and 4.2). As argued, hydrophobic interaction between F150 molecules and talc particles could be responsible for the coalescence promoting effect of talc that led to increased bubble size and decreased gas holdup. The fact that silica is hydrophilic indicates that such interaction is not possible. For the same reason, the speculated slowing down of bubbles in 1-pentanol with talc cannot occur either as silica is not collected by the bubbles. Silica appears to be in suspension in the slurry with no effect on bubble size and gas holdup.



This lack of impact is different from the conclusion of Banisi et. al. (1996) who, under flotation-related conditions, showed a consistent increase in gas holdup. The finer particle size here may be a factor in the difference.

Figures 5.3 and 5.4 summarize the effect of silica and talc in terms of effect on bubble size and gas holdup. Gas holdup for 1-heptanol and DowFroth 250 is similar for both talc and silica. Bubble size measurements for 1-heptanol, DowFroth 250 and 1-pentanol are similar as well for both solids. Based on the success correlating bubble size in air-water systems with measurements in presence of typical minerals in sulphide processing (Finch et. al., 2008) where hydrophobicity is induced by collector addition, it seems that a natural hydrophobic character of the solid may be the key.

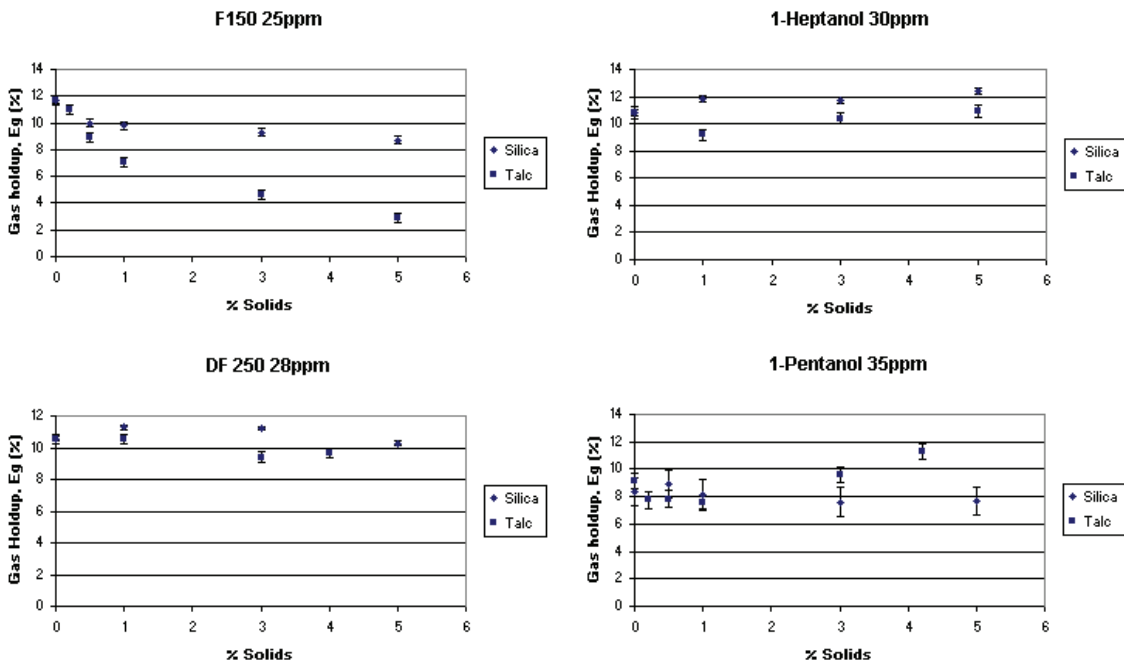


Figure 5.3 - Effect of silica and talc on gas holdup with different frothers

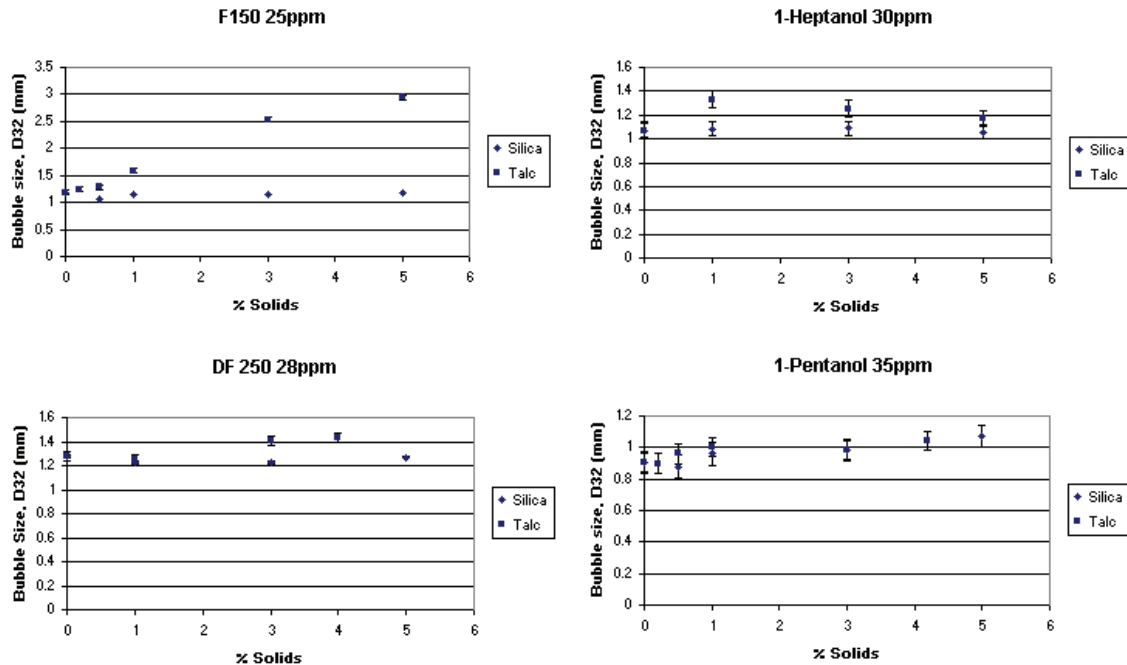


Figure 5.4 - Effect of silica and talc on Sauter mean bubble size with different frothers

## 5.2 – Froth zone

### 5.2.1 – Water and Solids Overflow Rates

In flotation systems, froth stability is generally enhanced by particles attached to a bubble (Rao and Leja, 2004). Without solids, froth rarely forms with the frother types and dosages used in practice. Particles physically block water drainage and increase the effective viscosity of the inter-bubble film. Froth becomes stabilized by hydrophobic solids when they adhere to the air/water interface so closely packed together that the draining of the liquid is restricted (Rao and Leja, 2004). This packing effect more than counterbalances the coalescence-inducing effect of isolated hydrophobic particles (Figure 5.1c). It can be argued that bubbles coalesce in the froth until the reduced specific surface area (surface area/volume) results in sufficient particle packing to restrict water drainage

and retard coalescence. Even in the absence of frother, collected particles will produce a froth; e.g. with salts in place of frother (Quinn, et. al., 2007) or even just water alone (Pugh, 2006).

Results have shown that for DowFroth 250 and 1-heptanol, while there were no changes in bubble size and gas holdup, water overflow rate increased when talc was added to the system (Figures 4.15 and 4.16). This increase in water overflow must be attributed to an increase in water retention in the froth due to the talc restricting water drainage from the froth back to the pulp. The same effect was probably at work in the F150 system but was counteracted by the changes in the pulp zone (larger bubbles, decreased gas holdup) which resulted in less water entering the froth and, in the end, resulted in a fluctuating water overflow rate.

With 1-pentanol the increase in water overflow rate was most pronounced. A froth stabilizing effect is no doubt at play but there is also an increase in gas holdup in the pulp zone (bubbles are rising more slowly) that may be playing a role in increasing the amount of water entering the froth. Moyo et. al. (2007), for instance, showed water overflow rate increased as gas holdup increased.

