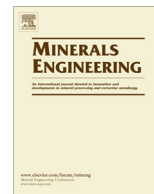




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Gas dispersion properties of collector/frother blends

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ABSTRACT

The addition of frothers in the flotation process has a significant effect on the hydrodynamic properties of the pulp phase as well as the stability of the froth phase. Some flotation collectors have been reported to have similar hydrodynamic effects, but there are few published quantitative studies. In this paper, the pure forms of the main families of sulfhydryl collectors from thiophosphates, xanthates and xanthate derivatives are tested. The aim was to determine the hydrodynamic properties of each reagent and to examine frother-collector interactions in terms of hydrodynamics. Experiments were carried out in a two phase air-water system using a 3 in. diameter column. Gas holdup (ϵ_g) and foam height (H_f) were measured to construct hydrodynamic characterization curves. The collectors tested have varied hydrodynamic activity with the xanthogen subgroup of collectors showing different degrees of hydrodynamic activity depending on their structure. In general, sulfhydryl collectors used in salt formulations showed very little hydrodynamic activity, while the oily collectors showed varied hydrodynamic effects from frother-type behavior to defoaming. In all frother-collector blends tested, a variable degree of foam stability loss was observed with this effect more pronounced in blends of weak frother and collector. A slight increase in ϵ_g was measured for almost all of the collector-frother blends.

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1. Introduction

Flotation performance is typically controlled by the addition of chemical reagents such as collectors and frothers. A frother has two main functions: generate and preserve small bubbles in the pulp phase; and provide stability to the froth phase. Three mechanisms of particle recovery into the froth have been proposed: by particle-bubble attachment; by water entrainment; and by entrapment between attached particles (Wills and Finch, 2016). Understanding frother hydrodynamic characteristics has become essential as the selection of an optimum frother is dependent on the needs of a given flotation circuit. Different methods exist to characterize frother strength, based on parameters such as gas holdup (ϵ_g), Sauter mean bubble diameter (D_{32}) and water carrying rate (Cho and Laskowski, 2002; Azgomi et al., 2007; Moyo et al., 2007). These parameters are strongly influenced by frother type. A strong frother is characterized as having a significantly higher water carrying rate, smaller bubble size and a high gas holdup, but increased frother strength also results in less selective mineral recovery (Azgomi et al., 2007; Melo and Laskowski, 2006; Moyo et al., 2007).

Cappuccitti and Finch (2008) proposed a characterization process termed the “hydrodynamic characterization curve” which correlates foam height as a function of gas holdup, *i.e.* H_f vs. ϵ_g . In this paper, the two-phase liquid-air system is referred to as “foam”, with “froth” reserved for the three-phase solid-liquid-air system typical of industrial flotation systems. The relationship between gas holdup (which provides an indication of the bubble size and rise velocity in the pulp phase), and foam height (which reflects foam stability), defines the “hydrodynamic characterization” curve of a frother. The utility of this characterization method can be understood in terms of the rate constant relationship proposed by Gorain et al. (1998), where the rate constant depends on three factors: ore floatability, bubble surface area flux and froth recovery. Thus hydrodynamic characterization curves of gas holdup versus foam height provide indications of the bubble surface area flux and froth recovery, respectively.

Relative hydrodynamic strengths can be determined from the curves produced for multiple frother concentrations. A hydrodynamically strong frother will have a sharp increase in foam height as concentration increases within a relatively small range of gas holdup (a steeper hydrodynamic curve), whereas a hydrodynamically weaker frother will have a gradual increase in foam height as concentration increases over a broader range of gas holdup.

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The shape of the curve therefore gives an indication of the relative strength of a frother.

