AGGLOMERATION AND LEACHING OF A CRUSHED SECONDARY SULFIDE COPPER ORE

by

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STATEMENT OF THESIS APPROVAL

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ABSTRACT

A crushed, secondary sulfide copper ore was provided by a large mining company. The agglomeration of the ore was studied in three phases. In phase one, a series of scoping experiments were conducted to characterize agglomerates produced in terms of agglomerate size distribution, electrical conductivity, hydraulic conductivity, and a test of binding strength. Using these tools and visual appearance, optimum agglomeration conditions were determined to be 13.0% moisture determined on a wet basis, 60 g/L H_2SO_4, 30% critical speed, and 3 minute agglomeration time.

Upon determination of agglomeration conditions, the study entered a second phase where agglomerates were subjected to leaching in order to determine the relation of leaching behavior to agglomeration conditions. Acid concentration was found to be important for the initial week of leaching. None of the agglomeration conditions affected copper recovery at 90 days of leaching.

Following leaching, acid-resistant agglomeration aids were evaluated to identify those with a potential to improve agglomerate stability and potentially, leaching behavior. Two cationic polymers were selected from an assortment of potential binders. Both polymers greatly improved agglomerate strength and hydraulic conductivity when used during agglomeration at a dosage of 0.5 kg polymer per tonne of ore.

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CHAPTER 1

INTRODUCTION

1.1 Brief History

Metals have been used by man for the past several thousand years. It is believed that the first metals to be used were copper, silver, and gold. These metals can be found in their native form, which allows the metal to be shaped immediately without any further refining[1]. Initially, metals were gathered in their native form with copper being the most useful due to the average size of native copper nuggets versus those of silver and gold. In addition, copper is much harder than silver or gold and would function better as a tool. As time passed, copper-containing oxide ores were discovered and smelted, which yielded elemental copper[2]. Perhaps by accident, tin made its way into the copper and the alloy bronze was formed. Bronze became very useful because it was stronger than either pure copper or tin. Iron was also obtained by smelting iron-containing ores with wood or charcoal, which yielded elemental iron. Most historians believe iron was discovered millennia after copper was being extracted from ore, while others suggest the two were being used around the same time[3].

1.2 Copper Production

While copper was one of the oldest metals discovered, it still remains important in modern times. World production of copper was 15,900,000 metric tonnes in 2009[4]. Because of its high electrical and thermal conductivity, copper is found in electrical devices, electrical transmission systems, and heat exchangers. The decreasing quality of copper ore has encouraged the development of new technologies to recover copper. There are currently two major methods for producing copper, one using pyrometallurgy and the other using hydrometallurgy.

Around 80% of the world's copper is produced using pyrometallurgical techniques. Production via pyrometallurgy requires several steps. Ore that is initially mined typically has a concentration of 0.5-1% copper[5]. Smelting this small concentration of copper would require enormous amounts of energy and would not be cost effective. The copper must first be upgraded. This process is typically performed using flotation, which produces a copper-rich slurry of around 30% copper[6]. This slurry is smelted to produce an even higher grade copper matte. Subsequent converting and fire refining occur to further upgrade the copper content. The final molten copper is cast into anodes and subjected to electrorefining where copper is transferred from the anodes to plate as cathodes. The plated copper has typically less than 20 ppm impurities, which is suitable for most electronics applications[7].

1.3 Heap Leaching

While the majority of the world's copper is produced using more traditional pyrometallurgy, approximately 20% is produced using hydrometallurgy. Heap leaching is the primary method for hydrometallurgical copper extraction. Heap leaching allows copper extraction from ores without the need to grind down to dumps; however, current leaching methods involve building engineered heaps to optimize extraction[8]. During heap leaching, ore is stacked onto a protective pad where it is irrigated with a leaching solution. The solution percolates through the heap and dissolves the copper-containing minerals (often with the aid of microorganisms). The copper-containing pregnant leach solution (PLS) is collected and through solvent extraction, the copper is transferred from the PLS to an electrowinning electrolyte solution. The copper is subsequently deposited as cathodes with purity similar to that produced by electrorefining. A flow sheet illustrating copper production through heap leaching is shown in Figure 1.1.

Before a heap can be constructed, a site must first be prepared for leaching. A geomembrane barrier, often 1.5 mm polyethylene, is usually put in place to allow for PLS collection and to prevent the solution from entering the environment[9,10]. The barrier must be able to withstand the force of the ore that will be placed on it. Heaps can be designed in a natural valley, on a dedicated pad intended for multiple lifts, or using on/off pads where leached ore is removed, disposed of, and replaced with fresh ore[10]. A pond or several ponds are designed near the heap to allow for collection of the PLS where it will be drawn from for solvent extraction.

After the leaching site has been prepared, an aeration system may be put in place if leaching will be performed on sulfide ores. Heap leaching is typically performed on copper oxide and secondary copper sulfide ores. While copper oxide ores are readily leached by dilute sulfuric acid, copper sulfide ores are not. Leaching of copper sulfide ores is accomplished by utilizing microbes, which

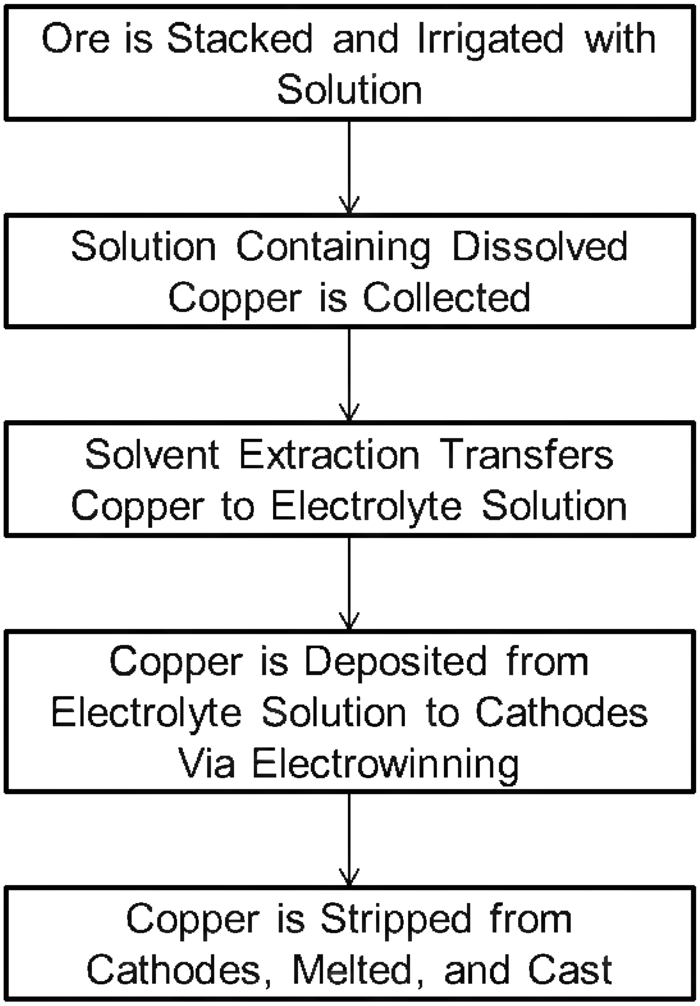

Figure 1.1 Flow sheet of copper production from heap leaching.

convert ferrous iron in the ore to ferric iron. Ferric iron reacts with the copper sulfide mineral to form copper ion, which is then recovered from solution[11,12]. In order for the microbes to grow, it is often necessary to supply the heap with air. Traditionally, heaps were designed using natural air infiltration. Forced air is now more commonly used and is important for supplying CO_2, which serves as a carbon source for microbes, and for supplying O_2, which is used to oxidize ferrous iron[12]. Copper production from sulfide ores is related to the amount of oxygen available in the ore body[13]. Many operations utilize piping at the base of the heap and fans to supply air[14]. Without utilizing an aeration system, heaps would be limited in height and area[15].

1.4 Agglomeration

Prior to stacking crushed copper ore for leaching, it is often treated through a process known as agglomeration. In copper leaching, ore agglomeration serves two main purposes: it introduces acid or ferric ion into the ore and improves the permeability in the subsequent heap. As acid or ferric is introduced at the top of a heap during leaching, it is consumed, resulting in reduced extraction rates in the lower portion of the heap. Initial mixing of acid or ferric into the ore prior to stacking allows copper to begin to dissolve even before the irrigation solution reaches that ore[16,17]. Agglomeration is needed to improve heap permeability when a significant amount of fine particles are present in the ore. Fine particles tend to be transported down the heap and can clog pores, which leads to ponding, a situation where solution builds up, and channeling, a condition where solution flows down a specific series of paths[18,19].

Figure 1.2 shows an example of channeling compared to desired flow. Both of these conditions can cause reduced recovery or increase the amount of time required to effectively leach the ore[18]. When ore contains at least 5% of -75 µm fines, agglomeration is suggested. When the content of -75 µm fines is greater than 10%, a binding agent should be used[20].

Agglomeration is typically referred to as a process of binding small particles to other small particles or to larger particles. This has been described as occurring via four methods: solid bridges, liquid bridges, mechanical interlocking, and attraction forces[17,21]. Currently, the most common binder for copper ore is sulfuric acid, which forms liquid bridges caused by capillary forces between adjacent particles. In gold heap leaching, Portland cement type II is mixed with the ore to create agglomerates. The chemical reaction which occurs with the cement curing forms strong solid bridges[21].

Agglomerates are typically formed by mixing crushed ore with a binder and leaching solution. Mixing is usually done in continuously fed drums, although some mining sites utilize a series of conveyor belts. As the particles become saturated with solution and contact other particles, capillary forces create bonds, which allow the agglomerate to form. When utilizing conveyor belts, this process typically occurs at transfer points, whereas in drum agglomeration, this is a continuous process as the mixture rolls in the drum[17,21].

When agglomerates formed using liquid bridges are leached, the liquid bridges can fail and the agglomerates can break apart. Other binders have been suggested for use in agglomeration. While Portland cement has been successful

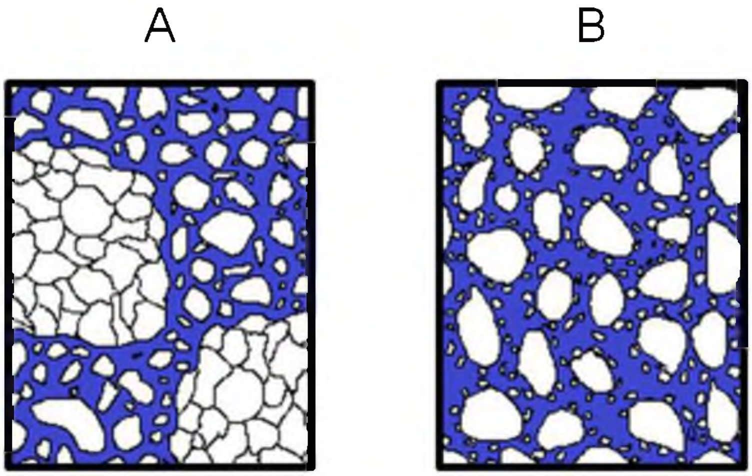

Figure 1.2 Proposed solution flow for ore bodies. A) An example of channeling where ore is not interacting with the leaching solution. B) Desired flow pattern where all ore is interacting with leaching solution.

for leaching gold and silver, the low pH conditions of copper leaching would easily break the solid bridges formed[22]. As a result, binders would need to be developed which are both acid resistant and, in the case of copper sulfide leaching, not harmful to the microorganisms present. The binders would also need to be relatively inexpensive to justify their use in copper leaching. Binders which have been tested with copper ore agglomeration include polyvinyl acetate, tall oil pitch, stucco, various polyacrylamides, among others[22–24]. Binders such as stucco and certain polyacrylamides appear to have improved agglomerate strength, although different ores may behave differently from those tested[23,24]. Binders will be investigated to see if they can improve the behavior of the ore I will be working with. Binders will be investigated to see if they can improve the behavior of the ore I will be working with.

1.5 Agglomerate Characterization

Regardless of how agglomerates are made and what binders they are made with, the agglomerate needs to be characterized to determine its stability, relative behavior to agglomerates made under varying conditions, and overall benefit to the leaching process. While some tests have been suggested and implemented in research settings, the quality of most heap leaching agglomerates is determined subjectively by operators[19].

Laboratory tests to determine the strength of an agglomerate include a soak test, attrition test, and bulk density test. The soak or dip test is performed by placing agglomerates on a screen and allowing them to soak in solution for a set amount of time. The strength of the agglomerate is related to the amount of material remaining on the screen relative to the amount passing through the screen[22,23,25]. The attrition test subjects agglomerates to mechanical forces which break down weaker agglomerates[19]. Bulk density tests show how well agglomerates hold their structure when subjected to wetting[22,25]. Electrical conductivity has also been used to correlate other test results and identify what amount of moisture produces the best agglomerates[19].

1.6 Project Objectives and Thesis Organization

In conducting the research, specific goals were determined based on objectives established in conjunction with AMIRA project P986. The project objectives were as follows:

- Identify which factors affect agglomerate formation
- Evaluate how varying agglomeration conditions affects leaching behavior
- Identify potential agglomeration aids to increase agglomerate strength

One of the project sponsors provided a crushed, secondary sulfide copper ore to be studied. This ore was studied in three phases. In phase one, a series of scoping experiments were conducted to characterize agglomerates produced with this ore using a set of quality control tools. Using these tools, optimum agglomeration conditions were determined. Upon determination of the effect of agglomeration conditions on agglomerate formation, the project entered a second phase where the agglomerates were subjected to leaching in order to determine the relation of leaching behavior to agglomeration conditions. Following leaching, acid-resistant agglomeration aids were evaluated to identify those with a potential to improve agglomerate stability and potentially leaching behavior.

1.7 Ore Characteristics

The ore provided primarily contains copper sulfides but has a small fraction (about 15% of copper content) of copper oxides. The gangue material is largely composed of silicates including quartz, orthoclase, muscovite, and a large amount of kaolinite. Of the feed material, 18.5% will pass a 75 μm screen.

CHAPTER 2

SCOPING EXPERIMENTS

2.1 Experimental

2.1.1 Sample Preparation

A large mining company provided two tonnes of secondary sulfide copper ore to examine in the project. The ore was screened and bagged prior to delivery to the university. The company provided a typical feed size distribution for material from the mine sampled, shown in Table 2.1, which was to be investigated and that distribution was reproduced from the supplied ore.

2.1.2 Liquid Retention Capacity

Liquid retention capacity (LRC) provides an indication of how much liquid is required to saturate the capillaries of a specific ore[26]. This was measured using a PVC column with an internal diameter of 24.9 cm, which had a rigid water permeable cloth attached to the bottom. Approximately 2 kg of ore was added to the column and the device was placed in a dish filled with water. Water was periodically added as needed to maintain a depth of about 1 cm. Once the top of the ore was completely wetted, the device was removed and excess water was allowed to drip for about 10 minutes. The amount of water absorbed by the ore

Table 2.1 Size distribution of ore to be tested.

Top Size (mm)	Bottom Size (mm)	Percent of Ore
	19.1	9.00%
19.1	12.7	24.00%
12.7	6.4	24.00%
6.4	1.7	13.50%
1.7	0.50	4.37%
0.50	0.15	0.94%
0.15		24.19%

was then determined. The LRC was determined to be 23.6%, as calculated using equation 2.1.

$$\text{LRC }(\%) = \frac{\text{Mass}_{\text{Water}}}{\text{Mass}_{\text{Ore}}} \tag{2.1}$$

2.1.3 Agglomeration

The following factors on agglomeration were considered in the scoping experiments:

1. Mixing Time
2. Drum Rotational Speed
3. Moisture Content
4. Acid Concentration

Mixing time was fixed at 3 minutes in the first set of experiments, and then to examine the effect of time, it was varied at 6 and 9 minutes. Shorter mixing times were not attempted due to equipment limitation and the rate of solution addition needed. Mixing time in industrial agglomeration is typically between 1 and 3 minutes. As the amount of time was increased, the flow of each solution was adjusted to deliver the proper volumes over a period of 1/3 of the mixing time.

The drum speed used for crushed ore agglomeration is typically between 30 to 50% of the critical speed, where the critical speed is the point where inertial forces overcome gravitational forces and the feed material follows the drum rotation. In this study, 30% of the critical speed was used in the first set of experiments and then 20% and 50% of the critical speed were considered to observe the effect of drum speed. The critical speed (NC) of an agglomeration drum is found by using equation 2.2.

$$NC=\frac{42.3}{\sqrt{D}} \tag{2.2}$$

where D is the internal drum diameter in meters.

A plastic drum was used as the agglomerator. The drum has dimensions of 26.7 cm diameter and a length of 36.2 cm. The agglomerating drum was equipped with 4 lifters of 0.5 cm thickness and 20 mm width, which ran the length of the drum and were equally spaced around the drum's internal circumference. The critical speed of the drum was calculated as 81.9 rpm. Thus, 40.9, 24.6, and 16.4 rpm were used to produce 50%, 30%, and 20% of critical speed, respectively.

For the initial agglomeration batch, the drum was filled with 4 kg of sample (copper ore) which resulted in about 12.8 % volume fill of the drum. After completing the first experiment, it was found that more agglomerates would be needed for the quality control tests. All additional tests were performed with 5 kg batches, which resulted in about 15.9% volume fill of the drum. To achieve homogeneity, the sample was mixed for 15 minutes in the drum at the speed to be used in the specific experiment prior to solution addition.

Deionized water and concentrated sulfuric acid were applied simultaneously during the first one-third of the mixing time using peristaltic pumps and separate hoses for the respective solutions. The moisture contents selected to study were 16.7%, 14.5%, 13.8%, 13.0%, and 9.1%. At a later point, 11.5% was also tested and 13.0% was retested. Moisture content is calculated using equation 2.3.

$$\text{Moisture content } (\mu,\ \%) = \frac{\text{Mass}_{\text{wet}} - \text{Mass}_{\text{dry}}}{\text{Mass}_{\text{wet}}} \times 100 \qquad (2.3)$$

where $\text{Mass}_{\text{wet}} - \text{Mass}_{\text{dry}}$ is the weight of the solution used in the agglomeration and Mass_{wet} is the weight of the ore samples after solution has been added.

The acid concentration initially used for moisture scoping experiments was 25.6 g/L. Upon completion of the moisture tests, the condition which produced agglomerates with the best visual appearance was 13.0%. This condition was tested with various mixing times and drum rotational speeds as described above, and various acid concentrations. In order to investigate the effect of acid concentration on agglomeration, additional acid concentrations of 50 g/L, 100 g/L, and 200 g/L were tested at 13.0% moisture.

The amount of the acid solution required for agglomeration was first calculated, and then the acid solution volumes were used to calculate the volume of sulfuric acid and water due to the fact that the acid and water were applied to the drum via different hoses. The amount of acid solution in mL used in an experiment was calculated via equation 2.4.

$$D=\left(\frac{\frac{(A-B)}{100}}{\rho_{\text{acid solution}}}\right)(C) \tag{2.4}$$

where A = Target moisture content (%)

B = Natural moisture content (%); 2.5% for the studied copper ore

$\rho_{\text{acid solution}}$ = Density of the acid solution (g / mL)

C = Ore amount (g); 5,000 g per batch

D = Acid solution volume to be added (mL)

The density of acid solution used in equation 2.4 varied depending on the desired acid concentration of the final solution. The density of the acid solution was obtained via a relationship between the sulfuric acid concentration and density provided in the open literature. The specific acid strengths examined in this study and their calculated densities are shown in Table 2.2. The obtained acid solution volume was used to calculate the concentrated sulfuric acid and water volumes via equations 2.5 and 2.6. Tables 2.3 and 2.4 show the volumes of concentrated sulfuric acid and water used to agglomerate 5 kg batches of copper ore during phase one.

$$\text{Concentrated sulfuric acid (F, mL)}=\left(\frac{D\times E}{1840\times G}\right) \tag{2.5}$$

$$\text{Water (mL)}=\left(\frac{D\times\rho_{\text{acid solution}}-\frac{F}{1.840}}{\rho_{\text{pure water}}}\right) \tag{2.6}$$

Table 2.2 Acid solution concentration, weight percentage, and density.

Sulfuric acid solution concentration (g/L)	Sulfuric acid solution wt% (%)	Sulfuric acid solution density (g/mL)
25.6	2.5	1.015
50	4.8	1.031
100	9.4	1.062
200	17.8	1.123

Table 2.3 Volume (mL) of concentrated sulfuric acid used to agglomerate 5 kg of copper ore at different moisture content; the number in the parenthesis is kg acid per tonne of ore used.

Moisture Content (%)	Acid solution concentration (g/L)			
	25.6	50	100	200
9.1	5.4 (2.0)	-	-	-
11.5	7.5 (2.8)	-	-	-
13.0	8.9 (3.3)	17.0 (6.3)	33.1 (12.2)	62.5 (23.0)
13.8	9.6 (3.5)	-	-	-
14.5	10.4 (3.8)	-	-	-
16.7[b]	10.0 (4.6)	-	-	-

[a] The number in parenthesis is kg acid per tonne of ore used

[b] This condition was prepared with a 4 kg batch

[-] Indicates conditions not tested

Table 2.4 Volume (mL) of water used to agglomerate 5 kg of copper ore at different moisture content.

Moisture Content (%)	Acid solution concentration (g/L)			
	25.6	50	100	200
9.1	366	-	-	-
11.5	513	-	-	-
13.0	605	591	563	515
13.8	659	-	-	-
14.5	708	-	-	-
16.7[a]	684	-	-	-

[a] This condition was prepared with a 4 kg batch

- Indicates conditions not tested

where D = Acid solution volume to be added (mL)

E = Acid concentration desired (g/L)

F = Concentrated sulfuric acid volume to be added (mL)

G = Purity of acid (Assumed to be 96%)

For each condition studied, three agglomeration batches were prepared in order to obtain 15 kg of the agglomerates. Approximately 1 hour after agglomeration, 600-700 g of agglomerate from each batch, obtained by a cone and quartering method, was allowed to air dry to determine size distribution. After cone and quartering, the agglomerate not designated for feed size determination was mixed with respective samples from the other batches. The remaining agglomerates were used for electrical conductivity, permeability, and TBS testing.

2.1.4 Size Distribution

Agglomerate size distributions (ASDs) were determined by hand screening. The smallest sieve size used was 1.0 mm. About 600-700 g of agglomerates were collected using a cone and quartering method[27]. The samples were allowed to air dry for a minimum of 48 hours. A series of sieves from 31.5 mm to 1.0 mm was used in the experiments. The samples were gently shaken by hand to screen while avoiding agglomerate breakage. The agglomerates were collected from each sieve and weighed. Upon determination of the size distribution, the D_{50} and D_{10} can be determined where these values indicate the size of screen that 50% and 10% of the agglomerates will pass through, respectively.

2.1.5 Electrical Conductivity

The electrical conductivity measurement was implemented because in has been reported that one copper operation uses it to control the amount of water added during agglomeration[19]. As water is added, conductivity is expected to increase gradually until the agglomerates are saturated. At this point, the conductivity is expected to increase dramatically.

The electrical conductivity was measured approximately 1.5 hours after agglomeration. To measure electrical conductivity, about 650 g of agglomerated sample were placed in the electrical conductivity measurement cell. The cell is dried prior to introduction of the sample to avoid changes in output voltage due to extra moisture. Six different direct current voltages were applied to the circuit with a maximum voltage of 3 V to avoid corrosion of the stainless steel electrode in the cell. The measured ampere was recorded and then plotted versus the applied voltage. Resistance values are obtained as the slope of the voltage against current graph. The resistivity of the bed of agglomerates is calculated using equation 2.7.

$$p=\left(\frac{R\times A}{l}\right) \tag{2.7}$$

where p = resistivity (Ωm), R = resistance (Ω), A = area of electrode (m^2), and l = distance between electrodes (m). Conductivity is simply the inverse of resistivity and is expressed in Siemens per meter ($S\ m^{-1}$).

After finishing the first conductivity tests when no mechanical load is applied, a second conductivity test was performed with a weight of 18.2 kg applied (this simulates the weight that the agglomerates face at about 1.5 m into the real heap). The amount of load was calculated from the average bulk density of the agglomerates and area of the applied force. The conductivity measurement was performed the same as when no load was applied. A schematic of the measurement device is shown in Figure 2.1.

2.1.6 Permeability

Between 10 and 11.5 kg of agglomerates were placed inside the 20.0 cm inner diameter test cell. The cell was flooded, and then water was allowed to flow downward through the test cell. A head change through the agglomerates was obtained by reading two manometers. A schematic of the permeability apparatus is shown in Figure 2.2. The values of area and the height of the agglomerated ore bed were measured and therefore, the hydraulic permeability was calculated using equation 2.8 (Darcy's Equation). The system must be in equilibrium, $Q = Q_{in} = Q_{out}$, for Darcy's law to be valid for this experiment.

$$Q=AK\frac{\Delta h}{L} \tag{2.8}$$

where Q = volumetric flow rate (cm^3 / s)

A = flow area perpendicular to L (cm^2)

K = hydraulic conductivity (cm/s)

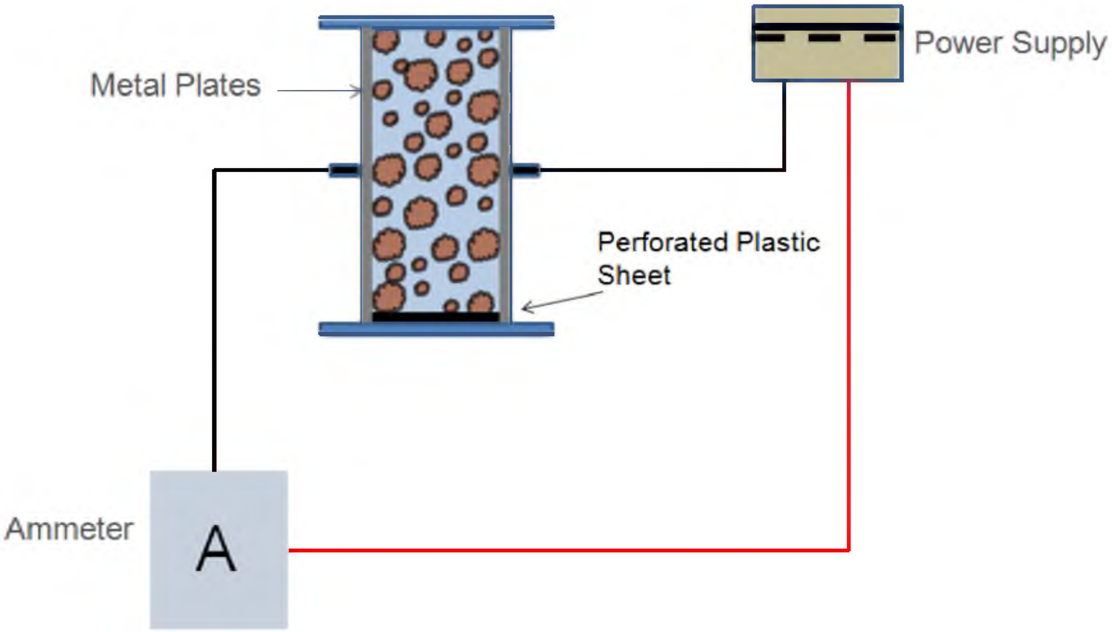

Figure 2.1 Schematic of electrical conductivity apparatus. A complete circuit is formed where electricity is passed through an agglomerate bed at a known voltage and current is measured to determine conductivity.