### **5.2.2 – Ranking of Water Overflow Rate with Addition of Solids**

Gredelj et. al. (2009) showed that in the presence of carbonaceous matter, polypropylene glycol (PPG) frothers of the lowest molecular weight and highest ratio of hydrophilic to hydrophobic groups adsorbed at the solid/liquid interface the least and produced the most

stable froth. Froth stability in this context was measured in terms of froth half-life. It is known that as the froth retains more water, bubble coalescence is inhibited which retards froth collapse, and hence the froth becomes more stable. A more stable froth means higher water content in the froth and thus, higher water recovery. Gredelj et. al. (2009) found that this was in contrast to the data reported by Tan et. al. (2005) where PPGs of higher molecular weights produced more stable foams than PPG of lower molecular weight. The contrast was attributed to the fact that Tan et. al. (2005) studied frother in solution only, in the absence of particles that interacted with the frothers. Similarly in this thesis, comparison of the water overflow rate ranking showed that in the 2-phase system, water overflow rate was higher for the frother with higher molecular weight (F150) while as soon as hydrophobic talc was introduced, the frother with lower molecular weight (1-pentanol) started to increase in water overflow rate and eventually overtake the high molecular weight frother (Figure 4.18). Though in Gredelj et. al. (2009) carbon was used and in this thesis talc, both solids are hydrophobic and we see a similar change in water overflow rate ranking with the introduction of these solids, a change related to the frother molecular weight. Gredelj et. al. (2009) proposed that further decreases in hydrophobicity, and hence decreases in adsorption of the frother onto the carbonaceous material, may be achieved by decreasing the chain length of the polymer backbone. Interestingly, in this thesis, frother adsorption onto talc was found with F150 but not with 1-pentanol and one evident difference between the two is the longer hydrocarbon chain of F150 (21 carbon atoms) compared to 1-pentanol (5 carbon atoms).

A change in water overflow ranking of frothers induced by the presence of coal, another naturally hydrophobic material, was reported by Melo and Laskowski (2006). They found that two alcohol frothers, diacetone alcohol and MIBC, gave the lowest water recoveries in 2-phase experiments but gave the highest water flotation rate constants (and highest coal flotation rate constants) in the experiments with coal. The high molecular weight polyglycol, DF-1012, the most surface active frother of the tested suite which gave the highest water recoveries in the 2-phase experiments, gave low flotation rates for water in the presence of coal and proved to be the worst agent for flotation of the coal being tested.

Based on the literature review and results of this thesis, one possibility to explain the results of Melo and Laskowski is that DF-1012 with a longer hydrocarbon (more hydrophobic) chain adsorbs more onto coal particles compared to the frothers of shorter chain length (MIBC and diacetone alcohol). This reduces coalescence inhibition in the case of DF-1012, thus decreasing the amount of water carried to the overflow. There was no frother adsorption, bubble size or gas holdup data in support, however.

### **5.3 – Overall Inference**

Based on the above, the following inference is made: Naturally hydrophobic solids adsorb certain frothers and promote coalescence. Adsorption of F150 by talc is seen in Figures 4.20 and 4.21. An indication of talc promoting bubble coalescence is found in Figure 4.21 where F150 concentration of 8-10ppm left in solution after adsorption is normally enough to produce small bubbles in water alone but did not do so in the

presence of talc ( $D_{32} = 4\text{mm}$  at 3% w/w). An additional coalescence promoting effect due to the presence of talc is therefore required. An interaction of talc with F150 at the bubble surface is hypothesized, the ultimate location where coalescence prevention/promotion occurs.

As a result of the adsorption, the following sequence of events is set in motion: less frother is found in solution, bubble coalescence rate increases, larger bubbles rise faster and gas holdup decreases, bubble surface area flux decreases and less water is carried into pulp and through the froth, resulting in less water reporting to overflow. This explains the higher amount of water overflow rate in 2-phase and lower amount of water overflow rate in 3-phase for frothers with higher molecular weights (i.e., longer more hydrophobic hydrocarbon chains) compared to frothers with lower molecular weights, e.g. F150 vs. 1-pentanol in this work, DF-1012 vs. diacetone alcohol & MIBC in Melo and Laskowski (2006) and PPG 425<sup>1</sup> & PPG 1000 vs. PPG 192 in Gredelj et. al. (2009). The reverse is true for frothers with a lower molecular weight (i.e., shorter hydrocarbon chain); because of the shorter hydrocarbon chain, there is no adsorption onto the hydrophobic solids (coal or talc), bubble coalescence is still inhibited, there is no increase in bubble size and gas holdup stays the same. However, due to the froth stabilizing effect of hydrophobic solids, water overflow does increase which explains the case for 1-heptanol and DowFroth 250 in this thesis. For 1-pentanol, the shorter hydrocarbon chain does not induce adsorption by talc (shown by test results in Figures 4.22 and 4.23) though there is an increase in gas holdup which is attributed to terminal velocity not being

<sup>1</sup>PPG 425 and F150 refer to the same frother type, i.e., the same chemical formula; PPG is just an accepted shorthand for polypropylene glycol while for F150 the letter indicates the supplier, Flottec, and the number the PPG series.

reached at the frother concentration and height of column used, as supported by Rafiei (2009). The increase in gas holdup coupled with the froth stabilizing effect of talc in the case of 1-pentanol may have further increased the water overflow rate.

In summary, the adsorption criteria leading to interaction between solids and frother are:

- The frother should have a long hydrocarbon chain ( $C \geq 15$ ).
- The solids should be naturally hydrophobic.

The second criteria suggests that solids be naturally hydrophobic. Collector induced hydrophobic solids is the common situation, for example in processing sulphide ores. As far as is known, no comparison of frothers showing reversal of ranking of water overflow rate in these cases has been undertaken. Given the successful use of long chain frothers like F150 in sulphide systems, it suggests that in these cases there is little to no interaction between solids and frother that produces 'unexpected' consequences. This remains to be verified.

## Chapter 6 – Conclusions

The following conclusions were reached as a result of this work:

- When silica was used as solids (0 to 5 w/w%), no change was observed in bubble size and gas holdup with 1-heptanol at 30ppm, DowFroth 250 at 28ppm and 1-pentanol at 35ppm.
- Of all four frothers, when talc as a model naturally hydrophobic solid was added, only F150 showed a significant change in both bubble size, which increased, and gas holdup, which decreased. For 1-pentanol, the bubble size remained the same while gas holdup increased upon addition of talc. For 1-heptanol and DowFroth 250 there was no impact on either property.
- The observed behaviour of gas holdup and bubble size with F150 upon addition of talc is tentatively attributed to two factors: adsorption of frother by talc, as demonstrated (i.e., an indirect effect) and talc promoting coalescence (i.e., a direct effect).
- The observed behaviour of gas holdup and bubble size with 1-pentanol upon addition of talc can be attributed to talc loading and showing the bubble which has not reached terminal velocity. Bubbles in the other frother systems do reach terminal velocity (Rafiei, 2009).
- At 25ppm F150, as talc was added, bubble size distribution started off as unimodal and transformed to bimodal. As more talc was added, the larger mode increased in size while the smaller mode remained unchanged. The appearance of the smaller mode can be attributed to coalescence-mediated bubble break-up (Tse et. al., 2002).