The main function of a collector is to selectively adsorb on a mineral surface, altering its hydrophobicity such that bubble-particle aggregation can take place for minerals which are not readily floatable in the absence of absence of collector addition. A number of recent studies investigating the influence of collectors on flotation hydrodynamics have shown that amine and many oxhydryl collectors exhibit frother-like properties (Espinosa-Gomez et al., 1988; Laskowski, 1993; El-Shall et al., 2000; Atrafi et al., 2012; Ravichandran et al., 2013; Corona-Arroyo et al., 2015). Atrafi et al. (2012) quantified the frother-like properties of sodium oleate (a common anionic fatty acid collector) and determined it to be a very weak frother over the pH range 6.2–10 with a critical coalescence concentration (CCC) in excess of 70 ppm. The CCC is an indication of bubble size reduction, readers unfamiliar with this concept should consult Cho and Laskowski (2001) and Zhang et al. (2012). Corona-Arroyo et al. (2015) determined the CCC of dodecylamine (DDA), a cationic surfactant, to be 6.2 ppm in a column with a down-comer (pH 7), slightly stronger than MIBC. They also analyzed the hydrodynamics of frother-collector mixtures, and reported a synergistic effect on bubble size with small additions of DDA in the presence of frother.

This work characterizes the foaming properties of the main families of sulphydryl collectors, by measuring gas holdup and foam heights to establish their hydrodynamic strength profiles. The collectors are compared to frothers of known strength. Mixtures of selected collectors with common weak and strong frothers are then analyzed to determine possible synergetic effects.

2. Experimental

2.1. Apparatus

Fig. 1 shows the 3 in. (76.2 mm) diameter laboratory flotation column used to measure hydrodynamic properties in this work. Bubbles were generated with a stainless steel porous sparger installed at the base of the column. Gas holdup (ε_g) was measured using the differential pressure method with a Bailey PTSDDD differential pressure transducer installed at the middle of the flotation column. Calculated ε_g data were determined from the average pressure value over the first 10 min of measurement from the start of air injection.

Gas superficial flowrates (J_g) of 1.0 cm/s and 1.75 cm/s (corrected to standard conditions) were selected to represent low and high flowrate conditions normally found in a flotation cell. Along with a pressure sensor, a temperature sensor was installed on the column wall for the air flowrate correction.

Foam height was measured using a ruler taped to the upper section of the flotation column. The foam height was measured exactly 3 min after the gas holdup reached equilibrium. This time was selected in order to ensure that the foam height had reached equilibrium and to minimize variations between tests. All solutions were mixed with 25 L of Montreal tap water at ambient temperature (20–22 °C) and were manually stirred for a minimum of 5 min for highly soluble collectors and a minimum of 10 min for less soluble collectors. For insoluble collectors, the collectors were first dispersed in a 1 L beaker, stirred using a magnetic stirrer for a minimum of 30 min, and then dispersed into the feed tank. For frother-collector blends, collector concentration was kept constant while hydrodynamic measurements were made across a range of frother concentrations. Frother and collector were first mixed in a 500 mL beaker then dispersed in the solution tank for further conditioning. All tests were conducted in batch and repeated to give a total of

