

# **Methods of Improving Oil Agglomeration**

Sarah Ann Smith

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Roe-Hoan Yoon, Chairman

Gerald H. Luttrell, Co-Chairman

Gregory T. Adel

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## **Abstract**

A simple thermodynamic analysis suggests that oil can spontaneously displace water from coal's surface if the coal particle has a water contact angle greater than  $90^\circ$ . However, the clean coal products obtained from laboratory-scale dewatering-by-displacement (DbD) test work assayed moistures substantially higher than expected. These high moisture contents were attributed to the formation of water-in-oil emulsions stabilized by coal particles. Four different approaches were taken to overcome this problem and obtain low-moisture agglomeration products. These included separating the water droplets by screening, breaking emulsions with ultrasonic energy, breaking agglomerates with ultrasonic energy, and breaking agglomerates using vibrating mesh plates. On the basis of the laboratory test work, a semi-continuous test circuit was built and tested using an ultrasonic vibrator to break the water-in-oil emulsions.

The most promising results were obtained agglomerates were broken using the ultrasonic probe and the vibrating mesh plates. Tests conducted on flotation feed from the Kingston coal preparation plant gave a clean coal product containing 1% by weigh of moisture with a 94% combustible recovery. The separation efficiency of 93% is substantially higher than results achievable using froth flotation. When agglomerates formed from thermal coal from the Bailey coal preparation plant were broken using either ultrasonic energy or vibrating mesh plates, the obtained results were very similar: clean coal products assayed less than 5% moisture with separation efficiencies of 86% in average.

## **Acknowledgements**

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# Chapter 1: General Introduction

## 1.1 Preamble

In 2011, 42% of the electricity in the United States was generated from coal (EIA 2011). Additionally, projections show that 39% of US electricity will still come from coal in 2035 (EIA 2012). Over the next 23 years it is expected that electricity generation will increase by approximately 1 trillion kilowatt hours (EIA 2012). With the majority of electricity coming from coal, it is more important than ever that coal processing becomes as economic and efficient as possible.

In older coal preparation plants, the recovery processes for fine and ultrafine coal were not as efficient as today; therefore, many impoundments contain recoverable coal as large as 600  $\mu\text{m}$  (National Research Council 2002). The larger material (+44  $\mu\text{m}$ ) can be re-mined and recovered using current operations in the plant; however, some ultrafine material is still lost in reprocessing.

While improvements in coal cleaning technology have led to an increased recovery of fine particles within the preparation plant, plant refuse still contains a large number of particles finer than 44  $\mu\text{m}$  (National Research Council 2002). Because the -44  $\mu\text{m}$  size class is so well liberated, its lower ash content makes it an extremely desirable product. These particles have a large surface area, and therefore have a higher moisture content. Thermal dryers afford the opportunity to dewater and dry this ultrafine coal; however, dryers also create an array of problems including high operating cost, potential safety concerns from explosions, and regulatory restrictions. As a result, most ultrafine particles are discarded with the tailings stream and ultimately end up in impoundments. A new and innovative process to recover these ultrafine particles has two main benefits: the ultrafine coal is a sellable product that will pay for the recovery process and recovering the coal decreases the amount of material being sent to the impoundment.

## 1.2 Objectives

The goal of the project is to improve the oil agglomeration process in order to replace current technologies used to clean and dewater fine and ultrafine coal. An energy source is used to disperse coal particles into a phase comprised of hydrophobic liquid, while water and ash remain in a separate phase. The hydrophobic phase will be recovered, and the liquid recycled. The resulting clean coal product should contain less than 10% moisture. All experiments will use Pentane as the hydrophobic liquid since it is affordable and can be easily recycled through evaporation and condensation.

This project focused on conducting laboratory-scale batch tests using four separation methods to determine the best method for scale-up. The separation methods focused on cleaning coal via agglomeration or emulsification. During the experimental processes, the produced agglomerates or emulsions were dispersed into hydrophobic liquid using mechanical vibrations from an ultrasonic probe or vibrating mesh plates. Additionally, a laboratory-scale continuous testing unit was developed and constructed to test the feasibility of the process over a long operating period.

## 1.3 Organization

This thesis is divided into five chapters. The first chapter discusses the need and benefits of the Dewatering by Displacement method and the objectives of the laboratory testing methods employed.

*Chapter 2: Literature Review* is designed to bring readers up to date on the necessary background needed to understand the scope of the project. The review discusses prior research that evolved into the current project, thermal drying, the theory of Pickering emulsions, and the theory and practice of oil agglomeration.

The third chapter reviews the laboratory batch testing methods tried throughout the project. Each subsection of chapter three is dedicated to one of the four methods tried. For each of the testing methods, subsections will be further divided to discuss the experimental apparatus, experimental methods, and results obtained using the method. The fourth chapter is organized similar to the third chapter, but focuses on a continuous laboratory testing unit. Finally, the fifth

chapter summarizes the findings of the project and recommends future modifications and testing of the agglomerate dispersion process.

## Chapter 2: Literature Review

### 2.1 Prior Work

#### 2.1.1 Hydrophobic Displacement

Virginia Tech began developing the dewatering by displacement process in 1995 by researching hydrophobic displacement. Hydrophobic displacement uses a hydrophobic liquid with a higher affinity for the coal's surface to displace water presently on the surface of the coal. Figure 2-1a shows that when dewatering by hydrophobic displacement, a hydrophobic solid particle (in this case coal) of state 1, must leave an aqueous state, 3, and cross an interface into a non-polar hydrophobic (oil) phase, 2 (Yoon). For this displacement to occur, the change in Gibbs free energy,  $G$ , with respect to the particle surface area,  $A$ , must equal the difference in the surface free energy at the coal/oil interface and the surface free energy at the coal/water interface, represented by  $\gamma_{12}$  and  $\gamma_{13}$ , respectively. Additionally, for a reaction to be thermodynamically spontaneous, the change in Gibbs free energy must be less than zero. Therefore:

$$dG/dA = \gamma_{12} - \gamma_{13} < 0 \quad [1]$$

Young's equation can be used to define the relationship between  $\gamma_{12}$ ,  $\gamma_{13}$ , and the surface free energy at the oil/water interface,  $\gamma_{23}$  (Yoon):

$$dG/dA = \gamma_{12} - \gamma_{13} = \gamma_{23} \cos \theta \quad [2]$$

where  $\theta$  represents the contact angle between a droplet of oil on a coal surface and the surface itself, measured through the water phase (Figure 2-1b). Equation 2 can be substituted into Equation 1, so that:

$$dG/dA = \gamma_{23} \cos \theta < 0 \quad [3]$$

Therefore, when  $\theta > 90^\circ$ , the hydrophobic oil phase will displace water on the surface of the coal.

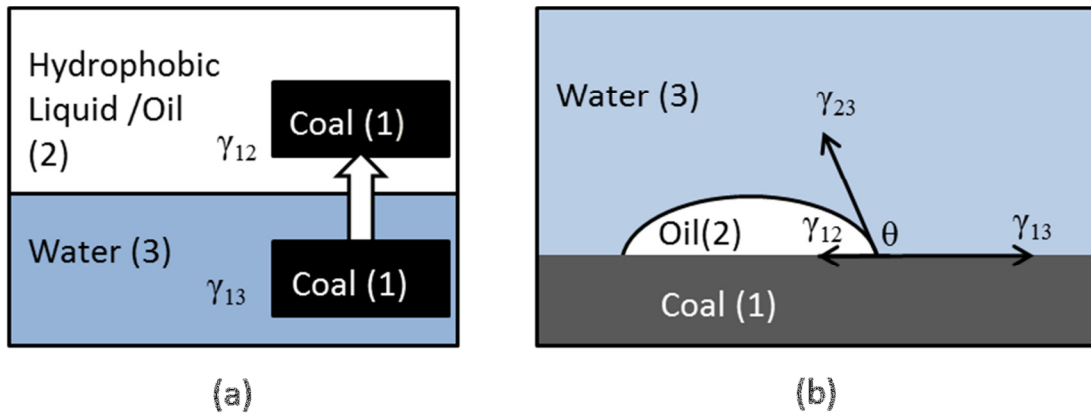


Figure 2-1: a) Schematic showing the displacement of coal, 1, from the water phase, 3, into the oil phase, 2; and b) the three-phase equilibrium between interfacial tensions (Redrawn from Yoon).

### 2.1.2 Low Temperature Drying

In 2010, a low temperature drying process was developed at Virginia Tech to reduce the moisture of displaced coal. The technology was applicable to coal agglomerates and filtered flotation concentrate with less than approximately 22% moisture (Freeland 2010). Three devices were developed to explore the process: a static breaker, air jet conveyor, and centrifugal fan. In each device, the coal agglomerates or cake were subjected to a high, mechanical shearing force. Compared to the other two methods, the centrifugal fan consistently produced a low-moisture product (less than 2%) without plugging.

Low temperature drying requires a high amount of airflow to dry the particles. The relative humidity and temperature of the ambient air have a large impact on the water carrying capacity of the air. It was discovered that the process worked best by heating the air to at least 48.89 °C (120°F) (Freeland 2010). Unfortunately, heating the air adds an additional cost to the process. Based on a mathematical model to calculate the cost of the an industrial scale low temperature dryer, it was discovered that a thermal dryer requires \$0.18/ton less energy than a low heat dryer (Freeland 2010).

## 2.2 Thermal Drying

For fine and ultrafine coals, mechanical dewatering is able to lower the surface moisture to approximately 15 to 25%, respectively (Korte and Mangena 2004). Due to customer specifications and difficulty in handling, these values are unacceptable. Thermal drying is currently the only operation which can give fine and ultrafine coals moistures in the single digit range.

### 2.2.1 Operation

All thermal dryers operate under the same condition: in order for moisture to evaporate from the surface of the coal, the coal must be brought into contact with a heat source. There are many different types of thermal dryers used in the coal industry including: rotary dryers, flash dryers, fluidized beds, and conveyor type dryers.

Rotary thermal dryers are one of the most common dryers used in industry. Of the two types of rotary driers, parallel flow and counterflow, parallel flow rotary driers are more commonly used due to higher fuel efficiency rates and larger capacity. Figure 2-2 shows a basic parallel flow rotary drier. The dryer consists of long, rotating, cylindrical shell. In direct heating dryers, feed and heat enter at the upper end of the dryer and flow downward by gravity due to the slight incline of the dryer drum. In indirect heating dryers, heat circulates around the rotating cylinder drum. At the end of the process, dry product is deposited onto a conveyor belt while the remaining hot air and fugitive dust particles are exhausted into a cyclone (Wills and Napier-Munn 2006).

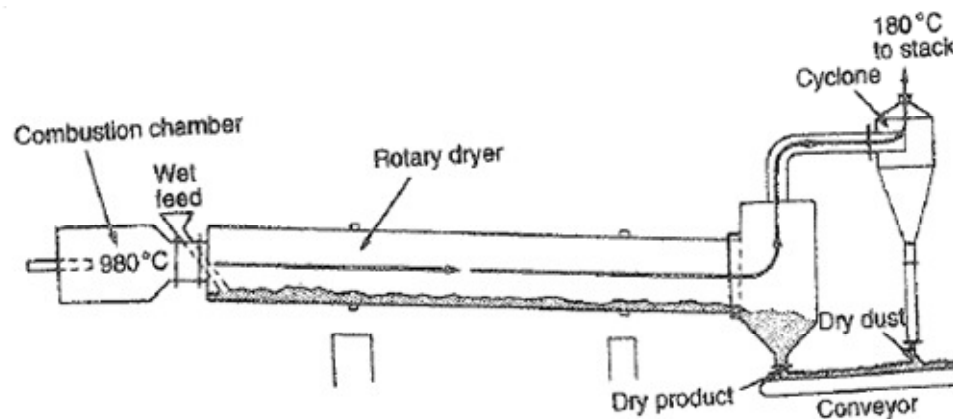


Figure 2-2: Example of a parallel flow rotary dryer (Wills and Napier-Munn 2006).

### **2.2.2 Operation Issues**

When using thermal dryers, it is possible for filter cake to clump within the dryer, creating a risk for a fire or explosion. In order for a fire to occur, fuel, heat, and oxygen must be present. For an explosion, two additional elements are required: dust suspension and confinement (Korte and Mangena 2004). To avoid suspension and clumping, coarser material (6.35 mm x 0.595 mm) is often mixed with fine coal in ratios of 2:1 to 4:1 (Luckie 1991). While this method keeps material from clumping in the dryer, the larger size class can normally be dried using cheaper methods, thus creating inefficiencies in the drying process.

### **2.2.3 Permitting Issues**

Sulfur dioxide (SO<sub>2</sub>), nitrogen oxides (NO<sub>x</sub>), and carbon monoxide (CO) are all gaseous byproducts of operating a thermal dryer. In addition to these gases, several other volatile compounds released by the coal when heating are considered to have carcinogenic effects. For thermal dryers built after 2008, the EPA has implemented very strict emission and monitoring guidelines for these gaseous byproducts. The guidelines are so strict that it is very difficult for a mine operator to obtain a new permit for a thermal dryer and operate below maximum emission levels (2012).

## **2.3 Pickering Emulsions**

In 1907, S.U. Pickering discovered that solid particles could be used to stabilize emulsions (Giermanska-Kahn, Schmitt et al. 2002). Finkle et al. were the first to make a distinction between the two types of emulsions formed (oil-in-water versus water-in-oil). His team discovered that in a particle-stabilized emulsion, one liquid will wet the particles more than the other liquid. The liquid with the poorer wetting properties will become dispersed in the phase that better wets the particles. The following sections provide a summary of the theories behind the formation of Pickering emulsions and factors that affect the determination of the type of emulsions formed.

### **2.3.1 Emulsion Formation**

When a group of particles are placed at an air-water interface, the particles are disordered with no geometric arrangement. However, when an alkane is layered onto the interface, the particles rearrange into an ordered monolayer displaying a hexagonal pattern. When electrolyte concentrations of up to 0.1M NaCl, high enough to cause particle aggregation at an air-water

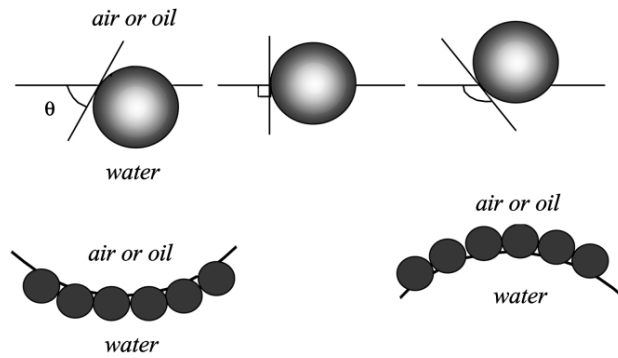


interface, were added to the aqueous phase of the oil-water system, the layer maintained its geometric arrangement (Binks 2002). Therefore, this arrangement is thought to be due to long-range repulsive forces acting over several micrometers through the oil phase (Binks 2002). It is believed that residual surface charges on the particle surface are responsible for the repulsion. The minimum charge required to cause repulsion was calculated to be only 1% of the total possible surface charge.

When the monolayer is compressed, the most hydrophobic and hydrophilic particles are expelled from the layer and report to the oil and water phases, respectively. Particles of intermediate hydrophobicity remain on the interface. Upon compression by additional particles, the monolayer rearranges from a hexagonal array to a rhombohedral array. Further compression of the rhombohedral array will first lead to the layer folding over on itself and upon even further compression the layer will create a wave shape (Binks 2002).

### **2.3.2 Emulsion Type**

There are two possible types of emulsions, oil-in-water (o/w) or water-in-oil (w/o). As shown in Figure 2-3, the contact angle,  $\theta$ , formed between the particle and the oil-water interface determines which type of emulsion is formed. For hydrophilic particles, the contact angle formed is normally less than  $90^\circ$ ; therefore, an oil-in-water emulsion will be formed. A larger portion of the particle surface area will exist in the water phase and particles will encapsulate the oil or air droplet. Similarly, if the particle is hydrophobic, the contact angle formed between the particle and the interface will be greater than  $90^\circ$ . In this case, the majority of the particle surface area will reside in the oil phase and water will be stabilized by particles. Only in cases when  $\theta$  is exactly  $90^\circ$ , will no emulsion form. The particle will have an equal surface area in both the oil and water phases and the net curvature will be zero.



**Figure 2-3: Emulsion type based on contact angle,  $\theta$ . For  $\theta < 90^\circ$ , oil- in-water emulsions form (left). For  $\theta > 90^\circ$ , water-in-oil Emulsions form. Used with permission of Binks. (Binks 2002).**

Once an emulsion is formed, it is possible to cause inversion and change the emulsion type. This can be accomplished by changing the oil:water ratio or by changing the average wettability of particles at the interface. By changing the ratio of oil to water, inversion can occur so that an o/w emulsion will change to a w/o emulsion or vice versa, despite particle hydrophobicity. Figure 2-4 shows how the fraction of water,  $\phi_w$ , present in the system affects the conductivity of the emulsions and the type of emulsions formed. At low water volumes, conductivity of emulsions is low and emulsions disperse in the oil phase (w/o emulsions formed). As the volume of water increases, the type of emulsions formed change-to-oil in water emulsions and conductivity greatly increases. The experiment was conducted using particles with 50% SiOH. Open points on the graph represent cases where the ratio was changed by adding water to oil while filled points represent oil added to water. Based on this study, it was determined that the most stable emulsions were formed at points near inversion (Binks 2002).

