Entry to Stockholm Junior Water Prize 2017

Fluid Foam Development: *A Novel Approach to*

Heavy Metal Decontamination from Effluent Water

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2. Preliminary Matters

2a. Project Abstract

This project investigates the application of bulk bubble foam when implemented in the process of heavy metal removal from industrial effluent wastewater. In order to achieve successful metal removal, experimentation was designed to increase overall foam stability and longevity while also reducing internal liquid film instabilities that would increase the rate of foam decay. The criteria required for high-volume particle-stabilized foam creation was analyzed, and it was concluded that the addition of hydrophobic particles into the solution reduced overall foam drainage, improving stability. It was also concluded that the introduced aggregated particle network within the fluid system was responsible for a deceleration of foam structure decay. The simplicity and versatility of this novel approach is expected to aid the future formulation of stable wet foams for a variety of physical applications such as materials manufacturing, food, cosmetics, and oil recovery, but possesses greatest application potential for mass heavy metal decontamination from industrial effluent water as the process can be easily scaled to accommodate large quantities of water.

2b. Table of Contents

2c. Key Words

Agglomeration · Bubble Foam · Coalescence · Coarsening · Colloidal silica · Continuous foam separation · Flock Particle · Fluid film · Foam fractioning · Heavy metal · Induced gas flotation · Interfacial pressure gradient · Liquid Interface · Particle recovery/ removal · Pickering emulsion · Plateau Boarders ·Silicon Dioxide (Silica)

2d. Abbreviations and Acronyms

FPS- Frames per Second; MCL- Maximum Contaminant Levels

2e. Acknowledgements

2f. Biography

My name is Nicole Schmidt and I am an IB high school student from Alberta, Canada. My interest in science and engineering began at an early age and throughout high school has since developed into an avid love for fluid mechanics. I began my current project after learning about the difficulties that several prominent Canadian industries face when processing effluent wastewater, and the need for a sustainable, cost-effective solution. Outside of my science pursuits, I am involved extensively with my high school's Model United Nations team and have had the opportunity to attend conferences across Canada. I am also an internationally ranked Open-Championship level Irish dancer, and I will be competing at the North American championships later this year. My other extracurricular activities include visual arts pursuits, in-school clubs, and community volunteering efforts. Next year I will be beginning a combined undergraduate degree in arts and sciences at the University of Calgary. I am incredibly excited about being able to attend the SJWP, as I believe that this event is a fantastic platform to promote the importance of water science to youth on a global scale.

3. Introduction

3.1 Problem: Currently, several international industrial water-processing operations are seeking novel barrier-free technologies for selective removal of hard-to-eliminate heavy metal particle contaminants from residual processing water without chemical addition [1]. The wastewater from industrial operations such as mineral extraction, petroleum refinement, and electroplating is filtered to remove prominent contaminants such as solvents and cleaning agent, but remaining permeate typically consists of ultrafine hazardous heavy metal particles [2]. Heavy metals in waste streams don't naturally degrade and are toxic to organic life, even at low concentrations. This can include metals such as copper II, nickel, zinc II, and lead II. The current cost of filtering and removing heavy metal contaminants from the water phase currently is considerably more than liquid disposal, however improper disposal can result in long-term environmental consequences such as seepage and potential ground water contamination. This project aims to achieve a sustainable, efficient, cost effective approach to remove commonly found heavy metals from industrial wastewater by developing a technique that would ensure a high percentage metal recovery rate.

3.2 Process: Bulk bubble foam is an agglomeration of gas bubbles separated from each other by thin liquid films in a continuous liquid phase [3]. Bubble foam- when utilized as an afterthought to induced gas flotation- has immense application potential for multiphase wastewater refinement efforts as it has the potential to effectively capture ultrafine suspended particles. This paper is restricted to studying the application potential of closed-cell fluid foams, which are formed by injecting air bubbles into wastewater contained in a flotation chamber. The air bubbles created by a pump or sparger at the base of the tank adhere to the particulate suspended matter in the solution-such as heavy metal contaminants- and float to the surface where the dense bubble foam is formed. The foam can then be skimmed, and the heavy metal particles separated through evaporation, centrifuge or gravity separation [4].

3.3 Project Approach: Traditionally, a surfactant is used in the solution in order to stabilize the foam formation and also to aid in the adherence of heavy metal contaminants to the rising bubbles, however surfactants pose long-term environmental risks if not removed properly, and are costly due to the large volume that has to be injected into the wastewater. Alternatively, as demonstrated by Pickering Emulsions, foams can be stabilized by dispersing particles into the solution which adhere to the rising gas bubbles and serve to reduce their surface tension, making them less susceptible to instabilities that would

