5-1-1931

Studies in the Development of Dakota Lignite

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STUDIES IN THE DEVELOPMENT OF DAKOTA LIGNITE

I. Further Studies on Steam Drying.

II. Beneficiation by Treatment with Oil Emulsions at Elevated Pressures.

by

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A thesis submitted to the faculty of the College of Engineering of the University of North Dakota in partial fulfillment of the requirements for the degree of Master of Science in Chemical Engineering.

May 20, 1931 Grand Forks, North Dakota

This Thesis, presented by Albert M. Cooley as a partial fulfillment of the requirements for the degree of Master of Science in the University of North Dakota, is hereby approved by the Committee in charge of his work.

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STUDIES IN THE DEVELOPMENT OF DAKOTA LIGNITE

Part I.

FURTHER STUDIES ON STEAM DRYING.

INTRODUCTION.

The improvement of Dakota lignite as a fuel for domestic as well as industrial use has been the subject of much concern to those intimately connected with the welfare of the State. The potential value of this fuel to the development of the Northwest has long been realized. It is for this reason that the School of Mines has been so actively engaged in studies that will ultimately lead to the solution of this perplexing as well as interesting problem.

The late Dean E. J. Babcock (1) and his assistants worked out a process for the carbonization and briquetting of Dakota Lignite. Due, however, to economic conditions, this process has not been commercialized.

Within the past few years, the School of Mines, under the active direction of Dr. A. W. Gauger, has attacked this problem from another point of view; namely, the improvement of this fuel by the direct removal of a large fraction of its moisture. It was realized that any process to be successful will have to minimize the number of steps required in the processing and, likewise, it must not involve too great a cost.

In reviewing the literature it was found that Fleissner (2), had developed a process whereby Austrian brown coal was dried successfully by the use of saturated steam at elevated pressures. Since a consideration of the underlying principles of steam drying indicated its possibilities it was decided to study this process with Dakota lignite.

This work was first started at the University of North Dakota during the year 1928-29. During the year 1929-30 the work was continued at the University of Minnesota by Dr. Irvin Lavine under a cooperative agreement reached by Dr. A. W. Gauger of the University of North Dakota and Dr. C. A. Mann of the University of Minnesota. The results of this study are to be found in the Journal of Industrial and Engineering Chemistry (3).

The work during 1929-30 was limited to the development of an apparatus suitable for study with Dakota fuel and to a detailed study of the mechanism of this process. As a direct result, only three different Dakota lignites were studied.

In outlining the work for the present year, it was decided to extend this study with lignite from other deposits within this state. The results of this study are reported in Part 1 of this thesis.

EXPERIMENTAL

Apparatus.

The assembly of the apparatus used in the present investigation is shown in Figure 1. A gas fired boiler A was mounted on a

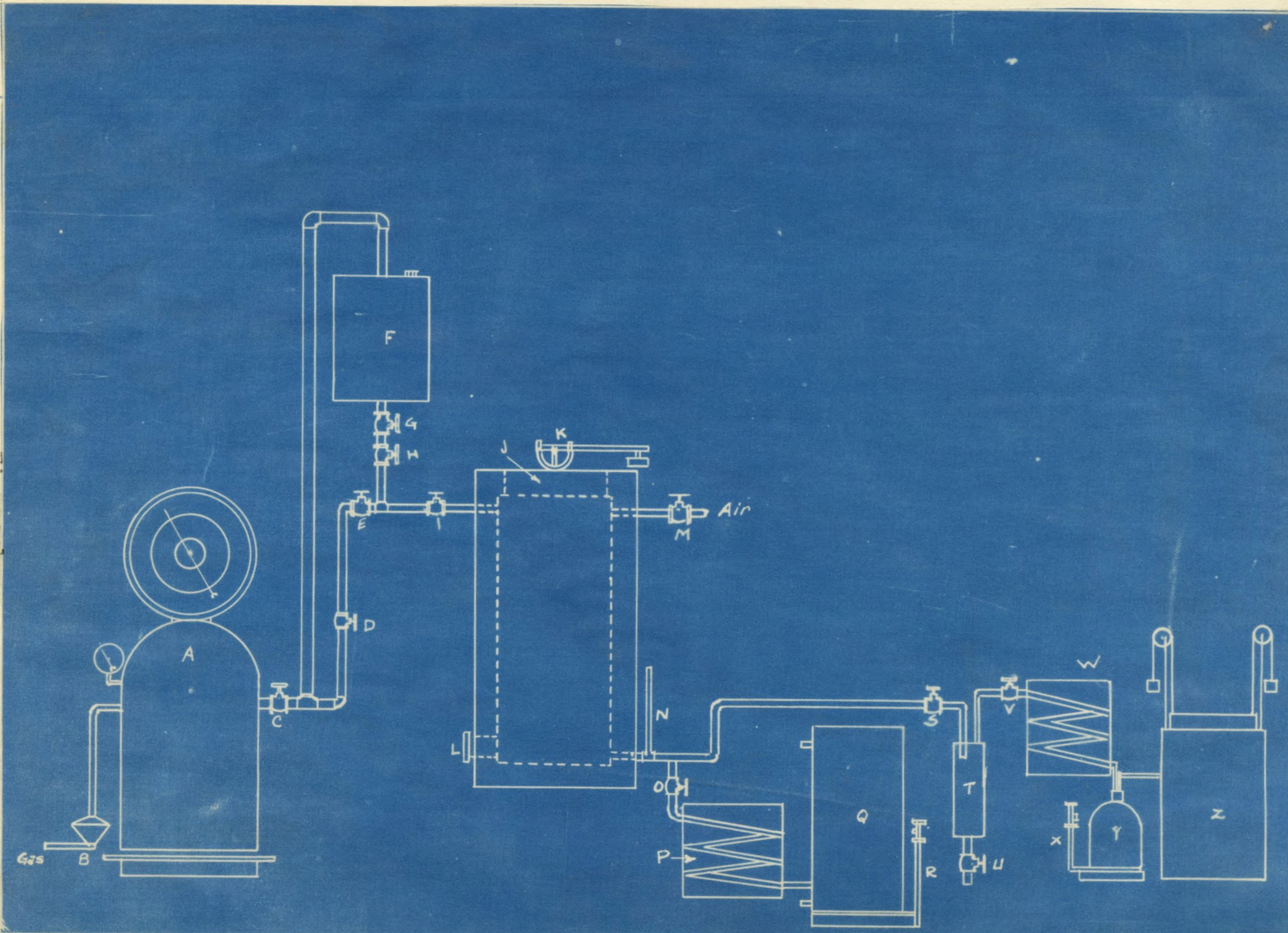


Figure 1.

