

CLEANING AND DEWATERING FINE COAL USING HYDROPHOBIC DISPLACEMENT

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ABSTRACT

A new processing technique, known as hydrophobic displacement, was explored as a means of simultaneously removing both mineral matter and surface moisture from coal in a single process. Previous thermodynamic analysis suggests that coal moisture will be spontaneously displaced by any oil with a contact angle greater than ninety degrees in water. Based on these results, six methods of hydrophobic displacement were evaluated: hand shaking, screening, air classification, centrifugation, filtration, and displacement. In the first five methods hydrophobic displacement took place during the cleaning stage. A recyclable non-polar liquid (i.e. pentane) was used to agglomerate coal fines followed by a physical separation step to remove the coal agglomerates from the mineral-laden slurry. Bench-scale tests were performed to identify the conditions required to create stable agglomerates. Only the last method, displacement, did not utilize agglomeration and performed hydrophobic displacement during dewatering, not cleaning. A procedure was also developed for determining moisture content from evaporation curves so that the contents of water and pentane remaining in a sample could be accurately distinguished.

Two primary coal samples were evaluated in the test program, i.e., dry pulverized 80 mesh x 0 clean coal and 100 mesh x 0 flotation feed. These samples were further screened or aged (oxidized) to provide additional test samples. The lowest moisture, 7.5%, was achieved with centrifugation of the pulverized 80 mesh x 0 clean coal sample. Centrifugation provided the most reliable separation method since it consistently produced low moisture, high combustible recoveries, and high ash rejections. Hand shaking produced the next lowest moisture at 16.2%; however, the low moistures were associated with a drop in combustible recovery. There was also a great deal of error in this process due to its arbitrary nature. Factors such as oxidation, size distribution, and contact angle hysteresis influenced the concentrate moistures, regardless of the method utilized.

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1. INTRODUCTION

1.1. Preamble

Cleaning and dewatering of fine coal is currently one of the coal mining industry's greatest challenges. The United States' dependence on coal for 52% of its electricity in 2006 mandates that coal be utilized as efficiently as possible (EIA, 2007). Though 92% of coal in the United States is used for the generation of electricity, the remaining percentage is used for a variety of purposes including heating and coke for steel blast furnaces (EIA, 2007). As an integral component to many industries, it is vital to the country's economy that a steady, low-cost supply of coal is maintained. Many industries would be hard pressed to come up with an economic substitute for coal.

As easily accessible, high quality coal reserves are depleted, the mining industry needs a low cost solution which will allow it to utilize more of the fines generated during processing and to recover fine coal stored in refuse impoundments (Hazra, 1988). The high water content of ultrafine coal often makes it uneconomical to sell due to the associated contract penalties, and this coal contributes to the 70-90 million tons of fine wastes produced each year (Orr, 2002). In 2002, there was already more than 2.5 billion tons of fine coal wastes discarded into impoundments.

Conventionally, fine coal undergoes separate cleaning and dewatering phases. Water-based density separators such as spirals and/or water-only cyclones are used to treat small particles in the 1 mm x 100 mesh size range, while froth flotation is used to clean minus 100 mesh coal. The water-rich products from these cleaning processes are dried using centrifugation, filtration, or thermal drying. Unfortunately, these methods become increasingly expensive as the material becomes finer.

Screen-bowl centrifuges, which are commonly used to treat 1 mm x 0 coals, are the most popular fine coal dewatering method used in the United States. The amount of minus 325 mesh material in the feed controls the final product moisture. For example, only 30% ultrafines results in a surface moisture around 18% (Osborne 1988). Also, screen-bowl centrifuges cannot achieve high coal recoveries since they lose nearly half of the ultrafines present in the feed as an effluent stream.

Vacuum filtration, which can achieve nearly complete recoveries of coal solids, usually results in a product with 20-35% moisture. This value rises as the amount of ultrafines (minus 325 mesh material) increases, resulting in higher water penalties. The fines also filter more slowly and require more power. Unfortunately, the higher capacity, lower cost filtration units, such as disc filters, often produce higher moisture products than lower capacity, higher cost filtration processes (Wills, 1997). As a result, the popularity of vacuum filters has declined dramatically in the United States where high moisture values cannot be tolerated due to strict contract specification and difficulties associated with handling and freezing of damp coal.

Thermal drying can produce single digit moisture without the same size restrictions as centrifugation and filtration. However, this process is expensive and increasingly harder to permit (Osborne, 1988). As a result of the difficulties associated with dewatering ultrafines, the minus 325 mesh stream is often discarded in coal plants even though it contains the most well-liberated material. This not only wastes valuable coal, but also creates potential environmental problems associated with the disposal of fine coal wastes.

1.2. Objectives

This project seeks to replace conventional coal cleaning and dewatering technologies with a single solid-solid and solid-liquid separation process which results in products with less than 10% moisture. The basis for this process is displacement of water and hydrophilic material by a hydrophobic liquid. For this study, pentane was selected as the hydrophobic liquid since it was affordable, met the thermodynamic requirements (i.e. a contact angle on coal greater than ninety degrees in water), and could be easily recycled via evaporation and condensation. Though the volatility of pentane necessitates a more complicated, closed system, it makes recovery of the oil less energy intensive and less expensive.

Six cleaning-dewatering processes were evaluated: hand shaking, screening, air classification, centrifugation, filtration, and displacement. Most of the tests revolved around oil agglomeration with pentane in which hydrophobic displacement took place during the cleaning stage; the dewatering stage then consisted of physical separation of oil-coated agglomerates and free water droplets. Only the displacement process utilized hydrophobic displacement during the dewatering stage in which oil sought to strip moisture from fine coal's surface. This project consists of completing the bench-scale and batch testing and evaluating the best method for

continuous operation. The project also includes an investigation into how to accurately measure moisture due only to water, the effects of oil dosage, and the optimum dosage for the chosen dewatering method.

1.3. Organization

This thesis is divided into seven major sections. The proceeding introductory section seeks to explain the need for improvement in fine coal processing and how this project will attempt to fill the gap in technology.

The second literary review section summarizes the current states of technology. This section contains four subsections: conventional fine coal cleaning, conventional fine coal dewatering, oil agglomeration, and hydrophobic displacement. The cleaning section includes information on froth flotation, the current industry practice for fine coal recovery. The dewatering section covers drying through centrifugation, filtration, and thermal drying. The oil agglomeration section reviews the history, theory, and practice of coal agglomeration and also touches on previous testing with pentane, the chosen oil for this project. The hydrophobic displacement section reviews the precursor to this project at Virginia Tech.

The third, experimental section covers the samples, apparatus, and procedures used in this study. In particular, this section covers water content determination, agglomerate formation, and removal of agglomerates in both bench-scale and batch testing.

The fourth and fifth sections contain the experimental results and subsequent discussion of them. These sections focus heavily on the feasibility of the different methods and the results of the final batch centrifuge testing.

The sixth section contains a brief summary of the project, while section seven provides recommendations for future testing of this process.

2. LITERATURE REVIEW

The literature review is split into four subsections: conventional fine coal cleaning, conventional fine coal dewatering, oil agglomeration, and hydrophobic displacement. The first two sections are meant to provide a brief overview of the currently accepted practices. However, the main focus of this literature review is oil agglomeration since it is the basis for much the reported work. The final section reviews this project's precursor at Virginia Tech. Though it did not utilize pentane, it contained a thermodynamic analysis of the hydrophobic displacement process.

2.1. Conventional Fine Coal Cleaning

Currently, froth flotation is the only commercially practiced method for cleaning fine coal in the United States. This section will not cover other novel cleaning methods. It should be noted that oil agglomeration is also an extensively studied method for cleaning coal; however, due to its combined cleaning and dewatering capabilities and importance to this project, it will be reviewed in a separate section of this document.

2.1.1. Froth Flotation

Froth flotation (Figure 1-1) is currently the preferred method for cleaning minus 100 mesh coal. It is based on the differential wettability of particles; this surface-based process distinguishes between hydrophobic coal and hydrophilic clays. Air bubbles passing through a coal slurry selectively attach to coal particles, carrying them to the surface froth phase, while hydrophilic tailings remain in the water or pulp phase. The froth phase is then removed, effectively separating the coal and impurities.

Flotation is controlled by chemical, operational, and design variables. Chemical variables include coal rank, pulp chemistry, surface oxidation, and reagent dosage. Operational variables include particle size, feed rate, pulp density, pulp level, froth height, impeller speed, aeration rate, and conditioning time. Design variables are based on the type of cell and configuration.

Since flotation is a surface-based process, the ability to control surface chemistry is essential. This is done through reagents known as collectors, depressants, activators, pH modifiers, and frothers. Though complex ore flotation applications may utilize all of these reagents, coal flotation is simpler and often only requires a collector and frother. A collector is a chemical that adsorbs on coal rendering it more hydrophobic in order to facilitate bubble attachment (Wills, 1997). A frother is a surfactant which helps to stabilize a froth and prevent bubble breakage once the loaded bubble reaches the surface (Wills, 1997). Without a frother, bubbles would break or coalesce and release coal back into the pulp phase, preventing separation.



Figure 1-1. Conventional flotation bank used to clean ultrafine coal.

Next to surface chemistry, particle and bubble size are two of the most important variables. Flotation works best for fine particles about 0.1-0.25 mm in diameter. Larger particles have a high probability of bubble-particle detachment, while smaller ones have a low probability of bubble-particle collision. While particle size determines which particles are most likely to float, bubble size controls the amount of particles that are able to float. The total surface area of the bubbles determines the carrying capacity of the froth. If there is no free area on a bubble for a coal particle to attach to, it will be misplaced to the tailings. Since surface area can be drastically increased by decreasing the size of a bubble, modern flotation equipment are typically designed to produce small bubbles to maximize flotation kinetics and the carrying capacity of the air volume.

Though flotation is a selective process, not all of the material reporting to the concentrate is coal. Three mechanisms contribute to the concentrate: attachment, entrapment, and entrainment. Attachment refers to selective bubble-particle attachment. These particles may be coal or a combination of unliberated coal and ash. Attachment is the only mechanism which selectively contributes to the desired components of a concentrate. Entrapment, the most rare of

the mechanisms, refers to small particles that are trapped between agglomerated, coarser particles or between coarser particles and a bubble. Entrainment occurs due to hydraulic transport of ultrafine material. Water is required to form the liquid films in the froth, and free floating, ultrafine material may exist in these channels.

There are two major types of froth flotation cells: mechanical and column. A mechanical or conventional cell produces bubbles through mechanical agitation. The froth layer is relatively short and is scrapped off by paddles. Due to the large water recovery and possibility for entrainment, large banks of cells are usually setup with multiple stages of flotation (Osborne, 1988). Column cells produce bubbles through spargers and allow bubbles to rise through tall quiescent tanks. Froths are considerably deeper in column cells, so wash water may be used to remove entrained material from the froth (Osborne, 1988). Both types of cells are currently in use in the United States.

2.2. Conventional Fine Coal Dewatering

Three methods of drying will be reviewed: screen-bowl centrifugation, vacuum filtration, and thermal drying. Though screen-bowls are reviewed and able to handle some ultrafines, they are usually reserved for coarser feeds than those studied in this project. Vacuum filtration is the most common method for dewatering ultrafines and is the mostly likely candidate for treating the types of feed size distributions used in the project. Finally, though thermal drying produces the driest product, it is the least used of the three methods due to problems with expense and permitting.

2.2.1. Screen-Bowl Centrifugation

Centrifuges combine centrifugal sedimentation and filtration. High g-forces cause solids to settle quickly into a compact cake and force water out through the pores (Osborne, 1988). Screen-bowl centrifuges (Figure 1-2) consist of a horizontal tube with a screw inside to

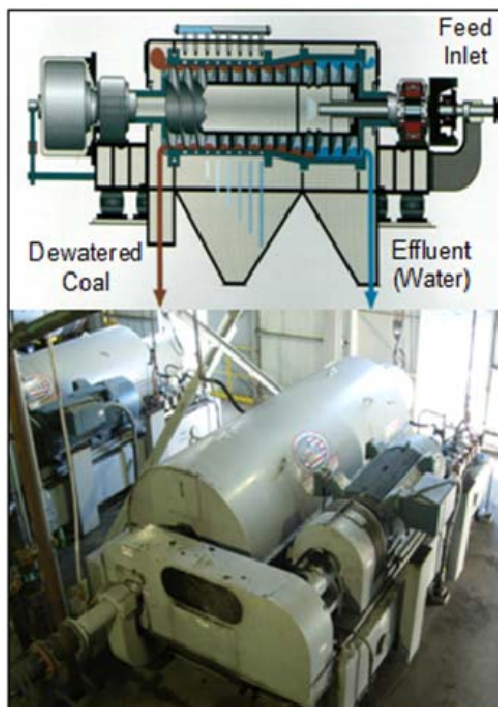


Figure 1-2. Screen-bowl centrifuge used to dewater fine coal.

move the material. The first section of the tube is solid and removes the bulk of the water. As the feed comes into this section, it quickly forms a cake while the majority of the liquid and about half of the minus 325 mesh material flow over the adjustable weirs in the back of the machine. The screw pulls the material up a small ramp to the front section of the machine which consists of a screen for further dewatering. A screen-bowl centrifuge is a hybrid centrifuge; the solid bowl section enables the machine to handle large volumes of water with a high solids recovery while the screen basket section allows drainage aided by centrifugal force (Osborne, 1988).

These centrifuges are high capacity, long life machines that can provide low moistures. The final moisture is directly related to the amount of minus 325 mesh feed material. For example, if a feed contains 30% minus 325 mesh, the product's moisture will be around 18% (Osborne, 1988). It should also be noted that some of this ultrafine material is discarded with the main effluent. Typically this effluent is not recycled, and any material in it is lost to the tailings. Final product moisture is also dependent on the centrifugal force. A higher operating speed will lead to lower moisture and a finer cut; however, screen-bowl centrifuges are generally not operated above 500g due to excessive wear. Due to the strong dependence of product moisture on feed size and limited centrifugal force, screen-bowl centrifuges are generally used for dewatering fine material coming off of spirals.

2.2.2. Vacuum Filtration

Vacuum filtration (Figure 1-3) is the most effective method for dewatering fine coal containing a large proportion of minus 325 mesh solids. Disc filers, the most common type in the United States, consist of vertical discs with fan-shaped sectors covered in fine cloth or mesh. The hollow discs are under vacuum and submerged about half way in slurry. As the discs rotate, they pick up solids from the slurry, the cake dries as it is carried into the air, and then the dried cake is



Figure 1-3. Disc vacuum filter used to dewater fine coal.

blown off before the fan segment is again dipped into the slurry (Osborne, 1988). Fine solids are trapped in the cake against the filter cloth, and recoveries are usually greater than 97%. They typically produce moistures in the 25-35% range, and reagents may be needed to reach the lower moistures. Flocculants are usually added to reduce screen blinding, reduce ultrafine losses, and aid in cake release, while cationic coagulants are occasionally used to increase the filtration rate.

Disc filters are popular in the United States due to their small footprints, high capacities, and low cost; however, they produce higher moistures and require more maintenance compared to some other filters. The vertical nature of disc filters also prevents cake washing (Wills, 1997). Other continuous vacuum filters include rotary drums and horizontal belt filters. Filtration may also be done by applying positive pressure instead of a vacuum; however, these filters are more expensive and are used rarely in the coal industry for dewatering clean coal products.

2.2.3. Thermal Drying

Thermal drying (Figure 1-4) is not common in the United States. It is the most expensive unit operation in coal preparation (Osborne, 1988), and it is extremely difficult to permit new units. They are generally only used on ultrafine coals whose large surface areas lead to high moisture contents. Thermal dryers are the only unit that can consistently provide single digit moisture with ultrafine feed. This low moisture may be worth the cost to reduce the possibility of freezing, to reduce heat loss during combustion, and to prepare the coal for coke making among other reasons (Osborne, 1988). Industrial coal dryers usually employ convection in direct heat-exchange type dryers in which wet coal is continuously brought into contact with hot gases in order to evaporate surface moisture (Osborne, 1988).



Figure 1-4. Thermal dryer used to dry coal to low moisture contents.

2.3. Oil Agglomeration

2.3.1. History of Oil Agglomeration

Oil agglomeration was first performed on coal in the early 1920's (Mehrotra et al., 1983); however, it was not until the 1970's energy crisis that the United States invested significant amounts of time and money into the potential uses of oil agglomeration. The sharp increase in oil prices spurred the need for an alternative source of energy to run equipment such as turbines and diesel engines. It was discovered that the fine particulates in coal slurry were not problematic, but the residue due to ash was unacceptable. Oil agglomeration was investigated as a method to produce the ultraclean coal needed. Though most of the testing during the 1980's focused on the cleaning ability of oil agglomeration, dewatering and oil recovery were also explored. Several pilot plants were even created to test the feasibility of continuous, larger scale processing (Mehrotra et al., 1983). The inherently expensive process could not compete with the falling oil prices in the late 1980's, and oil agglomeration was largely abandoned.

Much of the prohibitive cost associated with oil agglomeration is due to the need to finely grind the feed, sometimes as fine as a few microns, for ultracleaning (Nguyen et al., 1983). The price and consumption of refined oil are also major disadvantages. The finer the feed, the more surface area is created, increasing the oil consumed. Complete recovery of the oil is often impossible or prohibitively expensive. For these reasons, commercial oil agglomeration with coal is not currently practiced in the United States.

2.3.2. Theory of Oil Agglomeration

Oil agglomeration is based on the ability of an oil to preferentially wet hydrophobic or oleophilic surfaces. This selectivity enables an oil to coat the hydrophobic sites of coal particles, while rejecting hydrophilic material such as clays and pyrite (Good et al., 1991). The hydrophilic particles remain in an aqueous suspension, while the hydrophobic coal particles combine into agglomerates to minimize the surface in contact with water.

Despite the fact that oil agglomeration has been studied extensively, the microscopic interactions are still not well understood. Coal is not homogenous and consists of a patchwork of hydrophilic and hydrophobic sites (Keller et al., 1987); therefore, several conflicting theories exist on which liquid, oil or water, acts as the bridging mechanism to form the agglomerates. The

first popular theory is that oil acts as a liquid bridge between coal particles (Keller et al., 1987). The oil envelopes the coal and bridges over the hydrophilic sites. Though small droplets of water may remain bound to the hydrophilic sites, oil displaces the water from the hydrophobic sites and remains the dominant liquid in the agglomerates. As two oil coated particles collide during mixing, the oil and capillary attraction of the oil causes the particles to stick together and eventually form agglomerates.

The second opposing theory is that water actually acts as the bridging liquid. Many oils simply spread on hydrophobic coal surfaces. In contrast, when surrounded by oil water sticking to the hydrophilic sites forms water droplets with contact angles greater than 90 degrees (Good et al., 1991). When two of these droplets meet, they form a bridge and the surface tension of the water pulls the coal particles together. The more the particles are pulled apart, the more the surface tension increases and forces the particles back together. In contrast, hydrophobic liquids will break apart into two droplets when the bridge is stretched (Good et al., 1991). Oil simply coats the particles and provides an environment for the water bridges. Finally, there is little discussion on whether these theories are mutually exclusive or may both contribute to agglomerate formation.

The location of agglomerate water is dependent on which theory of bridging liquids is ascribed to. If oil is considered to be the bridging liquid, then only minor amounts of water will be trapped in the agglomerates at the hydrophilic sites. If water is considered to be the bridging liquid, then there is an inherent amount of water needed for an agglomerate to keep its shape. In both cases, free water droplets may be located in the voids and pores between agglomerates. As more oil is added, these water droplets may be completely enclosed and trapped.

The macroscopic interactions of oil agglomeration are better understood than interactions within agglomerates, and most of the papers and patents associated with oil agglomeration are written at this level. The appearances of the agglomerates vary greatly with changes in the operating parameters. These operating parameters include oil type, oil dosage, mixing time, mixing speed, coal surface oxidation, particle size, and solids content (Boni et al., 1994; Hazra et al., 1986).

The type of oil being used makes a significant impact on the speed and selectivity of agglomeration. Oils are commonly divided into light and heavy oils. Heavy oils are less selective and used with lignite and other low-rank coals. Lighter low viscosity paraffinic oils are more

selective and are used with higher rank coals such as anthracite and bituminous coal (Capes, 1991). The type of oil also affects whether it will be recoverable and whether a binder may need to be added for dewatering. Oil dosage varied widely in previous testing and has a large effect on the appearance of the agglomerates. Lower dosages create small, compact, rounded agglomerates while higher dosages tend to result in large, soft, irregular curds (Osborne, 1988). Enough oil is needed to cover the coal and displace water from its surface, but too much oil may actually trap water globules between the curds. Mixing time and speed also vary greatly in oil agglomeration. High shear mixers are often used under the theory that more energy provides more opportunity for the oil to come into contact with the coal (Keller et al., 1987). The high shear reworks and cleans the agglomerates. However, lower shear mixing may also be used to prevent the agglomerates from being ripped apart.

Coal surface oxidation affects the ability of oil to coat the coal. Oxidation makes coal more hydrophilic and may prevent oil from binding to its surface (Drzymala et al., 1994). The more oxidized sites, the more water will be incorporated into the agglomerate. Particle size affects the dosage of oil needed. As particles become smaller, the surface area increases rapidly. It is the surface of the coal that determines how much oil will be needed to coat the particles (Osborne, 1988). Particle size also influences the amount of water in the agglomerates. Smaller particles will be more liberated and may have less hydrophilic sites for water. Conversely, if the coal is oxidized, the increase in surface area will lead to an increase in trapped water and perhaps suspended hydrophilic material. There is not a single optimum set of operating conditions. The amount of interdependent operating parameters means several sets of conditions can produce similar results and may be customized for the particular coal being used.

2.3.3. Practice of Oil Agglomeration

There are several different aspects of oil agglomeration that have been the focus of research including cleaning, dewatering, and oil recovery. The ability of oil agglomeration to clean a coal has been the dominant and original focus of research in the United States. Oil agglomeration cleans a coal by agglomerating only hydrophobic coal and leaving hydrophilic waste in suspension. Cleaning often involves finely grinding the coal to as little as five microns in order to liberate all of the clays, pyrite, and other hydrophilic impurities (Mehrotra et al., 1983). A typical process uses higher dosages of oil and high shear mixing to ensure that all of

hydrophilic material is expelled (Mehrotra et al., 1983). The curds are then skimmed from the surface or rinsed on a screen. The Otisca-T Process is one of the most well-known patents in this particular area.

The dewatering aspect of oil agglomeration has not received as much attention in research. Dewatering assumes that water is not the bridging liquid and may be expelled from the interior of the agglomerates if enough oil is present. Only minor amounts of water remain at the hydrophilic sites; the majority of the water is located in the pores between the individual agglomerates. This water may be removed with conventional dewatering equipment including centrifuges and vibratory screens (Mehrotra et al., 1983). Binders may be added to strengthen the agglomerates before they are dewatered. Research by the National Research Council of Canada resulted in some of the lowest moistures ranging from 3-12%. They focused on spherical agglomeration to simplify dewatering. Micro-agglomerates, or flocs, were dewatered on a screen before being pelletized with heavy, cheap oil binders (Mehrotra et al., 1983).

Oil recovery is a popular research topic for oil agglomeration. Oil agglomeration consumes large amounts of oil, and this may be prohibitively expensive. Some processes do not attempt to recover the oil. Instead, the oil is allowed to burn with the concentrate, contributing to the overall heating value of the coal product. There are even multi-stage oil agglomeration processes in which a light refined oil and a heavier binder are utilized together (Mehrotra et al., 1983). While processes such as these make dewatering and cleaning easier, their expensive nature prompted research into oil recovery through heating and condensation. Many papers in the late 1980's were based on the economics of oil agglomeration, a topic closely related to the loss and cost of oil (Mehrotra et al., 1983).

2.3.4. Previous Testing with Pentane

Pentane is not a widely used hydrocarbon in oil agglomeration. Most labs, including the Convertol Lab, NRCC Lab, Shell Lab, and BHP Lab, tended to focus on cheaper oils including diesel, kerosene, fish oil, and fuel oil. Although lighter oils are more selective, heavier oils were utilized in order to minimize loss due to evaporation (Mehrotra et al., 1983). One of the most popular processes utilizing pentane is the Otisca-T process. This process focused on coal beneficiation, not dewatering. The ultrafine feed was agglomerated using large amounts of pentane or heptane in order to ensure coal particles were coated in oil and hydrophilic material

was rejected. When using finely micronized coals, the resulting curd-like agglomerates formed by this process often contained less than 1% ash with a combustible recovery greater than 95% (Keller et al., 1990).

2.4. Hydrophobic Displacement

Studies in hydrophobic displacement were initiated at Virginia Tech in 1995 and included thermodynamic analysis and batch testing with butane. The thermodynamic analysis compared a beginning state of coal (1) in water (3) and an end state of coal in a hydrophobic liquid (2). Application of Young's equation yielded the following condition for spontaneous dewatering (Sohn et al., 1997):

$$dG / dA = \gamma_{23} \cos \theta < 0 \quad [1]$$

In other words, a hydrophobic liquid will displace water from coal when its contact angle θ is greater than 90 degrees (Figure 2-1.). In response the contact angles for several hydrocarbon liquids (C4-C10) were measured or calculated. The contact angles increased as the carbon number decreased. Liquefied butane (C4) had the greatest contact angle at 110°. Pentane had the next highest at 106° (Sohn et al, 1997).

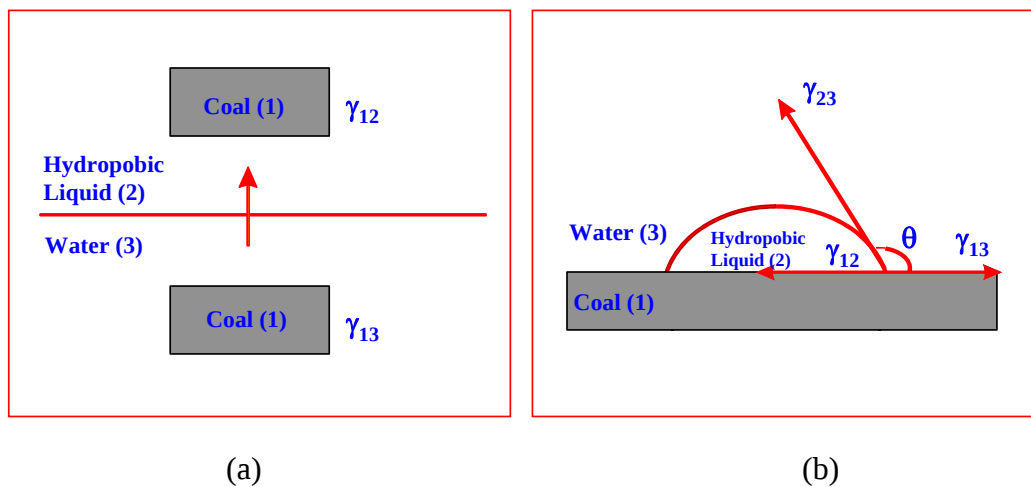


Figure 2-1. a) A schematic representation of the removal of a coal particle from an aqueous phase to a hydrophobic liquid phase with a change in free energy from γ_{13} to γ_{12} . b) The angle θ represents the equilibrium hydrophobic liquid-in-water contact angle (Sohn et al., 1997).

Batch testing was performed with liquefied butane due to its large contact angle and ease of recovery. When clean coal slurry was gently agitated with large amounts of butane in a pressurized vessel, the resulting 'dry' coal powder gathered on top of the water phase. The concentrate (i.e. 2 grams) was removed from the top of the powder, and the initial weight for the moisture was taken after the sample sat at room temperature for 90 minutes. Testing indicated this was the approximate amount of time needed for butane to evaporate. The best moisture, 1%, occurred with a butane-to-coal mass ratio of 2.0, a solid content of 5%, and a settling time of 10 minutes (Sohn et al., 1997). Initial testing indicated that butane recovery would be high due to ease of evaporation and minor loss of butane to water.

3. EXPERIMENTAL

3.1. Coal Samples

The majority of the tests were performed on coal from a single preparation plant in order to maintain consistency for comparison. The minus 100 mesh coal sample was collected from the flotation feed at Alpha Natural Resources' Tom's Creek Preparation Plant in Coeburn, Virginia. The Tom's Creek plant processes coal from two mines operating in the Lower Banner and Dorchester seams. The feed slurry contained 6.0-7.1% solids with an average ash of 38%. The slurry contained about 72% minus 325 mesh, and it was not deslimed at the plant site before flotation due to its ease of cleaning. The slurry contained minor amounts of frother since it was obtained from the cyclone overflow sump where frother was first introduced before flotation. Most of the samples did not undergo further processing before being utilized. In order to vary the solids content for testing, the buckets of slurry were allowed to sit until the solids settled and water could be decanted. Chemicals such as flocculants or coagulants were not added to speed settling because they would interfere with the oil agglomeration process. Three solid contents were used for the extraction by shaking tests, i.e., the original solids content, 15%, and 30%. In order to prevent oxidation, bucket samples were used within a few weeks. Samples that were not used immediately were stored in sealed metal drums.

Two other samples were prepared from the original minus 100 mesh Tom's Creek flotation feed. One bucket was wet screened at 325 mesh, and the 325 mesh x 0 product was used in order to evaluate the dewatering ability of oil agglomeration on slime. It was only used for the extraction by shaking method. The second sample was used only for dewatering by centrifugation. Though it remained a 100 mesh x 0 sample, it only contained about 28% minus 325 mesh material. The sample was created by screening the feed to remove all of the minus 325 mesh ultrafines. Some of the original unscreened feed was then mixed back into the new, screened agglomeration feed. The final feed sample was about 6.1% solids.

Since previous hydrophobic displacement testing at Virginia Tech was performed on dry coal, a dry coal sample was also prepared. The coal originated from the clean coal stockpile at Tom's Creek Preparation Plant in Coeburn, Virginia. The ¼" coal was ground using a pulverizer. After going through the pulverizer once, the product was dry screened. The minus 80 mesh material was saved and the oversize was discarded. The product was approximately 19% ash and

34% minus 325 mesh. Water was added to create a 6.0% solids slurry, assuming the original solids were dry. Two samples were prepared with this material. The first was wetted by shaking the coal and water in a closed container for 1 minute and used immediately. Each 900 ml feed was made separately. The second sample was created by letting the water and coal sit for three days in a bucket. It was stirred occasionally with a rod and all of the lumps were broken to allow wetting.

Two other preparation plants provided samples for testing. The first came from Dickenson-Russell Coal Company's Moss No. 3 Prep Plant in Clinchfield, Virginia. The minus 100 mesh flotation feed came from the same coal splits as those feeding Tom's Creek. The slurry was 6.6% solids. The second sample originated from Arch Coal's Pardee Preparation Plant in Appalachia, Virginia. It was cut from the six inch cyclone overflow in order to produce an ultrafine, minus 325 mesh sample. The slurry was 9.3% solids. In both cases, chemicals, likely cationic coagulants, were added at the plant in order to decant water and increase the solids content.

Agglomeration tests were also performed on several anthracite and bituminous tailing pond samples. Their origin was undisclosed. The semi-dry material was mixed with water to create a 7.0% solids slurry and a coarse screen was used to remove foreign twigs and leaves. The anthracite sample was 55% ash, the Pittsburgh seam sample was 33% ash, and the Sewell seam sample was 15% ash.

3.2. Materials and Supplies

3.2.1. Pentane

The oil used in agglomeration was *n*-Pentane produced by Alfa Aesar Co. The pentane is HPLC grade and 99% min. The clear liquid has a density of .626 g/ml and is immiscible in water. Its boiling point is 36°C, and explosive mixtures of air and pentane exist when pentane composes 1.4-8.0% by volume. Pentane is considered a very fast evaporating liquid and has a vapor pressure of 416 mm Hg at 20°C. It is very flammable and may cause skin irritation. More detailed information may be found in Alfa Aesar's MSDS.

3.2.2. Miscellaneous Chemicals

Three other chemicals were used: Magnafloc 356, fluorescein, and ethanol. Ciba Chemical's Magnafloc 356 is a cationic polymer. The chemical was used to coagulate tailings to speed filtering. Cole-Parmer's fluorescein is a yellow-green fluorescent dye. It is miscible in water, not pentane. It can be seen by the naked eye in large concentrations; however, ultraviolet light enhances its visibility. Small amounts of ethanol were used to aid in rewetting dry coal before Microtrac sizing.

3.3. Experimental Apparatus

3.3.1. Moisture Determination

In order to report moisture due only to water, not pentane and water, a special weighing platform was designed. A bottom-loading Denver Instruments scale was placed on top of a small oven within a fume hood. A metal platform was connected to the base of the scale via a hole in the top of the oven. The scale was connected to a laptop so changes in weights could be recorded and graphed in real time. A remote thermometer was used to monitor the temperature in the oven, and the scale was partially surrounded by Plexiglas to protect it from the breeze in the fumehood (Figure 3-1).

A condenser was also setup to capture the evaporating moisture. The liquid was condensed in graduated container, so the volumes of pentane and water could be compared. A 500 ml flask, large enough to hold the centrifuge concentrate, held the original sample and sat on a laboratory heater. Quarter inch tubing connected the flask to a condenser. The condenser was chilled by cold tap water, and the condensed liquid fell into a graduated container chilled by ice water. The system was closed to prevent vapor loss (Figure 3-2).



Figure 3-1. Weighing platform used for moisture determinations.



Figure 3-2. Condenser apparatus.

3.3.2. Agitation and Mixing

3.3.2.1. *Hand Shaking*

Most oil agglomeration was done by simple hand shaking. Pentane is a volatile liquid and evaporates quickly if open to the atmosphere. Hand shaking provided a simple solution to mixing in a closed container. The original oil agglomeration tests were performed in a 500 ml cylindrical separatory funnel. The cylindrical shape was chosen because it created a thicker bed of agglomerates. Later centrifuge testing required larger samples and a 1000 ml separatory funnel was utilized. The funnels were each used with a rubber stopper and stopcock.

3.3.2.2. *Impeller Mixing*

Impeller mixing was also explored in order to provide more consistent agitation than hand shaking. First, a glass separatory funnel with a ground glass and o-ring sealed bearing was used. Though the bearing held against the vapor pressure, the mixing blades hit the edges of the container at high speeds. For safety reasons, a cylindrical plexiglass container with a top and bottom port was created for continuous mixing. A ground bearing with vacuum grease was used to seal the mixing shaft. However, the bearing around the mixing shaft could not withstand the vapor pressure, and the container leaked pentane. Finally, impeller mixing open to atmosphere was attempted with a laboratory Denver flotation cell. Air was not used during the mixing.

3.3.3. Cleaning and Water Transfer

During the centrifugation process, a pseudo-enclosed water transfer system was created to minimize pentane loss. After mixing, the 1000 ml separatory funnel used for hand shaking was connected between two other containers for liquid exchange. The top container held clean wash water and the bottom flask held the exchanged clay water. Quarter inch rubber tubing completed the circuit between the three containers to prevent a vacuum, allow liquid exchange, and prevent pentane loss. Stopcocks above and below the funnel containing the agglomerates allowed it to be sealed while water was added or emptied from the other two glasses. Small amounts of pentane vapor were lost from these transfer containers whenever they were open to atmosphere (Figures 3-3 and 3-4).



Figure 3-3. Base of water transfer apparatus with Fluorescein dye.



Figure 3-4. Top of water transfer apparatus with Fluorescein dye.

3.3.4. Centrifugation

A sealed centrifuge was the main component of this process. The centrifuge was driven by a $\frac{1}{2}$ hp, variable speed DC motor. A one to four pulley ratio enabled the centrifuge to operate between 0 and 4360 rpm. The centrifuge contained an inner basket lined with centrifuge bars, and the sample was poured into this basket through a port in the middle of the lid. The inner basket contained an insert in the middle which forced the coal to the sides of the container. It was added to assist in even cake formation. The inner basket was enclosed in a solid outer shell. The entire centrifuge was made of aluminum and was held by bearings on the top and bottom. Though the centrifuge could hold about 450 ml of liquid while still; if it was spinning, it could only hold 160 ml before the liquid would touch the outside of the inner basket. The centrifuge contained a plug in the bottom so water could be drained after centrifugation was complete, and a rubber stopper in the top of the lid sealed the centrifuge (Figures 3-5 and 3-6). Scaled drawings of the centrifuge are located in the Appendix (Figures A-1 through A-6).



Figure 3-5. Centrifuge and stand.