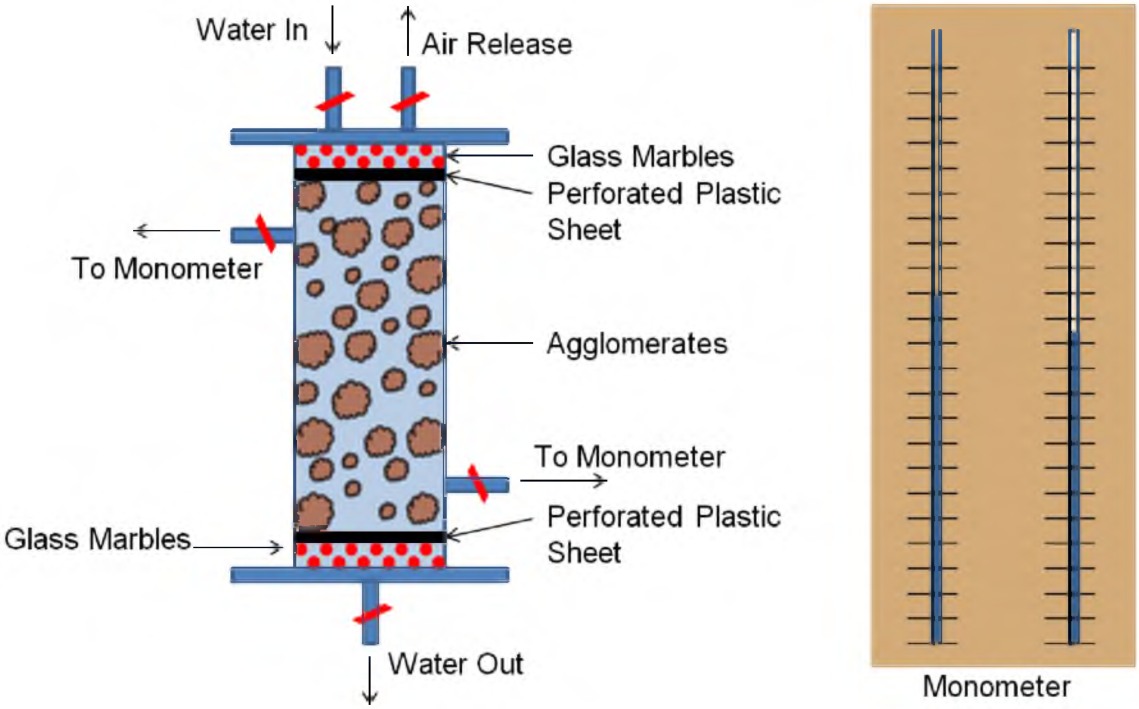

Figure 2.2 Schematic of permeability apparatus. Permeability of the agglomerate bed is determined by measuring flow rate and pressure drop and using Darcy's equation.

L = flow path length (cm)

Δh = change in hydraulic head (cm)

The permeability (k) of the ore body can be calculated using equation 2.9.

$$k=K\left(\frac{\mu}{\rho g}\right) \qquad (2.9)$$

where k = Permeability (cm^2)

K = Hydraulic conductivity (cm/s)

p = Solution density (water); $1000\ \frac{kg}{m^3} = \frac{kg}{1000\ cm^3}$

g = Earth gravitational constant; $9.81\frac{m}{s^2} = 981\frac{cm}{s^2}$

μ = Solution viscosity (water);1 centipoise=0.001 Pa(s)=$0.001\frac{kg}{m \cdot s}$=$0.00001\frac{kg}{cm \cdot s}$

Therefore, permeability (k) is equal to $k=K\left(\frac{\mu}{\rho g}\right)=K\left(\frac{0.00001}{\frac{981}{1000}}\right)=\left(1.02\times10^{-5}\right)K$

It is convenient to work with hydraulic conductivity rather than permeability at times. It should be noted that trends in hydraulic conductivity and permeability will be equivalent.

2.1.7 Test of Binding Strength (TBS)

The test of binding strength was developed by our research group. It is performed by immersing agglomerates in water and observing the time required for disintegration. For each condition, agglomerates of about 4 cm, 2 cm, and 1 cm diameter were selected and photographed. Each agglomerate was immersed in water and a timer was started. Once the agglomerate had visually disintegrated, the timer was stopped and the time recorded.

2.2 Results and Discussion

2.2.1 Agglomerate Size Distributions

2.2.1.1 Effect of Moisture and Acid

One of the objectives of agglomeration prior to heap leaching is to reduce the amount of fines in the ore. Reducing fines can enhance solution flow, reduce channeling, increase recovery rate, and reduce leaching time. In the initial scoping experiments, different moisture contents were examined to observe its effect on agglomeration.

The effect of different moisture contents at the same acid concentration on the resulting ASDs is shown in Figure 2.3. As expected, the sizes of agglomerates were coarser as moisture content increases. As the moisture content increases, the volume of liquid also increases. An increase in liquid volume was expected to increase the ability to form liquid bridges and thus increase agglomerate sizes. The agglomerate particle size as characterized by D_{50} was in the range of 15 mm to 28 mm.

The ASDs for agglomerates produced with varying acid concentration at constant moisture content is shown in Figure 2.4. There appears to be an inverse relationship between agglomerated particle size and acid concentration. The volume of liquid plays an important role in the formation of agglomerates. As the amount of liquid added decreases (e.g., increasing acid concentration at the same moisture content), the agglomerates become finer.

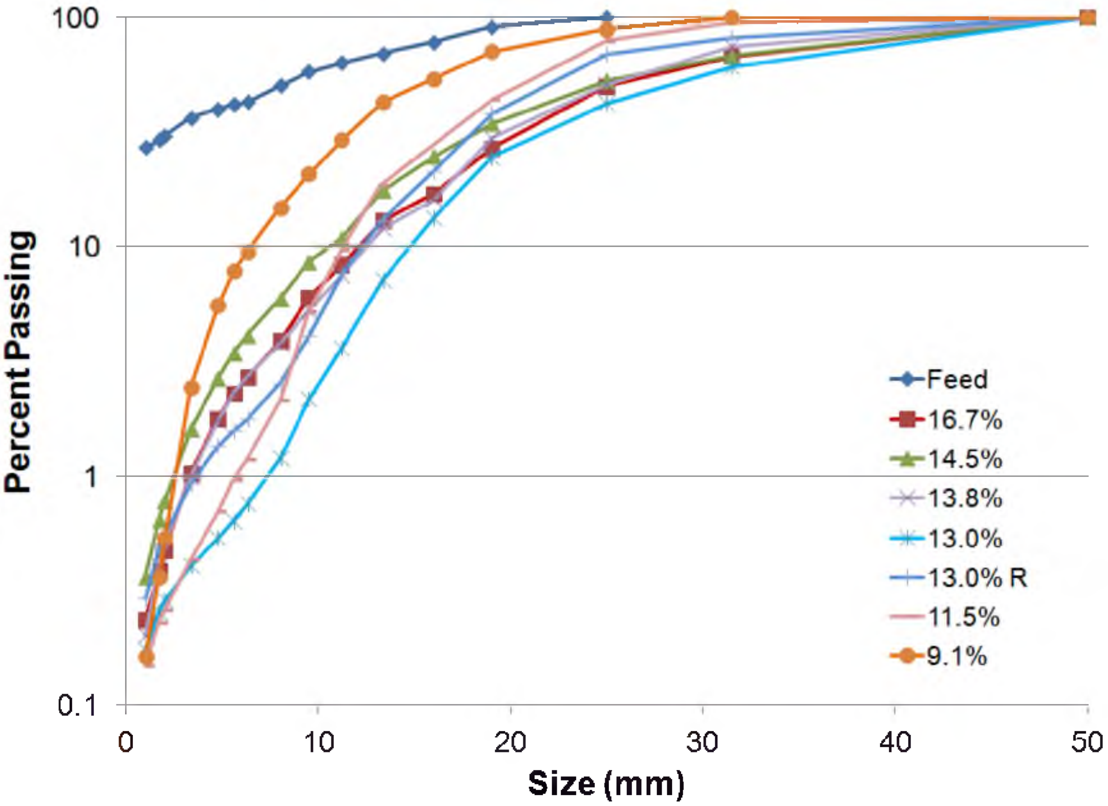

Figure 2.3 ASDs at various moisture contents, 25.6 g/L acid concentration, 30% NC, and 3 minute mixing time. R indicates a repeat of conditions.

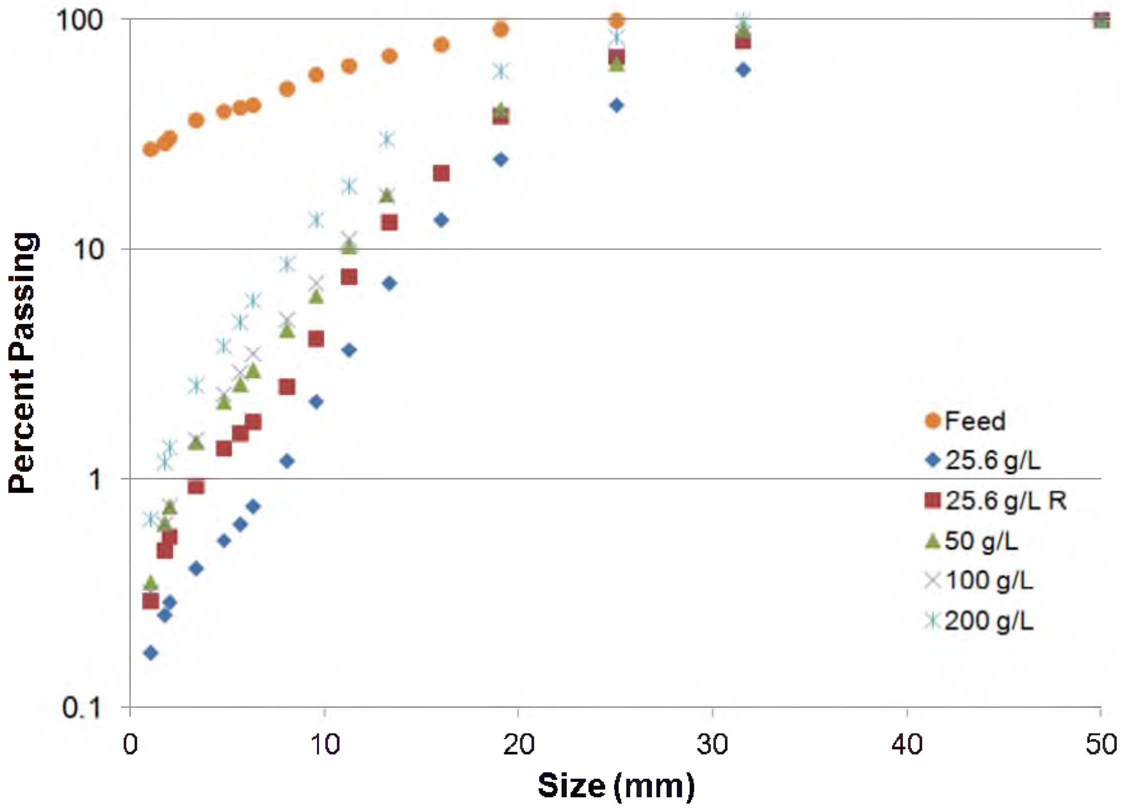

Figure 2.4 ASDs at 13% moisture, 30% NC, 3 minute mixing time, and various acid concentrations. Legend displays g H_2SO_4/ L solution. R indicates a repeat of conditions.

A relationship between liquid volume added and agglomerate size was observed as seen in Figure 2.5. As a greater volume of solution is added, the visual appearance of the agglomerates changes from small and dry to moderately large and completely coated to becoming very large and sticky. The sticky nature of the agglomerates is likely due to the high fraction of kaolinite. These observations can be seen in Figure 2.6. The optimal moisture content was selected as 13.0% because agglomerates produced at this moisture were completely wetted but not sticky.

The Liquid Retention Capacity test indicated an optimal moisture of 23.6% (dry basis), which calculates to 19.1% (wet basis as used in agglomeration). This value is higher than the optimal of 13% found during agglomeration. Perhaps, the difference between the moisture content predicted by the LRC test and what was actually observed is due to the difference in the amount of time the moisture is interacting with the ore. In the LRC, the ore is exposed to water for several hours versus 3 minutes in agglomeration. During the LRC test, Vethosodsakda et al. observed a continued increase in moisture uptake over a 90 minute period for an ore with fine particles[26]. The difference could also be related to the stickiness of the ore, which was factored into the optimal moisture of agglomeration.

2.2.1.2 Effect of Drum Speed and Mixing Time

In the second part of the scoping experiments, the moisture content and acid concentration were held constant at 13.0% and 25.6 g/L H_2SO_4,

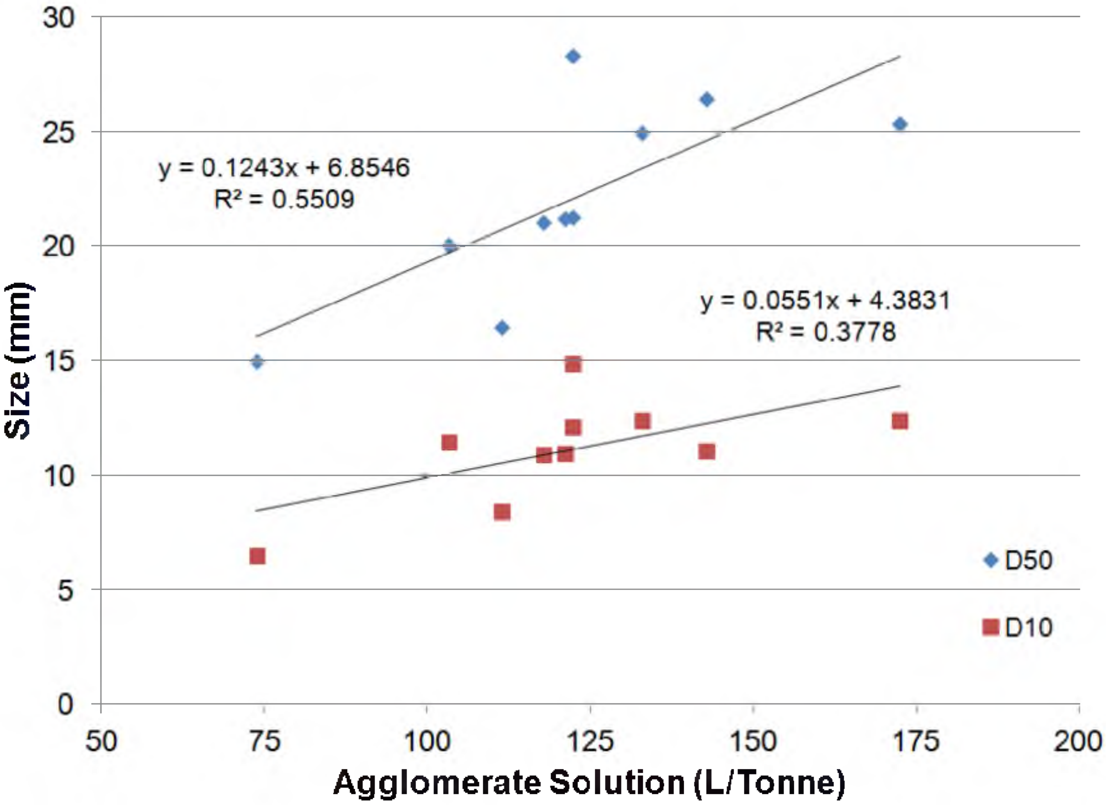

Figure 2.5 Relationship between volume of solution added and agglomerate particle size.

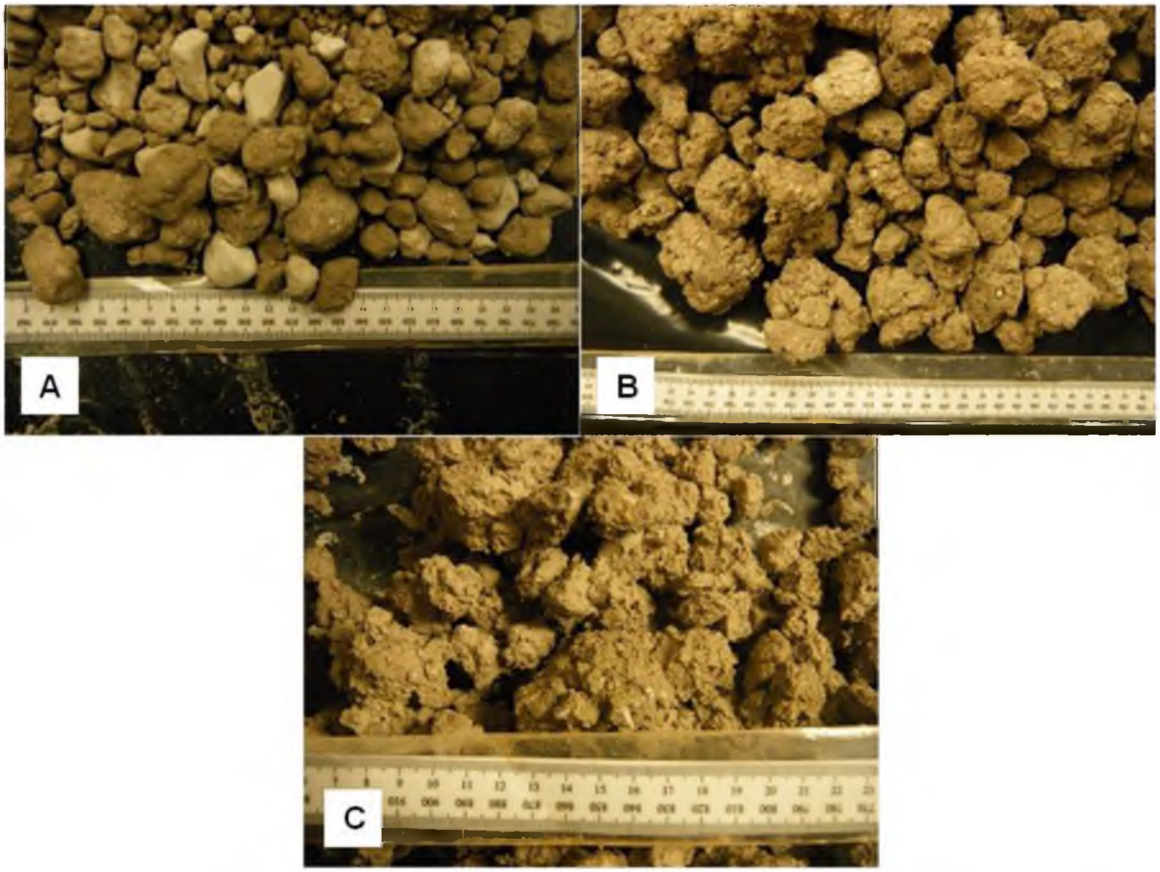

Figure 2.6 Pictures of agglomerates (A) 9.1% moisture content –too dry (B) 13.0% moisture content –good looking and, (C) 16.7% moisture –too wet.

respectively, and mixing time and drum speed were examined. With a 3 minute mixing time, the drum rotation speed was examined from 20 to 50% of drum critical speed. The ASDs produced at different drum rotation speeds are shown in Figure 2.7. The agglomerates produced at 20% and 30% critical speed are similar; however, the agglomerates produced at 50% critical speed are slightly smaller.

Mixing times were varied from 3 to 9 minutes while maintaining a 30% NC drum speed, 13.0% moisture, and 25.6 g/L H_2SO_4. The D_{10} and D_{50} of these agglomerates are summarized in Table 2.5. The agglomerates tend to be less coarse when produced with longer mixing times, as is observed by smaller D_{10} and D_{50} values.

The agglomerate size distribution curves at different retention times are shown in Figure 2.8. In general, for the conditions examined, mixing time had minimal effects on the fine end of the resulting particle size distribution. Longer mixing times appear to have produced less coarse agglomerates. This may indicate breakage of the agglomerates.

2.2.2 Electrical Conductivity

For each agglomeration condition, a sample of the combined batches was evaluated for electrical conductivity. The measured conductivities and a conductivity ratio are provided in Tables 2.6 and 2.7. The conductivity ratio is the measured conductivity divided by the literature conductivity of the acid solution used. This value is useful when comparing data where acid concentrations are different.

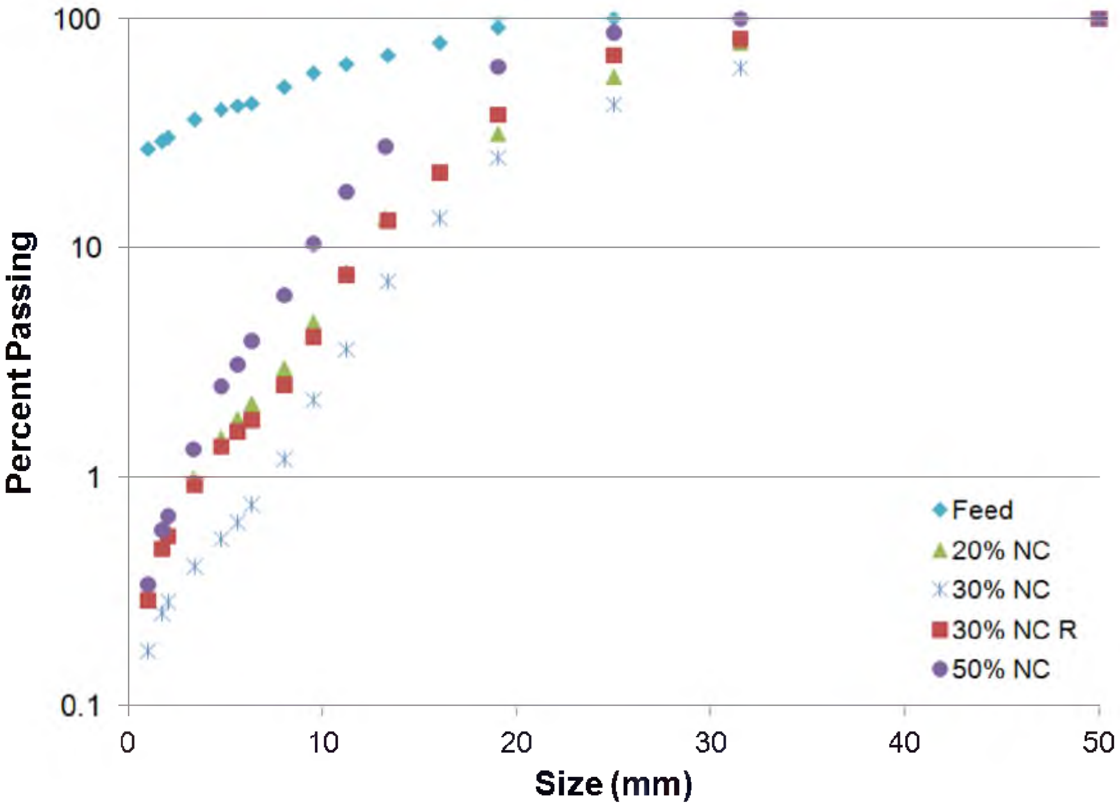

Figure 2.7 The ASDs produced at different drum rotation speeds using 13.0% moisture and 25.6 g/L H_2SO_4. R indicates a repeat of conditions.

Table 2.5 D_{10} and D_{50} of agglomerates at different retention times.

Mixing Time (min)	D_{10}	D_{50}
3	14.9	28.3
3 (Repeat)	12.1	21.3
6	11.4	20.6
9	11.0	20.5

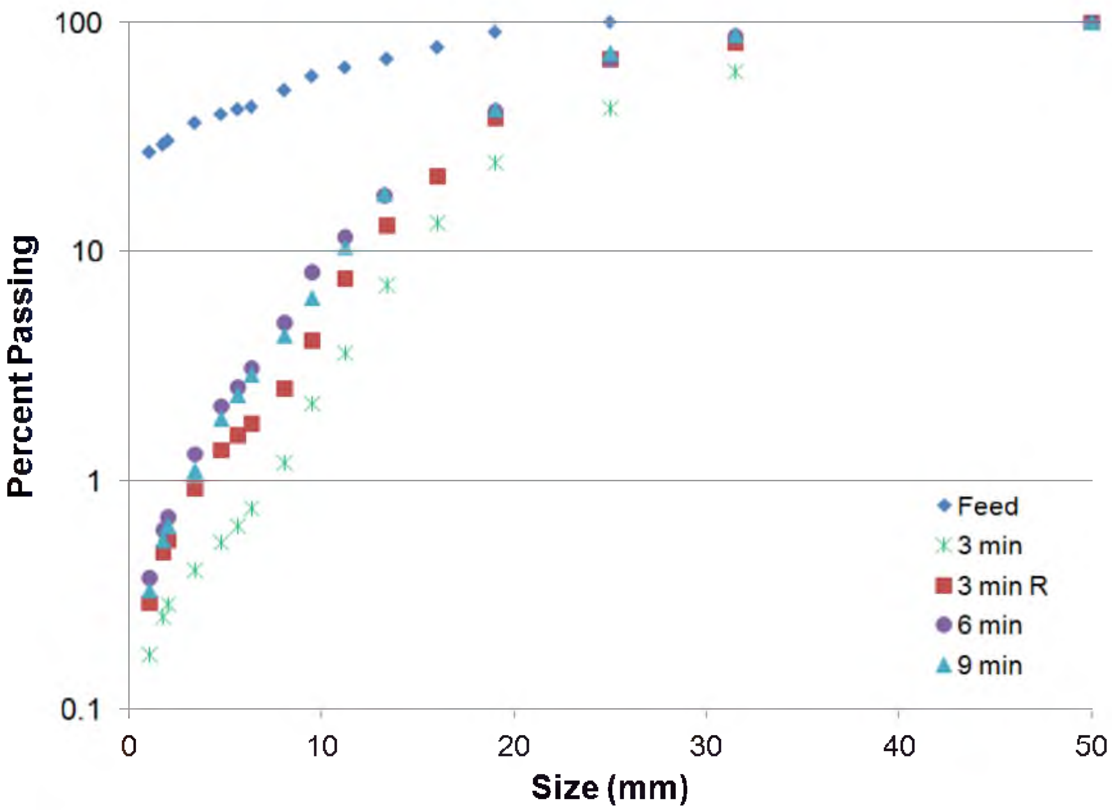

Figure 2.8 The ASDs produced at different mixing times using 13.0% moisture, 25.6 g/L H_2SO_4, and 30% critical speed. R indicates a repeat of conditions.

