



Hot water drying and briquetting of lignites
by Kevin Jess Poor

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Science in
Chemical Engineering
Montana State University
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Abstract:

The objective of this research was to characterize the effects of hot-water drying on a Montana lignite and to see if this drying process changes the briquetting ability of the coal. A Savage lignite was hot-water-dried at temperatures between 200°C and 300°C in a cold-charge autoclave. Hot and cold off-gases of the drying processes were tested for their contents. The dried coals were tested for their moisture content, ash content, gross heating value and ultimate elemental contents. Briquets were made from the raw lignite and the dried coals using 5 different binders in varying concentrations. Water was included in the briquetting mixture in some trials. All briquets were tested for compressive strength.

The off-gases mainly consisted of CO₂ and nitrogen. With increasing drying temperature the dried coals exhibited a higher carbon content, lower oxygen content and a higher heating value than did the raw lignite. The moisture content of the lignite decreased with increasing drying temperature. An increase in the moisture reduction occurred at a drying temperature between 240°C and 260°C. This is a temperature where significant CO₂ production has been shown previously to occur.

Compressive strength of briquets made without water decreased with increasing lignite drying temperature. Briquets made with water as part of the briquetting mixture showed a compressive strength minimum with respect to drying temperature for briquets made with a lignite dried at 240°C. Binder choice made little difference in briquet strength for briquets made using lignite dried at 300°C. Binder choice had a larger impact for briquets made with the raw lignite. Water in the briquetting mixture didn't increase briquet moisture content.

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A thesis submitted in partial fulfillment
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of

Master of Science

in

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Bozeman, Montana

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ABSTRACT

The objective of this research was to characterize the effects of hot-water drying on a Montana lignite and to see if this drying process changes the briquetting ability of the coal. A Savage lignite was hot-water-dried at temperatures between 200°C and 300°C in a cold-charge autoclave. Hot and cold off-gases of the drying processes were tested for their contents. The dried coals were tested for their moisture content, ash content, gross heating value and ultimate elemental contents. Briquets were made from the raw lignite and the dried coals using 5 different binders in varying concentrations. Water was included in the briquetting mixture in some trials. All briquets were tested for compressive strength.

The off-gases mainly consisted of CO₂ and nitrogen. With increasing drying temperature the dried coals exhibited a higher carbon content, lower oxygen content and a higher heating value than did the raw lignite. The moisture content of the lignite decreased with increasing drying temperature. An increase in the moisture reduction occurred at a drying temperature between 240°C and 260°C. This is a temperature where significant CO₂ production has been shown previously to occur.

Compressive strength of briquets made without water decreased with increasing lignite drying temperature. Briquets made with water as part of the briquetting mixture showed a compressive strength minimum with respect to drying temperature for briquets made with a lignite dried at 240°C. Binder choice made little difference in briquet strength for briquets made using lignite dried at 300°C. Binder choice had a larger impact for briquets made with the raw lignite. Water in the briquetting mixture didn't increase briquet moisture content.

INTRODUCTION

Large quantities of easily mined, relatively low sulfur lignite and sub-bituminous coal lie mostly unused in Montana. In fact, as of 1976, Montana had 120.6 billion tons of demonstrated coal reserves (1), largest of any state in the U.S. Despite these huge reserves, only about 32.87 million tons were mined last year in Montana (2). It is estimated a similar amount will be mined in 1986. This would rank Montana tenth nationally out of a total estimated U.S. production of 901.9 million tons.

Several reasons appear to account for the apparent under-use of these vast energy reserves. In addition to the currently very soft market for coal of all kinds, the coal in Montana is generally high in moisture content, 20-40 percent, and correspondingly low in heating value. These attributes make it uneconomical to transport coal over the great distances to the major coal markets in the mid-west. However, new federal pollution standards (3) of 1 pound of SO_2 per 10^6 B.T.U., as opposed to the previous standards of 1 pound of sulfur per 10^6 B.T.U., make Montana's low sulfur lignite more attractive to major coal users.

The thrust of this investigation is to obtain technical

data on steam/hot-water drying of a Montana lignite in order to analyze if it could become a more marketable product. Most of the previous work in this area have been looking at hot water drying, as a step in a proposed coal-slurry system. Because problems with water acquisition and right-of-way acquisition for pipeline siting have arisen for coal-slurry systems this investigation also explores the effect of hot water drying on the briquetting ability of the lignite. Briquetting the dried lignite will make it a more weather resistant and transportable product. Parameters being investigated include the effect of drying temperature on the coal moisture content, the reabsorption of water in the dried samples, the off-gas composition during drying, the elemental content of the product coal, and the briquet strength.

REVIEW OF LITERATURE

Major advantages of coal dewatering include decreased transportation costs, improved heating value, and a reduction of the likelihood of freezing during transport. In the case of fine coals, it is estimated that a 1% moisture reduction can reduce transportation costs by \$310,000 to \$1 million per year (4). It can be shown that a 1% increase in the moisture content of a fine coal can offset a 4.5% decrease in ash content.