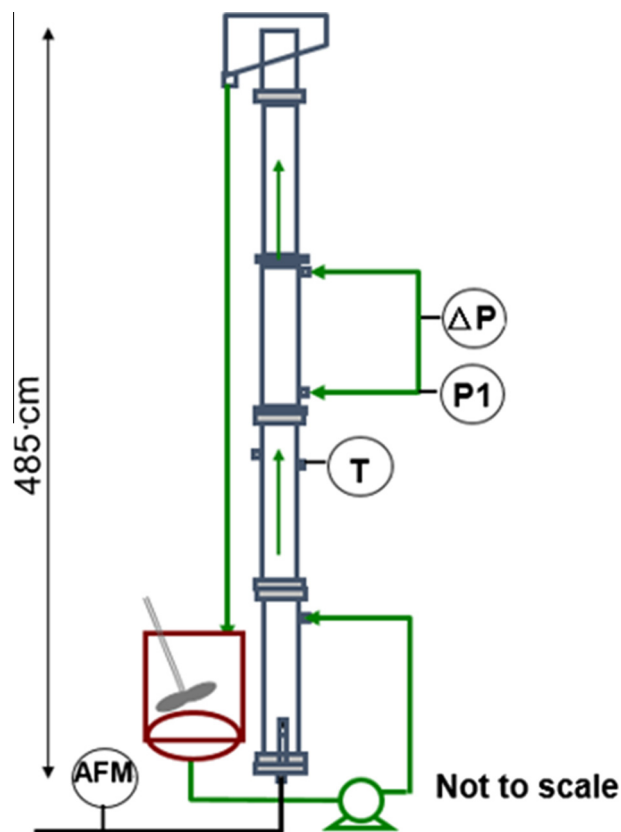


Fig. 1. Schematic diagram of the flotation column.

five measurements under each condition to ensure reproducible results.

The apparatus was thoroughly cleaned after each experimental run as most of the collectors used are known to form sticky precipitates. The top section of the column was dismantled and scrubbed of any residual precipitate as required.

2.2. Reagents

Methyl isobutyl carbinol (MIBC) and Flottec F150 (a polypropylene glycol) were selected to represent a weak and strong frother, respectively. The collectors were commercial grade provided by Flottec. They were modified to contain the lowest possible levels of solvents such that the results best represented the collector chemistry and not that of the solvents typically present in the commercial products. Table 1 lists the collectors selected from each of the main chemical families. The collectors represent chemistries from the xanthate derivative and thiophosphate family of products. Several dithiophosphate collectors of various hydrocarbon chain lengths, Flottec 2020, 2034, 2041 and 2054, were selected to determine the effect of carbon chain length on the hydrodynamic strength of this type of collector. Xanthates, dithiocarbamates, mercaptobenzothiazole, and thiophosphates are soluble aqueous salts while the xanthate derivatives and kerosene are insoluble oily collectors.

It is appreciated that many of the collectors investigated have very strong affinities for the minerals they are typically used to float (e.g., base metal sulphides) and would therefore be expected to adsorb onto the mineral surfaces during the course of a typical flotation process. The results discussed here can be interpreted as representing the behavior of the residual collector (i.e., the fraction

Table 1
Summary of reagents properties.^a

Reagents	Chemical family	Molecular formula ^b	R groups	Molecular weight, g/mol	Solubility in water ^a
SIBX 90	Sodium Isobutyl Xanthate	R ₁ X Na	R ₁ = C ₄ H ₉	172.3	53 g/100 g at 20 °C
X60-01	Sodium Dibutyl Dithionocarbamate	R ₁ R ₂ NCS ₂ Na	R ₁ = R ₂ = C ₄ H ₉	227.4	Complete
Flottec 34	Isopropyl Ethyl Xanthogen Formate	R ₁ R ₂ OCS ₂ CO ₂	R ₁ = CH(CH ₃) ₂ , R ₂ = CH ₂ CH ₃	208.3	Dispersible
Flottec 305	Amyl Allyl Xanthate Ester	R ₁ R ₂ X	R ₁ = CH ₂ CHCH ₂ , R ₂ = C ₅ H ₁₁	204.4	Negligible
Flottec 1234	Isopropyl Ethyl Thionocarbamate	R ₁ R ₂ OCSNH	R ₁ = CH(CH ₃) ₂ , R ₂ = CH ₂ CH ₃	147.3	Negligible
Flottec 1344	Allyl Isobutyl Thionocarbamate	R ₁ R ₂ OCSNH	R ₁ = CH ₂ CHCH ₂ , R ₂ = C ₄ H ₉	173.3	497 mg/L
Flottec 500	Sodium Mercaptobenzothiazole	R ₁ NCS ₂ Na	R ₁ = C ₆ H ₄	189.2	Complete
Flottec 2644	Sodium Diisobutyl Monothiophosphate	R ₁ R ₂ O ₃ PS Na	R ₁ = R ₂ = C ₄ H ₉	248.3	Complete
Flottec 2020	Sodium Diethyl Dithiophosphate	R ₁ R ₂ O ₂ PS ₂ Na	R ₁ = R ₂ = CH ₂ CH ₃	208.2	Complete
Flottec 2034	Sodium Diisopropyl Dithiophosphate	R ₁ R ₂ O ₂ PS ₂ Na	R ₁ = R ₂ = CH(CH ₃) ₂	234.3	Complete
Flottec 2041	Sodium Disecbutyl Dithiophosphate	R ₁ R ₂ O ₂ PS ₂ Na	R ₁ = R ₂ = C ₄ H ₉	260.3	Complete
Flottec 2054	Sodium Isoamyl Dithiophosphate	R ₁ R ₂ O ₂ PS ₂ Na	R ₁ = R ₂ = C ₅ H ₁₁	292.4	Complete
Flottec 2200LF	Sodium Dicresyl Dithiophosphate	R ₁ R ₂ O ₂ PS ₂ Na	R ₁ = R ₂ = CH ₃ C ₆ H ₄	332.4	Complete
MIBC	Alcohol frother	(CH ₃) ₂ C ₃ H ₄ OHCH ₃		102.2	1.6% m/m at 20 °C
F150	Polypropylene glycol frother	H(OC ₃ H ₆) _n OH		424.6	Complete
Kerosene	Oil	Sigma-Aldrich (CAS # 8008-20-6)			Negligible