Toledo platform scale. Connection to the autoclave was made by a flexible steam hose (which was later replaced by ½ inch pipe so arranged that it served as the injector for the oil emulsions used in the work described in Part 3 of this paper). The rate of flow of steam from the boiler to the autoclave was controlled by valve G. The autoclave was equipped with; (1) a removable head J through which the coal was charged, K being a safety valve; (2) a pressure gauge (not shown in the illustration) which served to indicate the working pressures; (3) a coal discharge opening L; (4) air inlet valve M; (5) condensate valve S; (6) thermometer N to read the temperature of condensate leaving the autoclave; (7) trap T (constructed of 4 inch wrought steel pipe having boiler plate welded over the ends) which served to separate the water from the steam that was released from the autoclave; (8) valve V which regulated the rate of release of the steam. Condenser W served to condense steam emitted as condensate from autoclave. The condensated steam drained into bottle Y which was mounted on scale X. The non condensable vapors passed into gas holder Z.

Air from a blower (not shown in the diagram) was forced through valve M into the autoclave, through the coal mass, and out through valve O. The water vapor picked up by the air was partially removed in condenser P and further removed in calcium chloride tower Q, which was mounted on a platform scale.

Procedure.

Since it was realized that the previous history of the coal determined to a large extent its behavior during the drying process, the coal used in this work was shipped directly from the mines in steel drums having welded heads. The drums were opened as the coal was used, care being taken to minimize the drying in the atmosphere.

About 60 pounds of graded lignite (3 to 4 inch lumps) were taken for each charge. A representative sample was also taken from each barrel for use in determining its moisture content and proximate analysis.

The coal was charged into the autoclave through the head, which was then fastened in position by means of eight ⅞ inch bolts. Valves C, H and G were closed and S and V opened. The desired working pressure was then set by regulating the pressure diaphrum B. When the desired steam pressure had been reached the weight of the boiler was read directly on the Toledo scale, valves D, E and I were then opened full and the rate of flow regulated by valve C.

Preheating period. The rate of flow of steam to the autoclave could be determined approximately by the volume of air displaced into gas holder R. The steam consumption was obtained directly every five minutes from readings on the Toledo scale. Valve U was opened every five minutes and the condensate weighed. The weight of steam accompanying the condensate was simultaneously determined by weighing on scale Y. The volume of non-condensable gas was read directly to tenths of a cubic foot on gas holder R every five minutes.

At the first indication of steam, valve V was closed and the condensate removed every five minutes by first closing valve S and then opening valves V and U. Operating in this manner minimized the loss of steam considerably.

Heating period. As soon as the pressure in the autoclave had reached the maximum value (as determined by the set boiler pressure) the heating period commenced. It was found that fluctuations of the pressure during this period tended to check the coal and, therefore, condensate was not removed during the heating period. The procedure consisted in maintaining the pressure for a length of time sufficient to heat the entire lump of coal to the temperature of the steam.

The steam consumption and pressure was recorded every hour.

Release period. At the end of the heating period valve C was closed and the scale reading taken. The steam release was regulated by valve V and the condensate removed through U. The following readings were recorded every 5 minutes until the autoclave pressure was reduced to atmospheric; (1) weight of water removed through U; (2) the condensed steam in V; (3) volume of non-condensable gas; (4) and autoclave pressure.

Aeration period. As soon as there was zero gauge pressure in the autoclave, valve S was closed and valves M and O opened. Air was then forced through the coal mass. The weight of the calcium chloride tower was then recorded every thirty minutes.

Removal of coal. At the end of the aeration period the contents of the autoclave were removed through head J and opening L. A representative sample was taken for moisture and proximate analysis. The coal was first critically examined for physical appearance and was then weighed. A ten pound sample was taken for a hardness test. The remainder was burned under standard conditions to determine its adaptability for domestic use.

Results.

<u>Preheating period</u>. The procedure was changed from time to time to determine the best conditions for each particular lignite. It was found that a long slow preheat, while consuming a relatively larger amount of steam, gave a tougher product. Table 1 shows the effect of time on the steam consumption. It should be noted that none of the different lignites behaved similarly and, therefore comparisons must be made on the same kind of lignite.

Table 1.

Effect of time on Steam Consumption during preheat.

Time	Boiler pressure lbs. per sq. in.	Autoclave pressure atmospheres	Steam Consumed pounds	Condensate Water pounds	Condensate Steam pounds	Non-Condensable gas cu. ft.
Exp. 8						
0	200	0	0	0	0	0
5	190	0	2	0	0	.5
10	140	0	4	1 3/8	0	1.25
15	140	0	6	4	0	1.5
20	140	0	10	2 3/4	0	1.75
25	140	1	13	3 1/8	0	1.75
30	140	6.5	16	3 1/4	0	1.75
35	140	8	18	2	0	1.75
40	160	10	22	0	0	1.75
45	180	11	25	3	0	1.75
50	180	11 1/2	27	3	0	1.75
55	190	12	30	3	0	1.75
60	200	13	32	2 3/4	3/4	1.75
Exp. 9						
0	200	0	0	0	0	0
5	180	0	6	5	0	2.1
10	150	7	15	6	3/4	2.1
15	150	9	18	3 1/2	1	2.5
20	160	10	22	3 1/4	1	2.5
25	170	11	24	2	1/2	2.5
30	190	12	28	2	1 1/8	2.5
35	200	13	29	0	0	2.5

Note: Experiments 8 and 9 were performed with Knife River lignite.

Of particular interest in regard to the pre-heating period data is the fact that different lignites behave quite differently with respect to steam consumption. This is illustrated by the data given in Table 2.

Table 2.

Pre-Heating Period Data for Two Different Dakota Lignites.

Time:	Boiler pressure lbs. per sq. in.	Autoclave pressure atmospheres	Steam consumed pounds	Condensate Water pounds	Condensate Steam pounds	Non-Condensable gas cu. ft.
Exp. 18 - Knife River Lignite.						
0	200	0	0	0	0	0
5	190	0	3	0	0	1 1/4
10	190	0	5	3	0	2
15	180	2	9	4 1/4	0	2
20	170	5	13	5 1/2	0	2
25	170	10	19	4	0	2
30	170	10 1/2	21	2	1	2
35	180	11	23	1	1/4	2
40	180	11 1/2	25	2	1/2	2.1
45	190	12 1/2	27	1 3/4	1	2.1
50	200	13	29	1 1/2	1/2	2.1
Exp. 21 - Velva Lignite.						
0	200	0	0	0	0	0
5	190	0	2	0	0	0
10	180	0	4	0	0	1
15	190	0	5	0	0	1 1/2
20	185	0	7	4	0	1.8
25	180	0	10	2 1/2	0	1.9
30	180	1/2	14	2 1/2	0	1.9
35	180	3	17	0	0	1.9
40	175	6	20	0	0	1.9
45	175	9	24	0 1/4	0	1.9
50	180	10 1/2	25	0	0	1.9
55	190	12	30	0	0	1.9
60	200	13	33	0	0	1.9

A consideration of this table shows that Knife River lignite does not absorb steam during the preheat since the steam consumed and the condensate removed were nearly equal. Velva lignite, on the other hand, shows a decided absorption as manifested by 22 pounds of steam being consumed with only 18 1/4 pounds of condensate removed. This phenomenon is of particular importance since it may explain the decided difference in the success of steam drying with these two coals.