Table 2.6 Conductivities and conductivity ratios for agglomerates produced with various moisture contents and acid concentrations.

Moisture Content (%)	Acid Concentration (g/L)	Conductivity (S/m)		Conductivity Ratio	
		No Load	18.2 kg Load	No Load	18.2 kg Load
9.1	25.6	7.12×10^{-4}	2.75×10^{-3}	6.25×10^{-3}	2.42×10^{-2}
11.5	25.6	4.74×10^{-4}	1.32×10^{-3}	4.16×10^{-3}	1.16×10^{-2}
13.0	25.6	7.73×10^{-4}	3.70×10^{-3}	6.79×10^{-3}	3.25×10^{-2}
	25.6 (Repeat)	2.17×10^{-3}	7.66×10^{-3}	1.91×10^{-2}	6.73×10^{-2}
	50	9.19×10^{-4}	3.20×10^{-3}	4.35×10^{-3}	1.51×10^{-2}
	100	7.08×10^{-3}	1.77×10^{-2}	1.76×10^{-2}	4.40×10^{-2}
	200	7.20×10^{-3}	2.35×10^{-2}	1.08×10^{-2}	3.53×10^{-2}
13.8	25.6	1.35×10^{-3}	8.55×10^{-3}	1.19×10^{-2}	7.51×10^{-2}
14.5	25.6	4.64×10^{-3}	1.18×10^{-2}	4.08×10^{-2}	1.04×10^{-1}
16.7	25.6	6.29×10^{-3}	9.44×10^{-3}	5.52×10^{-2}	8.29×10^{-2}

Table 2.7 Conductivities and conductivity ratios for agglomerates with varying drum rotational speeds and mixing times at 13.0% moisture and 25.6 g/L acid concentration.

Factors		Conductivity (S/m) No Load	Conductivity Ratio 18.2 kg Load	Conductivity (S/m) No Load	Conductivity Ratio 18.2 kg Load
Drum Rotation Speed (%NC)	20	1.03×10^{-3}	4.99×10^{-3}	9.08×10^{-3}	4.38×10^{-2}
	30	7.73×10^{-4}	3.70×10^{-3}	6.79×10^{-3}	3.25×10^{-2}
	30 (Repeat)	2.17×10^{-3}	7.66×10^{-3}	1.91×10^{-2}	6.73×10^{-2}
	50	1.31×10^{-3}	2.74×10^{-3}	1.15×10^{-2}	2.41×10^{-2}
Mixing Time (min)	3	7.73×10^{-4}	3.70×10^{-3}	6.79×10^{-3}	3.25×10^{-2}
	3 (Repeat)	2.17×10^{-3}	7.66×10^{-3}	1.91×10^{-2}	6.73×10^{-2}
	6	1.12×10^{-3}	2.94×10^{-3}	9.83×10^{-3}	2.58×10^{-2}
	9	2.42×10^{-3}	3.50×10^{-3}	2.12×10^{-2}	3.07×10^{-2}

The conductivity of the agglomerates tends to increase as the volume of solution added increases. This trend can be seen in Figure 2.9 where the conductivity ratio is plotted to allow comparison of different acid solution.

2.2.3 Permeability

The ore was initially tested without agglomeration to observe behavior in the permeability test. During this test, channeling was observed. The apparatus became clogged and only one data point could be obtained.

In general, permeability for a bed of agglomerates produced from the ore studied increases with increasing moisture at a constant acid strength, as seen in Figure 2.10. The permeability at 16.7% moisture could not be obtained because the agglomerates had slumped below the top monometer point. Valid readings could not be obtained at 9.1% moisture because the sample was too dry and the outlet became plugged. These values indicate that agglomeration for this ore should occur with moisture contents between 16.7% and 9.1%. In addition, agglomeration at or below 9.1% moisture will lead to very poor permeability.

The permeabilities of samples produced with various acid concentrations are shown in Figure 2.11. The agglomerates prepared at 200 g/L were too dry, which again caused the outlet on the apparatus to become plugged. There appears to be a u-trend where the permeability is at a maximum around 50 g/L H_2SO_4 and permeability decreases as acid concentration either increases or decreases.

The permeability data for varying rotational speed are found in Table 2.8. These results may indicate permeability is at a minimum at 30% NC. In

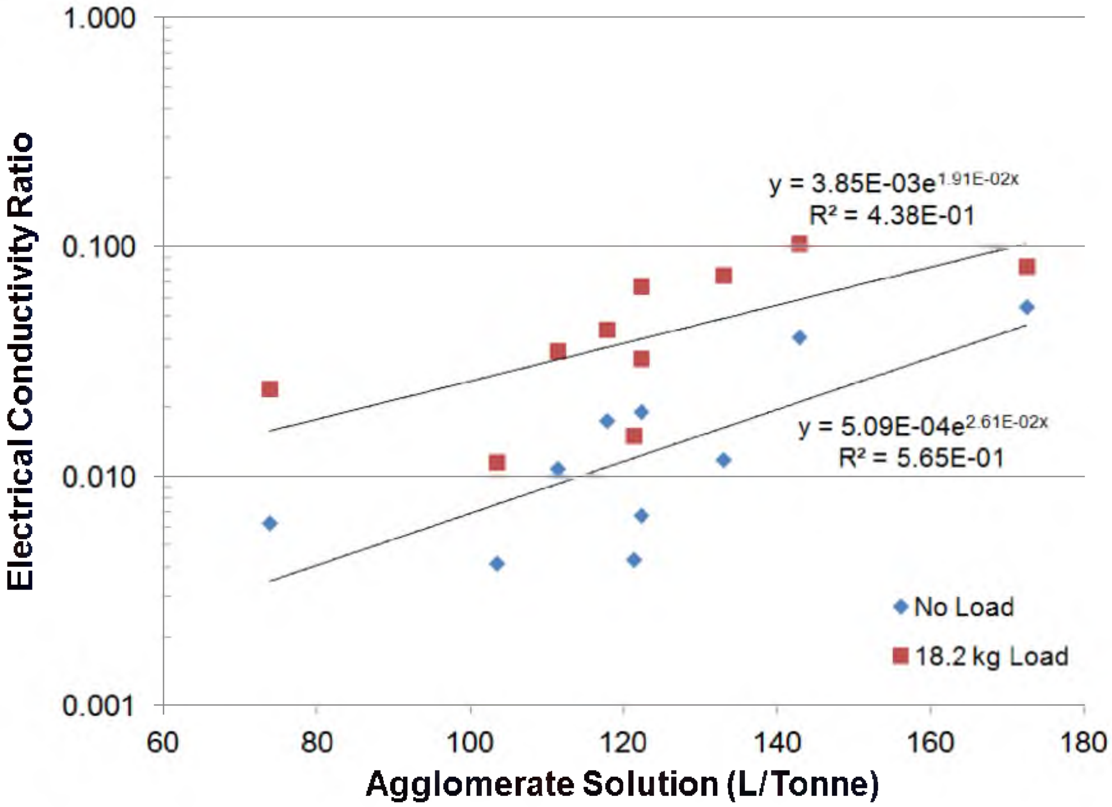

Figure 2.9 Relationship between agglomerate solution added and electrical conductivity. The 18.2 kg load simulates the weight the agglomerates support at 1.5 meters in the heap.

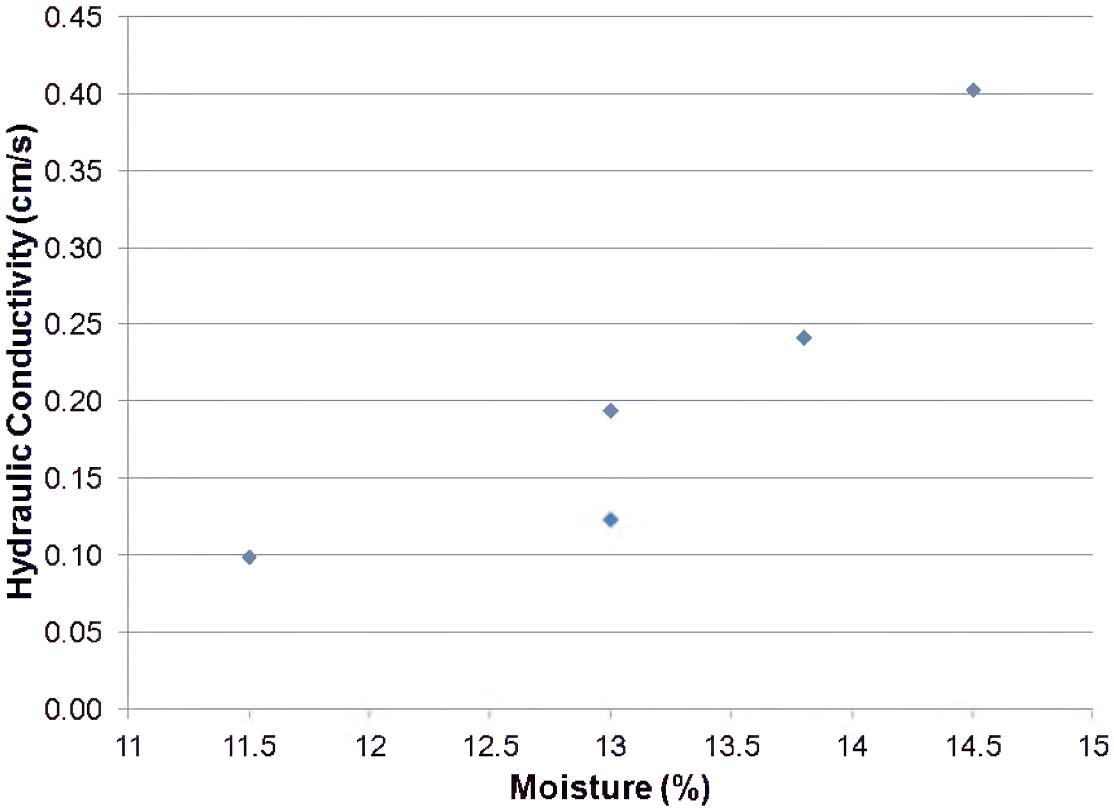

Figure 2.10 Hydraulic conductivity of agglomerates with varying moisture content.

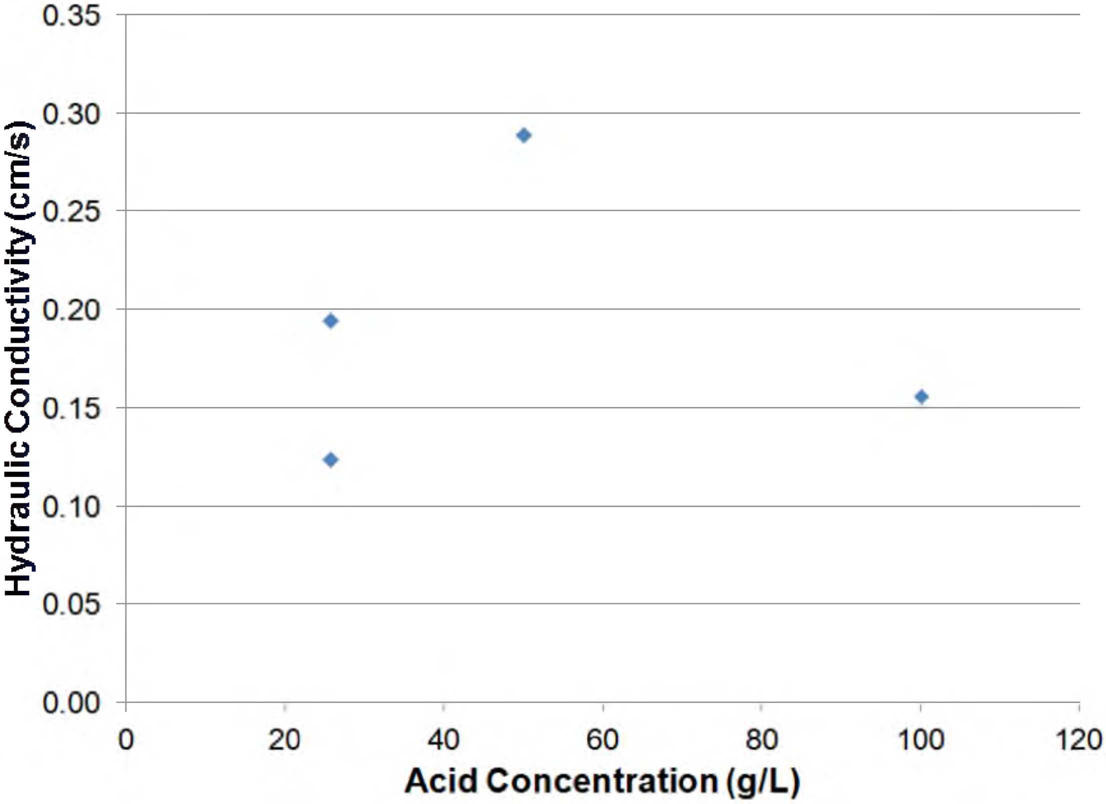

Figure 2.11 Hydraulic conductivity of agglomerates produced with various acid concentrations at 13.0% moisture.

Table 2.8 Permeability data for agglomerates produced with varying rotational speed.

Rotational Speed (% NC)	Hydraulic Conductivity
20	0.24
30	0.19
30 (Repeat)	0.12
50	0.28

Figure 2.12, the permeability of samples produced with varying retention time is shown. There appears to be a trend of increasing permeability with increasing retention time. This is unexpected because permeability should be a function of agglomerate size and these agglomerates had similar size distributions. Perhaps the agglomerates with a higher retention time are more compacted and able to maintain their structure longer during the permeability test.

2.2.4 Bulk Density

The bulk density of agglomerated ore was obtained at the beginning and at the end of the permeability test. Bulk density was calculated using equation 2.9. The porosity of the agglomerate bed was calculated using equation 2.10.

$$\text{Bulk Density } \left(\frac{\text{g}}{\text{mL}}\right) = \frac{\text{Mass of Agglomerates (g)}}{\text{Volume of Agglomerates (mL)}} \tag{2.9}$$

$$\text{Porosity} = 1 - \frac{\text{Bulk Density of Agglomerates } \left(\frac{\text{g}}{\text{mL}}\right)}{\text{Particle Density } \left(\frac{\text{g}}{\text{mL}}\right)} \tag{2.10}$$

The average particle density for the ore was measured at 2.28 g/mL.

All of the agglomerates experienced an increase in bulk density and decrease in porosity during the permeability test. This observation is shown in Tables 2.9 and 2.10. While performing the permeability test, one can watch the agglomerates slump as they are submerged in water and degrade. Thus, the

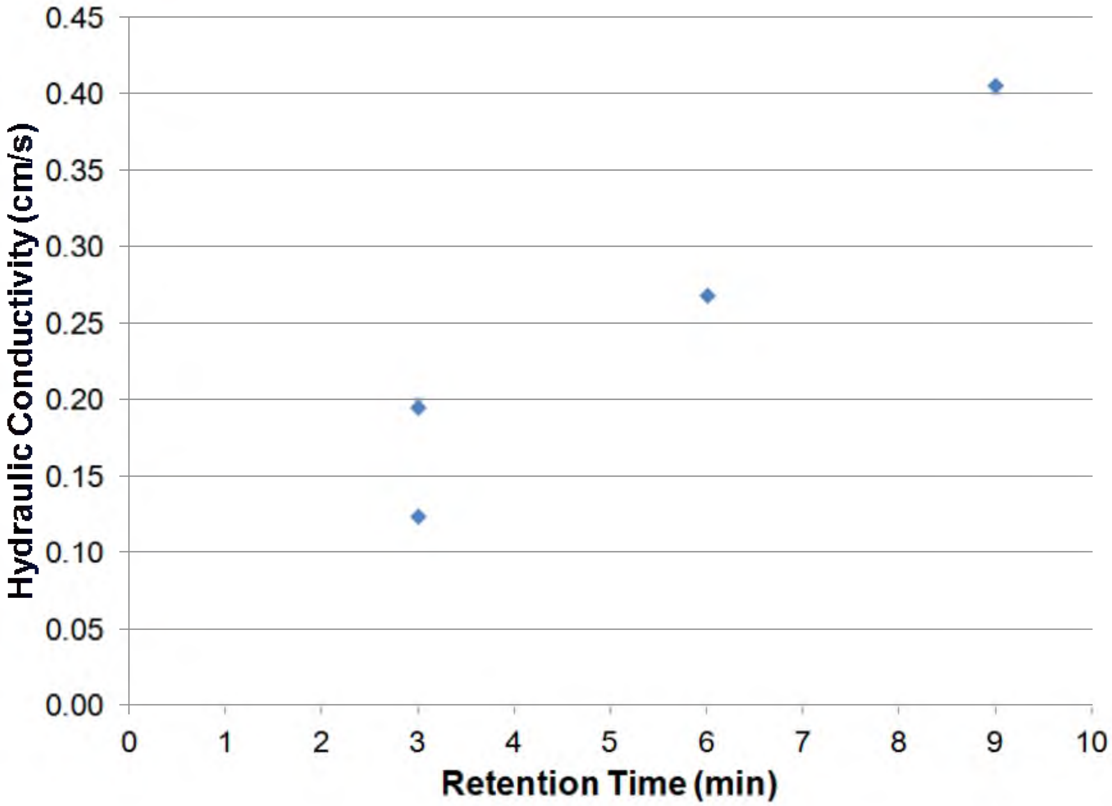

Figure 2.12 Hydraulic conductivity of agglomerates produced with various retention times.

Table 2.9 Bulk Density and Porosity data for agglomerates of varying moisture contents and acid concentrations.

Moisture Content (%)	Acid Concentration (g/L)	Bulk Density (g/mL)		ΔBulk Density (%)	Porosity		ΔPorosity (%)
		Before Perm. Test	After Perm. Test		Before Perm. Test	After Perm. Test	
Unagglomerated	-	1.55	-	-	0.32	-	-
9.1	25.6	1.30	1.60	18.8	0.43	0.30	30.2
11.5	25.6	1.24	1.49	16.8	0.46	0.35	23.9
13.0	25.6	0.97	1.18	17.8	0.58	0.48	17.2
	25.6 (Repeat)	1.21	1.51	19.9	0.47	0.34	27.7
	50	1.19	1.51	21.2	0.48	0.34	29.2
	100	1.18	1.51	21.9	0.48	0.34	29.2
	200	1.18	1.58	25.3	0.48	0.31	35.4
13.8	25.6	1.17	1.48	20.9	0.49	0.35	28.6
14.5	25.6	1.26	1.59	20.8	0.45	0.30	33.3
16.7	25.6	0.99	1.41	29.8	0.57	0.38	33.3

Table 2.10 Bulk Density and Porosity data for agglomerates with varying drum rotational speeds and mixing times at 13.0% moisture and 25.6 g/L acid concentration.

Factors		Bulk Density		ΔBulk Density (%)	Porosity		ΔPorosity (%)
		Before Perm. Test	After Perm. Test		Before Perm. Test	After Perm. Test	
Unagglomerated		1.55	-	-	0.32	-	-
Drum Rotation Speed (%NC)	20	1.22	1.58	22.8	0.47	0.31	34.0
	30	0.97	1.18	17.8	0.58	0.48	17.2
	30 (Repeat)	1.21	1.51	19.9	0.47	0.34	27.7
	50	1.20	1.52	21.1	0.47	0.33	29.8
Mixing Time (min)	3	0.97	1.18	17.8	0.58	0.48	17.2
	3 (Repeat)	1.21	1.51	19.9	0.47	0.34	27.7
	6	1.24	1.53	19.0	0.46	0.33	28.3
	9	1.25	1.54	18.8	0.45	0.33	26.7

values after the permeability test for all of the samples except one were very similar. This one condition when repeated was not found to be reproducible.

2.2.5 TBS Test

The TBS test was performed after allowing the agglomerates to cure for 24 hours in a sealed plastic bag. Agglomerates that were "good looking" (e.g., formed from multiple particles and roundish in shape) were tested. Based on previous research with other ores, agglomerate stability was considered to be "good", if it remained stable in this test for 30 minutes. An example of a test for an agglomerate about 2 cm in diameter at the 50% NC condition can be seen in Figure 2.13. None of the agglomerates tested can be considered "good", as indicated in Tables 2.11 and 2.12. One agglomerate for the 6 minute mixing time almost reached 30 minutes, but when this was repeated, the result was very different. When reviewing the data, some sizes of agglomerates seem to last for different times than other sizes produced under the same agglomerating conditions. This variation is considered experimental scatter. Since this test did not produce results that varied with agglomeration conditions, it was not used later in the study.

2.3 Conclusions

The purpose of the scoping experiments was to learn the agglomeration characteristics of the copper ore in order to better select conditions for the column leach experiments. The scoping experiments indicated that for this ore, the agglomerates produced using sulfuric acid and water as a binder are not very

Figure 2.13 TBS test where changes can be seen A) at the beginning B) during the test and C) at the end of the test. The agglomerate can be seen degrading as the test progresses.

Table 2.11 TBS results for agglomerates of varying moisture contents and acid concentrations.

Moisture Content (%)	Acid Concentration (g/L)	~4 cm (min)	~2 cm (min)	~1 cm (min)
9.1	25.6	8.0	4.1	2.6
11.5	25.6	10.7	7.9	3.4
13.0	25.6	6.4	6.4	9.3
	25.6 (Repeat)	7.4	3.5	5.4
	50	4.6	2.8	3.6
	100	2.7	2.9	2.7
	200	4.2	1.1	2.6
13.8	25.6	12.5	8.0	3.7
14.5	25.6	8.4	3.4	10.3
16.7	25.6	10.7	3.6	11.9

Table 2.12 TBS results for agglomerates with varying drum rotational speeds and mixing times at 13.0% moisture and 25.6 g/L H_2SO_4 concentration.

Factors		~4 cm (min)	~2 cm (min)	~1 cm (min)
Drum Rotation Speed (%NC)	20	8.2	10.7	1.3
	30	6.4	6.4	9.3
	30 (Repeat)	7.4	3.5	5.4
	50	16.3	11.8	13.4
Mixing Time (min)	3	6.4	6.4	9.3
	3 (Repeat)	7.4	3.5	5.4
	6	14.8	29.0/6.4[a]	7.0
	9	6.6	6.9	9.4

[a] Repeated test result

stable and easily degrade. Furthermore, if the agglomerates are too dry, channeling and fines migration were detectable in the permeability testing. Finally, drum speed and mixing time had minimal effects on the agglomerates.

CHAPTER 3

COLUMN LEACHING EXPERIMENTS

3.1 Experimental

3.1.1 Sample Preparation

The same size distribution as used for the scoping experiments was used to prepare samples for column leaching. An insufficient amount of material smaller than 0.15 mm was provided by the mine, so additional material had to be manufactured. Material was taken from the following sizes for use in fines manufacture: 6.4 mm to 1.7 mm, 1.7 mm to 0.50 mm, and 0.50 mm to 0.15 mm. The material was ground using a continuously fed ball mill to produce approximately 200 kg of fines smaller than 150 μm. The resulting size distribution along with the size distribution of the fine material originally provided by the mine is shown in Table 3.1.

3.1.2 Liquid Retention Capacity

Because the behavior of the ore could have changed with the introduction of manufactured fines, the liquid retention capacity (LRC) was measured again. The LRC was determined to be 25.2%, as described in Section 2.1.2. This number differs from the 23.6% obtained from measuring the original mine-supplied ore, which indicates changing to the manufactured fines has affected the LRC and could affect the optimal moisture in agglomeration.

Table 3.1 Size distribution of material smaller than 150 µm.

Mesh Size (µm)	Company Size Distribution (% Passing)	Milled Size Distribution (% Passing)
150	100%	95%
106	96%	90%
75	87%	85%
45	75%	80%
Pan	0%	0%

3.1.3 Agglomeration

The agglomeration procedure was modified somewhat from the procedure used during the scoping experiments to address a problem encountered with fines sticking to the agglomeration drum. Total mixing time was fixed at 4.5 minutes. Solution was applied for the first 30 seconds of mixing. After 30 seconds of mixing without solution addition, solution was applied for an additional 30 seconds. After a total of 3 minutes of mixing, the agglomeration drum was removed and fines stuck to the drum were removed by hitting the outside of the drum. The drum was replaced and agglomeration continued for an additional 1.5 minutes.

A plastic drum was used as the agglomerator. The drum had dimensions of 45.2 cm diameter and a length of 71 cm. The agglomerating drum was equipped with 4 lifters of 0.9 cm thickness, 2.5 cm width, and 60 cm length, which was centered between the two ends of the drum. The lifters were equally spaced around the drum's internal circumference. The critical speed of the drum was calculated as 67.2 rpm. Thus, 20.2 rpm was used to produce 30% critical speed.