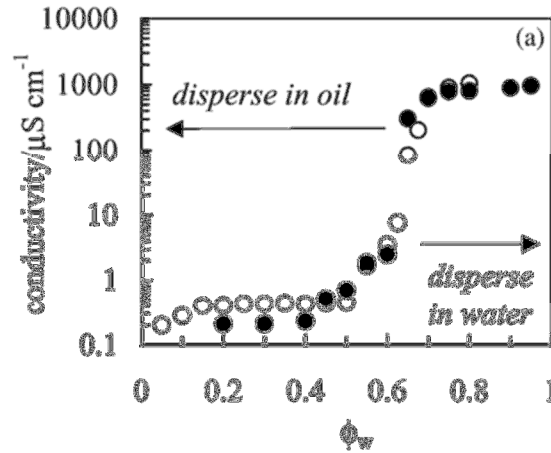


Figure 2-4: The effect of the volume fraction of water present on the type of emulsion formed. Used with permission of Binks.

Emulsion inversion is also possible through the addition of particles with differing hydrophobicity. Using a mixture of particles can affect the inversion by changing the overall average wettability of particles at the interface (Binks 2002). Figure 2-5 shows the inversion the inversion of emulsions from o/w to w/o by the addition of hydrophobic silica. Increasing the amount of hydrophobic silica causes the particles to change from mainly water-wettable to oil-wettable (Binks 2002).

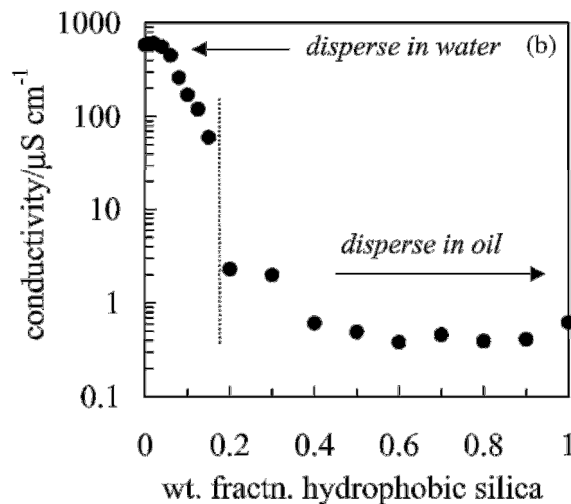


Figure 2-5: The effect of the weight fraction of hydrophobic silica present on the type of emulsion formed. Used with permission of Binks.

### 2.3.3 Energy of Detachment

For particles small enough so that gravity effects are negligible (less than a few microns in diameter), the energy of detachment,  $E$ , to remove the particle from the oil water interface can be calculated using Equation 1 (Binks 2002):

$$E = \pi r^2 \gamma_{ow} (1 \pm \cos \theta_{ow})^2 \quad [4]$$

where  $r$  equals the particle radius and  $\gamma_{ow}$  is the tension at the oil/water interface. For removal into the water phase, a negative sign is used inside the bracket while a positive sign is used for removal into the oil phase. A series of calculations to determine the effect of contact angle on the energy of detachment has been conducted by Binks. It was found that particles are most strongly held at the interface when  $\theta_{ow} = 90^\circ$ . As the contact angle moves away from  $90^\circ$ , the energy necessary to hold particles at the interface greatly decreases. For angles between  $0$  to  $20^\circ$  and  $160$  to  $180^\circ$  the energy of detachment was nearly thirty times less than the energy required at  $90^\circ$ .

As shown in Equation 4, the detachment energy is dependent on the square of the particle radius. Figure 2-6 shows the relationship between particle radius and energy of detachment at an alkane-water interface when  $\theta = 90^\circ$  and  $\gamma_{ow} = 50$  mN/m. The graph shows that low energy is required to detach very small particles (less than  $0.5$  nm), leading to the conclusion that smaller particles create less stable emulsions compared to larger sized particles.

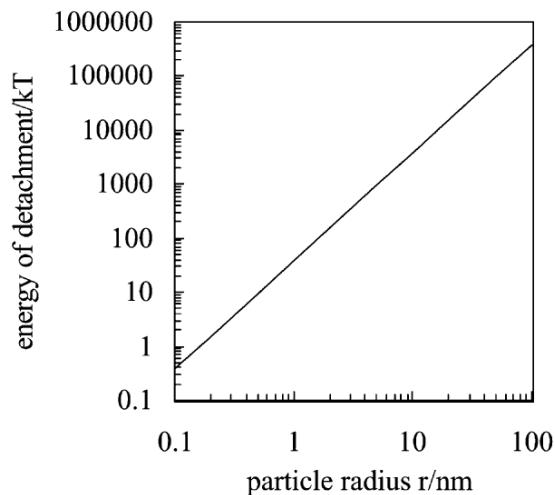


Figure 2-6: Required energy of detachment as a function of particle radius. Used with permission of Binks.

## **2.4 Oil Agglomeration**

An increase in low-seam mechanized mining methods has resulted in an increased production of coal fines mixed with high ash tailings (Hazra, Rao et al. 1988). Due to the small size of the particles ( $-44\ \mu\text{m}$ ), froth flotation is not always a suitable cleaning method. Compared to oil agglomeration with an oil recovery system, research has shown that using froth flotation to decrease the ash in ultrafine coal decreases yield and increases processing cost (Hazra, Rao et al. 1988). The main factors affecting the cost of the agglomeration process are the cost of the coal, the cost of the oil, and the selling price of the final product (Hazra, Rao et al. 1988). Oil agglomeration is most economical when performed on refuse material leaving as effluent since the material is essentially “free” and would otherwise be sent to impoundments (Nicol 1979).

### **2.4.1 History**

Oil agglomeration for coal cleaning first began in the early 1920's as the Trent Process. This first process used a 40% solid,  $-100$  mesh ( $150\ \mu\text{m}$ ) slurry mixed with 30% fuel oil (by weight of dry coal). Mixing time was in the range of 6-24 minutes and oil loss was around 10 to 50%. The costs of creating the agglomerates were so high that the agglomerates could not be sold to the steam market and could only be sold as home “Superfuel” after briquetting. Therefore, the process was abandoned after only a few years of operation (Mehrotra, Sastry et al. 1983).

Oil agglomeration was heavily researched and developed during the 1970's due to a sharp increase in oil prices. Though all oil agglomeration processes operate under the same theory, a large number of oil agglomeration patents were filed during the 70's and 80's due to significant process variations. During this time period, the oil agglomeration process was improved, oil dosages, and mixing times were decreased while combustible recovery remained in the upper 90<sup>th</sup> percentile. Several pilot plants were constructed to test the feasibility of the process as a method of cleaning fine coal. As oil prices began to decrease in the late 1980's the process once again became uneconomical as an oil substitute was no longer needed (Mehrotra, Sastry et al. 1983).

### **2.4.2 Factors affecting agglomeration**

Oil agglomeration depends on the surface hydrophobicity of a coal particle and its ability to be preferentially wetted by hydrophobic oil (Mehrotra, Sastry et al. 1983). The interaction that

occurs between the hydrophobic particles and hydrophobic liquid is controlled by three factors: the free energy at the three phase interface (the interface between water, coal, and the hydrophobic liquid), the amount of hydrophobic liquid used, and the mixing intensity (Capes and Darcovich 1984). These interactions allow the oil to wet the coal and suspend it in a hydrophobic phase while the ash and oxide matter remain in the aqueous phase. With sufficient oil and mechanical agitation, coal particles collide and an oil bridge is formed between particles. The interfacial tension of the oil and capillary attraction of the bridges helps keep the agglomerates stable so that they may continue to grow and attract new particles (Mehrotra, Sastry et al. 1983).

A wealth of research has been conducted on the type of oil used for agglomeration; however, results vary over researchers. Oils are divided into light and heavy classes depending on viscosity. One set of research shows that oils with specific gravities between 0.7 and 0.85 are most effective in agglomeration coals while oils with gravities below 0.64 or above 0.97 are ineffective. The same research states that heavy oils are too viscous to disperse in the slurry while light weight oils were unable to make the coal hydrophobic enough; however, this research was later disputed when it was discovered that heavy oils could produce agglomerates and high recoveries if long mixing times were used. Multiple researchers have confirmed that heavy oils result in a higher combustible recovery, but also give a poor ash and sulfur rejection. In the 1970's, it was discovered that oils with medium surface tension and medium viscosity were most effective in agglomerating and that oils with lower or high viscosities and surface tensions were ineffective at producing agglomerates. Due to such variations in research, an appropriate oil can only be selected after the properties of the coal surface are known and through experimentation to understand how the oil will interact with the surface (Mehrotra, Sastry et al. 1983).

### ***2.4.3 Dewatering Agglomerates***

The moisture content within the agglomerates comes from two sources: internally trapped moisture and surface moisture. The internal moisture which heavily depends on the mixing method, intensity, and oil dosage, commonly makes up 5 to 10% of the total agglomerate weight. Therefore, most of the agglomerate moisture is on the surface of the agglomerate. For agglomerates larger than about 2 mm, gravity drainage on a dewatering screen can reduce the total agglomerate moisture to 10% by weight or less. Since most moisture comes from the

surface of the agglomerates, it is desirable to produce larger diameter agglomerates. For agglomerates less than 1 mm in diameter, surface moisture is best removed using a centrifuge (Capes and Darcovich 1984).

### **Chapter 3: Batch Testing Methods**

Batch tests were conducted using four different methods to clean and dry coal: breaking agglomerates via screening, breaking emulsions with ultrasonic energy, breaking agglomerates with ultrasonic energy, and breaking agglomerates using vibrating mesh plates. This chapter is divided into four subsections, one for each method. The subsection for each experimental process is further divided to include a description of the apparatus used for the process, the methods followed, the results obtained, and a discussion of the results.

For each testing method, n-Pentane, produced by Alfa Aesar, was used to produce either agglomerates or emulsions. Pentane is a colorless, immiscible liquid with a density of 0.631 g/cm<sup>3</sup>. The liquid and vapors are highly flammable. Pentane has a boiling point of 36°C. When pentane enters the air and concentrations of 1.8% to 8.0% by volume are reached, an explosive environment is created. The pentane used in the following experiments was HPLC grade and was composed of a minimum of 99% pentane by volume (Alfa Aesar 2009). Additional supplies and apparatuses used for each individual method will be discussed in the method's individual section.



### **3.1 Breaking Agglomerates via Screening**

#### ***3.1.1 Experimental Apparatus: Breaking agglomerates via screening***

To form the agglomerates for the process, a Black and Decker kitchen blender was used. The blade of the blender “cut” the agglomerates and was therefore replaced with a custom-made flat paddle. A variable speed control was used with the blender so that both high and low shear mixing environments could be created. To dewater and break agglomerates, 8 inch laboratory sieves of varying apertures were used.

#### ***3.1.2 Experimental Methods: Breaking agglomerates via screening***

To form the agglomerates, a volume of coal slurry was poured into the blender and mixed on a high speed setting for 30 to 50 seconds after the pentane was added. The volume of pentane added varied based on the requirements of each sample to form the agglomerates. Immediately following the addition of the pentane, an obvious phase separation could be observed. Heavier, ash-containing water remained in the lower portion of the blender while black, while less-dense coal agglomerates rested on the top of the blender.

Larger agglomerates were desired to enhance the dewatering stage; therefore, the blender speed was reduced using a variable speed controller to create a low-shear mixing environment. The agglomerates were then mixed in the low shear environment for an additional 4.5 minutes or until the total mixing time was five minutes. The long mixing time allowed large (approximately 0.5 cm diameter) agglomerates to form.

The agglomerates and ash-containing water were poured across a large-mesh screen (ranging between ten and thirty mesh depending on agglomerate size) to dewater the agglomerates. The dewatered agglomerates were then transferred to a larger mesh (smaller opening) screen. Next, the screen was shaken by hand so that individual dry coal particles or small groups of particles sheared off the agglomerates and fell through the screen and wet coal remained on the screen. Shaking times were varied based on the amount of coal that fell through the screen. Screens were shaken until the majority of original material fell through.

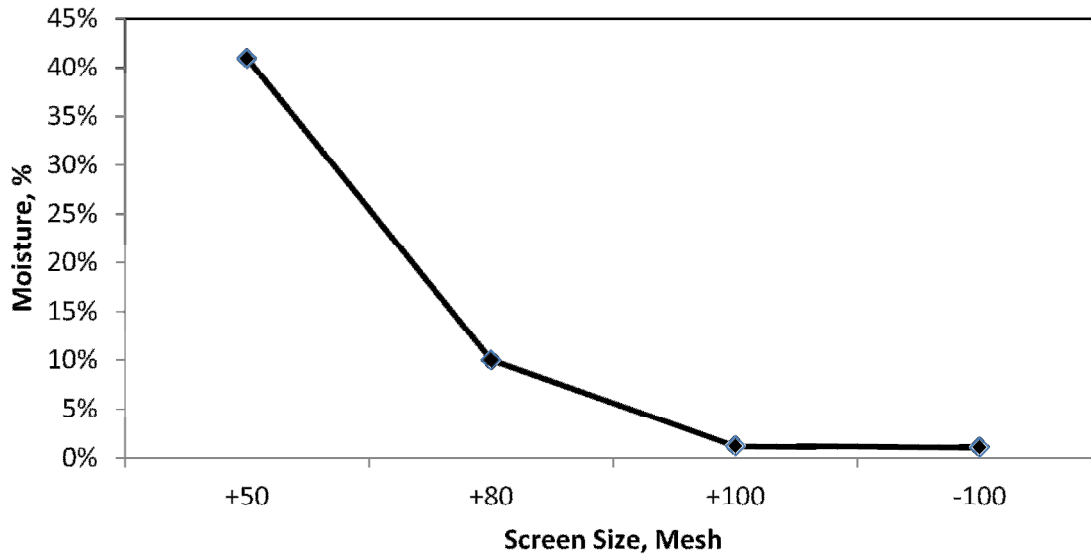
To further reduce the product moisture, the dry coal particles were placed on another, smaller-opening screen and shaken. This step was repeated for one to two additional stages using increasingly smaller screens than the previous stage. Depending on the moisture of the oversized material, the oversized material was either added into the final product or recycled and used to form new agglomerates.

### **3.1.3 Results: Breaking agglomerates via screening**

Screening was originally tried on sample from the Blue Creek preparation facility. Size analysis done on the sample showed that 71% of the sample was smaller than 500 Mesh. Agglomerates were made via handshaking in a separatory funnel using a 6% solids (by weight) slurry containing 39.1% ash. The agglomerates were dewatered by opening the valve at the bottom of the funnel to remove ash. After dewatering, the ash of the product was reduced to 4.65%. The agglomerates were shaken by hand on 50, 80 and 100 mesh screens until sample would no longer pass through the screen. By combining the -50 + 80, -80 + 100, and -100 x 0 size classes, the overall moisture was reduced to 2.10 % and cumulative ash was reduced to 3.88%. Table 3-1 shows the results obtained by screening the Blue Creek agglomerates. With each new screen, the moisture of the particles passing continued to decrease. Figure 3-1 shows the steady decrease as screening progressed.

**Table 3-1: Results of agglomerating and screening Blue Creek Coal.**

<b>Screen Size, Mesh (micron)</b>	<b>Dry Weight, g</b>	<b>Moisture, %</b>	<b>Ash, %</b>	<b>Yield, %</b>	<b>Recovery, %</b>
+50	6.06	40.95%	4.65	-	-
-50 + 80	0.20	10.00%	-	50.36%	79.06%
-80 + 100	1.16	1.19%	3.85		
-100 x 0	0.73	1.08%	3.96		
Tails	8.40	-	74.31	-	-
Feed	19.27	-	39.1	-	-

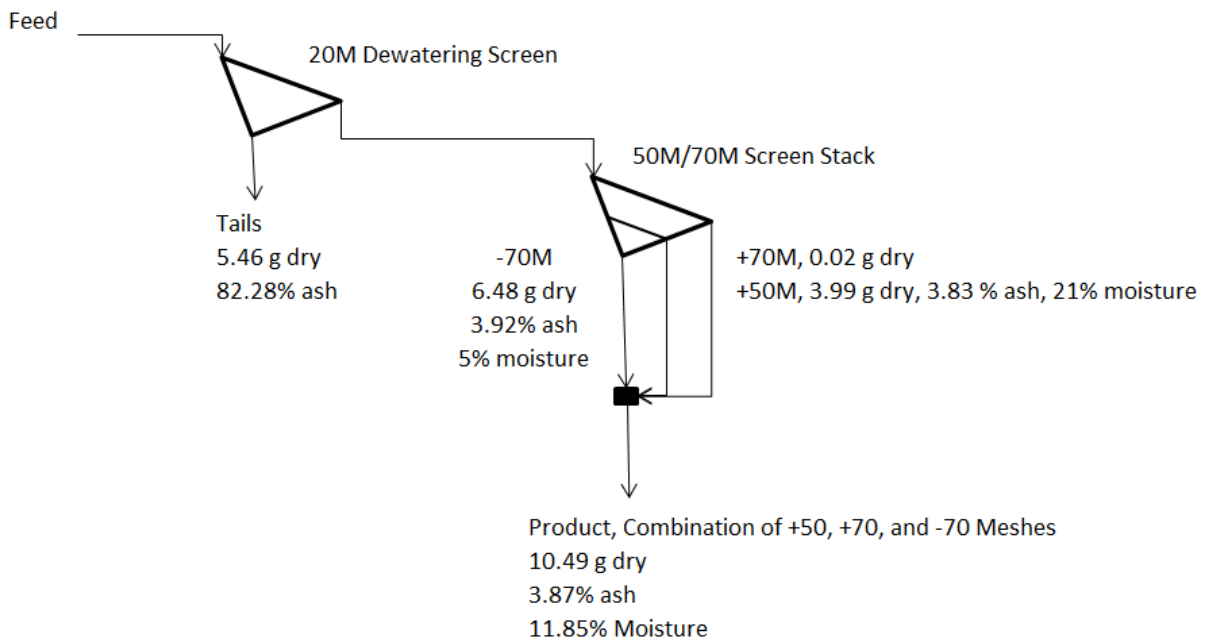


**Figure 3-1: The effect of screen size on moisture.**

In an attempt to lower the moisture and increase the recovery, different screen configurations were tried. In the next experiment, agglomerates were created in the blender using flotation feed from CONSOL's Bailey preparation plant. The agglomerates were dewatered on a 20 mesh screen, and then poured on a set of 50 and 70 mesh stacked screens. The screens were shaken until no more material fell through the openings (due to screen blinding). Table 3-2 shows the results obtained from the experiments while Figure 3-2 shows a flow sheet of the process followed. Approximately 22% (by weight) of the original feed remained on the 50 mesh screen and had a moisture content of 21%. The material which passed through the screen fell onto a 70 mesh screen. The 70 mesh screen was hand shaken until no material would pass the screen. The majority of the material passed through the screen very quickly. After shaking, nearly all of the material passed through. The material remaining on the screen had a very high moisture content of 33%. The majority of the original feed (35.24% by weight) was able to pass the screen and report to the -70 x 0 mesh size class. This material had the lowest moisture, 4.85%.