Heating period. The steam consumption during the heating period is dependent to a large extent upon the time and the rate of removal of the condensate. It was found that the steam consumption was considerably reduced when no condensate was removed. Further, the removal of condensate during the heating period tends to check the lumps of lignite. It is reasonable to assume that the time of processing must be fixed by the quality of the product. Thus, if the lump is not heated thoroughly to its interior, the strength of the dried product will be much impaired.

Release period. During the release period the pressure was gradually released to atmospheric. The water and steam were separated by means of a trap, but due to the construction of this apparatus the separation was not accurate and little may be said of the economics of the process. Actual drying of the lignite probably takes place during this period. It was found that the greater the working pressure the greater was the degree of drying.

Table 3 shows representative release data from three different lignites.

Table 3.

Release Period Data

Exp.	Kind of Lignite	Time Minutes	Condensate Water-lbs.	Steam-lbs.	Non-Condensable Gas - cu. ft.
19	Velva	15	2 1/2	8	.4
10	Kincaid	20	1	9	.25
9	Knife River	25	2 1/4	7 3/4	.4

__Aeration period.__ According to the theory of steam drying, the passage of air over the thoroughly heated lump of lignite should cause a further drying. Lavine, Gauger and Mann (2) found, however, that for maximum drying during this period it is necessary to use an air velocity of at least 11 cubic feet per minute. In the present study it was not possible to obtain this velocity because the air compressor available supplied a much smaller quantity. The quantity of water removed during the aeration period in this study was nearly equal in every experiment and amounted to 4-5 pounds for the charge of lignite used.

Table 4.

PHYSICAL DATA FOR LIGNITE DRIED WITH SATURATED STEAM.

Run No.	Pressure Atm.	Kind of Coal	Size Inches	Physical Appearance	Sizing Test on 1½" Screen Per cent	Moisture in Processed Lignite Per Cent	Moisture in Raw Lignite Per Cent
6	13½	Beukol	2-3	Badly checked	47.5		35.9
7	13½	"	2-3	" "	48.0		35.9
8	13½	Knife River	2-4	" "	60.0		35.5
9	13	"	3-4	" "	48.0	10.8	30.0
10	13½	Kincaid	3-4	" "	48.5	15.9	36.6
11	13½	"	3-4	" "	47.0	15.0	36.6
12	13½	Knife River	3-4	" "	-----	12.9	35.5
13	13	Kincaid	2-3	Disintegrated (None above 1½" screen)			36.6
14	11½	Knife River	3-4	Better than 8 and 9	50.0	14.0	35.5
16	8	"	3-4	Better than 14	50.0	16.0	35.8
17	9	Kincaid	3-4	Disintegrated. None above 3"		----	38.6
19	14	Velva	3-4	About 10% good	50.0	12.50	38.3
20	9½	"	3-4	Badly checked	----	----	38.3
21	13	"	4	About 25% very good	57.5	14.7	30.3
22	13	"	3-4	" " "	----	----	

PHYSICAL PROPERTIES OF STEAM DRIED LIGNITE.

As each lump of lignite was removed from the autoclave it was examined critically and tested for strength by breaking. It was found that a poorly dried lump splits up into a series of laminations, which are bound together loosely and easy to break. The percentage by weight of the good lumps in an experiment was recorded and this was used as a basis for comparison. Data as to the physical properties of the coal from various experiments is given in Table 4.

Drop tests. In column 6 of Table 4 the results of drop tests are given. The apparatus used in making this test consisted of a tight wooden enclosure 18 inches square at the base and 8 inches square at the top, and approximately 7 feet in height. The top terminated in a small reservoir with a double drop gate so arranged that the whole charge of lignite could be simultaneously released and dropped. The lignite struck on a 2 inch slab of concrete fitted tightly to the bottom of the box.

A ten pound charge of lignite was graded between 3 and 4 inches and placed in the reservoir, the gates were released and the mass dropped. The floor of the apparatus was swept clean and the charge again placed in the reservoir. This procedure was repeated 5 times after which the charge was screened and the weights recorded. The percentage above 1½ inch screen was taken as a basis for comparison. Table 5 shows the results of drop tests on samples of freshly mined lignites.

Table 8.

DROP TEST DATA FOR FRESHLY MINED LIGNITE

Knife River Lignite.
Weight 10 pounds.