At present, moisture reduction in coal is either done by mechanical means, such as filtration or centrifugation, or by thermal processes by heating the coal with hot gases or steam. An alternate dewatering technique is hot-water-drying of coal. It has been called the most under-used coal drying technology available today.

Steam drying is the treatment of low rank coal with saturated steam at pressure. It was first investigated by Fleissner in the 1920's (5). Treatment with steam removes water, causes shrinkage, and results in a stable lump of coal with improved weathering properties (6).

Klein (7) first speculated as to the mechanisms involved in steam drying. During treatment, carboxylic

groups in the coal decompose, forming carbon dioxide which pushes water out of the pores of the coal. Secondly, chemical reaction causes the coal surface to become more hydrophobic. Additionally, water has a lower viscosity at high temperatures, allowing it to run more freely from the coal.

The Fleissner (steam drying) process has been studied extensively with a variety of coals, and has been used commercially in Europe since 1927 (6).

Rao and Wolff steam dried a sub-bituminous C coal and found a 20 percent weight reduction of 2 inch chunks in 5 minutes with 550 psia steam (8). They reported a 30 percent increase in heating value and found their dried coals to be as stable as their raw coals. Although they had some moisture readsorption at 98 percent humidity, the weight gain was much less than the weight loss during drying.

Rozgonyi and Szigeti steam dried a high moisture content lignite at pressures up to 4.053 MPa, 588 psia (9). About 80 percent of the total lignite moisture was removed. Smaller sizes of coal released their water more easily and exhibited better heat transfer characteristics. A smaller coal particle will also have a shorter distance for the water to travel for it to get from the inside of the coal particle to the surface. Rozgonyi and Szigeti assumed that the moisture content of a coal cannot be reduced to zero

without a radical reorganization of the coal matrix. Each grain size of a lignite has a minimum water content at different pressures and temperatures. This critical water content cannot be reduced without a drastic change in the coal. A gross calorific value increase of 50 to 75 percent was achieved in their work.

Koppelman(10) has patented a lignite drying process where the coal is put into an autoclave and heated to between 750°C and 1250°C. During cooldown, the coal is then contacted with the off gases that were created during heating. This allows the deposition of any of the condensable gases onto the coal.

Hot-Water Drying

More recently, hot water, under pressure, has been used to dewater coal. Hot-water drying is advantageous in that all of the water expelled from the coal comes out in the liquid form. The latent heat of vaporization for the water in the coal does not have to be supplied. Additionally, because the coal is under water, the explosion problems associated with the thermal drying of fine coal are avoided. Hot-water drying reduces the sodium content of the coal,

leaving it dissolved in the water. This lower sodium content helps reduce ash fouling during combustion. Significant sulfur extraction has also been reported (10,11).

Baria, Maas, and Paulson (12) have shown that hot-water drying would work well in a slurry system as some of the slurry water could come from the water driven out of the coal during the drying process. Baria et al. studied hot-water drying of lignite with cold-charge and hot-charge autoclaves. They found that moisture reduction was completed in the first five minutes of drying and that the moisture removal is a function of the interior temperature of the particle. In the cold-charge tests, moisture reduction was a linear function of drying temperature. The hot-charge tests showed a higher drying rate above 240°C, coinciding with the temperature at which significant carbon dioxide forms to increase the water removal in the coal pores. A decrease in carboxylic acid groups was found with increasing temperature. They found a significant decrease in carboxyl groups in coals dried above 260°C. Baria, Maas, and Paulson found that the gross heating value of the coal increases with temperature. They also reported significant sodium removal during treatment.

Maas (11) hot-water dried a North Dakota lignite in a cold-charge system, and found that the major portion of the gases left in the autoclave after cool down was carbon

dioxide, but that there were also very significant concentrations of nitrogen. In this case, the gross heating value ranged from 6225 calories per gram when dried at 200°C, to about 6500 calories per gram when dried at 280°C. Maas found a decrease in the sodium content of the coal, but no significant decrease in the ash content of the coal.

T.A. Potas et al. (13) found that the amount of organic matter lost to gases and liquids vary, depending on the processing time and temperature. They also found reductions in the mineral content of the coal. Under certain conditions with given coals, all of the sodium could be removed. However, they also found a reduction in ash content occurred during treatment in a hot-charge autoclave. The particle sizes of their coal also became smaller with increasing processing temperature.

Lynch and Webster (14) used proton nuclear magnetic resonance, N.M.R., to show that there is a rapid reduction in the water binding ability of coals under heat treatment above 150°C.

Cole et. al.(15) patented a technique for upgrading low rank coals. In this system a coal/water slurry is heated to above 300°F. After cooldown, a hydrocarbon liquid is added to the slurry. The hydrocarbon forms an agglomerate with the coal, making it easier to separate from the water.

Coal Briquetting

Briquetting converts a solid fuel into one of higher quality. Briquetting consists of applying pressure to a mass of particles to compact them into a single agglomerate, sometimes with the aid of a binder (6). The important interactions in briquetting are the cohesive forces between solids, and the adhesive properties of the binders.