The drum was filled with 25 kg of sample (copper ore), which resulted in about 14.2 % volume fill of the drum. To achieve homogeneity, the sample was mixed for 30 minutes in the drum prior to solution addition. Deionized water and concentrated sulfuric acid were applied simultaneously using peristaltic pumps and separate hoses for the respective solutions. The moisture contents, acid concentration, and acid dosages for agglomerates produced are shown in

Table 3.2. The moisture content was calculated using equation 2.3. Acid and water volumes were calculated using equations 2.4, 2.5, and 2.6, as described in Chapter 2.

3.1.4 Quality Control Tests

Agglomerate size distribution, electrical conductivity, and permeability tests were performed using the same methods described in Chapter 2. The TBS test was not performed on agglomerates produced during this phase of the project as the test was not found to be helpful.

3.1.5 Column Construction

Columns were constructed using 20 cm internal diameter PVC pipe. A cap was placed on the bottom with two holes drilled into it, one for a drainage tube and the other for an air line. A perforated, flexible air tube is placed at the bottom of the pipe. Glass marbles are placed on top of the air tube, followed by a perforated plastic sheet. Agglomerates are loaded on top of the plastic sheet. Another perforated plastic sheet is placed on the agglomerates, followed by additional glass marbles and five layers of cloth. The marbles and sheet are used to ensure adequate drainage. The cloth allows leaching solution to be evenly dispersed. A schematic of the column is provided in Figure 3.1. An image of the flexible air tube is shown in Figure 3.2.

Agglomerates not collected as samples for the quality control tests were weighed and loaded into the column the same day they were prepared. Columns were left for 8 days (cured) before leaching was started.

Table 3.2 Moisture content, acid concentration, and acid dosage of agglomerates.

Label	Moisture %	Acid Concentration (g/L)	Acid Dosage (kg/tonne)
4	11.5	30	3.2
2	11.5	90	9.3
1,3,7[a]	13.0	60	7.5
6	14.5	30	4.5
5	14.5	90	12.9

[a] This condition was produced in triplicate

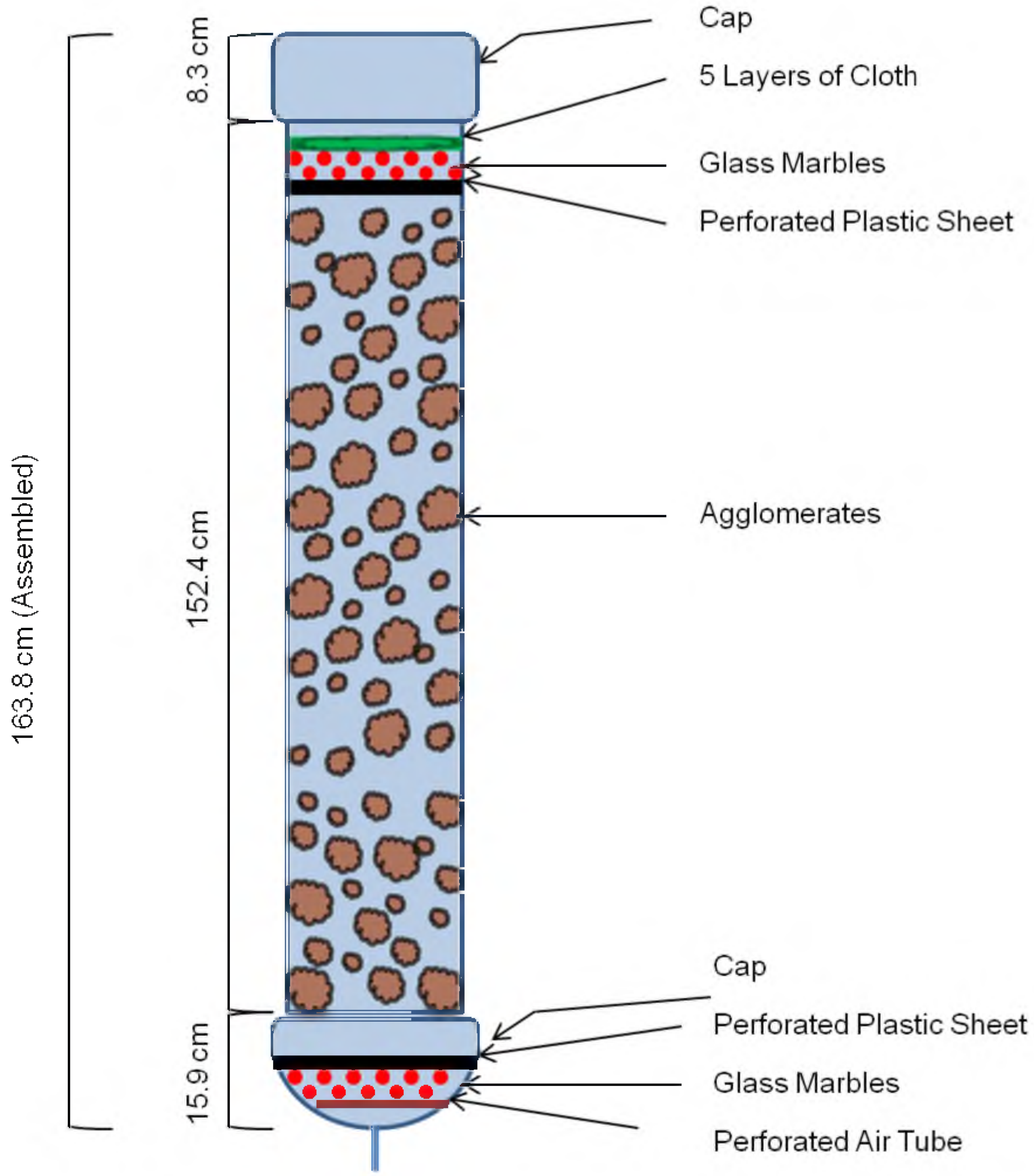

Figure 3.1 Schematic of column used in leaching experiments.

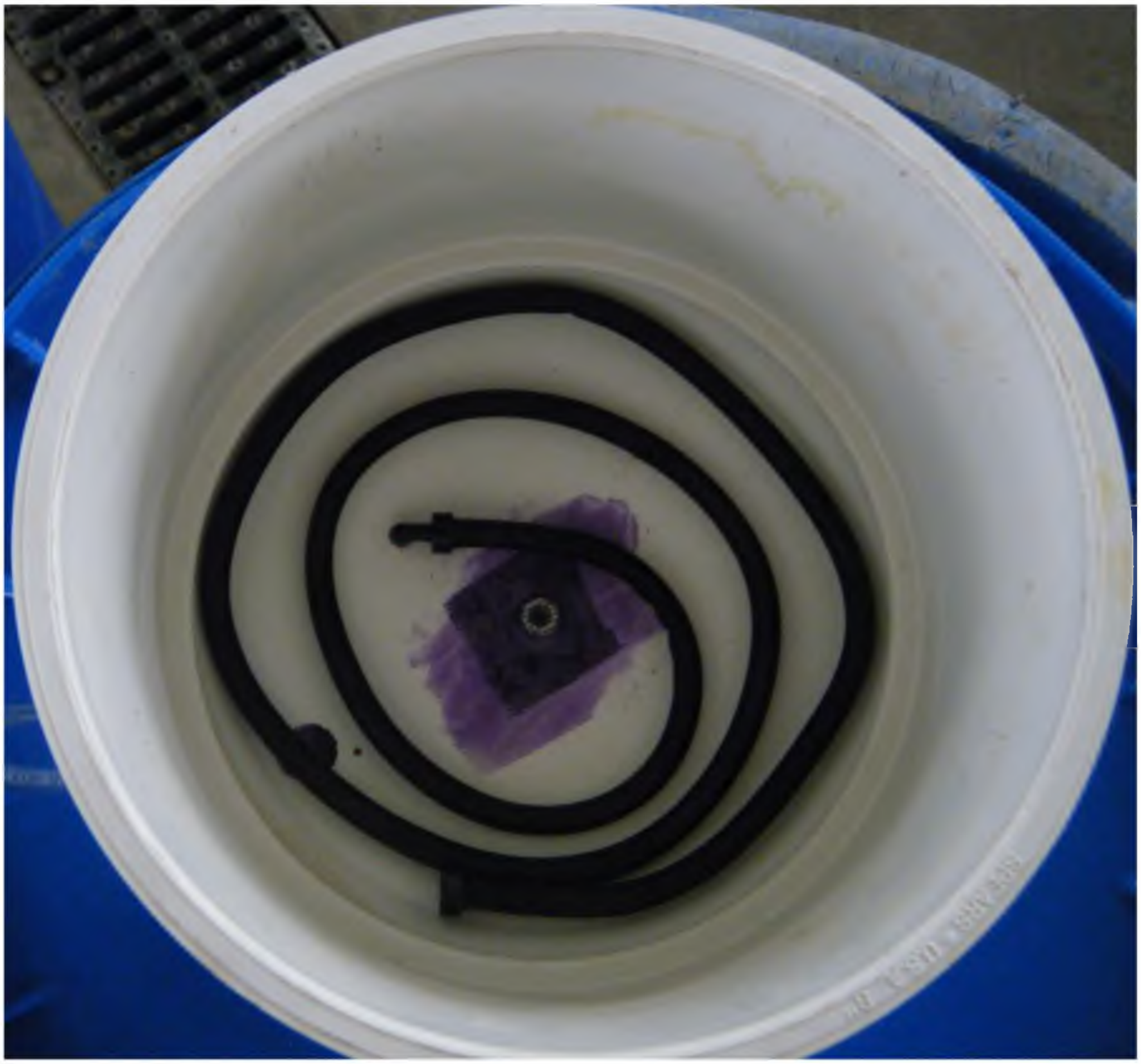

Figure 3.2 Flexible air tubing in column end cap.

3.1.6 Column Leaching

Leaching solution was prepared by dissolving sulfuric acid (H_2SO_4), ferric sulfate ($Fe_2(SO_4)_3$), and sodium chloride (NaCl) in deionized water. The target concentration of each component was: 2.2 g/L Cl^-, 2.6 g/L Fe^{3+}, and 7.4 g/L H_2SO_4. Leaching solution was delivered to each column using plastic tubing and a peristaltic pump. Solution was drawn from the same container and delivered to each of the columns. The target delivery rate of solution was 4.4 mL/min or 8 $L/hr \cdot m^2$. After passing through the column, solution was collected in a bucket under the column. To reduce evaporation and for safety reasons, the bucket was equipped with a lid. Leaching solution was only passed through a column once and after sampling, was disposed.

Air was delivered to the column using an oil-less air compressor. A flow meter with a control valve was connected to the air tube for each column to allow adjustment of air delivery. To provide even air distribution, air was delivered at the bottom of each column through a flexible perforated tube. The target air delivery rate was 250 mL/min or 0.47 $m^3/hr \cdot m^2$.

3.1.7 Column Startup Procedure

Following the cure period, solution and air flow to the column was started and the current time recorded. The drainage tube for the column was inserted into a custom-made detection device. When solution flows into the device, an electrical circuit is completed and starts a clock. The time the first solution exits the column, known as the breakthrough time, can be determined from the clock reading.

3.1.8 Column Shutdown Procedure

After 90 days of leaching, the solution and air delivery were stopped. The draining solution was collected periodically for a period of 24 hours. Columns were then washed with deionized water in order to reduce the acid concentration prior to disassembling the column.

3.1.9 Column Monitoring

On a daily basis, data were collected, which included air flow rate, height of ore in the column, oxidation reduction potential (ORP), solution volume, and solution weight. In addition, a sample was taken to be analyzed for pH and copper content. The air flow rate fluctuated over time and was adjusted back to the desired flow (250 mL/min) for each column on a daily basis. Periodically, solution flow rate was measured. Because a single pump controlled the flow to all columns, the solution flow varied somewhat from column to column throughout the experiment. Solution flow rates were within about 10% of the desired value (4.4 mL/min) for all columns.

3.1.10 Analysis for Copper Recovery

Samples collected from column leaching were analyzed for copper content using a Spectro model Genesis FES ICP-OES. Samples were diluted to produce a copper concentration measurement between 20 and 100 ppm. The instrument was calibrated using standard solutions ranging from 20 to 100 ppm. Samples were analyzed five times with the average concentration being used to calculate copper recovery. The percent of copper recovered was calculated using equation 3.1.

$$\text{Copper \% Recovered}=\frac{\sum_{i=1}^{N}\text{Cu Avg}_i\times\text{Vol}_i}{\text{Mass}_{\text{Agg}}\times\text{Assay}} \tag{3.1}$$

where N is the number of samples collected, Cu Avg is the average copper concentration of the sample, Vol is the volume of solution at the sample concentration, and Mass_{Agg} is the mass of feed ore loaded into the column. Assay is the fraction of copper in the feed ore obtained from the assay provided by the mine.

3.1.11 Statistical Analysis

The significance of moisture and acid on each of the agglomerate characteristics or behaviors was evaluated using JMP® 10.0.0 statistics software.

3.2 Results and Discussion

3.2.1 Agglomerate Size Distribution

After producing agglomerates for column 1, a sample was taken from each of the three batches to compare size distribution and agglomerate reproducibility from one batch to the next. The ASDs for these batches are shown in Figure 3.3. The ASD for each of the batches is quite similar, which indicates the agglomerates are reproducible.

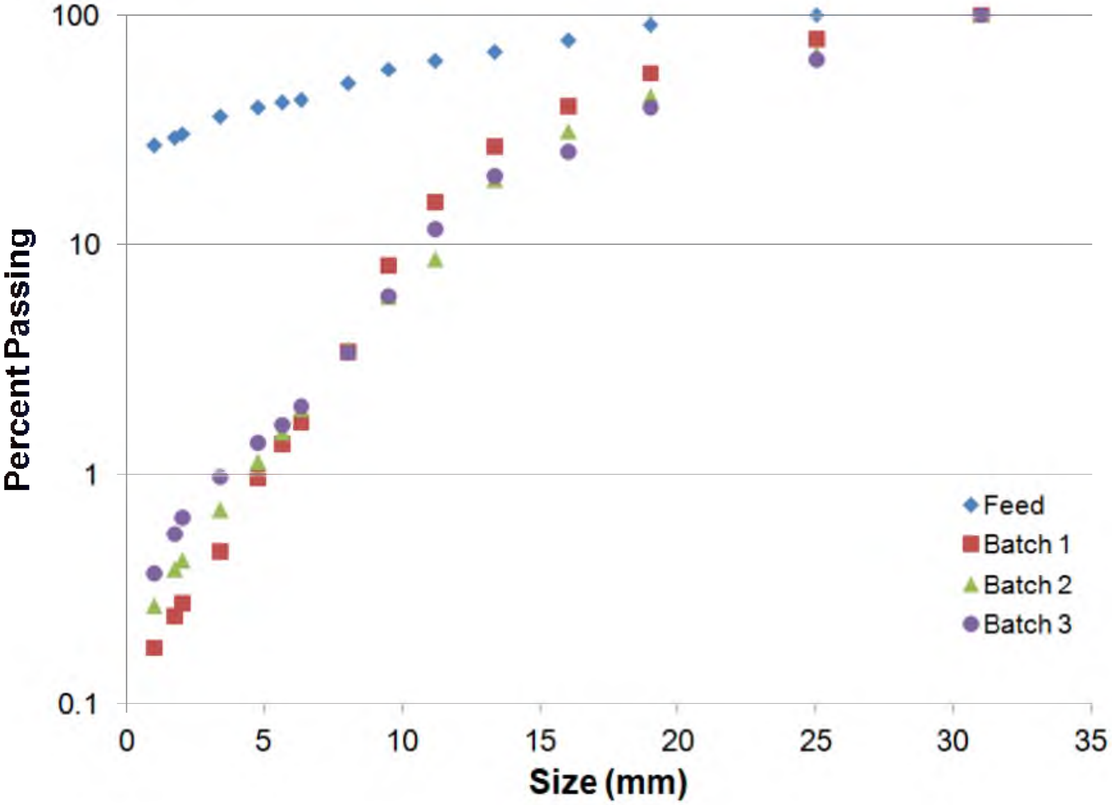

Figure 3.3 ASDs of batches for column 1, which was produced at 13.0% moisture, 60 g/L H_2SO_4, and 30% NC

Because columns 1, 3, and 7 were all made using the same condition, it is expected the ASDs of these three columns will be similar. The ASDs for columns 1, 3, and 7 are shown in Figure 3.4. The ASDs for these columns are similar, which also indicates reproducibility of agglomerates from one column to another.

The ASDs for all conditions are shown in Figure 3.5. As was encountered during the scoping experiments, when the moisture is lower, the ASD approaches the feed size distribution. This same trend is observed when comparing the total amount of solution added to the D_{50}. This is confirmed with statistical analysis indicating moisture content affects the D_{50} ($P=0.0074$). It should be noted that the trend does not exist with the D_{10}. The D_{50} and D_{10} values for each column are shown in Figure 3.6.

While the trend is the same for the column agglomerates as it was during the scoping experiments, the agglomerate D_{50} for a given volume of solution was smaller for the column agglomerates than it was for scoping experiment agglomerates. This is likely due to a change in the mineralogy of the ore caused by the substitution of manufactured fines. The LRC measurement indicates the manufactured ore can support a larger amount of water. This may suggest a higher moisture is required to produce the same size of agglomerates made during the scoping experiments at a given moisture value. The D_{50}s and D_{10}s of agglomerates produced during the scoping experiments are shown with those of the columns in Figure 3.7. Photographs of the agglomerates are shown in Figures 3.8 and 3.9.

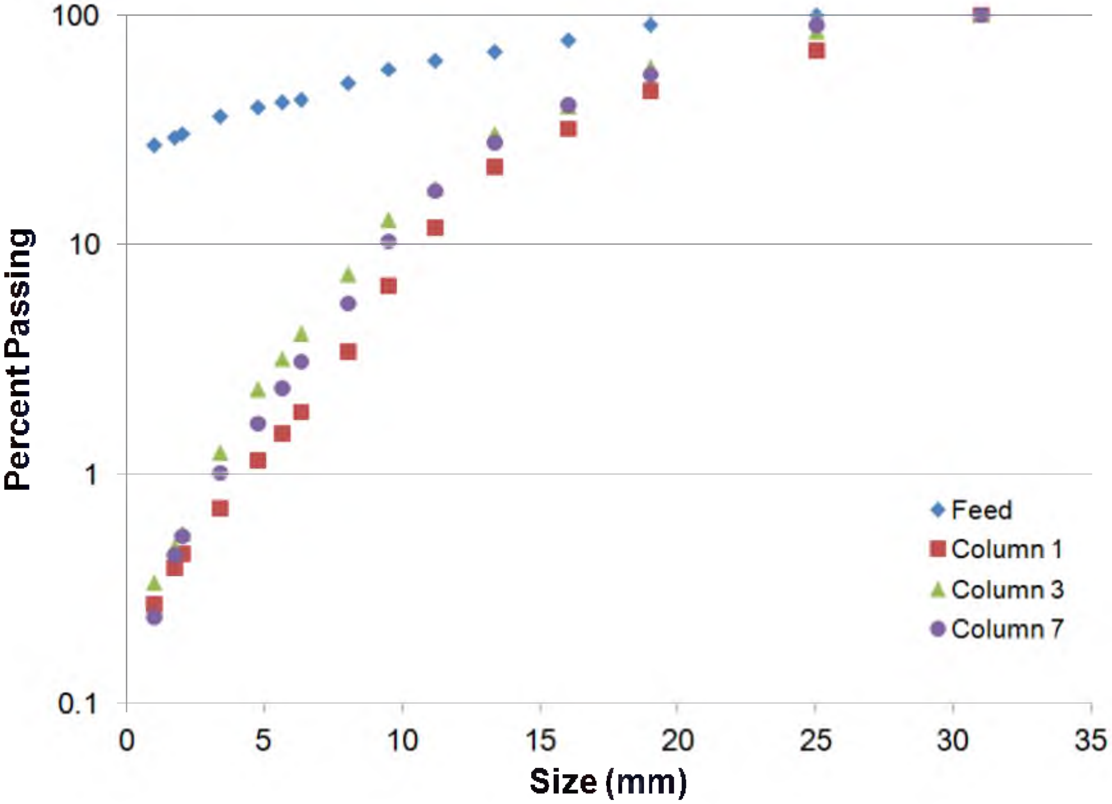

Figure 3.4 ASDs of midpoint columns produced at 13.0% moisture, 60 g/L H_2SO_4, and 30% NC.

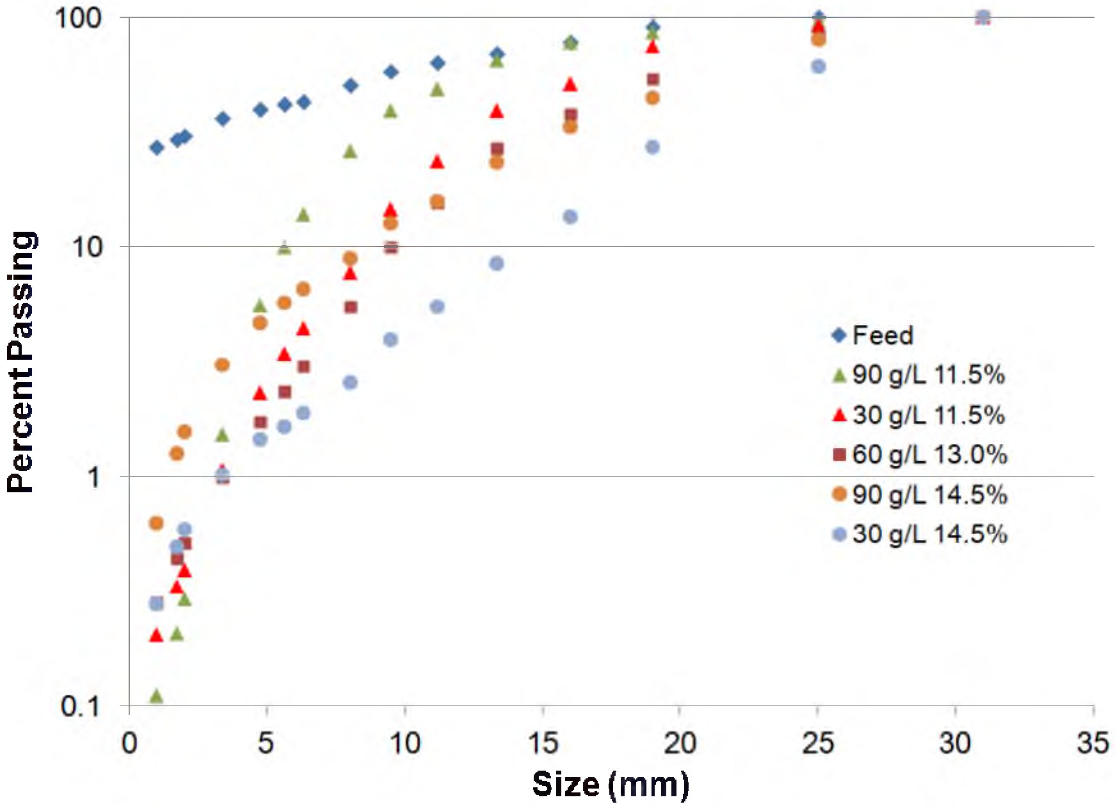

Figure 3.5 ASDs of all conditions with an average shown for the midpoint columns at 13.0% moisture, 60 g/L H_2SO_4, and 30% NC. Legend is read as acid concentration followed by moisture percent.

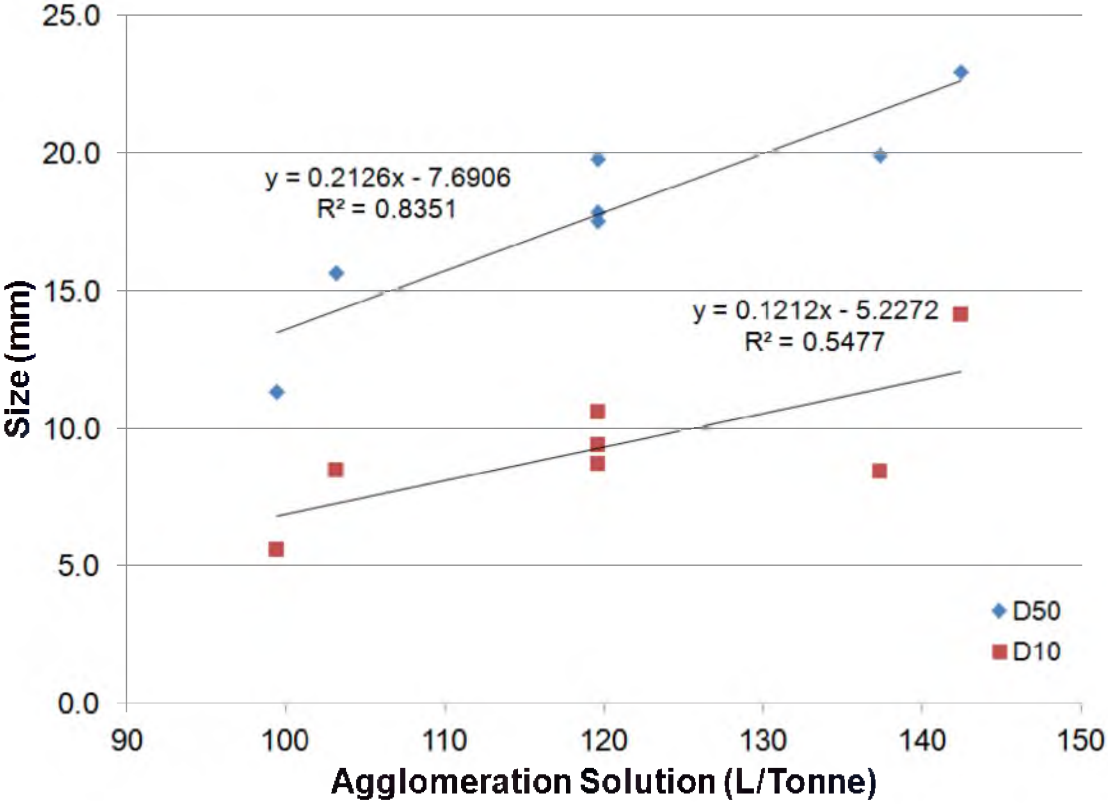

Figure 3.6 Relationship between volume of solution added and agglomerate particle size for agglomerates produced for column leaching.