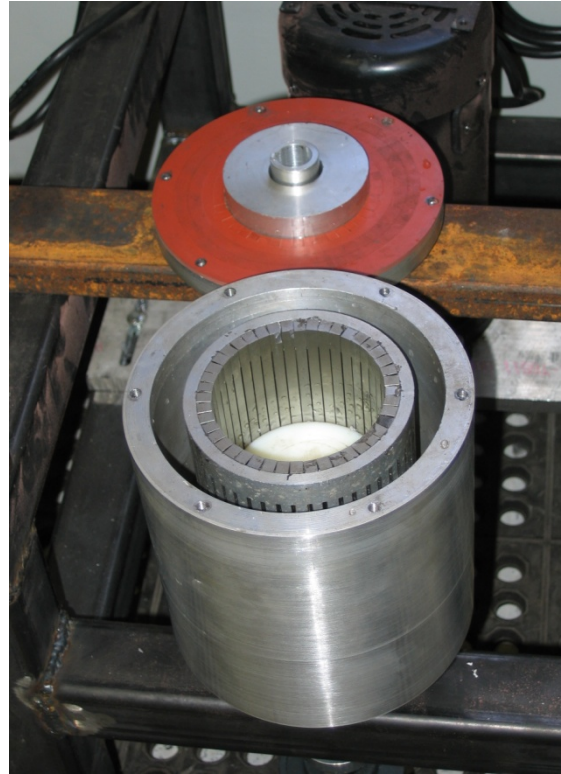


Figure 3-6. Inner centrifuge assembly.

3.3.5. Filtering and Displacement

Two types of vacuum filters were used in the testing. i) The main Denver filter was approximately twelve inches in diameter and was used with filter paper to dewater tailings before they were placed in the oven. ii) A small Peterson filter was used to measure concentrate moisture due to filtering and to determine the void space of a packed coal cake. The Peterson filter was only 6.3 cm in diameter and could hold about 500 ml of slurry prior to filtering. It was generally used with filter paper. Both filters were connected to a four liter flask which stored the removed water, and a flask of desiccant was located before the vacuum pump to protect the machinery. Stiff, quarter inch tubing connected the components. The vacuum pump could pull up to 20 in Hg.

The displacement process also utilized the Peterson filter, but it was powered by a water aspirator. The Peterson filter sat on a four liter flask which was connected directly to the water aspirator. Since displacement utilized large amounts of oil, all of the filtering equipment, including the aspirator, was located within a fumehood (Figure 3-7).

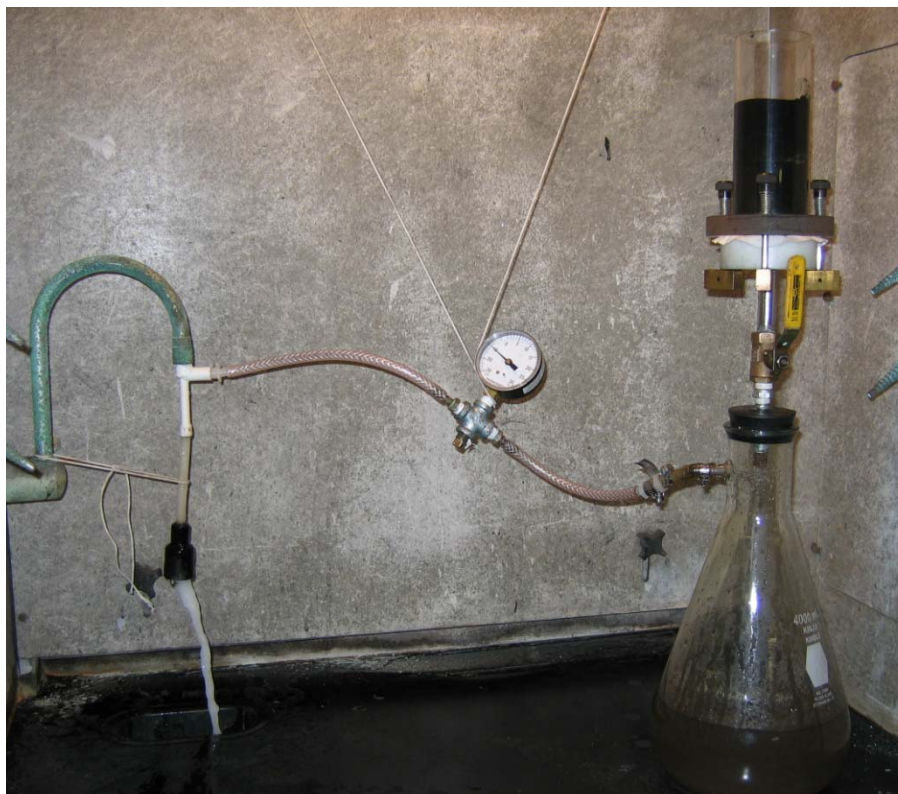


Figure 3-7. Displacement setup (Peterson vacuum filter and water-driven aspirator).

3.3.6. Miscellaneous

A variety of standard lab tools were utilized including beakers and buckets for carrying water, glass dishes for drying concentrate, glass syringes for measuring pentane, metal pans, brushes, and a stopwatch. Most tests were performed in a fumehood due to the presence of pentane. Eight screens, 28, 65, 80, 100, 150, 200, 270, and 325 Tyler mesh, were used for sizing with a vibrator. An Ohaus moisture balance was used to record the evaporation rates of small amount of water, pentane, and coal. It had a capacity of 50g; an aluminum dish was cut to fit inside the balance and enabled it to hold about 50ml instead of only 10ml. A Microtrac was used to size small feed, concentrate, and tailing slurries. A laboratory Denver flotation cell was used for release analysis and single-stage flotation. Finally, a briquetter was used to press the agglomerates into an easily handleable product.

3.3. Experimental Procedures

3.3.1. Moisture Determination

Correct moisture determination played a critical role in the experiments. In a commercial application, pentane would be recovered before the product was shipped; therefore, it would be deceptive if the reported moisture included anything other than water. Four different methods were explored to determine the product's water content. A moisture balance was used to identify the original problem. A weighing platform constituted a solution to the problem, and the condenser and fluorescein methods were introduced to attempt to validate the weighing platform method.

3.3.1.1. *Moisture Balance*

The original purpose of the Ohaus moisture balance was to confirm the variable evaporation rates of pentane and water. Its other purpose was to determine if the produced baseline evaporation curves could be used to predict the time needed to remove pentane. In order to reach these goals, the balance was set at a constant 40°C and was connected to a laptop for live recording of data. Thirty tests were run with mixtures of 0g, 5g, 10g, or 20g of water and/or pentane and 0g or 15g of coal. Once the desired weights were determined, the coal (a dry, clean flotation concentrate) was placed into the aluminum weighing dish. Densities of 1 g/ml for water and .626 g/ml for pentane were used to calculate the desired volumes of the liquids. Next, the appropriate amount of water was measured and stirred into the coal. Pentane was added next, and after a quick mixing, it was placed into the balance before too much pentane could evaporate. Weights were recorded until either the sample weight remained constant or 60 minutes passed. Once the testing was completed, the resulting change in weights and evaporation rates were plotted against time to identify trends and attempt to predict needed evaporation times.

3.3.1.2. Weighing Platform

The weighing platform was created to estimate the water-only moisture content in large concentrate samples. Before beginning, the oven was set at 40°C. This temperature is close to the boiling point of pentane (36°C); a higher temperature was not used due to the risks associated with the flammable liquid. A remote digital thermometer was used to monitor the temperature, and once the oven reached the desired temperature, the scale was connected to a laptop. Concentrate samples were placed in deep, 500ml glass petri dishes. After the concentrate was placed on the platform, the oven temperature was manually adjusted to maintain the temperature within 1°C of 40°C. As pentane evaporated, it chilled the container and surrounding air. To compensate for this immediate drop in temperature and the heat loss due to opening the oven door, the oven was usually set a few degrees above the desired temperature and changed back to the desired set point after the concentrate was in the oven.

Excel was used to plot the changing weight against time and calculate the evaporation rates (Figure 3-1). Unlike water, pentane is considered a very fast evaporating liquid. The

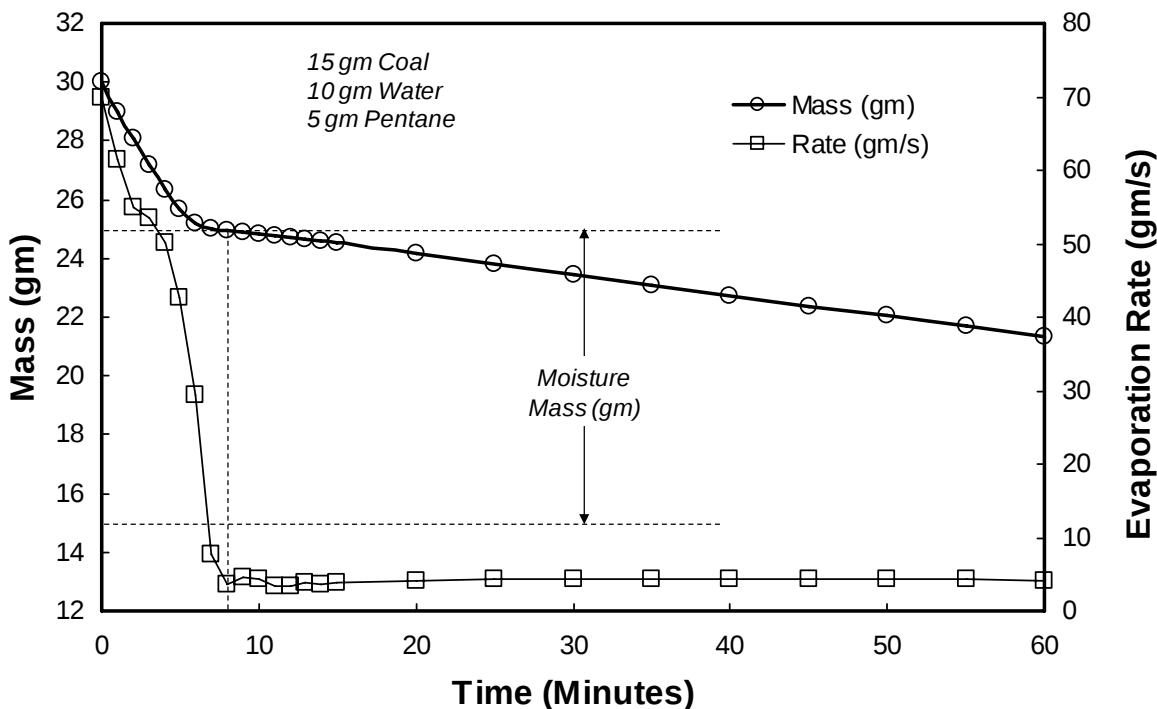


Figure 3-8. Example of an evaporation curve obtained from the weighing platform.

discrepancy in their evaporation rates means pentane will evaporate quickly, leaving only water behind. The difference in evaporation rates creates an elbow in the weight versus time curve. The first leg and curve of the graph are assumed to represent evaporating pentane. The gentler slope of the second leg is assumed to represent slower evaporating water.

In order to determine the moisture due to water, an initial and final weight were needed. The initial weight was taken where the calculated evaporation rates first become constant in the second leg. On the graph this point appears after the elbow of the curve. This point was chosen based on previous moisture balance testing (Appendix C) and is explained in the results and discussion sections. Once the second leg of the curve was established, the concentrate was transferred to a larger oven. The sample remained in the oven for 20 hours at 90°C to remove all of the surface moisture. The final weight was measured after this second stage of drying. Surface moisture was calculated with the difference in weights divided by the initial weight and multiplied by 100.

3.3.1.3. Condenser

The condenser was created to check the accuracy of the water content determined by the weighing platform. Due to its bulky setup, it was not meant to be used as a permanent method for moisture determination. After checking that all of the connections were sealed, the concentrate sample was placed in the flask, and the heater was turned on. When the condensed liquid level in the graduated container became constant, the volumes of the water and pentane were recorded. They were used to find the weights associated with pentane and water based on density. The weights were used to calculate moisture and compared to similar drying tests performed with the weighing platform.

3.3.1.4. Fluorescein

When the weighing platform returned higher than expected moistures from a series of centrifugation tests, fluorescein was used to determine if and where that water was present. It was only used in conjunction with centrifuge testing; the centrifuge procedure is described in detail in a later section. Fluorescein dye was added in two places. Several drops of dye were added and shaken with the original slurry before oil agglomeration began. Later when clay water

was exchanged for clean water, the clean water also contained dye. The sample underwent centrifugation, and the resulting cake was examined in a dark room with an ultraviolet light. Several agglomerates were also removed from the cake and examined under a microscope with ultraviolet light.

Finally, fluorescein-rich water was mixed with dry centrifuge concentrate to create a 20% moisture mixture. This represented the high moistures coming out of the centrifuge. The sample was stirred until the coal was thoroughly wetted, and the sample was examined in a dark room to see if fluorescein was visible when spread over such a large surface area.

3.3.2. Agitation and Mixing

3.3.2.1. *Hand Shaking*

In this project, the standard method for agglomeration agitation was hand shaking. It was difficult to create a small sealed mixing container for pentane, so a simple separatory funnel and rubber stopper were used. Since hand shaking is not consistent and to ensure complete agglomerate formation, the slurry sample was shaken for about five minutes. It was shaken the full time regardless of whether or not the agglomerates formed in a shorter amount of time.

3.3.2.2. *Impeller Mixing*

Two impeller mixing methods were attempted for oil agglomeration. First, a plexiglass container described in the apparatus section was used to agglomerate with pentane. After the two ports were sealed, slurry and pentane were added, and a variable speed motor provided mixing. Second, agglomeration open to atmosphere was attempted. Since pentane evaporates quickly, heptane was used instead. Slurry was placed in a 1.5 liter Denver flotation cell. The flotation cell was used only for agitation; air was not used.

3.3.3. Cleaning and Water Transfer

Water transfer consists of three stages: clay water removal, cleaning, and water replacement. Before transfer began, the stopper in the shaking separatory funnel was replaced with a new stopper that had a tube running through it. The tube was designed to allow clean water to enter while clay water drained through the stop cock at the base of the glass. The first

step in cleaning was to remove the bulk of the clay water. The upper chamber remained empty during this stage. Both stop cocks were opened, and the agglomerates were allowed to gravity drain until no water remained. Then the stop cocks were closed again. To begin the cleaning stage, about 600 ml of clean water were placed in the upper chamber while the bottom was emptied of the clay water. The top stop cock was opened, allowing water to enter the agglomerate chamber, and the agglomerates were shaken slightly to free the clay trapped between them. The bottom stop cock was opened once about 400 ml of clean water had been transferred. The remaining clean water continued to wash the agglomerates as water drained through the base. Once all of the water had been drained from the agglomerates, the stop cocks were closed again to isolate the chamber. To begin the water replacement stage, about 100 ml of clean water were added to the top chamber. The top stop cock was opened, and the clean water moved into the agglomerate chamber. The stopper was quickly exchanged for the original, solid stopper, and the separatory funnel was disconnected from the bottom chamber. The water transfer was complete.

3.3.4. Agglomerate Extraction and Dewatering

3.3.4.1. *Shaking*

This method was used in conjunction with agitation by hand mixing. No cleaning stage was utilized; instead clay water was simply drained from the bottom stop cock. The agglomerates were gently shaken up and down a few times to release the remaining trapped water. Once the bulk of the water was removed, the cylindrical separatory funnel was held above a metal pan at about 20 degrees from horizontal. The glass was shaken in an irregular motion. The agglomerates were thrown up the glass and out the opening while the bulk of the water remained in a pool at the other, lower end. The shaking time was arbitrary; since the goal was dewatering, shaking stopped when too many water droplets exited the glass. The material shaken out was considered the concentrate, and it was scraped into a glass dish with care being taken to also remove the water smears from the metal pan. The concentrate's moisture was then measured with the weighing platform. Any solids still in the original container were considered middlings. Though they were clean, they had a high moisture content due to the pooled water. Unfortunately, the middling's moisture could not be determined because wash water was used to remove the solids from the glass, raising the water content.

It should be noted that small variations in this process were needed based on the dosage of oil used. As the oil dosage increased and the balls became soft, additional shakes were not used to remove extra water after the main draining because shaking collapsed the drainage channels. As dosage further increased, the consistency of the material was not conducive to shaking. In these cases, all of the material was simply poured into the concentrate dish, essentially eliminating the middlings.

3.3.4.2. Screening

This screening method is different from the classical method; instead of water falling through the screen, water rich solids remained on top of the screen while dry material fell through. This method also used the finest screens first and proceeded coarser, the opposite of traditional screening used for sizing. Four screens were utilized: 28, 65, 100, and 270 Tyler mesh. Fitted solid metal pans were placed under each screen to prevent material loss. The powdery flocs constituting the feed were produced by oil agglomeration with a low dosage of oil. Some of the flocs were poured over the smallest screen, and the screen was gently shaken back and forth to sieve the dry powder through. More feed was slowly added until the entire batch had been screened. Shaking caused the water droplets to coalesce and roll on the surface of the screen. Screening stopped once no more material passed through, the screen became blinded, or the screen began to wet. The undersize was weighed for moisture; the weighing platform was not utilized because initial weighing with it indicated no pentane was present after screening. Meanwhile, the oversize was placed on the next larger screen, and the process was repeated for each screen. Care was taken when transferring the oversize in order to prevent the water droplets from wetting the metal. Since water could not be used to rinse the oversize onto the next screen, any solids or water remaining on the screens were considered lost.

3.3.4.3. Air Classification

In this process, agglomerates were removed from the water phase with an air hose. The hose was inserted into the separatory funnel and used to blow the top agglomerates out. The aim was to remove the agglomerates sitting above the liquid level. As agglomerates were removed from the top of the bed, more filled the gaps due to buoyancy.

3.3.4.4. Centrifugation

Centrifugation of spherical agglomerates was performed only after a sample had been cleaned with the water transfer procedure. A funnel was placed in the top of the sealed centrifuge, and the agglomerates were poured out of the separatory funnel. Due to the limited volume in the centrifuge, only 50 ml of wash water were used in addition to the original 100 ml of water with the agglomerates. The rubber stopper was replaced once the agglomerates were inside the centrifuge, and the time required to pour the material was recorded in case a sample took an excessive amount of time to pour, allowing pentane to evaporate. The variable speed centrifuge was then turned on for a specified amount of time. It should be noted that the centrifuge was generally off during the pouring stage in order to create a more even cake.

3.3.4.5. Filtering

Filtering was performed with clean compact spherical agglomerates floating in clear water. Once the slurry was placed in the Peterson filter, the vacuum pump was turned on, and the valve was opened. The initial filtering time was recorded, and the agglomerates were generally allowed to dry for an additional one minute after the entire cake's surface was exposed to air. The pump operated at maximum vacuum in all of the tests, and the vacuum pressures were recorded for the filtering and drying phases. The dewatered sample was collected from the filter and subjected to a standard moisture determination. After initial testing with the weighing platform indicated pentane had evaporated during filtering, the initial weight for moisture determination was simply taken at time zero.

3.3.4.6. Displacement

Displacement consisted of filtering coal slurry with large amounts of pentane. Dry pulverized clean coal from Tom's Creek was utilized for all of the tests. Feed samples were prepared by shaking coal and water together for one minute; the resulting 400 ml slurry contained 6.0% solids. After the slurry was poured into the filter, about 80 ml or one inch of pentane was slowly poured on top. Care was taken to pour the oil slowly to prevent the formation of coal-covered water droplets in the pentane phase. The valve was then opened to begin filtering. Several times corresponding to important events were recorded during the course

of each experiment. These included the approximate time when water finished filtering, the total filtering time, and the drying time, usually 30 or 60 seconds. Since a water aspirator was used and multiple liquids were being filtered, the pressure varied throughout the test. Only the starting pressure could be controlled by water flow. Pressures were recorded at the beginning and end of the water and pentane phases.

3.3.5. Conventional Cleaning and Dewatering

3.3.5.1. Flotation

Two sets of flotation testing were performed: release analysis and single-stage cleaning. A release analysis was performed on the minus 100 mesh flotation feed from Tom's Creek and on each of the tailing pond samples. In each case, the collector was diesel, and the frother was MIBC. Though recorded, the amount of collector was not regulated during release analysis since the goal was complete cleaning. Each sample underwent five cleaning stages; the concentrate was cleaned five times with one cumulative tailings. During the fifth stage, the concentrate was removed in eight batches of approximately equal weights. These samples were weighed and ashed, and the resulting recoveries were plotted against the ash content.

Single stage cleaning was also performed to represent conventional cleaning. The product then underwent filtration, and the final moisture could be used to compare conventional and pentane dewatering. The 1.5 liter flotation cell was agitated at 1100 rpm, and all of the tests used 3µl of MIBC frother. Several Nalco reagents and diesel were screened as collectors at 200 and 400 g/ton or at 20 and 40 µl. The cells were run to exhaustion.

3.3.5.2. Filtering

Three conventional filtering series were performed with the use of a Peterson filter. The first filter series utilized flotation concentrate from single-stage laboratory flotation. The dry concentrate was mixed with water for a solids content of 10%. Each feed sample contained 250ml of slurry. Baseline filtering was performed without any dewatering aids. Tests were also performed with the RV and RW dewatering aids developed at Virginia Tech. The samples were conditioned with the chemicals for two minutes prior to filtering. All filtration was performed at maximum vacuum pressure with a drying time of two minutes.

Filtration was also regularly performed on both agglomerate feed and concentrate samples. The agglomerate feeds required no special preparation. Maximum vacuum pressure was used to filter 400 ml feed samples with one minute drying times. The concentrate samples required more preparation. Oil agglomeration was performed with 500 ml, and the subsequent spherical agglomerates were cleaned. The concentrate was then placed in a blender and left open to atmosphere for about an hour to ensure pentane evaporation. Afterwards the blender was turned on for 20 seconds at a low speed to shear the agglomerates and rewet the coal. Filtration was performed with a Peterson filter at maximum vacuum pressure with one minute of drying time.

4. RESULTS

4.1. Samples

Testing revolved around three main samples from Alpha Natural Resources' Tom's Creek Plant. A Microtrac size analyzer was used to determine the size distributions for each sample. The surface areas of the concentrates were then estimated from the size distribution data by assuming spherical particles.

The first sample consisted of unaltered flotation feed from the Tom's Creek plant. This minus 100 mesh sample was used to evaluate all of the dewatering methods explored in this study. The sample had an average ash of 38% and was approximately 72% minus 325 mesh. Figure 4-1 shows the relative size distributions of a centrifuge feed, concentrate, and tailings originating from this sample. It has an estimated surface area of 14,300 m². This sample also underwent BET analysis, which used gas adsorption to determine the total surface area, including pore area, of the sample. Unfortunately, the data from the BET analysis was not available at the time this report was prepared.

The second sample also originated from Tom's Creek's minus 100 mesh flotation feed; however, a portion of the ultrafines were removed by sieving. The deslimed sample contained 19% ash, 28% minus 325 mesh, and had an estimated surface area of 320 m². Figure 4-2 shows the relative size distributions of a centrifuge feed, concentrate, and tailings originating from this sample.

The third sample was obtained by dry pulverizing a sample of clean coal from the Tom's Creek plant. The resulting minus 80 mesh sample contained 19% ash, 34% minus 325 mesh, and had a surface area of 380 m². The centrifuge size distributions are provided in Figure 4-3.

Figure B-1 in the Appendix provides further information on the Microtrac's sizing methods and accuracy, and Figures B-2 through B-4 contain further size distribution information for the three samples.

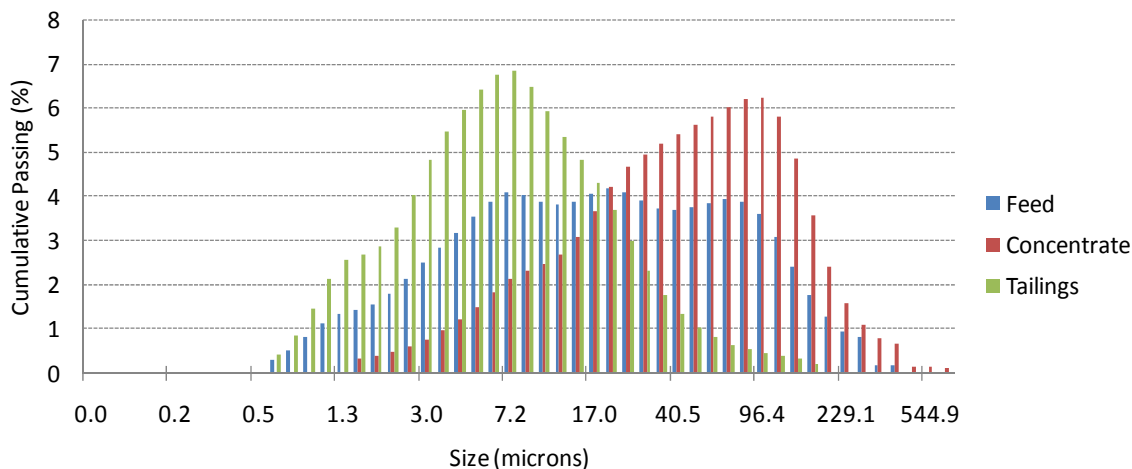


Figure 4-1. Size distributions of centrifuge feed, concentrate, and tailings for 100 mesh x 0 flotation feed.

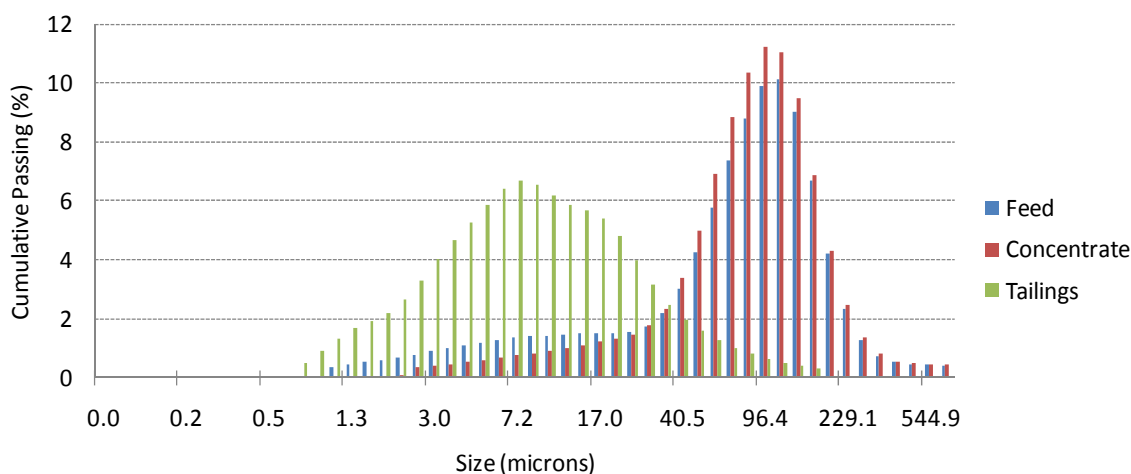


Figure 4-2. Size distributions of centrifuge feed, concentrate, and tailings for deslimed 100 mesh x 0 flotation feed.

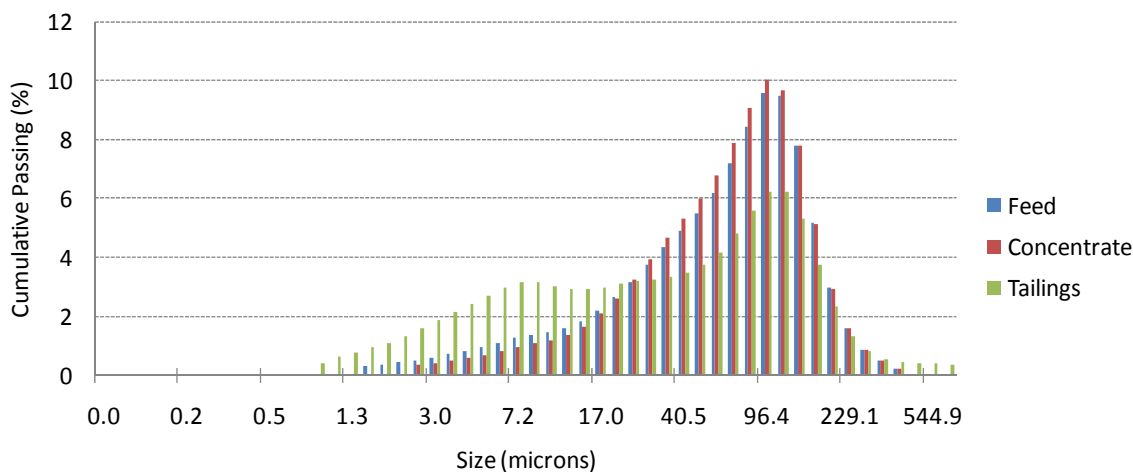


Figure 4-3. Size distributions of centrifuge feed, concentrate, and tailings for pulverized 80 mesh x 0 clean coal.

4.2. Moisture Determination

The preferred method of moisture determination utilized a weighing platform. Before this method was implemented, the initial weight for moisture determination was found by placing a sample in an oven for 20 minutes at 40°C to evaporate pentane. No water was assumed to evaporate during this initial period. Any loss in weight after this period was attributed to moisture remaining in the sample. Unfortunately, this approach tended to misrepresent moisture. Moistures were underestimated for concentrates produced with small oil dosages and were overestimated for concentrates with large oil dosages. To overcome this problem, moisture determinations for future samples were conducted using the weighing platform. This device provides an evaporation curve showing the sample weight loss as a function of time. Evaporation rates were also calculated from the recorded weights and times (Figure 4-4).

The example evaporation curve in Figure 4-4 is from a concentrate with a large oil dosage. It displays a sharp elbow, and the two legs are due to two distinct evaporation schemes. The first leg has high evaporation rates which fall from 2.9 g/min to .4 g/min. The initial rise in

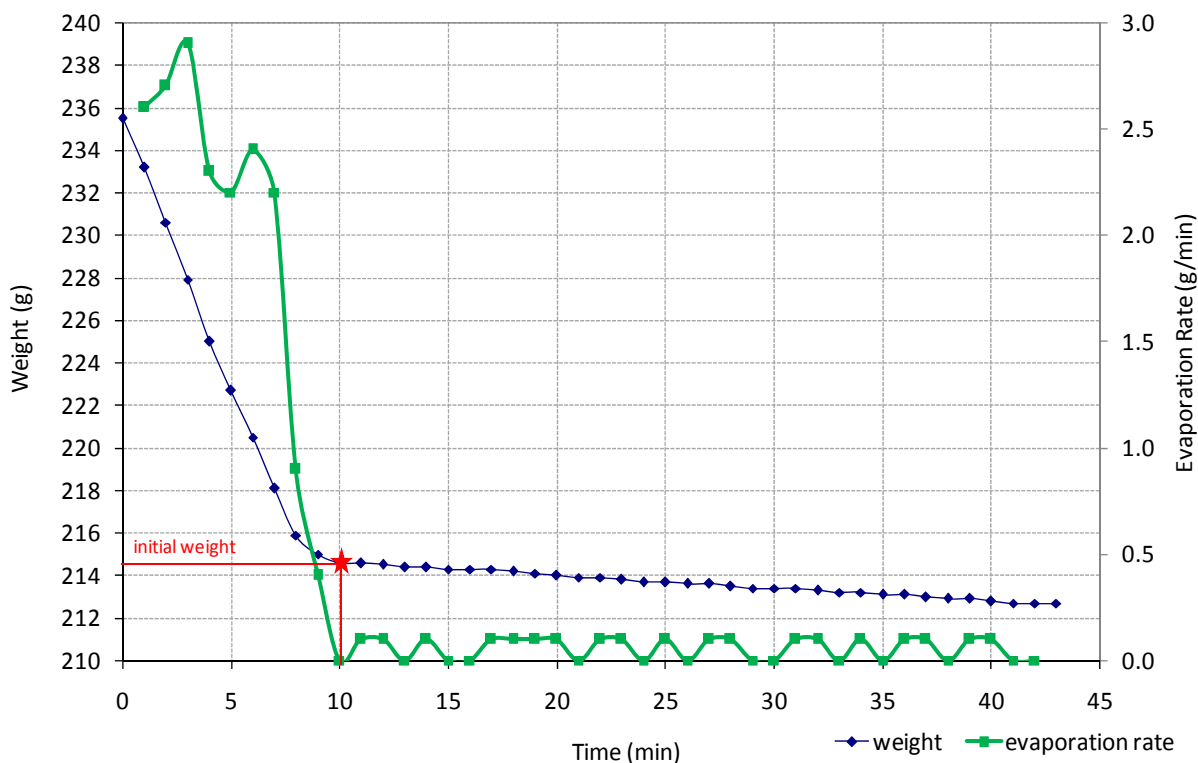


Figure 4-4. Example how the initial weight is chosen for moisture determination from a weighing platform evaporation curve.

evaporation rate may attributed to scale error when the sample was first loaded; the weights fluctuate slightly in the first two or three minutes as the platform sways. The second leg displays a consistent evaporation rate between .1 g/min and 0 g/min. Similar graphs are produced when samples with smaller oil dosages are weighed. They display shorter first legs. Eventually, if the oil dosage is small enough, the curve will begin on the elbow. In some cases no deflection is visible which indicates that all of the pentane evaporated during handling, and the initial weight is simply taken at time zero.

These curves aided in identifying the moisture due to water. As described in the procedure section, the initial weight for the water-only moisture was taken when the evaporation rate first became constant in the second leg. Experiments conducted using specially prepared samples containing known amounts of dry coal, water and pentane indicated this point was preferable to the projected intersection of the two legs of the weight curve. Detailed graphs and explanations of this moisture balance data may be found in Appendix C.

It should be noted that some water likely evaporated during the first leg of the weight curve. It is impossible to only evaporate pentane at 40°C; however, the volume of water evaporated is negligible compared to pentane. Not only is water's evaporation rate low in the second leg (0-.1 g/min), but it should be even lower in the first leg due to chilling as pentane evaporates.

Table 4-1 highlights the impact the determination method had on the calculated moistures. As expected, the fixed drying time (original) method used to calculate the moistures substantially underestimated the true (corrected) moisture contents of the samples determined using the weighing platform. These corrected moistures utilized the weights at time zero since

Table 4-1. Average original and corrected hand shaking moistures for 100 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Original Moisture (%)	Corrected Moisture (%)
0.16	33.72	37.6
0.21	28.21	33.4
0.29	12.92	20.3
0.36	6.45	14.2
0.48	8.25	--
1.45	47.32	--

**Note: All tests conducted at 6.6% solids.*

small amounts of pentane were used. It was assumed the pentane evaporated during handling. The original moistures displayed errors of 10-55%. Therefore, all of the moisture contents reported in this document were determined using the weighing platform. Tables B-1 and B-2 in the Appendix contain detailed data on the original incorrect moistures.

4.3. Agitation and Mixing

Agglomeration using pentane was performed with a wide range of dosages. The dosages were roughly based on pentane-to-solid mass ratios of 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Based on a feed of 400 ml at 15% solids and an ash of 39%, these ratios correspond to pentane-to-coal mass ratios ($M_p:M_c$) of 0.18, 0.23, 0.32, 0.40, 0.53, and 1.60.

Figure 4-5 roughly shows the trend in agglomerate size as dosage changes. The lowest dosage produces floc-like agglomerates less than 0.5 mm in diameter. The agglomerates appear powdery and float on the surface of the water phase; however, large amounts of water are trapped within the powder. The next three dosages produce spherical agglomerates that increase in size from 0.5 to 2 mm in diameter as the oil level increases. The agglomerates float in the

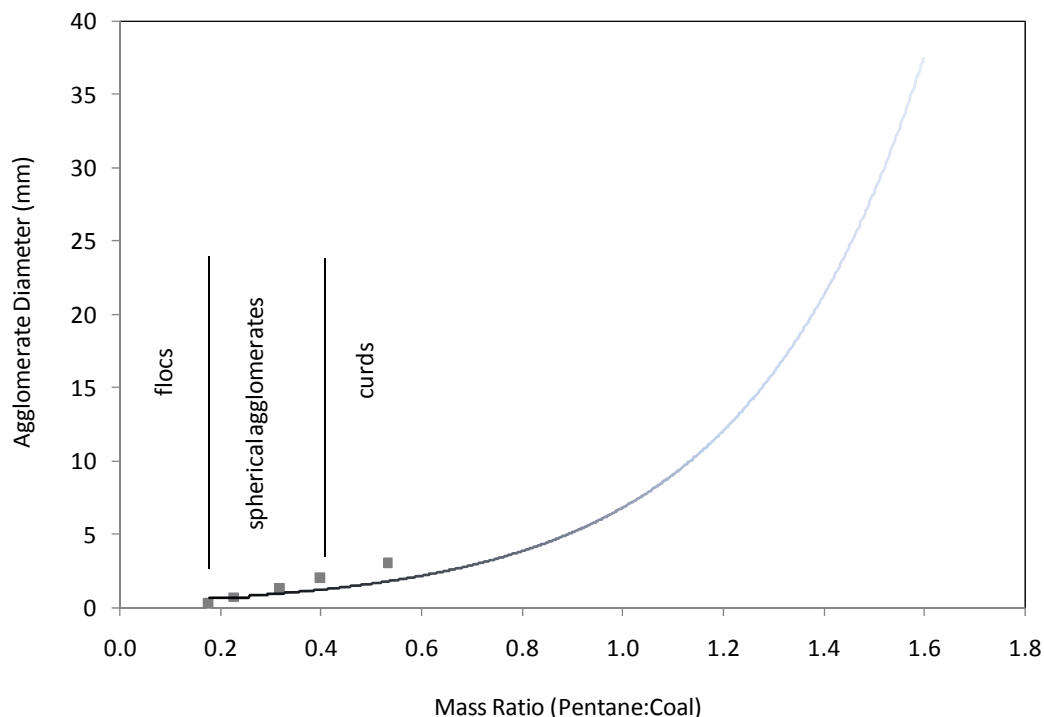


Figure 4-5. Estimated change in agglomerate size and appearance with pentane dosage.

water near the water-air interface. As the oil level increases, they become shinier and clump slightly. There are distinct drainage channels between the agglomerates. The next dosage produces black, cottage-cheese-like curds. These semi-solid curds are extremely soft and tend to trap large amounts of water. Finally, the highest dosage of pentane tends to form a semi-solid layer of coal, water, and pentane near the top of the water phase. There are no distinct agglomerates, and a clear layer of pentane lies on top of the coal.