^a Collector physical properties taken from supplier MSDS.^b X = OCS₂.

of collector remaining in solution after conditioning with mineral particles).

3. Results and discussion

3.1. Hydrodynamic strength of collectors

Although two different gas superficial flowrates were tested ($J_g = 1 \text{ cm/s}$, 1.75 cm/s), only results at the lower J_g are discussed in detail in this work. The reason for this was the suspected “wall effect” at the higher J_g , which contributed to high variability in the Hf data.

Table 2 shows the results of the hydrodynamic properties of all tested collectors. The oily xanthate derivatives family appears to be more hydrodynamically active than the thiophosphate collectors.

However, their hydrodynamic strength tends to decrease over time. The aqueous thiophosphate type collectors (Flottec 2020, 2034, 2041 and 2054) differ by hydrocarbon chain length (from 2 to 5). The longer chain Flottec 2041 and 2054 show some limited hydrodynamic properties at high gas flowrates while the shorter chain lengths have no effect on hydrodynamics. In contrast, Flottec 2200LF, a sodium dicresyl dithiophosphate has a hydrodynamic strength similar to that of an intermediate strength frother at high J_g but was observed to be hydrodynamically inactive at low J_g . Being a water soluble thiophosphate collector, Flottec 2200LF produced a more stable foam than xanthate collectors.

Fig. 2 compares collectors which exhibited significant hydrodynamic strength with the two known frothers, F150 and MIBC. Only Flottec 1234 exhibited comparable frother-like hydrodynamic strength. Flottec 1344 exhibited weak foaming properties, but was able to increase gas holdup, indicating that it causes a signif-

Table 2
Effect of collectors on hydrodynamic properties in two phases.

Collector group	Collector	Concentration range tested	Hydrodynamics, J_g 1 cm/s	Hydrodynamics, J_g 1.75 cm/s	Remarks
Xanthate derivatives	SIBX 90	5–30	Null ^a , n/a ^b	Null, n/a	Strong defoaming property decreasing over time
	X60-01	5–30	Semi, –	Null, n/a	
	Flottec 34	5–30	Semi, ++	Semi, +++	
	Flottec 305	5–30	Semi, --	Semi, --	
	Flottec 1234	5–30	Full, +++	Full, +++	
Thiophosphates	Flottec 1344	5–30	Semi, ++	Semi, ++	Rapid collapse of foam (within minutes)
	Flottec 500	5–50	Null, n/a	Null, n/a	
	Flottec 2644	5–50	Null, n/a	Semi, +	
	Flottec 2020	5–30	Null, n/a	Null, n/a	
	Flottec 2034	5–30	Null, n/a	Null, n/a	
	Flottec 2041	5–50	Null, n/a	Semi, +	
	Flottec 2054	5–50	Null, n/a	Semi, +	
	Flottec 2200LF	5–50	Null, n/a	Full, +++	
Hydrocarbon	Kerosene	5–25	Null, n/a	Null, n/a	