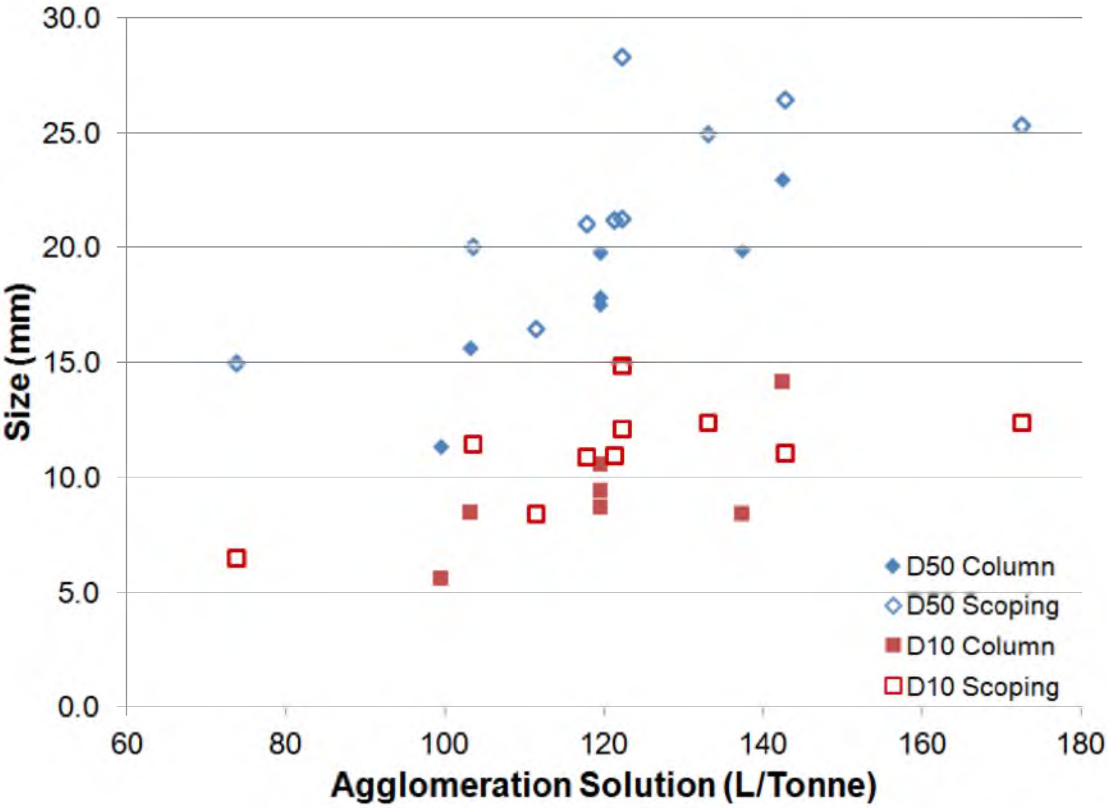

Figure 3.7 Comparison of agglomerates produced during scoping experiments with those produced for columns. Mixing speed was fixed at 30% NC. Mixing time was 3 minutes for scoping experiments and 4.5 minutes for columns.

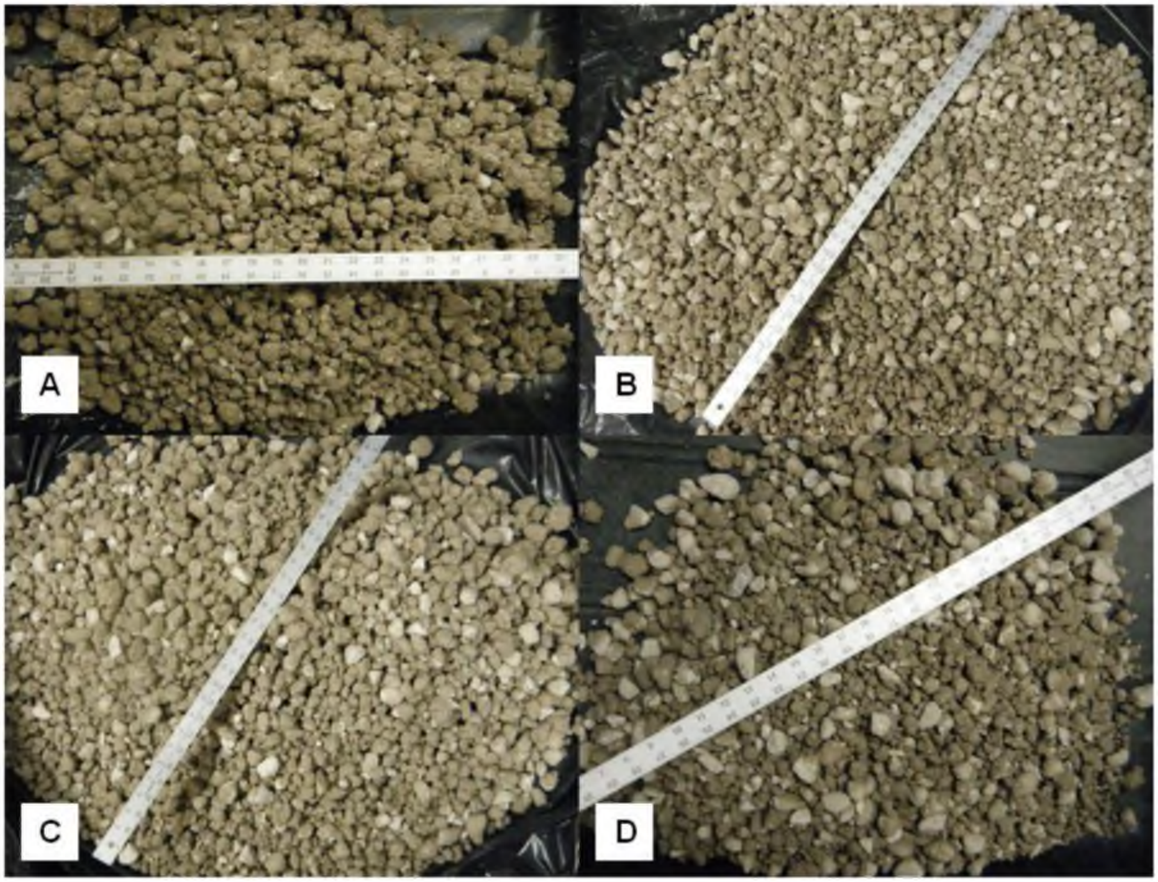

Figure 3.8 Photos of agglomerates from A) Column 1, B) Column 2, C) Column 3, and D) Column 4

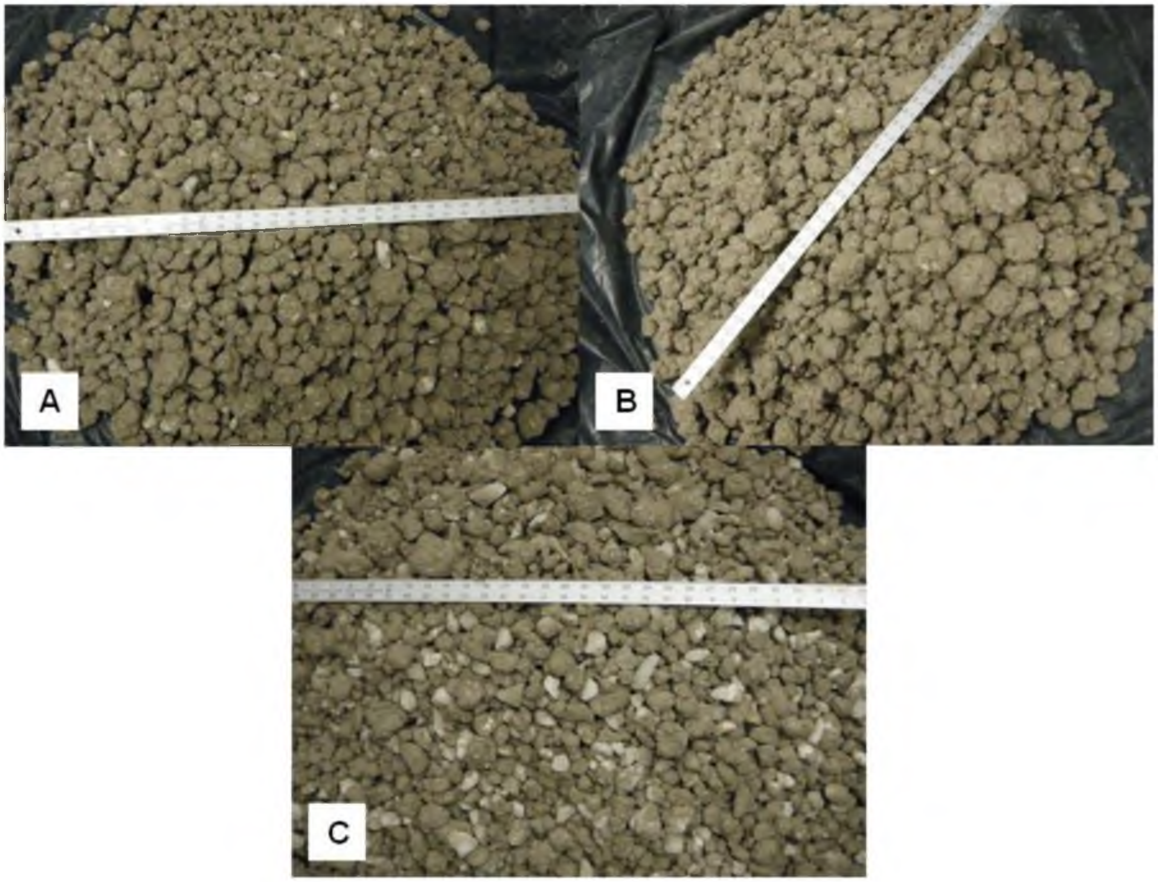

Figure 3.9 Photos of agglomerates from A) Column 5, B) Column 6, and C) Column 7.

3.2.2 Electrical Conductivity

The electrical conductivity of the agglomerates was measured and the results are summarized in Table 3.3. As expected, the conductivity ratio tends to increase as the total volume of solution added increases. This trend is shown in Figure 3.10. The conductivity ratio produced when a load is applied is more consistent than when no load is applied. The statistical analysis did not show a significant effect of acid concentration or moisture content on the electrical conductivity ratio regardless of the presence of a load.

The conductivity ratios obtained from these agglomerates are consistent with those obtained for scoping experiment agglomerates. This consistency can be seen in Figure 3.11. It should be noted that because of the large amount of scatter observed, this test may be difficult to utilize during agglomerate characterization in a production setting for this ore.

3.2.3 Permeability

The hydraulic conductivity was measured on a sample of agglomerates produced for column leach testing. The results in Figure 3.12 show a trend of increasing hydraulic conductivity as the volume of agglomerate solution is increased. It is expected that larger agglomerates would be more permeable and these results agree with those shown in Figure 3.7. The statistical analysis performed indicates neither moisture content nor acid concentration are significant factors affecting hydraulic conductivity. This was likely due to the variation present at 13.0% moisture.

Table 3.3 Conductivities and conductivity ratios of agglomerates produced for column leaching.

Acid Conc. (g/L)	Moisture Content (%)	Label	Conductivity (S/m)		Conductivity Ratio	
			No Load	18.2 kg Load	No Load	18.2 kg Load
30	11.5	4	1.04×10^{-3}	4.69×10^{-3}	4.74×10^{-3}	2.14×10^{-2}
90	11.5	2	2.90×10^{-3}	7.05×10^{-3}	7.86×10^{-3}	1.91×10^{-2}
60	13.0	1	9.71×10^{-4}	4.89×10^{-3}	4.43×10^{-3}	2.23×10^{-2}
		3	8.03×10^{-4}	2.63×10^{-3}	6.02×10^{-3}	1.97×10^{-2}
		7	3.45×10^{-3}	1.05×10^{-2}	9.37×10^{-3}	2.86×10^{-2}
30	14.5	6	1.47×10^{-3}	4.02×10^{-3}	1.10×10^{-2}	3.02×10^{-2}
90	14.5	5	2.79×10^{-3}	4.95×10^{-3}	1.27×10^{-2}	2.26×10^{-2}

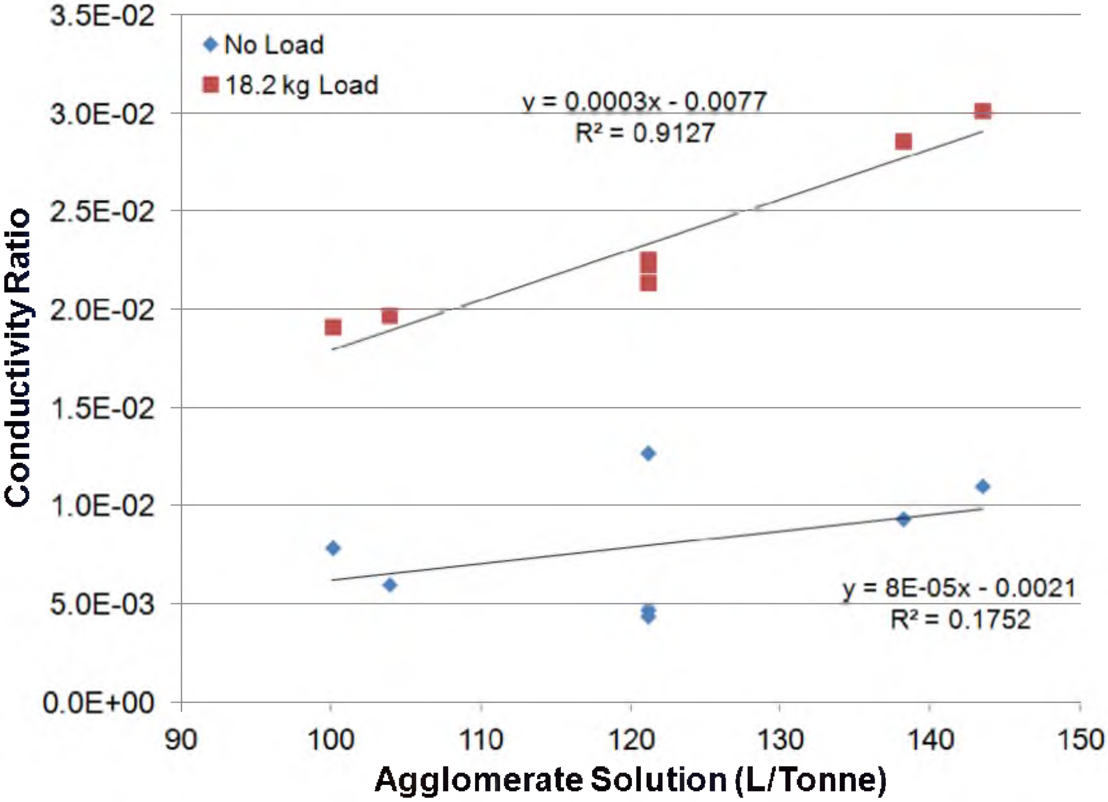

Figure 3.10 Electrical conductivity ratio of agglomerates produced for column leaching.

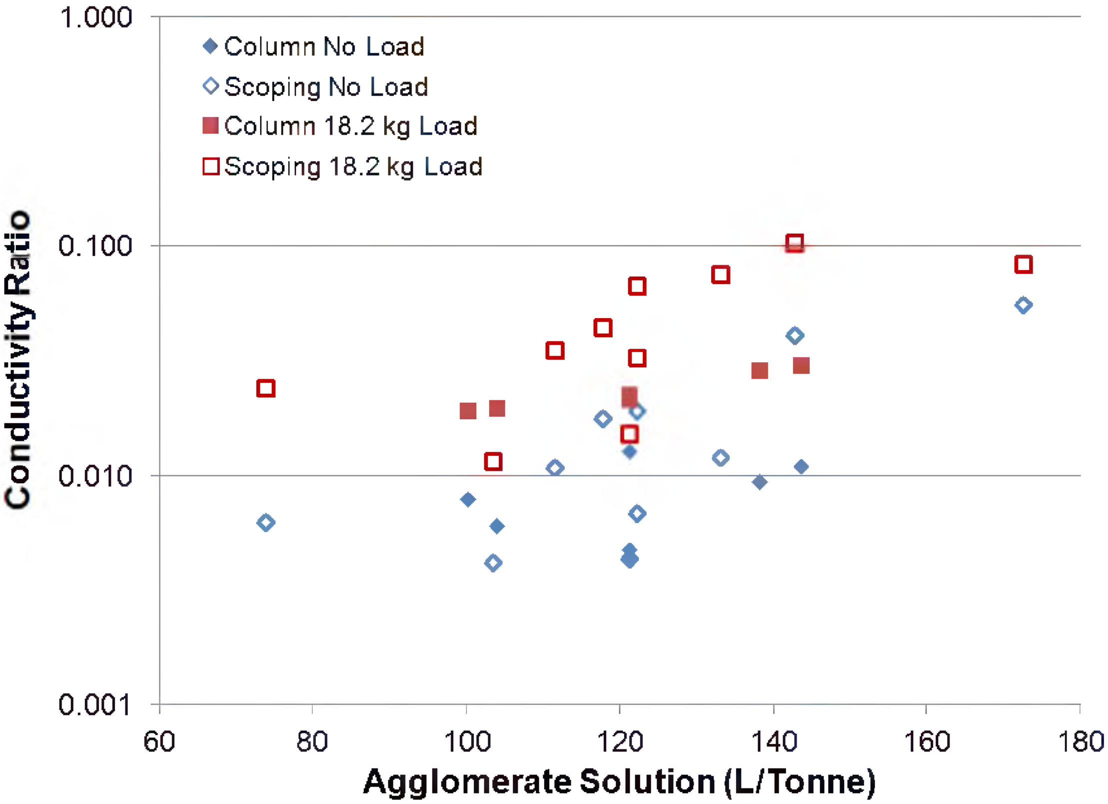

Figure 3.11 Comparison of conductivity ratio of agglomerates produced during scoping experiments with those produced for columns. Mixing speed was fixed at 30% NC. Mixing time was 3 minutes for scoping experiments and 4.5 minutes for columns.

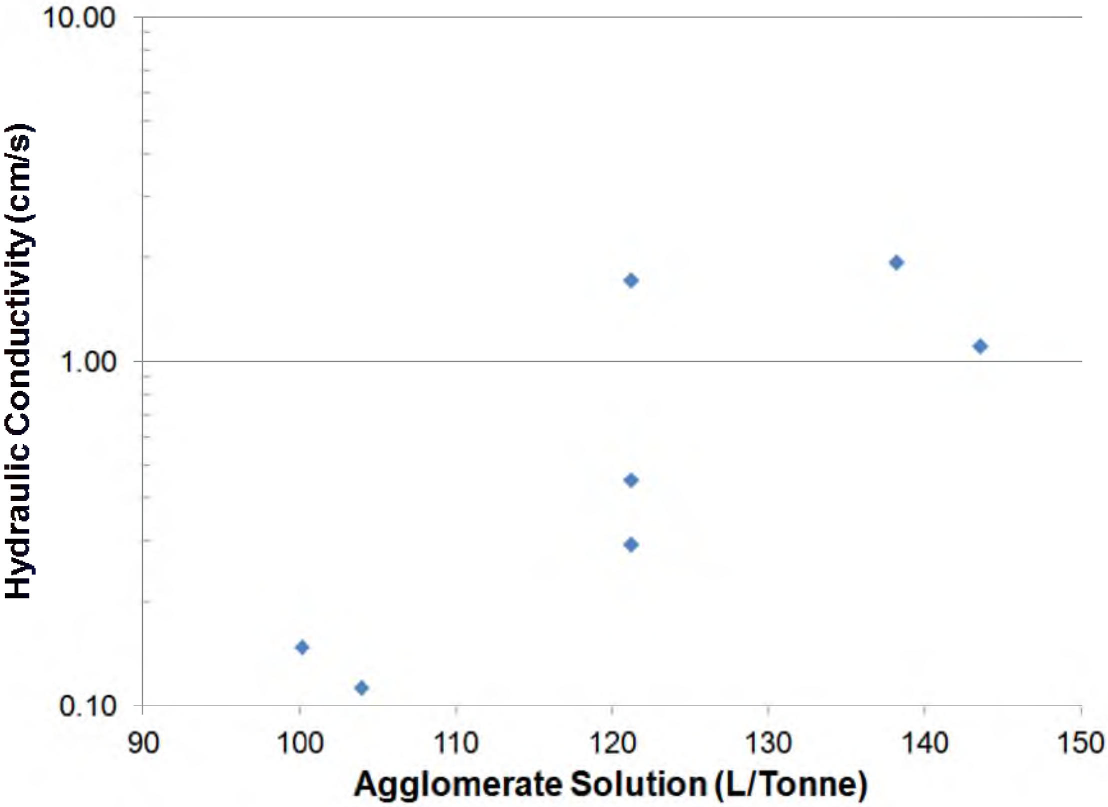

Figure 3.12 Hydraulic conductivity of agglomerates produced for column leaching.

Figure 3.13 shows a comparison of the hydraulic conductivity values measured during the scoping experiments to the agglomerates produced for column leaching. The hydraulic conductivity results from agglomerates produced for column leaching differ from results obtained from scoping experiments. This may be due to the increase in retention time of agglomerates produced for column leaching. Data from Chapter 2 suggest an increase in hydraulic conductivity with retention time. The difference likely is not due to added strength caused by mineralogy differences between the samples used in the column leaching experiments and those used in the scoping experiments. A student's t-test performed on the bulk densities after the permeability test for both the scoping tests and the column tests indicates the values are the same at 95% confidence.

3.2.4 Bulk Density

The bulk density of agglomerated ore was obtained at the beginning and at the end of the permeability test. The bulk density and porosity were calculated as described in Chapter 2. The results are listed in Table 3.4. All of the agglomerates experienced an increase in bulk density and decrease in porosity during the permeability test. While the initial values vary somewhat from one condition to another, they all appear to approach the same value after the permeability test. Following the test, the bulk density and porosity values are only slightly better than they would be for unagglomerated ore. The statistical analysis indicates moisture content significantly affects the initial bulk density (P=0.0191). Because this is a function of agglomerate size and moisture is

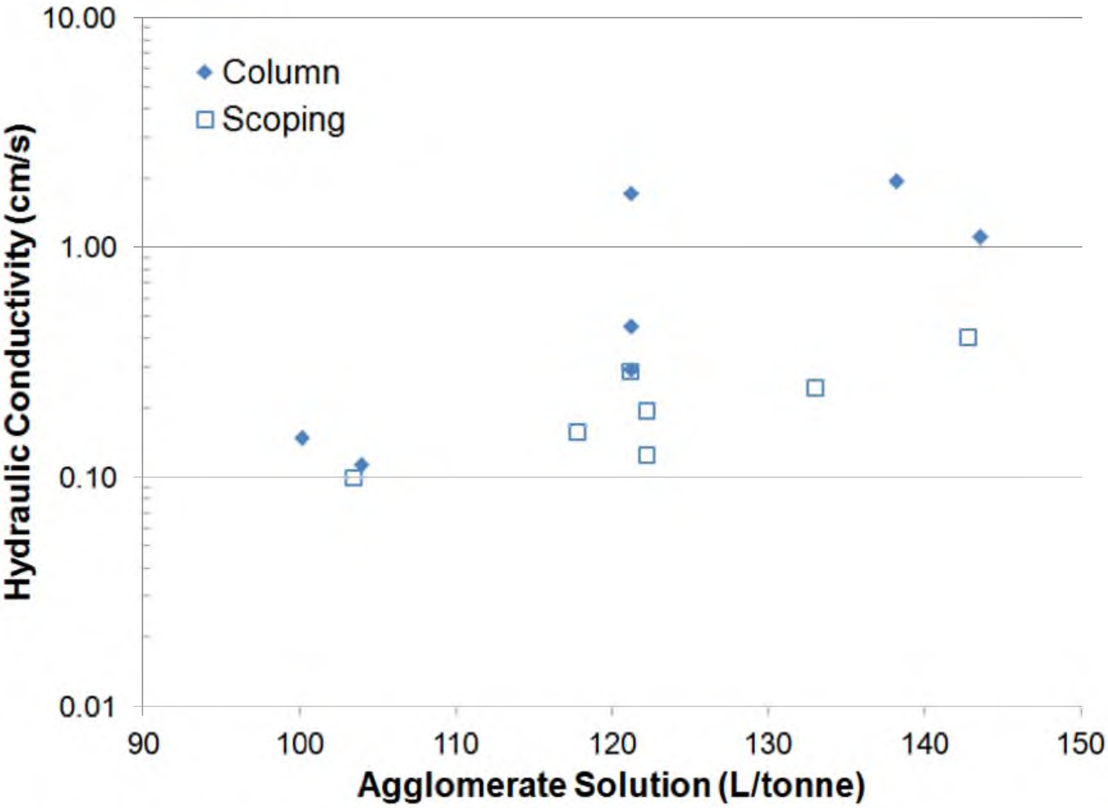

Figure 3.13 Comparison of hydraulic conductivity of agglomerates produced during scoping experiments with those produced for columns. Mixing speed was fixed at 30% NC. Mixing time was 3 minutes for scoping experiments and 4.5 minutes for columns.

Table 3.4 Bulk Density and Porosity data for agglomerates produced for column leaching.

Acid Concentration (g/L)	Moisture Content (%)	Label	Bulk Density (g/mL)		ΔBulk Density (%)	Porosity		ΔPorosity (%)
			Before Perm. Test	After Perm. Test		Before Perm. Test	After Perm. Test	
-	Unagglomerated	-	1.55	-	-	0.32	-	-
30	11.5	4	1.22	1.47	16.9	0.46	0.35	23.5
90	11.5	2	1.24	1.44	13.9	0.46	0.37	19.1
60	13.0	1	1.18	1.43	17.2	0.48	0.37	22.4
		3	1.22	1.49	18.2	0.47	0.35	25.6
		7	1.18	1.39	14.9	0.48	0.39	18.9
30	14.5	6	1.15	1.40	17.9	0.50	0.39	22.2
90	14.5	5	1.12	1.41	20.4	0.51	0.38	24.9

significantly affecting agglomerate size, this is expected. There was no significant effect observed due to moisture or acid on the final bulk density values.