**Table 3-2: Screening results for each individual size class when screening agglomerated Bailey coal.**

Sample	Sample Dry Weight, g	Weight Percent, %	Moisture, %	Ash, %
Screen Losses	2.44	13.27%	-	-
+50 M	3.99	21.70%	21.15%	3.83
-50 + 70 M	0.02	0.11%	33.33%	-
-70 x 0 M	6.48	35.24%	4.85%	3.92
Tails	5.46	29.69%	-	82.28
Feed	18.39	-	-	29.74



**Figure 3-2: Flow sheets and results of screening process using Bailey sample with a dewatering screen and stacked 50 and 70 Mesh screens.**

By combining the +50 mesh, -50 + 70 mesh, and -70 x 0 mesh materials, 78% of the combustible material in the feed was recovered in the product; however, nearly 7.5% of the of the combustible material in the product reported to the tailings. The final product had a moisture content of 11.85% and an ash content of 3.88%. Table 3-3 shows a summary of the specifications of the final product.

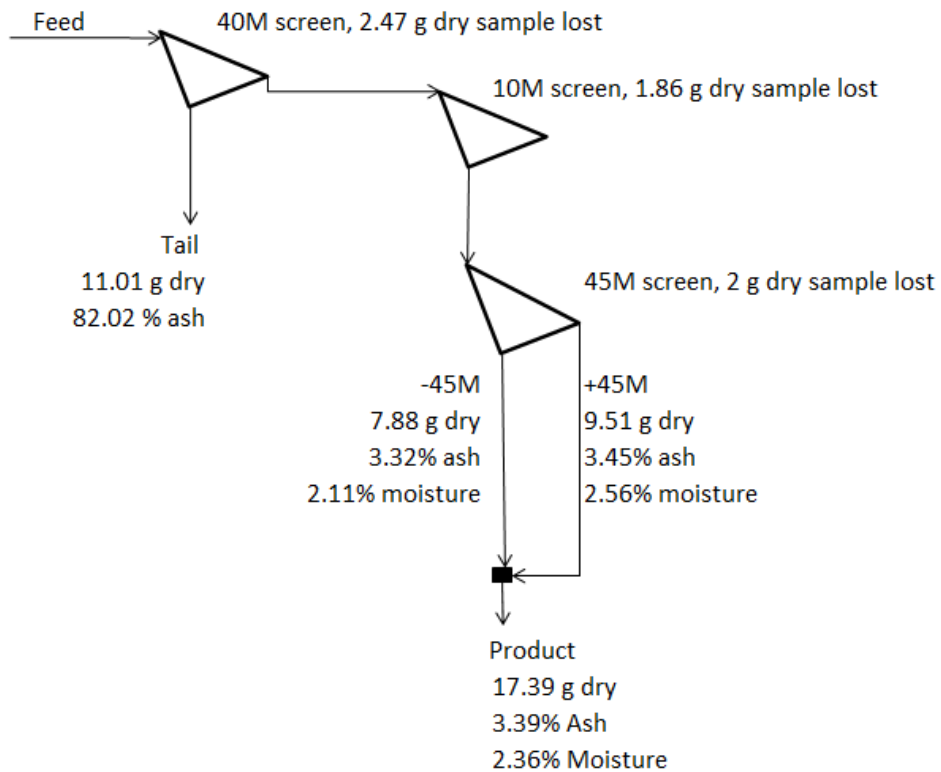
**Table 3-3: Cumulative product ash, moisture, and combustible recovery for screening process using Bailey coal.**

	<b>Sample</b>	<b>Dry Weight, g</b>	<b>Weight Percent, %</b>	<b>Ash, %</b>	<b>Cumulative Moisture, %</b>	<b>Combustible Recovery, %</b>
Lost	Screen Losses	2.44	13.27%			14.56%
Product	+50 M	10.49	57.04%	3.88%	11.85%	78.04%
	-50 + 70 M					
	-70 x 0 M					
Tailing	Tails	5.46	29.69%	82.28%		7.40%
Feed	Feed	18.39	100.00%	29.74%		

A second experiment was tried using flotation feed CONSOL’s Bailey preparation plant. Larger screen sizes were used to see if combustible recovery could be increased. The agglomerates for this experiment were much larger in diameter than in the previous experiment. The agglomerates were drained on a 40 mesh screen and then transferred to a 10 mesh screen. The agglomerates were shaken until all coal particles had passed through the screen and no material remained on top of the screen. Next, the coal particles were placed on a 45 mesh screen for shaking. After just over half of the material had passed through the screen, shaking was stopped because the +45 mesh material appeared dry. Moisture and ash analysis was conducted on the 45 mesh overflow and underflow, as well as the material which remained plugged in the screen openings. Analysis results are shown in Table 3-4. Figure 3-3 shows the generalized flow sheet for the process with the results for each size class. Please note in Table 3-4 that the samples containing “screen” in the name are the dry material which was left in the screen openings due to blinding. The screens were dried weighed wet and then placed in the oven so that the moisture of the product left on the screen could be calculated. After the screens were dry, each screen was tapped over a sample pan to dislodge the blinded material.

**Table 3-4: Screening results for individual size classes using larger mesh screens with Bailey sample.**

Sample	Dry Weight, g	Weight Percent, %	Moisture, %	Ash, %
40M Screen	2.47	7.11%	57.71%	3.31
10M Screen	1.86	5.36%	38.41%	4.02
45M Screen	2.00	5.76%	6.98%	3.39
+45M Material	9.51	27.38%	2.56%	3.45
-45M Material	7.88	22.69%	2.11%	3.32
Tails	11.01	31.70%	-	82.02
Feed	34.73	-	-	29.87



**Figure 3-3: Flow sheet variation for dewatering process using Bailey's screen bowl effluent.**

After screening, the +45 mesh material contained 2.56% moisture while the -45 mesh coal had a moisture value of 2.36%. These two materials were combined to make the final product for the process. Table 3-5 shows the cumulative values of the product, feed, tailings, and material lost due to screen blinding. The final product had cumulative moisture of 2.36%, but had a very low combustible recovery of 69%. Just over 8% of the combustible material reported

**Table 3-5: Screening results using Bailey sample after combining material to create final product.**

	<b>Sample</b>	<b>Dry Weight, g</b>	<b>Weight Percent, %</b>	<b>Average Ash, %</b>	<b>Cumulative Moisture, %</b>	<b>Combustible Recovery, %</b>
Lost	Screen Losses	6.33	18.23%	-	-	22.77%
Product	-10 + 45 M	17.39	50.07%	3.39%	2.36%	69.13%
	-45 x 0 M					
Tailing	Tails	11.01	31.70%	82.02%	-	8.10%
Feed	Feed	34.73	100.00%	29.87%	-	-

**Table 3-6: Screening results using Bailey sample with screen losses neglected.**

	<b>Sample</b>	<b>Dry Weight, g</b>	<b>Weight Percent, %</b>	<b>Ash, %</b>	<b>Cumulative Moisture, %</b>	<b>Combustible Recovery, %</b>
Product	-10 + 45 M	17.39	61.23%	3.39%	2.39%	84.35%
	-45 x 0 M					
Tailing	Tails	11.01	38.77%	82.02%	-	9.94%
Feed	Feed	28.4	100.00%	29.87%	-	-

to the tails. Nearly a quarter of the combustible material was lost due to screen blinding. As shaking progressed, the screen openings plugged, leaving behind 18 % of the total material fed into the system and 23% of the total combustible material in the system.

If screen losses are neglected (assuming a screen cleaning method would be available in a continuous process), the combustible recovery increases by 15 percentage points to nearly 85%. The percentage of combustible material in the tailings increases to 10%. Table 3-6 shows the results of the experiment if the screen losses are neglected.

In the next experiment, Agglomerates were created with screen bowl effluent from the Bailey preparation plant and screened using five different sieve sizes: 20 mesh for dewatering, and 10, 45, 60, and 80 meshes for drying. More screen sizes were used than in previous experiments to test the theory that more screening stages would decrease the product moisture. A flow sheet of the procedure is shown in Figure 3-4. The smaller, 20 mesh sieve was used for dewatering to ensure that no whole agglomerates passed through to the tailings. A large 10 mesh screen was used as the first stage of drying. After shaking, no material was left on top of the

screen; however, 5.5% of the initial feed weight remained in between the mesh openings. The underflow of the 10 mesh screen was poured onto a 45 mesh screen. All screens in the process were shaken until material stopped passing through the openings. The 45 mesh screen became completely plugged, preventing material from passing. Nearly 5.5 grams, or 24% of the feed by weight, remained on the screen. The -45 mesh coal was transferred onto a 60 mesh screen and then an 80 mesh screen. All of the material passed through both screens at a very fast rate. The -10 + 45, -45 + 60, -60 + 80, -80 x 0 mesh materials were all combined to create the final product. Table 3-7 shows the individual results for each step in the process. The first 5 rows, which include the word “screen” in the sample name, represent the coal that was left on the screen due to blinding.

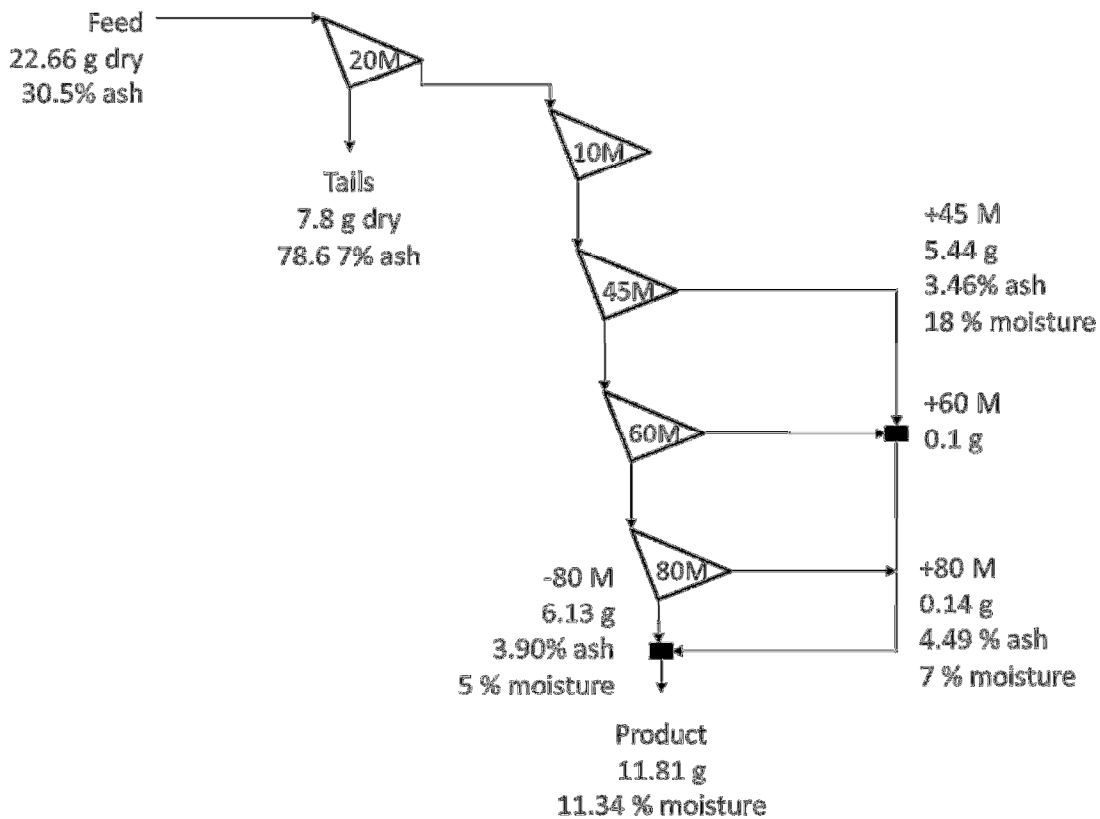


Figure 3-4: Experimental flowsheet for multistage screening processes.



**Table 3-7: Individual sizeclass results for multistage cleaning process.**

<b>Sample</b>	<b>Dry Weight, g</b>	<b>Weight Percent, %</b>	<b>Moisture, %</b>	<b>Ash, %</b>
20M screen	1.12	4.94%	-	3.34
10M screen	1.24	5.47%	38.00%	3.58
45M screen	0.67	2.96%	22.99%	-
60M screen	0.00	0.00%	-	-
80M screen	0.02	0.09%	0.00%	-
-10 + 45M material	5.44	24.01%	17.58%	3.46
-45 + 60M material	0.1	0.44%	0.00%	8.14
-60 + 80M material	0.14	0.62%	6.67%	4.49
-80 x 0 M material	6.13	27.05%	5.26%	3.9
Tails	7.8	34.42%	-	78.67
Feed	22.66	100.00%	-	30.50

Based on the screen data, most of the moisture in the sample was lost on the 10 and 45 mesh screens. The material left in between the openings of the 10 mesh screen had a moisture content of 38% while the moisture content of the blinded 45 mesh material was 23%. The -10 + 45 mesh material that fell in between these two screens had a moisture content of 17.5%. The 60 and 80 mesh screens had no material left between the openings and the material that passed the screens had low moisture contents of 6.6% and 5.3% for the -60 and -80 mesh size classes, respectively.

Though the -10 + 45 mesh size class had high moisture content, the large amount of dry material that fell into the -80 x 0 mesh class was able to “balance” out the moisture contents so that both materials could be used in the final product. The -45 + 60 and -60 + 80 mesh materials were also combined into the final product. Cumulatively, the final product had a moisture value of 11.3%. Table 3-8 shows the specifications of the product, screen losses, tailings, and feed. Seventeen percent of the combustible material was lost on the screens, with the majority being lost on the 20 and 10 mesh screens (Table 3-7). Ten percent of the combustible material passed through the dewatering screen, into the tailings. Of the combustible material fed into the process, 72% reported to the final product. Using a recovery of 72%, the separation efficiency for the process is 62.52%. If screen losses are neglected (assuming screens will be cleaned and the material will be recovered), the combustible recovery increases to 87.23% and the separation

**Table 3-8: Multistage screening results for cumulative product and tailings.**

	<b>Sample</b>	<b>Dry Weight, g</b>	<b>Weight Percent, %</b>	<b>Average Ash, %</b>	<b>Cumulative Moisture, %</b>	<b>Combustible Recovery, %</b>
Lost	Screen Losses	3.05	13.46%	-	-	17.24%
Product	-10 + 45M	11.81	52.12%	3.74%	11.34%	72.16%
	-45 +60 M					
	-60 + 80 M					
	-80 x 0 M					
Tailing	Tails	7.8	34.42%	78.67%	-	10.60%
Feed	Feed	22.66	100.00%	30.50%	-	-

efficiency rises to 77.59%. Neglecting screen losses will also raise the combustible recovery of the tailings to 12.77%. However, during the dewatering stage, any coal which falls through the dewatering screen forms a thin layer on the surface of the tailings. This material could be skimmed off and recycled into increase combustible recovery in the product and decrease combustible recovery in the tailings.

Using multiple stages of screening seemed to have no effect on the product moisture. Therefore, an experiment utilizing only two shaking stages was tried to see how the moisture would be affected. A 30 mesh screen was used as the dewatering screen. In all previous experiments, the dewatered agglomerates were removed from the screen so that the majority of agglomerates were sent to the subsequent shaking stages. Most of the agglomerates directly against the screen mesh have higher surface moisture content compared to agglomerates near the top of the material on the screen. As the water from the upper levels of agglomerates drain through the screen, some water collects along the bottom of the screen and between the openings, wetting the agglomerates. These wetter agglomerates tend to “stick” to the screen due to the surface tension between the water on the coal surface and the water on the screen.