Briquetting has been used commercially for over 200 years. Most briquetting knowledge is empirical in nature, as opposed to exact physical or chemical theory. Generally, the higher the rank of coal, the harder it is to briquet without a binder. In many places, especially Europe, brown coal briquets are made without any binder. Most U.S. coals are of high enough rank that a binder is required.

A review of the basic fundamentals of coal briquetting by Berkowitz (16) contrasts the differences between briquetting with, and without, a binder. Without a binder the strength of the briquet is limited by the characteristics of the coal; with a binder, the briquet strength is determined by the coal characteristics, the binder dispersal, and by the interaction between the coal and the binder.

Berkowitz goes on to define the variables involved in briquetting. In binderless briquetting, maximum briquet strength will occur when both the modulus of elasticity and the rigidity modulus are low. First order variables in binderless briquetting include time, pressure, temperature, degree of comminution, and the presence of a film-forming material in the solid.

Time is involved because any process where deformation of a solid is involved is a rate process. Pressure increases produce larger deformations of the solid, and temperature increases reduce the modulus of elasticity of the solid.

The degree of comminution is a variable involved with the size of the coal particles. It involves the particle size range, the maximum particle size, and the particle size distribution. Briquet strength is proportional to the packing density of the particles. The maximum briquet strength is where the particle size distribution allows the maximum packing density. Generally, the larger the top particle size, the weaker the briquet. However, if the particles are too small they can act as air traps and reduce briquet strength.

The presence of a film-forming material, such as water, influence briquet strength by 1. acting as a lubricant, allowing the particles to rearrange into a more dense briquet, 2. reducing the surface tension of the solid

allowing for increased deformability, and 3. extending the range of the solid-solid cohesive forces.

When briquetting with binders, interactions between the binder and the coal particles can become much more important than the characteristics of the coal. Many substances can be used as binders: asphalts, pitches, starches, cement binders, and sulphate liquor are common. For coal briquetting, binders should be cheap and should not increase ash content or decrease heating value. Binders should have an affinity for the coal surface. Different binders have different affinities for different coals. The best binder for one coal-briquetting application may not be the best binder for a different coal.

In binderless briquets moisture was shown to be a factor in high briquet strength, but in briquetting with a binder it can reduce briquet strength. A binder may adhere to the water, instead of to the coal surface. The contact angle a binder forms with the coal surface can affect the strength of the briquet. Binders with contact angles less than 90° will wet the coal surface and give higher adhesion energies than binders with contact angles greater than 90° . Surface roughness will change the contact angle between the liquid and the solid (17). If the contact angle is less than 90° , roughness lowers the angle, while if it is greater than 90° , roughness increases the contact angle.

Lin (18) studied thermally dried coals and briquetting ability. He used varying concentrations of corn starch, Kraft sulfate liquor, and #6 fuel oil as binders. A briquet made with 5 percent corn starch and 95 percent dried coal was found to be best.

Research Objective

The structure of this study will be first to characterize a hot-water-dried Montana lignite, and second to investigate the dried lignite's briquetting ability using various binders. The coal will be hot-water dried over a range of temperatures. Samples of the hot off-gases will be tested for their contents. Elemental content of the dried coals will be determined, along with moisture content and heating value. Briquets will then be made and tested for strength, using five different binders in varying concentrations.

EXPERIMENTAL

The coal-water contacting vessel consists of a high pressure bomb with four ports in the top: one port for a pressure gauge, another for a thermowell, a third port for a helium flush, and the fourth port for a gas exit line. The vessel is surrounded by a steel sleeve, a heating mantle, and insulation to facilitate good heating (Fig 1).

As illustrated in Figure 1, the gas exit line has one valve adjacent to the outlet of the bomb to allow a gas sample to be obtained during an experiment. A second needle valve, further up the line, traps a sample and acts as a water trap. Downstream from the second valve are a desiccant water trap and a third valve leading to a vacuum pump and allowing for evacuation of the entire system. The gas exit line continues on to a gas sampling valve leading to a gas chromatograph and ends in a valve allowing for a completely closed system. All valves are needle valves. Up to the desiccant water trap the exit line is constructed of 1/4 inch stainless steel tubing, i.d. 0.167 inch. The balance of the line is constructed with 1/8 inch tubing, i.d. 0.042 inch.