- With 1-pentanol (35ppm), 1-heptanol (30ppm) and DowFroth 250 (28ppm) systems, no significant changes were observed in bubble size distributions as talc was added: they remained unimodal throughout.
- Hydrophobic interaction between F150 molecules and talc particles could be responsible for the coalescence promoting effect of talc that led to increased bubble size and decreased gas holdup. The fact that silica is hydrophilic indicates that such interaction is not possible. For the same reason, the speculated slowing down of bubbles in 1-pentanol with talc cannot occur either. Silica appears to be suspended in the slurry with no effect on bubble size and gas holdup.
- The increase in water overflow rate in 3-phase talc systems with 1-pentanol, 1-heptanol and DowFroth 250 indicates an increase of water content in the froth due to the talc blocking water drainage back to the pulp. The same effect was probably at work in the F150 system but was countered by an increase in bubble size in the pulp zone which reduced the volume of water entering the froth.
- Test results seem to show that the water overflow rate ranking for frothers in 2 phase is reversed in 3 phase with talc. A similar observation was also seen in the solids overflow rate. The higher amount of water overflow rate in 2-phase and lower amount of water overflow rate in 3-phase for frothers with high molecular weights (i.e., long hydrocarbon chains) compared to frothers with lower molecular weights was also observed in Melo and Laskowski (2006) and Gredelj et. al. (2009).

## 6.1 - Recommendations

The following are suggestions for future work:

- Study of water drainage from the froth back into the pulp with the presence of solids. Is the large increase in water overflow when talc was added in the case of 1-pentanol and 1-heptanol, due to more water being carried into the froth from the pulp or due to the same amount of water entering but different amounts of drainage?
- Study of effect of solids on frother bubble thin films. If the first inference above is true, then the addition of a naturally hydrophobic solid like talc would increase the amount of water carried into the froth. This could be confirmed by measuring the bubble thin film with the presence of a naturally hydrophobic solid like talc.
- There exists the opportunity to study the effect of solids on bubble terminal velocity in flotation. Rafiei (2009) showed interesting revelations of the bubble terminal velocity with different frother types and frother concentrations. Adding solids as a parameter to the work will provide further insight into the complicated nature of flotation hydrodynamics.

*'Vous êtes belles, mais vous êtes vides.... On ne peut pas mourir pour vous.'*  
- Antoine de St Exupéry

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## **Appendices**

# Appendix 1: Gas Holdup Data

Table A1- Gas holdup tests with talc

Frother type	Frother conc (ppm)	Frother conc (mmol/L)	% solids	Eg (%)	95% conf. int.(%)
F150	2.5	0.006	0	5.37	0.13
			0.2	4.67	
			0.5	4.02	
			0.5	4.02	
			0.5	4.45	
			1	3.64	
			1	3.69	
			3	3.36	
			5	1.33	
	15	0.035	0	9.88	0.01
			0.2	9.01	
			0.5	7.54	
			1	6.11	
			1	6.11	
			3	4.15	
			5	2.31	
	25	0.059	0	11.64	0.18
			0	11.8	
			0.2	11.02	
			0.5	8.82	
			0.5	9.04	
			1	6.73	
			1	6.78	
			1	7.49	
			1	7.11	
			3	4.23	
			3	4.72	
			3	4.88	
			5	2.39	
			5	3.36	
5	2.9				
1-pentanol	21	0.238	0	6.05	0.08
			0.2	5.45	
			0.5	5.59	
			0.5	5.56	
			1	6	
			1	5.78	
			3	7.46	
			5	10.34	
	35	0.397	0	9.14	0.37
			0	7.57	
			0.2	7.76	
			0.5	7.33	

			0.5	8.33	
			1	7.92	
			1	7.49	
			1	7.27	
			3	9.61	
			4.2	11.29	
1-heptanol	30	0.258	0	11.31	0.43
			0	10.35	
			1	9.52	
			1	9.01	
			1	9.04	
			1	9.12	
			3	10.5	
			3	10.61	
			3	10.12	
			5	10.93	
DF250	28	0.106	0	10.55	0.32
			1	10.47	
			1	10.64	
			3	9.8	
			3	9.02	
			4	9.66	

**Table A2 - Gas holdup tests with silica**

<b>Frother type</b>	<b>Frother conc (ppm)</b>	<b>Frother conc (mmol/L)</b>	<b>% solids</b>	<b>Eg (%)</b>	<b>95% conf. int.(%)</b>
F150	25	0.059	0	11.72	0.28
			0.5	9.74	
			0.5	10.23	
			1	9.82	
			3	9.28	
			5	8.71	
1-pentanol	35	0.397	0	9.14	1.04
			0	7.57	
			0.5	8.93	
			1	9.33	
			1	7	
			3	7.6	
			5	7.71	
1-heptanol	30	0.258	0	10.66	0.24
			1	11.75	
			1	11.88	
			3	11.99	
			3	11.4	
			5	12.43	
DF250	28	0.106	0	10.55	0.14
			1	11.18	
			1	11.37	
			3	11.37	

			3	11.07
			5	10.28

## Appendix 2: Bubble Size Data

Table A3 - Bubble size tests with talc

Frother type	Frother conc (ppm)	Frother conc (mmol/L)	% solids	$D_{32}$ (mm)	95% conf.int.(mm)	
F150	2.5	0.006	0	2.3	0.09	
			0.2	2.52		
			0.5	3.13		
			0.5	2.91		
			0.5	2.84		
			1	3.7		
			1	3.59		
			3	3.97		
	15	0.035	0	1.25	0.06	
			0	1.31		
			0.2	1.3		
			0.2	1.34		
			0.5	1.43		
			0.5	1.71		
			0.5	1.62		
			1	2.02		
			1	1.92		
			3	2.69		
	25	0.059	0	1.18	0.03	
			0.2	1.24		
			0.5	1.25		
			0.5	1.32		
			1	1.61		
			1	1.56		
			3	2.53		
			5	2.95		
	1-pentanol	21	0.238	0	1.08	0.02
				0.2	1.08	
0.5				1.06		
0.5				1.1		
1				1.06		
3				1.09		
5				1.1		
35		0.397	0	0.85	0.04	
			0.2	0.9		
			0.5	0.96		
			1	0.99		
			1	1.02		

			3	0.98	
			4.2	1.04	
1-heptanol	30	0.258	0	1.07	0.07
			1	1.27	
			1	1.39	
			3	1.19	
			3	1.32	
			5	1.17	
DF250	28	0.106	0	1.28	0.04
			1	1.24	
			1	1.26	
			3	1.36	
			3	1.46	
			4	1.43	

**Table A4 - Bubble size tests with silica**

<b>Frother type</b>	<b>Frother conc (ppm)</b>	<b>Frother conc (mmol/L)</b>	<b>% solids</b>	<b><math>D_{32}</math> (mm)</b>	<b>95% conf.int.(mm)</b>
F150	25	0.059	0	1.18	0.01
			0.5	1.07	
			0.5	1.07	
			1	1.15	
			3	1.16	
			5	1.19	
1-pentanol	35	0.397	0	0.85	0.07
			0	0.96	
			0.5	0.88	
			1	0.89	
			1	1.03	
			3	0.98	
1-heptanol	30	0.258	0	1.13	0.06
			1	1.02	
			1	1.15	
			3	1.04	
			3	1.14	
			5	1.06	
DF250	28	0.106	0	1.28	0.01
			1	1.23	
			1	1.23	
			3	1.23	
			3	1.22	
			5	1.27	