^a “Null” indicates collectors with no impact on either gas holdup or foam height, “Semi” indicates collectors with an effect on gas holdup only and “Full” indicates collectors with an effect on both gas holdup and foam height.^b “+” indicates a positive effect on either one or both hydrodynamic properties (bubble size reduction or foam stability) and “–” indicates a negative effect on the gas holdup (defoaming action). Multiple “+” or “–” indicates an increase in the magnitude of the effect, n/a indicates collectors with minimal hydrodynamic strength.

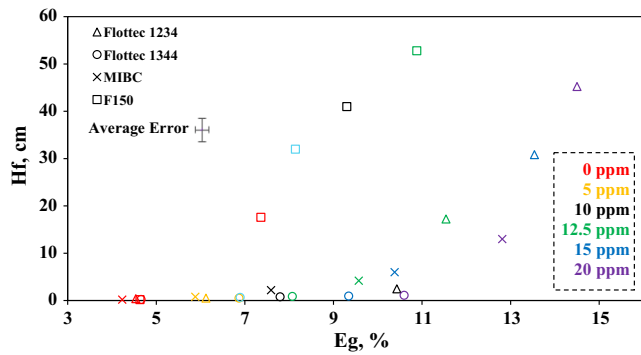


Fig. 2. Hydrodynamic strength for some collectors compared to two common commercial frothers, the colour coding in the figure represents MIBC concentration. Error bars represent 95% confidence intervals determined from all measurements.

icant reduction in bubble size. Xanthate derivatives, which are water insoluble oily liquids, are more hydrodynamically active compared to water soluble thiophosphates. The xanthate derivative collectors, however, show varied activity and for the most part, are only partially hydrodynamically active, in that they only affect the pulp phase, most likely by preventing bubble coalescence and therefore increasing ε_g . These collectors do not produce any stable foam, with most of the foam collapsing within the first minute, indicating that oily collectors might have a different mechanism to frothers, preventing bubble coalescence but breaking down the foam (Wills and Finch, 2016).

The Flottec 305 collector exhibits some interesting hydrodynamic properties: increasing collector concentration shows a gradual decrease in ε_g , to a level well below the ε_g of pure water, with a more pronounced effect observed at increased J_g and collector concentration. This phenomenon suggests a coalescence-promoting action in the pulp phase. Like other oily xanthate derivative collectors, Flottec 305 would be expected to be stripped out of the pulp phase by the bubbles in a relatively short time which explains the observed decrease in hydrodynamic properties over time.

3.2. Hydrodynamic properties of MIBC-collector and F150-collector blends

3.2.1. MIBC-Flottec 1234

Fig. 3 shows the hydrodynamic curves of MIBC-Flottec 1234 blends. All blends measured were significantly weaker than either the MIBC or the Flottec 1234 alone. If gas holdup is considered independently of foam height, then Fig. 2 indicates that MIBC-Flottec 1234 blends increase the ε_g with increasing MIBC concen-

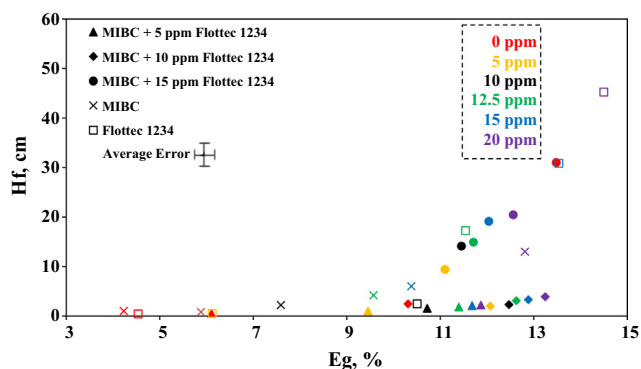


Fig. 3. Hydrodynamic strength for MIBC-Flottec 1234 frother-collector blends, the colour coding in the figure represents MIBC concentration. Error bars represent 95% confidence intervals determined from all measurements.