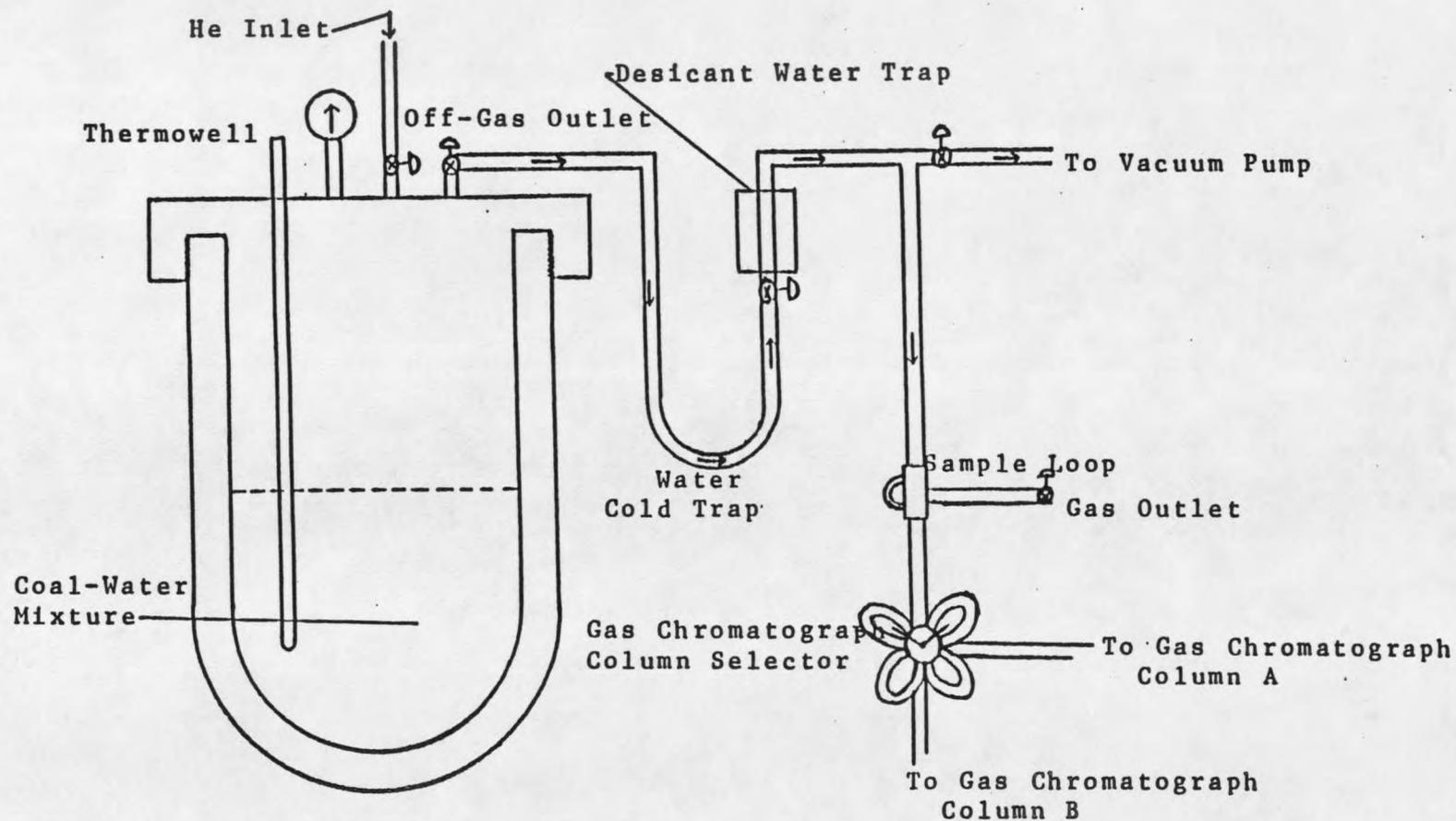


Figure 1. Coal/Water Contacting Vessel and Gas Sampling Apparatus.

The gas sampling valve, at the end of the gas exit line, is connected to an eight port valve which allows injection of the sample into either of the two columns in the gas chromatograph. One column is packed with Moleseive 5A, and detects oxygen, nitrogen, methane, carbon monoxide, and ethane. The second column is packed with Porpack Q, and detects ethane, oxygen, nitrogen, carbon monoxide, ethane and carbon dioxide.

Briquets were made on a Rimac spring tester, (Rinck-McIlwaine Inc.), with a stainless steel die. In the making of briquets every effort was made to insure consistency of conditions between experiments. In all cases a hand press was used to make cylindrical pellets of diameter 0.5 cm. A pressure of 245 p.s.i. was sustained on the briquetting mixture for a period of 20 seconds. All briquets were stored in airtight containers.

Drying Procedure

Before making a run the entire gas exit line is evacuated overnight to remove water, air, or any other contaminants.

The experiments in this investigation were done using a

Savage lignite supplied by Montana-Dakota Utilities. This coal was ground with a mortar and pestal to between 0.0328 inches and 0.0098 inches, 20-60 mesh, and dried within 24 hours of grinding. The ground coal was kept in a sealed container until just prior to use.

For the coal-water slurry a known weight of coal is put in the bomb with an amount of demineralized water such that the liquid-solid ratio is 2:1 by weight. The bomb is sealed and put in the heating mantle where it is connected to the gas exit line, helium flush, and thermowell.

Helium, at thirty p.s.i.g., is let into the vessel to check the vessel for leaks. Helium is then let into the gas exit line and all connections are pressure tested for leaks. The end valve in the gas exit line is then opened and the entire system is purged for fifteen minutes. This rids the system of much of the air that was in the bomb when it was sealed. The gas purge is turned off and the pressure in the bomb is reduced so that an excessive overpressure is not present during the coal-water contacting. To ensure that the majority of the air is out of the system a sample of gas from the exit line is put into the gas chromatograph. Any air in the system will come out as a peak on the recorder chart. If all of the air is not out of the system, the helium flush is continued.