## Appendix 3: Water and Solids Overflow Rate

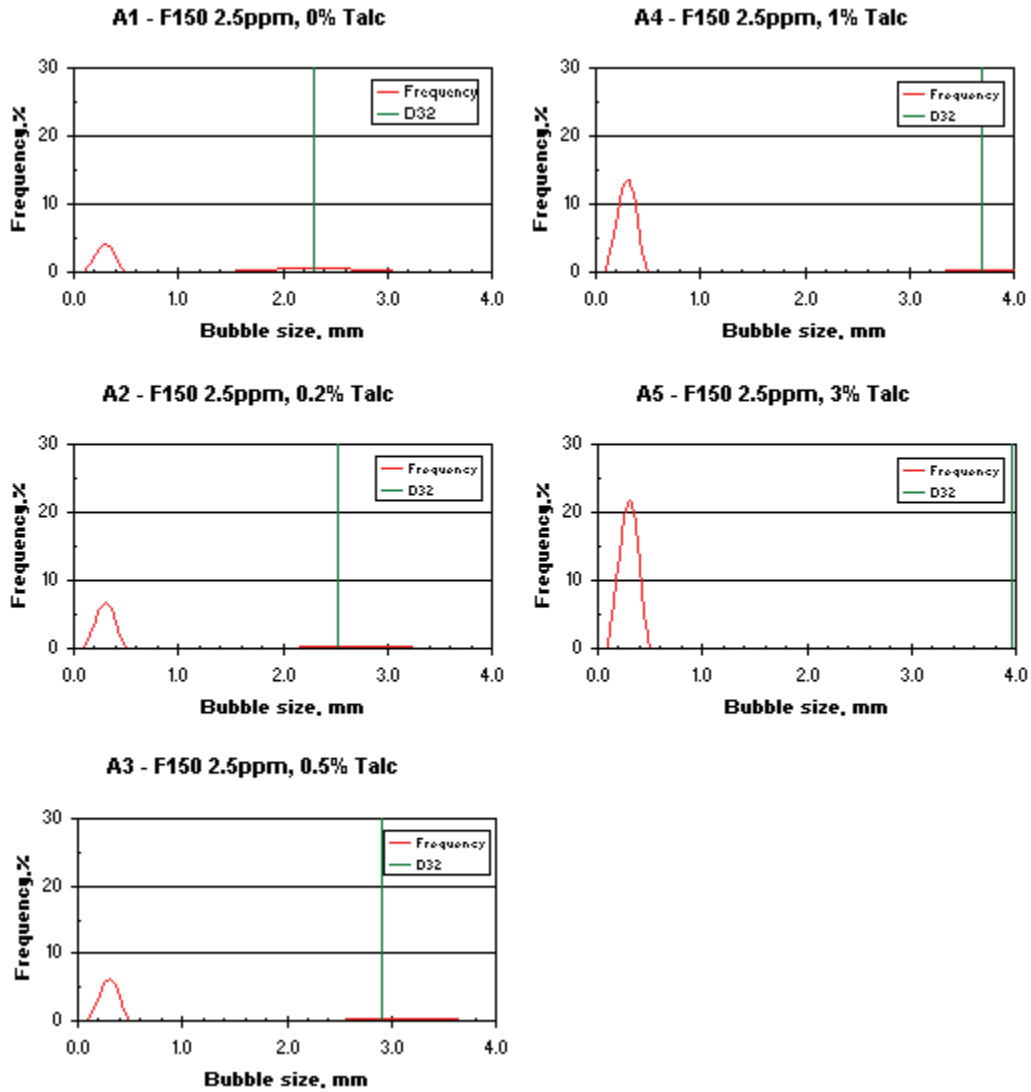
Table A5 - Water and solids overflow tests with talc

Frother type	Frother conc (ppm)	Frother conc (mol/L)	% solids	Water OF (g/min)	Solids OF (g/min)	95% conf.int. water OF (g/min)	95% conf.int. solids OF (g/min)
F150	25	0.06	0	475.5	0	6.8	1.5
			0	462	0		
			0	466.5	0		
			0	470.5	0		
			0	469	0		
			0	470.5	0		
			1	379.6	20.4		
			1	388.1	21.4		
			1	360.1	16.4		
			1	401.5	26		
			1	394.5	23.5		
			1	366.5	18		
			3	483.1	53.4		
			3	465.2	53.3		
			3	444.3	44.7		
			3	480	55		
			3	443.1	48.9		
			3	412.4	41.6		
			5	302.6	42.4		
			5	280.2	37.3		
5	299.9	36.1					
5	443	52.5					
5	374.1	46.4					
5	389.1	42.4					
1-pentanol	35	0.40	0	102	0	36	3.4
			0	118.5	0		
			0	117	0		
			1	353.4	31.6		
			1	288.2	26.8		
			1	287.2	24.8		
			1	293	26.5		
			1	252	20.5		
			3	1194.2	102.8		
			3	1282	103		
			3	1427.2	94.8		
			3	1410.6	90.4		
			3	1385.6	85.4		
			3	1510.2	78.8		
			5	1191	144		
			5	1140	127		
5	1085	139					

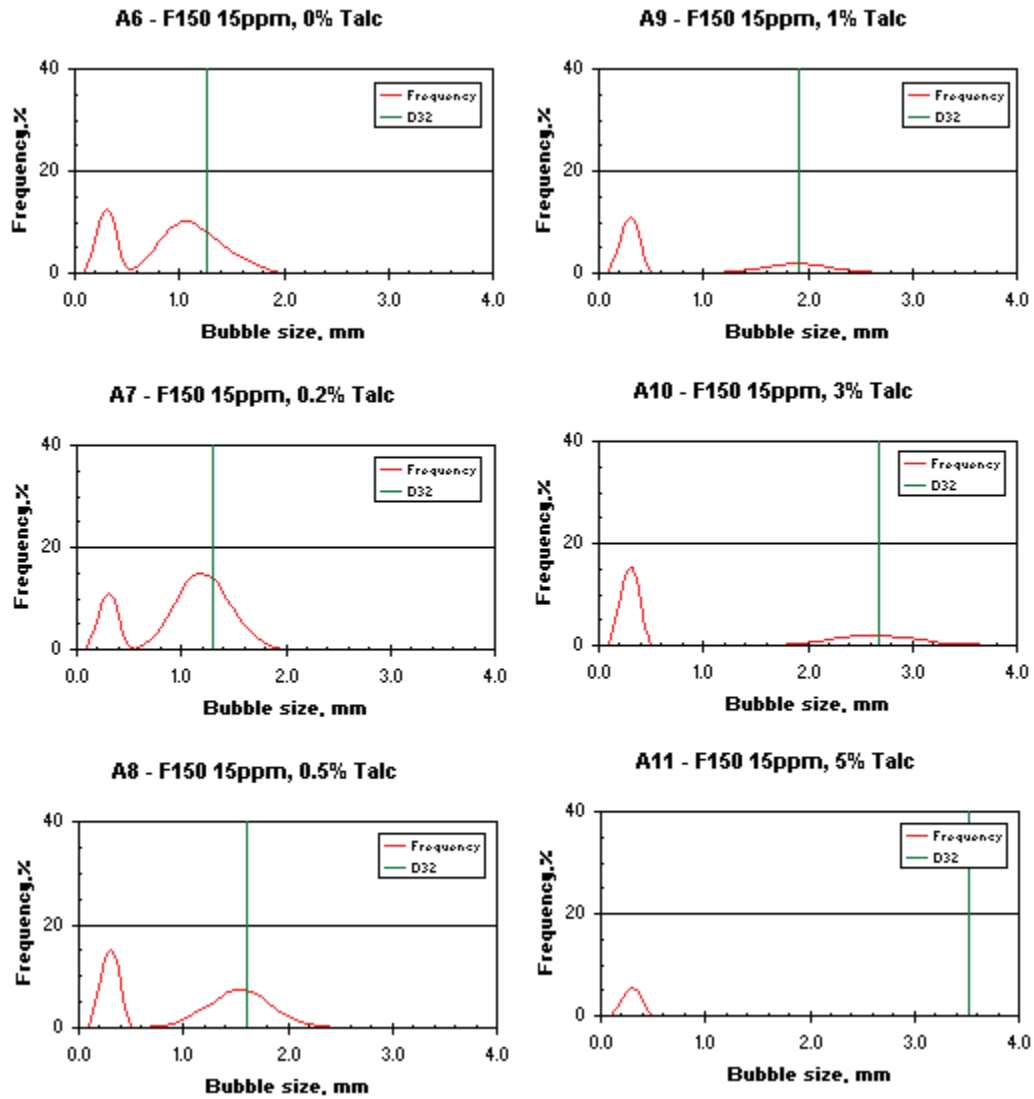
1-heptanol	30	0.26	0	18.5	0	39.6	4.6
			0	30.66667	0		
			0	80.57143	0		
			0	61	0		
			1	109.95	5.8		
			1	83.15	4.85		
			1	82.5	5.75		
			3	901.8	74.2		
			3	926.2	92.8		
			3	751	63		
			3	937.4	87.6		
			3	973.8	89.2		
			3	1027.2	89.8		
			5	1146.2	114.8		
			5	912.6	87.4		
			5	1112.4	103.6		
DF250	28	0.11	0	485.5	0	18.9	3.0
			0	475	0		
			0	473	0		
			1	463.2	17.8		
			1	497.2	17.3		
			1	459.4	14.1		
			3	1114	65.5		
			3	1173.4	59.6		
			3	1136.6	51.4		
			4	1154.6	106.4		
			4	1043.8	97.2		
			4	1089.6	91.4		