tration to a limited degree. At low frother dosage (<15 ppm MIBC) these blends offer a higher ε_g than MIBC alone. Although Flottec 1234 has considerable foaming strength, blending with MIBC results in a near complete destruction of its foaming character, as observed in Fig. 3. The negative impact on the foaming performance was significant enough to counteract the slight improvement recorded in ε_g to give a hydrodynamically very weak frother-collector blend. At higher collector dosages, the blend gives a significant increase in foam height, as shown in Fig. 3, but decreases ε_g compared to blends at lower collector concentrations (e.g., at 10 ppm Flottec 1234). Tan et al. (2005) proposed that a better foaming performance would result with a MIBC-strong frother blend due to the formation of a cohesive film at the air-liquid interface. Blending MIBC with Flottec 1234, a strong frother-like collector, results in blends which have almost no foaming strength over a range in collector concentration and J_g values. This suggests that frother-collector mixtures do not behave in a similar manner to frother-frother mixtures, possibly due to different adsorption mechanisms at the bubble-liquid interface.

3.2.2. MIBC-Flottec 305

Fig. 4 shows that MIBC-Flottec 305 mixtures have significantly weaker hydrodynamic strength compared to MIBC alone. Increasing the Flottec 305 concentration has a negligible effect on the hydrodynamic strength. When considering the individual components of the hydrodynamic curves, Fig. 4 shows that the ε_g of MIBC-Flottec 305 blends is slightly higher compared to MIBC alone at lower MIBC concentration (<20 ppm). However, increasing the Flottec 305 concentration in the blends results in a decrease in overall ε_g with a more pronounced effect at higher collector concentrations. Similarly to ε_g , an increase in Flottec 305 concentration decreases foam height significantly, as shown in Fig. 4, with the small residual foam layer observed to deepen with increased frother concentration. The amount of foam generated in these blends remains negligible compared to even the weak MIBC frother.

3.2.3. F150-Flottec 1344

Flottec 1344 alone has good bubble size reduction capability (i.e., increases ε_g), but does not create much foam. Fig. 5 shows that F150-Flottec 1344 blends have a hydrodynamic strength similar to a weak frother. Increasing the Flottec 1344 concentration in the blend has a negligible effect on the overall hydrodynamic strength of the blend. The blend produces an ε_g which is larger in magnitude compared to either the frother or the collector. However, the foam height associated with F150 alone is significantly reduced upon blending with Flottec 1344, resulting in a weaker overall foaming

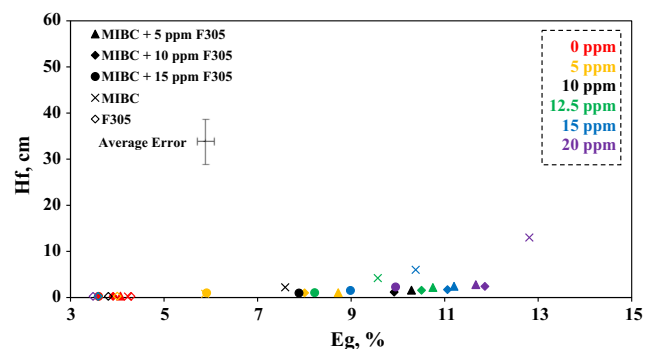


Fig. 4. Hydrodynamic strength for MIBC-Flottec 305 frother-collector blends, the colour coding in the figure represents MIBC concentration. Error bars represent 95% confidence intervals determined from all measurements.