The bomb is then isolated from the exit line. The

heating mantle is turned on, and the bomb is allowed to heat to the desired temperature. During the coal-water slurry heating the exit line is again evacuated with a vacuum pump.

The bomb is allowed to heat to four degrees below the desired temperature, which takes approximately two hours. A heating profile of the bomb is provided in Table 5, located in the Appendix. The heating mantle is then turned down to twenty percent of full power. The bomb continues to heat to the desired temperature and stays there for at least fifteen minutes before starting to cool. If the heat is completely turned off, the bomb starts to cool off too rapidly. The bomb is considered to be at the desired temperature when it is plus or minus 2°C of the nominal temperature.

After the coal-water slurry has been at the desired temperature for fifteen minutes a sample of the steam and off gases are let into the cold trap. Here they are cooled to approximately -30°C with an antifreeze-water mixture frozen with liquid nitrogen. After the off-gas sample has been frozen for at least five minutes to get rid of the majority of the water, it is let into the rest of the gas exit line. Any water not frozen out in the cold trap is absorbed on the desiccant. The desiccant was periodically changed to insure a water-free gas sample. Samples were then drawn and injected into the gas chromatograph for exit gas analysis. On some runs an additional gas sample was

taken after the bomb was allowed to cool to room temperature and compared with those taken hot.

After the bomb had cooled it was disassembled, and the coal-water mixture was filtered for one hour in a 11.5 cm Buchner funnel. The filtered, hot-water-dried coal was then stored in air-tight containers while awaiting further testing.

Briquetting

Although finding the optimum binder was not a goal of this research, several different binders were tried in an attempt to produce a viable product. Fuel oil, corn starch, wheat paste, vinyl wallpaper paste, and a water-soluble concrete binder (Elmers Concrete Bonder) were tried in varying concentrations. Briquetting schemes with, and without, the addition of water were tried. Water was added in some cases to see if any of the binders could be used where the binder is added directly to the coal slurry and immediately briquetted without further filtering or treatment.

A good quality corn starch paste was prepared to combine with the dried coal in the corn starch pellets. The corn starch paste was made by first heating a mixture of 5 parts water, one part dry corn starch at 60°C with constant

stirring. After the mixture thickened to the point where the magnetic stirrer would no longer turn, the lid was removed. The mixture was then stirred by hand until a good quality paste was obtained, less than one minute. Initially the lid was in place to reduce the amount of water evaporating during heating, thus keeping the paste close to the original 5-1 ratio.

The starch paste is an amalgam of dry starch and water. The briquets are based on the weight of dry starch in the paste, not on the weight of the paste. To make the briquets using corn starch as the binder, an amount of starch paste was weighed out and an appropriate weight of coal was added to make the desired briquetting mixture. The starch paste was then folded into the coal until the paste was evenly distributed. The mixture was quickly briquetted and stored in airtight containers.

A similar procedure was followed in making the briquets with the other binders. A quantity of binder was weighed and combined with the desired amount of coal, water, quickly briquetted and stored until testing. In all cases the briquetting was done at the ambient air temperature. This was done because heating the briquetting mixture would change the moisture content of the briquets.

Analyses

Proximate analyses were done in accordance with standard ASTM methods D3172-73(19). A sample of coal weighing approximately 1 gram was put into a crucible and placed in an oven at 104 to 110°C for one hour. The weight change corresponds to the moisture content of the coal. This moisture-free coal was then covered and put in a 930-970°C oven for 7 minutes. The weight loss from this heating is the volatile matter. The sample was then placed, uncovered, in a 700-750°C oven until a constant weight is reached. The weight of the sample left is the ash content of the coal. The percent fixed carbon equals 100% minus the percentages of moisture, volatile matter, and ash.

Elemental analyses were done using a Carlo Erba Strumentazione ultimate analysis instrument (20). Samples of coal of between 0.5 and 1.5 mg were placed in tin containers cleaned with water, acetone, and carbon tetrachloride. This cleaning eliminated most of the dust and dirt on the containers, providing for more consistent, reliable results. These filled containers were incinerated at 1025°C for carbon, nitrogen, and hydrogen determination and at 930°C for sulfur determination. Elemental fractions are determined by comparing the exhaust-gases gas chromatograph signal of

these samples with known samples. Because of the inconsistencies inherent with coal work, multiple samples of all coals were incinerated to obtain a good average value of the elemental contents.

Heating value data were obtained using a Parr adiabatic bomb calorimeter in accordance with ASTM method D2015-66(21). Corrections were made for heat of formation of nitric acid, amount of fuse wire used, and for the amount of sulfur in each sample.