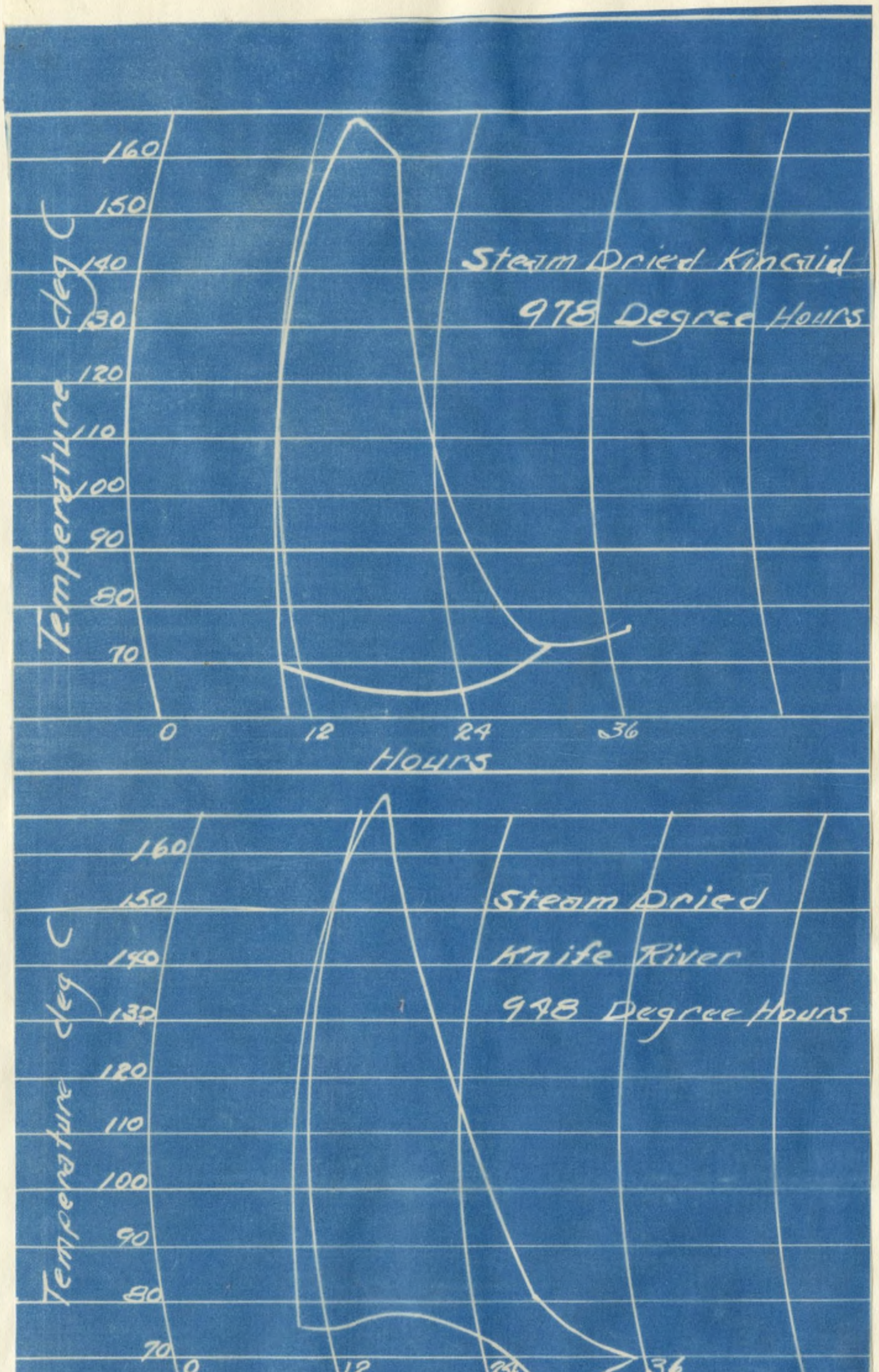
	Weight	Per cent	
Remaining same size	7.500	75.00	
Retained on 1½ inch screen	.750	7.50	82.5%
" " 1 " "	.375	3.75	
" " 4 mesh "	.600	6.00	
" " 6 " "	.500	5.00	
" "14 " "	.250	2.50	
Passing through 14 mesh	.125	1.25	

Beulah Lignite.
Weight 10 pounds.

	Weight	Per cent	
Remaining same size	4.250	42.50	
Retained on 1½ inch screen	2.345	23.45	65.95%
" " 1 " "	1.125	11.25	
" " 4 mesh "	.250	2.50	
" " 6 mesh "	.345	3.45	
" "14 " "	.250	2. 50	
Passing through 14 mesh	.025	0.25	

Velva Lignite.
Weight 10 pounds.

	Weight	Per cent	
Remaining same size	7.75	77.50	
Retained on 1½ inch screen	.25	2.50	80.0%
" " 1 " "	.50	5.00	
" " 4 mesh "	.25	2.50	
" " 6 " "	.25	2.50	
" "14 " "	.50	5.00	
Passing through 14 mesh	.50	5.00	



Temperature deg C
160
150
140
130
120
110
100
90
80
70
Steam Dried Kincaid
978 Degree Hours
0
12
24
36
Hours
Temperature deg C
160
150
140
130
120
110
100
90
80
70
Steam Dried
Knife River
948 Degree Hours
0
12
24
36
Hours

Figure 2-A.

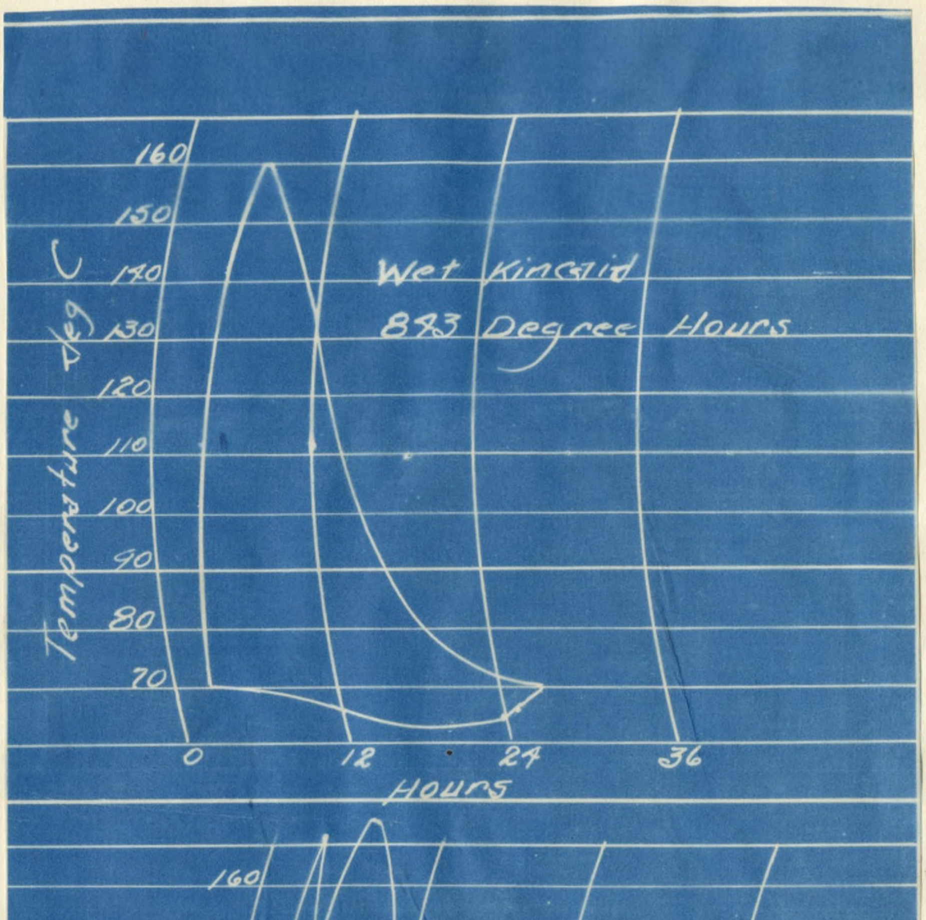

160
150
140
130
120
110
100
90
80
70
Temperature deg C
Wet Kincaid
883 Degree Hours
0
12
24
36
Hours
160
150
140
130
120
110
100
90
80
70
Temperature deg C
Columbus Wet Lignite
869 Degree Hours
0
12
24
Hours

Figure 2-B.