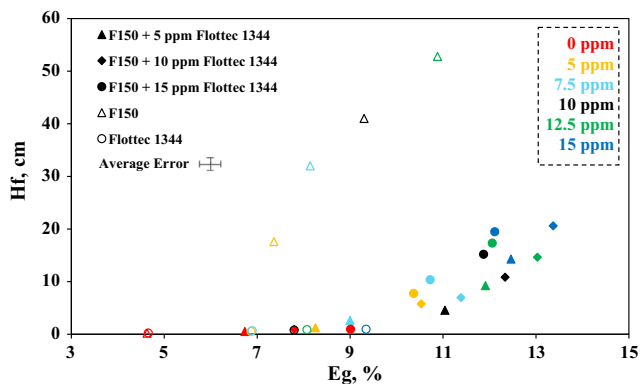


Fig. 5. Hydrodynamic strength for F150-Flottec 1344 frother-collector blends, the colour coding in the figure represents MIBC concentration. Error bars represent 95% confidence intervals determined from all measurements.

strength for the blend. At higher collector concentrations, the foam height increases slightly but the ϵ_g is significantly reduced.

3.2.4. F150-Flottec 305

Flottec 305 was observed to have a detrimental effect on both ϵ_g and H_f when blended with MIBC (Fig. 4). Fig. 6 shows that blends of Flottec 305 with the strong F150 frother result in a relatively weak hydrodynamic strength. Increasing Flottec 305 concentration has little impact on the hydrodynamic strength. Fig. 6 also shows a significant reduction in foam height for the blends compared to F150 alone. Although Flottec 305 slightly improves ϵ_g in some instances (higher collector and F150 concentrations), the significant reduction in foam height indicates that the blends are overall weaker than F150 alone.

Corona-Arroyo et al. (2015) reported co-adsorption of a cationic dodecylamine (DDA) collector and frother giving rise to a synergistic effect in reducing bubble size. For the anionic collectors studied here, gas holdup did show some degree of increase depending on the collector concentration and J_g , possibly indicating some synergistic response in bubble size reduction. Foam stability loss that lead to a drastic reduction in foam height was observed in all blends. It is known that hydrophobic particles, or simply oil droplets, when attached to a bubble, can lead to film breakage and a subsequent reduction in foam stability (Ross and McBain, 1944; Farrokhpay, 2011; Kuan and Finch, 2013). It is possible that very hydrophobic collector molecules act the same way, and cause film breakage and rupture in the foam layer. This effect is less significant when blending with a strong frother as opposed to the negligible foam observed in blends with a weak frother, suggesting the

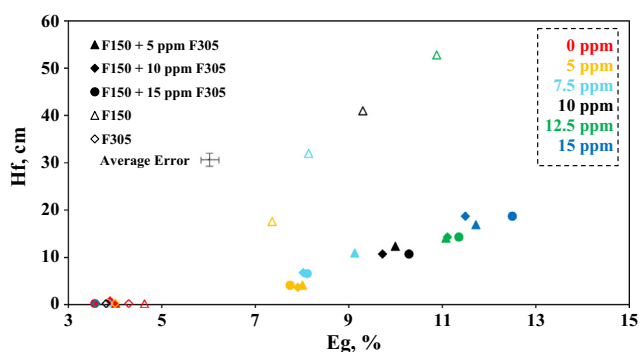


Fig. 6. Hydrodynamic strength for F150-F305 frother-collector blends at $J_g = 1$ cm/s, the colour coding in the figure represents MIBC concentration. Error bars represent 95% confidence intervals determined from all measurements.