3.2.5 Column Breakthrough

The column breakthrough times are listed in Table 3.5. Columns of greater moisture content experience a shorter breakthrough time. This is expected given our permeability results indicate that greater moisture content leads to higher permeability. Statistical analysis also indicated moisture content affects breakthrough time (P=0.0168). The two columns temporarily shut down at 45 days experienced a dramatic reduction in breakthrough time after being restarted. This was likely due to the ore body already being saturated with leaching solution.

A few problems were encountered during this part of the experiment. Solution delivery to column 1 ceased shortly after start-up. When the column was checked the following day, little slump had occurred and the solution to this column was not flowing. The lab aid assisting with column maintenance mistakenly started column 7 instead of column 5. This resulted in a cure time of 11 days for column 5 and 2 days for column 7. The breakthrough time observed by the lab aid for column 5 seems very short compared to the other columns. When disassembling the columns, it was observed that a perforated plate had not been placed at the bottom of column 5. The conductivity of solution exiting column 6 was too low to complete the circuit and start the clock. This is an indication that ions (ferric, dissolved copper, etc.) are precipitating out of solution.

Table 3.5 Agglomeration conditions for each column and the respective breakthrough times.

Acid Concentration (g/L)	Moisture Content (%)	Label	Breakthrough Time (hours)
30	11.5	4	25.8
90	11.5	2	23.3 / 13.3[a]
60	13.0	1	19.6[b] / 13.9[a]
		3	21.3
		7	21.8
30	14.5	6	14.9[b]
90	14.5	5	3.7[c]

[a] Breakthrough time following temporary shutdown at 45 days
[b] Estimated based on start time and collected volumes
[c] Unusual based on other values

Too little acid used during agglomeration will lead to acid consumption and no initial recovery of copper.

3.2.6 Column Sample pH and ORP

The pH measurements for each column are plotted in Figure 3.14 and ORP measurements are plotted in Figure 3.15. Acid consumption is observed for all columns with consumption very high for the columns prepared at 30 g/L H_2SO_4 (4 and 6). Acid consumption in these columns caused many ions to precipitate out of solution, as was observed both physically with transparent solutions and in the low ORP values. The dramatic change in pH and ORP values observed in columns 1 and 2 between 45 and 50 days of leaching is due to the columns being temporarily shut off for additional testing. Agglomeration conditions will affect the initial leaching behavior; however, after the first month of leaching, the agglomeration conditions have little effect on leaching behavior.

3.2.7 Column Slump

The column slump or decrease in agglomerate bed height is shown in Figure 3.16. All of the columns experienced the majority of slumping during the initial day of leaching. There appears to be a trend of increasing slumping as total solution added during agglomeration increases. Slumping on columns 1 and 2 around day 45 is likely due to settling during transport for additional testing. Statistical analysis performed on the percentage of slump recorded for the first day indicates moisture content strongly affects percentage of slump (P=0.0179). The first day column slump was chosen because most of the slump for all of the

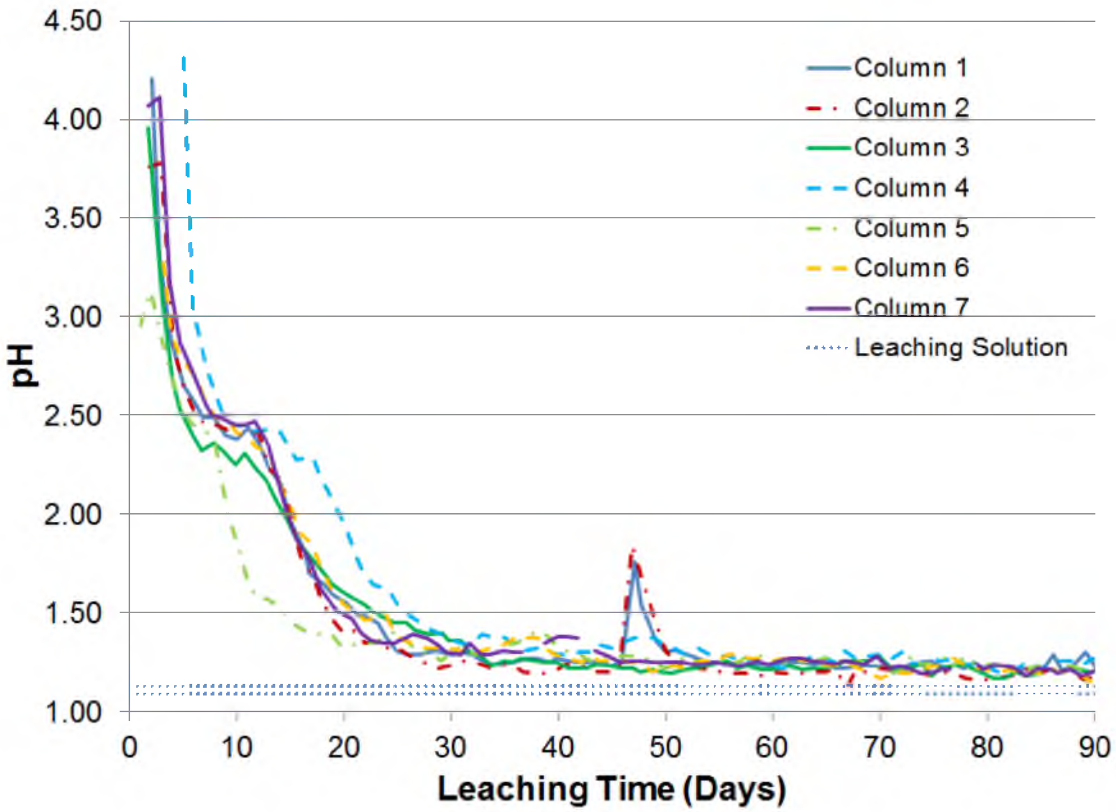

Figure 3.14 pH of column samples and leaching solution. The peak for columns 1 and 2 at 45 days is due to the columns being temporarily shut down for additional testing.

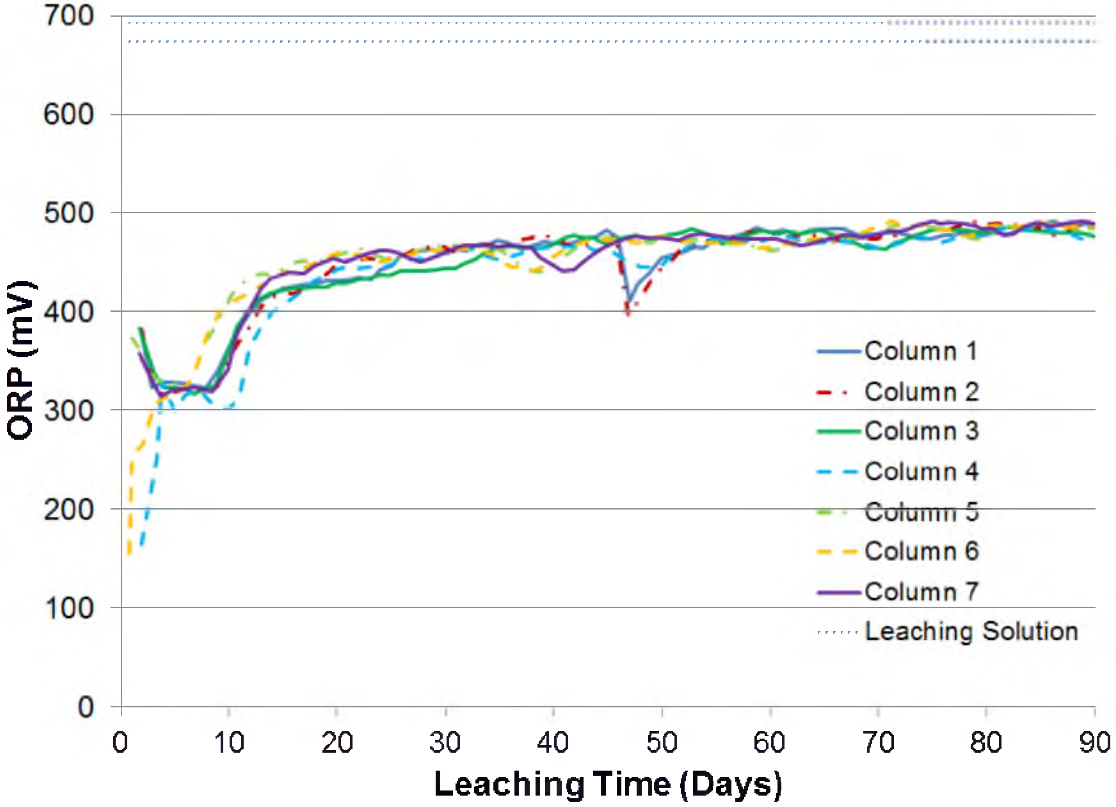

Figure 3.15 ORP values (Standard Hydrogen Electrode) of column samples and leaching solution. The peak for columns 1 and 2 at 45 days is due to the columns being temporarily shut down for additional testing.

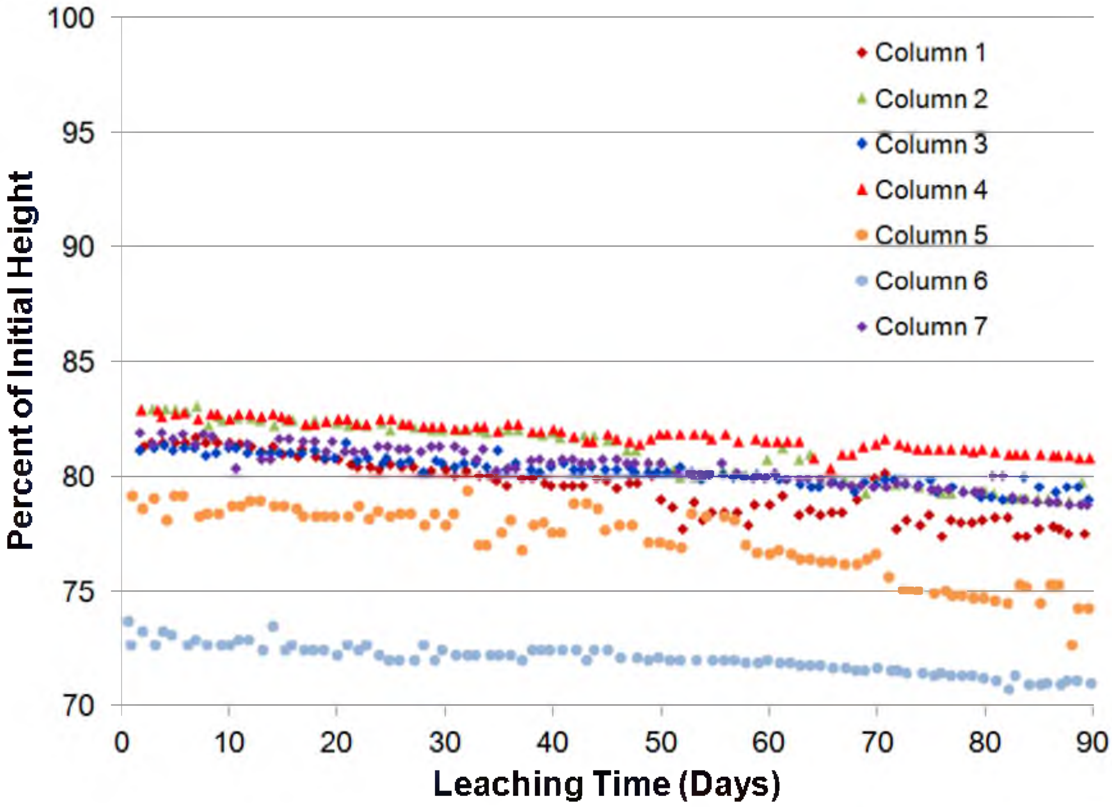

Figure 3.16 Column slump as a percentage of initial agglomerate bed height.

columns had taken place at this point. Again, the significance of moisture on slump is expected because a bed of larger agglomerates should contain larger void spaces and experience a greater slump.

3.2.8 Column Draindown

The column draindown volumes are listed in Table 3.6. The draindown volumes do not appear to correlate with acid concentration or moisture content of agglomerates. Statistical analysis indicates moisture content and acid concentration do not affect draindown volume. The draindown volume measured prior to temporary shutdown on columns 1 and 2 is greater than the volume measured when those columns were shut down at the end of the leaching cycle. This may be due to compaction or other disturbances introduced because of taking down and transporting the columns. The percent of draindown volume with respect to time is show in Figure 3.17. The draindown behavior of each column also did not appear to depend on acid concentration or moisture content.

3.2.9 Copper Recovery

Copper recovery is plotted versus leaching time in Figure 3.18. The curve is based on the original assay of the ore provided by the mine. Since the ore that was leached included fine material manufactured from other size fractions rather than fine material originally provided by the mine, the actual copper content will differ. However, the general trends observed should be valid. Initial recovery increases as H_2SO_4 added during agglomeration increases. After approximately 50 days of leaching, the recoveries of all columns except column 5 are quite

Table 3.6 Draindown volumes after 24 hours

Acid Concentration (g/L)	Moisture Content (%)	Label	Draindown Volume (mL)
30	11.5	4	908
90	11.5	2	886 / 1093[a]
60	13.0	1	775 / 928[a,b]
		3	1060
		7	935
30	14.5	6	719
90	14.5	5	934

[a] Draindown volume at temporary shutdown

[b] Interpolated between 16.25 and 48 hours

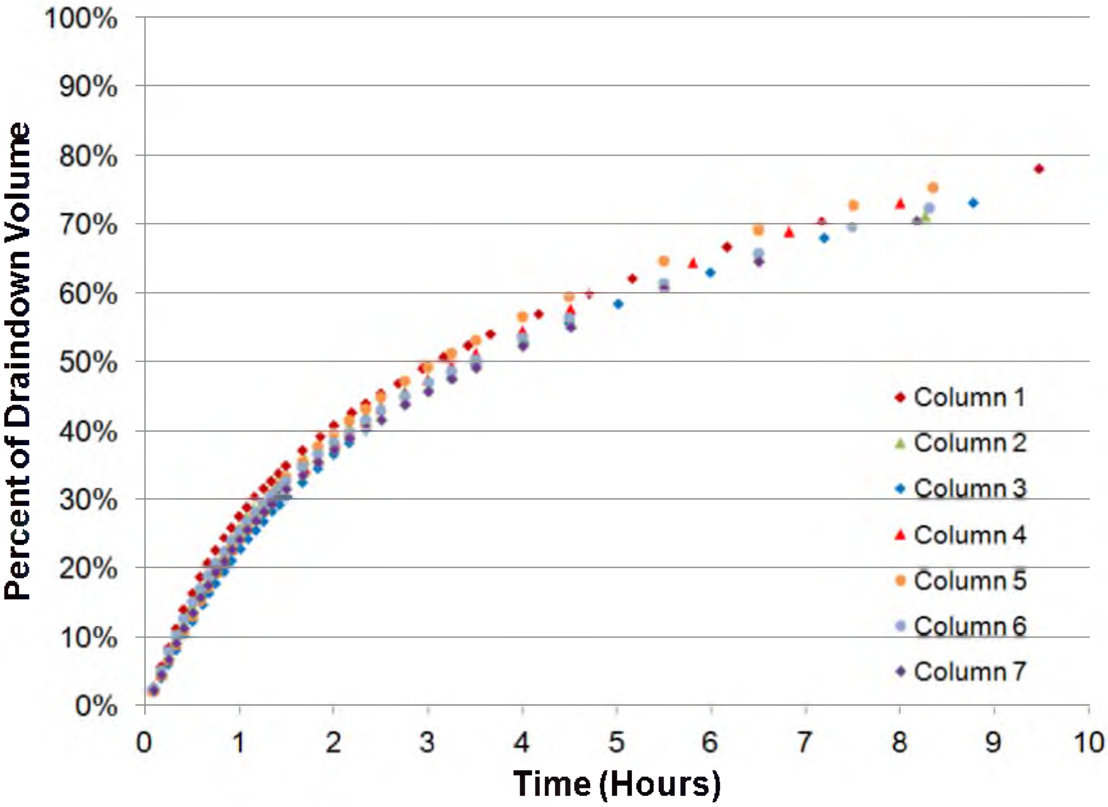

Figure 3.17 Percent of draindown volume as a function of time

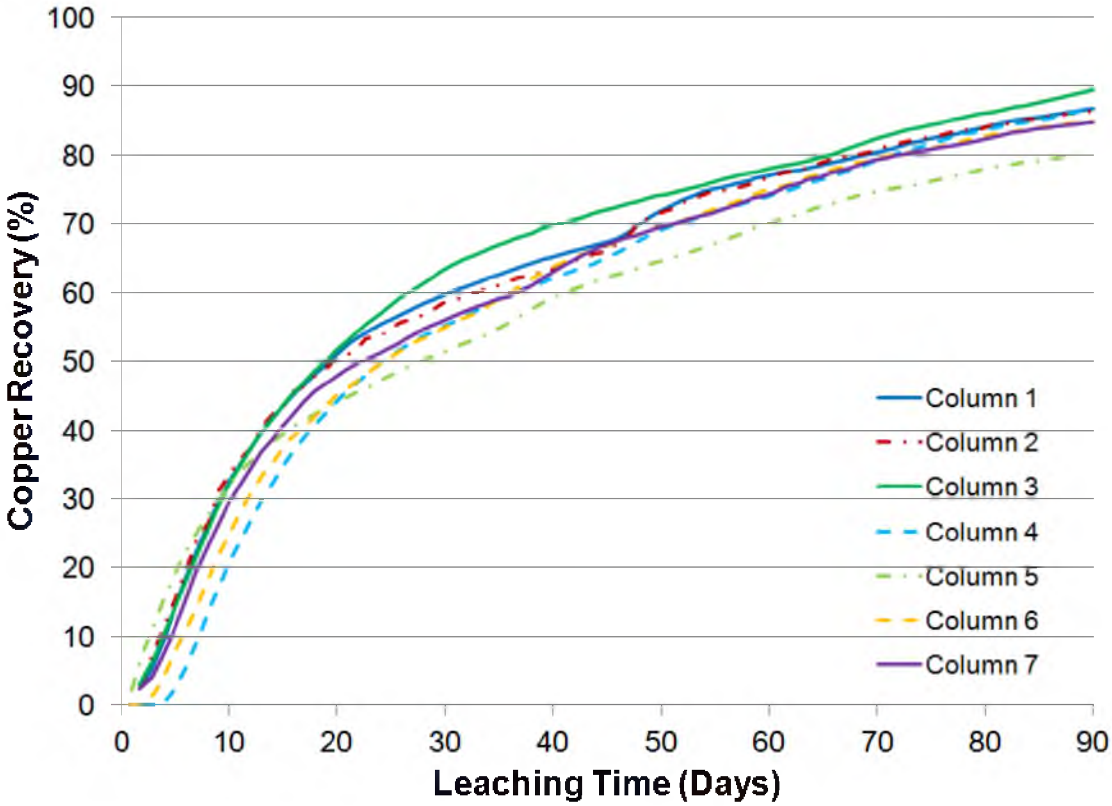

Figure 3.18 Copper recovery from column leaching based on the mine's original assay.

similar. The midpoint conditions bracket all other conditions except column 5 for the remainder of the experiment. These data indicate the columns behave the same over 90 days regardless of agglomerations conditions. The statistical analysis also shows neither acid concentration nor moisture content affect copper recovery after 90 days of leaching. The deviation of behavior for column 5 from the other columns may be due to the longer cure time and presence of air flow without solution flow when column 7 was started by mistake.

3.2.10 Statistical Analysis Results

The statistical analysis showed significant effects from moisture content on measurements relating to agglomerate size. These effects have been discussed in their respective sections. The results of the statistical analysis are recorded in Table 3.7

3.3 Conclusions

Agglomerates produced with a larger volume of solution are larger in size and display a greater hydraulic conductivity prior to leaching. Agglomerates from this ore are highly unstable, as is confirmed by the large increase in bulk density during permeability testing and the magnitude of slumping of the agglomerate bed during the first day of column leaching. Increasing the amount of acid added during agglomeration will reduce acid consumption experienced during leaching and avoid precipitation, which could cause problems in actual heap operation. The agglomeration conditions selected during this experiment did not have an impact on copper recovery.

Table 3.7 P values obtained during statistical analysis

Measurement	P value		
	Moisture Content	Acid Concentration	Moisture Content*Acid Concentration
D50	0.0074[a]	0.0547	0.6319
No Load Conductivity Ratio	0.1397	0.4496	0.8148
Load Conductivity Ratio	0.1986	0.2775	0.5297
Hydraulic Conductivity	0.1379	0.4416	0.5363
Bulk Density Before Test	0.0191[a]	0.7962	0.4182
Bulk Density After Test	0.3409	0.8060	0.6098
Delta Bulk Density	0.0762	0.8730	0.1493
Breakthrough Time	0.0168[a]	0.1179	0.2611
First Day Slump	0.0179[a]	0.1409	0.1460
Draindown Volume	0.6138	0.4982	0.4150
Draindown Volume with 45 days[b]	0.1063	0.0785	0.8564
90 Day Recovery	0.2415	0.4243	0.4899

[a] Significant factor (<0.05)
[b] Substituting 45 day draindown volume for columns 1 and 2

CHAPTER 4

BINDER PROBING EXPERIMENTS

4.1 Experimental

4.1.1 Sample Preparation

For the initial probing experiments to evaluate potential polymers for use as a binder in agglomeration, 200 gram samples were prepared. Because the samples would be agglomerated in a very small drum, the size distribution was modified to remove feed particles larger than 12.7 mm (1/2 inch). In order to maintain particle surface area similar to the original size distribution, additional 0.635 mm (1/4 inch) material was added. The resulting size distribution is shown in Table 4.1. Upon identifying potential polymers, the size distribution for scale-up and validation is the same as has been used in previous sections, as shown in Table 4.2.

4.1.2 Probing Agglomeration

The agglomeration procedure was modified to generate reproducible agglomerates for polymer probing. Total mixing time was fixed at 2 minutes. Solution was applied for the first minute of mixing. A plastic bottle was used as the agglomerating drum. The drum has dimensions of 9.0 cm diameter and a length of 17.0 cm. The agglomerating drum was equipped with 3 lifters of 0.6 cm

Table 4.1 Modified size distribution used in polymer probing work

Top Size (mm)	Bottom Size (mm)	Percent of Ore
	19.1	0.00%
19.1	12.7	0.00%
12.7	6.4	50.19%
6.4	1.7	15.64%
1.7	0.50	5.06%
0.50	0.15	1.09%
0.15		28.02%

Table 4.2 Size distribution used in scale-up

Top Size (mm)	Bottom Size (mm)	Percent of Ore
	19.1	9.00%
19.1	12.7	24.00%
12.7	6.4	24.00%
6.4	1.7	13.50%
1.7	0.50	4.37%
0.50	0.15	0.94%
0.15		24.19%

thickness, 2.0 cm width, and 14.3 cm length, which was centered between the two ends of the drum. The lifters were equally spaced around the drum's internal circumference.

The critical speed of the drum was calculated as 141.8 rpm. Thus, 42.6 rpm was used to produce 30% critical speed. The drum was filled with 200 g of sample (copper ore), which resulted in about 12.0 % volume fill of the drum. To achieve homogeneity, the sample was mixed for 5 minutes in the drum prior to solution addition. A 60 g/L acid solution was mixed with the polymer and delivered to the drum using a peristaltic pump. The samples were prepared at 13.0% moisture, which resulted in the delivery of 24 mL of solution.

4.1.3 Scale-Up Agglomeration in 5 kg Batches

A plastic drum was used as the agglomerator. The drum has dimensions of 26.7 cm diameter and a length of 36.2 cm. The agglomerating drum was equipped with 4 lifters of 0.5 cm thickness and 20 mm width, which ran the length of the drum and were equally spaced around the drum's internal circumference. The critical speed of the drum was calculated as 81.9 rpm. Thus, 24.6 rpm was used to produce 30% of critical speed. All tests were performed with 5 kg batches, which resulted in about 15.9% volume fill of the drum. To achieve homogeneity, the sample was mixed for 15 minutes in the drum prior to solution addition.

The polymer solution, consisting of deionized water and polymer, and concentrated sulfuric acid were added using peristaltic pumps and separate hoses for the respective solutions. Solution was applied for the first 30 seconds of mixing. After 30 seconds of mixing without solution addition, solution was applied for an additional 30 seconds. Mixing occurred for another 90 seconds without solution addition. The total mixing time was 3 minutes. The moisture content was fixed at 13.0% for all scale-up experiments.

4.1.4 Polymer Details

Several polymers were investigated for use as a binder in agglomeration. The polymers were either anionic, cationic, or nonionic charged molecules. Polymer charge and an identifying label are listed in Table 4.3. The identity of the polymers has been withheld for proprietary reasons.

4.1.5 Polymer Preparation

During the probing experiments, polymer solution was prepared using a 60 g/L sulfuric acid solution and the polymer. The dosages and polymer solution concentrations initially studied using four polymers are shown in Table 4.4. Following these experiments, a target dosage of 0.5 kg polymer per tonne of ore was used for the examination of the other polymers.

During the 5 kg batch scale-up experiments, polymer solution was prepared in deionized water. The dosage used in the scale-up experiments was 0.5 kg polymer per tonne of ore (4.27 g polymer per liter of water). For all experiments, the polymer was stirred for at least 24 hours to allow the polymer to dissolve, resulting in transparent homogeneous solutions.