4.4. Pellet Extraction and Dewatering

4.4.1. Shaking

Hand shaking tests were performed on four different samples, which all originated from the flotation feed at the Tom's Creek plant. The first two were 100 mesh x 0 samples of flotation feed containing solids contents of about 6% and 15% by weight. The third was identical to the first two except that the samples had been aged (oxidized) for four months prior to testing. The fourth was a much finer sized sample (i.e., 325 mesh x 0). In order to evaluate any reproducibility errors in moisture determination, each series of tests was performed multiple times. The first two series of tests were repeated three times; however, the tests performed with oxidized and 325 mesh x 0 samples were only performed once due to lack of sample.

Tables 4-2a and 4-2b show the results of the first series of tests which were performed on fresh 100 mesh x 0 feed without using the weighing platform to determine moisture. The tests were repeated using the weighing platform and the results are summarized in Tables 4-3a and 4-3b. A comparison of the data from Tables 4-2 and 4-3 show that the true moistures obtained using the balance are significantly higher than those obtained without the balance. The tests conducted at lower percent solids provided slightly lower moistures. The lowest, at an average moisture of 16.2%, occurred at a $M_p:M_c$ ratio of 0.32, which corresponded to a dosage of about 1:5 pentane to solids by weight.

Table 4-2a. Measured values for the original hand shaking with 100 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Ash Content (%)		
	Tailings	Middlings	Concentrate
6.3% solids by weight			
0.16	89.61	6.57	6.50
0.21	91.43	7.78	7.73
0.29	90.85	7.03	6.06
0.36	90.59	7.16	5.31
0.48	88.74	11.50	5.15
1.45	88.69	8.90	6.92
15.0% solids by weight			
0.16	90.95	11.67	9.58
0.21	91.10	11.70	8.86
0.29	88.33	11.14	7.56
0.36	88.04	22.54	6.27
0.48	88.10	47.30	7.56
1.44	82.69	34.11	11.55

Table 4-2b. Calculated values for the original hand shaking with 100 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Yield (%)		Recovery (%)		Ash Rejection (%)		Moisture (%)
	M+C	C	M+C	C	M+C	C	
6.3% solids by weight							
0.16	65.1	44.1	94.4	63.6	88.4	91.4	33.7
0.21	67.0	42.2	95.6	60.0	85.2	90.3	28.2
0.29	65.9	34.0	95.2	49.4	87.6	94.1	12.9
0.36	65.4	35.8	95.0	52.6	88.6	94.8	6.5
0.48	64.9	53.0	93.9	77.8	88.0	92.3	8.2
1.45	65.4	58.4	93.9	84.1	86.8	88.6	47.4
15.0% solids by weight							
0.16	67.8	10.7	95.4	15.2	79.4	97.2	20.9
0.21	67.5	18.4	95.4	26.5	80.3	95.6	15.3
0.29	65.3	25.4	93.6	37.2	83.2	94.8	12.5
0.36	64.0	56.7	93.2	84.2	86.0	90.4	14.5
0.48	64.1	63.0	93.2	92.2	85.9	87.1	25.3
1.44	65.0	62.8	90.4	88.0	78.3	80.3	62.1

**Note: M+C indicates the yield, combustible recovery, or ash rejection's concentrate consists of the shaking middlings and concentrate. C indicates only the shaking concentrate is used in the calculations.*

Table 4-3a. Summary of measured values for hand shaking with 100 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Ash Content (%)		
	Tailings	Middlings	Concentrate
6.3% solids by weight			
0.18	91.82	6.47	5.87
0.23	92.21	6.18	5.28
0.32	91.30	5.70	4.64
0.40	91.91	8.74	4.57
0.53	91.76	11.65	5.07
1.60	89.89	25.47	8.26
14.8% solids by weight			
0.18	91.71	8.94	6.99
0.23	90.22	13.06	6.21
0.32	90.59	11.89	5.26
0.40	90.59	7.59	5.19
0.53	89.01	16.55	6.16
1.59	81.57	28.49	26.48

Table 4-3b. Summary of calculated values for hand shaking with 100 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Yield (%)		Recovery (%)		Ash Rejection (%)		Moisture (%)
	M+C	C	M+C	C	M+C	C	
6.3% solids by weight							
0.18	61.5	52.1	94.8	80.5	90.6	92.2	31.2
0.23	61.5	33.8	95.1	52.6	91.0	95.4	17.5
0.32	60.7	34.0	94.4	53.3	92.0	96.0	16.2
0.40	60.8	55.2	94.8	86.4	92.3	93.5	17.5
0.53	61.0	58.0	94.7	90.3	91.6	92.5	26.4
1.60	62.7	60.9	93.8	91.5	86.0	87.1	59.3
14.8% solids by weight							
0.18	62.0	34.1	94.8	52.6	87.9	94.1	26.1
0.23	62.0	36.2	93.8	56.3	86.5	94.4	19.7
0.32	60.2	50.8	93.8	79.8	91.0	93.3	18.9
0.40	59.9	51.9	93.7	81.5	91.6	93.2	19.9
0.53	59.6	59.4	92.6	92.3	90.6	90.8	31.9
1.59	75.9	75.0	92.4	91.4	49.2	49.9	82.8

**Note: M+C indicates the yield, combustible recovery, or ash rejection's concentrate consists of the shaking middlings and concentrate. C indicates only the shaking concentrate is used in the calculations.*

The data reported in Tables 4-4a and 4-4b utilized the same 100 mesh x 0 flotation sample after it had been aged four months. This particular set of experiments was conducted at 14.9% solids, which is very close to the solids content of 14.8% solids used to obtain the lower set of data for the fresh sample reported in Table 4-3. As shown, the moistures obtained using the oxidized samples increased up to 137% when compared to the moistures obtained using the fresh samples. The difference was particularly notable for the experiments conducted at the lower range of pentane dosages. On the other hand, the moisture values obtained at the higher dosage levels were relatively unaffected by the aging and potential oxidation of the sample.

The last series of agglomeration tests were conducted using a 325 mesh x 0 split of the Toms Creek flotation feed. The results obtained for the sized sample are shown in Tables 4-5a and 4-5b. As shown, the moisture values obtained using the minus 325 mesh split were

Table 4-4a. Measured values for hand shaking with oxidized 100 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Ash Content (%)		
	Tailings	Middlings	Concentrate
14.9% solids by weight			
0.12	90.76	15.45	8.71
0.16	81.50	9.52	7.44
0.22	83.68	9.62	6.43
0.28	82.02	10.31	5.46
0.37	84.90	27.09	6.67
1.11	76.18	48.32	21.22

Table 4-4b. Calculated values for hand shaking with oxidized 100 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Yield (%)		Recovery (%)		Ash Rejection (%)		Moisture (%)
	M+C	C	M+C	C	M+C	C	
14.9% solids by weight							
0.12	77.7	70.7	97.1	89.2	73.3	77.7	46.2
0.16	73.5	52.3	93.2	67.0	78.1	85.9	46.6
0.22	73.2	62.7	93.9	81.0	81.2	85.4	35.4
0.28	71.7	63.2	93.0	82.5	83.9	87.5	25.2
0.37	73.5	72.5	94.5	93.5	81.4	82.5	42.0
1.11	88.7	88.1	96.3	95.9	31.1	32.4	82.9

**Note: M+C indicates the yield, combustible recovery, or ash rejection's concentrate consists of the shaking middlings and concentrate. C indicates only the shaking concentrate is used in the calculations.*

Table 4-5a. Summary of measured values for hand shaking with 325 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Ash Content (%)		
	Tailings	Middlings	Concentrate
7.3% solids by weight			
0.22	91.39	6.68	7.59
0.28	91.46	6.22	6.63
0.40	90.52	6.26	6.22
0.50	89.78	7.53	6.63
0.66	84.52	14.38	9.31
1.99	81.24	14.82	14.83

Table 4-5b. Summary of calculated values for hand shaking with 325 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Yield (%)		Recovery (%)		Ash Rejection (%)		Moisture (%)
	M+C	C	M+C	C	M+C	C	
7.3% solids by weight							
0.22	47.4	30.7	90.7	58.6	93.4	95.5	34.0
0.28	47.0	24.6	90.7	47.3	94.1	96.9	28.7
0.40	46.3	23.4	89.5	45.2	94.4	97.2	27.7
0.50	46.3	28.5	88.7	54.9	93.7	96.3	35.1
0.66	44.1	41.2	82.2	77.0	91.7	92.6	47.6
1.99	44.8	42.9	78.6	75.3	87.0	87.7	72.0

**Note: M+C indicates the yield, combustible recovery, or ash rejection's concentrate consists of the shaking middlings and concentrate. C indicates only the shaking concentrate is used in the calculations.*

considerably higher than those obtained using the coarser 100 mesh x 0 sample. This finding shows that agglomeration procedure is sensitive to particle size. The lowest moisture for the ultrafine sample occurred at a $M_p:M_c$ ratio of 0.4, which provided a moisture of 27.7%. The 27.7% moisture was 70% higher than that obtained for the 100 mesh x 0 sample. This large increase cannot be attributed to only the slight increase in solids content of the sample.

Figure 4-6 shows the recovery-rejection curve for the hand shaking tests conducted using the fresh 100 mesh x 0 sample at different pentane dosage levels. Only the data from this sample was included in the summary plot since it provided the lowest overall moisture values. As shown, most of the data points fall along a single recovery-rejection curve, which suggests that increasing the oil dosage does not shift the separation curve. On the other hand, higher dosage values did tend to move each grouping of data points to a higher recovery level on the curve.

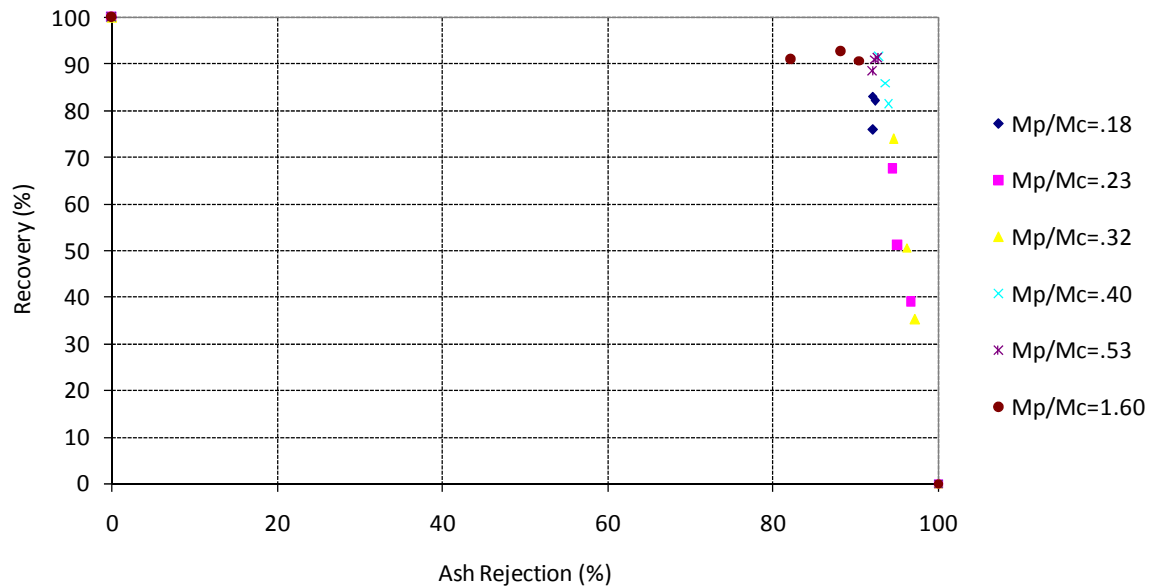


Figure 4-6. Recovery-rejection curve for 100 mesh x 0 flotation feed at different dosage levels.

*Note: M_p/M_c refers to the pentane-to-coal mass ratio.

The effect of pentane dosage on moisture, ash, and recovery for the fresh 100 mesh x 0 sample is more clearly shown in Figures 4-7 and 4-8. The data plotted in these two graphs correspond to feed solids contents of 6.3% and 14.8%, respectively. Error bars (50% confidence) were included to show the consistency of the moisture data. In both cases, an optimum dosage level occurs for moisture and ash at a ratio of 0.3-0.4. However, recovery tends to drop off sharply as the dosage ratio drops below about 0.5. Therefore, a very narrow window occurs at a dosage ratio of about 0.4 at which a reasonably low moisture and good recovery can be achieved.

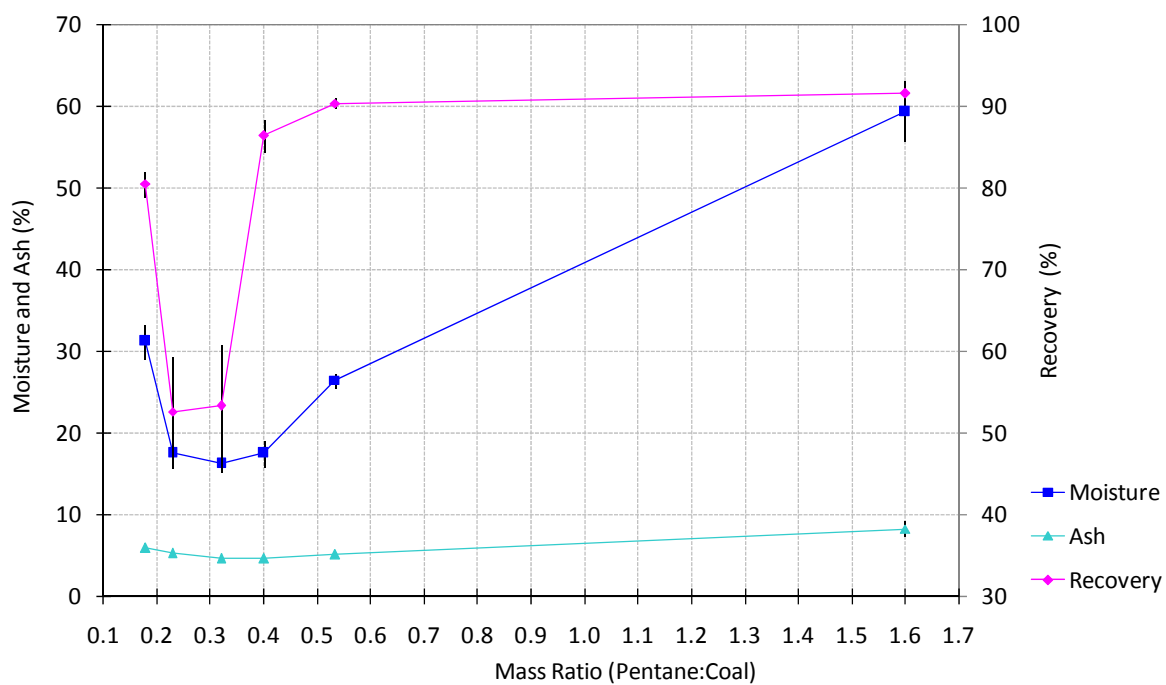


Figure 4-7. Comparison of hand shaking moisture, ash, and recovery for 100 mesh x 0 flotation feed at 6.3% solids.

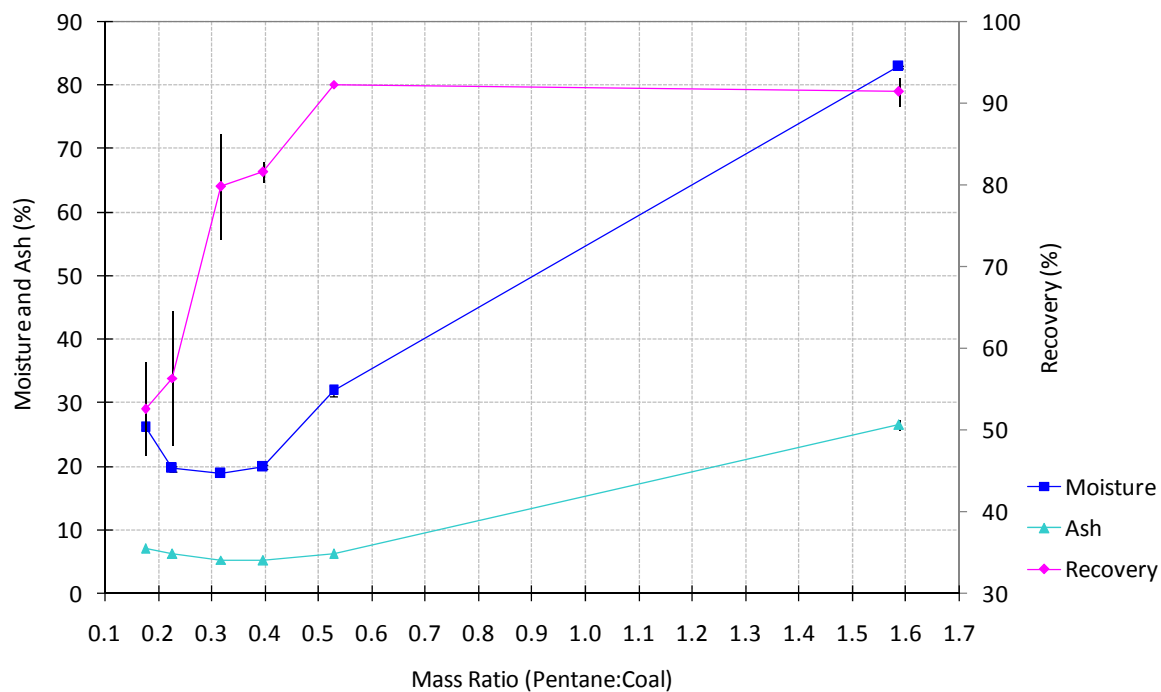


Figure 4-8. Comparison of hand shaking moisture, ash, and recovery for 100 mesh x 0 flotation feed at 14.8% solids.

4.4.2. Screening

Another method used to recover pentane agglomerated coal was screening. In this method, floc-like agglomerates were placed into individual screens with aperture openings ranging from 30 to 270 mesh. Dry solids passed through the sieve and were collected in pans, while coal-covered water droplets were retained atop the sieve (i.e., shaking caused the water droplets to coalesce and roll over the surface of the sieve). The screening tests were performed using a 100 mesh x 0 sample of flotation feed from the Toms Creek facility.

The test results from the screening tests are summarized in Table 4-6. It should be noted that many of the size classes contained less than one gram of sample; therefore, there are high errors associated with all of this data. Nevertheless, this procedure generated products with moistures contents ranging from a high of 50% for the coarsest sieve (30 mesh) down to a low of 6.1% for the finest sieve (270 mesh). Unfortunately, the procedure provided an overall combustible recovery of only 67.8%. Moreover, the bulk of the recovered solids were obtained from size fractions that corresponded to relatively high moisture values. The recoveries associated with products having 6.5% moisture or lower accounted for just 29.3% of the cumulative recovery (i.e., 23.1 + 6.2 = 29.3).

Table 4-6. Example of screening with 100 mesh x 0 flotation feed.

Particle Size Class			Measured	Calculated			
Pass (mesh)	Retain (mesh)	Mean (mm)	Ash (%)	Dry Wt (g)	Moisture (%)	Yield (%)	Recovery (%)
	30	0.600	6.44	0.30	50.0	3.7	5.9
30	70	0.406	5.91	0.74	27.5	9.2	14.6
70	100	0.181	5.10	0.91	12.5	11.3	18.1
100	270	0.102	4.76	1.16	6.5	14.4	23.1
270		0.053	4.60	0.31	6.1	3.8	6.2
Tailings			87.49	4.65	97.4	57.6	12.2
Total						42.4	67.8

**Note: The feed consisted of 200 ml of 41% ash 5.5% solids slurry. Approximately 8.5% of the material was lost to the screens and was not included in the yields and combustible recoveries. All of the screens are US mesh.*

4.4.3. Centrifugation

One of the most promising methods used to recover agglomerates was centrifugation. Three samples were dewatered using this technique: (i) the original 100 mesh x 0 sample containing 49% minus 325 mesh solids, (ii) a sample of 80 mesh x 0 pulverized coal containing about 32% minus 325 mesh solids, and (iii) deslimed 100 mesh x 0 sample containing 19% minus 325 mesh solids. The original and deslimed samples were conditioned with pentane using an average $M_p:M_c$ ratio of 0.32, while a slightly lower average $M_p:M_c$ ratio of 0.21 was utilized for the coarser 80 mesh x 0 sample. Pentane was occasionally added after the cleaning stage to replace any pentane that may have been lost to evaporation. Although the added pentane helped to reduce the moisture, it also made the agglomerates softer and harder to handle. When an excessive dosage of pentane was added, the agglomerates tended to clump together and become stuck when feeding the centrifuge.

The data given in Table 4-7 shows that the moistures obtained by centrifugation using the original 100 mesh x 0 sample were in the 18-19% range. These moisture values are significantly

Table 4-7. Reduction in moisture based on centrifuge sample.

Spin Time (sec)	Moisture (%)	Moisture Reduction (%)
original 100 mesh x 0 with 49% -325 mesh		
15	20.3	--
30	18.5	--
60	18.8	--
120	19.4	--
deslimed 100 mesh x 0 with 19% -325 mesh		
5	12.6	38.2
15	13.3	28.2
30	13.2	29.5
60	14.0	27.7
pulverized 80 mesh x 0 with 32% -325 mesh; soaked 1 min		
5	9.9	51.4
15	8.4	54.5
30	7.8	58.3
60	7.5	61.1
pulverized 80 mesh x 0 with 32% -325 mesh; soaked 3 days		
5	8.6	57.5
15	8.2	55.8
30	8.6	54.4
60	8.7	55.2

**Note: Moisture reduction was based on the difference from the original 100 mesh x 0. The minus 325 mesh values refer to the ultrafines in the concentrate.*

better than those obtained using the other techniques evaluated in this study (i.e., hand shaking and screening), particularly in light of the fact that the centrifuge provided very good coal recoveries (typically >90%). The data also show that a reduction in minus 325 mesh from 49% to 19% via sieving of the 100 mesh x 0 sample made it possible to further reduce the moisture from 18-19% down to 13-14%. By reducing the amount of fines, the moistures dropped an average of 56%.

The last two sets of data given in Table 4-7 show that switching to a slightly coarser, dry feed had the greatest impact on moisture. In fact, it was possible to reduce the moisture values down to single digit values when using the dry pulverized 80 mesh x 0 coal. Similar results were obtained regardless of whether the dry sample was soaked in water for just 1 minute prior to testing or soaked thoroughly for 3 days. The lowest moisture of 7.5% achieved using this sample represented a 61.1% reduction in moisture as compared to the original sample. For reference, Tables 4-8 through 4-10 contain summaries of the operating conditions, ash content, recovery, and ash rejection for each of the three samples tested using the centrifuge technique. More detailed versions of the tables are also included in Appendix B.

Table 4-8a. Measured centrifugation results for 100 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Pentane (ml)	Centrifuge Drying		Ash Content (%)	
		Time (sec)	Speed (rpm)	Tailings	Concentrate
0.32	0	30	3280	79.70	4.11
0.32	1	30	3280	81.20	4.11
0.32	2	30	3280	77.69	4.00
0.32	0	60	3280	81.41	4.08
0.32	1	60	3280	79.12	3.93
0.32	2	60	3280	76.93	4.07
0.32	3	60	3280	75.73	3.74
0.32	2	60	3280	73.78	3.82
0.33	2	15	880	75.65	3.95
0.33	2	30	880	83.41	4.15
0.33	2	60	880	81.98	4.05
0.33	2	120	880	84.94	4.15
0.32	2	15	3280	82.05	4.00
0.32	2	30	3280	80.17	3.87
0.32	2	60	3280	81.04	3.89
0.32	2	120	3280	81.06	3.92
0.31	2	15	2040	79.00	0.90
0.31	2	30	2040	80.76	3.92
0.31	2	60	2040	85.15	4.04
0.31	2	120	2040	82.44	4.08

**Note: All of these tests were conducted with 900 ml at 6.0% solids. The variations in mass ratio were due to changes in feed ash; 17.2 ml of pentane were used with each test. The pentane listed above was added after the agglomerates were cleaned. The centrifuge was still during feeding except for the eighth test which was fed at 880 rpm.*

Table 4-8b. Calculated centrifugation results for 100 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Yield (%)		Recovery (%)		Rejection (%)	Moisture (%)
	Cleaning	Centrifuge	Cleaning	Centrifuge		
0.32	54.1	100	84.8	100	94.3	21.1
0.32	55.0	100	86.2	100	94.2	19.7
0.32	52.8	100	82.8	100	94.6	19.1
0.32	55.1	100	86.4	100	94.2	19.9
0.32	53.7	100	84.2	100	94.6	19.4
0.32	52.4	100	82.1	100	94.5	19.4
0.32	51.3	100	80.7	100	95.0	17.5
0.32	50.0	100	78.6	100	95.1	20.7
0.33	47.8	100	78.3	100	95.4	23.6
0.33	53.0	100	86.7	100	94.7	24.2
0.33	52.1	100	85.3	100	94.9	21.8
0.33	53.9	100	88.2	100	94.6	21.0
0.32	54.3	100	86.4	100	94.5	20.3
0.32	53.1	100	84.6	100	94.8	18.5
0.32	53.7	100	85.4	100	94.7	18.8
0.32	53.7	100	85.5	100	94.7	19.4
0.31	54.7	100	85.1	100	98.6	19.4
0.31	57.9	100	87.3	100	93.7	19.6
0.31	60.3	100	90.7	100	93.3	20.5
0.31	58.9	100	88.7	100	93.4	21.6

**Note: The yields were calculated based on ash content. The centrifuge yield and combustible recovery are 100% because there were no measureable tailings. The ash rejection refers to the cleaning stage since no tailings were produced in the centrifuge. The concentrate for the cleaning stage calculations include the centrifuge concentrate and the material lost during centrifugation.*

Table 4-9a. Measured centrifugation results for screened 100 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Centrifuge Drying		Ash Content (%)	
	Time (sec)	Speed (rpm)	Tailings	Concentrate
0.34	5	3280	89.09	3.90
0.34	15	3280	88.53	3.91
0.34	30	3280	88.88	3.94
0.34	60	3280	88.99	3.91

**Note: All of these tests were conducted with 600 ml at 6.1% solids. No extra pentane was added after the agglomerates were cleaned.*

Table 4-9b. Calculated centrifugation results for screened 100 mesh x 0 flotation feed.

Mass Ratio (Pentane:Coal)	Yield (%)		Recovery (%)		Rejection (%)	Moisture (%)
	Cleaning	Centrifuge	Cleaning	Centrifuge		
0.34	81.7	100	97.5	100	83.6	12.6
0.34	81.6	100	97.4	100	83.6	13.3
0.34	81.7	100	97.5	100	83.5	13.2
0.34	81.7	100	97.5	100	83.6	14.0

**Note: The yields were calculated based on ash content. The centrifuge yield and combustible recovery are 100% because there were no measureable tailings. The ash rejection refers to the cleaning stage since no tailings were produced in the centrifuge. The concentrate for the cleaning stage calculations include the centrifuge concentrate and the material lost during centrifugation.*

Table 4-10a. Measured centrifugation results for pulverized 80 mesh x 0 clean coal.

Mass Ratio (Pentane:Coal)	Centrifuge Drying		Ash Content (%)	
	Time (sec)	Speed (rpm)	Tailings	Concentrate
coal soaked 1 minute				
0.21	5	3280	76.36	8.61
0.21	15	3280	79.30	8.02
0.21	30	3280	60.67	7.78
0.21	60	3280	40.45	7.71
coal soaked 3 days				
0.21	5	3280	77.08	6.59
0.21	15	3280	79.73	7.30
0.21	30	3280	84.69	7.21
0.21	60	3280	79.21	7.33

**Note: All of these tests were conducted with 900 ml at 6.0% solids. No extra pentane was added after the agglomerates were cleaned.*

Table 4-10b. Calculated centrifugation results for pulverized 80 mesh x 0 clean coal.

Mass Ratio (Pentane:Coal)	Yield (%)		Recovery (%)		Rejection (%)	Moisture (%)
	Cleaning	Centrifuge	Cleaning	Centrifuge		
coal soaked 1 minute						
0.21	82.6	100	94.8	100	65.2	9.9
0.21	82.6	100	95.5	100	67.6	8.4
0.21	76.1	100	88.2	100	71.0	7.8
0.21	61.1	100	70.9	100	76.9	7.5
coal soaked 3 days						
0.21	83.2	100	95.3	100	70.3	8.6
0.21	84.6	100	96.2	100	66.5	8.2
0.21	85.5	100	97.3	100	66.6	8.6
0.21	84.5	100	96.1	100	66.4	8.7

**Note: The yields were calculated based on ash content. The centrifuge yield and combustible recovery are 100% because there were no measureable tailings. The ash rejection refers to the cleaning stage since no tailings were produced in the centrifuge. The concentrate for the cleaning stage calculations include the centrifuge concentrate and the material lost during centrifugation.*

Figure 4-9 shows the effect of centrifuge speed and spin time on moisture. Three different rotation speeds (880, 2040 and 3280 rpm) and four different spin times (15, 30, 60 and 120 seconds) were examined. Since no replicate tests were performed, each point in the plot represents the data for only one test run. As a result, the impact of spin time on product moisture is not apparent due to the large degree of scatter in the experimental data. Nonetheless, logic would suggest that a longer spin time should result in lower overall moisture values (i.e., there is no reason to believe that a sample could regain moisture by extending the spin time). On the other hand, there appears to be a visible decrease in moisture content as the speed was increased from the lowest value of 880 rpm to the highest value of 3280 rpm. The lower moistures can be attributed to an increase in centrifugal g-force with increasing rotation speed. The ash and fines (minus 325 mesh) contents for these samples are summarized in Table 4-11.

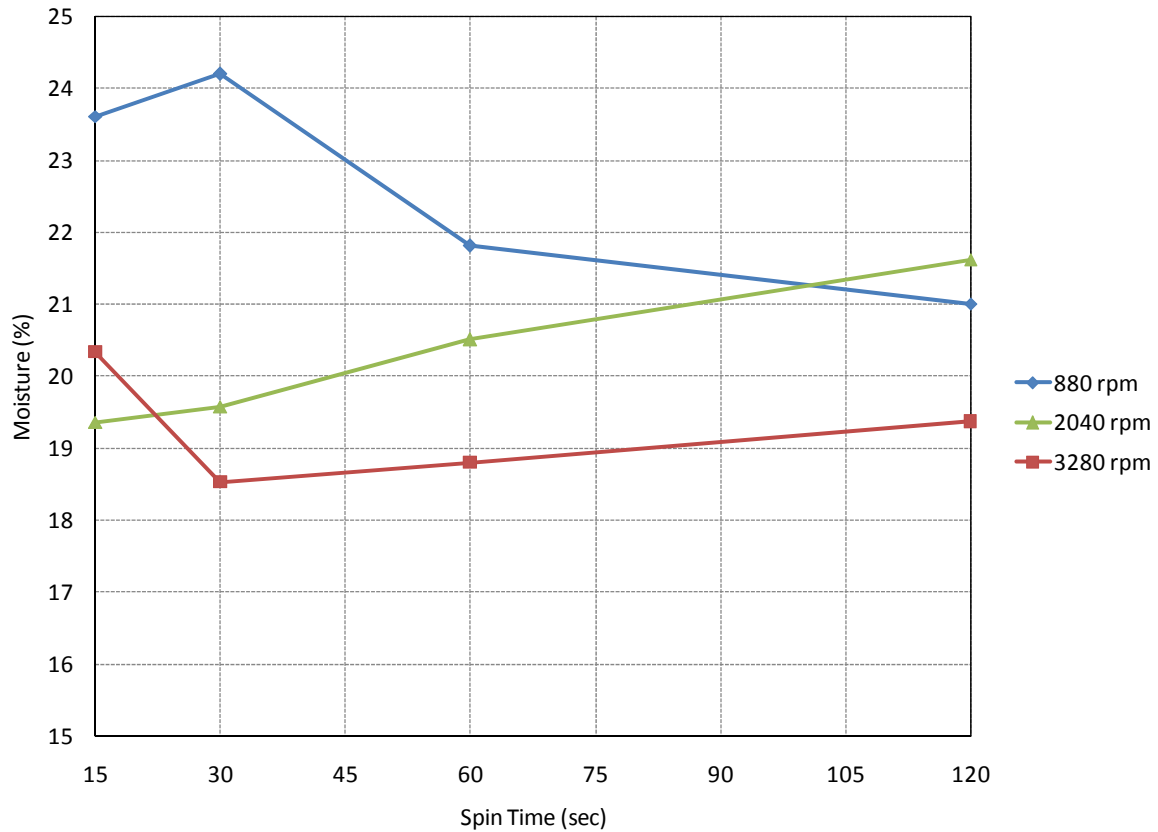


Figure 4-9. Effect of centrifuge speed and spin time on moisture for 100 mesh x 0 flotation feed.
**Note: Tests conducted with 0.31-0.33 pentane-to-coal mass ratio.*

Table 4-11. Summary of centrifugation samples.

Sample	Mass Ratio (Pentane:Coal)	Feed		Concentrate	
		-325 mesh (%)	Ash (%)	-325 mesh (%)	Ash (%)
original 100 mesh x 0	0.32	72	39.0	49	3.9
deslimed 100 mesh x 0	0.34	28	19.5	19	3.9
pulverized 80 mesh x 0	0.21	34	19.4	32	8.0

**Note: This contents of ash and ultrafines were calculated on the basis of averaged data obtained from Microtrac-R and proximate ash analyses conducted on three selected samples from the centrifuge tests.*

Finally, the data plotted in Figure 4-10 summarizes the effects of particle size and spin time on cake moisture for a fixed rotation speed of 880 rpm. The data clearly shows that moisture decreases sharply with increasing particle size. For the 100 mesh x 0 sample, a reduction in the ultrafines content reduced the moisture from about 18-20% down to 12-14%. A further reduction down to single-digit values in the 7-9% moisture range was obtained using the 80 mesh x 0 coal. It is interesting to note that this sample actually contained a higher percentage of minus 325 mesh solids than the deslimed 100 mesh x 0 sample (Figure 4-11). The data also tends to suggest that both the spin time and soaking time (for the case of the 80 mesh x 0 sample) has a comparatively low impact on the final moisture content.

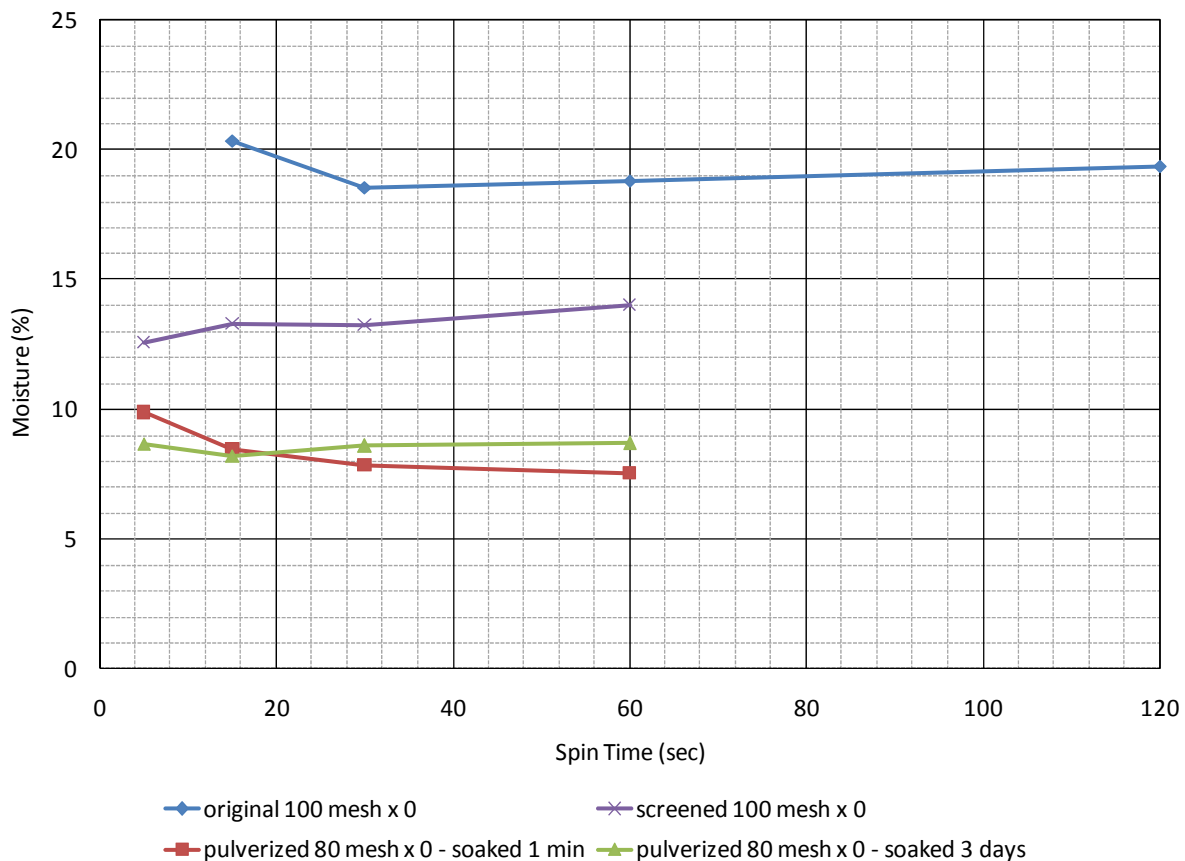


Figure 4-10. Effect of centrifuge sample on moisture.