result in their collapse [5]. If the injected particles have a moderate hydrophobicity, the foams can be extremely stable for long periods of time without any chemical additives. An otherwise non-foamable solution may be foamed through the addition of partially wettable particles dispersed in the solution. Large hydrophobic particles (diameter approximately between 1 and 8 mm), present on foam surface films, retard both foam and film drainage increasing overall film stability. Smaller hydrophobic particles (<1 mm) lack this property as they shorten film lifetime in foams, as they influence foam drainage [6]. The heavy metal particles dispersed in the solution adhere to these hydrophobic "floc" particles, which are already adhered through surface tension to the rising bubbles. The intentional dispersion of hydrophobic floc particles in the solution is important to preventing foam cell collapse and overall foam decay- as more stable foams ensures increased particle containment and removal [7]. The hydrophobic particles used in this project are colloidal silica particles [8]. Silica is the major constituent of sand and exists both as a mineral but also can be produced synthetically and poses no long term environmental dangers, making it the ideal floc particle.
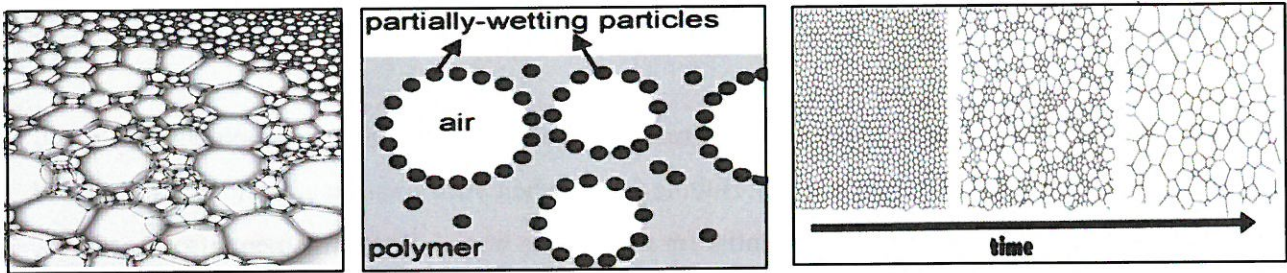

Figure 1. Photograph example of bulk bubble foam formation [9]

Figure 2. Particle-bubble stabilization diagram [10]

Figure 3. Foam decay as a function of time. The left cell is the most favorable bubble arrangement to ensure effective particle entrapment [11]

3.4 Experimental Objectives:

- Confirm that interfacially adsorbed hydrophobic floc particles can stabilize bubble foams for extended periods of time
- Confirm that hydrophobic floc particles are capable of contaminant removal from a fluid system by testing the effectiveness of silica particles in heavy metal particle entrapment, and the consequential removal from the fluid system utilizing a bubble column

- Confirm that hydrophobic silica floc particles are a viable means of foam stabilization, as opposed to surfactants by testing the effectiveness of the silica in film stabilization as a function of bubble coalescence
- Test the above scenarios with the development of an ideal approach to heavy metal removal in mind, and apply successful methods to industrial effluent waste water decontamination

3.5: Previous Scientific Work: Limited studies can be found referencing the success of Pickering Emulsions used in a real world context, and almost no literature exists on the success of dense bubble foam used for practical foaming operations such as heavy metal removal. Additionally, no registered patents based upon this new technology currently exist. One study examined the possibilities of heavy metal removal by electrostatically charged foam separation to target the different ion charges in the dispersed metals [12]. Additionally, some researchers have investigated the use of branched additives to modify the bulk rheology, e.g., increase the tensile elasticity, and hence improve foam ability [13,14,15]. P. Wilson also published a lengthy summary review of emulsion foam floatation in 1978 documenting recent advances in the stabilization of emulsions and foams by particles of nanoscale and microscopic dimensions [16]. Ongoing research is providing greater insight into (i) the molecular factors controlling particle wettability and adsorption, (ii) the structural and mechanical properties of particle-laden liquid interfaces, and (iii) the stabilization mechanisms of particle-coated droplets and bubbles.

4. Materials and Methods
4a. Differentiating Experimentations:

Experiment 1- Testing the capabilities of hydrophobic silica particles to stabilize the bubble film. The effectiveness of the dispersed silica particles will be gauged as a function of the rate of bubble coalescence, as reduced coalescence results in greater foam longevity and density. Coalescence occurs when the bubbles in are in contact with one another in an agglomerated setting (such as that which exists in foam), and the thin liquid film existing between the two bubbles drains until the point where the two bubbles merge into one another to create a single bubble (Figure. 4). It is possible to measure the stability of the fluid interface, and thus the foam, by measuring the coalescence time, which is measured from the point of bubble contact, to complete film rupture and merger. The frequency that coalescence occurs, and the time that it takes to occur will be measured as a factor of silica concentration increase in the solution.
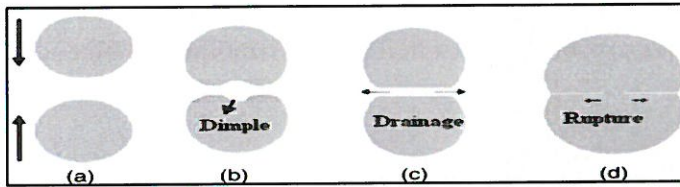
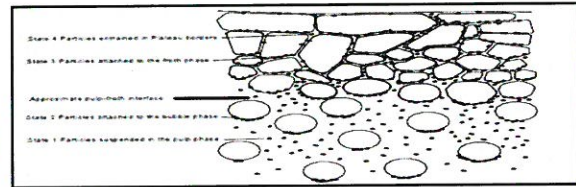**Figure 4.** Bubble coalescence process illustrated [17]