Compression tests on the briquets were performed on an Instron Universal Testing Instruments tension compression tester model TT-D, using a CCT compression cell(22). This cell has a resolution down to 0.01 pounds. Briquets were tested vertically, down the length of the cylinder. Results were given on a strip chart recorder, and given as a percentile of the total range of the instrument. The compression on the pellet at the moment of pellet failure was recorded. This corresponded to the maximum pressure placed on the pellet. The pressure at briquet failure is recorded in pounds force. To obtain the value in pounds per square inch the total pressure must be divided by 0.030 in.², the area of the end of a briquet.

RESULTS AND DISCUSSION

Table 1 shows the proximate analysis of a representative sample of the Savage lignite used in this study. It is reported in the as-received and moisture-free bases. The proximate analyses were performed on the same size coal as was used in the drying process, 20-60 mesh. As typical of lignites, this coal has a relatively high moisture content and correspondingly low fixed carbon content. High rank coals, like anthracite, can have fixed carbon contents of 95% or more.(23)

Table 1. Proximate Analysis of Savage Lignite.

Component	As Received %	MF %
Moisture	36.95	----
Volatile Matter	25.74	40.82
Fixed Carbon	30.78	48.82
Ash	6.53	10.36
	100.00	100.00

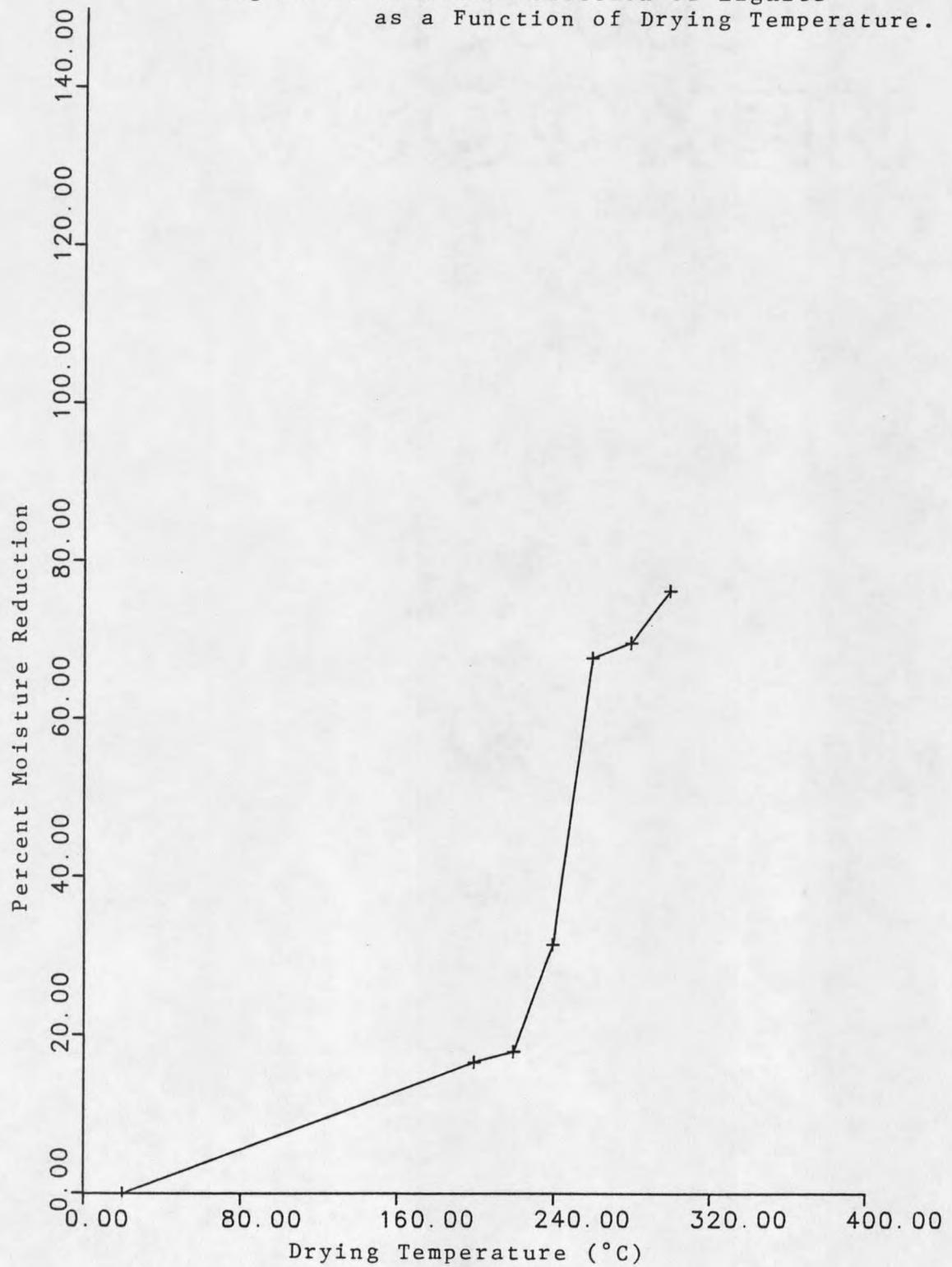
Effect of Drying Temperature on Moisture Content

Table 2 shows the moisture content of lignite samples after they have been hot-water dried at temperatures ranging between 200°C and 300°C. Figure 2 shows the moisture reduction in the coals as a function of drying temperature. In all figures where drying temperature is the ordinate of the graph the appropriate value obtained for the raw coal is listed at 20°C. The moisture content stayed relatively level through the 240°C run. Only small amounts of moisture reduction were obtained. At these lower temperatures the small amount of drying that takes place is just from a reduction in the physically adsorbed surface moisture associated with the coal.