Table 4.3 Polymer charges and label

Charge	Abbreviation	Charge	Abbreviation
Anionic	A-1	Cationic	C-10
Anionic	A-2	Cationic	C-11
Anionic	A-3	Cationic	C-12
Anionic	A-4	Cationic	C-13
Anionic	A-5	Cationic	C-14
Anionic	A-6	Cationic	C-15
Anionic	A-7	Cationic	C-16
Anionic	A-8	Cationic	C-17
Cationic	C-1	Cationic	C-18
Cationic	C-2	Cationic	C-19
Cationic	C-3	Cationic	C-20
Cationic	C-4	Nonionic	N-1
Cationic	C-5	Nonionic	N-2
Cationic	C-6	Nonionic	N-3
Cationic	C-7	Nonionic	N-4
Cationic	C-8	Unknown	U-1
Cationic	C-9	Unknown	U-2

Table 4.4 Polymer dosages investigated with four possible binders

Target Dosage (kg/tonne)	Polymer Concentration (g/L)
0.2	1.67
0.5	4.17
1	8.3
1.5	12.50
2	16.67
2.5	20.83

4.1.6 Soak Test

The soak test was used to determine if a given polymer affected agglomerate strength. The soak test was only performed during the probing experiments. Following agglomeration, the agglomerates were placed in a plastic bag for 24 hours of “wet curing.” After 24 hours, the agglomerates were placed as a monolayer on a tared 2.0 mm screen. The screen was carefully submerged in a 3 ½ gallon bucket containing approximately 3 L of 7.5 g/L H_2SO_4 solution so that all agglomerates were fully submerged. After 30 minutes of soaking, the screen was carefully removed and placed in an oven to dry for 24 hours. The remaining solution was passed through a tared Whatman® 1 filter using a filter press. The filter was also placed in the oven to dry for 24 hours. After 24 hours of drying, the screen and filter were weighed to determine the mass of the filter cake and the mass of the agglomerates. The percent of fines migration was determined using equation 4.1.

$$\text{Percent Fines Migration}=\frac{\text{Mass}_{\text{Filter Cake}}}{\text{Mass}_{\text{Filter Cake}}+\text{Mass}_{\text{Agglomerates}}}\times 100\% \qquad (4.1)$$

4.1.7 Quality Control Tests

Agglomerate size distribution, electrical conductivity, and permeability tests were performed using the same methods described in Chapter 2. For ASD determination, the only deviation from the method was that the samples were twice the size as previously used. These tests were only performed on the scale-up experiments. The TBS test was not performed on agglomerates produced during this phase of the project.

4.2 Results and Discussion

4.2.1 Dosage Scoping

In order to prepare for screening several polymers, an appropriate dosage needed to be determined. The four polymers selected for dosage scoping were A-5, C-18, N-3, and C-1. Replicates were produced for each condition. Some conditions were not tested for N-3 and C-1 because the polymer supply was depleted. The fines migration results from these scoping experiments are shown in Figures 4.1 through 4.4. The 0.5 kg/tonne dosage was selected for polymer screening because of its reproducibility and potential economic feasibility for use on an industrial scale.

4.2.2 Polymer Screening

All of the polymers listed in Table 4.3 were screened using a dosage of 0.5 kg/tonne. The average for samples tested previously during the dosage scoping experiments is used in this section. The fines migrations for samples agglomerated with anionic polymers are shown in Figure 4.5. Cationic polymer data are shown in Figures 4.6 through 4.8. The fines migrations for nonionic polymers and those where the charge is unknown are shown in Figure 4.9.

In general, cationic polymers appear to reduce fines migration the most. With the exception of one cationic polymer tested, all of the agglomerates prepared with cationic polymers had lower fines migrations than when no

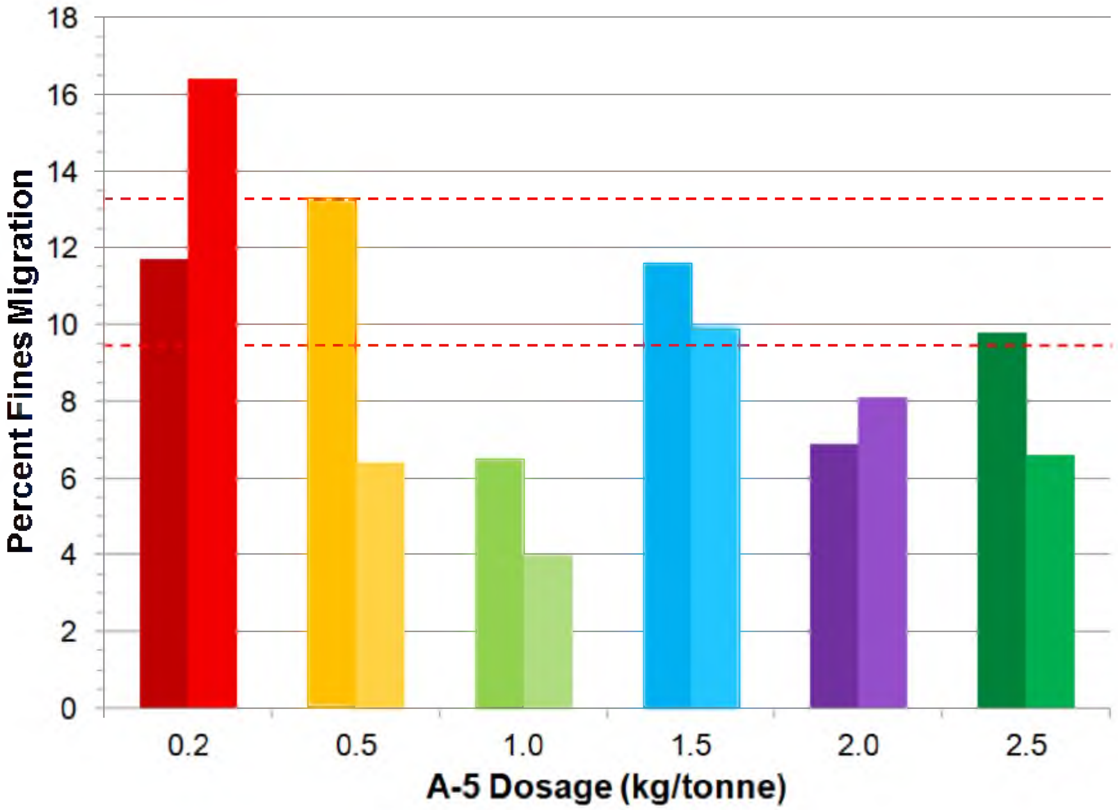

Figure 4.1 Fines migration observed at varying dosages of A-5. The dashed line is the range of migration from 11 experiments conducted with no polymer added.

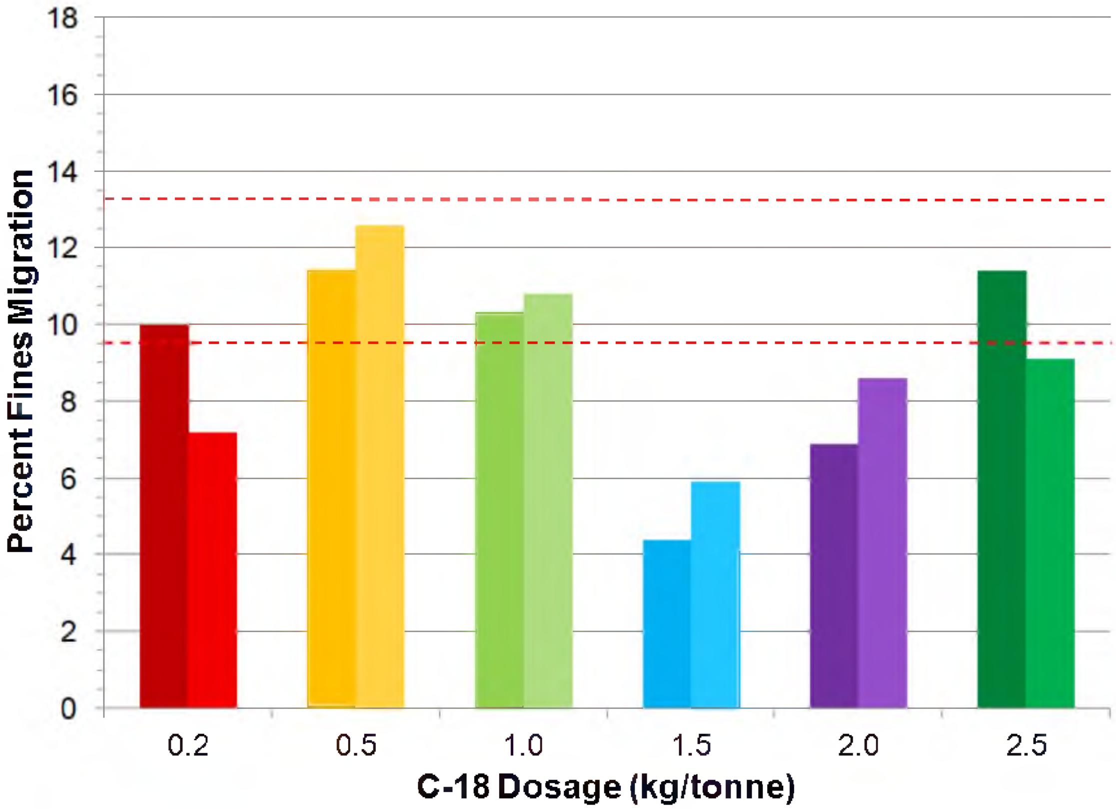

Figure 4.2 Fines migration observed at varying dosages of C-18. The dashed line is the range of migration from 11 experiments conducted with no polymer added.

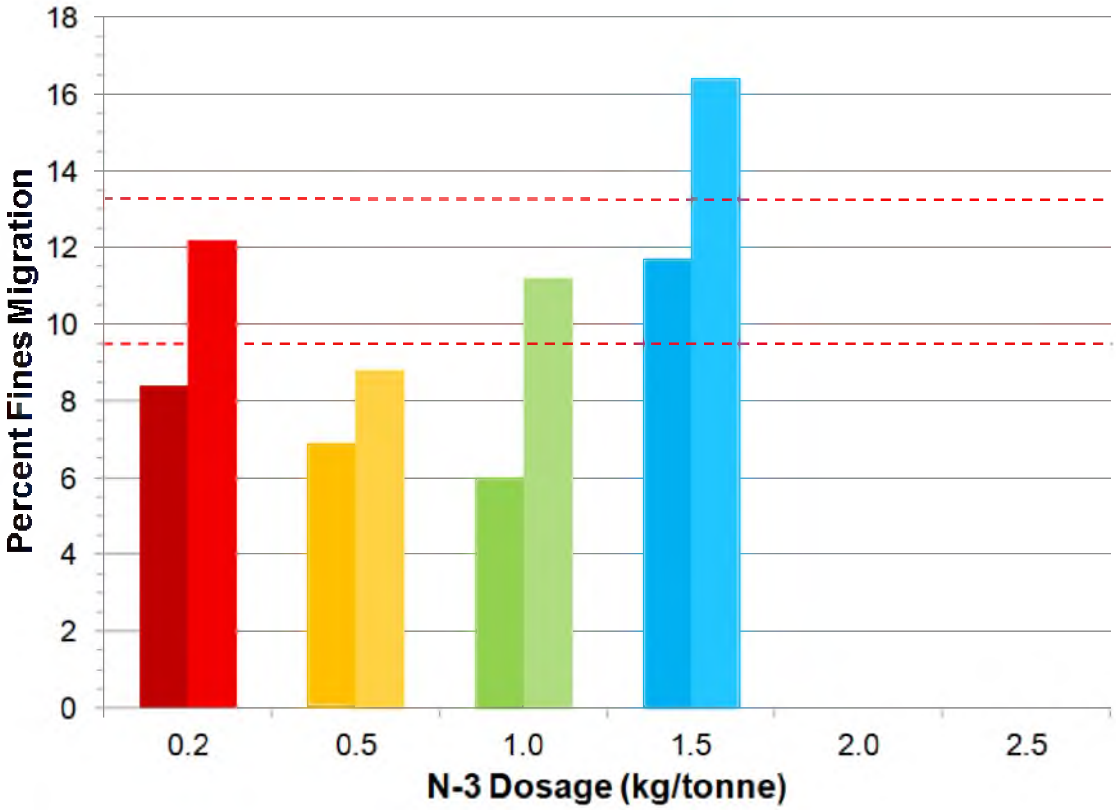

Figure 4.3 Fines migration observed at varying dosages of N-3. The dashed line is the range of migration from 11 experiments conducted with no polymer added.

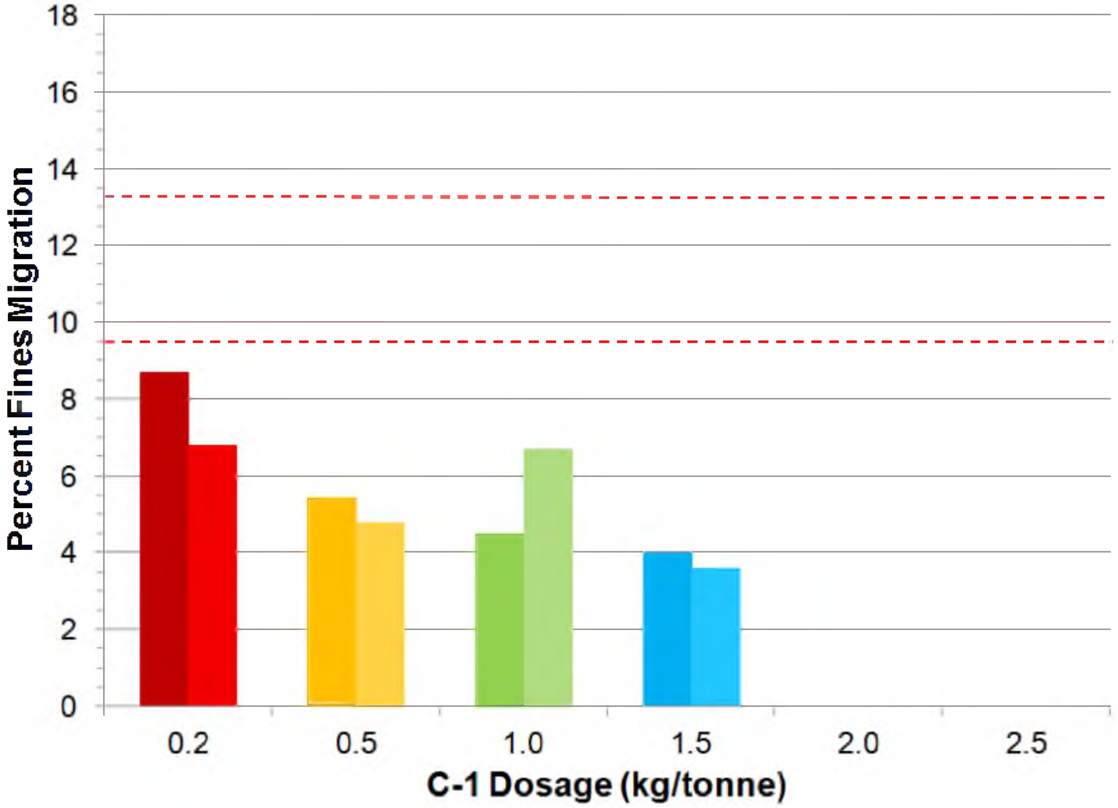

Figure 4.4 Fines migration observed at varying dosages of C-1. The dashed line is the range of migration from 11 experiments conducted with no polymer added.

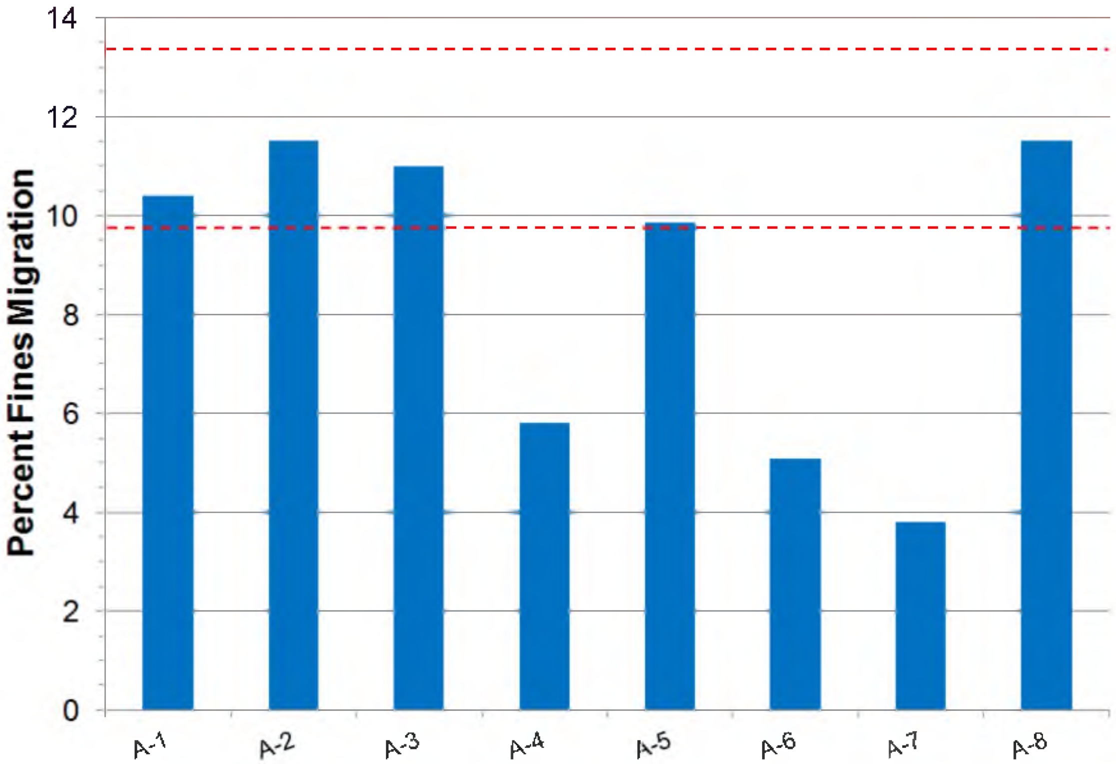

Figure 4.5 Fines migration observed at 0.5 kg/tonne dosage of various anionic polymers. The dashed line is the range of migration from 11 experiments conducted with no polymer added.

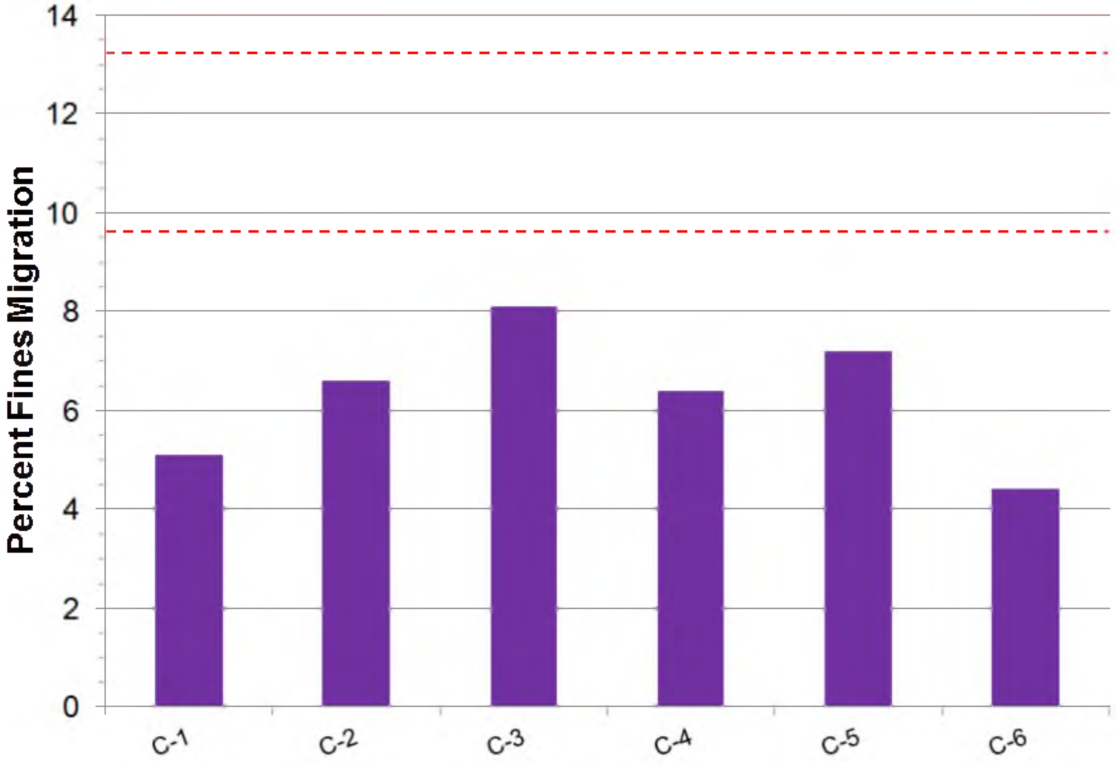

Figure 4.6 Fines migration observed at 0.5 kg/tonne dosage of various cationic polymers. The dashed line is the range of migration from 11 experiments conducted with no polymer added.

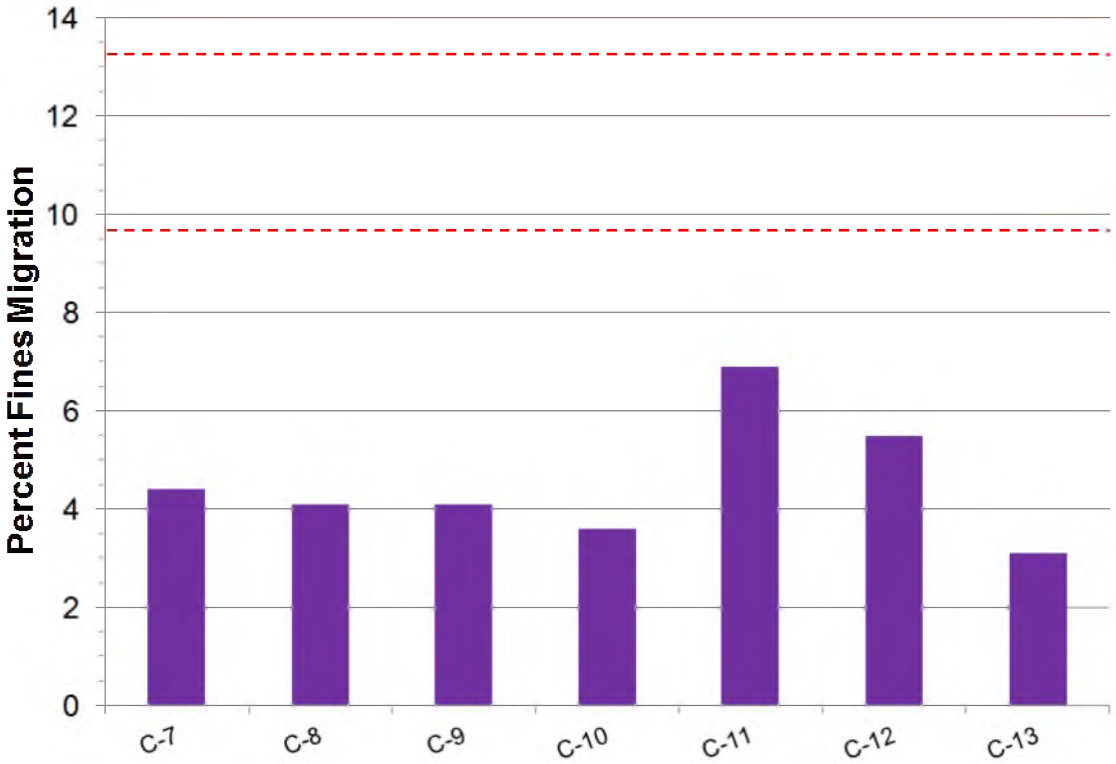

Figure 4.7 Fines migration observed at 0.5 kg/tonne dosage of various cationic polymers. The dashed line is the range of migration from 11 experiments conducted with no polymer added.

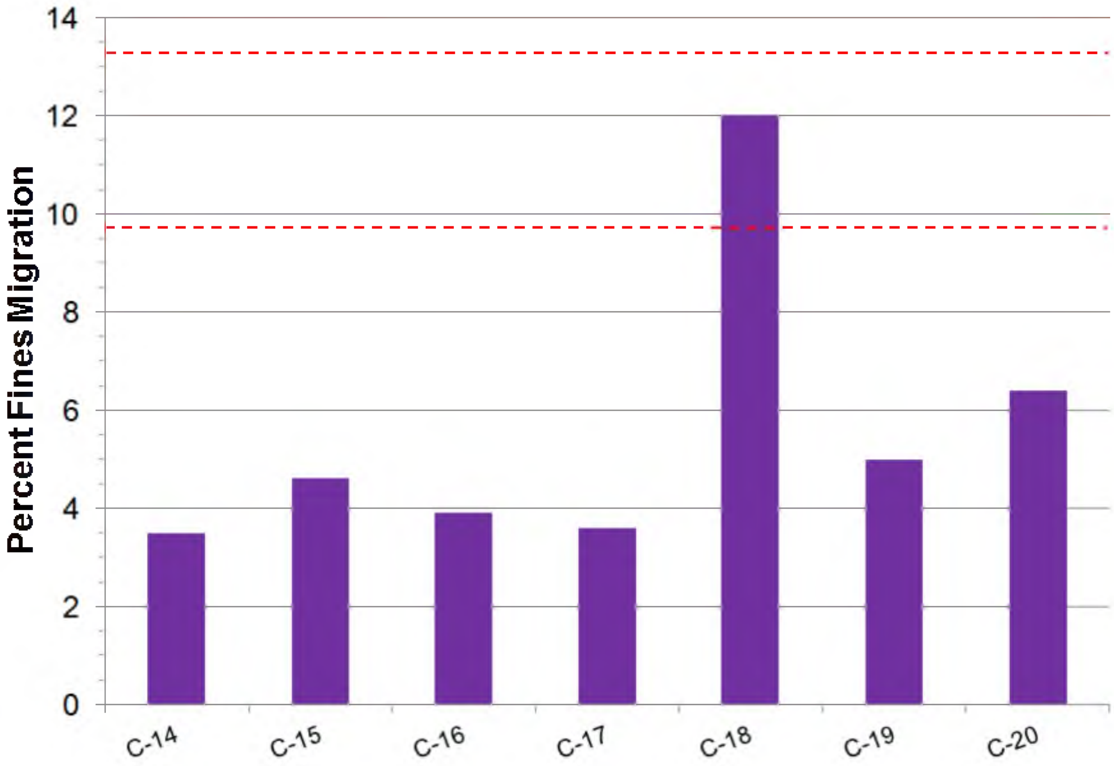

Figure 4.8 Fines migration observed at 0.5 kg/tonne dosage of various cationic polymers. The dashed line is the range of migration from 11 experiments conducted with no polymer added.