Figure 5. Diagram illustrating colloidal silica coating flotation bubbles [18]

Experiment 2- Testing the capacity to which silica particles can entrap and remove heavy metal particles from the fluid system. The ability for heavy metal particles to form a stable aggregate on the surface of flotation bubbles will be measured by gauging the amount of heavy metal recovered during experimentation compared to the amount of dispersed silica in the solution. Height of the foam in the bubble column will also be taken into consideration, as is an indicator of overall stability of the fluid-foam system.

4b. Solution Composition: This artificial wastewater solution was created in order to simulate industrial effluent as closely as possible and was kept constant in all experiments conducted. This solution was prepared in reference to preexisting measurements of a known wastewater solution [19]. It was prepared using 1000 mL of distilled water, and 10g/ 1000mL of NaCl in order to replicate a slight electrolyte concentration that exists in the wastewater (1%). Seventeen scientific PH drops/ 1000 mL were also used to mimic an acidity of 5 in the replica wastewater solution. For tests involving a surfactant, 100mL of sodium lauryl sulfate was used. It is a stand-alone effective foaming agent and anionic detergent found in many personal care products and industrial surfactants, which resultantly end up wastewater. The final solution viscosities were measured in confirmation to replicate wastewater by a stress-controlled rhemonter, and the final densities of the solution are measured by a 10ml specific gravity bottle.

Solution Number	Solution Composition	Silica dispersed in solution	Notes:
1	Replica wastewater solution	None	Control Solution
2	Replica wastewater solution and sodium lauryl sulfide	None	Control Surfactant Solution
3	Replica wastewater solution- 1% Silica	1000 ppm	-
4	Replica wastewater solution- 2% Scilica	2000ppm	-
5	Replica wastewater solution- 3% Silica	3000ppm	-
6	Replica wastewater solution- 4% Silica	4000ppm	-
7	Replica wastewater solution- 5% Silica	5000ppm	-
8	Replica wastewater solution- 6% Silica	6000ppm	-
9	Replica wastewater solution- 7% Silica	7000ppm	-
10	Replica wastewater solution- 8% Silica	8000ppm	-
11	Replica wastewater solution- 9% Silica	9000ppm	-
12	Replica wastewater solution- 10% Silica	10000ppm	-

Figure 6. Solution composition chart. The above solutions were used in both Experiments

4c. Colloidal Silica: Interfacial adsorption of a large number of hydrophobic particles at the bubbles surface causes a monolayer to develop at the air/liquid interface, and the significant mechanical robustness of the monolayer is then responsible for increased foam stabilization (Figure 6). Colloidal silica was chosen for experimentation. It is a suspension of fine amorphous, nonporous, spherical, evenly sized, slightly hydrophobic particles in a liquid phase. Colloidal silica's are prepared in a multi-step process where an alkali-silicate solution is partially neutralized, leading to the formation of silica nuclei. The colloidal silica used in experimentation was a 50 wt. % suspension in H_2O, with a surface area of ~140 m2/g, particle diameter of 2-3 mm, a PH of 9.0, and a density of 1.4 g/mL at 25 °C.

4d. Heavy Metal Samples:

	Copper II	Nickel	Zinc II	Lead II
Assay	≥99.5% (complex metric)	≥99% trace metals basis	99.5 % Trace metal basis	99.5% Trace metal basis
Form	Powder	Powder	Powder	Powder
Resistivity	1.673 μΩ-cm, 20°C	6.97 μΩ-cm, 20°C	5.8 μΩ-cm, 20°C	12.7 μΩ-cm, 20°C
Particle size	≤0.6 mm	~ 0.5 mm	~ 0.5mm	~ 0.6mm
bp	0.063-0.15 mm (sieve analysis)	2732 °C (lit.)	907 °C (lit.)	2672 °C (lit.)
mp	2567 °C (lit.)	1453 °C (lit.)	420 °C (lit.)	1857 °C (lit.)
Density	1083.4 °C (lit.)	8.9 g/mL at 25 °C (lit.)	7.133 g/mL at 25 °C (lit.)	7.14 g/mL at 25 °C (lit.)