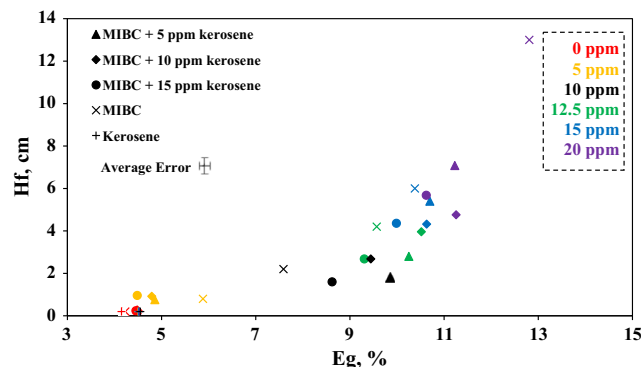


Fig. 7. Hydrodynamic strength for MIBC-kerosene frother-collector blends at $J_g = 1$ cm/s, the colour coding in the figure represents MIBC concentration. Error bars represent 95% confidence intervals determined from all measurements.

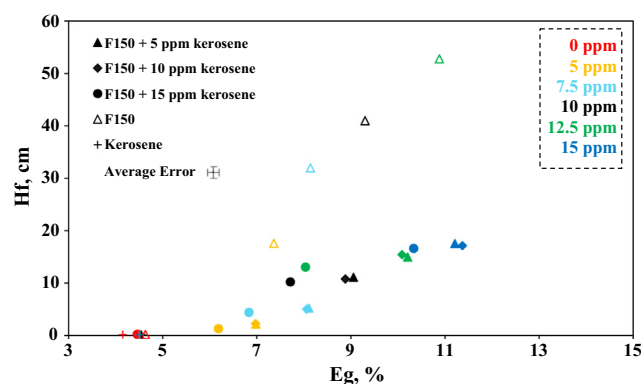


Fig. 8. Hydrodynamic strength for F150-kerosene frother-collector blends at $J_g = 1$ cm/s, the colour coding in the figure represents MIBC concentration. Error bars represent 95% confidence intervals determined from all measurements.

phenomena may be related to the frother type used in the blend. Evidence for such claims can be seen in Figs. 7 and 8. In the presence of a weak frother, such as MIBC, a drop in blend hydrodynamic strength can be observed with increasing kerosene concentration. Fig. 7 demonstrates that in the presence of kerosene, foam height decreases while ϵ_g stays relatively constant. This interference intensifies with increasing kerosene concentration. In the presence of F150, a stronger frother, an increase in kerosene concentration has negligible impact on the F150 frother properties (Fig. 8). In both cases, kerosene droplets are likely being carried into the foam resulting in film rupture, according to the mechanism proposed by Ross and McBain (1950) where oil droplets pierce the water film surrounding a bubble, spreading to form a weak oil film that ultimately results in film rupture. This mechanism may explain the observation here regarding the frother-oily collector blends. It must be reminded that the collector concentration in this work represents residual collector remaining in solution after adsorption on the target mineral surface. The observed effect of residual oily collector likely competes against the froth stabilization provided by the mineral particles themselves.

4. Conclusions

This study is the first attempt to characterize the hydrodynamic properties of sulfhydryl collectors along with investigating the possibility of synergistic effects with frothers. It was shown that Flottec 1234, a thionocarbamate collector, was the most frother-like in hydrodynamics yielding a similar hydrodynamic characterization

curve to MIBC. Flottec 2200LF, a dithiophosphate collector, was observed to have an intermediate strength hydrodynamic behavior at high J_g and high collector concentration. Divorcing the role of the dithiophosphate molecule from the associated soluble aqueous salt in affecting hydrodynamic properties was not possible in this work. Flottec 305, a xanthate ester, promoted bubble coalescence to levels such that the gas holdup fell below that of water alone. Most hydrodynamic properties of xanthates and their derivatives decreased over time due to their being stripped out of solution by adsorption on the bubbles. Most frother-collector blends showed small improvements in gas holdup according to the following order: MIBC-Flottec 1234 > F150-Flottec 1344 > MIBC-Flottec 305 > F150-Flottec 305. Foaming strength, as determined by foam height measurements, of all the tested frother-collector blends indicated a significant loss of foam stability. A possible mechanism is that hydrophobic oily collectors promote film breakage in the foam, as is the well-known case for oil droplets.

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