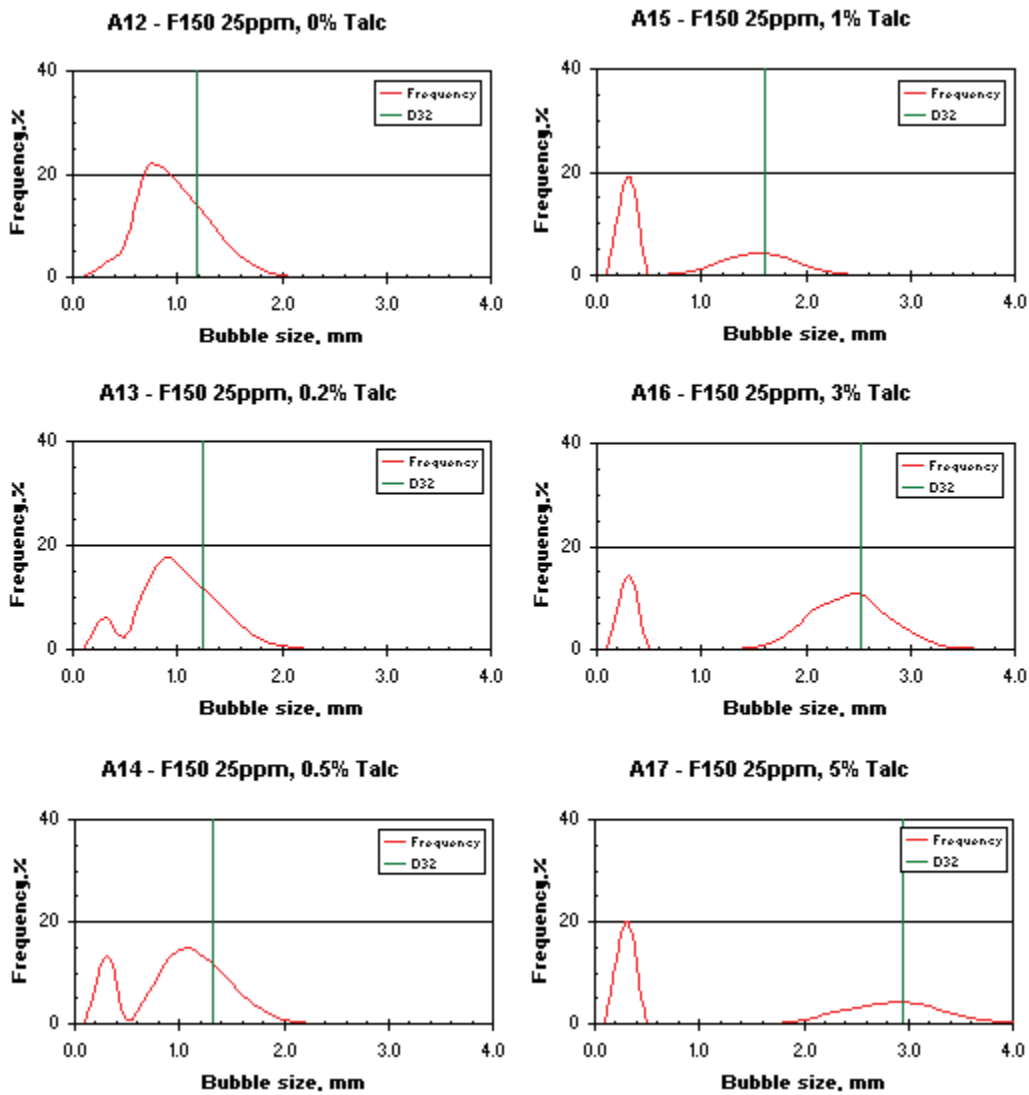
## Appendix 4: Bubble Size Distribution Data



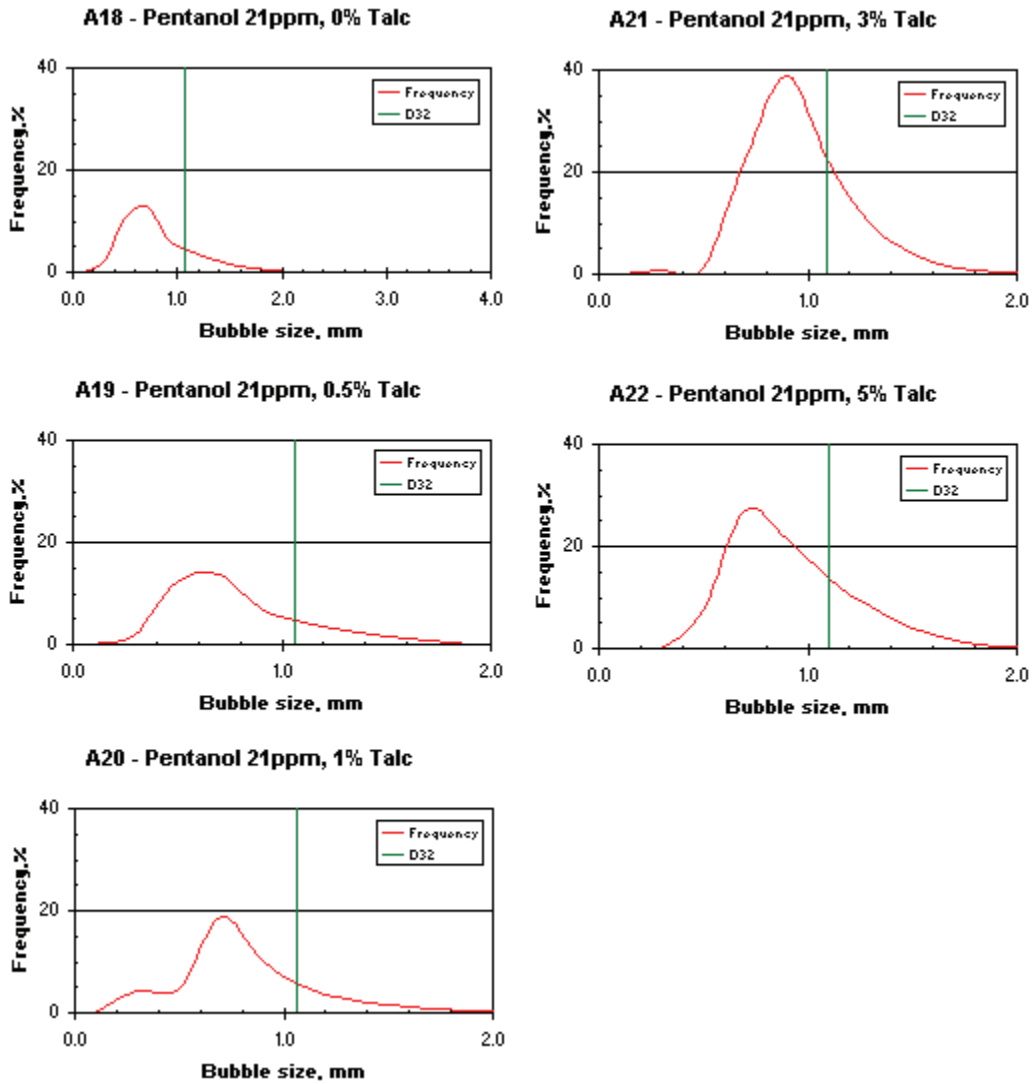
Figures A1 to A5 – Effect of talc on bubble size ( $D_{32}$ ) distribution in 2.5ppm F150