Figure 7. Physical characteristics of scientific grade heavy metal samples used

4e. Procedure:

Experiment 1- The objective of this experiment is to determine the effectiveness of colloidal silica particles in reducing the frequency of coalescence and mitigating overall bubble film collapse. A single one-on-one bubble interaction system was created for this experiment, as it was easier to study the bubble film rupture. The laboratory set up consisted of two adjacent syringes submerged in a fluid bath, connected to microair compressors (Figure 8). The syringes were separated by a 3cm gap, and submerged 2.65 mm below the fluid surface. Two bubbles were produced by pushing air from a syringe through a capillary tube attached to a moving plate, through which the approach velocity of the two bubbles could be controlled. The bubbles were grown until the film thickness between the bubble and liquid-air interfaces was about 2 mm. Using pairs of 2cm diameter bubbles (± 0.5-mm), the approach

velocity of the two bubbles was kept constant between all trails at 2 mm/s, until the point of fluid-film contact, at which the forward movement was stopped. The process from the point of bubble contact to the point of total film collapse was recorded with the aid of a microscope-video set with 15x magnification and a high-speed video camera recording at 17.4 fps. The size of the drop was later measured through the analyzed video images, as was time duration. Fluid mechanics software was then used for modeling the interactions of the two bubbles from the footage. Each trial (silica concentration increase) was repeated 100 times to ensure optimal accuracy, and minimal result variance. Before commencing testing, all system components including the glass cell, syringe, needle and tubing were rinsed thoroughly in distilled water. Once the solution was prepared it was then poured into the glass photography cell and the whole system is kept at 20 °C for 24 hours to ensure that thermodynamic equilibrium has been achieved throughout the system. Once testing has begun the fluid bath was covered by clear Plexiglas sheet to ensure that no additional particles land on the surface, altering the solutions overall stability.
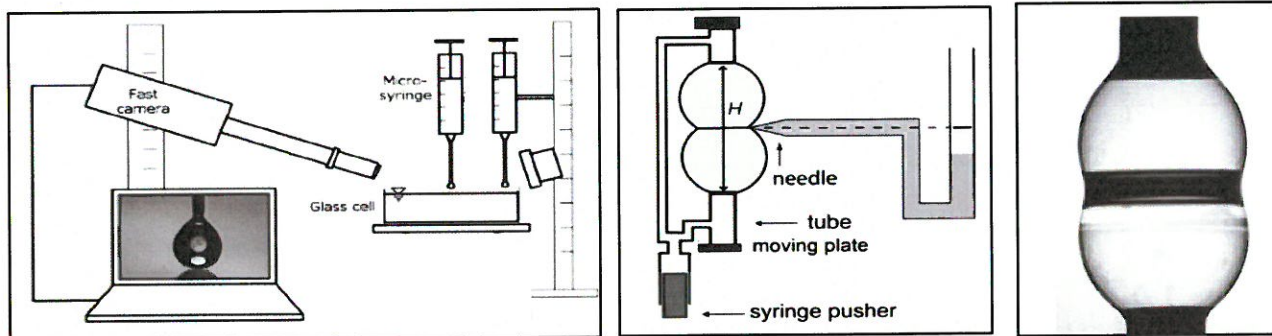

Figure 8. Experiment set-up diagram with glass cell and perpendicular syringes

Figure 9. Parallel approach plates with coalescing bubbles diagram

Figure 10. Photograph example of bubbles mid coalescence. The Plateu boarder is visible.

Experiment 2- The objective of this experiment is to determine the effectiveness of silica particles in entrapping and removing heavy metal particles from the fluid system, specifically the removal of copper II, nickel, zinc II and lead II. The laboratory set up consists of a 1m tall bubble column, and pump, which injected air into the column at a predetermined rate (Figure 11). The bubbles would rise to the surface, entrapping both silica particles and heavy metal contaminants dispersed in the solution. The process was filmed, and the height of the bubble foam analyzed from the footage to ensure accuracy, and the height of the foam was measured as a function of overall stability. The foam was then skimmed

from the surface of the solution, where gravitational separation was used to separate the recovered heavy metal particles from the silica particles. 100 mg/1000mL of each heavy metal type was individually dispersed in separate trial solutions, and recovery was gauged by how much metal could be recovered from the surface foam, and a totally system recovery percentage was calculated. The solutions tested in Experiment 2 were identical to those tested in Experiment 1, only with the addition of the previously specified heavy metal contaminants.
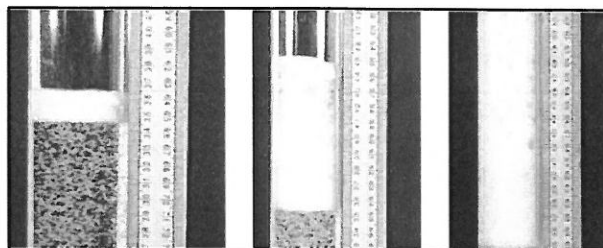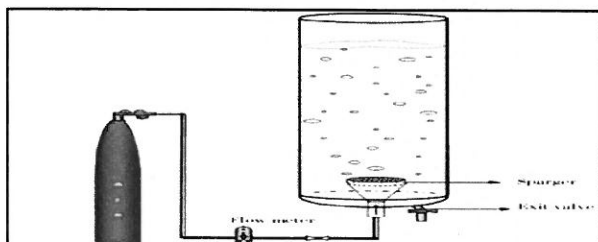