<u>Burning tests.</u> The apparatus used in the burning test consisted of a Montgomery Ward heater, specially designed for lignite, placed in a room 10 feet wide, 12 feet long and 10 feet high. A recording thermometer placed in the furnace room recorded the temperature from the beginning to the end of the test. Another recording thermometer on the outside of the testing room registered the air temperature outside the room.

Two pounds of dry pine kindling were placed on the grates with 10 pounds of coal. The temperature outside the building, the temperature inside and outside the testing room and the approximate wind velocity were all recorded at the time of ignition. Fifteen minutes after the coal was ignited 40 pounds more was added and the draft closed. The testing room was then kept closed until the recording thermometer in the testing room and in the building registered the same temperature. The ash was then removed, weighed and the combustible matter determined.

By plotting the curves of the two recording thermometers on one sheet the inclosed area so formed could be taken as a measure of comparison of the burning properties of different lignites. The results of typical burning tests, using this method, are shown in Figure 2.

The area of each square on the thermometer sheet was equal to 120 degree hours. The total degree hours of burning could then be computed from a simple proportionality between this factor and the inclosed area. In general it was found that steam dried lignite did not show an increase in the degree hours proportional to the moisture decrease, due perhaps to its very rapid burning and consequently, greater radiation of heat from the testing room at the higher temperature.

__Shrinkage.__ Steam drying causes a perceptible shrinkage in the lumps of lignite. It was thought advisable to determine the degree of shrinkage at various pressures and accordingly the following procedure was followed:

During the selection of the sixty or more pounds of freshly mined lignite for use in the experimental work, one lump was selected for a shrinkage determination. This lump was placed in a circular metal container of known volume. Standard sand was then poured over the lump and the contents gently shaken. Sand was again added to completely fill the container. The volume of sand was then measured in a graduated cylinder and the volume difference between that of the container and that of the sand was taken as the volume of the lump. This lump was next placed in a thin cotton bag, in order to identify it and prevent chipping during processing, and then carefully placed in the center of the charge in the autoclave.

Upon completion of the processing, the bag was carefully removed and the volume of the lump was determined according to the method just described.

The difference in volume of the lump before and after processing was then calculated. The ratio of this difference to the original volume was taken as the index of shrinkage. The following results were obtained:

Pressure (Atmospheres)	Shrinkage Index
15	35%
13	30%
8	16%

It is evident that the shrinkage increases with increase in the working pressure.

SUMMARY.

1. The steam drying process has been extended to lignite from Knife River and Beenan. It was found that the lignite from these deposits is not well suited for drying by this process.

2. Burning tests have been conducted with steam dried lignite.

3. Tests have been made to determine the degree of shrinkage due to steam drying. It was found that shrinkage increases with increase in working pressure.

4. The results of Lavine, Gauger and Mann have been verified.

REFERENCES

Part 1.

1. Babcock and Odell, U. S. Bureau of Mines Bulletin No. 221.

2. Fleissner, U. S. Patent No. 1,632,829 (June 21, 1927) and U. S. Patent No. 1,679,078 (July 31, 1928).

3. Lavine, Gauger and Mann, Journ. of Ind. and Eng. Chem., 22, 1347, (1930).

Part II.

BENEFICIATION BY TREATMENT WITH OIL EMULSIONS AT ELEVATED PRESSURES.

INTRODUCTION.

It has been pointed out already that the improvement of the relatively low heat value lignite of North Dakota into a fuel of high heat value with weathering, handling and burning qualities equal to the higher grade coals has been a major research problem at the School of Mines of the University of North Dakota. One phase of this problem has been the drying of lignite by saturated steam.

The results presented in Part 1 of this thesis as well as those of Levine, Conger and Mann (4) show that the steam drying of certain Dakota lignite yields a material much superior to the original fuel, but does not prevent checking entirely. Because of the construction of the apparatus it was impossible to determine at what period of the process this checking occurs. However, certain evidence presented itself to indicate that this takes place mainly during the early moments of the pre-heating period.

In considering means of eliminating this undesirable property it occurred that the presence of an oil phase during the preheat might work satisfactorily since it is a well established fact that colloidal materials such as lignite show a preferential absorption for certain oils. Also, it was thought that the addition of oil to the lignite would materially increase its heating value.

The use of a steam-oil emulsion at an elevated pressure for successfully processing lignite seemed logical from a theoretical consideration. In such an atmosphere the lump could be heated thoroughly to a high temperature without loss of moisture and furthermore the absorbed oil might serve as a cementing material within the cell walls, thus minimizing the checking of the lumps.

The theory of coal washing by froth flotation according to Bogue (1) is as follows:- "The coal particles being more readily wet by oil than water attach themselves to the oily bubble film and these 'armour plated' bubbles rise to the top in a stable froth, leaving the mineral matter in the water." According to this theory the coal has a greater affinity for oil than water. It was thought, therefore, that if an emulsion of oil and water was kept constantly in contact with the lumps of coal during the drying process there would be a selective absorption of the oil, which would displace the water. Shrinkage would then be materially reduced and consequently checking would be likewise minimized. It was decided, therefore, to study the effect of steam-oil emulsions at elevated pressures on the drying of lignite and the results of this study are presented in Part II.

EXPERIMENTAL

Apparatus.

The experimental apparatus was essentially the same as that used in the experimental work described in Part 1 (see Figure 1.). The emulsion to be injected was poured into the receiving tank F through a removable plug at the top. Two valves, G. and H served to control the rate of flow of emulsion from the tank. An injector, constructed as shown in Figure 3, drew the emulsion from the tank F into the autoclave.

Materials.

Emulsions. The following oil-water emulsions were used:

(1) No. 50 Iso-Vis Motor Oil emulsified with steam.

(2) Oklahoma Fuel Oil emulsified with steam.

(3) Boiled and Raw Linseed Oil emulsified with steam.

(4) Medium Heavy Barnsdall Motor Oil emulsified with steam.

(5) Medium Heavy Quaker State Motor Oil emulsified with steam.

(6) No. 3, Headley Emulsified Oil.

Lignite. Sealed samples shipped directly from the Truax-Traer mine at Velva and the Knife River mine at Beulah were used in these experiments. The containers were not opened until the apparatus was ready for charging; thus, minimizing any air drying effects.

Procedure.

About 60 pounds of fresh coal was charged into the autoclave through head J and the head bolted on. Valve H was closed, the predetermined weight of oil-water emulsion poured into tank F, and the plug replaced. Boiler A was brought to the desired steam pressure and the Toledo scale reading taken.