Table 2. Moisture Content Change In Lignite With Drying

<u>Temperature</u>	
<u>Drying Temp., °C</u>	<u>Moisture Content, %</u>
200	30.82
220	30.32
240	25.33
260	11.91
280	11.22
300	8.79

Figure 2. Moisture Reduction of Lignite
as a Function of Drying Temperature.



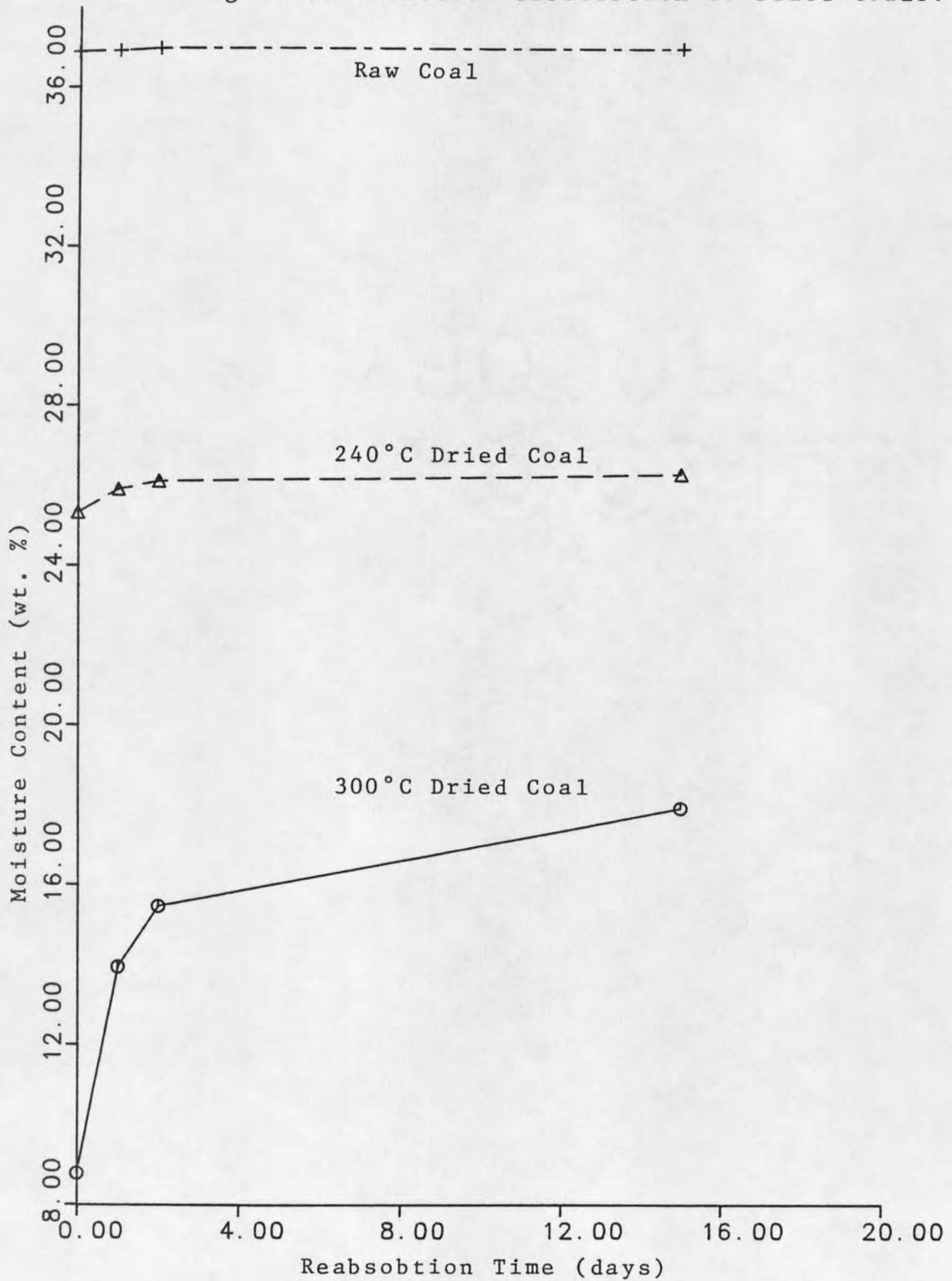
Above 240°C a rapid increase in moisture reduction takes place. The large increase in moisture reduction between 240°C and 260°C occurs at the temperature where an increase in the carbon dioxide formation has been shown to take place (12). This CO₂ formation removes many of the polar carboxylic groups from the coal allowing the chemically adsorbed moisture to escape. This accounts for the reduction in carboxylic groups found at 260°C in other lignites.(11,12)

Above 260°C, while still increasing, the rate of moisture loss slows. Thus, the majority of the moisture loss occurred in a small temperature range. Pressures during runs were nominally the saturated steam pressures corresponding to the temperature of the run.