Figure 11. Experiment set up diagram with bubble column

Figure 12. Bubble column foam formation and decay
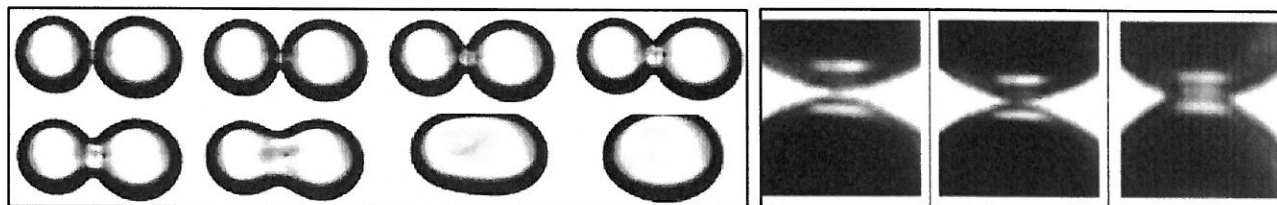
5. Results

5a. Experiment 1

Figure 12. Photographed bubble coalescence process in solution 2: replica wastewater solution and sodium lauryl sulfide (absent of silica)

Figure 13. Side view of bubble neck enlargement occurring during coalescence in solution 2: replica wastewater solution and sodium lauryl sulfide (absent of silica)
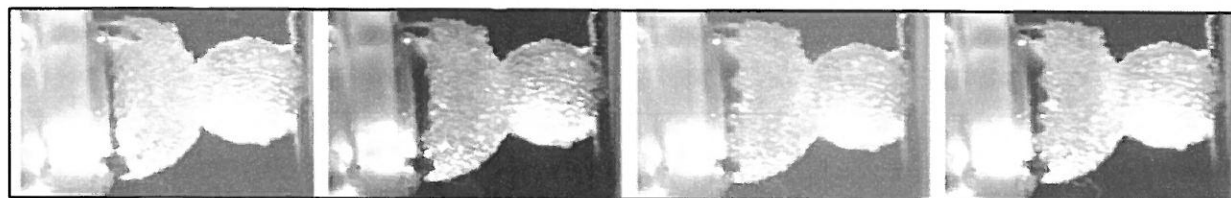

Figure 14. Silica particles coating two opposing bubbles at they approach one another. The bubbles are not coalescing due to the fact that the two interfacial films are not in direct contact, due to the coating of the silica
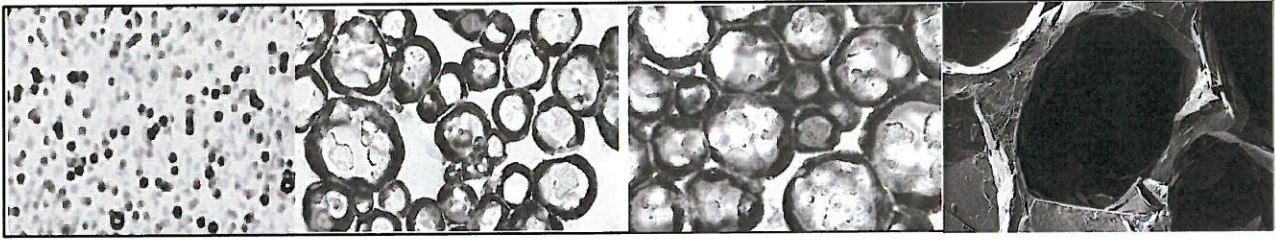
Figure 15. Silica particles from Solution 8: Replica wastewater solution- 6% Silica. *(Left to right)* Particles suspended in solution devoid of bubbles, 5x view of particles, 10x view of particles, 15x view of particles.
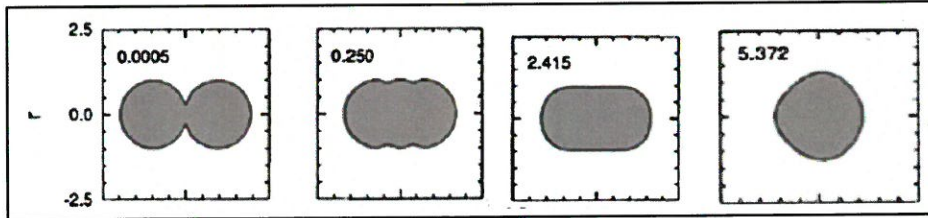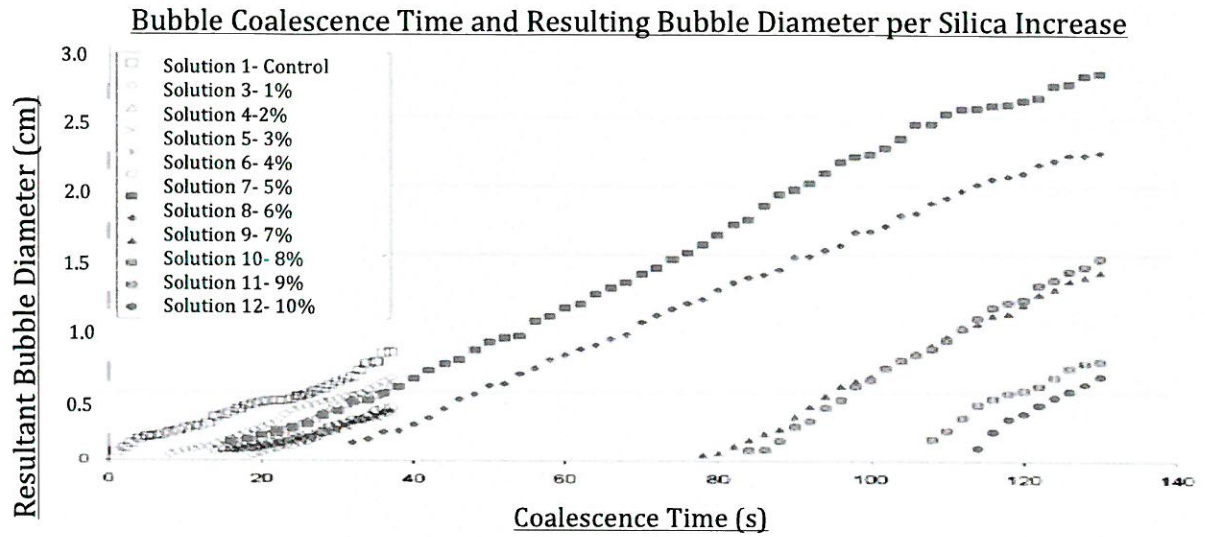