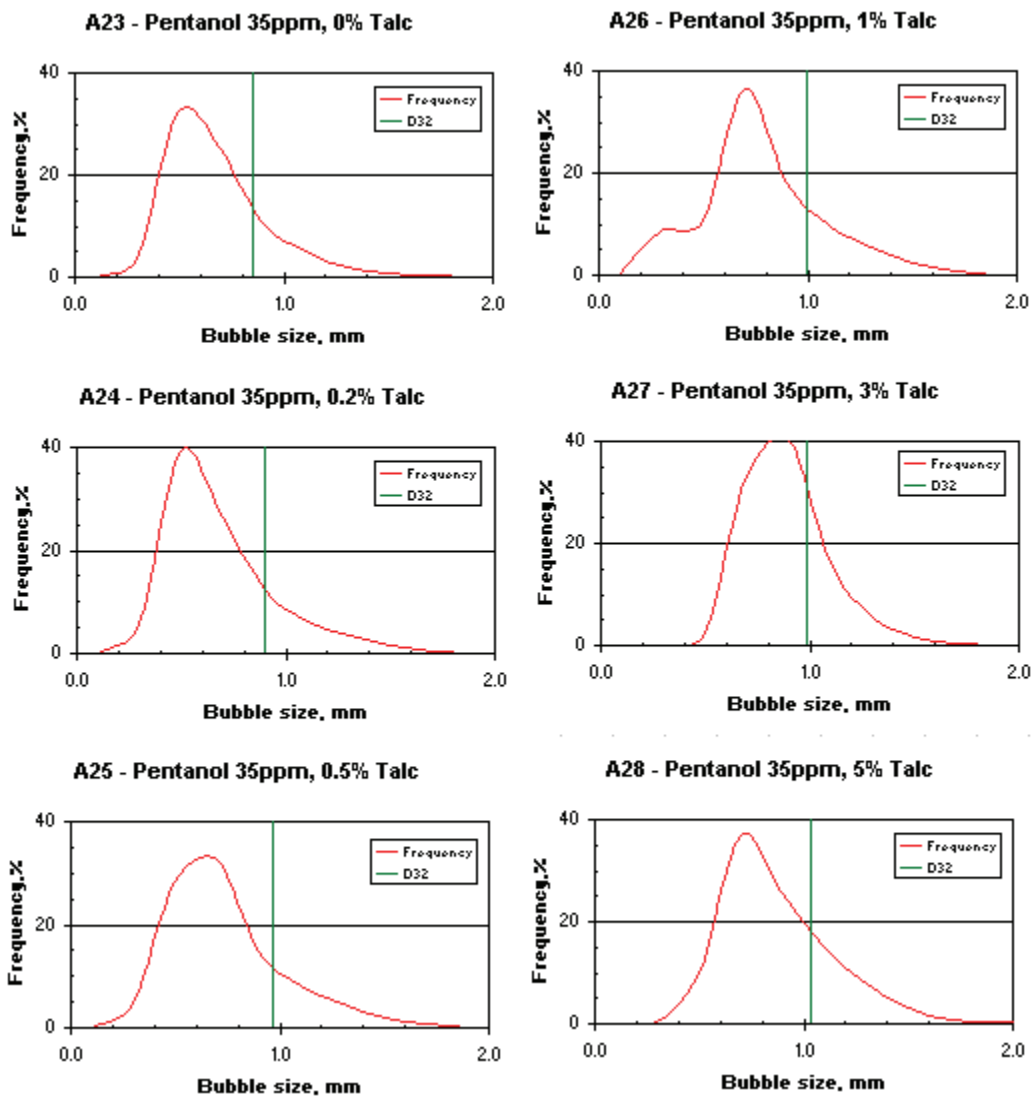
Figures A6 to A11 – Effect of talc on bubble size ( $D_{32}$ ) distribution in 15ppm F150



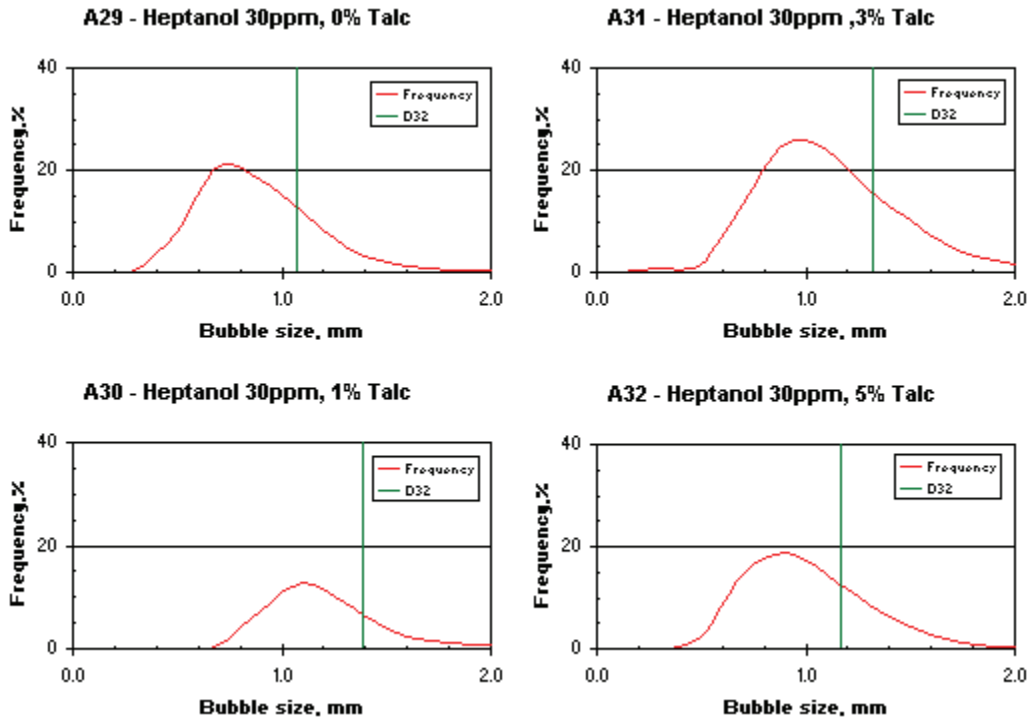
Figures A12 to A17 – Effect of talc on bubble size ( $D_{32}$ ) distribution in 25ppm F150



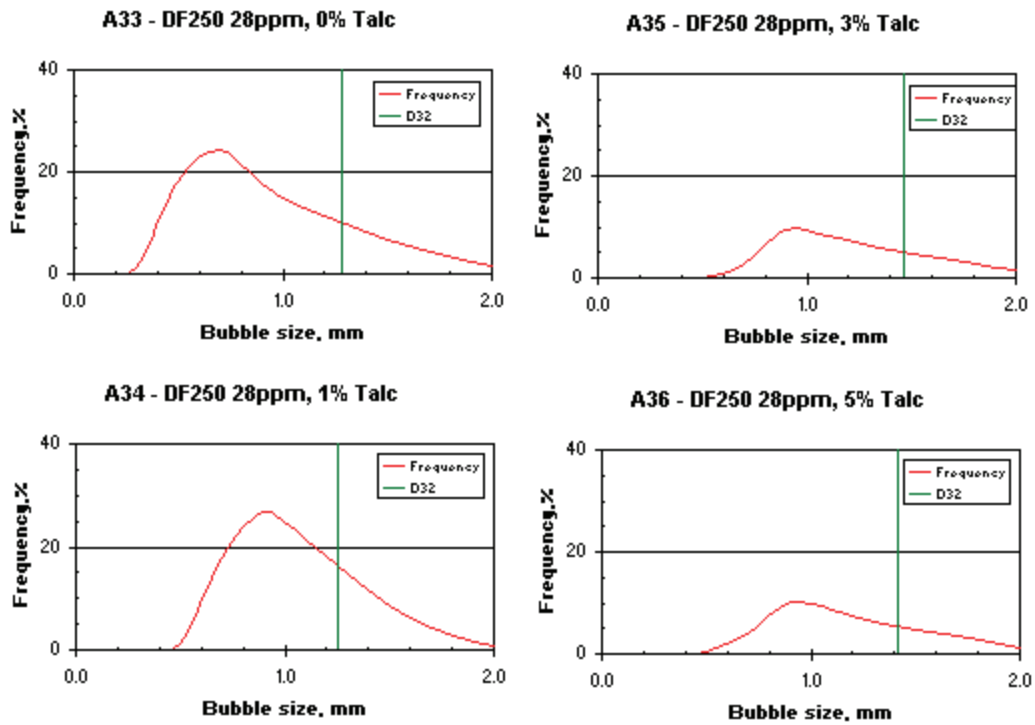
Figures A18 to A22 – Effect of talc on bubble size ( $D_{32}$ ) distribution in 21ppm 1-pentanol