Presaturation period. The presaturation period was then started by opening valves D, E, I and H and then closing valves C, G and H. The rate of steam flow was regulated by valve C. Valve G was opened sufficiently to permit the last of the emulsion to be injected into the autoclave just before the autoclave pressure had reached that of the boiler. The scale reading, boiler pressure, and autoclave pressure were recorded every five minutes until the autoclave pressure had attained the predetermined maximum. Condensate was removed in one experiment to determine its effect on the degree of drying.

Saturation period. The saturation period is defined as the period from the time that the autoclave pressure reaches that of the boiler until the release period is begun. In other words it is the period of maintenance of maximum pressure in the autoclave. It is during this period that the coal-oil system comes to equilibrium. The duration of the saturation period must be determined by the quality and moisture content of the product. The procedure was varied during this period to determine the effect of the removal of condensate on the degree of drying. The scale reading, boiler pressure and autoclave pressure were recorded at the beginning and at the end of the saturation period.

Release period. At the end of the saturation period valve C was closed and the final reading on the Toledo scale taken. The pressure was gradually reduced to atmospheric through valve S. The weight of the water removed (through valve U), the weight of condensed steam in T, and the volume of non-condensable gas were recorded every 5 minutes.

Cooling period. When the pressure in the autoclave had reached atmospheric, valve S was closed and valves H and O opened. Air was blown through the coal mass for a period of two hours. The moisture so removed was weighed in the calcium chloride tower on scale A.

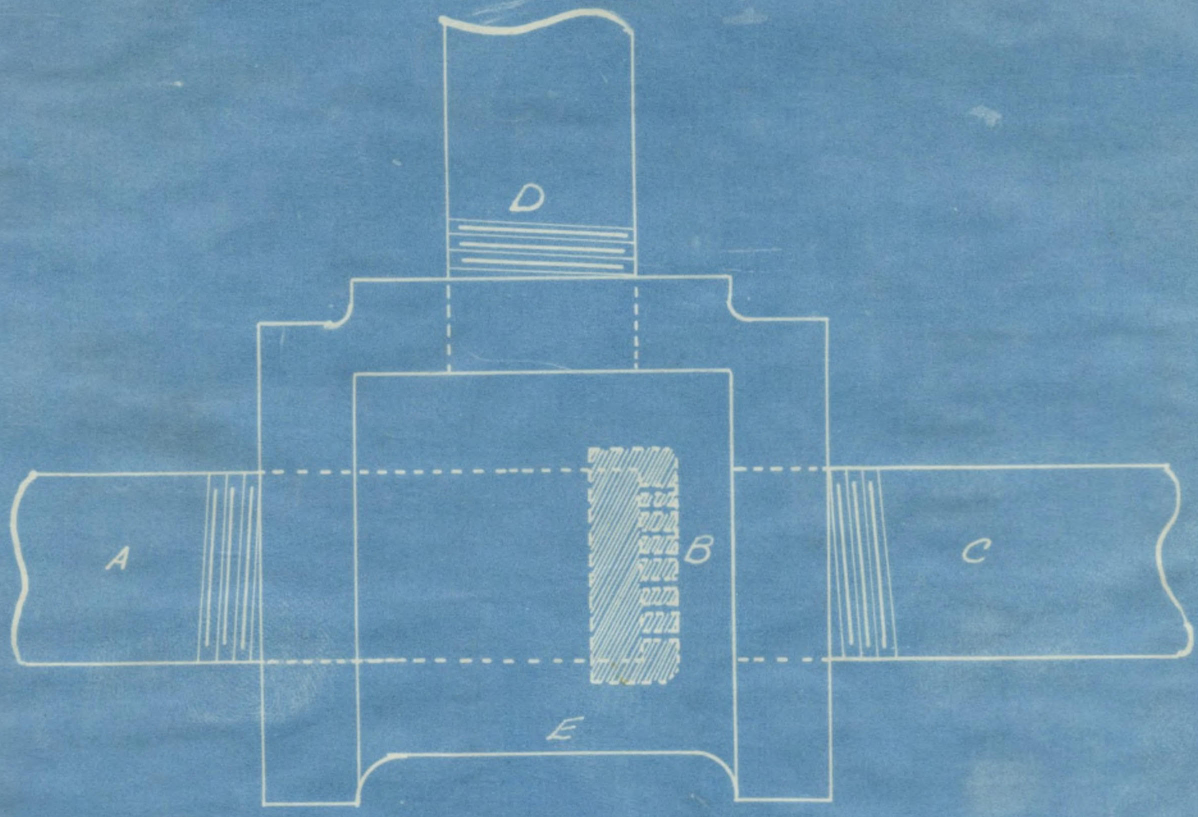


Fig 3

Oil Injector

A = $\frac{1}{2}$" pipe from Steam Line

B = $\frac{1}{2}$" Cap drilled with $\frac{1}{16}$" holes

C = $\frac{1}{2}$" pipe to Autoclave

D = $\frac{1}{2}$" pipe from Oil Reservoir

E = $\frac{3}{4}$" Tee

Results.

Preliminary experiments. Number 60 Iso-Vis motor oil was first tried but mechanical difficulties prevented proper conditions within the autoclave. A nozzle jet was used to inject and atomize the oil. As the pressure in the autoclave approached the maximum value the steam velocity was correspondingly decreased and the nozzle did not function efficiently. As a result the emulsified oil probably was not in contact with the lumps for a period of time sufficient to cause an even absorption throughout the mass of coal. However, about 25% of the product was very good. Analysis of the product showed that the B.T.U. value was considerably increased above the value of the unprocessed coal of the same moisture content. This is a further indication that some oil was taken up by the lignite during the processing.

An Oklahoma fuel oil was next used but with no success. The lignite showed very little absorbing capacity for this material.

Boiled and raw linseed oil were also unsuccessful, due no doubt, to the fact that this oil does not form a stable emulsion by mere mixing with water.

No. 2, Headley emulsified oil gave the best results. It may be pointed out that this oil oxidizes to a viscous mass when coming in contact with air. The excellent results obtained with the Headley emulsion may be in part due to the fact that the absorbed oil decreases the transfer of heat to the interior of the lump and thereby minimizes checking. The emulsion that penetrates into the lump may also serve as a binder, thus preventing warping and splitting.

Presaturation period. The early experiments were performed by first removing the condensate until steam appeared and then injecting the steam oil emulsion after valve 8 had been closed. However, it was found that the coal was checked very badly, due no doubt, to the early heating of the lump before the oil-steam emulsion was injected. Accordingly, the procedure was changed so that the emulsion was injected from the beginning of the presaturation period and no condensate was removed. This procedure gave a product of very good physical strength but of poor appearance. In the case of the Headley emulsion the oil on surface of the lumps had oxidized to a tar which partially prevented drying. The third variation in the presaturation procedure was to inject the emulsion from the beginning of the period as before but in this case condensate was removed continuously until the maximum pressure had been reached. The greatest degree of drying was obtained by this last procedure, but only about 20% of the product remained unchecked. The passage of steam through the autoclave during the removal of the condensate probably washed the emulsion from the surface of the lumps and carried it out with the condensate.