Figure 16. Computer imaging analysis done to observe the evolving coalescing bubbles from video footage recorded during experimentation of Solution 3: Replica wastewater solution-1% Silica. Diameter of the bubble and time elapsed is indicated.

Average Coalescence Times			
Solution	Composition	Mean Coalescence time/ 100 trials (s)	Standard deviation S^2 (s)
1	Replica wastewater solution	15.8	4.3
3	Replica wastewater solution-1% Silica	19.7	2.1
4	Replica wastewater solution- 2%Scilica	25.6	3.5
5	Replica wastewater solution- 3% Silica	31.6	2.8
6	Replica wastewater solution- 4% Silica	43.2	4.7
7	Replica wastewater solution- 5% Silica	53.6	5.6
8	Replica wastewater solution- 6% Silica	67.9	2.3
9	Replica wastewater solution-7% Silica	80.6	10.4
10	Replica wastewater solution- 8% Silica	100.8	4.9
11	Replica wastewater solution- 9% Silica	102.5	6.3
12	Replica wastewater solution- 10% Silica	118.9	3.9

Figure 17. Average coalescence time per 100 trials.

Bubble Coalescence Time and Resulting Bubble Diameter per Silica Increase

Figure 18. Coalescence time as related to resulting bubble diameter
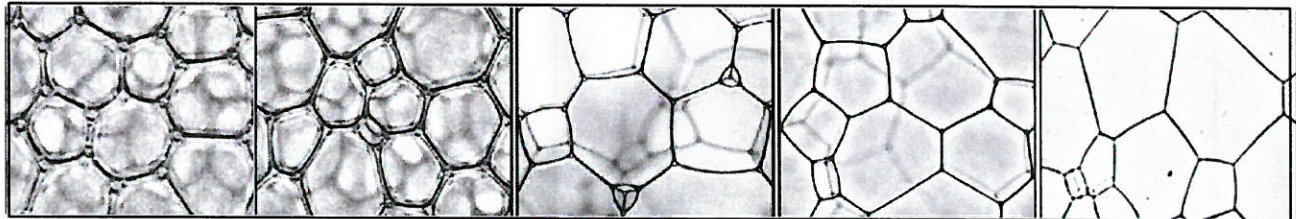
5b. Experiment 2

Figure 19. Foam evolution as a function of time illustrated by *solution 3:* 1% silica.
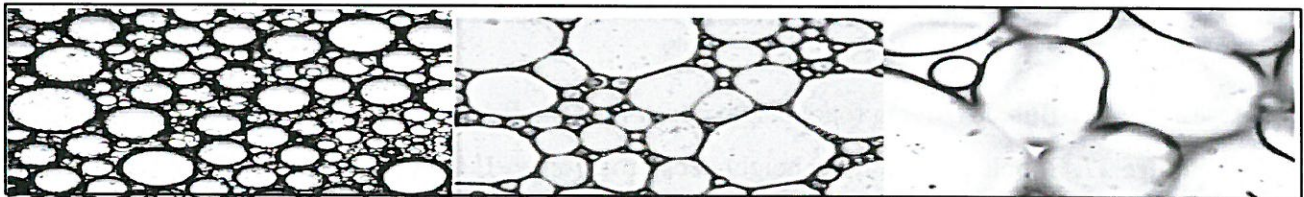

Figure 20. Slowed bubble coalescence as evidence by *solution 5*: 3% silica.
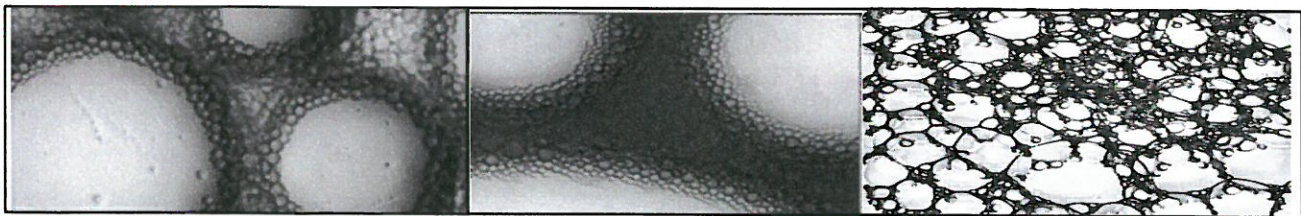

Figure 21. Sample photograph of most saturated silica solution with visible dispersed silica.