In this experiment, the agglomerates created with Bailey flotation feed were dewatered, and then the dewatering screen was tilted 90°. All of the agglomerates that fell from the screen were placed onto the 50 mesh screen. Agglomerates that were stuck to the screen with water were not manually removed from the screen, instead, they were assumed to be recycled back to the agglomeration stage in a continuous circuit because of their higher moisture content. The

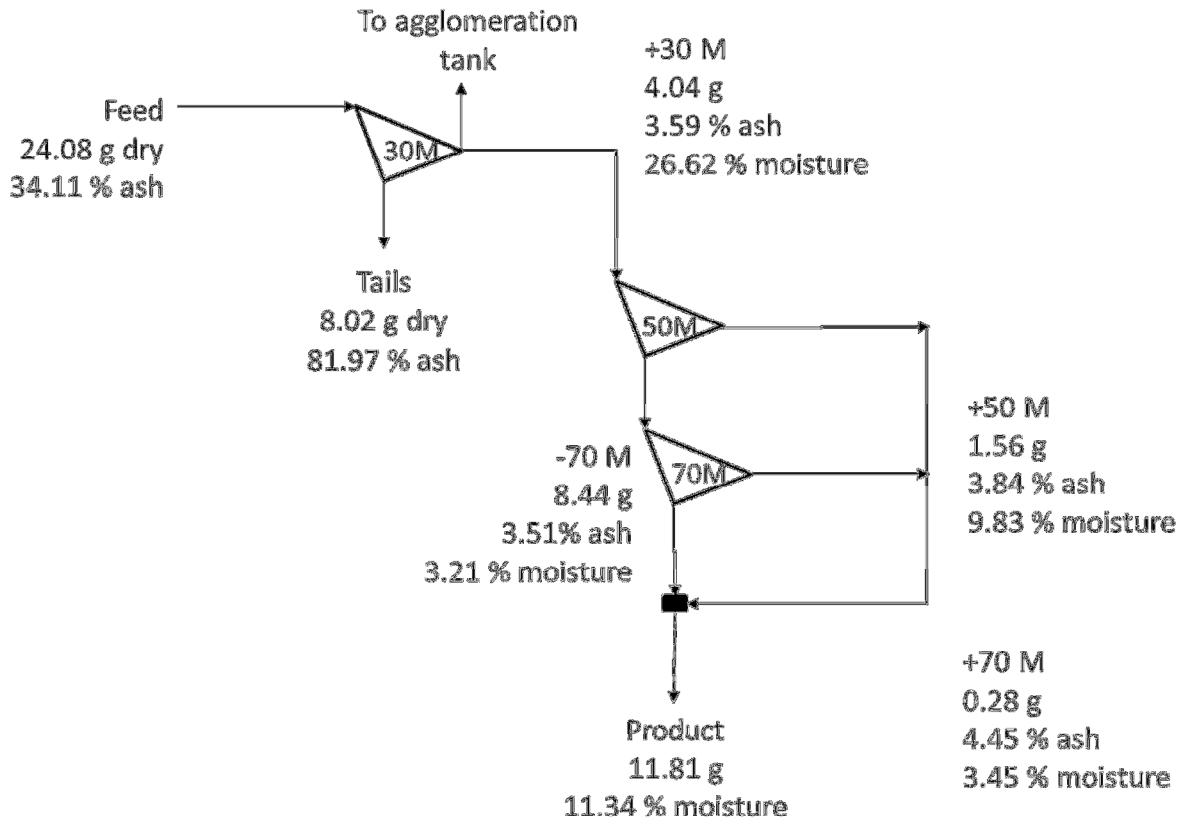


Figure 3-5: Process flow sheet for screening process with recycle stream used to dewater Bailey flotation feed.

agglomerates which went onto the 50 mesh screen were shaken until the screen blinded. The material passing the screen fell onto a 70 mesh screen. A flow sheet for the process followed is shown in Figure 3-5.

Table 3-9 shows the moisture, ash, and weight percent for each size class. The agglomerates which did not freely fall from the angled 30 mesh dewatering screen were manually removed for moisture and ash analysis. The +30 mesh agglomerates that were not removed from the screen had a moisture content of 28.62% and comprised nearly 17% of the original feed. The +50 mesh sample had a moisture content of 9.83% and was combined with the -50 + 70 and -70 x 0 mesh samples which had moisture contents of 3.45% and 3.21%, respectively, to create the final product. The specifications of the combined product, tailings, feed, and internal recycle streams are shown in Table 3-10 .

**Table 3-9: Individual size class results for screening process with recycle stream used to dewater Bailey flotation feed.**

Sample	Dry Weight, g	Weight Percent, %	Moisture, %	Ash, %
30M Screen	1.39	5.77%	61.50%	-
50M Screen	0.24	1.00%	40.00%	-
70M Screen	0.11	0.46%	60.71%	-
+30M Sample	4.04	16.78%	28.62%	3.59
+50 M Sample	1.56	6.48%	9.83%	3.84
-50 + 70M Sample	0.28	1.16%	3.45%	4.45
-70x 0M Sample	8.44	35.05%	3.21%	3.51
Tails	8.02	33.31%	-	81.97
feed	24.08	-	-	34.11

The combined product stream had a very low moisture content of 4.28%. The total combustible recovery of the product was 62.49%. Nine percent of the combustible material was lost to the tailings. The + 30 mesh agglomerates which stuck to the dewatering screen and were recycled accounted for nearly 25% of the combustible material that entered the system. If considering only the feed, product and tailings streams (the material from screen losses and the internal recycle stream are assumed to be recovered in a continuous process), the combustible recovery for the product stream increases to 87.27% and the tailings stream ash increases to 12.72%.

**Table 3-10: Cummulative results for screening process with recycle stream used to dewater Bailey flotation feed.**

	Sample	Dry Weight, g	Weight Percent, %	Average Ash, %	Cumulative Moisture, %	Combustible Recovery, %
Lost	Screen Losses	1.74	7.23%	-	-	10.96%
Recycle	+ 30 M	4.04	16.78%	3.59%	28.62%	24.57%
Product	-30 + 50M	10.28	42.69%	3.39%	4.28%	62.49%
	-50 + 70 M					
	-70 x 0 M					
Tailing	Tails	8.02	33.31%	81.97%	-	9.11%
Feed	Feed	24.08	100.00%	34.11%	-	-

### **3.1.4 Discussion: Breaking agglomerates via screening**

The screening method for breaking agglomerates can give a low moisture product; however, the process is very sensitive and has many drawbacks. Separation efficiencies were calculated for each of the screen configurations tested. The multiple stages of screening and the coal losses due to blinding make the process very inefficient. The average separation efficiency for all of the process was 66.3% with a standard deviation of 3.7%. If the screen losses are neglected, assuming the coal can be recovered, the average separation efficiency increases to 78.3% with a standard deviation of 1.9%; however, the process is still less efficient than other cleaning methods tested.

As mentioned, One of the largest problems with the process is screen blinding. As agglomerates are shaken on the screen, particles begin to shear off the surface of the agglomerate, exposing internal droplets of water. Some of the extremely small droplets evaporate into the air. When using traditional stainless steel screen mesh, the water droplets too large to evaporate will stretch across the screen opening and collect some loose particles until the screen is completely blinded. If a large amount of water was present in the agglomerates, the water in the screen openings would eventually become heavy enough to overcome the tension between the opening and fall into the product below. The trapping of moisture in the screen mesh is shown in Table 3-4. The material left in between the openings of the 45 mesh screen had 7% moisture. The +45 mesh and -45 mesh materials had moistures of just over 2%.

A Teflon coated mesh was tried to prevent the water droplets from stretching across the screen openings. The screen worked very well when shaking was first started. As shaking continued and water droplets became exposed, the droplets started to roll around on top of the screen with the agglomerates. Initially, the droplets would coalesce and look like a coal coated-water drop. As shaking continued, the droplet picked up more coal particles and become thicker in consistency. The water continued to pick up coal particles until everything on the screen was coalesced into a sticky mass. In industry, this wet mass could be recycled and sent back to the agglomeration tank. These blinding issues mean the process could never be truly continuous using traditional sieves. Screens would have to go through cycles of shaking/breaking agglomerates then be cleaned to remove dry material.

Another problem with this method is the loss of combustible material to the tailings. As much as 12% of the combustible material in the feed was lost to tailings. The tailings stream could be recycled to the agglomeration stage for a chance to recover the lost material. Proper and consistent agglomerates must be formed for the process to work properly. As discussed in the literature review, larger diameter agglomerates have lower moisture content than smaller agglomerates as the majority of agglomerate moisture occurs on the surface and larger agglomerates have a smaller surface area compared to smaller agglomerates. If the agglomerates are too small, excess material passes through the dewatering screen and is lost to the tailings.

## **3.2 Breaking emulsions with ultrasonic energy**

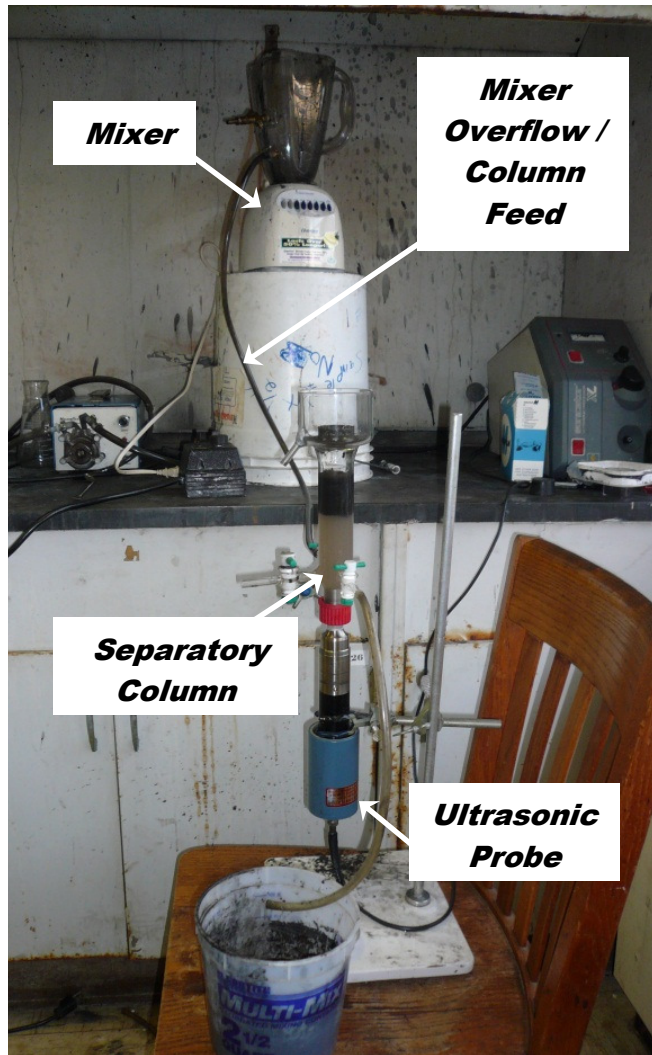
### **3.2.1 *Experimental Apparatus: Breaking emulsions with ultrasonic energy***

During separation, the plugging of feed lines by emulsions was a common problem; therefore, many different experimental setups were explored using the concept of breaking emulsions with ultrasonic energy. In all setups, a Qsonica Q700 ultrasonic probe operating at 20kHz was inserted into a separation column and used to break the emulsions. Different methods of creating and feeding the emulsions into the separation column were tried in an effort to prevent lines from plugging. The following sections will discuss each experimental setup in further detail.

#### **3.2.1.1 Experimental Apparatus: Emulsion formation with a traditional mixer**

In the initial experimental setup shown in Figure 3-6, two Masterflex peristaltic pumps were used to pump feed slurry and pentane into an Oster kitchen blender. A variable speed controller was used with the blender to control the rotational speed of the blender's mixing paddle. Two ports were located on the side of the blender pitcher. A lower port was used for incoming feed while the upper port was used as an overflow to the separation column.

The ultrasonic probe was mounted on the bottom of a custom-made, 1.5 inch diameter, 9 inch long, glass separatory column. Two ports were located at the bottom of the column directly above the tip of the probe, one for feed, and one for tailings. Originally, three ports were at the bottom of the column. Due to damage, one port was sealed off. Note that in Figure 3-6, the original third port is being used for feed and is located on the back side of the column.



**Figure 3-6: Experimental setup consisting of a kitchen blender for emulsion formation and gravity-fed separation column.**

Over time, the emulsions began to build up in the tubing between the blender and separation column. Eventually, the tubing would plug with material, creating a build-up in the blender. In order to remedy the problem, a peristaltic pump was installed between the blender and separation column to pump the emulsions from the blender to the column. The modified experimental setup is shown in Figure 3-7.



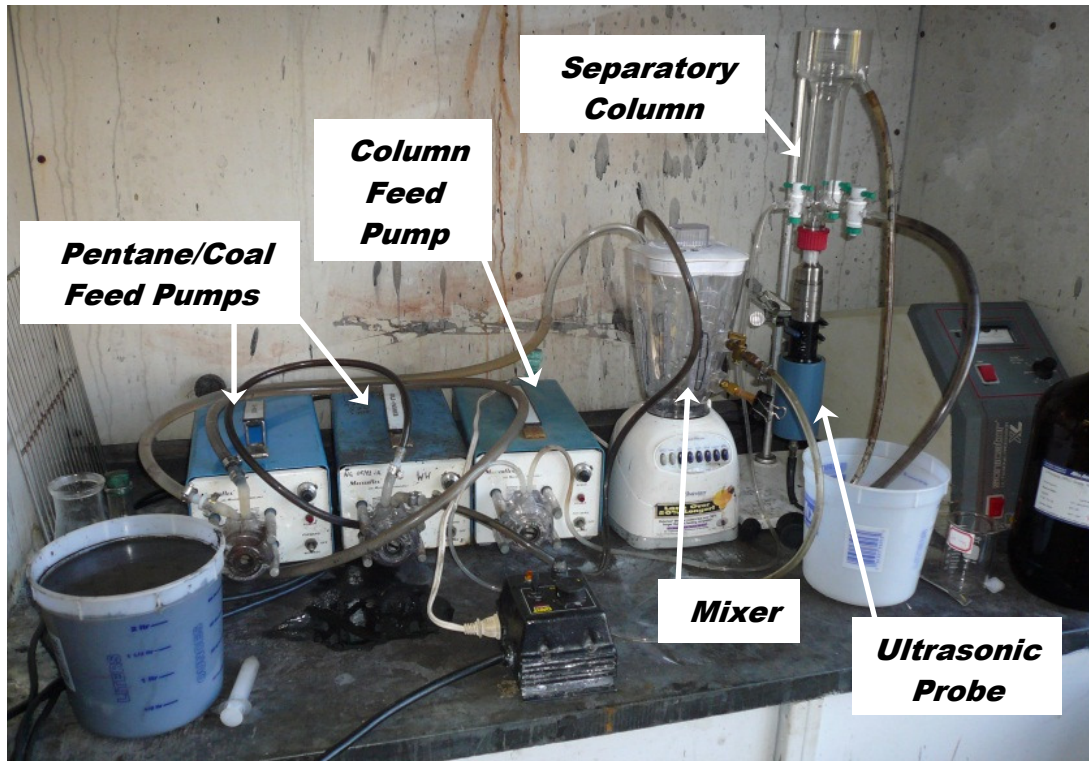


Figure 3-7 : Experimental setup consisting of feed pumps, kitchen blender for emulsion formation, column feed pump, and separatory column.

### 3.2.1.2 Experimental Apparatus: Emulsion formation with static mixer

In the second experimental configuration, shown in Figure 3-8, two peristaltic pumps were used to feed coal slurry and pentane into the mixing system. The two feed lines joined into a single line and were directed into a mixing loop. A 1/16 hp chemical resistant, magnetically coupled, pump was used to pump slurry and pentane across a 0.25 inch in-line static mixer. The rest of loop was comprised of 0.375 inch steel tubing and 0.625 inch hosing. A tube was connected to a 0.25 inch opening at the top of the static mixer so that a small amount of material could exit the loop, while the majority continued mixing. The same separation column and ultrasonic probe used with in section 3.2.2.1 was used in this experiment. Feed leaving the mixing loop entered the column through one of the two bottom ports. The tailings port was attached to an overflow system so that the tailings would exit the column at a rate necessary to maintain the pentane-water interface at a constant level.

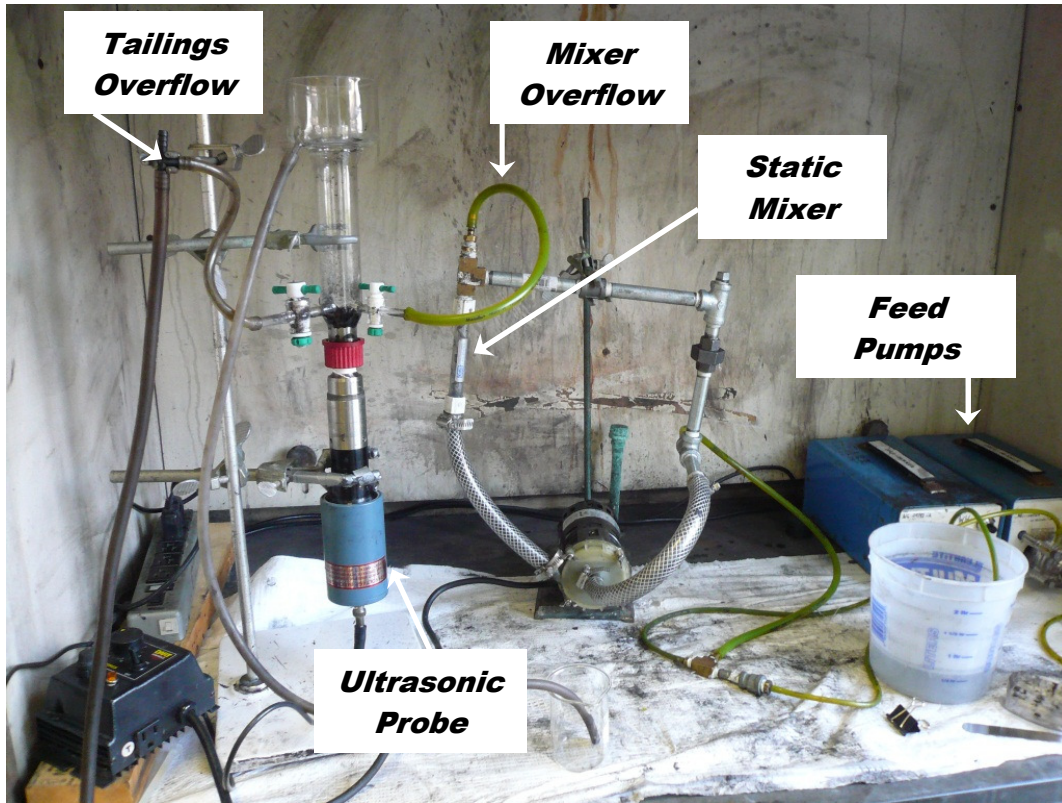


Figure 3-8: Experimental setup consisting of static mixing system and top-mounted separatory column.