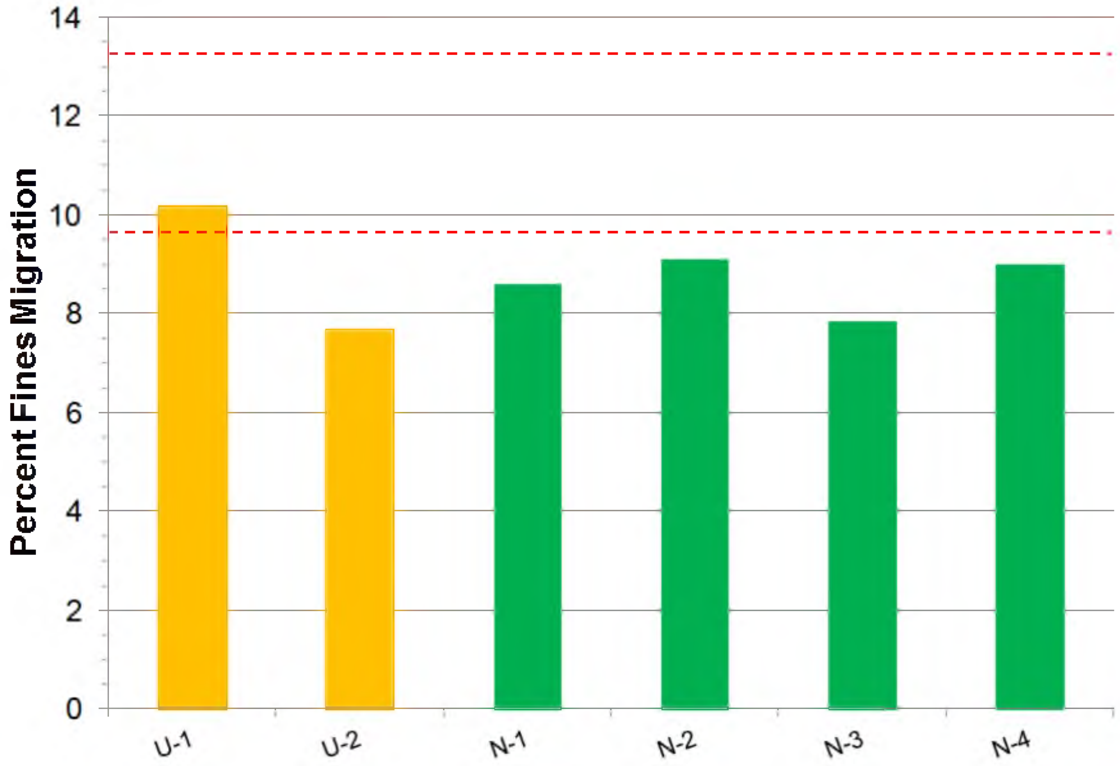

Figure 4.9 Fines migration observed at 0.5 kg/tonne dosage of various nonionic polymers (green) and polymers of unknown charge (yellow). The dashed line is the range of migration from 11 experiments conducted with no polymer added.

polymer was used. It has been shown that kaolinite surfaces contain silanol and aluminol groups[28]. Mineralogy data provided by the mine indicate the sample ore being tested contains approximately 40 % kaolinite. Nasser and James concluded that hydrogen bonds are formed with these groups. Their study of flocculation of kaolinite showed improved strength of flocculated suspensions produced with cationic polymers versus anionic polymers[29]. These scoping experiments indicate there are many polymers that could possibly be used to assist agglomeration of this ore.

4.2.3 Reproducibility Testing

4.2.3.1 First Selection

Polymers that produced agglomerates with fines migrations of 5% or less were retested. The fines migrations of both the original test and retest of these polymers are shown in Figures 4.10 and 4.11. Six of the twelve polymers tested were selected for additional reproducibility testing. The selection was based upon lower variability and lower average fines migration. For example, C-9 was selected over C-7 due to its slightly lower average fines migration even though its variability was slightly more.

4.2.3.2 Second Selection

The results of polymers which were tested in triplicate are shown in Figure 4.12. All of the polymers selected for triplicate testing were cationic. While any of the polymers which underwent triplicate testing would likely improve

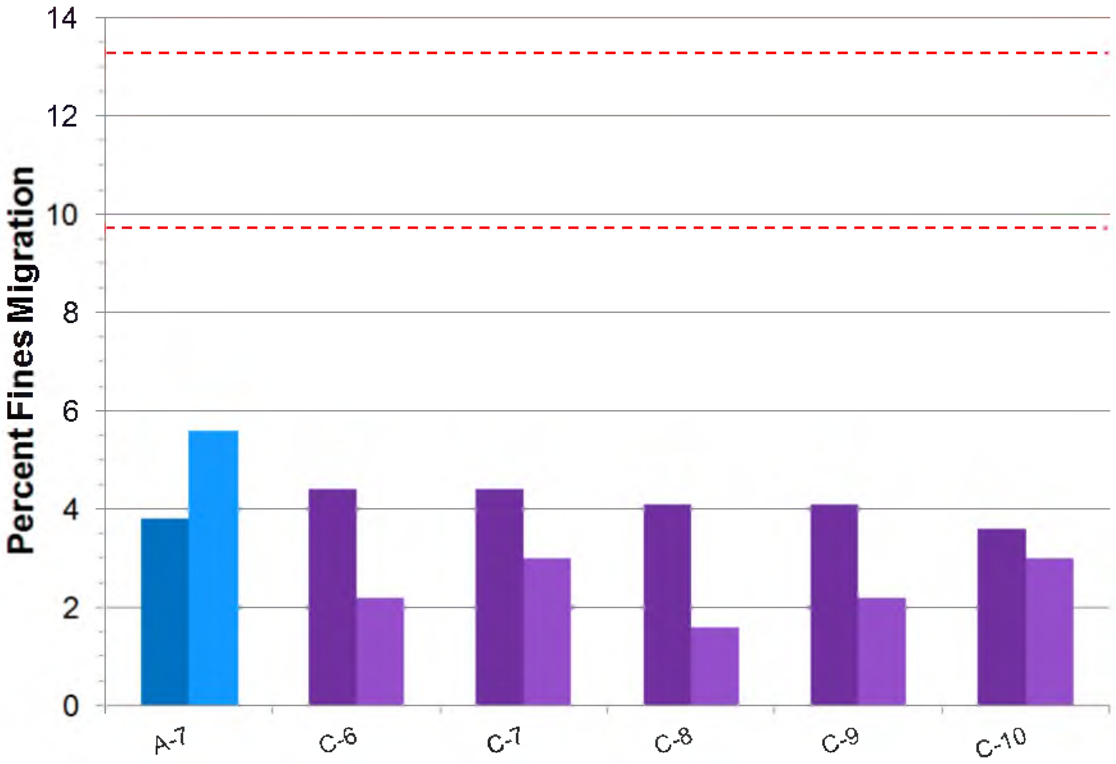

Figure 4.10 Fines migration observed at 0.5 kg/tonne dosage of polymers selected for duplicate testing. Blue is anionic. All others are cationic. The dashed line is the range of migration from 11 experiments conducted with no polymer added.

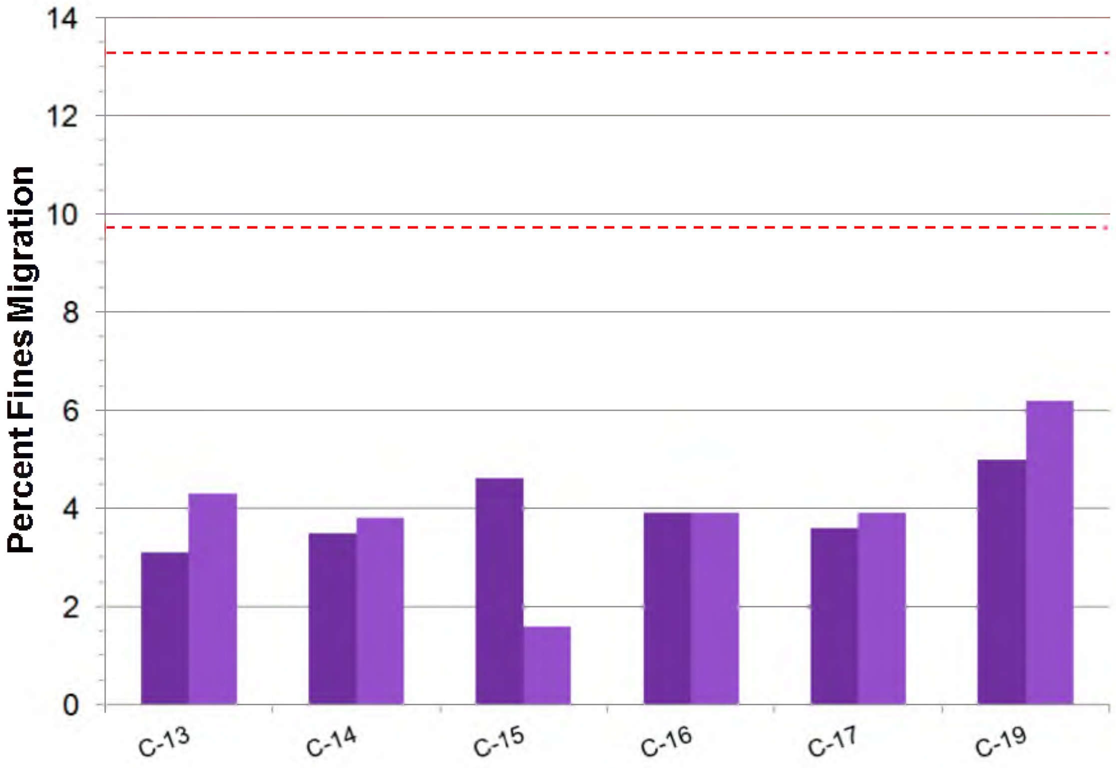

Figure 4.11 Fines migration observed at 0.5 kg/tonne dosage of polymers selected for duplicate testing. The dashed line is the range of migration from 11 experiments conducted with no polymer added.

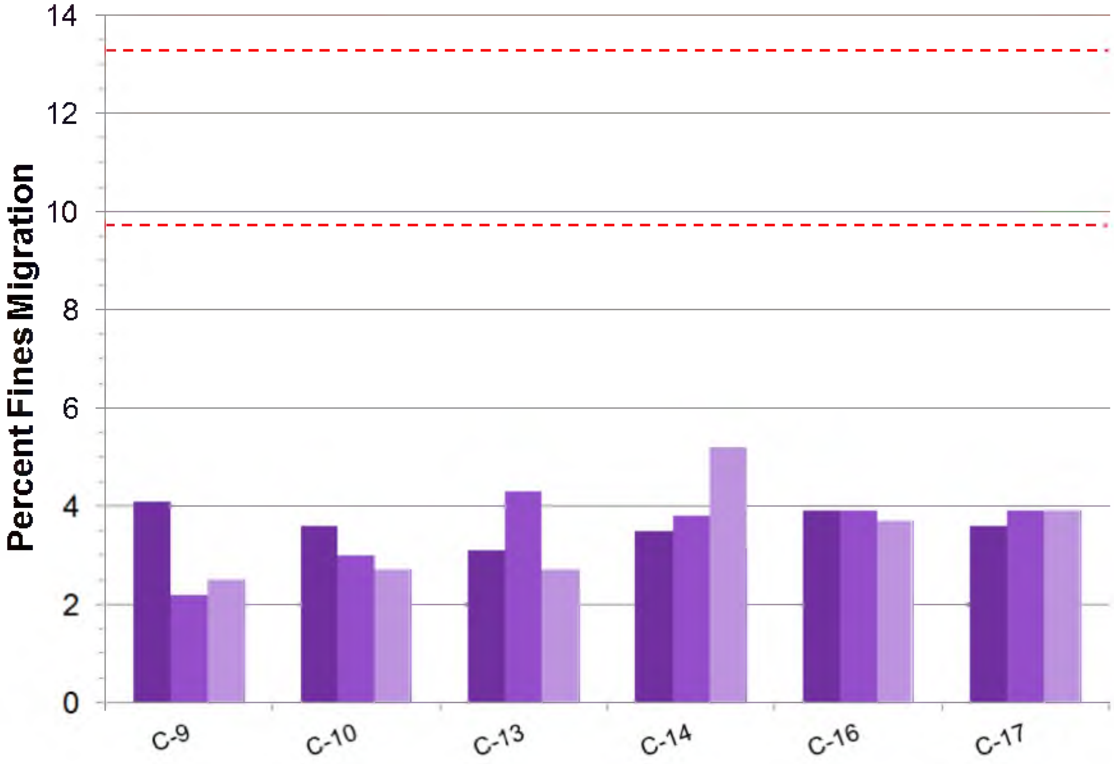

Figure 4.12 Fines migration observed at 0.5 kg/tonne dosage of polymers selected for triplicate testing. The dashed line is the range of migration from 11 experiments conducted with no polymer added.

agglomerate stability, the two most reproducible polymers, C-16 and C-17, were selected for scale-up testing.

4.2.4 Scale-Up Testing

The purpose of the polymer probing experiments was to select a polymer which would improve agglomerate strength the most. In order to compare agglomerates produced without polymer addition to those where a polymer binder is added, agglomeration was performed in 5 kg batches. Three batches were produced for each condition. Agglomeration was performed without polymer three separate times (9 batches total) to establish a control. The results of the quality control tests are presented in subsequent sections.

4.2.4.1 Agglomerate Size Distributions

The ASDs obtained from the scale-up experiments are shown in Figure 4.13. The D_{50} and D_{10} values are shown in Table 4.5. The agglomerates were smaller when produced using polymer than they were when no polymer was added. This may be due to the polymer solution being more viscous than the deionized water used when no polymer is added. Mills et al. also observed a decrease in agglomerate size of powders with higher viscosity fluids[30]. An increase in viscosity may reduce the ability of the solution to spread during agglomeration.

4.2.4.2 Electrical Conductivity

The electrical conductivity and conductivity ratio of agglomerates produced during scale-up are shown in Table 4.6. The agglomerates produced

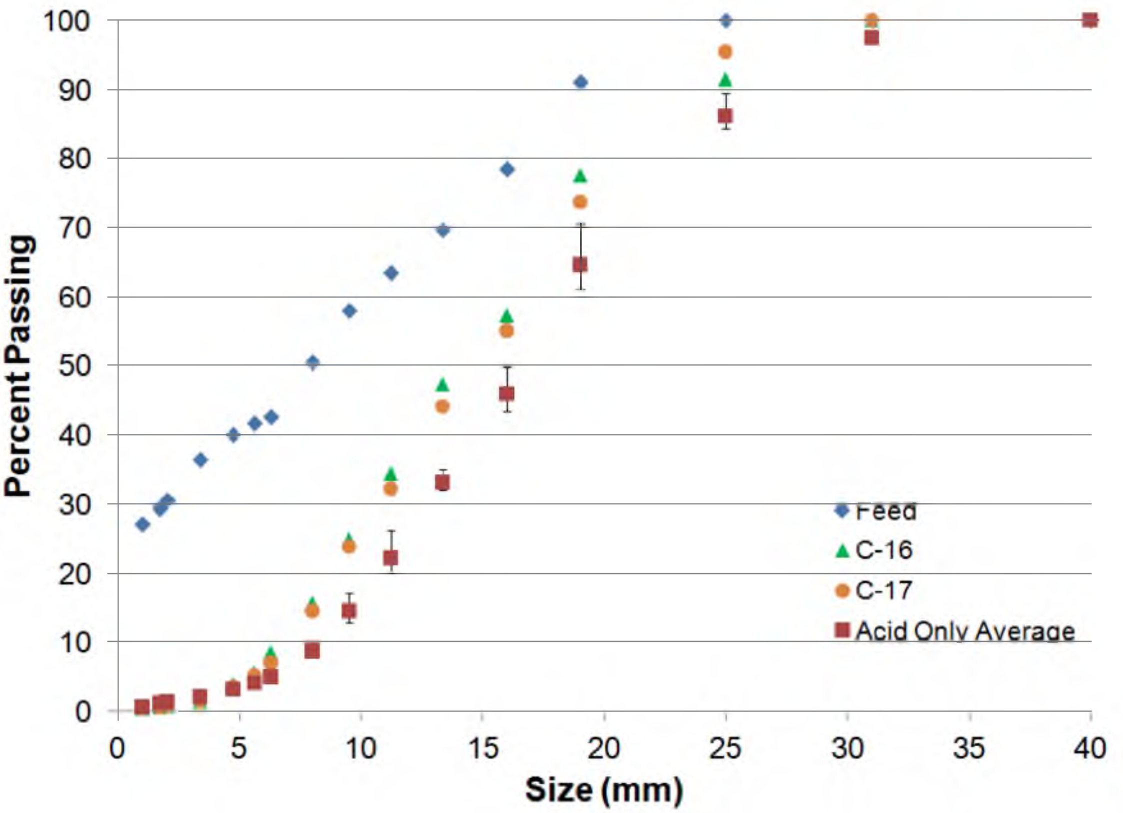

Figure 4.13 Size distribution of agglomerates produced with and without polymer at 13.0% moisture and 60 g/L H_2SO_4. Errors indicate minimum and maximum values observed.

Table 4.5 D50 and D10 for agglomerates produced in 5 kg batches

	D50 (mm)	D10 (mm)
C-16	14.0	6.7
C-17	14.8	7.0
Acid Only (Average)	16.7	8.4

Table 4.6 Conductivities and conductivity ratios for agglomerates produced with and without polymer.

Binder	Conductivity (S/m)		Conductivity Ratio	
	No Load	18.2 kg Load	No Load	18.2 kg Load
C-16	5.2×10^{-3}	8.32×10^{-3}	2.38×10^{-2}	3.79×10^{-2}
C-17	5.66×10^{-3}	8.52×10^{-3}	2.58×10^{-2}	3.88×10^{-2}
Acid Only (1)	2.58×10^{-3}	4.10×10^{-3}	1.18×10^{-2}	1.87×10^{-2}
Acid Only (2)	2.88×10^{-3}	4.95×10^{-3}	1.31×10^{-2}	2.26×10^{-2}
Acid Only (3)	3.31×10^{-3}	7.82×10^{-3}	1.51×10^{-2}	3.57×10^{-2}

with polymer appear to be slightly more conductive than those agglomerated with only acid, although the response from the control samples have some scatter. This may be due to the smaller size of the agglomerates produced with polymer. Smaller agglomerates should be more closely packed, which would increase electrical conductivity. The polymer may also be helping to retain moisture, which would increase electrical conductivity.

The electrical conductivities measured without a load applied are statistically different at 95% confidence for agglomerates produced with polymer compared to those with acid only, whereas the conductivity measured when a load is applied is statistically identical for agglomerates produced with and without polymer.

4.2.4.3 Bulk Density

The bulk density of agglomerated ore was obtained at the beginning and at the end of the permeability test, as described in Chapter 2. The bulk density results are shown in Table 4.7. While all of the agglomerates experienced an increase in bulk density and decrease in porosity during the permeability test, these changes were dramatically reduced for those produced with polymer.

Figures 4.14 and 4.15 show the permeability column before and after testing for each of the three scale-up conditions. It is seen that agglomerates produced with polymer maintain their structure during the test.

Table 4.7 Bulk Density and Porosity data for agglomerates produced with and without polymer

Binder	Bulk Density (g/mL) Before Perm. Test	Bulk Density (g/mL) After Perm. Test	ΔBulk Density (%)	Porosity Before Perm. Test	Porosity After Perm. Test	ΔPorosity (%)
C-16	1.16	1.28	9.0	0.49	0.44	10.4
C-17	1.22	1.30	6.2	0.47	0.43	7.5
Acid Only (1)	1.12	1.45	22.7	0.51	0.36	28.3
Acid Only (2)	1.04	1.40	26.1	0.55	0.39	29.3
Acid Only (3)	1.19	1.45	17.8	0.48	0.36	23.7

Figure 4.14 Permeability column before testing (A) with C-16 (B) with acid only (C) with C-17.

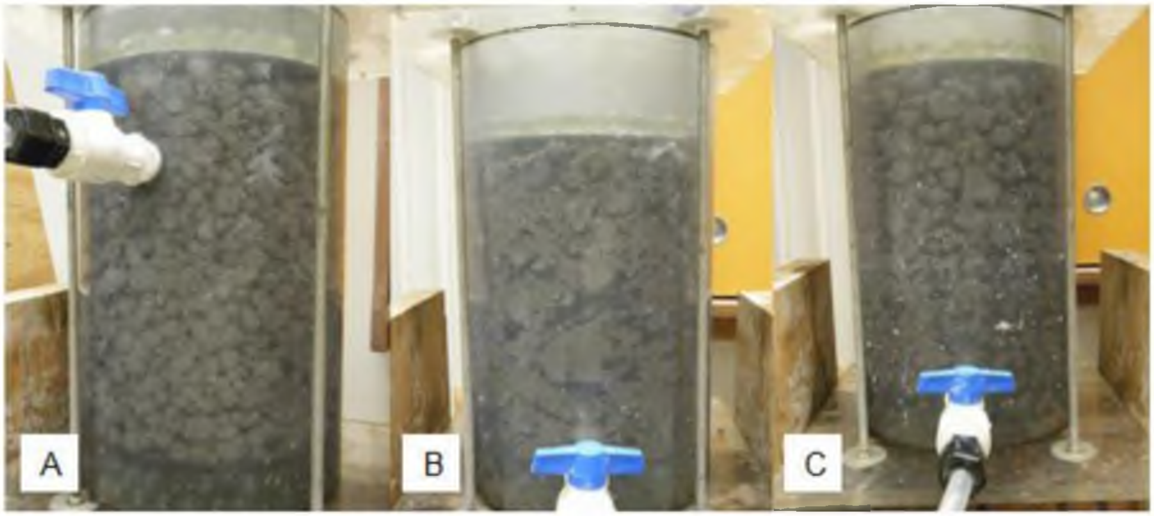

Figure 4.15 Permeability column after testing (A) with C-16 (B) with acid only (C) with C-17.

4.2.4.4 Permeability

The hydraulic conductivities of agglomerates produced during scale-up are shown in Table 4.8. One of the measurements for the acid only tests was compromised and that value is not reported. The agglomerates with polymer showed a hydraulic conductivity an order of magnitude larger than those without polymer. As is seen in Figure 4.15, agglomerates with polymer maintained their structure throughout the permeability test, which is likely the reason for the dramatic increase in hydraulic conductivity.

4.3 Conclusions

The addition of polymer during agglomeration can improve agglomerate strength and potentially heap permeability. Cationic polymers are most likely to improve agglomerate strength for the ore tested. While several polymers appear to be able to improve agglomerate strength, C-16 and C-17 were demonstrated to improve agglomerate strength, as has been shown primarily through the bulk density measurements before and after saturated permeability measurements. Because of this increase in strength, the agglomerate bed will be more permeable, as is seen in the increased saturated hydraulic conductivity measurement. The addition of polymer leads to a slightly less coarse size distribution of agglomerates compared to those produced without polymer.

Table 4.8 Hydraulic conductivity for agglomerates produced with and without polymer.

Binder	Hydraulic Conductivity (cm/s)
C-16	3.4
C-17	5.9
Acid Only (1)	-
Acid Only (2)	0.25
Acid Only (3)	0.21

CHAPTER 5

CONCLUSIONS

5.1 Agglomerate Size

The ore studied requires agglomeration in order to permit reasonable solution exposure during leaching. The amount of moisture added and, consequently, the total volume of agglomeration solution added will influence the agglomerate size distribution (ASD). Agglomerates will become coarser as a larger amount of solution is added during agglomeration. Low acid concentrations used during agglomeration do little to affect the size of the agglomerates; however, at higher acid concentrations, the agglomerates will become less coarse at a fixed moisture content. Longer retention times or higher agglomeration speeds have minimal impact on agglomerate size but may produce slightly finer ASDs. Addition of a polymer during agglomeration will generate slightly less coarse agglomerates.

5.2 Electrical Conductivity

Electrical conductivity of agglomerates was measured throughout the project. Generally, electrical conductivity increased as moisture content increased. Acid concentration was found to increase electrical conductivity but did not affect the normalized electrical conductivity (conductivity ratio) used for comparison in this study. Agglomeration speed and retention time also do not appear to affect electrical conductivity. When polymer is added during agglomeration, the electrical conductivity of an agglomerate bed without a load applied tends to increase. A large amount of variation was observed for the electrical conductivity measurements. As a result, this test needs more study before it can be used in a production setting.

5.3 Bulk Density

The bulk density before the permeability test is a function of agglomerate size. Therefore, any factor which increases agglomerate size will increase the initial bulk density. None of the agglomeration process variables impacted the final bulk density recorded following the permeability test. This indicates the agglomerates are weak and unable to maintain their structure. The addition of a polymer binder significantly increased the bulk density observed following the permeability test.

5.4 Permeability

The permeability of agglomerates increased as moisture content increased. The agglomerates created at low moisture content or high acid concentration had a critically low permeability. Longer retention times increase the permeability of agglomerates. Most importantly, the addition of a polymer binder increased permeability significantly.

5.5 Leaching Behavior

The initial behavior of the agglomerates during leaching is affected by the agglomeration conditions. Agglomerates made with a higher moisture content will experience a faster breakthrough of leaching solution. Lower acid concentrations used during agglomeration lead to a high degree of acid consumption during the first week of leaching. As a result, initial copper recovery will be reduced. Most of the slumping occurred during the first day of leaching. This is another indication that the agglomerates are weak and unable to maintain their structure. At 90 days of leaching, the agglomerates will behave the same regardless of agglomeration conditions.

5.6 Polymer Addition

The polymer tests indicate several polymers are likely suitable for increasing the strength of agglomerates produced with the ore being studied. Two polymers studied further, C-16 and C-17, increased agglomerate stability and greatly improved the permeability of the agglomerate bed.

5.7 Suggested Modifications for Future Work

Results from the permeability test may be more consistent if the agglomerate bed is allowed to stabilize for a period of time. If the permeability is being measured while the agglomerates are rapidly breaking down, the system is not at steady state and the permeability is a function of time. The device could also be modified to utilize a nondestructive fluid such as air to measure permeability.

Utilization of transparent columns during the column leach testing would allow for improved recording of slump height. It may also provide insight into the behavior of the agglomerates during leaching. In addition, if the aeration system is left running at the end of the leaching experiments, the agglomerates may become somewhat dried out, which may allow determination of an ASD following leaching.

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