Figure 22. Silica particles adhered to the outer bubble films.

Figure 23. Silica and Pb(II) particles adhered to surface foam in bubble column
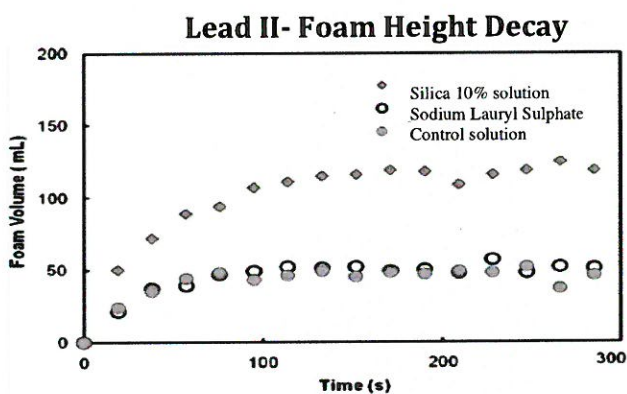
Figure 24. Bubble column foam height decay for Lead II in *solution 10*: 8% silica
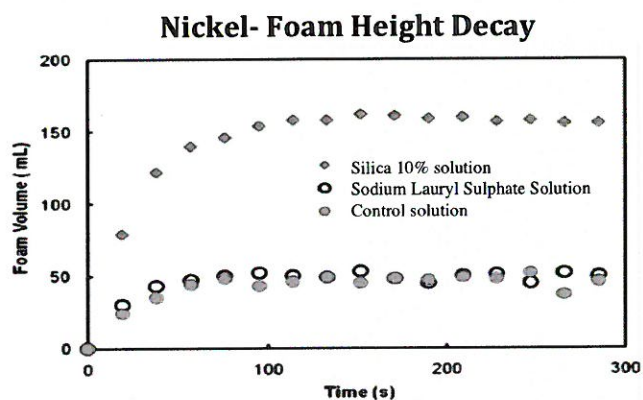
Figure 25. Bubble column foam height decay for Nickel in *solution 10*: 8% silica
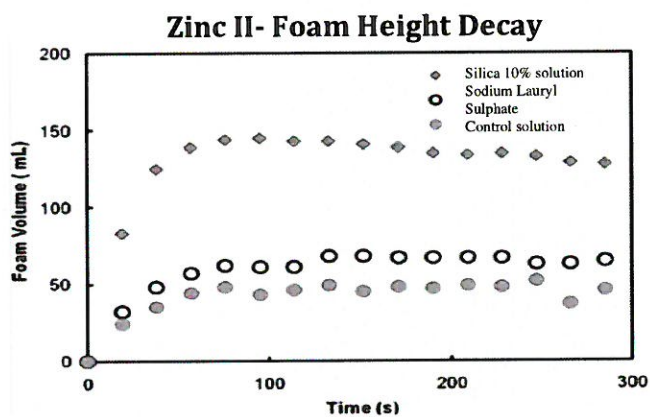

Figure 26. Bubble column foam height decay for Zinc II in *solution 10*: 8% silica
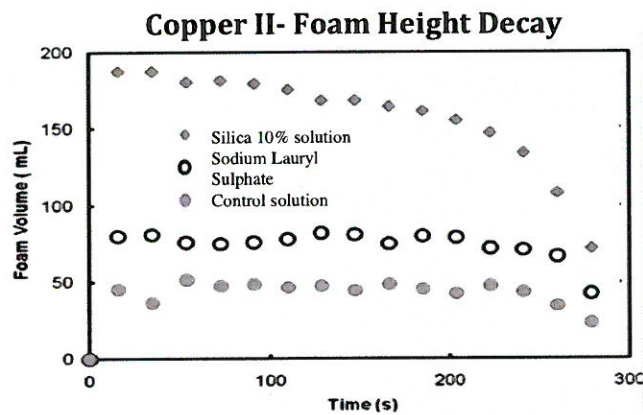
Figure 27. Bubble column foam height decay for Copper II in in *solution 10*: 8% silica

Average Heavy Metal Recovery Rate for Solution 12.