Saturation period. In the early experiments no condensate was removed during the saturation period. The product obtained by following this procedure was very tough but of high moisture content (17 to 21%). The temperature of the condensate was always around 110 degrees centigrade indicating that the presence of the condensed emulsion in the bottom of the autoclave may have prevented the attainment of equilibrium thus decreasing the amount of drying.

The removal of condensate every half hour during the saturation period improved the drying to about 10% but the product was partly checked, perhaps due to the variations in pressure caused by the removal of the condensate.

Release period. The rate of release was changed from time to time. It was found that a short period for release yielded a product of low moisture content.

Cooling period. The passage of air through the coal mass was found to accomplish very little further drying. About 4 pounds of moisture was removed during 2 hours of aeration.

Concentration of emulsion. The effect of processing with various concentrations of emulsion was studied in experiments 36 to 41 where different dilutions of the Headley emulsion were used. The original Headley emulsion consisted of approximately 30% water and was diluted for each of these experiments by mixing with a definite quantity of hot water. The 30% emulsion showed the greatest affinity for the coal but formed a dense tar over the surface of the product which prevented drying to a low degree. However, this coating seemed to water proof the individual lumps which increased their resistance to weathering. Lumps of this lignite which were placed out of doors during conditions of rain and hot sunshine showed little degradation over a period of two weeks. When dilute emulsions were used it was found that the degree of drying was much smaller and the steam consumption was increased. Likewise, the product was of poor physical strength. The results for these experiments are tabulated in Table 6.

A consideration of Table No. 6 indicates that the best results were obtained in experiment No. 41. In this experiment 10% by weight of Headley emulsion (calculated on the basis of the raw coal) was diluted with an equal weight of water and injected into the autoclave. The resulting product had the greatest physical strength and lowest moisture content (18%) of any of the processed lignites. The steam consumption was slightly greater than in the experiments where the original emulsion was used. This, no doubt, was due to the condensation caused by the cold emulsion when it came in contact with the steam. However, the actual penetration into the coal lump was greater than in the case of the original emulsion. Oil penetration was determined by breaking a lump normal to its bedding plane. The physical appearance of the broken lump indicated the extent of the oil penetration.

PHYSICAL PROPERTIES OF OIL EMULSION PROCESSED LIGNITES.

The lignite, upon its removal from the autoclave, was tested for physical strength by breaking. A representative sample was obtained and placed in a two quart Mason jar for moisture and B.T.U. analysis. Ten pounds of 3 to 4 inch lumps were used for drop tests to determine the handling qualities.

Table 6 tabulates the results of the different experiments.

A consideration of Table 6 shows that excellent results can be obtained with Headley emulsion. Thus in experiment 33, thirteen and one-quarter pounds of undiluted Headley emulsion were used for a charge of 63 pounds of raw coal. No condensate was removed until the release period. Approximately 80% of the product remained unchecked and was dried to 20.7% moisture. The surface of the lumps was covered with the oxidized material already referred to.

Table 6.

PHYSICAL DATA FOR LIGNITE PROCESSED WITH OIL STEAM EMULSIONS.

Exp. No.	Pressure Ats.	Kind of Coal	Size in-ches	Weight of Charge lbs.	Physical Appearance	On 1½ inch screen %
24	15	Velva	3-4	40	About 25% Good	60
25	15	"	3-4	65	Badly checked	--
26	15	"	3-4	55	" "	--
27	15	"	3-4	66	" "	--
28	15	"	3-4	51	" "	--
29	15	"	2-3	60	About 25% Good	62
30	15	"	2-5	65	" " "	--
31	15	"	3-5	50	" " %	60
32	15	"	3-4	62	Badly checked	--
33	15	"	3-6	62	About 50% Good	60
34	15	"	3-8	65	" 90% Good	65
35	15	"	2-5	60	Badly checked	--
36	15	"	3-5	60	" "	--
37	15	"	2-6	65	Better than 33	--
38	15	"	2-6	65	About 25% Good	--
39	15	"	3-6	66	" 50% "	--
40	15½	"	2-5	65	" 60% "	62
41	15½	"	2-5	60	" 75% "	--
42	15½	"	2-5	61	" 25% "	--
43	15½	"	2-5	61	" 25% "	--
44	15½	Knife River	3-4	61	Badly checked	--

Table 6 (continued)

Exp. No.	Moisture in raw lignite	Moisture in Product	B.T.U. of product	Kind of Emulsion Used	Weight of Original Emulsion lbs.
24	36.6	----	----	#50 Iso-Vis	7
25	36.6	----	----	Oklahoma Fuel Oil	15
26	36.6	17.8	8808	Med. Texaco	7
27	36.6	13.3	----	Boiled Linseed	14
28	36.6	----	----	Med. H. Barnsdall	8
29	36.6	23.3	8839	#50 Iso-Vis	8
30	36.6	21.0	8700	" " "	12
31	36.6	20.6	----	" " "	12
32	36.6	22.1	----	MH Quaker State	12
33	36.6	20.7	----	#2 Headley Emulsion	13½
34	36.6	24.9	8340	" " "	12(diluted to 8gal)
35	36.6	----	----	" " "	8
36	36.6	----	----	" " "	.6(diluted to 2gal)
37	36.6	----	----	" " "	4 " " 2 "
38	36.6	----	----	" " "	9½ " " 3 "
39	36.6	----	----	" " "	14 " " 3 "
40	36.6	----	----	" " "	9½ undiluted
41	36.6	----	----	" " "	9½ (diluted 50%)
42	36.6	20.7	8806	" " "	9½ undiluted
43	36.6	----	----	" " "	9½ "
44	37.2	----	----	" " "	9½ "

The emulsion removed as condensate from experiment 33 was used in experiment 34. The product so obtained was very good (Approximately 90% unchecked) but the moisture content was decreased only to 34.9%, which indicated that the large mass of liquid in the bottom of the autoclave prevented proper heating of the individual lumps. The surface of the lumps was covered also with the oxidized material as in experiment 33. The effect of dilute emulsions were next studied. Thus, a 1% Headley emulsion (calculated on the weight of the charge) was used in experiment 36. The product was badly checked. A 5% emulsion was used in experiment 37. A product slightly better than that of experiment 36 was obtained in this case. A 10% emulsion diluted with hot water to 5 gallons was used in experiment 38. The product was approximately 85% unchecked but the moisture content was high ( ). A 10% emulsion diluted 50% by weight with hot was used in experiment 41. The product was approximately 75% unchecked and of low moisture content. Condensate was not removed during the presaturation but was removed every 30 minutes during the saturation period. The surface of the lumps was not coated with the oxidized materials as in previous experiments.