In attempt to increase the rate of emulsion breakage, the ultrasonic probe was mounted in a downward position into a 1.5 inch diameter column with a solid bottom, shown in Figure 3-9. The same static mixing system previously discussed was reused. A magnetic stir bar was added to the bottom of the column to keep ash particles from settling and forming a layer across the bottom. Unlike the previous column, emulsions entered the column halfway up the column's length, as opposed to entering close to the probe tip. The feed port was added at to the center of the column to prevent the stir bar from remixing the ash and emulsions.

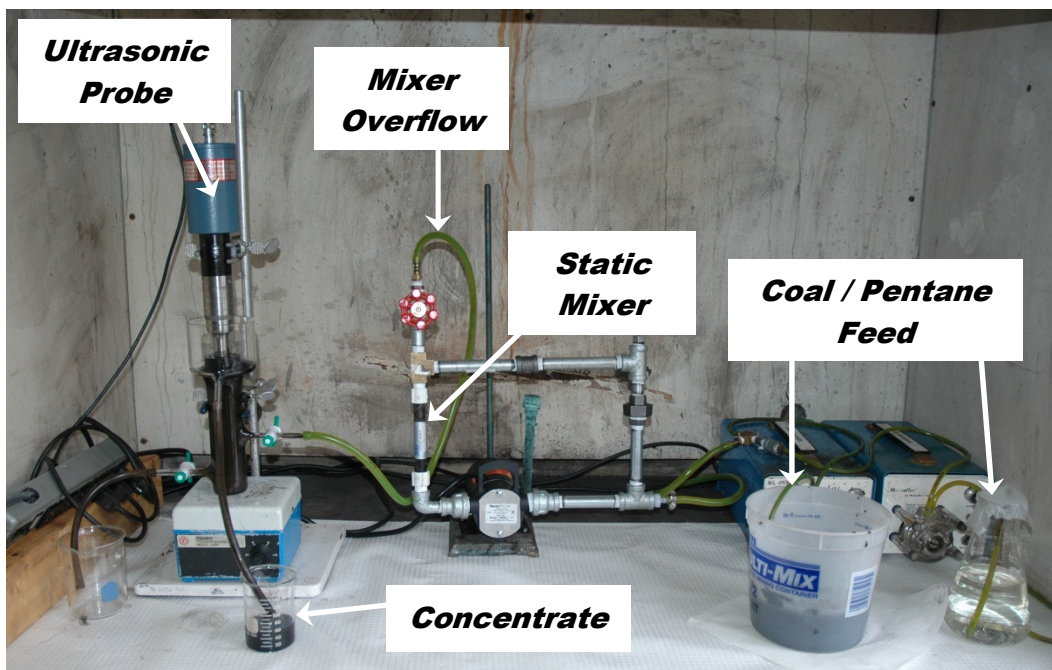


Figure 3-9: Experimental setup consisting of static mixing system and top-mounted ultrasonic probe.

### 3.2.2 Experimental Methods: Breaking emulsions with ultrasonic energy

#### 3.2.2.1 Methods: Emulsion formation with a traditional mixer

In the initial experimental setup shown in Figure 3-6, coal and pentane were pumped into an Oster kitchen blender at equal rates. The blender operated at high speed in order to form the emulsions. Initially, the blender operated at 2 minutes before opening the overflow port. The mixture of emulsions and water overflowed the blender from a side port and fed into the separation column via gravity.

Feed entered the column through one port while the second port was used to drain off tailings. After the initial feed entered the column, the feed port was closed for two minutes to allow for initial emulsion breakage. The feed pumps were turned off during the initial two minutes of operation to prevent a buildup of material in the mixer. The pumps were turned back on after the feed port was opened and the initial two minute sonication period had passed. As emulsions continued to enter the column, pentane and coal overflowed the weir and was collected for drying. In initial tests, coal was filtered on filter paper under the fume hood. In later

tests, the coal/pentane concentrate was evaporated in the evaporation circuit of the continuous unit discussed in Chapter 4.

### **3.2.2.2 Methods: Emulsion formation with static mixer**

Initially, the coal and pentane feed pumps were turned on to allow the system to fill. The feed valve on the column remained closed during this time. Once the pump and pipes comprising the mixing loop were full, the centrifugal pump was turned on and the feed pumps were turned off. The centrifugal pump ran for two to three minutes, giving time for the static mixer to form emulsions from the slurry and pentane.

After initial mixing, the feed valve to the column was opened and the slurry and pentane feed pumps were turned back on. Slurry entered the column at the same flow rate which fresh pentane and coal were being pumped into the mixing system. A gravity overflow system was added to the overflow valve. The height of the t-joint in the overflow system controlled the height of the pentane-water interface. This prevented the system operator from having to control the level manually by sporadically opening and closing the overflow valve.

As with the previous process, new feed entering the column caused the dispersed coal in the pentane phase to overflow. The dispersed coal was poured over a filter paper so that pentane could drain out of the coal. The tailings from the column were also filtered and dried.

## **3.2.3 Results: Breaking emulsions with ultrasonic energy**

### **3.2.3.1 Results: Emulsion formation with Laboratory Mixer**

The ultrasonic probe was consistently able to dewater the emulsions so that the final product had less than 2.0% moisture. The mean, standard deviation and confidence interval were calculated for the moisture values obtained. The moisture values were converted to a normalized Z value. Any samples more than four standard deviations away from the mean moisture value were discarded from the data set. The average moisture value obtained using the data in Table 3-11 was  $0.96\% \pm 0.15\%$  at a confidence interval of 50%. Please note that two samples do not have moisture values listed. For these samples, so little sample was collected that it was impossible for the scale to accurately read the change in weight after the sample was removed

Table 3-11: Results obtained by breaking emulsions in the ultrasonic separator.

Sample	Feed Ash (%)	Tailing Ash (%)	Product		Recovery (%)	Separation Efficiency (%)	Ash Rejection (%)
			Ash (%)	Moisture (%)			
Flotation Feed	9.91%	86.24%	3.30%	1.32%	98.80%	68.14%	68.14%
Flotation Feed	41.11%	82.66%	3.36%	0.28%	85.98%	81.70%	81.70%
Flotation Feed	42.73%	81.86%	3.27%	1.30%	84.10%	80.29%	80.29%
Flotation Feed	36.65%	84.43%	3.94%	-	90.32%	83.63%	83.63%
Screen Bowl Effluent	19.22%	73.06%	3.38%	-	92.42%	78.83%	78.83%

from the oven; therefore, the moisture content could not be calculated. These samples were not factored into statistical moisture calculations, but were included in recovery calculations.

The recovery values ranged between 84.10% and 98.80% with an average of 90.25%. The separation efficiency increased compared to the agglomerates screening method. The separation efficiency averaged 78.50%. Many experiments were performed to attempt to break the emulsions; however, throughput of the system was so low that for many tests there was not enough dry coal collected to properly measure the moisture and recovery.

### 3.2.3.2 Results: Emulsion formation with static mixer

Experiments tried using the static mixer were largely unsuccessful. Initially, as sample circulated through the mixing loop, emulsions exited through the discharge port at the top of the loop. As the material in the mixing loop continued to circulate, the discharge rate of the emulsions began to slow down. After running in the range of 5-10 minutes, emulsions completely stopped exiting the discharge port and only pentane was leaving the loop. As the pump continued to run, the coal plugged the static mixer and water traveled through the mixer via capillaries formed through the coal.

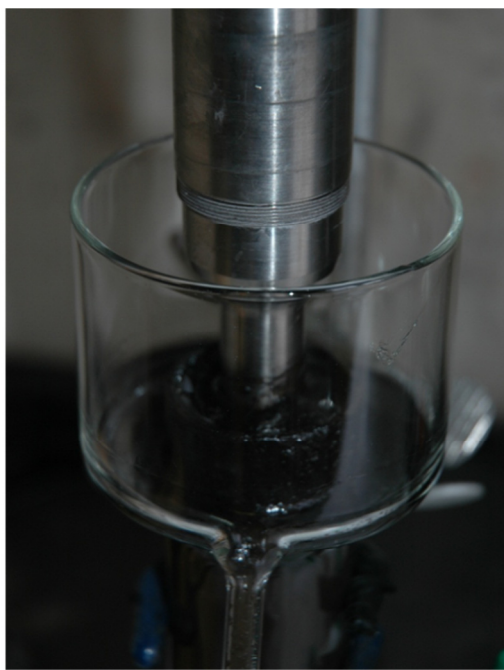
The plugging of the mixer could be prevented by drastically decreasing the feed ratio of coal to pentane. When the coal ratio was decreased, there was insufficient coal in the system to feed the separation column. As the coal in the system built up, the same plugging problem would be encountered, though the mixer was able to operate for a longer time period before plugging.



For coal that entered the separation column with the bottom mounted probe, the ultrasonic probe did a very poor job of breaking the emulsions. The concentration of coal in the pentane phase overflowing was extremely low, so that the overflow was practically clear. To combat this, a column with a flat bottom was fabricated and the probe was mounted downward, through the top of the column. It was theorized that the waves from the probe would reverberate off the bottom of the column, helping to better suspend coal particles. As the emulsions entered the column, the waves from the probe forced the particles downward. The pentane phase remained completely clear. As new emulsions entered the column, the pentane phase overflowed. After all of the pentane had left the column, a mixture of water and emulsions began to overflow. The energy from the probe caused the emulsions to remix with the tailings and form new oil-in-water emulsions. Figure 3-10a shows the remixing effect caused by the probe. When the probe was bottom-mounted a distinct color separation between coal and tailings could be seen (see Figure 3-6); however, with the top-mounted probe, the material in the column is uniform in color. Figure 3-10b shows an up close view of the emulsions overflowing the column during operation.



(a)



(b)

**Figure 3-10: (a) Picture showing the remixing of the sample caused by the ultrasonic probe. (b) Picture showing wet emulsions overflowing the separatory column**

### **3.2.4 Discussion: Breaking emulsions with ultrasonic energy**

Using the ultrasonic probe to break emulsions created a low moisture product at a high recovery; however, the process was inefficient in terms of throughput. The percentage of solids in the overflow was very low, less than 0.5%. The low solids concentration could be attributed to the ultrasonic probe. Instead of breaking the emulsions and sending all of the surface coal to the pentane phase, it is believed that the vibration waves from the probe actually split the majority of emulsions, so that one emulsion would be divided into multiple emulsions by the waves. Any coal into the pentane phase was “thrown” off the surface of the emulsion when the emulsion was split.

The rate of emulsion breakage was extremely slow. As the probe operated and new emulsions fed into the column, the thickness of the emulsions layer within the column continually increased. Eventually, emulsions would fill almost the entire length of column. The amount of time necessary for the emulsions to fill the column depended on the feed rate into the column, but the build-up of the emulsion layer was observed in every test, regardless of feed rate.

Another operating issue encountered in the process was heating of the probe tip. The ultrasonic probe operated by vibrating the steel tip at a frequency of 20 kHz, causing the tip to heat. The probe was always placed in the water phase to prevent pentane from exploding; however, after approximately 15 minutes of operation, the probe would generate enough heat to heat the water and cause the pentane layer to boil. After operating approximately 20 minutes, large cavities could be seen forming at the tip of the probe and the glass column was warm to the touch. It is believed that the water directly against the tip was hot enough to boil, causing the cavities. At this point, the tip of the probe was very hot. Experiments were never conducted for longer than 25 minutes due to the heat.

### **3.3 Breaking agglomerates with ultrasonic energy**

#### ***3.3.1 Experimental Apparatus: Breaking agglomerates with ultrasonic energy***

##### **3.3.1.1 Experimental Apparatus: Preliminary Testing**

Due to the low throughput and inability to continuously run the emulsion process described in Section 3.2, other coal/hydrophobic liquid products that could be fed into the separatory column were explored. Once again the kitchen blender was used to create the agglomerates for the process. The ultrasonic probe was mounted at the bottom of a 1.75 inch diameter, 5 inch tall glass separatory column with a single port at the top of the column for product overflow. To overflow the coal/pentane product, liquid pentane was manually poured into the top of the column by hand. The tailings were removed from the column using an overflow system designed to maintain the interface at a constant height within the column.

##### **3.3.1.2 Experimental Apparatus: Moisture Testing**

Tests were performed in a custom made, 1.5 inch diameter, separatory column. The ultrasonic probe mounted to the bottom of the column so that the distance between the tip of the probe and the overflow spout was 3 inches. A tube entering the top of the column was connected to a peristaltic pump set to pump pentane into the column at a rate of 48 mL/min. Figure 3-11 shows the experimental setup for the process.



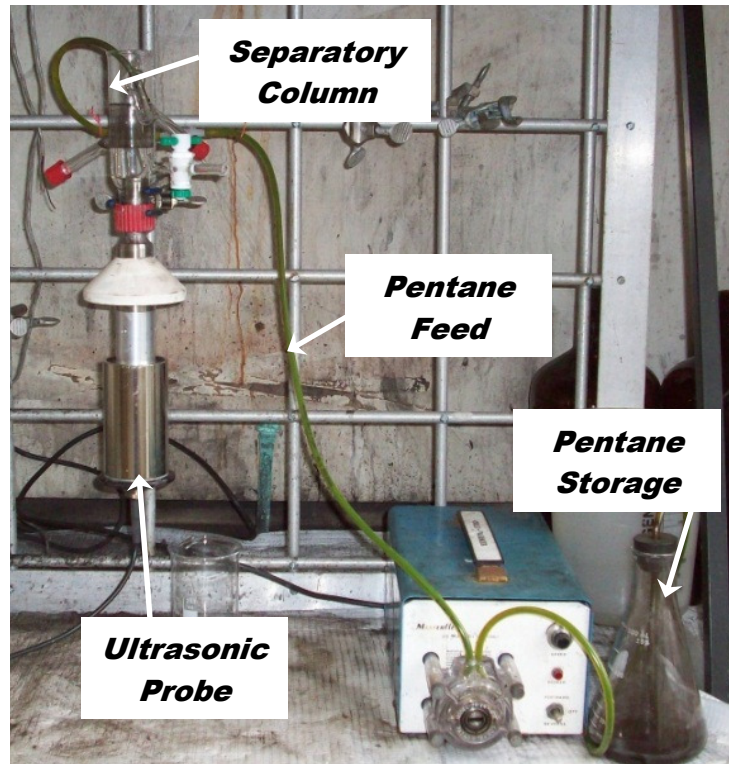


Figure 3-11: Experimental setup used to determine the effect of interface distance from probe tip on moisture.

### **3.3.2 Experimental Methods: Breaking agglomerates with ultrasonic energy**

#### **3.3.2.1 Experimental Methods: Preliminary Testing**

Initially, the screening process followed in Section 3.1.2 was modified so that the overflow agglomerates were poured into the ultrasonic separatory column to be cleaned and dried. Figure 3-12 shows the flow sheet of the process followed. Coal agglomerates were made by mixing 600mL of slurry (6% solids) from CONSOL’s Bailey preparation plant and 10mL of hydrophobic liquid in a kitchen blender. For the initial 60 seconds of mixing, during which the agglomerates formed, the blender was operated at a high speed to ensure a high shear mixing environment. To increase the diameter of the agglomerates, the blender was turned down and operated at low shear for 30 seconds. The agglomerates floated on top of the water and ash phase. The blender contents were poured across a 30 mesh screen to remove water (the “Primary Dewatering Screen” in Figure 3-12). The water stream was collected, dried, and assayed.

The agglomerates remaining on the screen were poured onto a second screen, smaller mesh screen and shaken so that dry particles fell through the mesh (the “Secondary Dewatering Screen” in Figure 3-12). For two of the three tests, a 50 Mesh screen was used as the secondary dewatering screen. In the final test, the secondary dewatering screen had 70 mesh openings. The screens were shaken until no new material fell through. The agglomerates remaining on the screen were hand-fed into the ultrasonic 1.75 inch diameter by 5 inch separatory column used in previous sections. Unlike emulsions which were fed into the column in the water phase, agglomerates were poured into the top of the column, into the pentane phase.

Additional pentane was poured into the top of the column to cause the dispersed particles to overflow the column. The pentane was separated from the coal through evaporation. The column overflow was combined with the secondary dewatering screen underflow to compose the final coal product.

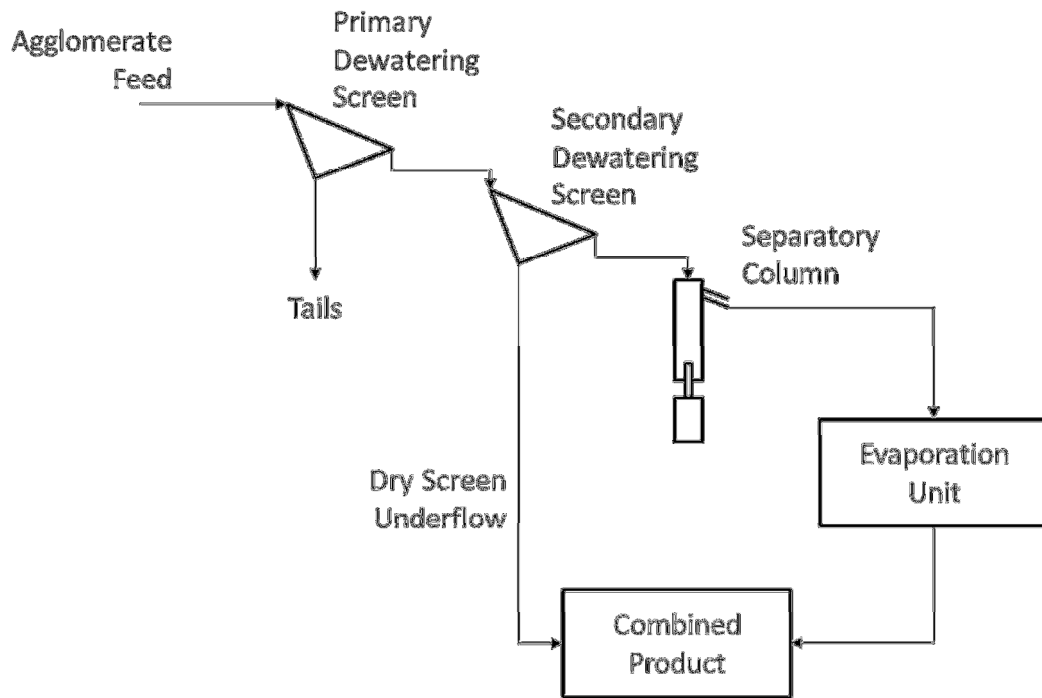


Figure 3-12: Flow sheet of process combining screening and ultrasonic separation to clean and dry coal.