Moisture Reabsorption

Samples of the raw coal and of the lignite hot water dried at 240°C and 300°C were kept in a 100% humidity chamber at room temperature for 15 days. The moisture content of these samples was checked after 1, 2, and 15 days in the chamber to check for moisture reabsorption. Some moisture reabsorption did occur as illustrated in Figure 3. The coal dried at 300°C exhibited the most moisture reabsorption. It reabsorbed 32.52% of the moisture lost

Figure 3. Moisture Reabsorption of Dried Coals.



during hot-water drying. The resulting lignite is still a vast improvement over the raw coal. The majority of the reabsorption took place in the first two days in the chamber. The raw coal showed no absorption of water during its time in the humidity chamber. It should be pointed out that actual conditions would be nowhere near those found in the humidity chamber. Moisture reabsorption in actual practice would be much less and much slower than was produced in the 100% humidity chamber.

Size Reduction

During the drying process a small amount of lignite particle size reduction was expected to take place. To test for size reduction the raw coal and coals dried at 240°C and 300°C were sized using standard mesh screens. The amount of coal in each screen was weighed and recorded. The raw coal put in the hot-water drying apparatus was all between 20 and 60 mesh. The dry coals were sized using standard mesh screens sizes 20, 32, 40, 60, 80, 100, and 140.

Both the mean particle size and the particle size distribution decreased during hot water drying of the Savage lignite. The raw coal had a mean particle diameter of 0.0259 inches, while the coal dried at 240°C had a mean particle diameter of 0.0236 inches. The lignite hot-water-dried at

300°C was even smaller, with a mean particle diameter of 0.0190 inches. This size reduction is expected as portions of the coals molecular structure react during drying to form CO₂, methane, and other off-gases, causing the coal to contract slightly. Pieces of coal will break off during repeated handling. However, the two hot-water-dried coal samples had equal handling. This indicates that the reduction in the mean particle diameter between the 240°C and 300°C runs is caused by the drying process.

Off-Gas Composition

Results of the off-gas determinations were largely inconclusive, however some interesting sidelights did appear. Because of limitations in the gas chromatograph and sampling system used in these experiments, small concentrations of many gases, which have been shown elsewhere to exist in similar systems, could not be detected.(11)

The gases that were readily detected were nitrogen/oxygen and carbon dioxide. In several runs one sample of gas was taken while the system was hot, and another sample was taken after the system had been allowed to cool overnight. On gas samples taken while the system was hot the majority of the gas was nitrogen with a small

fraction of carbon dioxide and carbon monoxide. However, on samples taken after cooldown the gas fractions had changed substantially. The amount of carbon dioxide was now equal or greater than the amount of nitrogen/oxygen present.

Because the system was thoroughly flushed and checked before each run, it is believed that some air had adsorbed onto the surface of the coal. This air then desorbed during heating. Nitrogen has been found elsewhere to be a large component of cooled off-gases in similar systems (11), but was assumed to be a function of the error in the system. Moore(24) found that, upon heating, all coals produced carbon dioxide and that a large proportion of them produced nitrogen. He also found that when peat was heated the gas given off consisted chiefly of nitrogen with smaller amounts of methane and carbon dioxide.

While the nitrogen desorbs rapidly during heating, the CO_2 formation may take more time. This would account for the reversal in gas fractions in the off-gas samples. The time needed for significant CO_2 formation depends on the coal and the treatment temperature.(12) For example, run 32, at 240°C , had a hot gas sample that was 89% N_2 and O_2 , and 11% CO_2 . After cooldown the gas sample was 63% CO_2 and 37% N_2 and O_2 .

Carbon dioxide is roughly twenty times as soluble in water at the pressure and temperature present when the

system is hot as it is when the system is at ambient temperature and pressure.(25) Houghton (26) showed that temperature affects CO_2 solubility in water much more strongly than for less soluble gases like nitrogen and hydrogen. This could help explain why the CO_2 concentration in the off-gases increases as the system cools. Carbon dioxide that was dissolved in the water at high temperatures is released into the off-gases at lower temperatures.

Ultimate Elemental Analyses

Table 3 is a listing of the ultimate elemental analyses of the raw and hot-water-dried lignites. The standard deviation of the sample, S, (27) is also listed for each entry. Elemental analyses are presented for the moisture-free coals and the coals after they have been left in a vacuum desiccator overnight. The lignites were left in the desiccator to see what differences, if any, resulted from removal of all of the moisture in the coal, as opposed to removing just the surface moisture. Results are listed as weight percents.

The carbon content of the hot water dried lignite is plotted as a function of drying temperature in Figure 4. For the moisture free samples the carbon content increased from 59.74%, when dried at 200°C , to 66.34% at 300°C . This 11%