**Note: Tests conducted with pentane-to-coal ratios of 0.32-0.34 for the 100 mesh x 0 samples and 0.21 for the pulverized 80 mesh x 0 samples.*

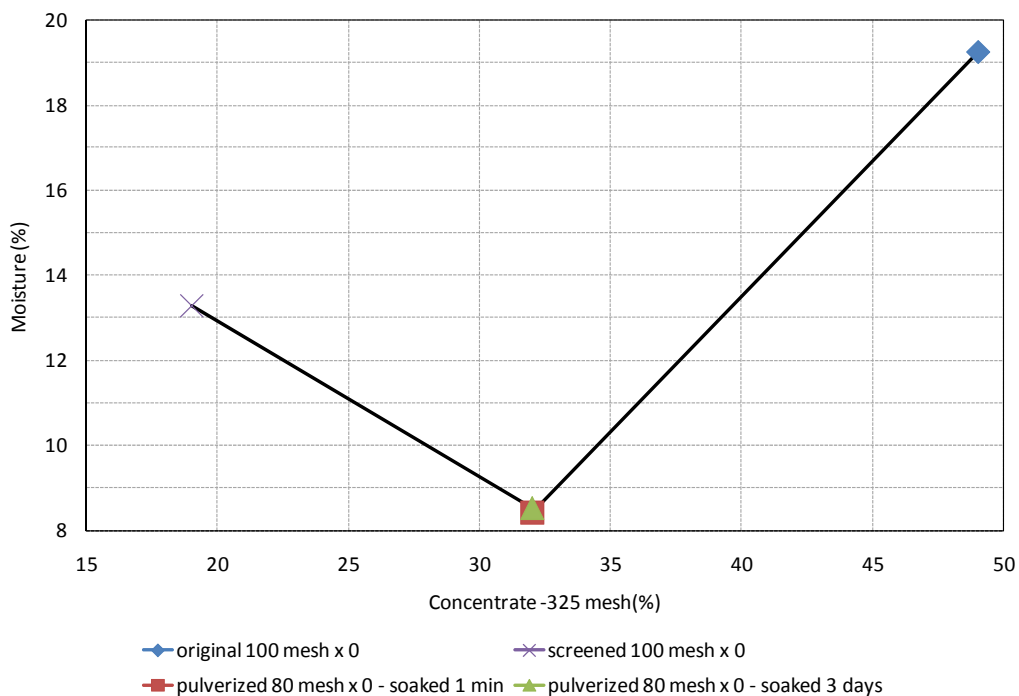


Figure 4-11. Effect of ultrafines on moisture.

**Note: Tests conducted with pentane-to-coal ratios of 0.32-0.34 for the 100 mesh x 0 samples and 0.21 for the pulverized 80 mesh x 0 samples.*

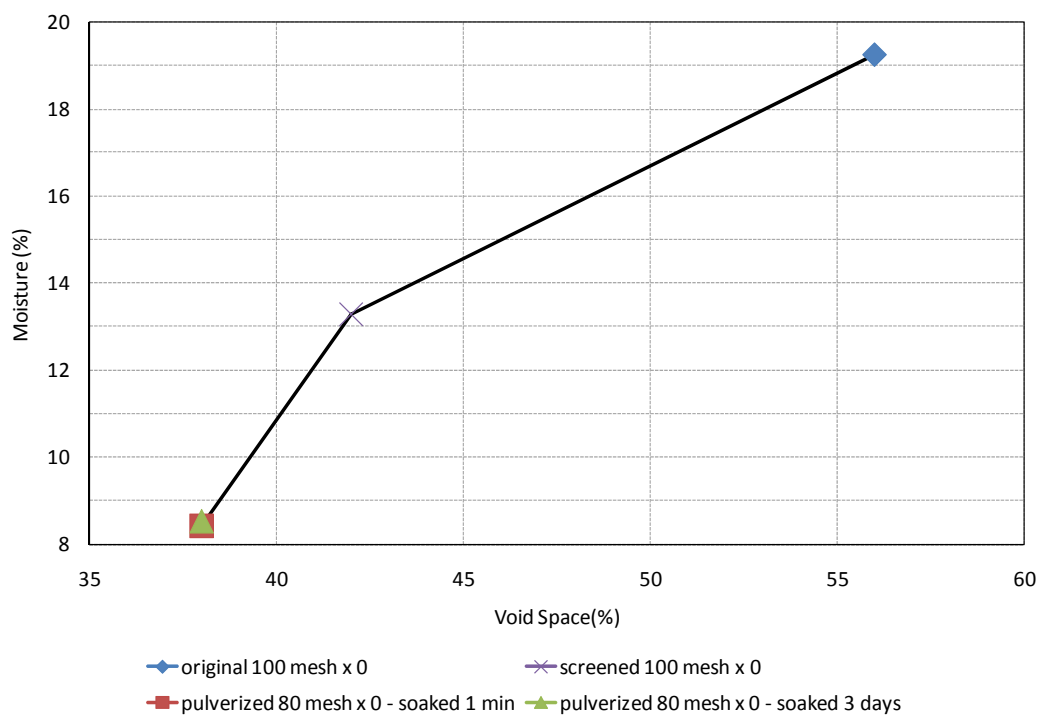


Figure 4-12. Effect of void space on moisture.

**Note: Tests conducted with pentane-to-coal ratios of 0.32-0.34 for the 100 mesh x 0 samples and 0.21 for the pulverized 80 mesh x 0 samples.*

It is interesting to note that though moisture does not appear to be directly related to ultrafines and surface area (Figure 4-11), which is unusual for a fine dewatering method, moisture does appear to have a semi-linear relationship to void space (Figure 4-12). In other words, the smaller the void space and available volume for water, the lower the moisture. However, this apparent dependence needs further investigation to ensure that void space is calculated correctly. It is currently based on the packing in a filter cake. It should also be noted that the pentane-to-voids volume ratio is higher for the 80 mesh (.67) than the 100 mesh (.52) due to different packing. The 80 mesh sample has the least available volume for water to fill. This may be a controlling factor instead of just void space.

4.4.4. Filtering

The use of vacuum filtration was also examined as a possible method for dewatering and recovering coal-pentane agglomerates. The filtration tests were performed using all three of the samples examined in the centrifugation tests (i.e., original 100 mesh x 0 sample, deslimed 100 mesh x 0 sample, and pulverized 80 mesh x 0 sample). As shown in Table 4-12, the filtration of the spherical coal-pentane agglomerates resulted in moistures varying from 20.1 to 32.0% moisture. Lower moisture values were obtained for samples with less ultrafines and more coarse solids. Also, no material was lost to tailings with this dewatering technique since pre-cleaned agglomerates were utilized. Unfortunately, the moisture values obtained from the filtration tests were considerably larger than those obtained in the centrifugation experiments. As such, this method is not as effective for agglomerate recovery and separation as centrifugation. The higher

Table 4-12. Summary of agglomerate filtration results.

Run	Sample	Thickness (mm)	Dry (g)	Filtration Time (sec)	Moisture (%)
c1	original 100 mesh x 0	15	15.71	2	32.0
c2	original 100 mesh x 0	12	14.66	2	27.2
c4	original 100 mesh x 0	13	15.88	3	27.3
c5	pulverized 80 mesh x 0	17	20.43	3	20.1
c6	pulverized 80 mesh x 0	20	21.11	3	21.7
c7	deslimed 100 mesh x 0	20	23.32	2	24.4

**Note: All tests were conducted with 500 ml and 60 seconds of drying time.*

moistures are attributed to the presence of coal-covered water droplets that were visually observed within the cake after vacuum filtration. Tests conducted using the centrifuge did not appear to suffer from the same problem.

4.4.5. Displacement

The displacement method of dewatering consisted of filtering coal slurry with large amounts of pentane. Unlike the previous techniques, oil agglomeration was not involved in displacement since pentane was added on top of a homogenous coal slurry phase being filtered. In this particular set of tests, only the slurry prepared from the dry pulverized 80 mesh x 0 Tom's Creek clean coal was used. The results obtained for this method of dewatering are provided in Table 4-13. Most of the tests conducted using pentane resulted in moistures values in the 22-28% range, while those conducted without pentane were substantially higher in the 31-34% range. There was no measureable material loss, so the recovery for this dewatering technique is 100%. The lowest moisture of 19.7% obtained using pentane was achieved by increasing the vacuum pressure and drying time to their largest values (i.e., 24 mmHg and 1 minute, respectively); however, the centrifuge method provided lower moistures in the single-digit range when used to treat the same sample.

Table 4-13. Summary of displacement results for pulverized 80 mesh x 0 clean coal.

Pentane	Thickness (mm)	Pressure (mmHg)				Filtering time (min)		Drying time (min)	(%)	
		A	B	C	D	Water	Total		Liquid	Moisture
yes	6	24	--	--	6	2.75	5.67	0.5	28.43	28.43
yes	6	24	21	8	8	2.58	6.63	0.5	25.75	22.21
yes	7	24	20	10	8	1.25	5.42	1.0	22.59	19.68
yes	8	20	15	8	7	2.08	8.50	0.5	27.66	27.66
yes	6	20	19	8	8	2.25	6.28	1.0	26.93	25.17
yes	7	12	10	8	6	4.33	9.83	0.5	27.87	26.01
yes	6	12	10	10	10	3.50	9.50	1.0	27.26	26.27
no	6	24	19	--	14	--	3.17	1.0	34.58	34.58
no	5	20	21	--	9	--	2.67	1.0	31.06	31.06
no	6	12	12	--	5	--	4.13	1.0	33.62	33.62

**Note: The first result displays moisture if all the water beads are not removed prior to the drying time; it was redone in the next test. The pressures correspond to different liquid phases: A-beginning of the test (water phase), B-end of the water phase, C-beginning of the pentane phase, and D-beginning of the air phase.*

4.5. Comparison with Conventional Cleaning and Dewatering

The original Tom's Creek 100 mesh x 0 flotation feed sample was subjected to conventional cleaning and dewatering tests in order to obtain data that could be used as a baseline to compare with data obtained from the various pentane dewatering tests. A flotation test conducted using a Denver Model D-12 laboratory flotation cell was used to evaluate the cleanability of the sample. Flotation tests were performed using the release analysis procedure to obtain the best possible separation results for flotation. Dewatering of flotation concentrate was assessed by performing laboratory vacuum filter tests. The dewatering tests were performed using two different dewatering aids (reagents RV and RW) in an attempt to achieve the lowest possible moistures for comparison to the pentane displacement technology.

The results of the laboratory flotation release analysis tests are plotted in Figure 4-13. As shown, the release analysis curve suggests that ash contents below 5% are attainable in a “perfect” flotation process. Very efficient real-world processes, such as column flotation, would be expected to provide ash contents that were slightly above this level. Fortunately, the pentane agglomeration tests also reduced ash contents to values that matched or exceeded this level of separation performance (see Table 4-8a). In fact, many of the pentane tests provided concentrates containing less than 4% ash. This finding indicates that this approach would be a very selective

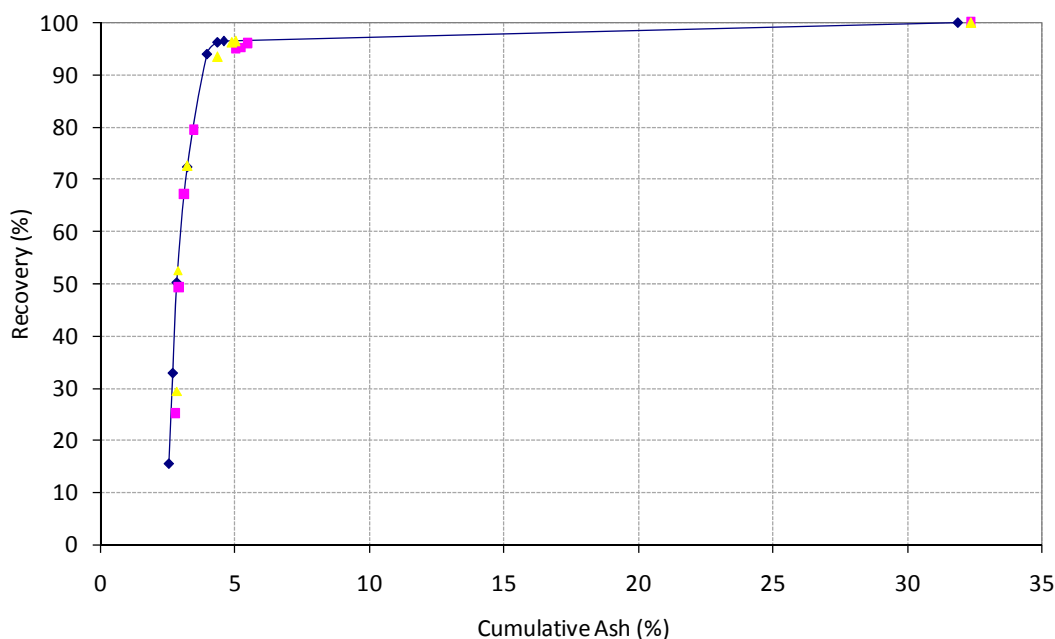


Figure 4-13. Release analysis for 100 mesh x 0 flotation feed.
**Note: Data from three different flotation trials are displayed.*

Table 4-14. Results for conventional vacuum filtration using flotation concentrate.

Dewatering Aid	Time (min)			Thickness (mm)	Dry (g)	Moisture (%)
	Conditioning	Formation	Drying			
--	0	5.8	2	7	18.44	29.2
RW	2	2.5	2	8	18.27	24.8
RV	2	3.7	2	10	20.16	30.5

**Note: Tests were conducted with 250 ml samples using a dewatering aid active-to-diesel ratio of 0.5. The ratio corresponds to 3 lb/ton or 39 μ l of prepared solution.*

process for separating unwanted mineral matter from valuable coal.

A more significant advantage of the pentane agglomeration process is the ability to provide cleaned products that are relatively low in moisture. The test results summarized in Table 4-14 show that conventional vacuum filtration was not capable of providing moisture contents lower than about 30% by weight. The addition of a dewatering aid (reagent RW) made it possible to further reduce the moisture down to about 25%, while another dewatering aid (reagent RV) did not significantly impact the resultant moisture. These values are significantly worse than the 18-20% moisture achieved using pentane agglomeration combined with centrifugation (see Table 4-7). The combined ability of the agglomeration process to reduce both ash and moisture make it a very attractive alternative for coal upgrading.

The vacuum filter technique was also utilized to perform a void space analysis on the concentrates from hand shaking and centrifuge agglomeration tests. Each sample was cleaned by agglomeration and then re-homogenized into slurry form. The compact cake produced by filtration was used to estimate the packing of coal particles within the agglomerates.

Table 4-15 shows the void spacing data obtained for the agglomerated samples produced by the centrifuge technique. As shown, the original 100 mesh x 0 flotation feed sample gave the largest void space (59.9% average), while the pulverized clean coal gave the smallest void spacing (38.0% average). The deslimed 100 mesh x 0 sample gave a void spacing of 42.3%, which is between these two values. It is interesting to note that the void spacing values fall in the same sequential order as the moisture values for these particular samples. This correlation suggests that the observed differences in moisture content for these three samples may be due to the variations in void spacing, particularly since no such correlation could be established

Table 4-15. Void space analysis for centrifuge concentrates.

Sample	Coal Volume (cm ³)	Thickness (mm)	Cake Volume (cm ³)	Void Volume (cm ³)	Void Space (%)
original 100 mesh x 0	12.3	9	28.1	15.7	56.0
original 100 mesh x 0	11.1	11	34.3	23.2	67.8
original 100 mesh x 0	12.3	9	28.1	15.7	56.1
average	11.9	9.7	30.1	18.2	59.9
pulverized 80 mesh x 0	18.2	8	24.9	6.8	27.2
pulverized 80 mesh x 0	15.9	10	31.2	15.2	48.9
average	17.0	9.0	28.1	11.0	38.0
deslimed 100 mesh x 0	18.0	10	31.2	13.2	42.3

**Note: The volumes were calculated with dry coal weights, a density of 1.31 g/cm³, thicknesses, and a diameter of 6.3 cm. The void space is a volume percentage.*

between moisture and the percentage of ultrafine (minus 325 mesh) solids in each of the three samples. Additional experimentation is recommended to further investigate this possibility.

For reference, Table 4-16 provides the void space data obtained for the sample of agglomerates recovered by hand shaking. An average void spacing of about 55% by volume was obtained for the 100 mesh x 0 sample used in these experiments. This void spacing value is very similar to that obtained using the centrifugation technique.

Table 4-16. Void space analysis for hand shaking concentrate from 100 mesh x 0 flotation feed.

Coal Volume (cm ³)	Thickness (mm)	Cake Volume (cm ³)	Void Volume (cm ³)	Void Space (%)
20.6	4	44.5	23.9	53.8
21.7	5	55.6	33.9	61.0
21.8	4	44.5	22.7	51.1
21.3	4.3	48.2	26.9	55.3

**Note: The last line contains the average values for the three tests. The volumes were calculated with dry coal weights, a density of 1.31 g/cm³, thicknesses, and a diameter of 11.9 cm. The void space is a volume percentage.*

5. DISCUSSION

Six methods of hydrophobic displacement were evaluated for their cleaning and dewatering capabilities: hand shaking, screening, air classification, centrifugation, filtration, and displacement. The first five methods all utilize oil agglomeration to perform displacement of water during the cleaning stage. The following extraction methods then removed the oil-coated agglomerates from the remaining bulk water. Only the last method, displacement, performed hydrophobic displacement during the dewatering stage.

5.1. Moisture Determination

Correct moisture determination is a critical step in this process. Since dewatering is one of the main goals, inaccurately portraying moisture content defeats the purpose of this project. The difficulty arises in reporting moisture due only to water, not pentane and water. Fortunately, the two clear liquids have very different evaporation rates. Though they have other widely distinguishable properties such as their refractive indices, evaporation rates are easy to record in real time and do not require a closed system.

This problem was identified early in the project. Originally, moistures were reported after twenty minutes at 40°C. However, this procedure had a major flaw: the time for pentane to evaporate varies widely with dosage and surface area. For example, with low dosages pentane will evaporate before the concentrate is even placed in the oven. Low dosage tests will therefore report a lower moisture than realistic because water will be evaporated for twenty minutes. Conversely, with large dosages twenty minutes is not enough time to remove all of the pentane. The reported moisture will be much higher than the real water-only moisture. This skews the moistures in favor of low oil dosages. Only the moisture due to water is important because in a commercial application, pentane will be evaporated and recovered.

The solution to the problem was to implement a weighing platform. The change-in-weights were plotted in real-time, and the moisture was chosen where the curve began to level off as seen in Figure 4-4. It should be noted that this procedure does remove some water. Unfortunately, though water evaporates more slowly than pentane, there is no way to prevent it from evaporating while the pentane does; however, in a commercial application it is still preferable to remove this negligible amount of water in favor of recovering all of the pentane due to the high cost of the oil.

Some error may be included in the moisture due to the location of the point taken on the graph. The leveling point of the weight versus time curve, where the evaporation rate became constant, was chosen to ensure that all of the pentane was removed. Traditionally the intersection of the two legs of the weight curve would be chosen. Appendix C contains more detail on why moisture was based on the end of the elbow instead of the center. This location does provide a slightly lower moisture. It should be noted that the needed evaporation times varied from zero to about fifteen minutes, and the ideal dosages for hand shaking and centrifugation only required zero to two minutes. The evaporation rate for water afterwards was about .1 g/min, so the loss of water during pentane evaporation was negligible compared to the sample weight utilized.

5.2. Agitation and Mixing

5.2.1. Hand Shaking

All of the oil agglomeration was performed by shaking slurry and pentane in an enclosed container. Unfortunately, hand shaking is not a consistent method; the force and shaking time are difficult to control. In order to offset some of this error, repetitive testing was implemented. Also, though spherical agglomerates were fully formed after one minute of shaking, shaking was continued for a full five minutes to ensure complete agglomerate formation in each test. Fortunately, the agglomerates formation time and size remained consistent according to their oil dosages. Figure 4-5 shows the variation in agglomerate diameter with dosage.

The most important factor during agitation was the oil dosage. It controlled the appearance of the agglomerates and the ease of handling them. The agglomerates varied from micro-agglomerates, or flocs, to spherical agglomerates to curds as the dosage increased. Further increase only resulted in a distinct pentane phase on top of the semi-solid agglomerates. The density of coal, about 1.3 g/cm^3 , was too great for coal to remain suspended in the 0.626 g/ml pentane, so the agglomerates usually sat in the top of the water phase.

Most of the dewatering techniques used spherical agglomerates. These hard, compact agglomerates formed with pentane-to-coal mass ratios of 0.21-0.34, depending on the coal's oxidation level and size distribution (i.e., the less oxidized pulverized coal required less oil than the screened flotation feed even though they had similar calculated surface areas). The spherical agglomerates were chosen for their durability and size. They were large enough, 1-2 mm, to provide channels for drainage but strong enough to maintain their shape under high g-forces.

Even after all pentane and moisture had been removed, the agglomerates maintained their shape until shaken.

The other main variable tested was the solids content of the feed slurry. Higher solid contents tended to speed the creation of agglomerates; however, similar size agglomerates formed for 6%, 15%, and 30% solids. It did not have a major impact on agglomerate appearance.

Finally, it is important that the feed be relatively clean of chemicals, especially cationic coagulants. Chemicals used to clarify water interfered with agglomeration and prevented spherical agglomerates from forming; therefore, most of the pond samples experienced difficulties due to the lingering presence of ions.

5.2.2. Impeller Mixing

Impeller mixing was tested in order to provide a more consistent method of agglomeration. Unfortunately, it was difficult to create such a small sealed mixing chamber. Heptane, which evaporates more slowly than pentane, was agitated using a Denver cell to demonstrate that similar spherical agglomerates formed with impeller mixing; however, once this was proved, it was decided to delay impeller mixing until a continuous, sealed process was designed.

5.3. Pellet Extraction and Dewatering

5.3.1. Shaking

Pellet extraction by hand shaking dominated the original dewatering methods tested. The lowest average moisture, 16.2%, occurred at an $M_p:M_c$ of .32. This moisture was obtained with the original Tom's Creek 100 mesh x 0 flotation feed with a solids content of 6.3%. Increasing the solids content to 14.8% raised the moisture by 14%. Oxidation and size distribution had an even greater impact. After the slurry sat for four months, the moistures increased up to 137%, depending on the dosage used. Using a screened 325 mesh x 0 sample increased the moisture by 70%. Most of the moisture in these samples was due to free water droplets being shaken out with the concentrate. The agglomerates also had to bounce up the damp glass of the container which increased the opportunity for agglomerates to pick up small water droplets. Tiny pin-pricks of water were occasionally seen on the agglomerates.

The hand shaking method has a high inherent error, hence the reason repetitive testing was utilized. Since shaking was not consistent, the decision on when to stop was arbitrary. This led to large variations in combustible recovery as material was left inside the container. The coal inside the container was considered middlings based on its higher water content even though it had a similar ash content. Since yield varied greatly depending on the amount of water shaken out, concentrate weights also varied greatly. Small concentrate weights could produce large errors as a single droplet would have a large impact on the moisture. Finally, the samples were shaken into a metal pan to prevent agglomerate loss, and the agglomerates were later transferred into a small glass dish for platform weighing. Though care was taken to transfer all of the water to the dish, loss was impossible to prevent. Droplets could easily smear on the metal and evaporate.

An attempt was made to mechanize this method, but the irregular movement was difficult to duplicate; eventually the method was abandoned in favor of the higher g-forces produced by a centrifuge.

5.3.2. Screening

This unique screening method was first used by Kerem Eraydin at Virginia Tech. The goal of the method is to screen out dry coal while coalescing water droplets on the surface of the screen. Coatings of coal protected the coalesced water from wetting the screen. This method has the ability to produce single digit moisture; however, the associated recoveries are also single digit. The method was not deemed practical due to the low recoveries and process difficulties. The process suffers from blinding due to fine dry particles, and there is always a risk of wetting the screen which would further encourage blinding. It would be difficult to prevent screen wetting in a plant environment.

5.3.3. Air Classification

Air classification was briefly examined as a method of pellet extraction. No quantitative testing was completed. This method utilized spherical agglomerates, and air was used to remove the top agglomerates sitting above the water. Unfortunately, the majority of the agglomerates sat in the water phase, so it was difficult and slow to blow out individual agglomerates. Also, there

was always a risk of blowing out water droplets. This method only removed the agglomerates from the main water phase. It did not exert any force to remove the tiny water droplets attached to the agglomerates.

5.3.4. Centrifugation

Dewatering by centrifugation was one of the main methods examined. Centrifugation provided a means to strip the agglomerates of their tiny water droplets by applying g-forces as high as 447 g's. The spherical agglomerates were surprisingly durable under the high pressure and maintained their shape. This was critical in order to maintain the pores for water to drain. A dosage higher than a $M_p:M_c$ of 0.21 to 0.34 would have resulted in agglomerates too soft; they would have deformed, effectively trapping the moisture. Centrifugation provided moistures as low as 7.5% with a combustible recovery of 70.9%, ash rejection of 76.9%, and concentrate ash of 7.71% (Table 4-11). These results were based on the pulverized 80 mesh x 0 Tom's Creek clean coal. The screened 100 mesh x 0 sample provided a higher moisture at 13.2% but a more favorable recovery of 97.5%, rejection of 83.5%, and ash of 3.94%. These recoveries represent the entire process, including cleaning. The centrifuge recoveries are about 100% since the centrifuge tailings did not contain measureable solids.

Oxidation, size distribution, and g-force all played major roles in centrifugation. The dry pulverized 80 mesh x 0 clean coal resulted in the lowest moistures (Figure 4-10). It was dry ground, unlike the flotation feed, and had been stored in a freezer to slow oxidation. Though it contained more fines than the screened 100 mesh x 0 sample, it still had a lower moisture by 5.7% for a 43% difference. Both of the samples had similar estimated surface areas. The original 100 mesh x 0 flotation feed produced the worst moistures, 18.5% being the lowest. This sample contained 72% minus 325 mesh versus the 34% and 28% in the previous samples. This resulted in a two orders of magnitude increase in surface area based on an estimation of spherical particles. Though moisture was not directly related to ultrafine content, it appears to be strongly related to void space and pentane-to-void volume ratio. The 80 mesh sample had the lowest moisture and void space and the highest pentane-to-void volume ratio (Figure 4-12). Finally, Figure 4-9 shows that an increase in speed and g-force tend to produce dryer cake. As speed increased, the cake became thinner and taller (Figure B-6). At slow speeds, the agglomerates simply filled the trough around the insert.

It is also critical that the spherical agglomerates are cleaned before undergoing centrifugation. With a homogenous coal slurry, the light weight ultrafines can produce a slime coating on the inside of the cake. In the case of pentane-agglomerated coal, the slimes form on the outside of the cake since the agglomerates float. This impermeable layer prevents the agglomerates from drying correctly, so it is essential that the agglomerates are thoroughly cleaned.

There are several problems with centrifugation. First, though the centrifuge was meant to be sealed, small amounts of pentane leaked from the top bearing under high pressures. Also, some pentane evaporated when the sample was open to atmosphere during pouring. Pentane evaporation allows coal to readsorb water and weakens the agglomerate structure. Without pentane, the agglomerates will powderize at high speeds. This powder is more difficult to dewater and may be lost between the centrifuge bars.

Second, in order to produce a relatively even cake, an insert is required in the middle of the centrifuge. The blank forced the agglomerates into a trough. Without the insert, the agglomerates would form a wide wedge at the base. Water drains too quickly, and the agglomerates have no time to travel up the sides of the chamber on the water. Increasing the speed did not make a difference. Since thin, even cakes traditionally provide lower moistures, the insert was added and the centrifuge was fed while still. It was fed while off to provide an opportunity for the agglomerates to fill the mold.

5.3.5. Filtering

Filtration of spherical agglomerates was meant to provide a baseline of moistures to compare centrifugation against. It resulted in moistures varying from 20.1 to 32%. Moisture was reduced by removing ultrafines and preventing oxidation (Table 4-12). No material was lost to tailings with this dewatering technique since pre-cleaned agglomerates were utilized.

5.3.6. Displacement

Dewatering by displacement does not utilize oil agglomeration; however, it is based on a similar principle in which oil rejects water. In each test, a coal slurry was filtered with a top layer of pentane. In theory, pentane should displace the last droplets of water as it filters through the

cake; however, it did not work as intended, and resulted in moistures of 20-35%. This is a standard range of moistures for filtration; therefore, pentane did not appear to have any impact.

There were a couple reasons why displacement did not work as intended. First, even when the pentane was poured slowly, coal-coated droplets of water tended to form and sit on top of slurry-oil interface. As filtration occurred, these droplets came to rest on the cake surface while the pentane filtered around them. They slowly filtered after the pentane was gone. Second, pentane likely filtered through the path of least resistance. While it is true that pentane rejects water, the solids interfere with the interface. Instead of a flat interface filtering down, the solids provide voids for droplets of water to exist. Once a path has been made, it is easier for the pentane to filter around these droplets than to displace them all.

5.4. Conventional Cleaning and Dewatering

Conventional cleaning and dewatering were performed to baseline all of the oil agglomeration testing. They were only performed on the original 100 mesh x 0 sample. The release analysis indicated that Tom's Creek is an easy-to-clean, black and white coal with good liberation; however, it was not as easy to dewater. Even with a dewatering aid, the lowest obtained moisture was 24.8%.

Conventional vacuum filtration was also used to calculate the void space in each of the three main coal samples. The cake simulated the compact coal composing the agglomerates, and the calculated void space was used to determine the pentane to void space ratios.

5.5. General Comparison of Methods

Of the six dewatering methods considered, centrifugation consistently provided the lowest moistures and highest recoveries. It achieved the goal of single digit moisture, unlike the other methods. Centrifugation produced a 7.5% moisture and 70.9% combustible recovery with the 80 mesh x 0 sample. Though the moisture increased with the other samples, recovery was generally in the high eighties and nineties. Hand shaking's lowest moisture was 16.2% with a recovery of only 53.3%. The lowest moistures occurred at a dip in the recovery curve, unlike centrifugation which had a relatively consistent recovery. Most of the variations in centrifuge recovery were due to poor feeding; the centrifuge feed tube became blocked if it was fed too

quickly. These variations could easily be eliminated to maintain high recovery. Unfortunately, low recovery cannot be eliminated for hand shaking.

Filtration and displacement were the next methods. They both produced moistures from 20-35%. These are standard vacuum filtration values and do not represent an improvement from conventional methods. Screening was the least practical of the five tested methods. Though it provided low, single-digit moisture, the recoveries were also single digit. The low recoveries and equipment difficulties prevent it from being a viable option. Finally, air classification never underwent quantitative testing. It needed special equipment and was deemed impractical in light of the centrifugation option.

Though moisture varied between the methods, they share some common points. In general it appears as though spherical agglomeration is preferable because it enables fine coal to be treated as coarse. Oxidation plays a key role in moisture elevation. Fresh samples contained significantly less water. Finally, size distribution also impacted the moisture; an increase in ultrafines resulted in higher moistures. However, oxidation was much more important. For example, in centrifugation the original 100 mesh x 0 sample was cleaned from 72% to 49% minus 325 mesh, the screened 100 mesh x 0 cleaned from 28% to 19% minus 325 mesh, and the pulverized 80 mesh x 0 from 34% to 32% ultrafines. The screened sample had the least ultrafines, yet the unoxidized 80 mesh x 0 provided the lowest moistures.

Ash content did not appear to have an impact on moisture, which is reasonable considering it was being cleaned before dewatering. The dry pulverized sample had a feed ash of 19% and a concentrate of 8%. It had the highest concentrate ash yet the lowest moisture. In contrast, the original 100 mesh x 0 had a feed of 38% and concentrate of 4% ash while the screened 100 mesh x 0 had a feed of 19% and similar concentrate ash of 3.9%.

The ability to achieve single digit moisture with centrifugation indicates that oil, not water is the bridging liquid; therefore, there is not an inherent amount of water needed for agglomeration to take place. The majority of the moisture occurring in the processes originated as free water droplets trapped between the agglomerates or as small droplets attached to the outside of the agglomerates. However, some moisture may remain in the agglomerates since the coal-to-void space volume ratios were only .50 to .65 for spherical agglomeration. This assumes the agglomerated coal was not packed more tightly than the vacuum cake. Unfortunately, the oil dosage cannot be increased to fill this void space because the agglomerates become soft and trap

large globules of water between themselves. It should be noted that some of the volume ratios are higher than reasonable; therefore, the void space determination may be inaccurate and should be re-examined.

The original thermodynamics calculated for this project indicated that dewatering should be spontaneous for any contact angle greater than 90 degrees (Sohn, 1997). Accordingly, the concentrates should contain no moisture since pentane's contact angle with coal in water is about 106 degrees. Though the thermodynamics examining the end states of the process are correct, they fail to take into consideration contact angle hysteresis. Hysteresis refers to the range of contact angles existing between the retreating and advancing angles. The measured contact angle likely represents an advancing angle; however, during draining a receding contact angle will occur. This angle may be lower than 90 degrees and would account for the inability of pentane to remove all water. With this lower contact angle, the process does not produce negative free energy. It is no longer thermodynamically favorable and spontaneous.

6. GENERAL SUMMARY

This purpose of this project was to seek alternatives to conventional fine to ultrafine coal cleaning and dewatering using hydrophobic displacement by pentane to produce moistures less than ten percent. Six dewatering methods were examined: hand shaking, screening, air classification, centrifugation, filtration, and displacement. The first five methods utilized oil agglomeration in order to perform hydrophobic displacement during the cleaning stage. The dewatering stage focused on removing the resulting oil-covered agglomerates from bulk water. These methods sought to use oil agglomeration as a basis for a single solid-solid, solid-liquid process which combined cleaning and dewatering. The last method, displacement, was the only process which performed hydrophobic displacement during the dewatering stage.

The desired bench-scale and batch testing were completed and succeeded in meeting the moisture requirement. Several pertinent conclusions are included below:

1. Spherical agglomeration provided most durable agglomerates for dewatering. They essentially enabled fine coal to be treated as coarse coal. Spherical agglomerates of 1-2 mm in diameter were produced with ratios of $.21-.34 M_{\text{pentane}}:M_{\text{coal}}$, $.42-.70 V_{\text{pentane}}:V_{\text{coal}}$, and $.50-.67 V_{\text{pentane}}:V_{\text{void}}$.
2. A weighing platform provided a flexible way to determine water-only moisture when evaporation times varied.
3. Cleaning with spherical oil agglomeration and dewatering by centrifugation produced the best moistures and recoveries. Centrifugation with the pulverized 80 mesh x 0 Tom's Creek clean coal produced a moisture 7.5% with a combustible recovery of 70.9%, rejection of 76.9%, and concentrate ash of 7.71%. Recoveries were usually in the mid eighties to nineties, regardless of oil dosage.
4. Hand shaking produced moistures as low as 16.2%; however, low moistures were associated with a dip in recovery. There is a great deal of error in this process due to its arbitrary nature.
5. Filtration and displacement produced moisture of 20-35%, and they did not show an improvement in conventional methods.
6. Though screening could produce single-digit moistures, it was not a practical process. The recoveries were too low and the screens risked blinding.

7. Oxidation and size distribution influenced the effectiveness of the dewatering stage. Oxidation had the largest effect.
8. During centrifugation, moisture appeared to be directly related to void space and pentane-to-void volume ratios. These factors had a greater impact on moisture than the ultrafines content which usually controls fine coal dewatering.
9. The majority of water existed as free water droplets trapped between the spherical agglomerates or as tiny droplets attached to the outside of the agglomerates. Little water existed inside the agglomerates, confirming that oil acts as the bridging liquid.
10. Contact angle hysteresis may explain why not all of the water is spontaneously removed by pentane. Though the advancing pentane and coal contact angle in water was about 106 degrees, the receding angle was likely less than 90 degrees.