The majority of preliminary tests were conducted modifying the process described above so that no secondary dewatering screen was used. Agglomerates were formed in the same manner discussed above using flotation feed from Alpha Natural Resources' Kingston preparation plant and flotation feed from CONSOL's Bailey preparation plant. After dewatering the agglomerates on the primary dewatering screen, the separatory column and ultrasonic probe described in the previous process were used to break the agglomerates. The column was filled with a small volume of water so that the water level was approximately 1 inch above the probe tip. The remainder of the column was filled with hydrophobic liquid. Agglomerates were removed from the screen and dropped into the hydrophobic liquid phase. The agglomerates broke up and coal dispersed into the hydrophobic liquid phase almost immediately. Additional hydrophobic liquid was poured into the top of the column to overflow the liquid and coal. The hydrophobic liquid was evaporated, leaving behind dry coal.

#### **3.3.2.2 Experimental Methods: Moisture Testing**

These experiments were focused on determining the effect of the interface distance from the probe tip on the moisture, recovery, and percent solids in the final product. The distance of the pentane/water interface from the probe tip was varied at 4 separate experimental distances. The first sample was taken when the interface was 1 inch away from the probe tip subsequent samples were taken at 0.5 inch increments until the interface distance was 2.5 inches away from the probe's tip.

Agglomerates were produced by mixing 500 mL of flotation feed from Alpha Natural Resource' Buchanan preparation plant with approximately 10mL of pentane. The sample was agitated at high speed for 30 seconds then low speed for 30 seconds. After the agglomerates had formed, the contents of the blender were poured across a 100 mesh screen and allowed to drain for 10 minutes. The tailing material was dewatered and dried for ash analysis.

A portion of the agglomerates were weighed and dried so that an initial moisture value could be calculated. The remaining agglomerates were weighed in aluminum dishes to serve as the feed for the column. One gram of agglomerates was weighed into the first dish to serve as the initial feed to the column. Half a gram of agglomerates were weighed into the remaining six dishes.

For all experiments, the ultrasonic probe was set to operate at 15 amps. Initially, the column was filled with enough water to reach the desired interface height. The remaining column volume was filled with pentane until pentane just started to exit through the overflow spout. The probe was turned on and the 1 gram sample was poured into the column. As soon as the sample entered the column, a timer was started. After 30 seconds of operation, the pentane pump was turned on and one of the 0.5 gram samples was added. Half a gram of agglomerates were added every 30 seconds. The last sample was added at a running time of 3:30. After the last sample was added, the pentane pump continued running for 2 minutes to allow the coal in the system to continue to overflow. After a total running time of 5:30, the collection beaker was removed from below the overflow spout and the pump and probe were turned off.

The total volume of sample collected was measured and the pentane/coal overflow was poured into the evaporation chamber of the continuous testing circuit. The continuous circuit was used to evaporate the excess pentane. After all pentane had evaporated and condensed, the coal was removed from the chamber and weighed for moisture analysis.

### **3.3.3 Results: Breaking agglomerates with ultrasonic energy**

#### **3.3.3.1 Results: Preliminary Testing**

For initial tests, the agglomerates were dewatered on a sieve, and then shaken on a screen similar to the process used in section 3.1. Results of the process are shown in Table 3-12. All tests used 30 mesh sieves to remove the tailings water from the agglomerates. The first two tests used a 50 mesh sieve as the secondary dewatering screen, while Test 3 used a 70 mesh sieve. The underflow moistures from the 50 mesh sieves were 16.53% and 15.68%, respectively. The underflow of the 70 mesh screen was much dryer, containing only 2.63% moisture.

**Table 3-12: Results obtained by screening agglomerates then dewatering overflow in ultrasonic separator.**

Test	Underflow Moisture	Agglomerate Moisture		Product Moisture	Recovery, %
		Before Sonication	After Sonication		
1	16.53	-	8.42	15.35	85.61%
2	15.68	30.98	1.01	13.97	84.09%
3	2.63	19.90	0.60	1.10	84.58%

The agglomerates which remained on top of the sieve were removed and poured into the separatory column. The column was very effective at the reducing the agglomerate moisture. For the second test, the agglomerates entering the column contained nearly 31% moisture while the overflow material had a moisture content of 1%. The +70 mesh material had a moisture content of nearly 20% when entering the column, and a product moisture of less than 1%.

The final product moisture was calculated by combining the screen underflow with the overflow from the separatory column. The screen underflow comprised a high percentage of the final product; therefore, the low moisture column overflow was unable to reduce the moisture for the first two tests below the target 10% value. For the first two tests with high moisture underflow samples, the total product moistures were approximately 15% and 14%, respectively. The third test had very promising results, yielding a final product with containing 1.10% moisture.

The recovery for all three tests averaged 84.76%. It was theorized that the low recovery was due to screen loses and entrained ash present in the small water droplets carried through with the screen underflow. Because the probe was so effective at reducing the moisture present in the overflow agglomerates, it was determined that screening the agglomerates before sonication to was not necessary. Therefore, no additional tests using this method were performed.

All other preliminary tests were performed by feeding dewatered and de-ashed agglomerates into the separatory column with no pre-screening. Table 3-13 shows the results for the individual preliminary tests performed using flotation feed from the Kingston preparation facility. Tests 1 - 4 were used to calculate the moisture and recovery of the overall process; therefore, the agglomerate moisture was not calculated in these initial tests. These tests were performed in the 1.75 inch diameter, 5 inch tall column. Tests 5 - 14 were used to assess the probe's effectiveness at reducing the moisture and ash within the agglomerates using the 1.5 inch diameter, 3 inch high column.

Pouring the agglomerates into the ultrasonic separatory column proved to be an extremely effective cleaning process. The flotation feed contained an average of 54.6% ash. After agglomeration, the ash was reduced to an average value of 7.13%. Pouring the dewatered agglomerates into the separation column reduced the ash to below 5%. The process was

extremely consistent in its separation and produced high recovery rates. The average recovery in the process was  $94.09\% \pm 1.22\%$ . The average ash rejection of the process was  $99.23\%$  with a standard deviation of  $0.003\%$ . The separation efficiency these tests were the highest off all the other experiments conducted. The average separation efficiency was  $93.33\%$  with a standard deviation of  $0.04\%$ .

The sonication process was also effective in creating a dry coal product. The largest moisture reduction can be found in Test 5. The agglomerates fed into the separatory column contained  $58\%$  moisture. After sonication, the sample contained  $0.60\%$  moisture. The average agglomerate moisture in the testing data was  $42.22\%$ . This number was reduced over 38 times so that the concentrate sample had an average moisture content of  $1.10\%$ . The histogram in Figure 3-13 shows that of the 14 tests run with the Kingston sample,  $90\%$  of the product samples had moisture contents less than  $1.75\%$ . The products most frequently contained between  $1.00\%$  and  $1.25\%$  moisture. All of the product samples had moistures below  $2.75\%$ .

Due to slight differences in testing methods, the percent solids of the concentrate samples are not included in Table 3-13; however, compared to breaking emulsions with the ultrasonic probe, the column overflow contained a much higher percentage of coal. When breaking agglomerates in the column, the percentage of solids in the overflow pentane was as high as  $2.26\%$  (compared to less than  $0.5\%$  when breaking emulsions). The percent solids depended heavily on the flow rate of the overflow pentane fed into the column.

Table 3-13: Preliminary results for cleaning and dewatering Kingston agglomerates in the ultrasonic separator.

Test	Feed Ash, %	Agglomerate Moisture, %	Concentrate Ash, %	Concentrate Moisture, %	Recovery, %
1	54.5	-	4.53	1.19	89.95
2	54.5	-	4.38	1.16	90.54
3	54.5	-	4.87	1.07	87.97
4	54.5	-	4.05	1.10	89.45
5	55.69	40.76	3.56	1.07	97.65
6	55.69	58.19	4.01	0.60	97.34
7	55.69	45.21	3.77	0.93	97.49
8	55.69	42.31	3.73	1.26	97.46
9	55.69	42.20	3.68	1.70	97.56
10	55.69	32.27	3.27	1.06	97.37
11	55.69	46.83	4.14	2.65	97.85
12	52.17	38.15	4.79	0.61	92.22
13	52.17	38.15	4.74	0.45	92.22
14	52.17	38.15	4.79	0.57	92.22

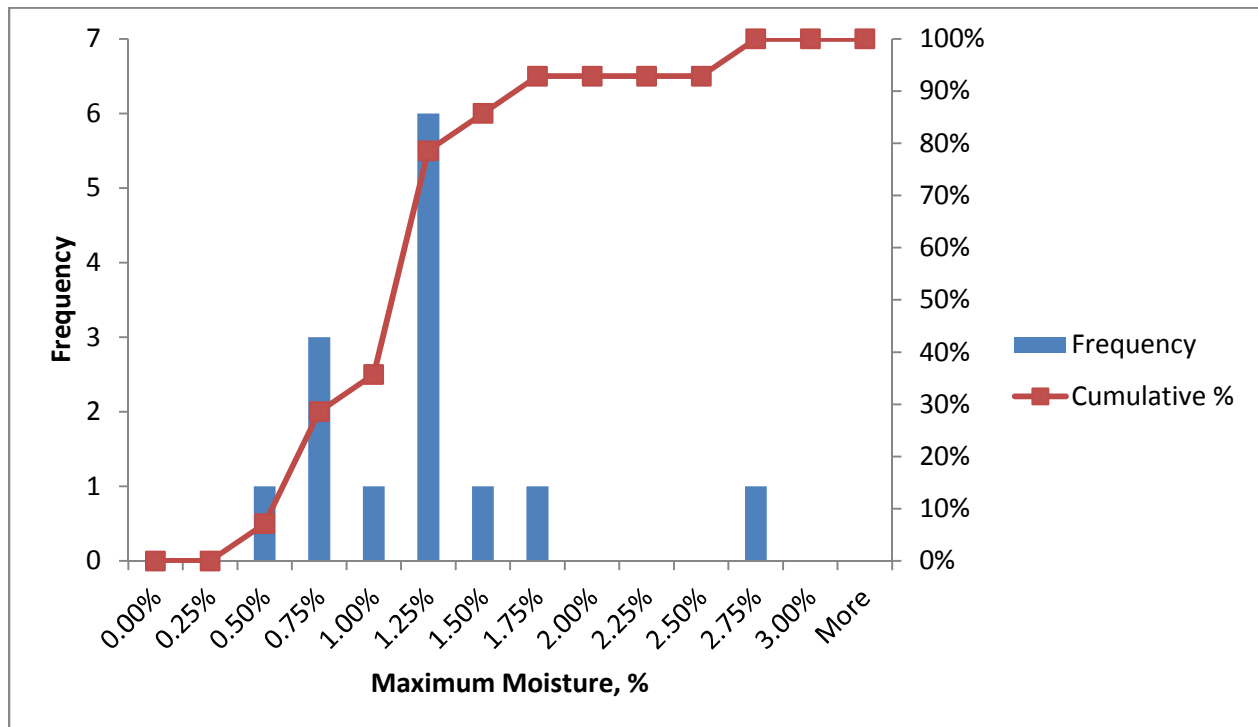


Figure 3-13: Histogram showing the distribution of product moistures for experiments performed in Table 3-13.

**Table 3-14: Data obtained by breaking agglomerated CONSOL coal with ultrasonic energy.**

<b>Feed Ash %</b>	<b>Product Moisture%</b>	<b>Product Ash %</b>	<b>Reject Ash %</b>	<b>Recovery %</b>	<b>Separation Efficiency %</b>	<b>Ash Rejection %</b>
41.11	1.15	3.06	86.94	89.94	88.45	98.51
41.11	8.42	2.43	84.19	87.30	75.65	88.35
41.11	0.60	3.36	86.97	90.01	89.24	99.23
41.11	1.01	3.60	87.41	90.43	89.11	98.68

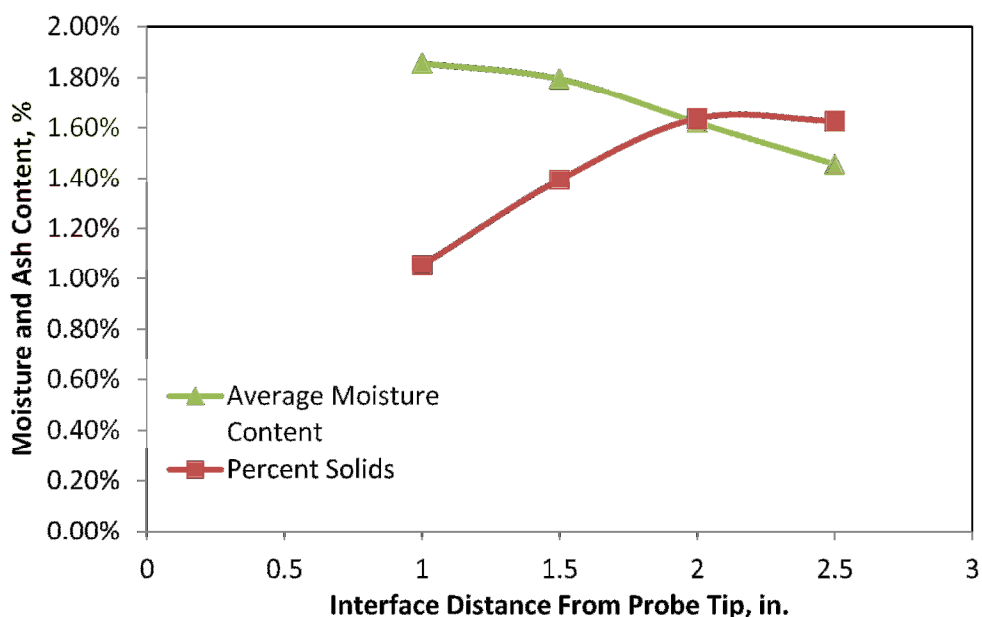
Four preliminary tests were performed using flotation feed from CONSOL’s Bailey Mine. The results are shown in Table 3-14. The average combustible recovery was 89.4%, slightly lower than the Kingston sample. With the exception of one data point, the process was able to reduce the moisture to below 2.0%. It is believed that the 8.42% moisture is due to improper agglomerate breakage; however, the data was included in the set due to the limited number of tests that were performed before sample. When excluding the high moisture point from the data set, the average moisture content was 0.9%. The ultrasonic separator was not as efficient at breaking the agglomerated Bailey coal as it was with the Kingston sample. With the separation efficiency for the 8.42% moisture point excluded, the average separation efficiency was 88.9% with a standard deviation of 0.35%

### **3.3.3.2 Results: Moisture Testing**

The interface distance from the probe’s tip was varied so that samples were taken when the interface was 1, 1.5, 2, and 2.5 inches from the tip, creating interface thicknesses of 2, 1.5, 1, and 0.5 inches, respectively. Figure 3-14 shows the results for the moisture determination tests. To calculate the data found in the plot, multiple tests were performed at each distance, and the results were averaged.

As the distance from the tip increased (decreasing interface thickness), the product moisture decreased. The total range of moistures was very narrow, ranging from an average low moisture of 1.46% at 2.5 inches away from the tip to a high average moisture of 1.86% at 1.0 inch away from the tip. Agglomerates were fed into the top of the column. It is believed that as the water droplets within the agglomerate fell through the pentane phase, heading towards the interface, the upward vibrations produced by the ultrasonic probe “pushed” the droplets further





**Figure 3-14: Plot showing the effect of interface distance from energy source and interface thickness on moisture content, ash content, and recovery of final product.**

upward in the pentane phase. It is believed that the cycle of the droplets falling, then getting pushed upward by vibrations is responsible for the slight increase in moisture in the thicker pentane layers. When the pentane layer is thinner, the droplets have less distance to travel and are not impacted by the probe's upward waves before reaching the water as many times as with a thicker pentane layer.

While the moisture of the sample decreases with increasing interface distance, the percent solids in the concentrate increases. When the pentane layer is thicker, there is a large volume through which the hydrophobic particles can disperse. Therefore, since the same amount of coal was added during each test, it is expected for the solids concentration to increase as the volume of pentane decreases. The concentrate contained 1.05% solids when the pentane thickness was 2 inches. When the thickness of the level was decreased to 1 and 0.5 inches, the percent solids increased to 1.64% and 1.63%, respectively. Thermodynamically, the coal will always prefer to be displaced into the hydrophobic phase as long as there is enough volume of hydrophobic liquid to accommodate all of the coal. It is believed that the percent solids will keep increasing, until the layer is so thin that water is being thrown into pentane phase, causing the overflow to be diluted and the moisture to increase.

Due to a malfunction of the ash analyzer, the ash contents of the concentrate and tailings samples of the latter sets of experiments could not be measured. Before the equipment malfunction, recovery rates and ash values were calculated for the first set of experiments and then used to calculate the data in Figure 3-14. The effects of interface distance on recovery and concentrate ash are shown in Figure 3-15. As the interface distance from the probe increased, the concentrate ash also increased; however, the increase was very slight. At a distance of 1 inch, the concentrate contained 1.98%. At 2.5 inch, the ash content had increased to 2.09%. Overall, the process produced an average concentrate moisture of 2.02% with a standard deviation of 0.06%.

The recovery of the sample peaked at distance of 1.5 inches; at which point the process recovered 84.54% of the combustible material. Over the four tests, the process produced consistent recoveries, ranging from 83.58% to 84.54%, depending on the interface level. The average recovery was calculated to be 84.14% with a standard deviation of 0.45%.