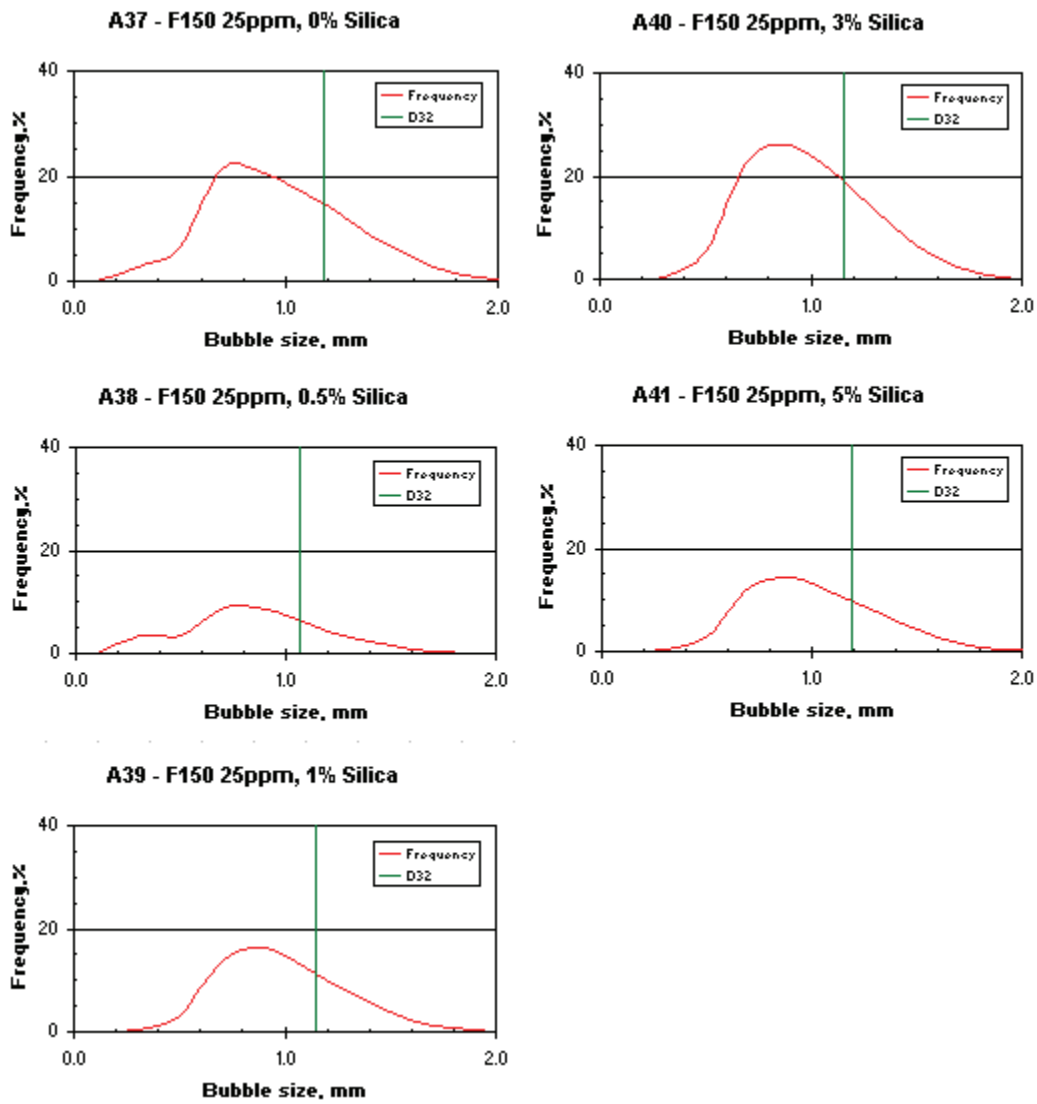
Figures A23 to A28 – Effect of talc on bubble size ( $D_{32}$ ) distribution in 35ppm 1-pentanol



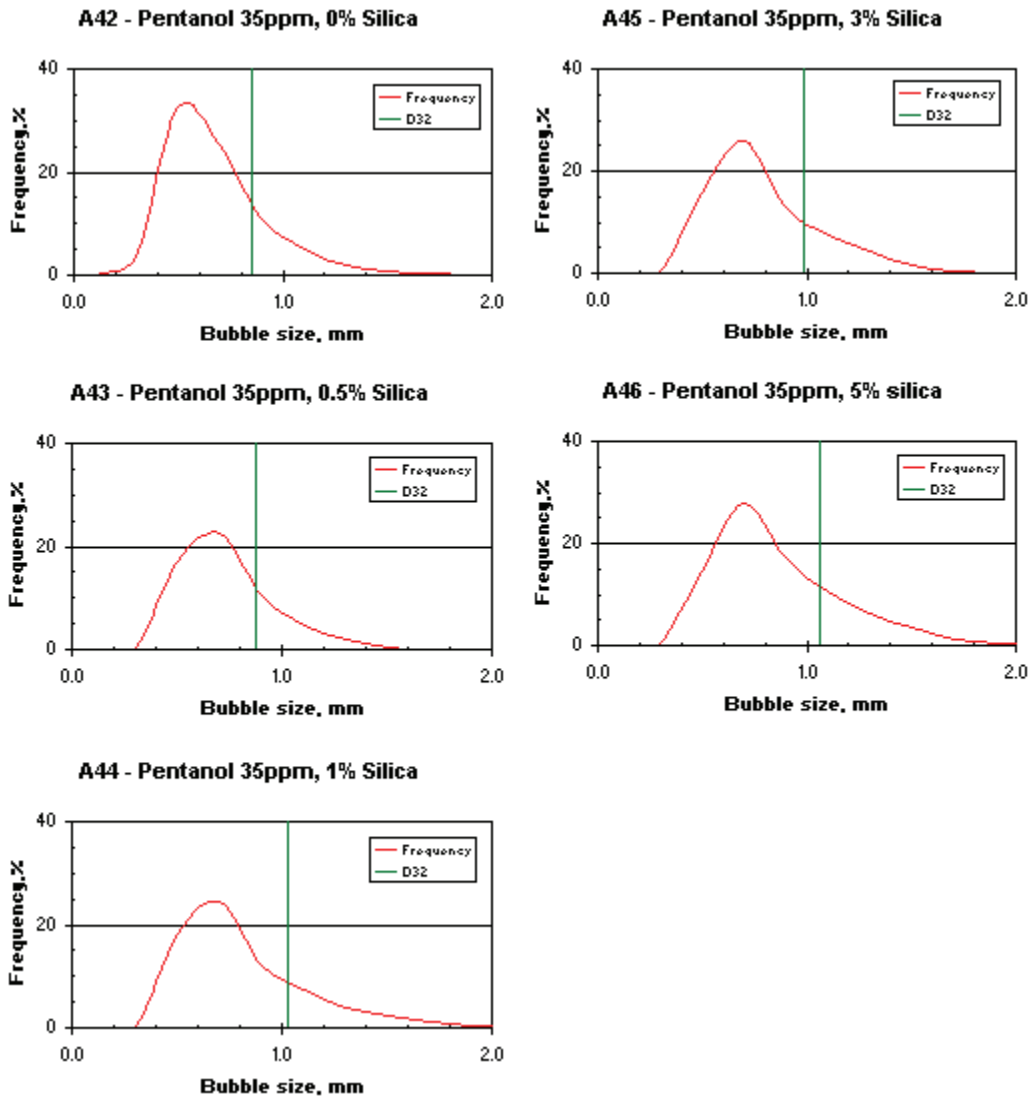
Figures A29 to A32 – Effect of talc on bubble size ( $D_{32}$ ) distribution in 30ppm 1-heptanol