Heavy Metal	Toxicities	MCL defined by The Agency for Toxic Substances and Disease Registry (mg/L)	Amount remaining in solution after silica flotation	Average removal efficiencies from experimentation (amount put in vs amount recovered) (standard deviation) (mg/L) % per 100 trials	Standard Deviation s^2 (%)
Copper II	- Insomnia - Dermatitis - Nausea - Chronic asthma - Coughing Human carcinogen	0.250	0.200	89	2.8
Nickel	- Asthma - Coughing -Human carcinogen	0.200	0.200	93	3.5
Lead II	- Brain damage - Disease of the Kidney -Disease of the circulatory system -Disease of the nervous system	0.060	0.050	94	4.2
Zinc II	- Neurological Disease - Nervous system disease	0.800	0.700	91	2.8

Figure 28. Heavy Metal Toxicities and total removal percentages

6. Discussion:

Experiment 1: The times that coalescence took to occur increased drastically as the concentration of silica particles increased- up to 10% silica solution dispersal. The increase in coalescence time evidenced in this experiment would be a key to increasing overall foam film stability and thereafter, improving particle entrapment. It was observed that as concentration increased, fluid film drainage slowed, increasing coalescence time. This was observed at 5% silica solution concentration, through to 10% concentration. The dispersed silica particles in the solution began to adhere through surface tension to the outermost surface layer of the two opposing bubbles, forming a monolayer. With increased silica particle dispersal in the system, this monolayer thickened and inhibited the surface film of the two opposing bubbles from contacting. This is believed to have helped drastically increase the time that the entire coalescence process took to occur, from 15.8 seconds, to 118.9 seconds. It appears that the addition of silica particles into the solution counteracted the effect of film thinning and drainage between the two bubbles in contact with one another. There was little to no rupture of films at higher concentrations of silica particles in the solution. This was proven to be true, as the increase in silica concentration resulted in greatly increased coalescence times, with the *solution 12* having a 10% silica concentration and an average bubble coalescence time increase of 103 seconds from the initial control test. While the focus of this experiment has been on the interfacial effects of added particles, it is well

recognized that particles can also affect the bulk rheology, which is believed to also have contributed to increased bubble stability and increased coalescence times. Such bulk rheological changes may be responsible for the fact that the two opposing bubbles appeared to be well separated from each other, and that they remained stable even though they appeared to have been only sparsely covered with particles, such as with *solution 7*- 5% silica concentration.

Experiment 2: The heavy metal particles attached themselves to the surfaces of the bubbles and formed stable aggregates in the order of 2-3cm in size. It is apparent that foam decay occurred faster in the solutions with lower silica particle concentrations, than occurred with solutions with silica concentrations up to 10%. There was less visible space between the between the bubbles in solutions with higher concentrations, so it appeared that overall stability was optimized as a result of a similar bubble size being retained throughout the entirety of the column. A higher heavy metal recovery percentage resulted from solutions with increased silica particle dispersal. Solution 12, with a 10% silica particle dispersal proved to be the most effective solution at entrapping and removing heavy metal contaminants from the solution with a recovery rate of up to 94% for Lead II.

In the bubble column for all experiments, air was injected up to t=100 s and then the foam was left to decay. The rate that it decayed at was in direct correlation to the amount of silica dispersed in the solution, with solution 12 (10% silica particle dispersal) maintaining the greatest foam height for the greatest period of time. It was also shown that foam decay time changed in response to the type of heavy metal dispersed in the solution. There is no concrete answer for why this occurred as the particles were in the approximate same size range, however it was concluded that this phenomena could in part be due to the ion charge of the metal impacting the surface tension of the bubbles to which they are adhered.

The conclusion of the testing is that heavy metal ore removal efficiency could be improved if foam stability is properly manipulated and maintained. Experiment 1 proved that the coalescence time was significantly slowed with an increase in silica particles. Experiment 2 proved that silica particle attachment improved overall foam stability, as both foam height, longevity, and recovery yield increased alongside a greater increase in silica-solution dispersal. The technique of foam-particle-removal

showcased in these experiments could be a sustainable, efficient, cost effective solution to remove fine particulate contaminants from residual processing water without harmful chemical addition.

7. Conclusions:

1. Hydrophobic silica particles dispersed in the bulk liquid result in a significant increase in long-term foam stability. Upwards of 1 hour were recorded.

2. An otherwise non-foamable solution may be foamed through the addition of hydrophobic, colloidal silica particles. Specifically, the partially wettable particles can adsorb at the air/polymer interface and confer long-term stability on a water-surfactant foam.

3. It is possible to stabilize polymer foams by an interfacial mechanism (dispersed particles). This strategy of foam stabilization appears to be broadly generalizable; the chief requirement is that the stabilizing particles have a lower surface energy than the polymer being foamed.

4. It is possible to extend and improve the range of conditions under which foaming may be conducted through the dispersal of increased silica particles, upto 400 mg/L.

5. It is possible to use particulate additives for interfacial modification, rather than bulk rheology modification, to achieve effective heavy metal recovery, upwards of 91%. Furthermore, a possible advantage of this approach is that a low-surface energy additive such as silica is non-specific and may be an effective foam stabilizer in a wide variety of liquid solutions.

6. This process possesses some distinct advantages- low effluent metal concentrations, rapid operation, limited space requirements, production of virtually no volumes of sludge's, flexibility of application to various metals at various scales, and moderate costs

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