Experiment 44 was performed with Knife River coal under conditions similar to those of experiment 41. The product was not only badly checked but also the lumps were softened to such an extent that they could be rubbed to powder between the palms of the hands.

In the experiments where Headley emulsion was used, it was found that lumps under 3 inch in size were successfully processed with minimum checking, whereas in the steam drying process lumps of this size always disintegrated rather severly during the processing.

Weathering properties. Accelerated slacking tests were made on the processed lignite by air drying 500 grams of 1½ inch lumps for 24 hours, immersing in water for one hour, drying 23 hours at 35° and then screening through a ¼ screen. This process was then repeated 8 times. (The 1½ inch lumps were obtained by breaking larger lumps to this size (3). The ratio of the weight of the undersize to the weight of the original sample was taken as the slacking index. The results indicate that the emulsion processed coal is very much superior to steam dried coal and raw lignite. This test also indicated that the emulsion penetrates very deeply into the lump since the entire surface of the small lumps appeared to be waterproofed.

Burning tests. Burning tests were made as described in Part 1. The results show an increase of from 150 to 300 degree hours for 50 pounds of lignite obtained with same weight of steam dried lignite of the same moisture content. The burning tests further indicate that the Headley emulsion processed lignite burned much slower than steam dried lignite. This is a decided advantage, since dry lignite burns too rapid for ordinary use. The amount of smoke liberated in the burning of the emulsion dried lignite was not greater than in the case of steam dried lignite.

Drop tests. Drop tests were made to determine the handling qualities of the coal and the results show that the emulsion dried lignite is very much better than the steam dried lignite. Table 6 shows that over 60% of this material is retained on a 1½ inch screen. In this respect, Headley emulsion processed lignite compares favorably with bituminous coal.

Critical Oxidation temperature. The critical oxidation temperature of lignite processed with Headley emulsions was determined in an apparatus similar to that used by Brady (2). Figure 4 shows the average curve of several determinations. The critical oxidation temperature was found to be around 180° C which is very little lower than unprocessed lignite of the same moisture content.

SUMMARY.

1. Processing Velva lignite with steam oil emulsions has the following advantages:

   a. The B.T.U. value is increased above that of unprocessed coal of the same moisture content.
   b. The yield of unchecked lumps is improved.
   c. The resistance to weathering is increased.
   d. The handling qualities are improved.

2. Studies made with this material show that the critical oxidation temperature is not materially lowered.

3. It has been found that Knife River lignite is not suited for this process under the conditions used.

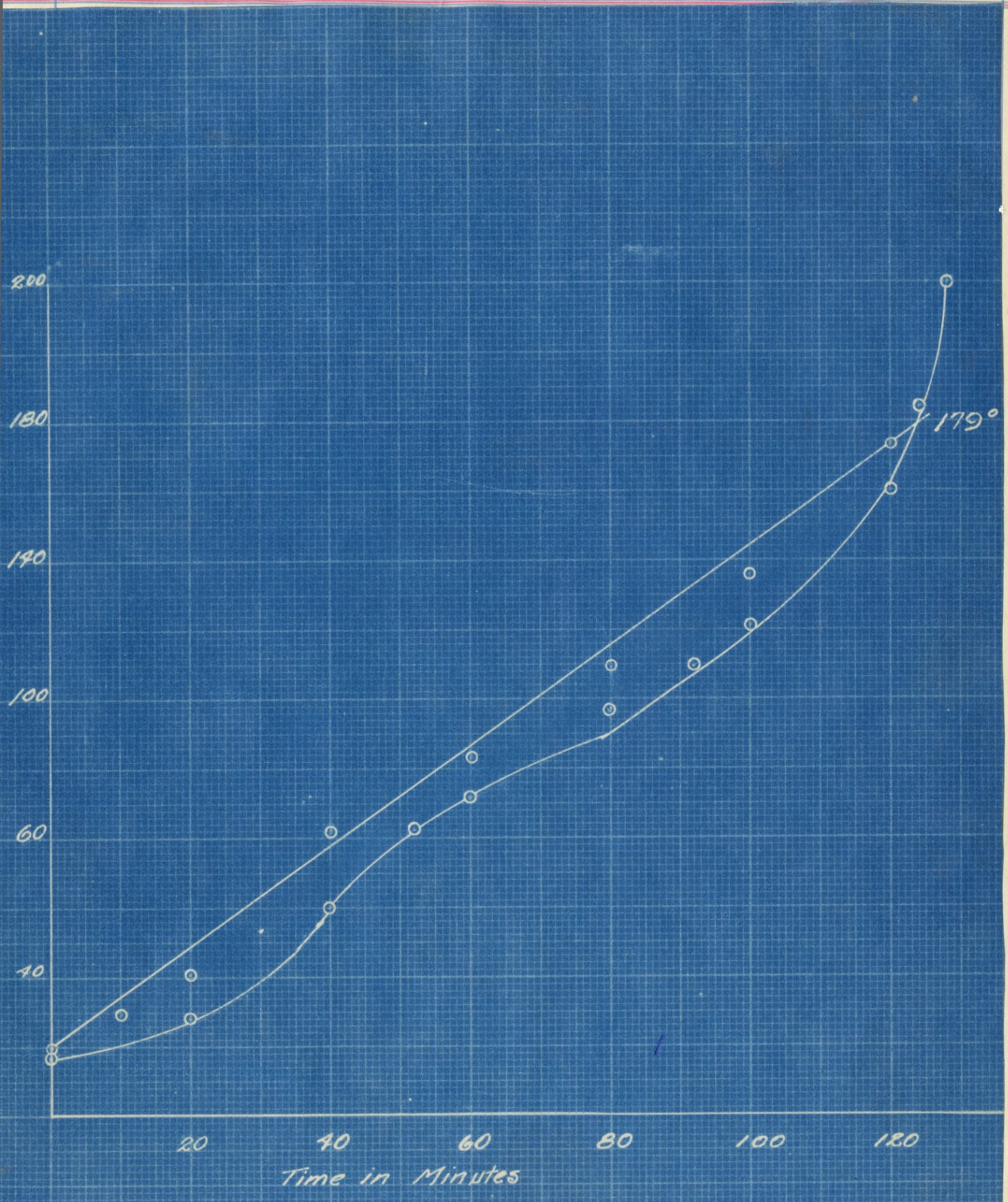


Figure 4.

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ACKNOWLEDGMENT.

The writer wishes to express his gratitude to Dr. Irvin Lavine and Dr. A. W. Gauger for their assistance and direction in this work.