Figures A33 to A36 – Effect of talc on bubble size ( $D_{32}$ ) distribution in 28ppm DowFroth 250

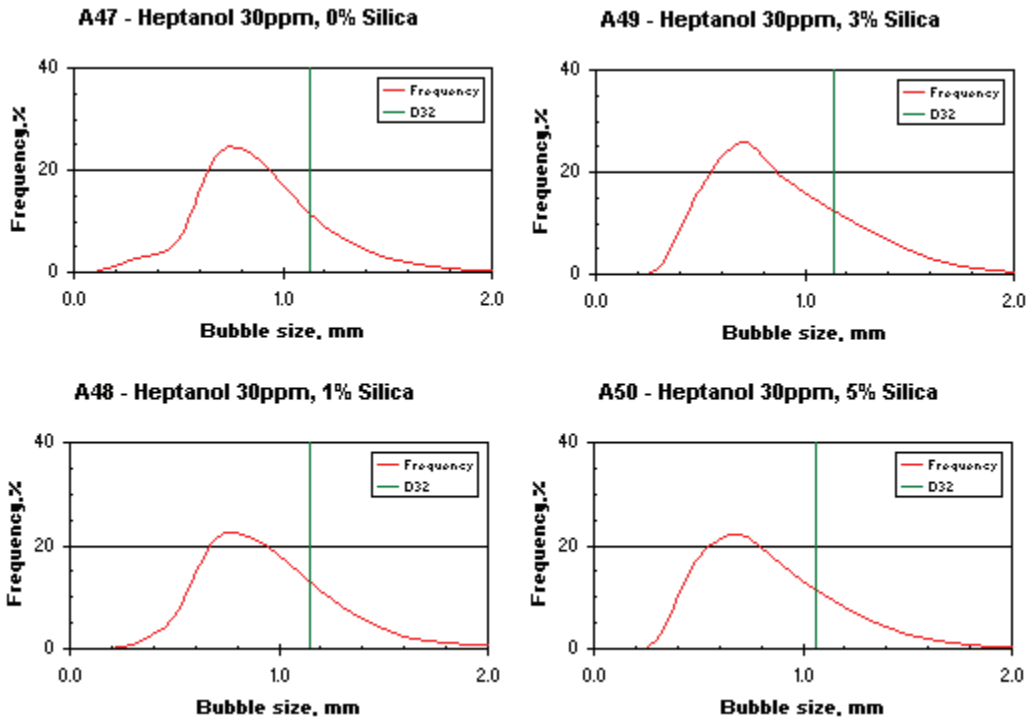


Figures A37 to A41 – Effect of silica on bubble size ( $D_{32}$ ) distribution in 25ppm F150

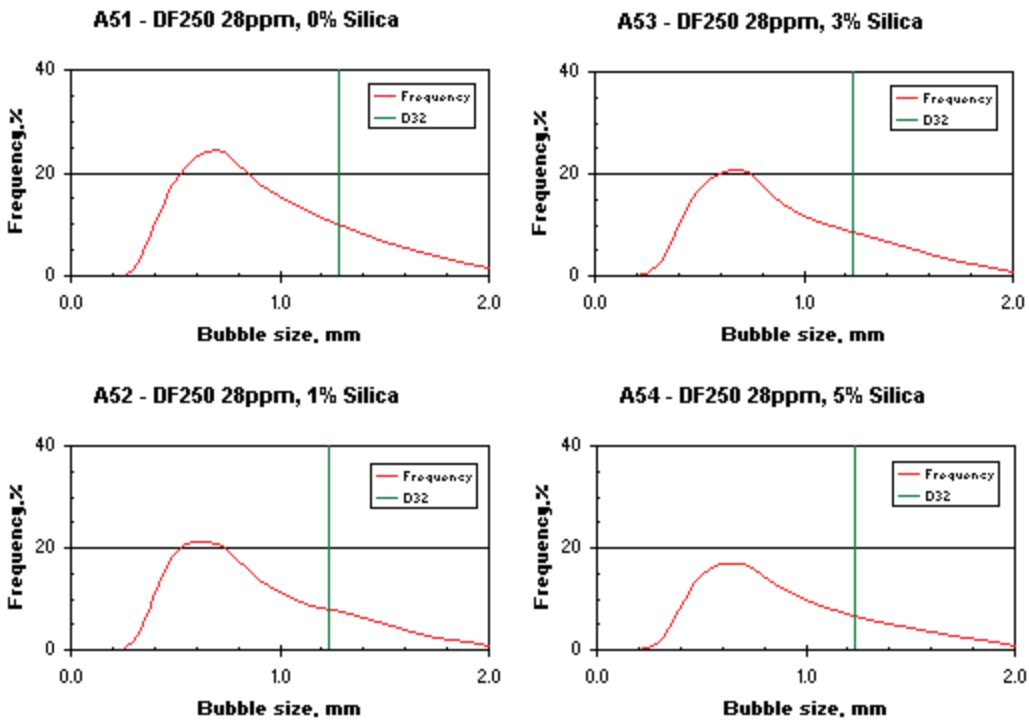


Figures A42 to A46 – Effect of silica on bubble size ( $D_{32}$ ) distribution in 35ppm 1-pentanol





Figures A47 to A50 – Effect of silica on bubble size ( $D_{32}$ ) distribution in 30ppm 1-heptanol



Figures A51 to A54 – Effect of silica on bubble size ( $D_{32}$ ) distribution in 28ppm DowFroth 250

## Appendix 5: Frother Concentration in the Pulp and Froth

**Table A6 - Frother concentrations in froth and pulp with the addition of talc in 2ppm F150**

% Talc	Frother conc. In froth(ppm)	95% conf. level(ppm)	Frother conc. In pulp(ppm)	95% conf. level(ppm)
0	30.53	0.71	1.89	0.28
0.2	5.63		1.94	
0.5	2.25		2.85	
1	2.42		5.76	
3	2.18		1.57	

**Table A7 - Frother concentration in froth and pulp with the addition of talc in 25ppm F150**

% Talc	Frother conc. In froth(ppm)	95% conf. level(ppm)	Frother conc. In pulp(ppm)	95% conf. level(ppm)
0	32.35	0.79	24.13	0.36
0.2	25.75		22.09	
0.5	23.04		21.57	
1	18.91		16.18	
3	8.67		7.38	

**Table A8 - Frother concentration in froth and pulp with the addition of talc in 8ppm 1-pentanol**

% Talc	Frother conc. In froth(ppm)	95% conf. level(ppm)	Frother conc. In pulp(ppm)	95% conf. level(ppm)
0	9.89	0.79	8.21	0.62
0.2	6.56		8.26	
0.5	13.25		9.91	
1	8.50		9.63	
3	10.27		8.69	

**Table A9 - Frother concentration in froth and pulp with the addition of talc in 25ppm 1-pentanol**

% Talc	Frother conc. In froth(ppm)	95% conf. level(ppm)	Frother conc. In pulp(ppm)	95% conf. level(ppm)
0	40.47	0.84	37.13	0.77
0.2	33.63		32.22	
0.5	31.11		37.77	
1	29.25		30.49	
3	47.01		39.07	