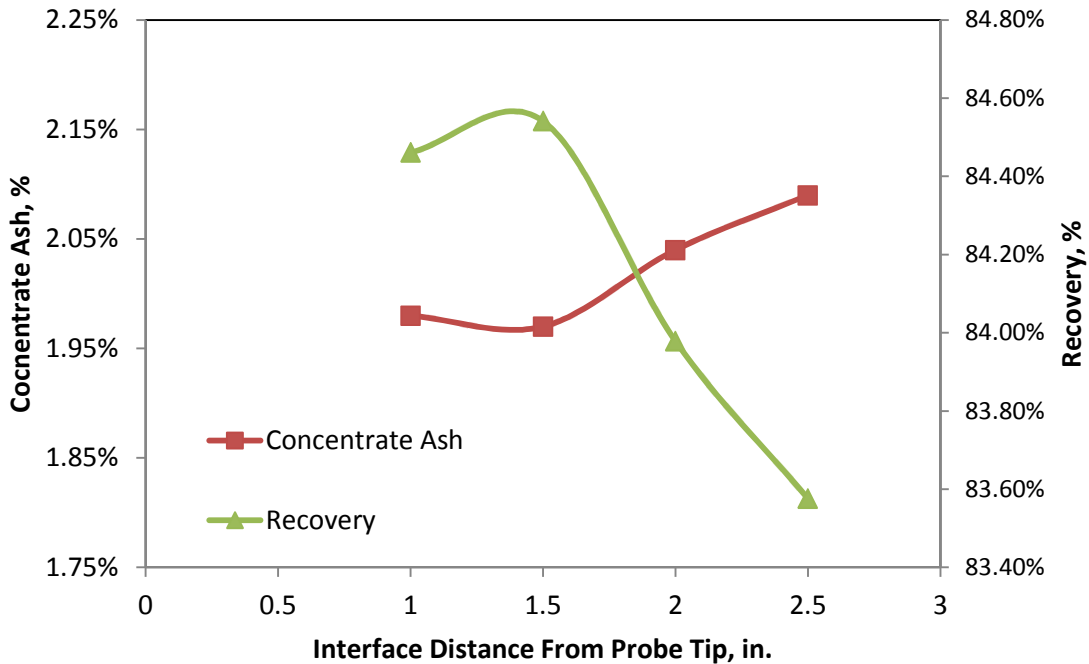


Figure 3-15: The effect of interface distance on recovery and concentrate ash.

### **3.3.4 Discussion: Breaking agglomerates with ultrasonic vibrations**

Breaking agglomerates using ultrasonic vibrations was successful in both cleaning and dewatering coal. The main advantage of using ultrasonic energy to break agglomerates over using ultrasonic energy to break emulsions is higher throughput. The probe was able to break the agglomerates at a much faster rate than the emulsions, causing more coal to be present in the column overflow. As previously mentioned in section 3.2.4: *Discussion*, it was believed, through visual observations, that the ultrasonic vibrations did not actually break the emulsions, but instead, split the emulsions into smaller and smaller emulsions. However, when agglomerates were introduced into the column, they broke immediately and coal dispersed throughout the pentane phase. The pentane phase in the column turned black, with no transparency, compared to being nearly clear at times when attempting to break emulsions.

While breaking the agglomerates was successful in drying the coal, the process also has some drawbacks. As previously discussed in section 3.2.4: *Discussion*, heating of the ultrasonic probe's tip proved to be a problem. It was impossible to operate the column for longer than 25 minutes due to the heat generated by the probe. Heating caused pentane to evaporate at a faster rate and eventually led to boiling in the water phase. Once the water began to boil, selectivity of the process was greatly reduced as the boiling water caused water and wet particles to be displaced into the pentane phase.

With most coal samples, emulsions began to form at the pentane/water interface after running for a certain time period. Coal from Alpha Natural Resources' Buchanan preparation plant was more hydrophobic than other samples used, and no emulsions formed. For all other samples, the emulsion layer which formed was very thin and did not seem to interfere with rate of agglomerate breakage. However, the probe was unable to run for a sufficient time period (due to overheating), to determine if the emulsion layer could eventually interfere with breakage.

Overall, the process was successful in meeting the goals of the project; however, one main concern regarding the process is the scalability of the ultrasonic probe. The largest ultrasonic probe which is currently being produced has a tip diameter of 2 inches, which will not allow for the throughput required to have an industrial scale process. Other industrial options which could be used in place of the ultrasonic probe were explored. One option considered included using ultrasonic transducers outside of a horizontal pipe and feeding agglomerates and

pentane into the pipe. However, due to the high energy consumption of the probe, ultrasonic vibrations were abandoned for a lower energy option, using a mechanical vibration generator to pulsate mesh discs within the pentane phase. This new option is discussed in section 3.4: *Breaking agglomerates with vibrating mesh.*

### 3.4 Breaking agglomerates with vibrating mesh

#### 3.4.1 Experimental Apparatus: Breaking agglomerates with vibrating mesh

Before separation, a Ninja kitchen blender was used to create the agglomerates. The separation took place in a custom-made glass column 5 inches high and 1.5 inches in diameter. A model 2007E electrodynamic shaker produced by the Modal Shop INC. was used to displace coal particles into a pentane phase. A shaft with three mesh discs extended from the bottom of the shaker. The openings in the mesh discs decreased in size going down the shaft (away from the shaker). The lowest plate had an opening of 0.5 millimeters and the upper plate had an opening of 80 micrometers. The mesh disks pulsed at 30 hertz with an amplitude of 0.5 inches. The experimental setup is shown in Figure 3-16.

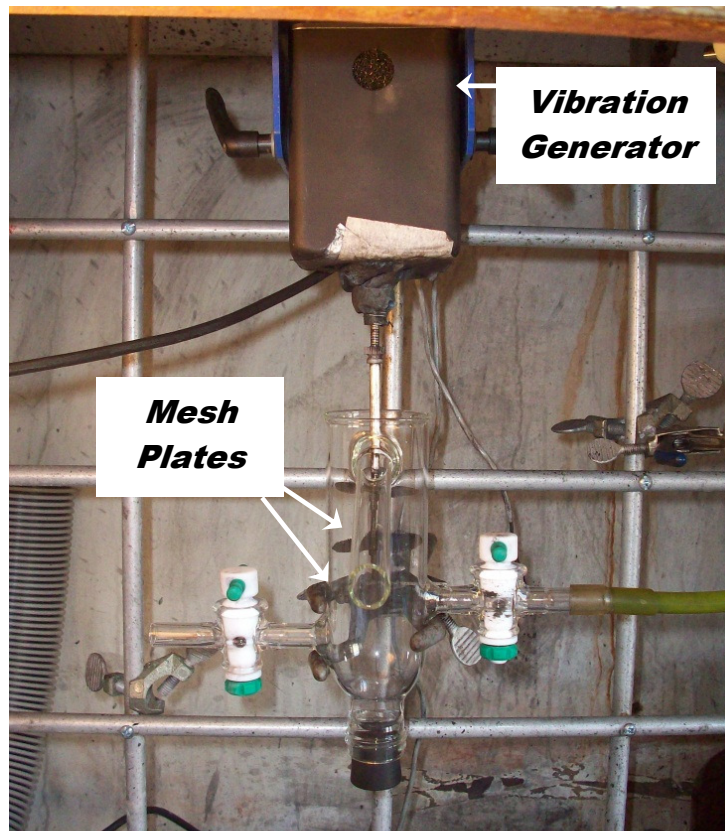


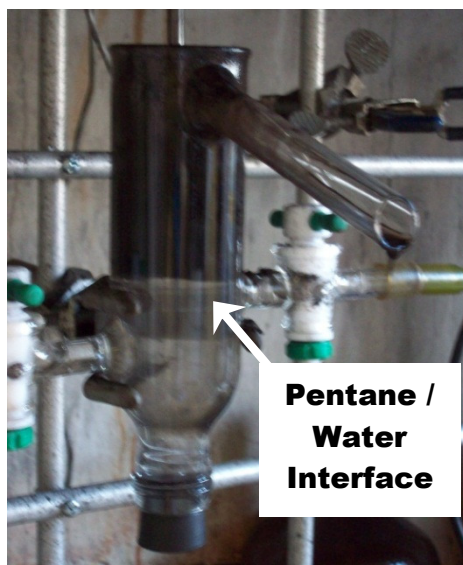
Figure 3-16: Experimental setup used for breaking agglomerates with vibrating mesh plates.

### **3.4.2 Experimental Methods: Breaking agglomerates with vibrating mesh**

Before the testing began, water was poured into the lower portion of a glass column and pentane was poured into the upper portion, creating an interface. The mesh discs were lowered into the column. The discs were positioned so that the lower disc sat at the pentane/water interface.

Before testing, a Ninja brand kitchen blender with a flat, three tiered paddle was used to create agglomerates. Pentane dosages were determined based on the coal sample used to create the agglomerates. Six hundred milliliters of coal slurry was poured into the blender. High shear mixing was started immediately after 20 ml of pentane was added to the slurry. The pentane and slurry were mixed for 30 seconds so that small, powder-like agglomerates could form. The agglomerates were dewatered on an 80 mesh screen.

The mechanical shaker was turned on and set to operate at 30 Hz. The agglomerates were removed from the screen on a laboratory spoonula and dropped into the top of the glass column. Immediately, the agglomerates dispersed into the pentane phase and ash could be seen falling into the water phase. The initial coal dispersion is shown in Figure 3-17. Pentane was slowly poured into the top of the column to overflow the coal-pentane mixture. The process of adding more coal particles and overflowing the column was repeated until enough product for ash analysis had been collected. If needed, ports at the bottom of the column were used to drain the ash containing water so that the interface could be maintained at the same level as the lower disc.



**Figure 3-17: Complete dispersion of coal in the pentane phase (above the pentane/water interface)**

The coal/pentane product was then poured into the evaporation circuit of the continuous separation unit discussed in *Chapter 4: Continuous Testing Unit*. The pentane was evaporated and condensed into a separate chamber, leaving behind dry coal. The dry coal was removed from the system and the final product moisture was calculated. Results from these experiments are discussed in section 3.4.3.

### **3.4.3 Results: Breaking agglomerates with vibrating mesh**

Breaking agglomerates with the vibrating mesh was successful in both cleaning the coal and creating dry final product. The process produced results similar to the ultrasonic probe used to break agglomerates. Flotation feed samples from both the Kingston preparation plant and the Bailey preparation facility were used to test the process. Screen bowl effluent samples from Bailey were also tested. Results of the experiments are shown in Table 3-15.

The Kingston sample produced an average recovery of 89.8%, approximately 5% lower than the average recovery obtained by breaking agglomerates from the same sample with the ultrasonic probe, with an average moisture content of 3.63%. The separation efficiency of

**Table 3-15: Results obtained for Kingston and Bailey samples using vibrating mesh to break agglomerates.**

Sample	Feed Ash, %	Tailings Ash, %	Concentrate Ash, %	Concentrate Moisture, %	Recovery, %
Kingston Flotation Feed	51.00	90.00	3.56	4.20	88.87
Kingston Flotation Feed	52.60	91.60	4.01	3.20	90.10
Kingston Flotation Feed	52.60	91.40	3.77	3.50	89.87
Kingston Flotation Feed	52.60	91.70	3.73	3.60	90.26
Bailey Flotation Feed	44.90	87.10	5.50	1.10	88.70
Bailey Flotation Feed	44.90	87.70	4.60	0.70	89.20
Bailey Screen Bowl Effluent	40.40	87.60	4.14	3.80	90.91
Bailey Screen Bowl Effluent	40.40	87.90	4.79	3.90	91.18

86.67% was also lower than obtained with the ultrasonic probe. The samples from the Bailey plant had an average recovery of 88.9% with a separation efficiency of 84.39%. Lower moisture contents were obtained with the flotation feed compared to the screen bowl effluent. The screen bowl effluent product moisture content was an average of 3.9% compared to an average of 0.90% for the flotation feed sample.

#### **3.4.4 Discussion: Breaking agglomerates with vibrating mesh**

Using the vibrating mesh to break agglomerates is a very effective process. The recovery and moisture results obtained using the vibrating mesh plates are not as good as the results obtained by breaking the agglomerates with the ultrasonic probe; however, the two processes are very comparable. As previously mentioned, there is currently no large-scale ultrasonic device to act as a substitute for the ultrasonic probe. The vibrating mesh plates are capable of being scaled up for use in pilot-scale and industrial-scale processes, giving this method of agglomerate dispersion a competitive advantage over the ultrasonic dispersion method.

The vibrating mesh separator tended to produce higher moisture contents than the ultrasonic probe for the Kingston sample, but produced an equivalent moisture content for the Bailey flotation feed sample. The screen bowl effluent was not tested in the ultrasonic separator;



therefore, the product moisture could not be compared. Though the Kingston product had higher moisture, the average moisture values of 3.6%, 3.9%, and 5.1% obtained for the Kingston and two Bailey samples, respectively, are well below the target 10% moisture and are acceptable values for a final product.

The vibrating mesh produced a lower average recovery for the Kingston flotation feed by nearly 5% compared to the ultrasonic probe. This lower recovery is most likely due to two factors: sample age and coal loss through the dewatering screen. Though both samples came from the Kingston preparation facility, the sample had aged for approximately 2 months before being agglomerated for use with the vibrating mesh separator. Aging of the sample leads to oxidation and decreased surface hydrophobicity. Particles with a high degree of decreased hydrophobicity will not attach to agglomerates and will therefore fall through the agglomerate dewatering screen. Any coal which passes through the screen will float on the surface of the tailings water. Therefore, any sample lost through the screen could easily be skimmed from the surface and recovered for additional conditioning.

Besides scalability, the vibrating mesh offers three other advantages over the ultrasonic process: decreased operating cost, lower operating temperatures, and higher throughput. The vibration generator used to move the discs uses less energy than the ultrasonic probe, resulting in a decreased energy cost. Unlike the ultrasonic probe which has the potential to overheat the sample in the separatory column, the vibrating mesh discs do not generate a significant amount of heat and no sample-overheating issues were ever encountered; therefore, the risk of boiling and subsequently displacing water particles into the product is greatly diminished. From a safety aspect, the risk of creating a pentane explosion is also virtually eliminated.

## Chapter 4: Continuous Testing Unit

### 4.1 Experimental Apparatus: Continuous Testing Unit

The continuous testing unit shown in Figure 4-1 was designed and built at Virginia Tech. The majority of the components are custom-made glassware. Tests were conducted using both emulsions and agglomerates. With the exception of the first mixing stages to form the agglomerates or emulsions, the process was the same for both materials. To create emulsions, feed slurry was pumped into a mixing chamber. The pentane reagent tank also pumped into the mixer to form the emulsions. To prevent pentane vapor from entering the mixing motor, a flexible shaft extended from the mixing motor, into the mixing tank. Agglomerates were first formed in a blender outside of the system, as the mixing tank did not provide enough shear for agglomeration to occur. The agglomerates were then pumped into the mixing tank to be mixed at

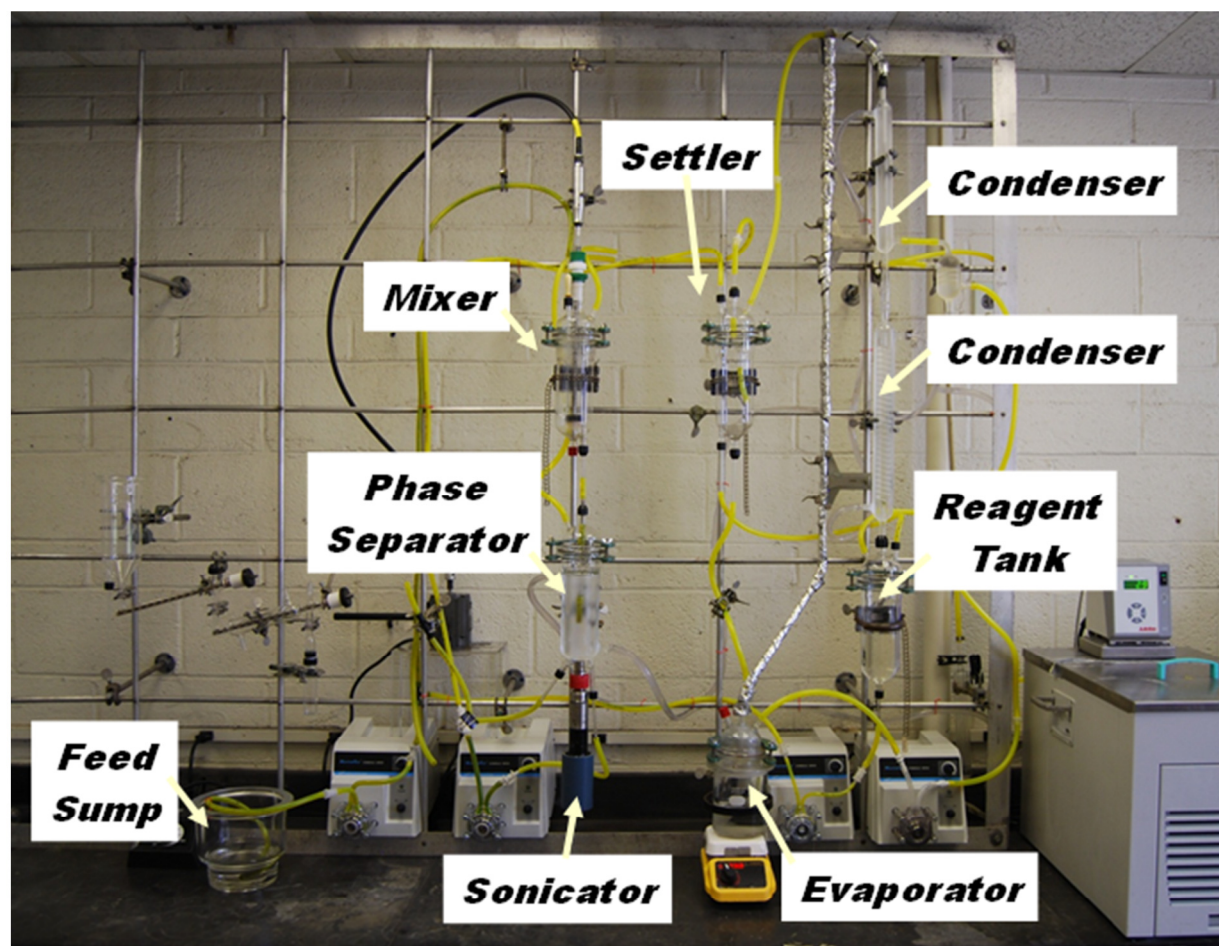


Figure 4-1: Continuous testing unit with labeled unit operations (photo by Jerry Luttrell).

low shear. No pentane was added to the mixing tank. The pump had a difficult time moving the dry agglomerates; therefore, in some trials, the agglomerates formed in the blender were poured into the tank. Emulsions or agglomerates in the mixing tank overflowed from a vertical pipe into the ultrasonic separatory column.