7. RECOMMENDATIONS FOR FUTURE WORK

There are five recommended trends for future work on this project. First, given the promising single-digit moistures produced by centrifugation with spherical agglomeration, a continuous version of this method should be explored. This would involve creating a closed system mixing chamber, cleaning chamber, and sealed centrifuge. Due to the high consumption of pentane in such a system, oil recovery by condensation should also be employed.

Second, considering the dependence of centrifuge moisture on void space, the void space determination method should be re-evaluated. Proctor testing should be performed to determine optimum void space and packing. Also, packing in pentane, not just water, should be examined.

Third, cleaning and dewatering by displacement should be re-examined. The theory is sound though the displacement-filtering method attempted here did not work. Instead, large amounts of oil should be used to create separate oil and water phases. Coal will gravitate to the interface, and if the surface area of this crowded interface is suddenly decreased, by jiggling with inverse triangular bars for example, the extra coal particles will be ejected into the pentane phase where they may be removed. This material should contain little to no water since each particle is completely enveloped in oil. Unfortunately, it takes very little agitation to create an emulsion or coal-coated water droplets in the pentane phase. The key will be to produce a gentle mechanical motion that can still handle large coal throughputs. Though a similar test tube method was attempted with dry coal in water and pentane and produced promising results, nothing was attempted with a coal slurry.

Fourth, multi-stage dewatering should be examined. This would provide more time for pentane to displace the remaining water. Also, as the coal is reworked, small water droplets may coalesce and become easier to remove.

Finally, it is critical that sample oxidization is minimized in order to produce low moistures. Fresh samples are also more representative of a preparation plant. Ideally, an enclosed pilot-scale process would be run at a site. If this proves difficult, coal may be ground in the lab, stored in a freezer, and mixed before each test. The samples should soak for a short time to encourage wetting before dewatering takes place.

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APPENDICES

A. Apparatus Appendix

Appendix A contains six mechanical drawings for the sealed centrifuge; they were all completed with Inventor software.

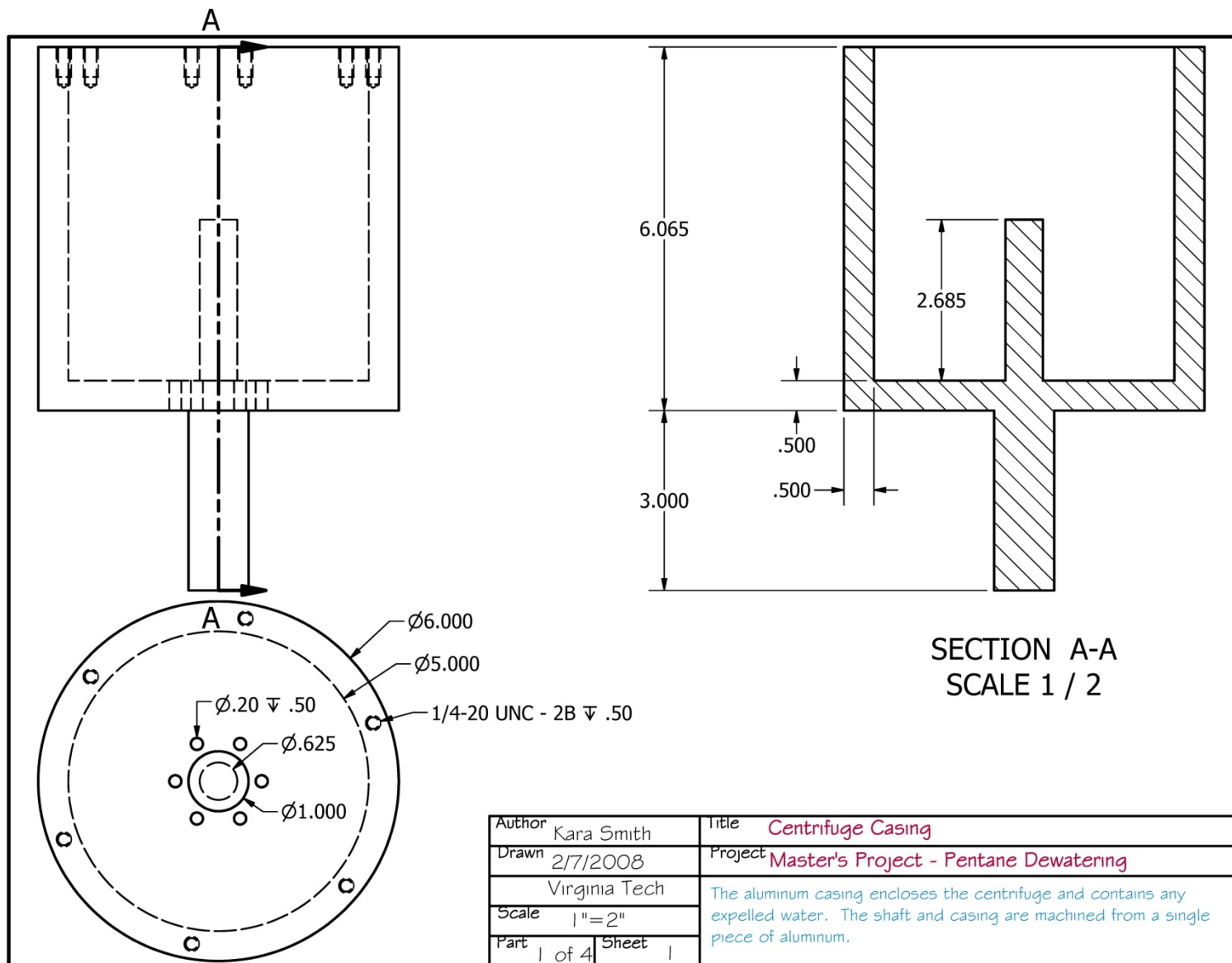


Figure A-1. Drawing of the centrifuge casing

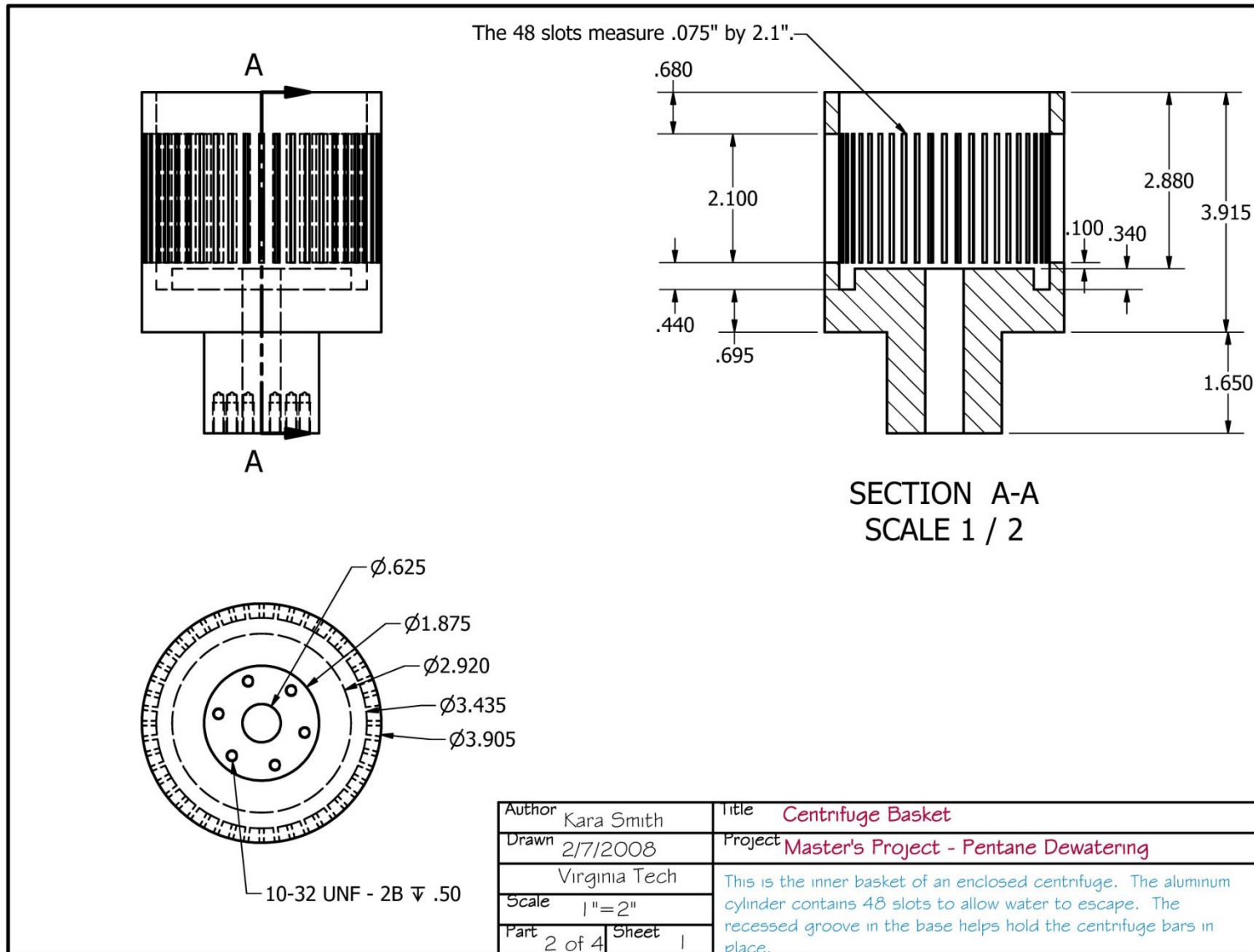


Figure A-2. Drawing of the centrifuge basket

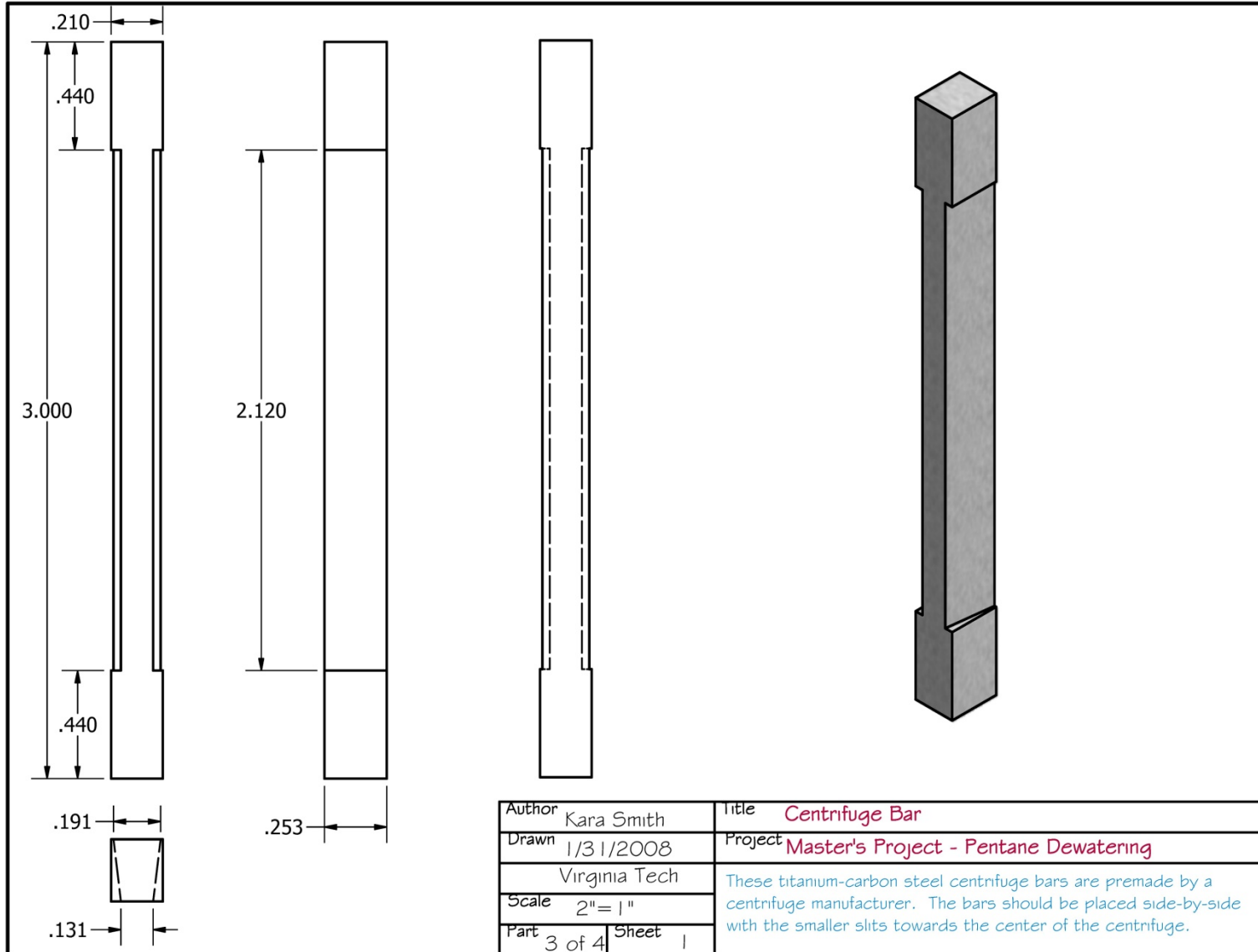


Figure A-3. Drawing of the centrifuge bar

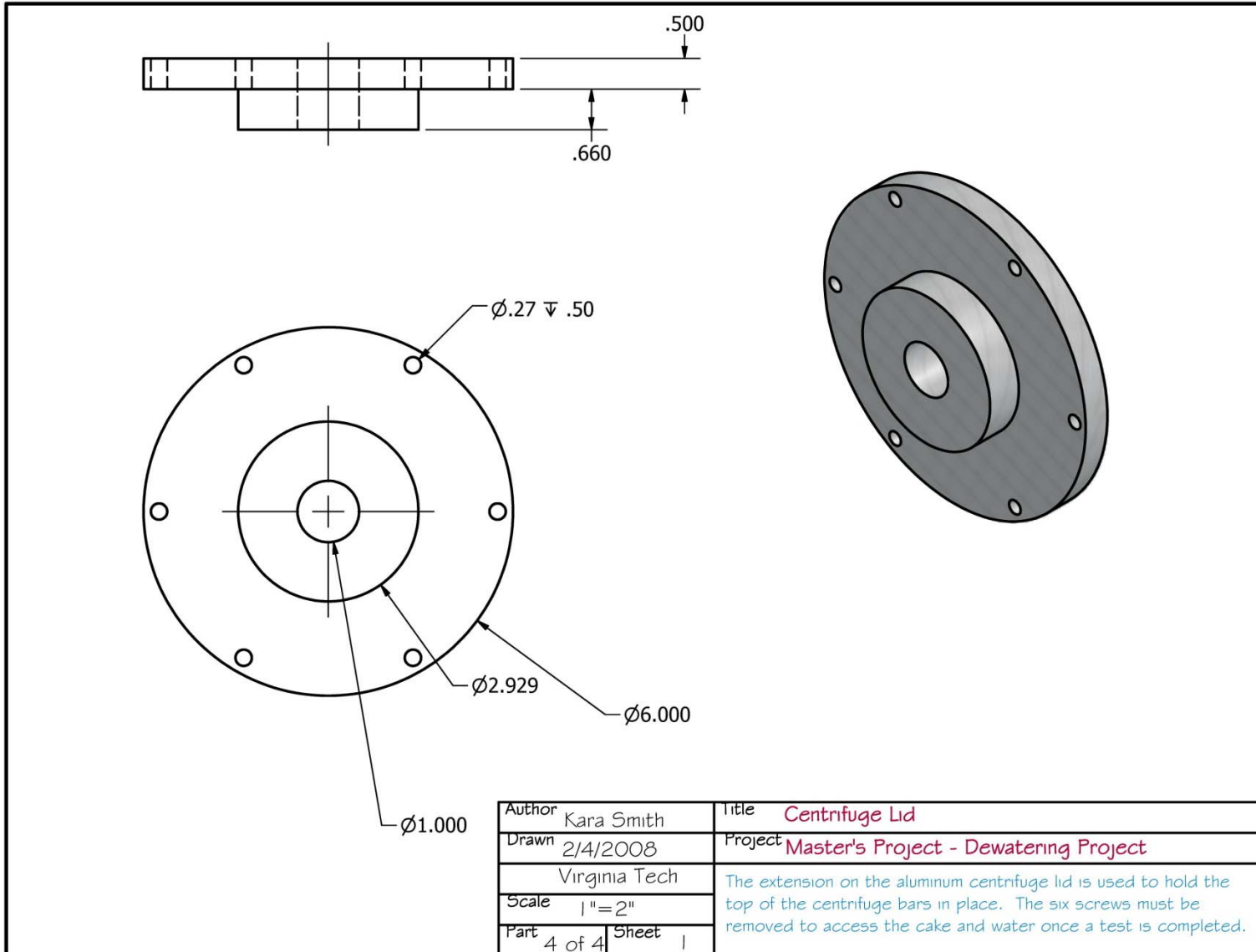


Figure A-4. Drawing of the centrifuge lid

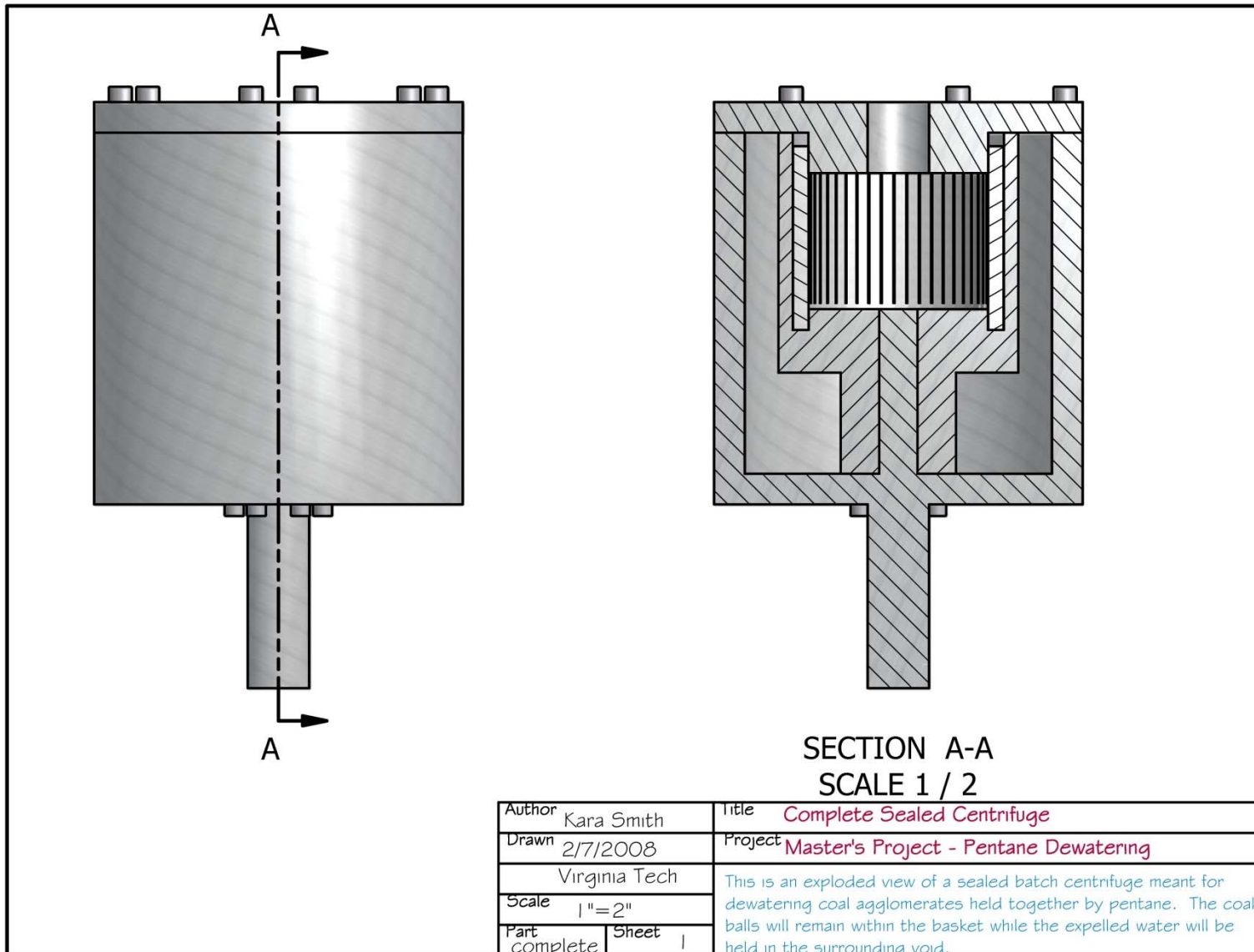


Figure A-5. Drawing of the completed sealed centrifuge

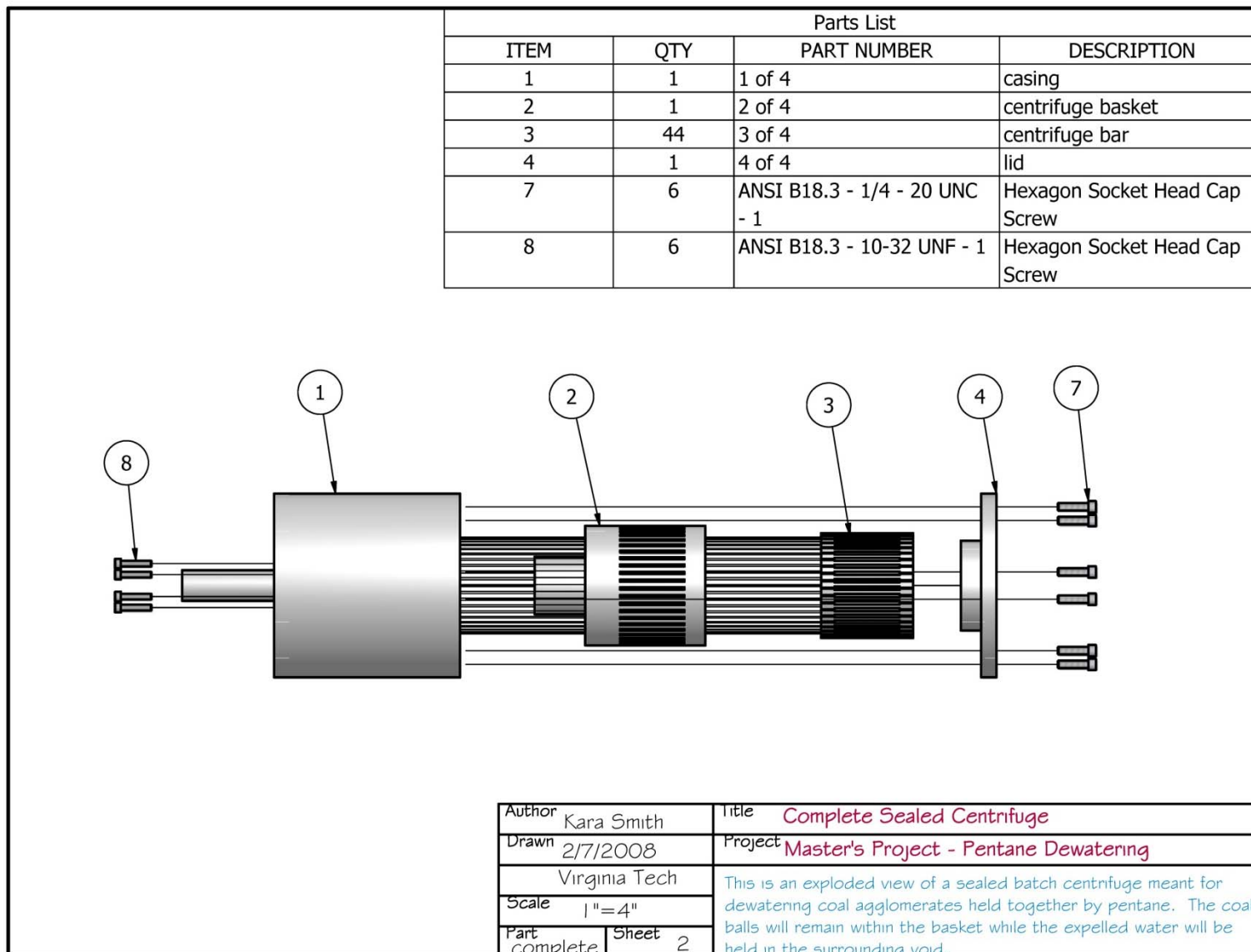


Figure A-6. Drawing of the exploded sealed centrifuge

B. Results Appendix

Appendix B contains detailed supporting information for the summary tables in the Results section.

Figures B-1 through B-4

The first figure compares two Microtrac sizing methods: reflection and transmission. Reflection does not use the refractive index of the particulate matter, so it is better suited to handling high ash contents as seen in Figure B-1. The next three figures contain further size distribution data obtained with the Microtrac reflection method.

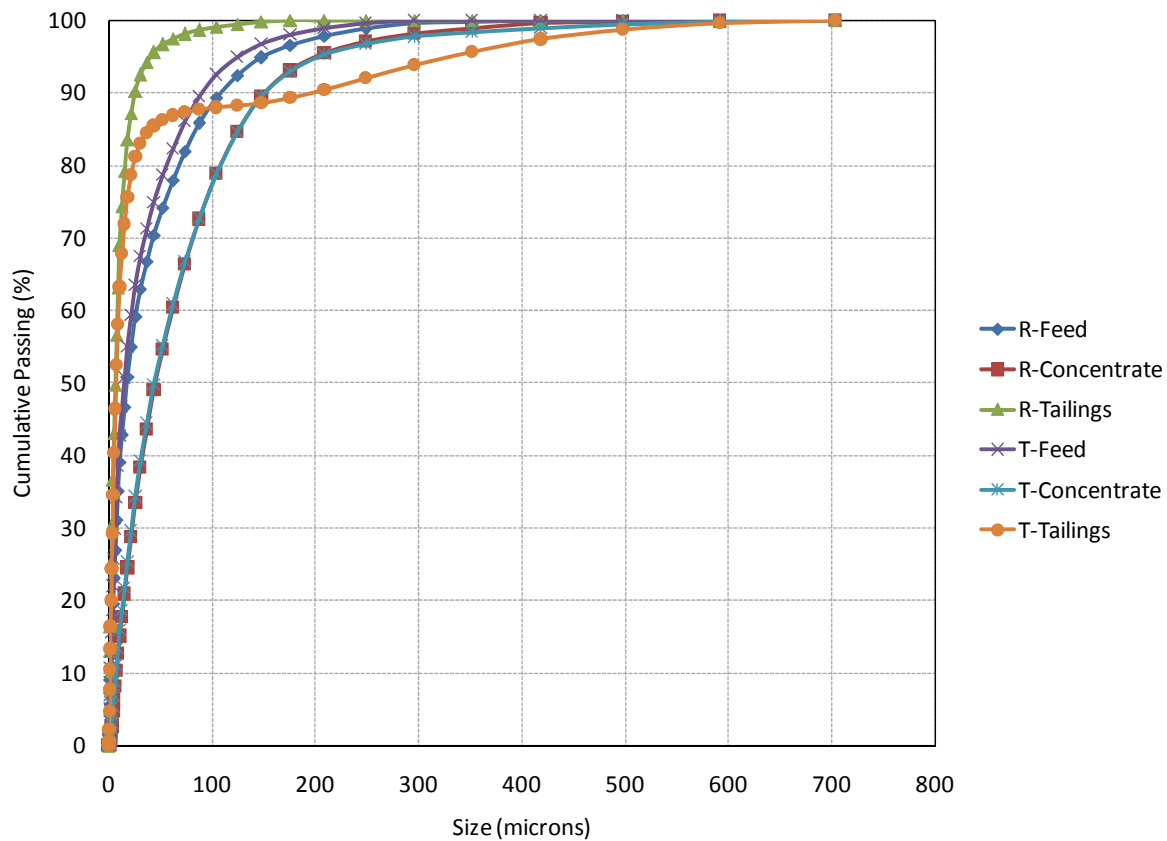


Figure B-1. Comparison of Microtrac reflection and transmission sizing methods.

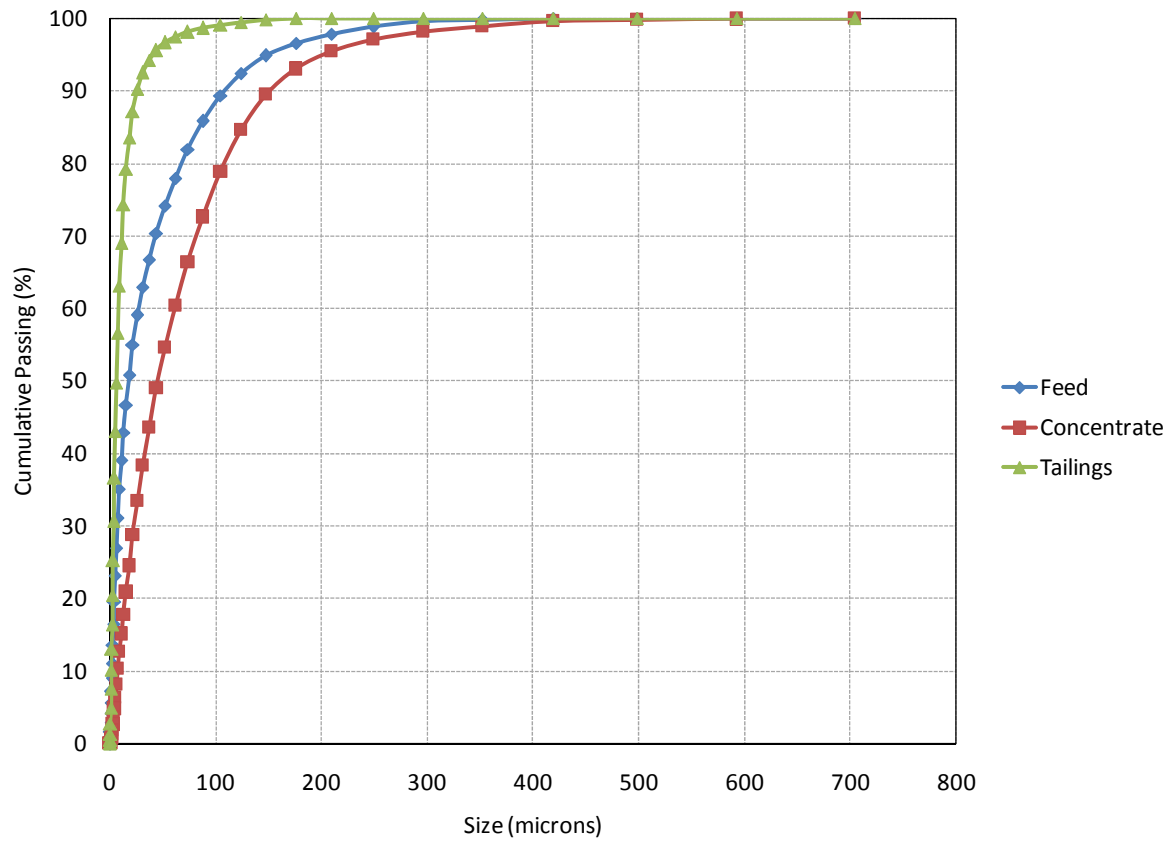


Figure B-2. Centrifuge size distributions for 100 mesh x 0 flotation feed.

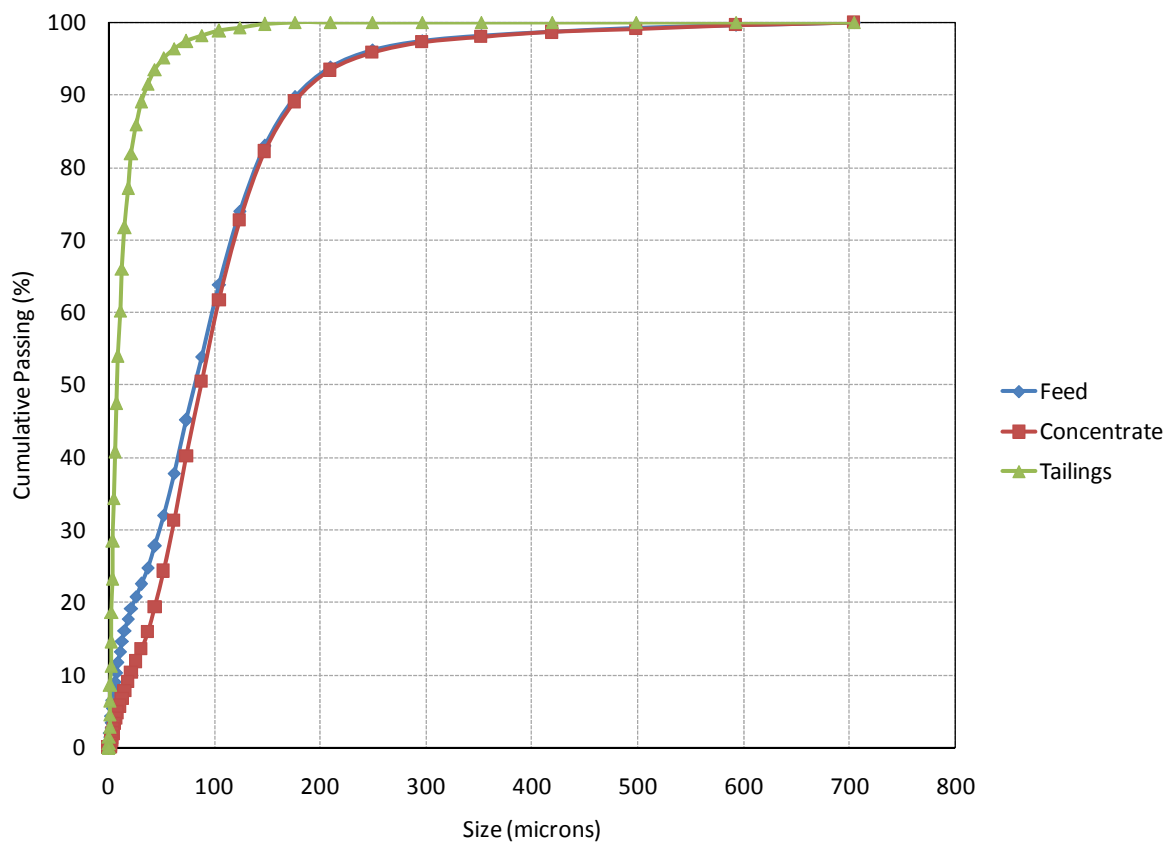


Figure B-3. Centrifuge size distributions for screened 100 mesh x 0 flotation feed.

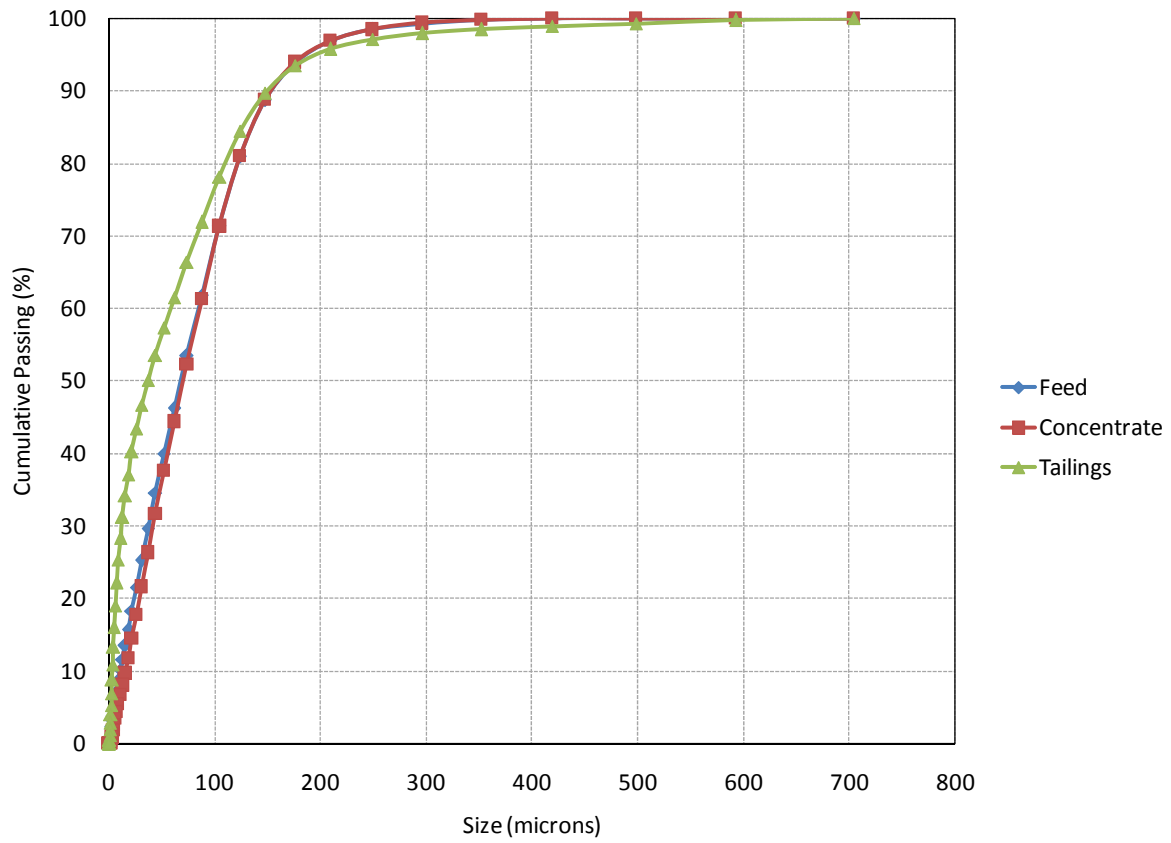


Figure B-4. Centrifuge size distributions for pulverized 80 mesh x 0 clean coal.

Tables B-1 to B-2

The next two sets of tables contain hand shaking moisture data obtained without using a weighing platform. The moistures were taken after 20 minutes at 40°C. These moistures are incorrect and are only included to demonstrate the importance of the moisture determination procedure. The tests were all performed on Tom's Creek 100 mesh x 0 flotation feed.

Table B-1. Comparison of incorrect and corrected hand shaking moistures for 100 mesh x 0

Run	Test	Mass Ratio (Pentane:Coal)	Original Moistures (%)	Corrected Moistures (%)
2	7	0.17	32.4	35.5
	8	0.22	28.7	--
	9	0.30	15.2	22.7
	10	0.38	2.1	9.7
	11	0.50	9.1	--
	12	1.51	41.4	--
3	13	0.17	41.2	43.2
	14	0.21	27.7	33.4
	15	0.30	11.5	17.9
	16	0.38	7.2	13.5
	17	0.50	7.3	--
	18	1.50	42.5	--

**Note: All conducted at 6.3% solids by weight. There is insufficient data to correct all of the moistures, especially those with large dosages.*

Table B-2a. Detailed hand shaking data with incorrect moistures for 100 mesh x 0

Run	Test	Solids (wt%)	Mass Ratio		Volume Ratio	
			(Pentane:Solids)	(Pentane:Coal)	(Pentane:Coal)	(Pentane:Voids)
2	7	6.3	0.11	0.17	0.34	0.27
	8	6.3	0.14	0.22	0.44	0.35
	9	6.3	0.20	0.30	0.62	0.49
	10	6.3	0.24	0.38	0.77	0.61
	11	6.3	0.33	0.50	1.03	0.82
	12	6.3	0.98	1.51	3.08	2.45
3	13	6.3	0.11	0.17	0.34	0.27
	14	6.3	0.14	0.21	0.44	0.35
	15	6.3	0.19	0.30	0.61	0.49
	16	6.3	0.24	0.38	0.77	0.61
	17	6.3	0.32	0.50	1.02	0.81
	18	6.3	0.97	1.50	3.07	2.43
4	19	7.1	0.10	0.15	0.30	0.24
	20	7.1	0.12	0.19	0.39	0.31
	21	7.1	0.17	0.27	0.54	0.43
	22	7.1	0.22	0.33	0.68	0.54
	23	7.1	0.29	0.44	0.91	0.72
	24	7.1	0.86	1.33	2.72	2.16
5	25	15.0	0.10	0.17	0.34	0.27
	26	15.0	0.13	0.21	0.44	0.35
	27	15.0	0.19	0.30	0.61	0.48
	28	15.0	0.24	0.37	0.76	0.60
	29	15.0	0.31	0.50	1.02	0.81
	30	15.0	0.94	1.49	3.05	2.42
6	31	15.0	0.10	0.17	0.34	0.27
	32	15.0	0.13	0.21	0.44	0.35
	33	15.0	0.19	0.30	0.61	0.48
	34	15.0	0.24	0.37	0.76	0.60
	35	15.0	0.31	0.50	1.02	0.81
	36	15.0	0.94	1.49	3.05	2.42
7	37	15.1	0.10	0.17	0.34	0.27
	38	15.1	0.13	0.21	0.44	0.35
	39	15.1	0.19	0.30	0.61	0.48
	40	15.1	0.24	0.37	0.76	0.60
	41	15.1	0.31	0.50	1.02	0.81
	42	15.1	0.94	1.49	3.05	2.42
8	43	30.3	0.10	0.15	0.30	0.24
	44	30.3	0.13	0.19	0.38	0.30
	45	30.3	0.18	0.26	0.54	0.43
	46	30.3	0.22	0.33	0.67	0.53
	47	30.3	0.30	0.44	0.90	0.71
	48	30.3	0.89	1.32	2.69	2.13

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1.*

Table B-2b. Detailed hand shaking data with incorrect moistures for 100 mesh x 0 continued

Pentane (ml)	Dry Weights (g)				Yield (%)	
	T	M	C	Total	M+C	C
4.5	8.77	2.48	14.12	25.37	65.9	56.3
5.8	8.55	2.19	14.75	25.49	68.5	60.4
8.1	8.87	7.60	9.01	25.48	65.2	35.4
10.1	8.91	3.75	12.70	25.36	64.3	49.3
13.4	9.30	1.53	15.22	26.05	63.9	58.0
40.3	9.36	2.05	14.97	26.38	64.9	57.2
4.5	9.02	3.98	13.53	26.53	66.2	51.2
5.8	8.56	9.41	7.89	25.86	66.8	30.3
8.1	8.80	6.01	10.95	25.76	66.1	42.9
10.1	9.08	8.39	8.79	26.26	65.3	33.3
13.4	9.35	3.82	13.19	26.36	64.1	49.4
40.3	9.52	1.96	15.32	26.80	64.7	57.4
4.5	8.83	9.13	7.93	25.89	63.2	24.8
5.8	8.46	7.39	8.71	24.56	65.8	35.9
8.1	9.11	11.49	5.87	26.47	66.3	23.8
10.1	9.57	11.85	6.52	27.94	66.5	24.9
13.4	9.39	4.22	13.75	27.36	66.7	51.7
40.3	9.52	1.69	16.87	28.08	66.5	60.5
10.7	19.16	35.85	8.38	63.39	67.7	7.7
13.7	19.03	27.64	15.90	62.57	67.0	19.5
19.2	20.23	23.86	18.39	62.48	65.6	25.3
24.0	21.34	5.41	37.23	63.98	64.8	56.0
32.0	21.82	0.91	41.80	64.53	64.3	62.8
96.1	21.38	1.34	40.93	63.65	63.3	61.1
10.7	19.49	33.15	12.01	64.65	67.9	13.6
13.7	19.57	31.20	14.64	65.41	67.9	17.3
19.2	20.57	23.43	20.35	64.35	64.9	25.6
24.0	22.63	3.59	38.45	64.67	63.2	57.4
32.0	21.81	0.44	40.36	62.61	63.8	63.1
95.9	19.82	1.39	43.10	64.31	66.8	64.5
10.7	16.13	5.72	41.95	63.80	66.0	54.1
13.8	15.05	8.74	38.86	62.65	67.3	49.8
19.3	17.22	2.06	44.23	63.51	65.6	61.9
24.1	18.57	2.65	42.33	63.55	63.4	58.4
32.2	18.17	0.71	44.73	63.61	64.0	62.7
96.5	15.45	1.01	48.38	64.84	65.2	63.0
21.5	33.46	6.37	87.52	127.35	73.9	68.9
27.6	31.90	13.28	85.14	130.32	74.6	64.1
38.7	36.86	14.81	81.03	132.70	73.5	62.8
48.4	39.61	5.09	92.16	136.86	71.3	67.6
64.5	37.39	0.62	89.11	127.12	72.2	71.7
193.5	--	--	--	--	--	--

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Table B-2c. Detailed hand shaking data with incorrect moistures for 100 mesh x 0 continued

Recovery (%)		Ash Rejection (%)		Concentrate Moisture	
M+C	C	M+C	C	(g)	(%)
94.5	80.9	86.3	88.6	6.7	32.4
95.6	84.3	81.0	83.3	5.9	28.7
94.4	51.3	88.2	93.6	1.6	15.2
94.6	72.9	90.9	93.6	0.3	2.1
93.7	85.2	90.5	91.7	1.5	9.1
93.7	82.8	87.7	89.5	10.6	41.4
94.2	73.1	84.9	88.8	9.4	41.2
96.0	43.7	86.5	94.1	3.0	27.7
95.9	62.4	88.3	92.6	1.4	11.5
94.9	48.9	88.6	95.2	0.7	7.2
93.3	72.8	89.2	93.2	1.0	7.3
93.5	83.2	87.9	89.6	11.3	42.5
94.5	36.7	94.1	96.8	3.0	27.6
95.3	51.9	88.2	93.4	3.4	28.2
95.2	34.7	86.4	96.2	0.8	12.1
95.4	36.1	86.4	95.6	0.7	10.1
94.6	75.6	84.2	92.0	1.3	8.4
94.6	86.3	84.8	86.5	23.8	58.4
95.4	11.2	79.7	98.1	1.7	16.3
95.3	28.1	81.3	95.3	2.8	15.1
94.2	36.9	83.4	94.6	3.1	14.5
93.3	82.9	84.2	90.0	6.3	14.4
93.1	91.6	85.0	86.3	13.0	23.7
89.1	86.8	80.9	82.9	67.2	62.0
95.4	19.3	79.1	96.2	4.1	25.4
95.6	24.9	79.4	95.8	2.7	15.5
92.9	37.6	82.9	95.0	2.4	10.4
93.0	85.6	87.8	90.7	6.6	14.7
93.4	92.9	86.8	87.8	14.9	27.0
91.6	89.1	75.8	77.6	71.4	62.3
95.8	78.7	84.9	88.0	12.0	22.2
96.0	72.9	81.8	89.7	10.3	20.9
95.0	90.9	84.7	87.7	9.8	18.1
94.0	87.2	89.0	90.9	11.8	21.7
94.4	92.8	88.1	89.0	18.4	28.9
91.3	88.3	79.4	80.2	126.1	72.0
97.3	92.3	74.9	79.9	25.0	22.2
97.3	85.4	73.0	80.5	21.9	20.4
96.9	83.8	75.5	81.2	20.0	19.8
96.2	92.7	80.6	84.9	25.8	21.8
96.6	96.4	78.7	79.8	68.4	42.1
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**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Table B-2d. Detailed hand shaking data with incorrect moistures for 100 mesh x 0 continued

Ash Content (%)				
T	M	C	M+C	T+M
89.60	8.46	7.17	7.36	71.71
90.91	9.81	9.80	9.80	74.37
89.67	6.48	6.35	6.41	51.28
90.20	6.42	4.58	5.00	65.38
88.78	6.96	5.09	5.26	77.22
88.43	8.21	6.49	6.70	74.02
88.83	9.06	7.76	8.06	64.41
92.19	7.35	6.91	7.15	47.76
92.12	6.56	6.11	6.27	57.40
90.48	7.23	5.13	6.16	50.50
87.86	9.70	4.88	5.96	65.19
88.12	8.41	6.39	6.62	74.51
90.39	2.20	4.57	3.30	45.56
91.18	6.17	6.49	6.34	51.54
90.75	8.06	5.72	7.27	44.63
91.10	7.82	6.21	7.25	45.03
89.57	17.85	5.48	8.38	67.33
89.52	10.08	7.87	8.07	77.54
91.01	11.55	8.91	11.05	39.23
90.91	11.14	8.83	10.30	43.67
89.43	10.46	7.85	9.32	46.69
88.08	25.79	6.57	9.01	75.48
87.76	35.96	8.02	8.62	85.69
81.24	36.99	10.30	11.15	78.63
90.88	11.78	10.24	11.37	41.07
91.28	12.26	8.88	11.18	42.72
87.22	11.82	7.26	9.70	47.07
88.00	19.29	5.97	7.11	78.59
88.43	58.63	7.10	7.66	87.84
84.13	31.23	12.79	13.37	80.66
92.17	10.34	8.16	8.42	70.75
92.36	20.25	7.66	9.97	65.87
90.78	36.36	7.30	8.59	84.97
89.63	16.81	5.75	6.40	80.54
90.17	29.68	6.49	6.85	87.90
84.23	15.75	11.56	11.65	80.03
92.93	32.24	9.47	11.01	83.22
92.95	23.73	9.86	11.73	72.60
92.21	16.84	9.72	10.82	70.61
90.97	37.41	7.25	8.83	84.87
91.67	68.85	9.15	9.56	91.30
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**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Tables B-3 to B-5

The next three tables provide detailed results for pellet extraction by hand shaking. These correspond with the previous Table B-2. The first table contains data on a fresh 100 mesh x 0 Tom's Creek flotation feed sample. Each test was repeated three times, and two solid contents, 6.3% and 14.8%, were examined. The second table contains data on an oxidized version of the first sample. The tests were not repeated, and only a slurry of 14.9% solids was tested. The last tables contains data on a 325x0 mesh sample removed from the original Tom's Creek flotation feed. These tests were repeated once, and the slurry's solids content was 7.3%. It should be noted that all of the yields are calculated by ash data, not weights.

Table B-3a. Detailed hand shaking data for 100 mesh x 0

Run	Test	Solids (wt%)	Mass Ratio		Volume Ratio	
			(Pentane:Solids)	(Pentane:Coal)	(Pentane:Coal)	(Pentane:Voids)
11	63	6.6	0.11	0.18	0.36	0.29
	64	6.6	0.14	0.23	0.47	0.37
	65	6.6	0.19	0.32	0.65	0.52
	66	6.6	0.24	0.40	0.82	0.65
	67	6.6	0.32	0.53	1.09	0.86
	68	6.6	0.97	1.60	3.26	2.59
12	69	6.2	0.11	0.18	0.36	0.29
	70	6.2	0.14	0.23	0.47	0.37
	71	6.2	0.20	0.32	0.65	0.52
	72	6.2	0.24	0.40	0.82	0.65
	73	6.2	0.33	0.53	1.09	0.86
	74	6.2	0.98	1.60	3.27	2.59
13	75	6.2	0.11	0.18	0.36	0.29
	76	6.2	0.14	0.23	0.47	0.37
	77	6.2	0.20	0.32	0.65	0.52
	78	6.2	0.24	0.40	0.82	0.65
	79	6.2	0.33	0.53	1.09	0.87
	80	6.2	0.98	1.60	3.27	2.60
14	81	14.8	0.10	0.17	0.35	0.28
	82	14.8	0.13	0.22	0.45	0.36
	83	14.8	0.19	0.31	0.64	0.51
	84	14.8	0.24	0.39	0.80	0.63
	85	14.8	0.31	0.52	1.06	0.84
	86	14.8	0.94	1.56	3.18	2.53
15	87	14.6	0.10	0.17	0.35	0.28
	88	14.6	0.13	0.22	0.46	0.36
	89	14.6	0.19	0.31	0.64	0.51
	90	14.6	0.24	0.39	0.80	0.63
	91	14.6	0.31	0.52	1.06	0.84
	92	14.6	0.94	1.56	3.19	2.53
16	93	14.6	0.10	0.17	0.35	0.28
	94	14.6	0.13	0.22	0.45	0.36
	95	14.6	0.19	0.31	0.64	0.51
	96	14.6	0.24	0.39	0.80	0.63
	97	14.6	0.31	0.52	1.06	0.84
	98	14.6	0.94	1.56	3.18	2.53

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Table B-3b. Detailed hand shaking data for 100 mesh x 0 continued

Pentane (ml)	Dry Weights (g)				Yield (%)	
	T	M	C	Total	M+C	C
4.7	9.67	3.13	11.36	24.16	61.8	49.4
6.0	9.58	9.08	6.29	24.95	61.5	25.1
8.4	9.67	3.18	11.80	24.65	60.3	47.3
10.5	9.38	0.47	13.81	23.66	60.6	58.6
14.0	9.45	1.03	14.08	24.56	61.2	57.0
42.0	8.75	0.54	15.66	24.95	64.6	62.4
4.4	9.72	1.98	12.96	24.66	61.1	53.1
5.7	9.55	7.15	8.24	24.94	61.7	33.1
8.0	9.60	9.42	5.40	24.42	60.9	22.5
9.9	9.70	1.47	13.71	24.88	60.8	54.9
13.3	9.54	0.51	14.55	24.60	60.6	58.5
39.8	9.22	0.50	14.28	24.00	61.2	59.1
4.4	9.52	1.92	11.92	23.36	61.6	53.9
5.7	9.09	4.17	10.70	23.96	61.2	43.4
7.9	9.48	6.86	7.90	24.24	60.8	32.4
9.9	9.06	2.09	12.72	23.87	61.0	52.0
13.2	9.26	0.68	14.66	24.60	61.3	58.5
39.6	8.98	0.28	15.57	24.83	62.2	61.1
10.5	23.06	23.73	13.95	60.74	62.2	23.4
13.5	22.43	15.69	21.90	60.02	63.4	37.7
18.9	23.79	13.22	23.47	60.48	60.6	38.7
23.6	24.66	6.14	30.46	61.26	59.8	49.8
31.5	24.70	0.26	36.58	61.54	59.1	58.7
94.6	10.88	0.35	48.39	59.62	74.3	73.6
10.3	23.17	14.40	24.42	61.99	62.2	38.8
13.3	22.01	9.66	27.91	59.58	62.8	46.5
18.6	23.96	2.14	35.23	61.33	60.3	56.8
23.3	21.95	3.51	32.99	58.45	60.3	53.9
31.0	23.66	0.15	36.21	60.02	60.3	60.0
93.1	7.29	0.25	53.80	61.34	79.3	78.6
10.4	22.61	12.64	25.23	60.48	61.6	40.1
13.3	23.18	20.23	16.16	59.57	59.6	24.5
18.7	23.77	1.48	33.93	59.18	59.6	57.1
23.4	23.70	4.47	30.82	58.99	59.6	52.0
31.1	24.30	0.05	35.84	60.19	59.5	59.4
93.4	7.55	0.32	52.14	60.01	74.1	72.9

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Table B-3c. Detailed hand shaking data for 100 mesh x 0 continued

Recovery (%)		Ash Rejection (%)		Concentrate Moisture	
M+C	C	M+C	C	(g)	(%)
94.9	75.9	90.0	92.1	6.5	36.3
94.8	39.0	90.4	96.7	1.3	16.9
94.3	74.0	92.8	94.6	1.9	13.6
94.6	91.6	92.5	92.9	2.8	17.0
94.7	88.5	91.0	92.2	4.7	24.8
94.0	91.0	81.3	82.3	29.1	64.8
94.4	82.2	91.0	92.4	4.4	25.4
95.4	51.1	90.9	95.2	--	--
94.3	35.2	91.3	97.2	1.1	16.2
94.9	86.0	92.4	93.7	2.2	13.6
94.4	91.2	92.2	92.7	4.9	25.3
93.5	90.9	89.3	90.5	13.5	48.3
95.2	83.3	90.8	92.1	5.6	31.9
95.1	67.6	91.8	94.5	2.3	18.1
94.5	50.7	91.9	96.2	1.8	18.9
94.9	81.4	92.0	94.0	3.5	21.9
95.2	91.2	91.5	92.5	6.1	29.2
94.0	92.7	87.4	88.3	28.4	64.8
94.8	35.7	87.3	95.4	5.1	26.9
95.0	58.6	84.6	94.0	5.1	18.9
93.8	60.7	90.1	94.8	5.4	18.6
93.4	78.2	91.4	93.6	7.6	19.9
91.9	91.4	90.9	91.2	15.4	29.6
93.4	92.7	54.6	55.4	220.6	82.0
95.0	60.1	87.6	93.8	8.7	26.4
95.0	72.4	86.3	93.0	7.2	20.7
94.5	89.1	91.8	92.5	7.9	18.4
94.5	84.6	91.8	92.8	7.8	19.2
93.7	93.3	90.6	90.7	17.4	32.6
96.1	95.1	46.2	46.7	263.7	83.1
94.6	61.9	88.8	93.1	8.4	25.1
91.4	38.1	88.7	96.1	3.9	19.5
93.0	89.6	91.3	92.5	8.3	19.6
93.4	81.7	91.8	93.2	8.0	20.6
92.3	92.2	90.4	90.5	18.2	33.7
87.8	86.3	46.8	47.6	262.2	83.4

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Table B-3d. Detailed hand shaking data for 100 mesh x 0 continued

Ash Content (%)				
T	M	C	M+C	T+M
91.88	6.60	6.23	6.31	71.03
91.70	6.76	5.15	6.10	50.37
91.31	5.19	4.48	4.63	70.00
91.64	8.74	4.73	4.86	87.68
91.62	10.58	5.36	5.72	83.66
89.60	18.32	11.08	11.32	85.46
91.27	6.41	5.62	5.72	76.91
92.65	5.89	5.69	5.78	55.50
91.12	6.05	4.81	5.60	48.99
92.10	8.60	4.46	4.86	81.11
91.28	10.01	4.85	5.02	87.15
89.79	23.32	6.26	6.84	86.37
92.32	6.41	5.76	5.85	77.90
92.29	5.88	4.99	5.24	65.12
91.46	5.85	4.62	5.19	55.52
91.98	8.88	4.53	5.14	76.40
92.38	14.36	5.01	5.42	87.04
90.27	34.76	7.45	7.93	88.59
91.61	8.29	7.82	8.12	49.35
91.69	14.28	6.32	9.64	59.83
90.56	8.58	5.34	6.51	61.28
90.03	8.84	5.11	5.74	73.84
88.11	25.67	5.97	6.11	87.46
84.37	48.83	24.03	24.21	83.26
91.95	10.46	6.35	7.87	60.72
91.86	16.41	5.99	8.67	68.85
91.64	8.84	5.21	5.42	84.85
91.61	6.43	5.28	5.39	79.87
90.40	14.46	6.15	6.18	89.92
88.70	16.13	26.91	26.86	86.29
91.58	8.06	6.79	7.21	61.63
87.11	8.49	6.31	7.52	50.47
89.58	18.26	5.24	5.78	85.40
90.12	7.49	5.18	5.47	77.01
88.51	9.53	6.36	6.36	88.35
71.65	20.50	28.49	28.44	69.57

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Table B-4a. Detailed hand shaking data for oxidized 100 mesh x 0

Run	Test	Solids (wt%)	Mass Ratio		Volume Ratio	
			(Pentane:Solids)	(Pentane:Coal)	(Pentane:Coal)	(Pentane:Voids)
9	49	14.9	0.11	0.12	0.26	--
	50	14.9	0.14	0.16	0.34	--
	51	14.9	0.19	0.22	0.47	--
	52	14.9	0.24	0.28	0.59	--
	53	14.9	0.32	0.37	0.79	--
	54	14.9	0.95	1.11	2.37	--
10	55	14.9	0.24	0.28	0.60	--
	56	14.9	0.96	1.13	2.40	--

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate. A void space analysis was not performed on the oxidized concentrate.*

Table B-4b. Detailed hand shaking data for oxidized 100 mesh x 0 continued

Pentane (ml)	Dry Weights (g)				Yield (%)	
	T	M	C	Total	M+C	C
10.6	15.20	4.60	34.60	54.40	77.7	70.7
13.6	17.70	13.90	22.10	53.70	73.5	52.3
19.0	19.70	7.50	28.40	55.60	73.2	62.7
23.8	18.00	5.30	29.70	53.00	71.7	63.2
31.7	17.30	0.60	35.60	53.50	73.5	72.5
95.0	9.60	0.40	43.00	53.00	88.7	88.1
24.1	19.69	3.69	39.04	62.42	73.4	68.3
96.2	5.47	0.34	58.51	64.32	96.1	95.7

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Table B-4c. Detailed hand shaking data for oxidized 100 mesh x 0 continued

Recovery (%)		Ash Rejection (%)		Concentrate Moisture	
M+C	C	M+C	C	(g)	(%)
97.1	89.2	73.3	77.7	28.1	46.2
93.2	67.0	78.1	85.9	19.7	46.6
93.9	81.0	81.2	85.4	16.6	35.4
93.0	82.5	83.9	87.5	11.1	25.2
94.5	93.5	81.4	82.5	23.5	42.0
96.3	95.9	31.1	32.4	210.8	82.9
95.3	89.2	84.0	86.3	10.5	21.3
98.8	98.6	11.2	11.7	287.1	83.0

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Table B-4d. Detailed hand shaking data for oxidized 100 mesh x 0 continued

Ash Content (%)				
T	M	C	M+C	T+M
90.76	15.45	8.71	9.50	90.76
81.50	9.52	7.44	8.24	81.50
83.68	9.62	6.43	7.10	83.68
82.02	10.31	5.46	6.19	82.02
84.90	27.09	6.67	7.01	84.90
76.18	48.32	21.22	21.47	76.18
87.27	11.47	5.53	6.04	87.27
78.37	38.05	25.50	25.57	78.37

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Table B-5a. Detailed hand shaking data for 325 mesh x 0

Run	Test	Solids (wt%)	Mass Ratio		Volume Ratio	
			(Pentane:Solids)	(Pentane:Coal)	(Pentane:Coal)	(Pentane:Voids)
1	113	7.2	0.11	0.22	0.47	--
	114	7.2	0.14	0.28	0.60	--
	115	7.2	0.19	0.40	0.85	--
	116	7.2	0.24	0.50	1.06	--
	117	7.2	0.32	0.66	1.41	--
	118	7.2	0.97	1.99	4.23	--
2	119	7.3	0.11	0.22	0.47	--
	120	7.3	0.14	0.28	0.60	--
	121	7.3	0.19	0.40	0.85	--
	122	7.3	0.24	0.50	1.06	--
	123	7.3	0.32	0.66	1.41	--
	124	7.3	0.97	1.99	4.23	--

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate. A void space analysis was not performed on the -325 mesh concentrate.*

Table B-5b. Detailed hand shaking data for 325 mesh x 0 continued

Pentane (ml)	Dry Weights (g)				Yield (%)	
	T	M	C	Total	M+C	C
5.1	15.46	3.50	10.43	29.39	47.7	35.9
6.6	14.38	5.56	7.16	27.10	47.2	26.8
9.2	15.75	5.00	8.71	29.46	47.1	30.3
11.5	15.73	4.83	8.77	29.33	46.5	30.0
15.4	16.78	0.61	11.71	29.10	43.5	41.4
46.1	16.59	0.42	11.61	28.62	43.2	41.8
5.2	15.27	6.22	7.02	28.51	47.1	25.6
6.7	15.41	7.10	5.79	28.30	46.8	22.3
9.4	15.21	8.10	4.54	27.85	45.5	16.5
11.7	15.70	5.55	7.30	28.55	46.1	27.0
15.6	12.14	0.86	11.83	24.83	44.8	41.0
46.8	16.25	0.72	12.38	29.35	46.4	44.0

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Table B-5c. Detailed hand shaking data for 325 mesh x 0 continued

Recovery (%)		Ash Rejection (%)		Concentrate Moisture	
M+C	C	M+C	C	(g)	(%)
91.3	68.6	93.4	95.0	5.3	33.6
91.3	51.8	94.3	96.8	2.7	27.1
91.1	58.7	94.4	96.4	2.7	23.5
89.3	57.8	94.0	96.2	3.9	31.0
82.0	78.2	92.8	93.2	9.0	43.3
75.5	72.8	87.2	87.4	31.0	72.8
90.0	48.5	93.3	96.0	3.7	34.5
90.0	42.8	94.0	97.0	2.5	30.4
87.9	31.8	94.4	98.0	2.1	32.0
88.0	52.0	93.4	96.5	4.7	39.1
82.4	75.8	90.7	91.9	12.7	51.8
81.7	77.8	86.8	87.9	30.6	71.2

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Table B-5d. Detailed hand shaking data for 325 mesh x 0 continued

Ash Content (%)				
T	M	C	M+C	T+M
91.97	6.75	7.18	7.07	76.24
92.00	6.09	6.23	6.17	68.05
91.83	6.29	6.04	6.13	71.22
90.33	6.94	6.53	6.68	70.74
84.50	10.55	8.47	8.57	81.91
79.07	7.77	15.49	15.22	77.31
90.80	6.61	8.00	7.35	66.43
90.91	6.35	7.02	6.65	64.24
89.20	6.22	6.39	6.28	60.37
89.23	8.12	6.73	7.33	68.05
84.53	18.20	10.15	10.70	80.14
83.40	21.86	14.17	14.59	80.79

**Note: Each test was conducted with 400 ml. The original estimated pentane-to-solids mass ratios are 1:9, 1:7, 1:5, 1:4, 1:3, and 1:1. Abbreviations: T-tailings, M-middlings, C-concentrate.*

Tables B-6 to B-8

The following three tables contain detailed information on dewatering by sealed centrifuge. The process yield is calculated by ashes; process refers to the cleaning stage. The centrifuge stage only considers cleaning concentrate that was fed to the centrifuge. Its yields are based on weights because there was not enough material to ash.

Table B-6a. Detailed centrifuge data for 100 mesh x 0

Run	Test	Mass Ratio		Volume Ratio		Added Pentane (ml)
		(Pentane:Solids)	(Pentane:Coal)	(Pentane:Coal)	(Pentane:Voids)	
c1	1	0.20	0.32	0.65	0.52	0
	2	0.20	0.32	0.65	0.52	1
	3	0.20	0.32	0.65	0.52	2
	4	0.20	0.32	0.65	0.52	0
	5	0.20	0.32	0.65	0.52	1
	6	0.20	0.32	0.65	0.52	2
	7	0.20	0.32	0.65	0.52	3
	8	0.20	0.32	0.65	0.52	2
c2	9	0.20	0.33	0.68	0.54	2
	10	0.20	0.33	0.68	0.54	2
	11	0.20	0.33	0.68	0.54	2
	12	0.20	0.33	0.68	0.54	2
c3	13	0.20	0.32	0.66	0.52	2
	14	0.20	0.32	0.66	0.52	2
	15	0.20	0.32	0.66	0.52	2
	16	0.20	0.32	0.66	0.52	2
c4	17	0.20	0.31	0.63	0.50	2
	18	0.20	0.31	0.63	0.50	2
	19	0.20	0.31	0.63	0.50	2
	20	0.20	0.31	0.63	0.50	2

**Note: Each test was conducted with 900 ml and 6.0% solids. Spherical agglomeration was performed with 17.2 ml. Additional pentane added after cleaning is listed above.*

Table B-6b. Detailed centrifuge data for 100 mesh x 0 continued

Feeding (sec)	Drying			Ash Content (%)	
	(sec)	(rpm)	(g's)	T1	C
21	30	3280	447	79.70	4.11
22	30	3280	447	81.20	4.11
53	30	3280	447	77.69	4.00
29	60	3280	447	81.41	4.08
20	60	3280	447	79.12	3.93
22	60	3280	447	76.93	4.07
150	60	3280	447	75.73	3.74
26	60	3280	447	73.78	3.82
22	15	880	32	75.65	3.95
23	30	880	32	83.41	4.15
64	60	880	32	81.98	4.05
55	120	880	32	84.94	4.15
90	15	3280	447	82.05	4.00
90	30	3280	447	80.17	3.87
70	60	3280	447	81.04	3.89
80	120	3280	447	81.06	3.92
70	15	2040	173	79.00	0.90
58	30	2040	173	80.76	3.92
58	60	2040	173	85.15	4.04
120	120	2040	173	82.44	4.08

**Note: Each test was conducted with 900 ml and 6.0% solids. During feeding, the centrifuge was still except for the 8th test which ran at 880 rpm. L2 and L1 are assumed to have the same ash as C. Abbreviations: T1-cleaning tailings, L1-lost while feeding the centrifuge, L2-cake lost to the centrifuge basket and not used in the moisture calculation, and C-centrifuge concentrate.*

Table B-6c. Detailed centrifuge data for 100 mesh x 0 continued

Dry Weight (%)				
T1	L1	L2	C	Total
23.85	0.96	0.95	25.31	51.07
23.80	2.18	0.87	25.06	51.91
24.68	0.87	1.44	25.13	52.12
23.90	1.15	1.17	25.98	52.20
25.22	0.66	0.77	25.94	52.61
25.03	0.05	0.67	26.44	52.22
25.95	15.89	0.62	8.25	50.71
26.43	-0.16	0.66	26.13	53.06
25.69	3.51	0.74	22.49	52.43
23.08	7.81	0.84	20.65	52.38
23.58	10.79	0.71	17.50	52.58
22.50	10.18	0.80	18.44	51.92
22.94	6.91	0.87	19.95	50.67
24.06	0.57	0.74	27.28	52.65
23.86	4.79	0.63	22.60	51.88
23.84	2.46	0.70	24.53	51.53
24.58	4.26	0.63	23.17	52.64
23.76	8.86	0.63	18.61	51.86
22.54	4.63	0.88	24.39	52.44
23.53	12.55	0.64	16.25	52.97

**Note: Each test was conducted with 900 ml and 6.0% solids. No centrifuge tailings (T2) were produced. Abbreviations: T1-cleaning tailings, L1-lost while feeding the centrifuge, L2-cake lost to the centrifuge basket and not used in the moisture calculation, and C-centrifuge concentrate.*

Table B-6d. Detailed centrifuge data for 100 mesh x 0 continued

Yield (%)		Recovery (%)		Ash Rejection (%)	Concentrate Moisture	
Cleaning	Centrifuge	Cleaning	Centrifuge		(g)	(%)
54.1	100	84.8	100	94.3	6.8	21.1
55.0	100	86.2	100	94.2	6.1	19.7
52.8	100	82.8	100	94.6	5.9	19.1
55.1	100	86.4	100	94.2	6.4	19.9
53.7	100	84.2	100	94.6	6.2	19.4
52.4	100	82.1	100	94.5	6.3	19.4
51.3	100	80.7	100	95.0	1.8	17.5
50.0	100	78.6	100	95.1	6.8	20.7
47.8	100	78.3	100	95.4	7.0	23.6
53.0	100	86.7	100	94.7	6.6	24.2
52.1	100	85.3	100	94.9	4.9	21.8
53.9	100	88.2	100	94.6	4.9	21.0
54.3	100	86.4	100	94.5	5.1	20.3
53.1	100	84.6	100	94.8	6.2	18.5
53.7	100	85.4	100	94.7	5.2	18.8
53.7	100	85.5	100	94.7	5.9	19.4
54.7	100	85.1	100	98.6	5.6	19.4
57.9	100	87.3	100	93.7	4.5	19.6
60.3	100	90.7	100	93.3	6.3	20.5
58.9	100	88.7	100	93.4	4.5	21.6

**Note: Each test was conducted with 900 ml and 6.0% solids. The yields were calculated based on ash content. The centrifuge yield and combustible recovery are 100% because there were no measureable centrifuge tailings. The ash rejection refers to the cleaning stage since no tailings were produced in the centrifuge. The concentrate for the cleaning stage calculations include the centrifuge concentrate and the material lost during centrifugation.*

Table B-7a. Detailed centrifuge data for screened 100 mesh x 0

Run	Test	Mass Ratio		Volume Ratio	
		(Pentane:Solids)	(Pentane:Coal)	(Pentane:Coal)	(Pentane:Voids)
c7	29	0.28	0.34	0.70	0.56
	30	0.28	0.34	0.70	0.56
	31	0.28	0.34	0.70	0.56
	32	0.28	0.34	0.70	0.56

**Note: Each test was conducted with 600 ml and 6.1% solids. Spherical agglomeration was performed with 16.4 ml, and no pentane was added after cleaning.*

Table B-7b. Detailed centrifuge data for screened 100 mesh x 0 continued

Feeding (sec)	Drying			Ash Content (%)	
	(sec)	(rpm)	(g's)	T1	C
32	5	3280	447	89.09	3.90
34	15	3280	447	88.53	3.91
40	30	3280	447	88.88	3.94
55	60	3280	447	88.99	3.91

**Note: Each test was conducted with 600 ml and 6.1% solids. During feeding, the centrifuge was still. L2 and L1 are assumed to have the same ash as C. Abbreviations: T1-cleaning tailings, L1-lost while feeding the centrifuge, L2-cake lost to the centrifuge basket and not used in the moisture calculation, and C-centrifuge concentrate.*

Table B-7c. Detailed centrifuge data for screened 100 mesh x 0 continued

Dry Weight (%)				
T1	L1	L2	C	Total
5.72	0.18	0.70	26.96	33.56
5.73	0.25	1.01	25.61	32.60
5.77	0.21	1.07	26.13	33.18
5.69	2.98	1.44	21.68	31.79

**Note: Each test was conducted with 600 ml and 6.1% solids. No centrifuge tailings (T2) were produced. Abbreviations: T1-cleaning tailings, L1-lost while feeding the centrifuge, L2-cake lost to the centrifuge basket and not used in the moisture calculation, and C-centrifuge concentrate.*

Table B-7d. Detailed centrifuge data for screened 100 mesh x 0 continued

Yield (%)		Recovery (%)		Ash Rejection (%)	Concentrate Moisture	
Cleaning	Centrifuge	Cleaning	Centrifuge		(g)	(%)
81.7	100	97.5	100	83.6	3.9	12.6
81.6	100	97.4	100	83.6	3.9	13.3
81.7	100	97.5	100	83.5	4.0	13.2
81.7	100	97.5	100	83.6	3.5	14.0

**Note: Each test was conducted with 600 ml and 6.1% solids. The yields were calculated based on ash content. The centrifuge yield and combustible recovery are 100% because there were no measureable centrifuge tailings. The ash rejection refers to the cleaning stage since no tailings were produced in the centrifuge. The concentrate for the cleaning stage calculations include the centrifuge concentrate and the material lost during centrifugation.*

Table B-8a. Detailed centrifuge data for pulverized 80 mesh x 0 clean coal

Run	Test	Mass Ratio		Volume Ratio	
		(Pentane:Solids)	(Pentane:Coal)	(Pentane:Coal)	(Pentane:Voids)
c5	21	0.17	0.21	0.43	0.67
	22	0.17	0.21	0.43	0.67
	23	0.17	0.21	0.43	0.67
	24	0.17	0.21	0.43	0.67
c6	25	0.17	0.21	0.42	0.65
	26	0.17	0.21	0.42	0.65
	27	0.17	0.21	0.42	0.65
	28	0.17	0.21	0.42	0.65

**Note: Each test was conducted with 900 ml and 6.0% solids. Spherical agglomeration was performed with 14.8 ml, and no pentane was added after cleaning. Coal in c5 soaked 1 min, and coal in c6 soaked 3 days.*

Table B-8b. Detailed centrifuge data for pulverized 80 mesh x 0 clean coal continued

Feeding (sec)	Drying			Ash Content (%)	
	(sec)	(rpm)	(g's)	T1	C
35	5	3280	447	76.36	8.61
30	15	3280	447	79.30	8.02
32	30	3280	447	60.67	7.78
25	60	3280	447	40.45	7.71
30	5	3280	447	77.08	6.59
25	15	3280	447	79.73	7.30
25	30	3280	447	84.69	7.21
30	60	3280	447	79.21	7.33

**Note: Each test was conducted with 900 ml and 6.0% solids. During feeding, the centrifuge was still. L2 and L1 are assumed to have the same ash as C. Abbreviations: T1-cleaning tailings, L1-lost while feeding the centrifuge, L2-cake lost to the centrifuge basket and not used in the moisture calculation, and C-centrifuge concentrate.*

Table B-8c. Detailed centrifuge data for pulverized 80 mesh x 0 clean coal continued

Dry Weight (%)				
T1	L1	L2	C	Total
4.42	0.00	0.89	22.92	28.23
4.30	0.00	0.99	23.26	28.55
5.14	0.00	1.40	18.95	25.49
2.59	0.00	1.43	20.61	24.63
4.32	0.04	0.89	21.35	26.60
3.44	0.00	1.07	22.55	27.06
3.77	0.34	1.54	21.54	27.19
3.82	0.00	1.42	22.00	27.24

**Note: Each test was conducted with 900 ml and 6.0% solids. No centrifuge tailings (T2) were produced. Abbreviations: T1-cleaning tailings, L1-lost while feeding the centrifuge, L2-cake lost to the centrifuge basket and not used in the moisture calculation, and C-centrifuge concentrate.*

Table B-8d. Detailed centrifuge data for pulverized 80 mesh x 0 clean coal continued

Yield (%)		Recovery (%)		Ash Rejection (%)	Concentrate Moisture	
Cleaning	Centrifuge	Cleaning	Centrifuge		(g)	(%)
82.6	100	94.8	100	65.2	2.5	9.9
82.6	100	95.5	100	67.6	2.1	8.4
76.1	100	88.2	100	71.0	1.6	7.8
61.1	100	70.9	100	76.9	1.7	7.5
83.2	100	95.3	100	70.3	2.0	8.6
84.6	100	96.2	100	66.5	2.0	8.2
85.5	100	97.3	100	66.6	2.0	8.6
84.5	100	96.1	100	66.4	2.1	8.7

**Note: Each test was conducted with 900 ml and 6.0% solids. The yields were calculated based on ash content. The centrifuge yield and combustible recovery are 100% because there were no measureable centrifuge tailings. The ash rejection refers to the cleaning stage since no tailings were produced in the centrifuge. The concentrate for the cleaning stage calculations include the centrifuge concentrate and the material lost during centrifugation.*

Table B-9

Conventional cleaning and dewatering were also performed on the original 100 mesh x 0 Tom's Creek flotation feed. The results from the release analysis can be seen below in Table B-I. Three tests were performed to ensure accuracy.