The column was mounted on top of the ultrasonic probe used in previous experiments. A cooling jacket was built around the column to prevent the pentane and water within from boiling. An overflow port at the bottom of the column was attached to flexible hosing and a t-joint open to the atmosphere to create interface level-control system used in previous experiments. Pentane from the reagent tank was pumped into the column to overflow the suspended coal through a port in the upper portion of the column. The overflow was pumped to a settling tank to give coal particles time to settle out of the pentane phase.

The settling tank had two ports, an adjustable overflow port so that clear pentane at the top of the settling tank could overflow into the reagent tank and an underflow port for thickened coal to flow into the evaporator. A valve was placed on the underflow tube so that the operator could control the rate at which thickened coal entered the evaporator. A double boiler was utilized in evaporating the pentane. The ignition temperature of pentane is 260°C (Alfa Aesar 2009). To fabricate the double boiler, a large beaker of water was placed on a hot plate. The sealed pentane evaporator was placed into the water. The maximum temperature the water would reach was 100°C before boiling. This system ensured that pentane would never reach its ignition point, only a maximum of 100°C. As the pentane evaporated, it traveled upward and was condensed by two condensers. The liquid pentane fell into the pentane reagent tank. In order for the pentane to evaporate and condense properly, a pump was used to pump the displaced gas from the reagent tank into the evaporation beaker; otherwise, the pentane would not condense.

## **4.2 Experimental Methods: Continuous Testing Unit**

Separate experiments were performed using emulsions and agglomerates as the feed material. Emulsions were formed by pumping coal slurry from the feed sump and pentane from the reagent tank into the mixer. The mixer was operated at high speed to form the emulsions. The level within the phase separator was controlled by hand using the overflow assembly discussed in Chapter 3. Once the sample in the evaporator was completely dry (no more pentane was condensing and falling into the reagent tank), the coal was removed and weighed for moisture analysis.

Through experimentation, it was discovered that the sealed mixer built into the continuous reactor was not capable of producing a high enough shear force to create the agglomerates. Therefore, the agglomerates were created outside of the system in a kitchen blender. Originally, it was intended to create the agglomerates via high shear mixing in the blender, then pump the agglomerates into the built-in mixer for low shear mixing. Once the pumps were turned on, it was discovered that the agglomerates collected at the mouth of the pumps inlet tube, creating a filter, so that only ash and water passed. Therefore, it was necessary to form the agglomerates in the blender, and pour the agglomerates into the mixer built-in to the reactor, creating a semi continuous process as opposed to a true continuous experiment. The pentane feed line from the reagent tank was rerouted so that it fed into the phase separator instead of the mixer. As with the emulsion method, the pentane level within the separator was controlled using the overflow system. Once the sample had completed drying, the moisture content and recovery rate was then calculated.

## **4.3 Results: Continuous Testing Unit**

Breaking emulsions in the continuous reactor was unsuccessful. The rate of breakage in the phase separator was unable to keep up with the flow rate of material entering from the mixer. After the flow rates of the feed were decreased, the phase separator was still extremely slow at breaking the emulsions. The layer of pentane was nearly clear. At times, the feed was completely turned off; however, in nearly all of the experiments performed, the phase separator completely filled with emulsions. Eventually, the emulsions began to exit the separator through the overflow

**Table 4-1: Agglomerate results from ultrasonic continuous reactor.**

Sample	Feed Ash %	Reject Ash %	Product		Combustible Recovery %	Separation Efficiency %	Ash Rejection %
			Ash %	Moisture %			
Bailey Screen Bowl Effluent	40.40	80.10	3.30	3.80	83.90	79.64	95.78
Bailey Screen Bowl Effluent	40.40	81.40	5.00	5.50	85.50	78.90	93.36
Bailey Screen Bowl Effluent	40.35	83.23	5.46	2.99	87.39	79.92	92.54
Bailey Screen Bowl Effluent	40.35	80.10	4.97	5.51	84.29	77.77	93.48
Kingston Flotation Feed	50.70	92.00	3.60	2.90	91.40	88.08	96.68

system, into the tailings. Of the samples that were collected, there was not enough weight to accurately measure the moisture content or recovery of the sample.

The reactor was very successful in creating dry product from the agglomerate feed. Table 4-1 shows the results obtained using flotation feed from CONSOL’s Bailey preparation plant as feed. Due to limited sample, one test was performed using flotation feed from Alpha Natural Resource’s Kingston preparation plant. The results from this single test are also included in Table 4-1.

For the Bailey samples, the continuous experiments produced slightly higher moisture values than in the batch tests. The average moisture content was 5.5%. Recovery and separation efficiency values were also slightly lower than the batch tests, with an average recovery value of 85.3% and an average separation efficiency of 79.06%.

#### 4.4 Discussion: Continuous Testing Unit

The continuous reactor offered promising results for removing the moisture from the coal agglomerates. Even when running the reactor with the agglomerate feed, emulsions still formed at the pentane/water interface. The formation of emulsions was faster than the rate of breakage and eventually, the column filled with emulsions.

The recovery was slightly lower than in the batch tests; however, a dewatering screen was not utilized to separate the ash from the agglomerates. The addition of a dewatering screen along with a recycle stream to recover any coal passing through the screen could help improve the recovery. The lower reject ash is likely responsible for the lower recovery and it is expected that the value was decreased due to coal emulsions exiting to the tailings through the column's overflow system. If a dewatering screen had been incorporated, the tailings from the column could then be recycled with the skimmed layer of coal taken off the top of the tailings which passed through the dewatering screen. This process would prevent coal leaving the reactor from reporting to the tailings.

In Table 3-14 and Table 3-15, where agglomerates were being broken by the ultrasonic probe and vibrating mesh, average ash values for the tailings were 86.4% and 87.4%, respectively. In the continuous testing unit, the average ash value for the Bailey sample was 81.2%. If the separation efficiency could be improved so that the tailings were comprised of 86% ash, the average combustible recovery would be increased to 89.7%, competitive to froth flotation circuits.

While the average moisture content was slightly higher than that obtained in the batch tests, it is believed that some modifications to the system could help decrease the moisture content. Originally feed tubes going into the pentane phase were included on the ultrasonic separator. However, the diameter of the tubes were too small for agglomerates to flow through without plugging; thus, when the tubes were removed. While the mixer was feeding the ultrasonic separator, some of the water entering the separator ran down the walls of the vessel. A small amount of the water flowed into the concentrate overflow spout. Enlarging the feed tubes to allow for agglomerates to flow without plugging will help decrease the moisture content in the product.

## Chapter 5: Conclusions and Recommendations

Breaking coal agglomerates with the ultrasonic probe and vibrating mesh yielded very similar results, especially when testing the Bailey sample. Figure 5-1 and Figure 5-2, given on pages 63 and 64, respectively, show the separation efficiencies for all of the tests conducted in the present work. The black diagonal lines on the plot represent the different areas of separation efficiencies. Breaking agglomerates formed with the Kingston flotation feed was the only method to achieve a separation efficiency of over 90%.

All of the final products obtained using the vibrating mesh plates to break agglomerates show separation efficiencies near 85%. Additionally, the Bailey agglomerates broken by the ultrasonic probe also show separation efficiencies near 85%. The average product moistures obtained using both breakage methods are approximately the same (2.05% and 3.63% average moisture for Bailey and Kingston agglomerates broken with mesh, respectively, versus 2.80% average moisture for Bailey agglomerates broken with the probe). The recovery values for the Bailey samples are also similar: 89.4% with the ultrasonic probe, and 88.9% with the vibrating plates.

Therefore, it is determined that the vibrating mesh plates provide the best method for breaking agglomerates and thus cleaning and dewatering fine coal. Since the results obtained with both methods are about the same, the decision as which dispersion method is best may be based on operational issues. The ultrasonic probe has four main drawbacks: overheating, emulsion formation at the interface, limited throughput, and higher energy consumption. These shortcomings can be overcome by using the vibrating mesh plates.

It is recommended that agglomerate dispersion by the vibrating mesh plates be further explored. It is believed that the separation efficiency of the Kingston agglomerates broken with the vibrating mesh was lower, in part, due to an older sample which had been slightly oxidized. Additional tests should be performed on this sample to determine exactly why the separation efficiency was nearly 10% lower than when using the ultrasonic probe to break the agglomerates. Additional sensitivity analyses should be performed to determine how certain parameters such as plate vibration frequency, plate position in regards to the pentane/water interface, agglomerate feed size and moisture, and column feed rate effect the final product. These findings could then be used to optimize the separator's performance.

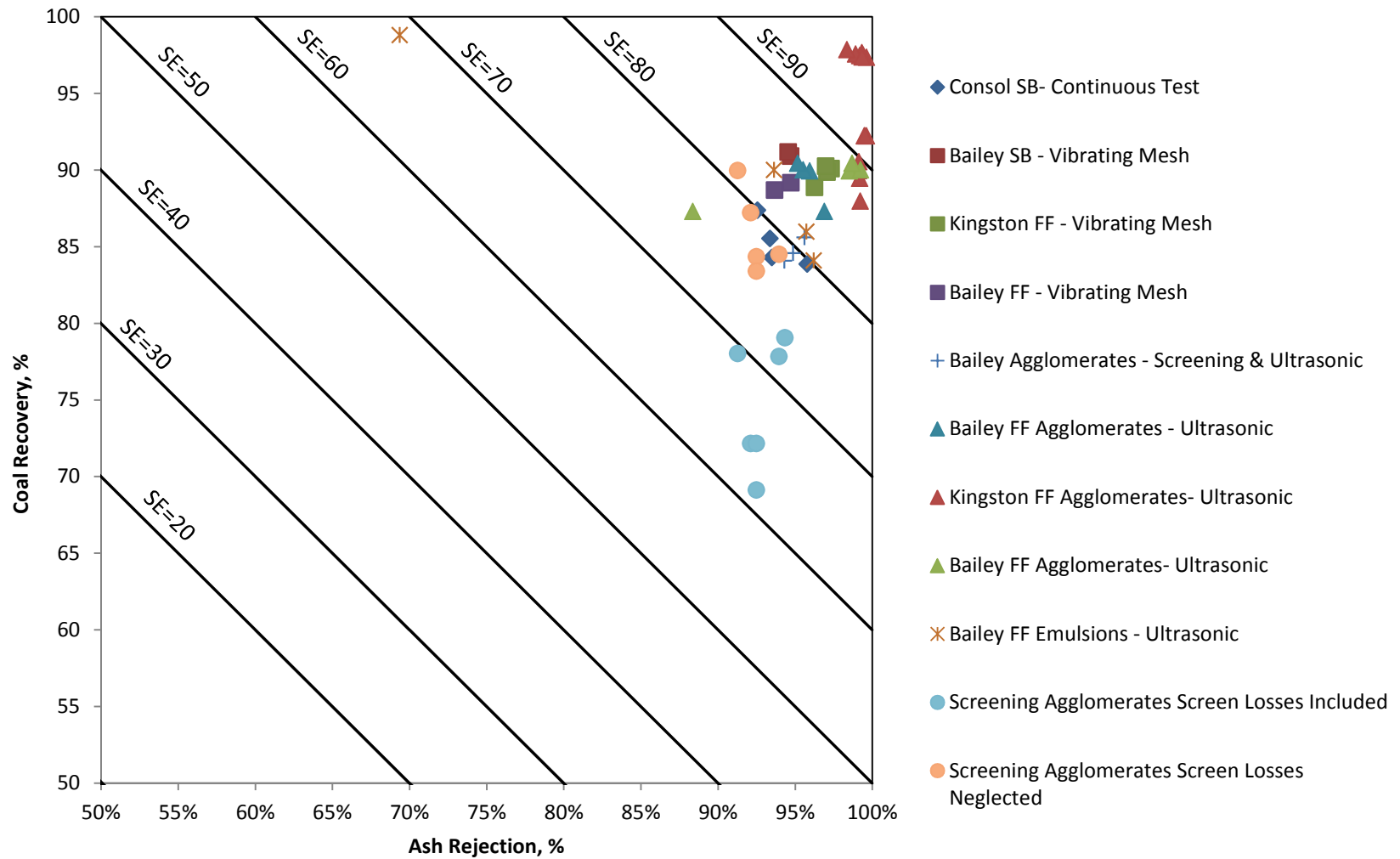


Figure 5-1: Plot showing the separation efficiency of all testing methods attempted. Please note: “FF” stands for “flotation feed” and “SB” stands for “screen bowl effluent.”



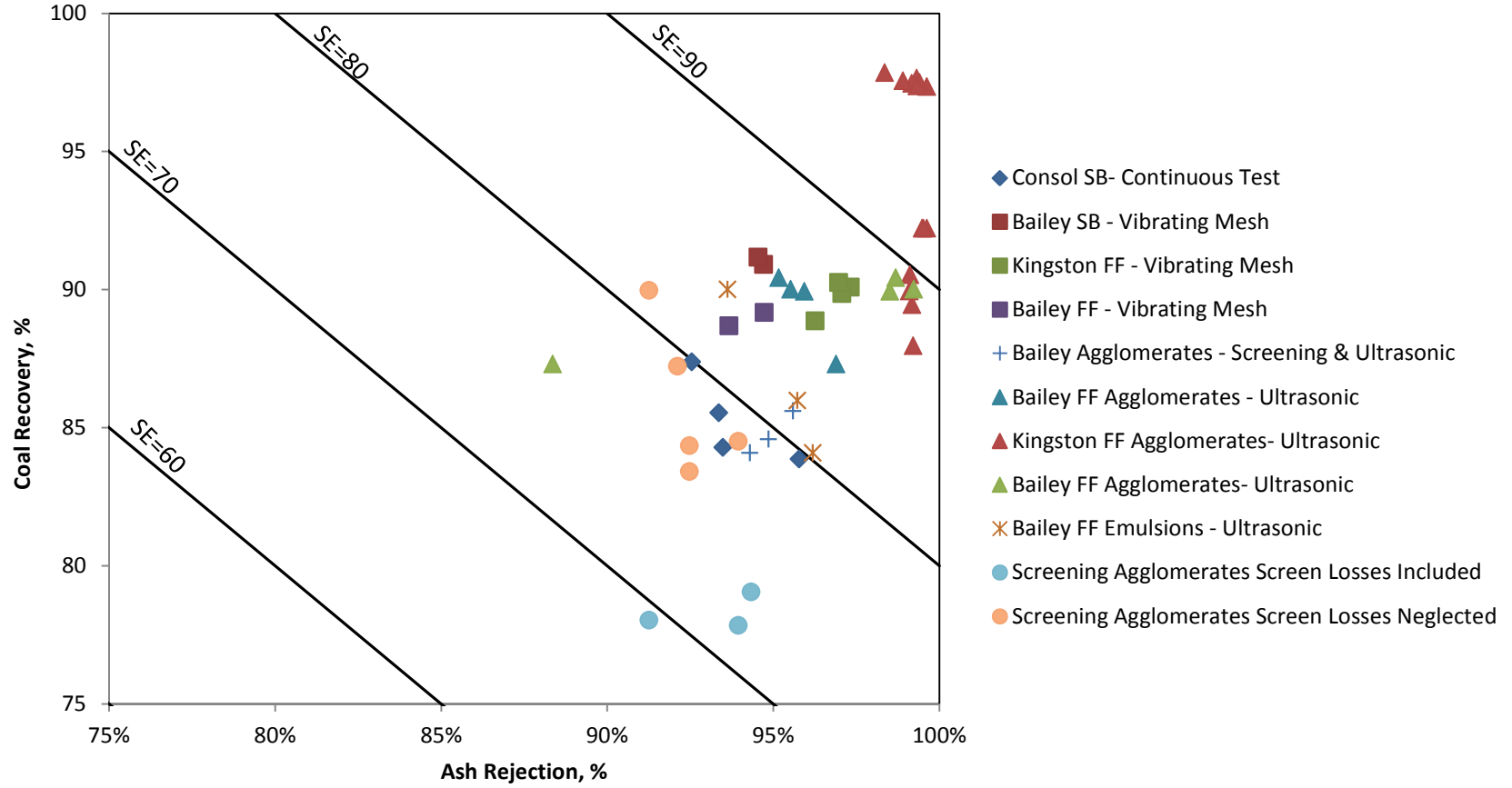


Figure 5-2: Zoomed in plot showing the separation efficiency of all testing methods attempted. Please note: “FF” stands for “flotation feed” and “SB” stands for “screen bowl effluent.”

Additionally, it is recommended that the vibrating mesh separator be integrated into the continuous circuit in place of the ultrasonic separator. One of the largest obstacles encountered when using the continuous circuit with the ultrasonic separator in its current configuration was that the formation and buildup of emulsions in the separator prevented the unit from being run for extended time periods. Since no emulsion build up has been observed when using the vibrating mesh plates, it would be beneficial to replace the ultrasonic separator to evaluate performance during long term, continuous operation.

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