Table B-9. Release analysis results for 100 mesh x 0 flotation feed

Trial	Time (min)	Bag (g)	Bag+Dry (g)	Dry (g)	Individual (%)		Cumulative (%)		
					Mass	Ash	Mass	Ash	Recovery
1	0.25	5.33	31.92	26.59	10.8	2.54	10.8	2.54	15.5
	0.5	5.28	35.12	29.84	12.2	2.81	23.0	2.68	32.8
	1	5.32	35.38	30.06	12.2	3.17	35.2	2.85	50.2
	2	5.32	43.79	38.47	15.7	4.06	50.9	3.22	72.3
	4	5.31	44.12	38.81	15.8	6.34	66.7	3.96	94.1
	8	5.33	9.74	4.41	1.8	19.00	68.5	4.36	96.2
	16	5.33	6.2	0.87	0.4	47.35	68.9	4.58	96.5
	Tailings	5.36	81.78	76.42	31.1	92.25	100.0	31.87	100.0
	Sum			245.47	100.0	31.87			
2	0.25	5.30	59.06	53.76	17.4	2.76	17.4	2.76	25.0
	0.5	5.32	57.33	52.01	16.9	3.12	34.3	2.94	49.2
	1	5.32	43.93	38.61	12.5	3.61	46.8	3.12	67.0
	2	5.31	32.67	27.36	8.9	5.24	55.7	3.46	79.4
	5	5.32	42.71	37.39	12.1	12.15	67.8	5.01	95.2
	8	5.26	6.25	0.99	0.3	51.57	68.1	5.23	95.4
	16	5.25	7.18	1.93	0.6	29.90	68.7	5.45	96.0
	32	5.18	5.44	0.26	0.1	8.35	68.8	5.46	96.1
	Tailings	5.32	101.59	96.27	31.2	91.64	100.0	32.34	100.0
	Sum			308.58	100.0	32.34			
3	0.25	5.5	57.0	51.5	20.5	2.83	20.5	2.83	29.5
	0.5	5.5	46.0	40.5	16.1	2.91	36.6	2.87	52.6
	1	5.5	41.0	35.5	14.1	4.19	50.8	3.23	72.7
	2	5.0	43.5	38.5	15.3	8.01	66.1	4.34	93.5
	4	5.0	11.0	6.0	2.4	20.20	68.5	4.89	96.3
	8	5.5	6.0	0.5	0.2	47.24	68.7	5.02	96.5
	Tailings	5.5	84.05	78.55	31.3	92.39	100.0	32.36	100.0
	Sum			251.1	100.0	32.35525			



Figure B-6. Centrifuge cake of spherical agglomerates.

C. Moisture Determination Appendix

Figures C-1 through C-16

Appendix C contains detailed supporting information and explanations for moisture determination. The preferred moisture determination utilizes a weighing platform, and this method is based on data collected from an Ohaus 50g-capacity moisture balance set at 40°C. The first section of this appendix displays moisture balance evaporation curves for known mixtures of water, pentane, and coal. Preparation of these samples is discussed in the procedures section. The following sixteen graphs have similar axes and are ordered to show trends.

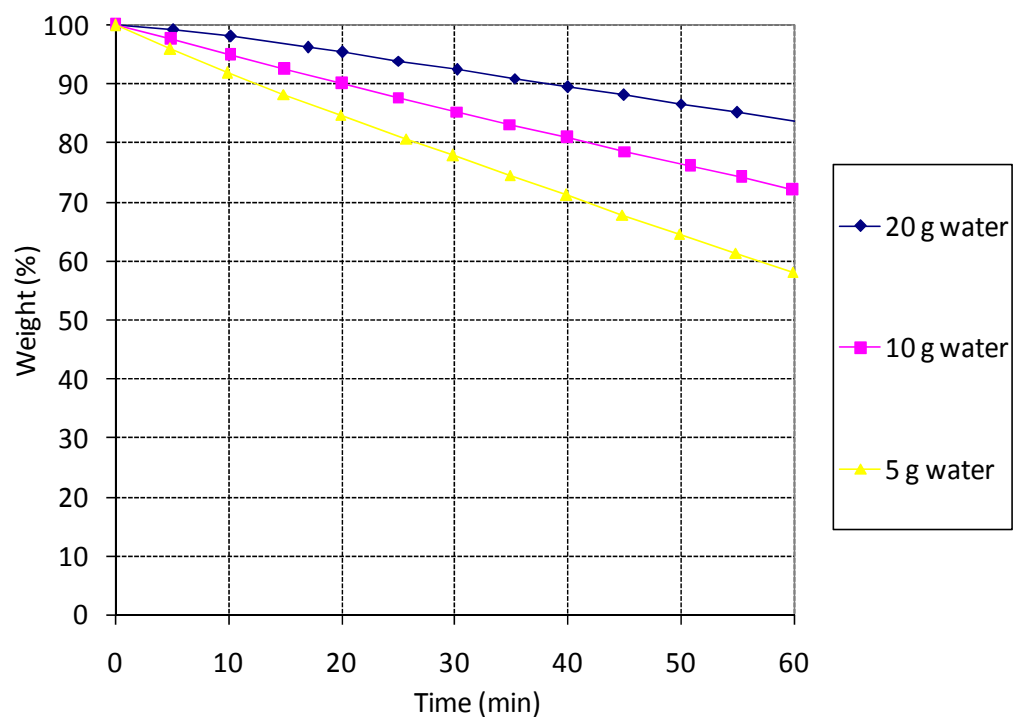


Figure C-1. Summary of vaporization curves: varying water.

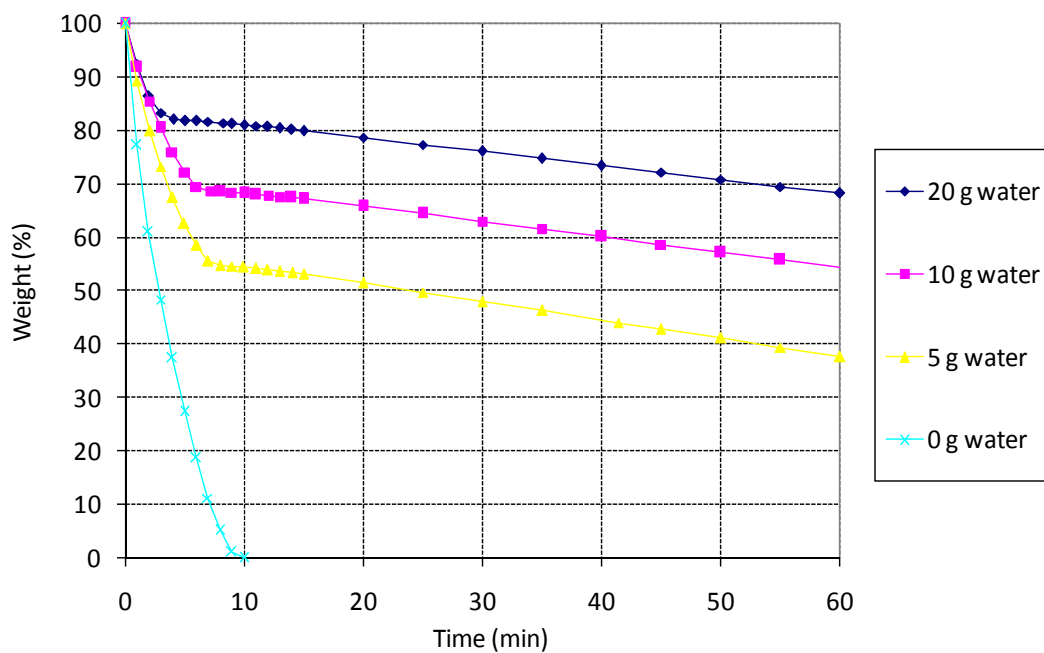


Figure C-2. Summary of vaporization curves: varying water + 5g pentane.

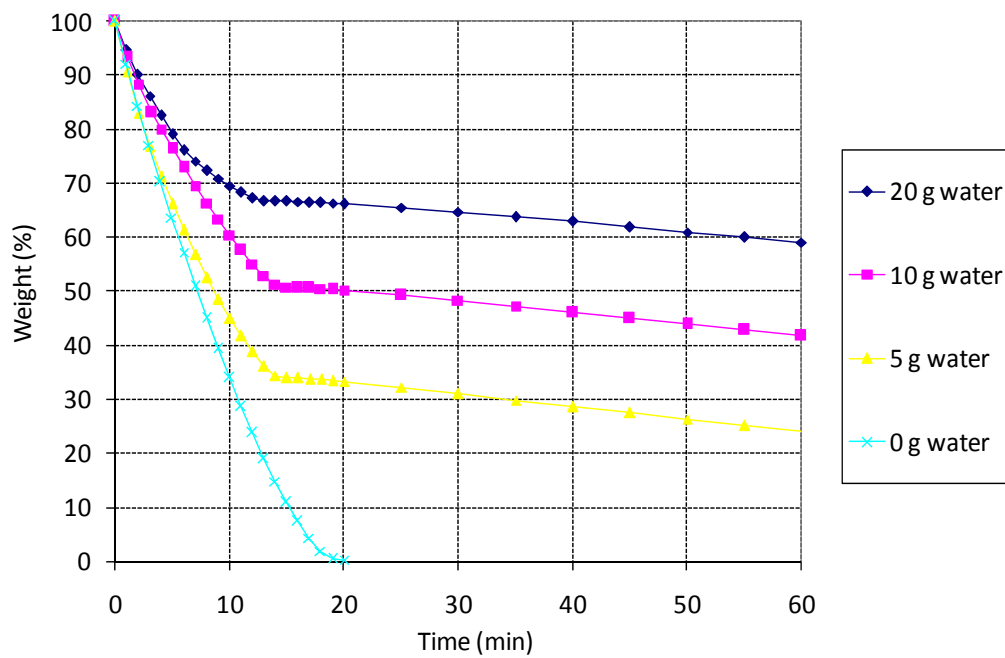


Figure C-3. Summary of vaporization curves: varying water + 10g pentane.

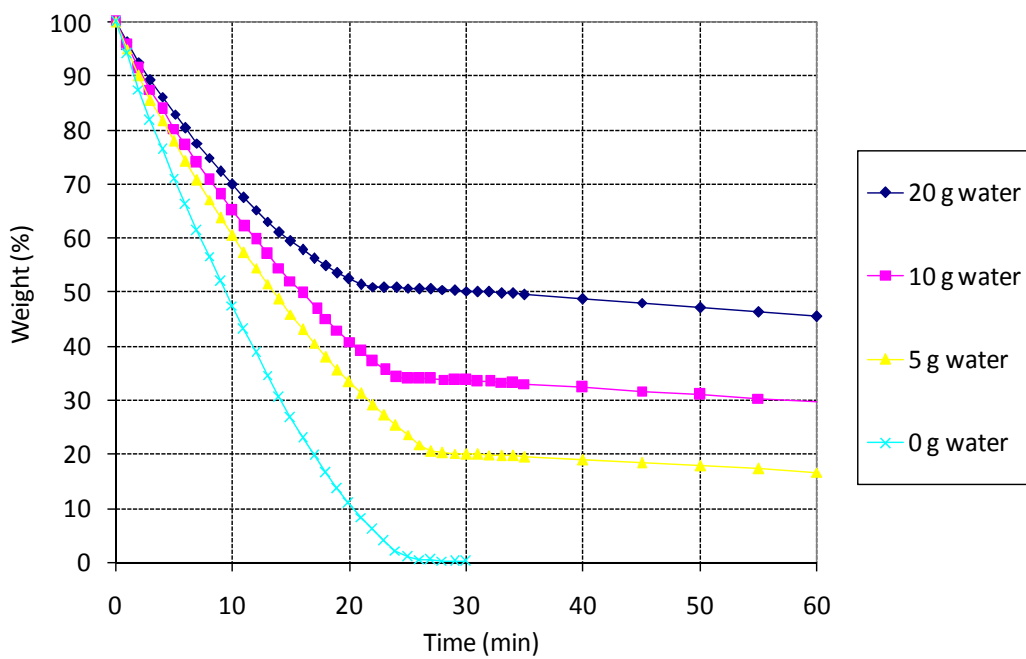


Figure C-4. Summary of vaporization curves: varying Water + 20g pentane.

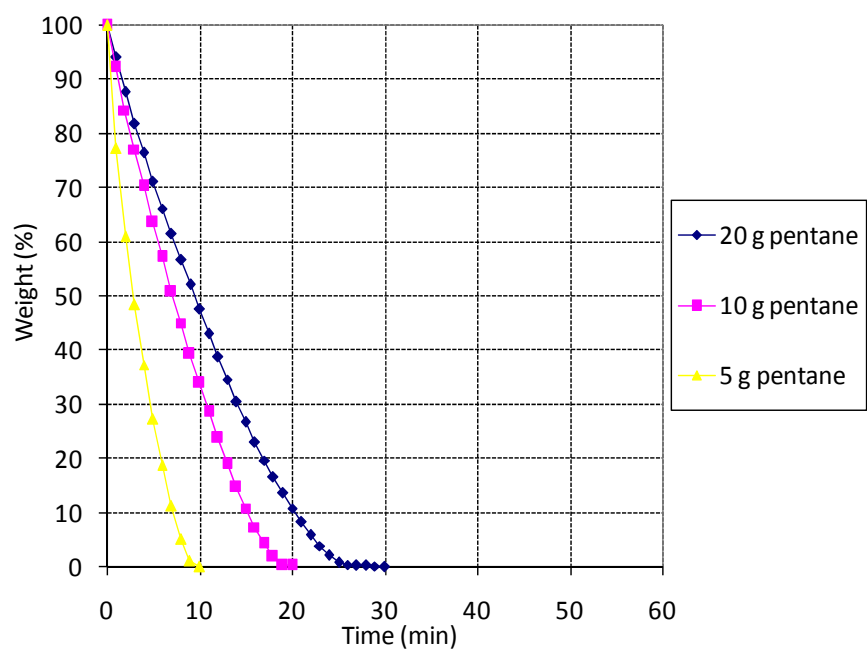


Figure C-5. Summary of vaporization curves: varying pentane.

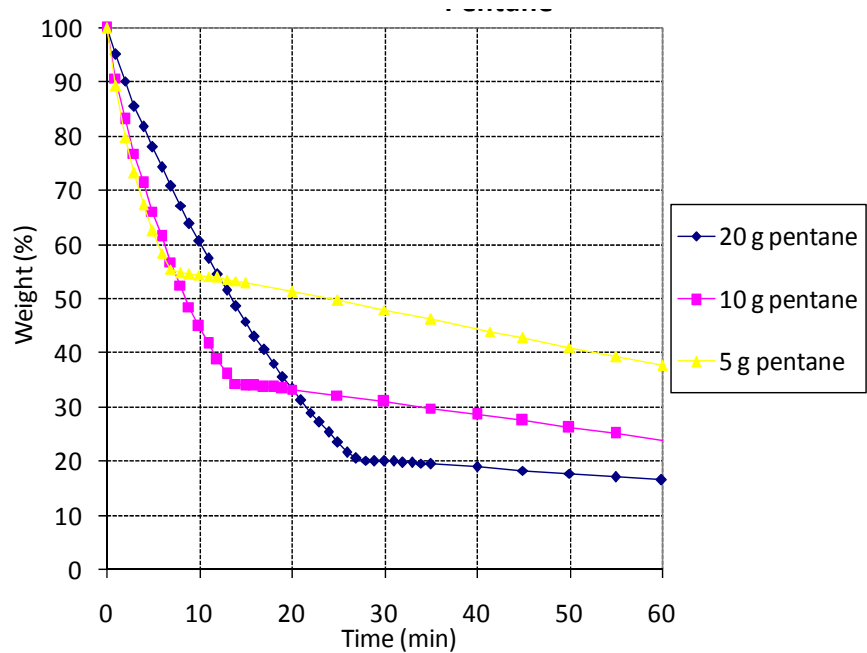


Figure C-6. Summary of vaporization curves: 5g water + varying pentane.

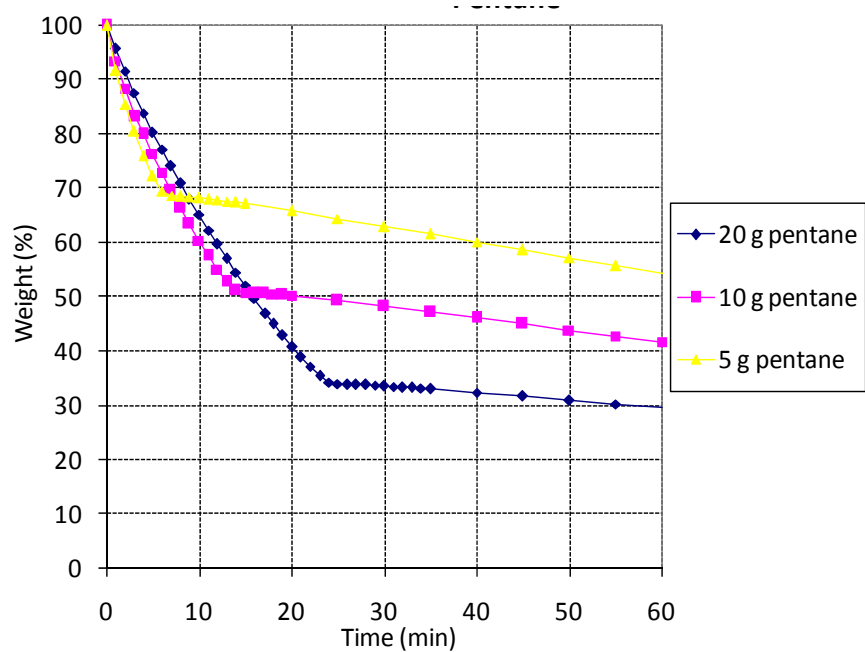


Figure C-7. Summary of vaporization curves: 10g water + varying pentane.

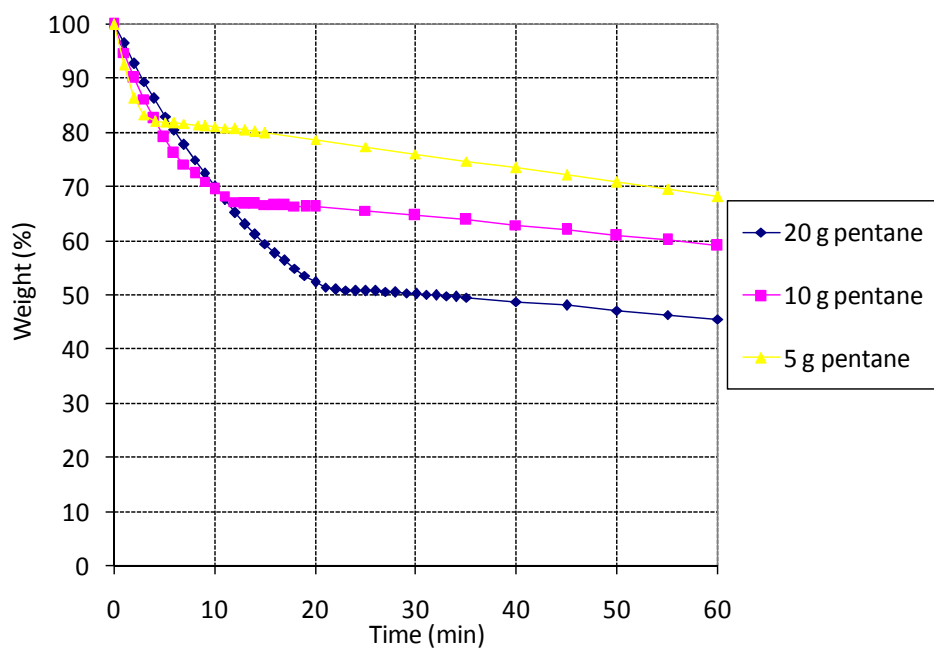


Figure C-8. Summary of vaporization curves: 20g water + varying pentane.

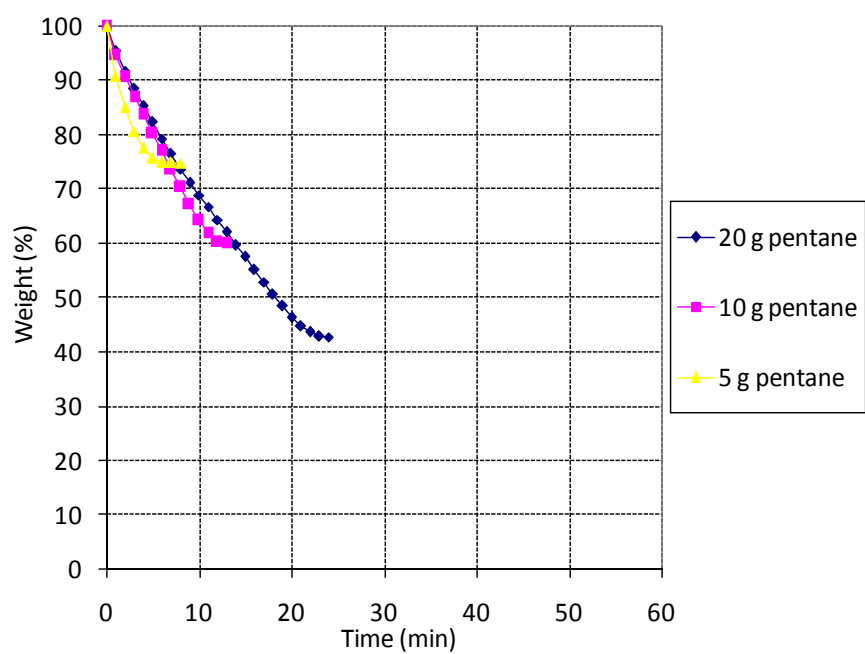


Figure C-9. Summary of vaporization curves: varying pentane + 15g coal.

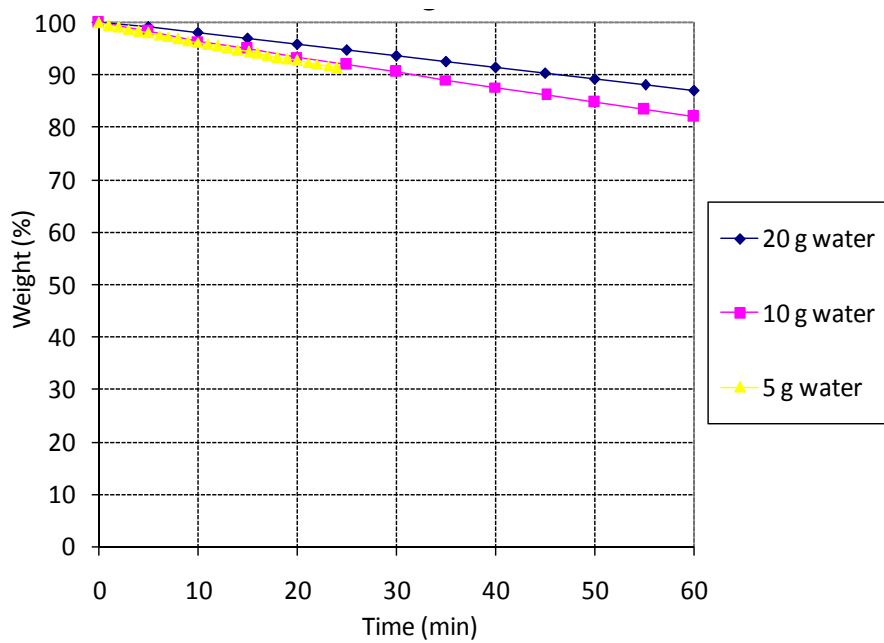


Figure C-10. Summary of vaporization curves: varying water + 15g coal.

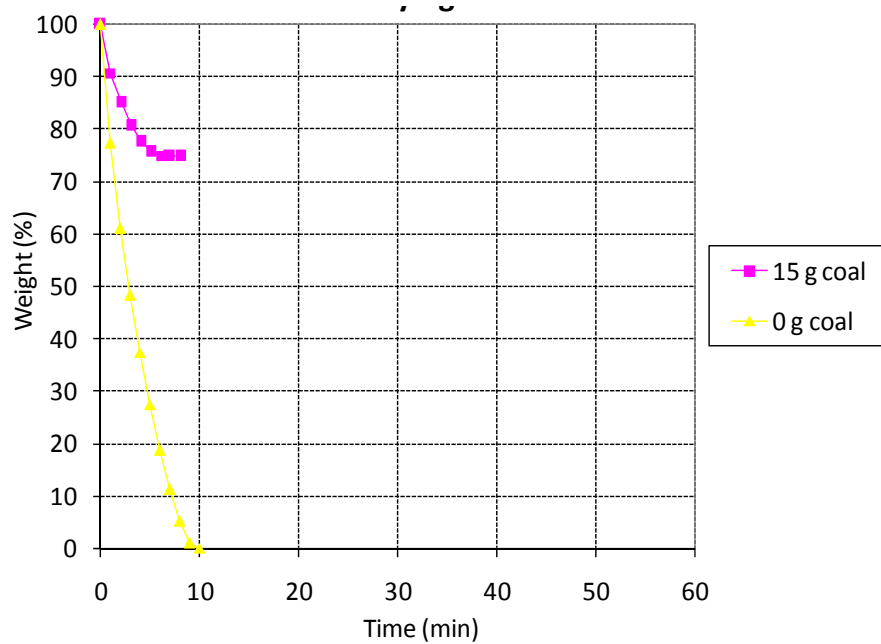


Figure C-11. Summary of vaporization curves: 5g pentane + varying coal.

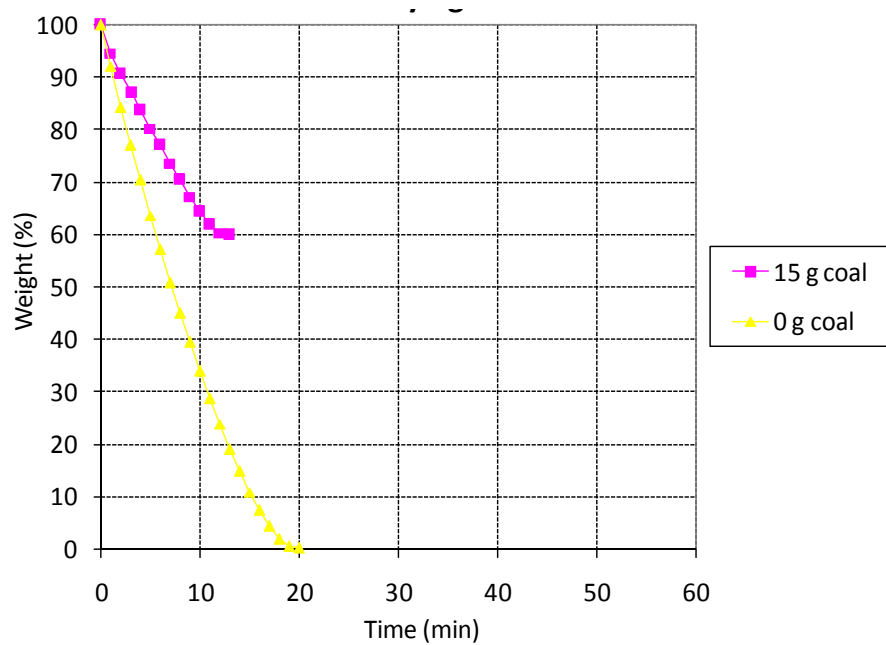


Figure C-12. Summary of vaporization curves: 10g pentane + varying coal.

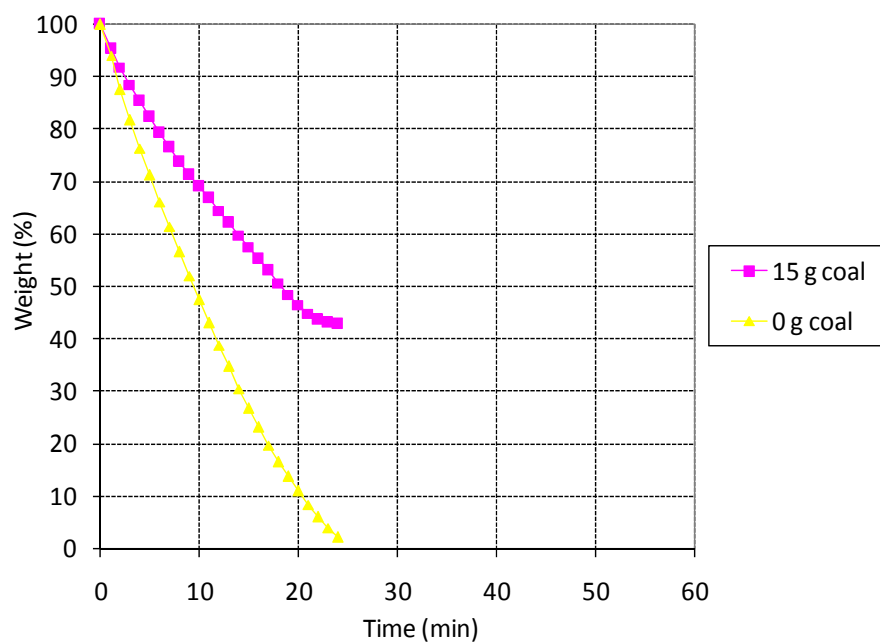


Figure C-13. Summary of vaporization curves: 20g pentane + varying coal.

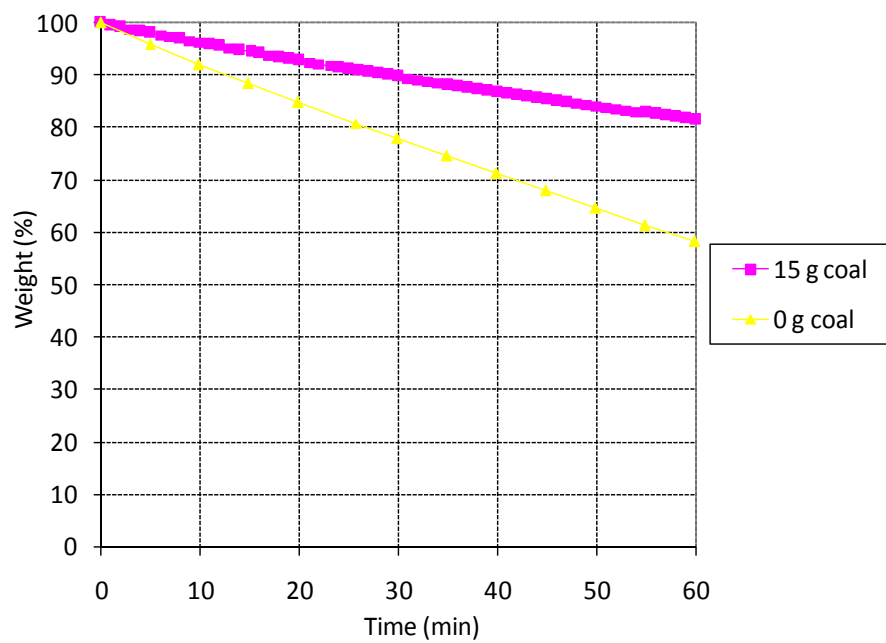


Figure C-14. Summary of vaporization curves: 5g water + varying coal.

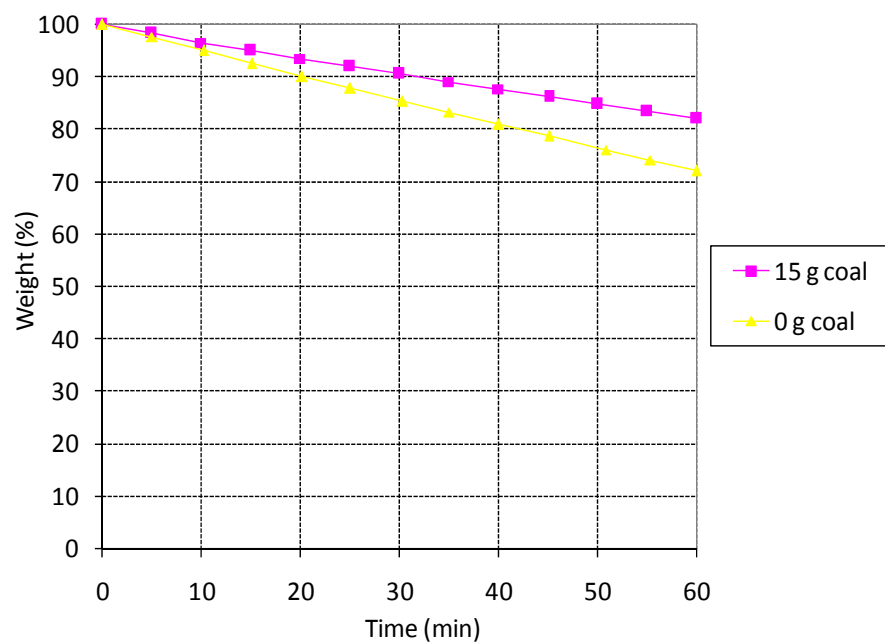


Figure C-15. Summary of vaporization curves: 10g water + varying coal.

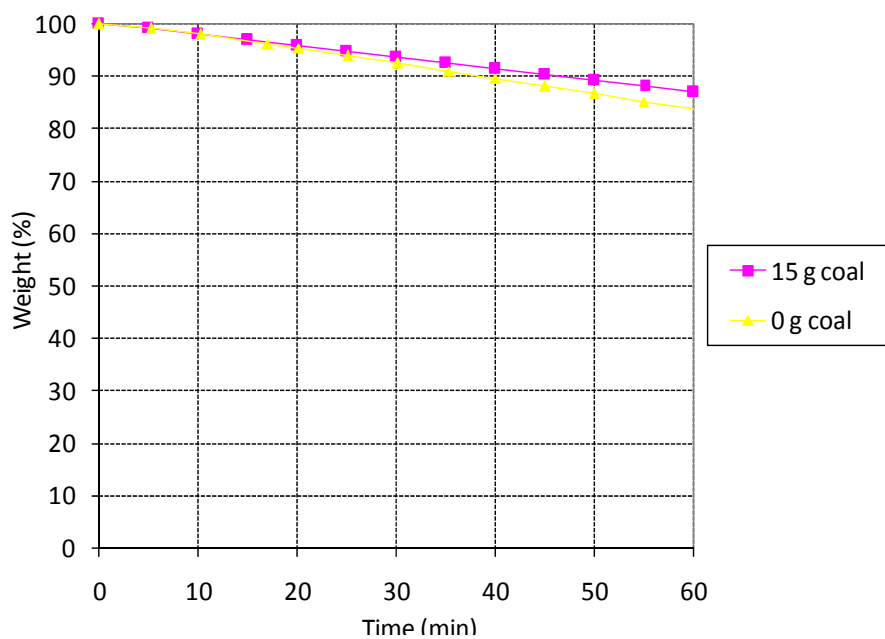


Figure C-16. Summary of vaporization curves: 20g water + varying coal.

Figures C-17 through C-32

The next sixteen graphs show trends in evaporation rates with known amounts of water, pentane, and coal. The graphs were produced with a moisture balance and correspond to Figures C-1 through C-16.

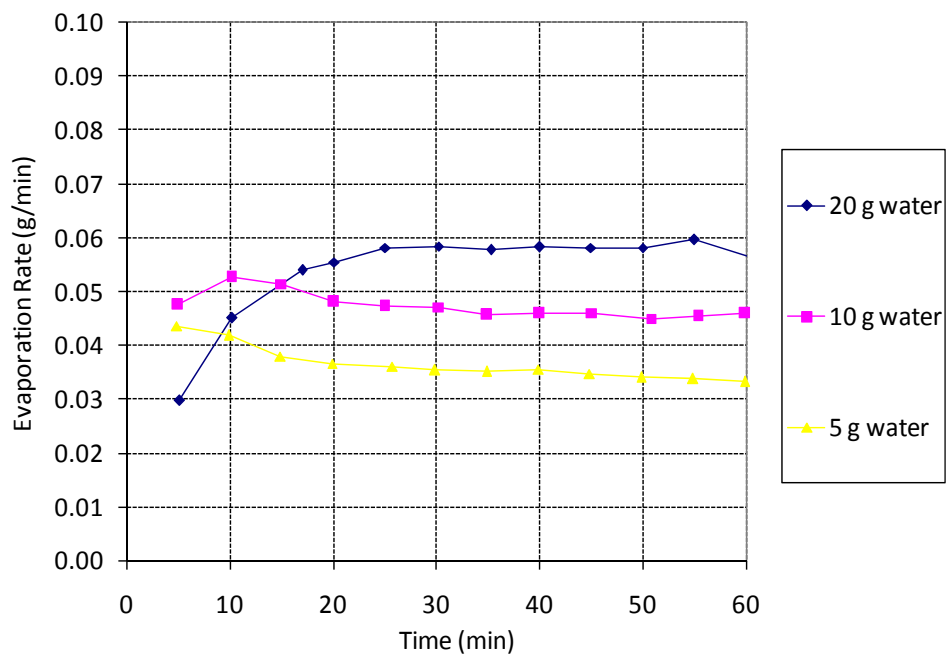


Figure C-17. Summary of vaporization curves: varying water.

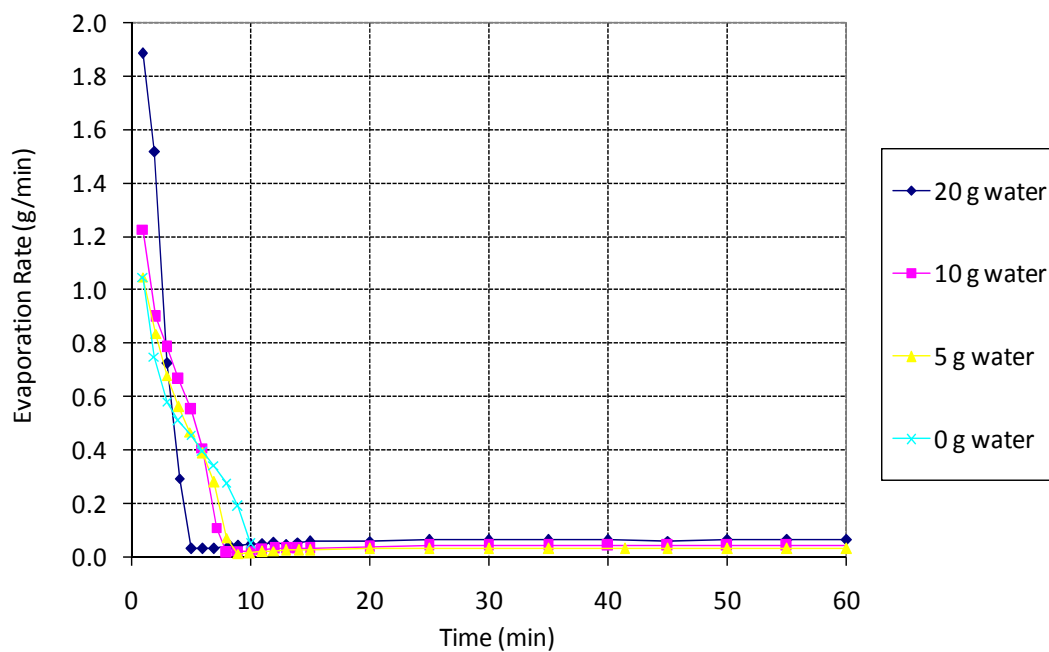


Figure C-18. Summary of vaporization curves: varying water + 5g pentane.

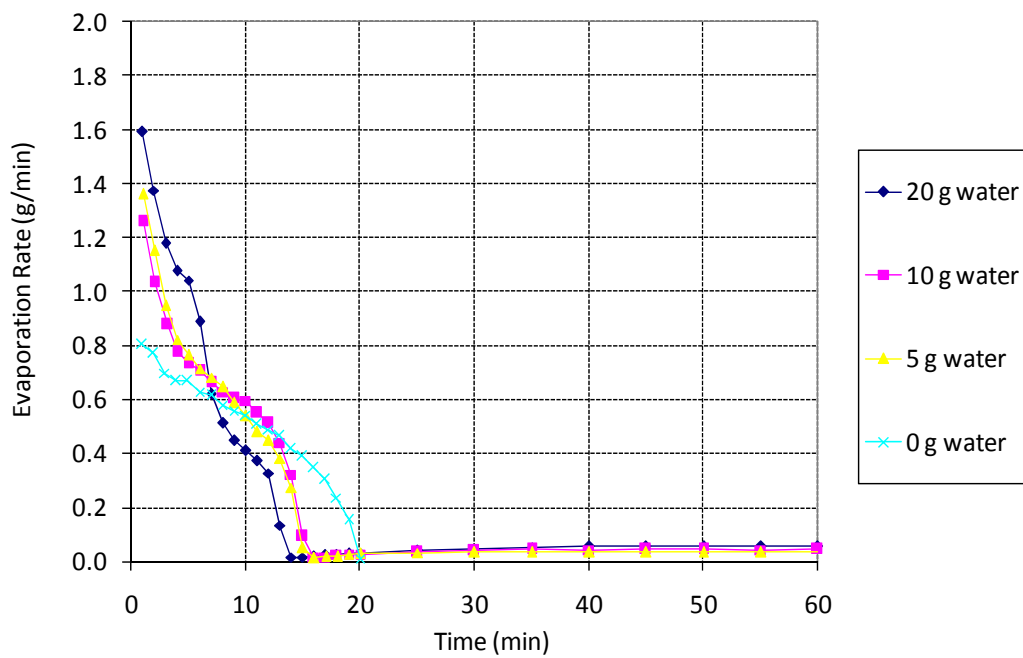


Figure C-19. Summary of vaporization curves: varying water + 10g pentane.

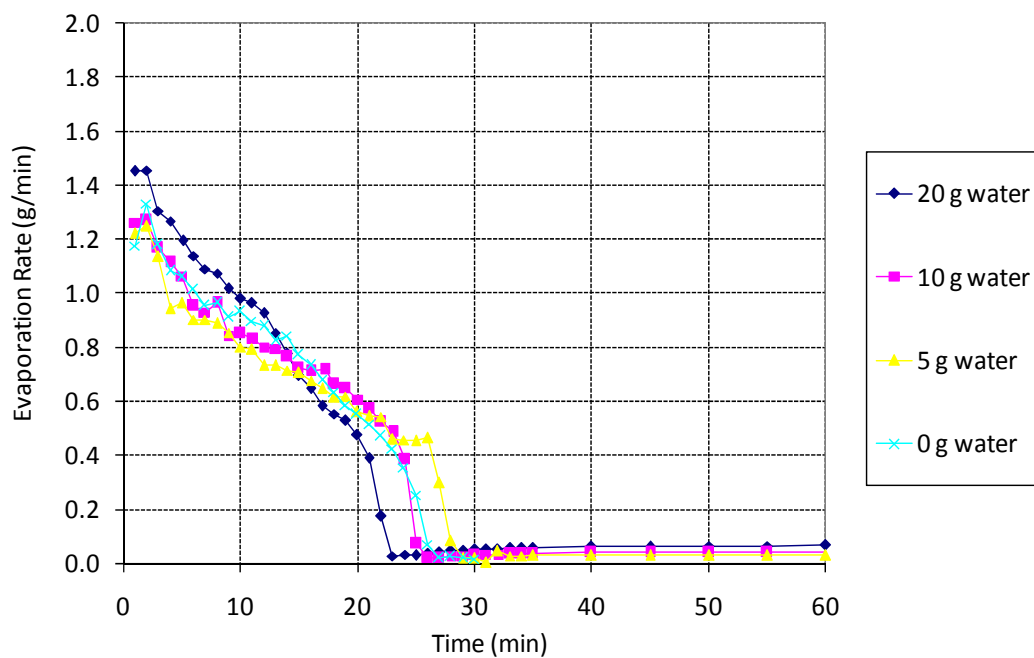


Figure C-20. Summary of vaporization curves: varying Water + 20g pentane.

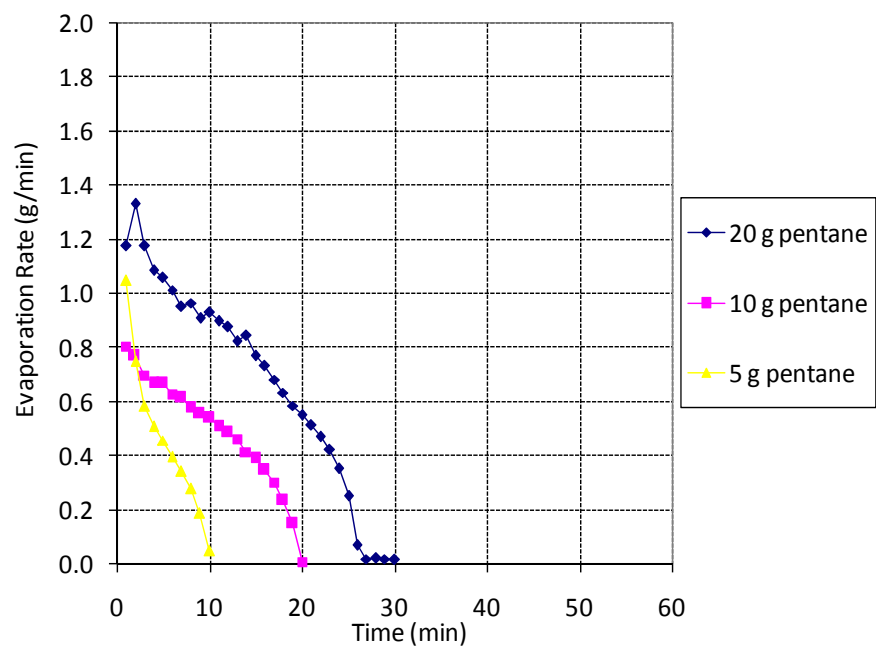


Figure C-21. Summary of vaporization curves: varying pentane.

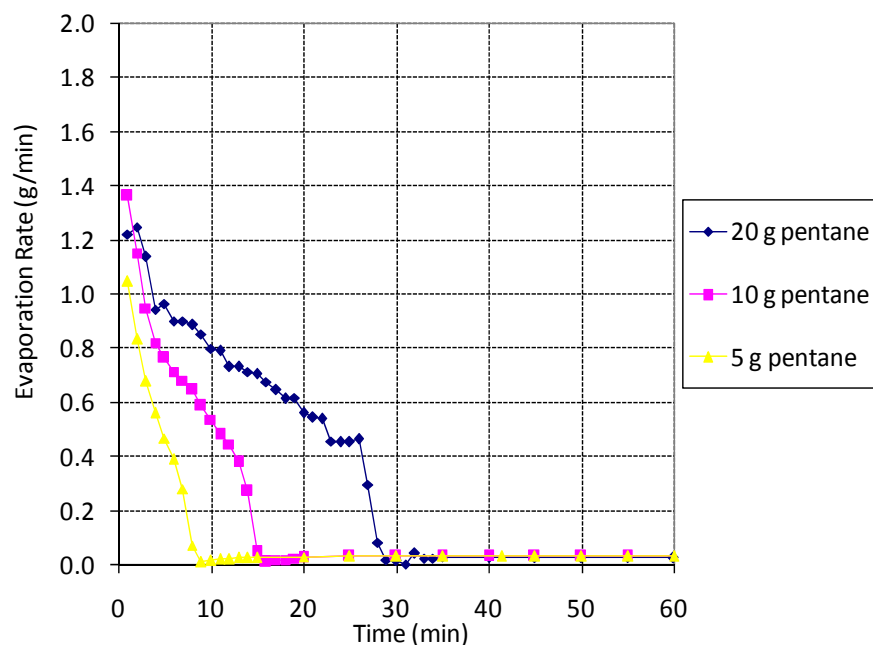


Figure C-22. Summary of vaporization curves: 5g water + varying pentane.

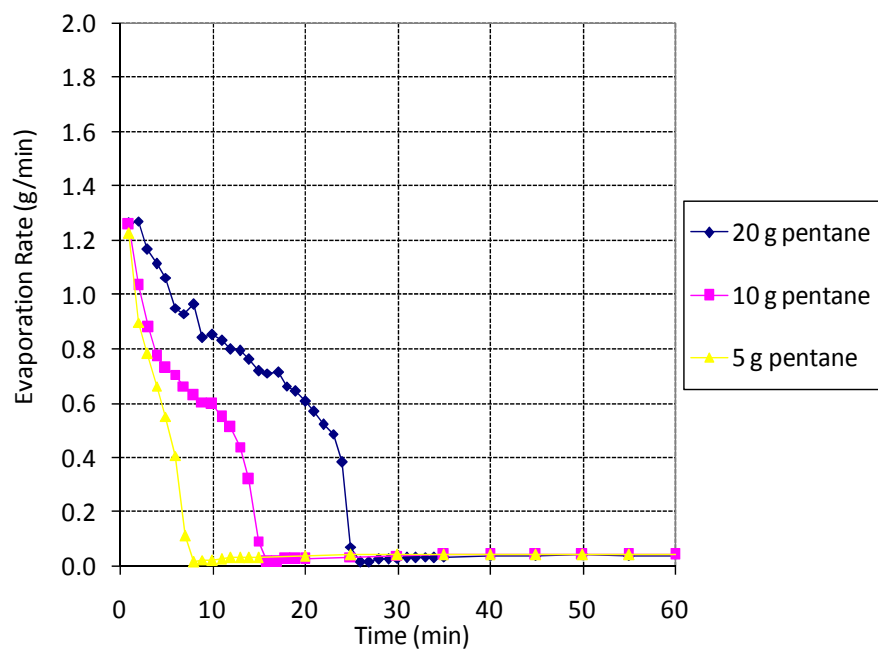


Figure C-23. Summary of vaporization curves: 10g water + varying pentane.

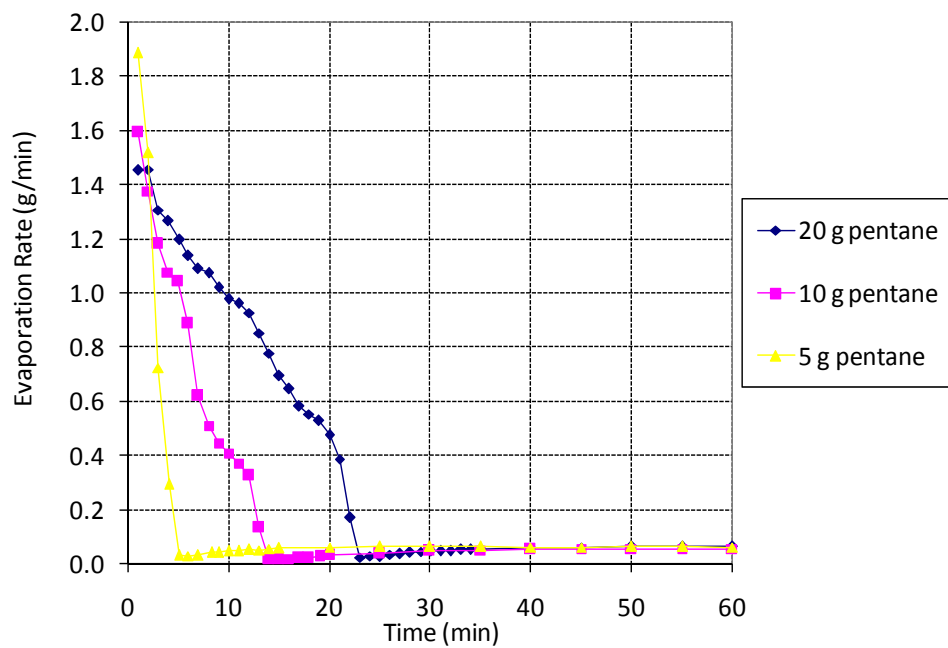


Figure C-24. Summary of vaporization curves: 20g water + varying pentane.

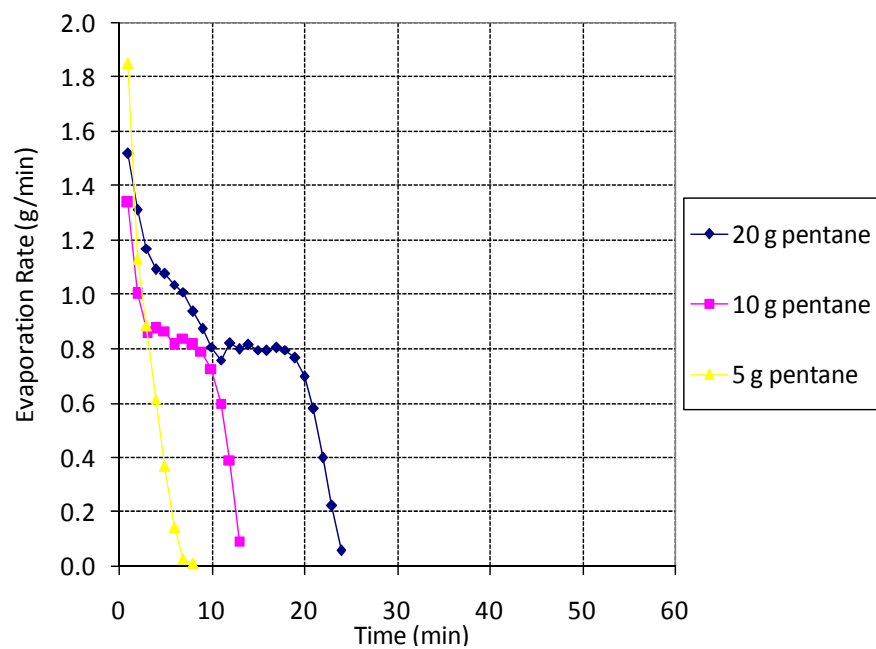


Figure C-25. Summary of vaporization curves: varying pentane + 15g coal.

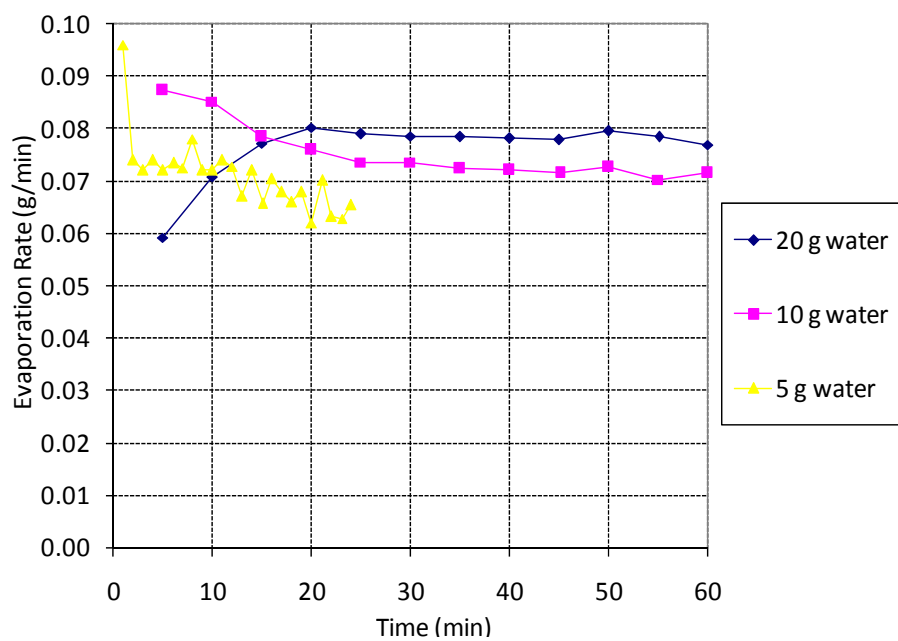


Figure C-26. Summary of vaporization curves: varying water + 15g coal.

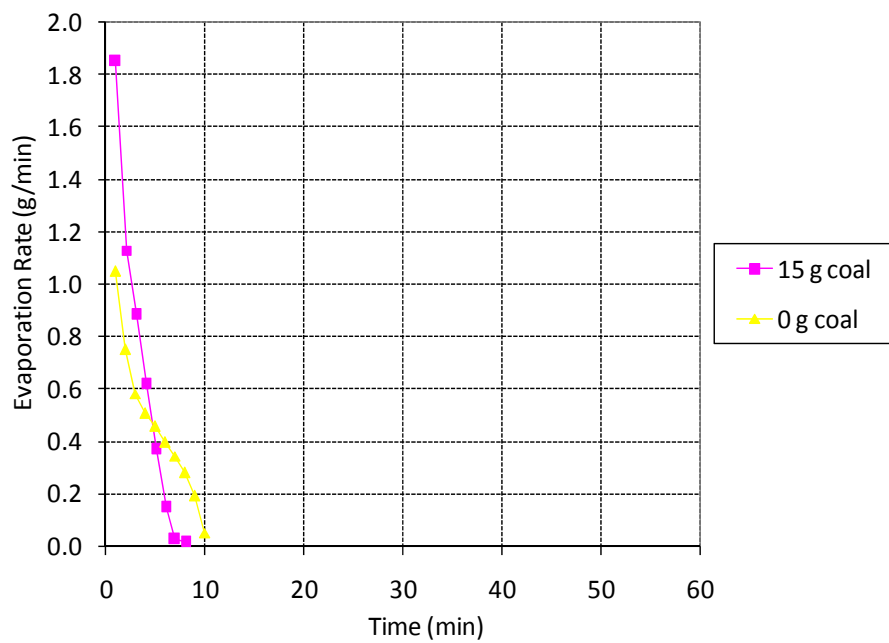


Figure C-27. Summary of vaporization curves: 5g pentane + varying coal.

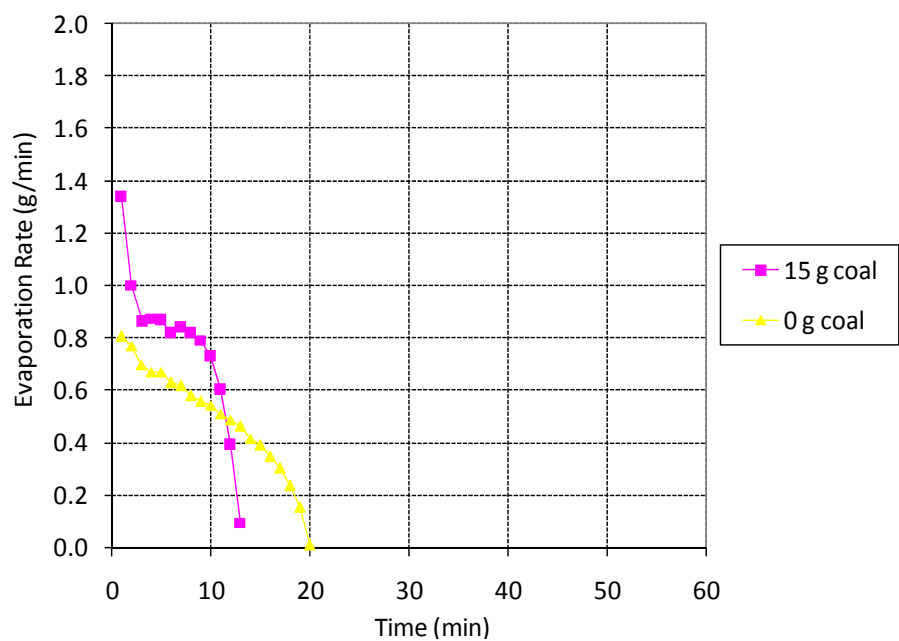


Figure C-28. Summary of vaporization curves: 10g pentane + varying coal.

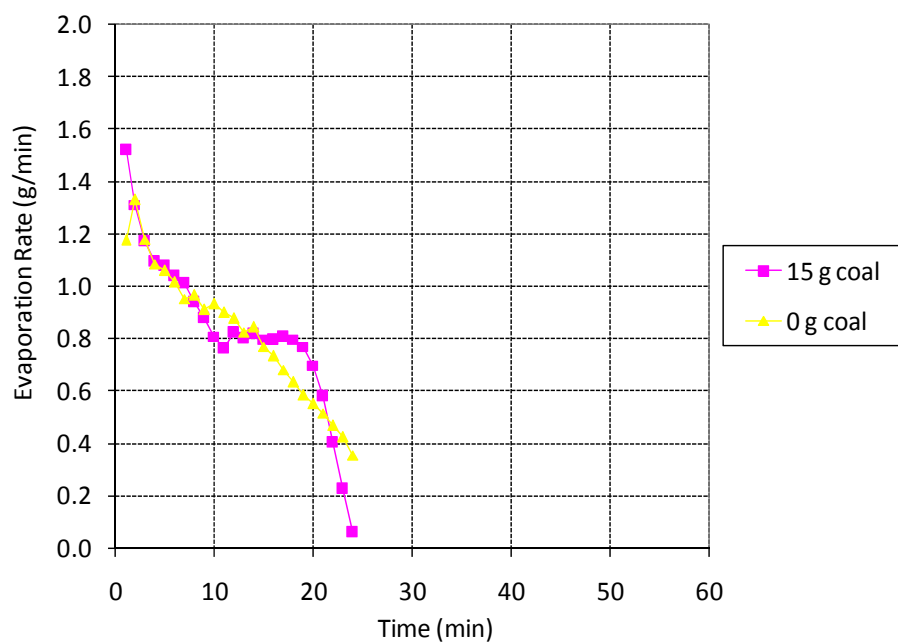


Figure C-29. Summary of vaporization curves: 20g pentane + varying coal.

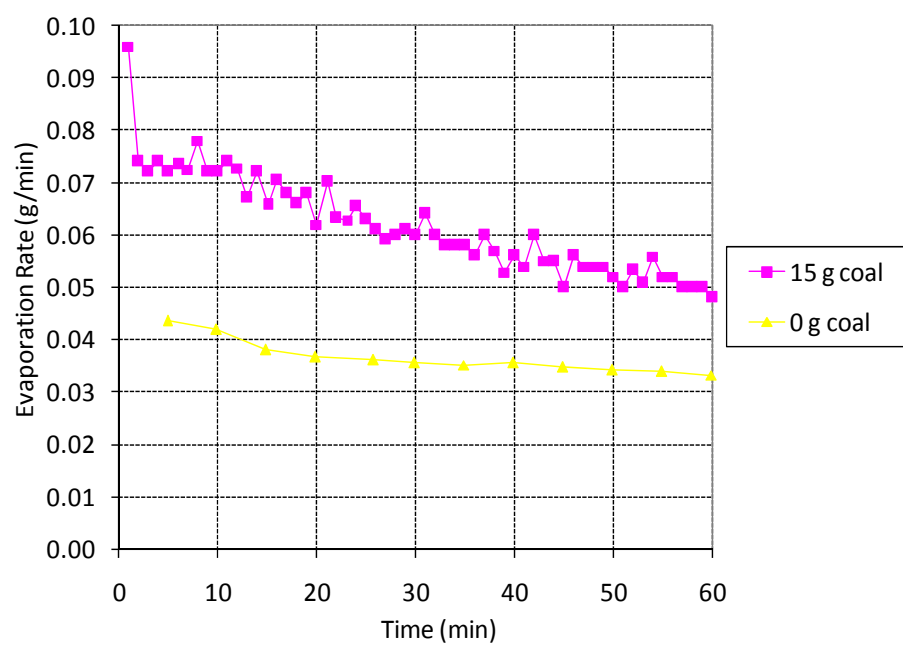


Figure C-30. Summary of vaporization curves: 5g water + varying coal.

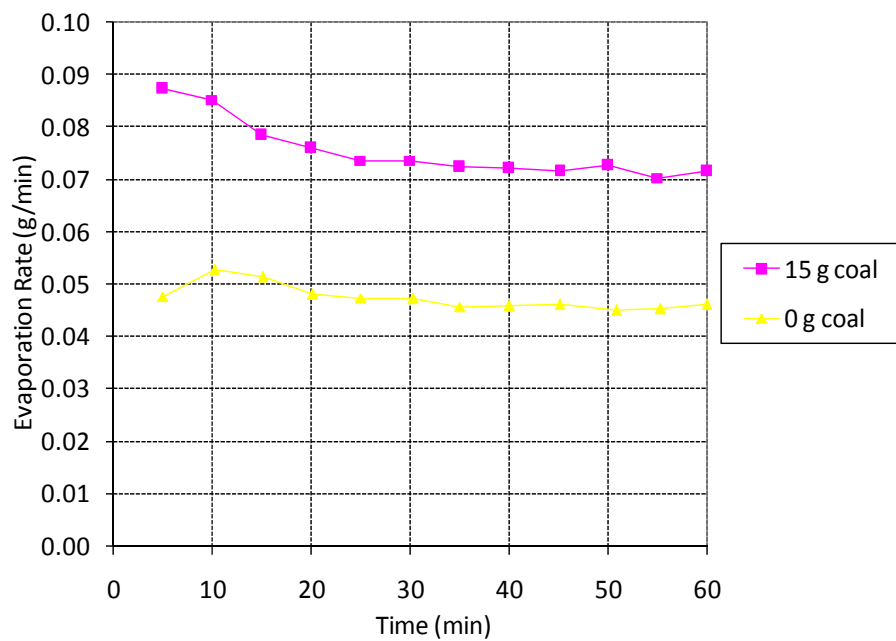


Figure C-31. Summary of vaporization curves: 10g water + varying coal.

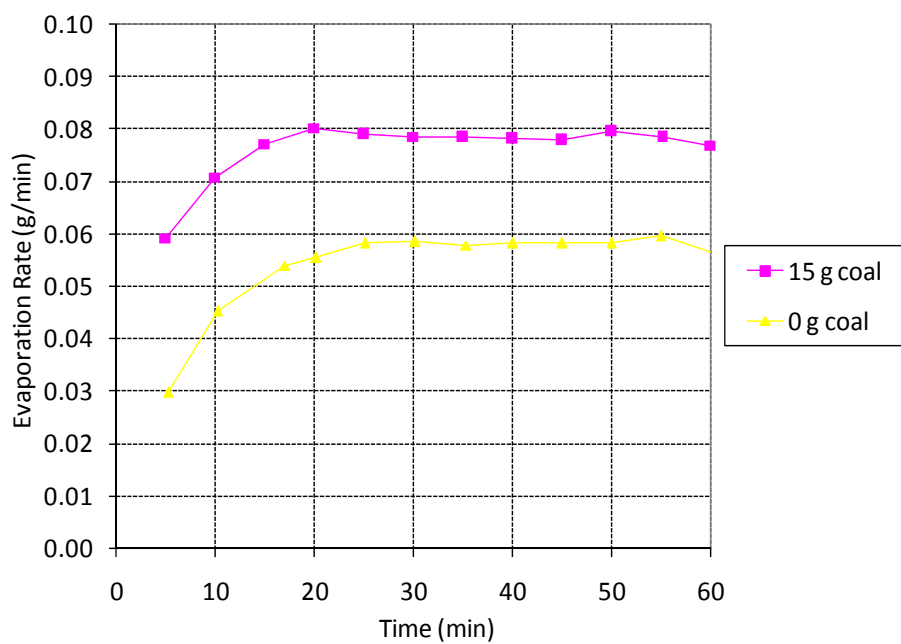


Figure C-32. Summary of vaporization curves: 20g water + varying coal.

Discussion

Several trends may be identified from the previous 32 figures. They are numbered below for ease of reading.

1. As the amount of water increases, the evaporation rate of pentane increases (Figures C-21 through C-24). In other words, the starting evaporation rate is higher, and the pentane tends to finish evaporating in a shorter amount of time. Pentane has no affinity for water, and the increased presence of water causes the pentane to evaporate faster in order to decrease the amount of liquid in contact with water.
2. As the amount of pentane increases, the starting evaporation rate of pentane decreases and the duration of the gently sloping, moderate evaporate rate of the first leg increases (Figures C-17 through C-20). In other words, changes in pentane's evaporation rate occur more slowly as the volume of pentane increases since pentane has an affinity for itself. The increase in pentane also minimizes the amount of pentane in contact with water; water tends to reject pentane and cause it to evaporate faster.
3. As the amount of coal increases, the evaporation rates of both water and pentane increase (Figures C-27 and C-28). The liquid is spread thinly over the large surface area which aids in evaporation and decreases the time needed for liquid removal.
4. Unlike water, pentane is not characterized by a constant evaporation rate (Figure C-21). Water's evaporation rate tends to remain relatively constant or displays small constant changes. Small variations may be noted at the beginning and likely the end (not seen here) of water evaporation (Figure C-17). This is consistent with pentane's trends; however, the change in pentane's evaporation rate vary more widely.

Pentane's weight versus time curves display a distinctive flattening of the slope as the last of the pentane evaporates. This trend is present when only pentane or pentane mixed in coal is being tested. Therefore, it is reasonable to assume that a similar flattening of pentane's weight curve occurs when water is present.

Reasons for Initial Weight Choice

The initial weight used in moisture determination was taken after the evaporation rate became constant in the second leg of the weight curve (Figure 4-4). Traditionally, this value would be taken at the intersection of the two legs of the graph instead of after the elbow. Several reasons motivated this choice:

1. Reason 4 in the previous section explained that pentane shows a distinctive flattening of its evaporation curve when by itself or mixed with coal. It is assumed that a similar trend occurs when water is present; therefore, pentane is still present until the elbow of the evaporation curve is complete.
2. Moisture balance testing was done with known amounts of liquids. The known masses of pentane tended to be associated with the end of the elbow in the evaporation curve.
3. Due to the expense of pentane, it would need to be recycled in a commercial setting. Plants would conservatively evaporate a little extra water to ensure that all pentane was recovered; therefore, it is reasonable that they would evaporate, at a minimum, to the end of the elbow in the evaporation curve.