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The physical and petrographic characteristics of formcoke produced experimentally from lignite and subbituminous coal

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THE PHYSICAL AND PETROGRAPHIC CHARACTERISTICS OF
FORMCOKE PRODUCED EXPERIMENTALLY FROM LIGNITE
AND SUBBITUMINOUS COAL

by

Bruce Lowell Ramsey

Bachelor of Science, University of North Dakota, 1972

A Thesis

Submitted to the Graduate Faculty

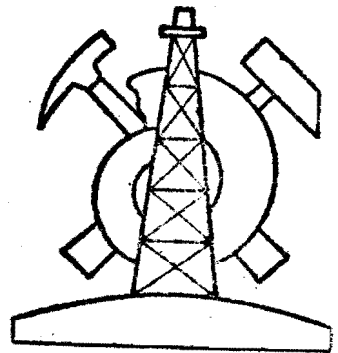
of the

University of North Dakota

in partial fulfillment of the requirements

for the degree of

Master of Science



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PRODUCED EXPERIMENTALLY FROM LIGNITE AND SUBBITUMINOUS COAL

Department Geology

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TABLE OF CONTENTS

	Page
ACKNOWLEDGMENTS	iv
LIST OF TABLES	vi
LIST OF FIGURES	vii
ABSTRACT	ix
INTRODUCTION	1
Statement of the Problem	
Previous Work	
Laboratory Methods	
Materials	
RESULTS	9
Effect of Final Carbonization Heating Rates on Formcoke	
Effect of Binder Percentage and Briquetting Pressure on Formcoke	
Effect of Initial Charring Temperature and Char Grain Size on Formcoke	
Proximate Analyses and Sulphur Content of the Chars and Some Formcoke	
Petrographic Study of the Chars and Some of the Formcoke	
CONCLUSIONS AND DISCUSSION	41
APPENDIX A. Compressive Strengths of Formcoke	45
APPENDIX B. Cumulative Frequency of Char Grain Size	49
APPENDIX C. Percentage Reflectance of Huminite Grains of The Chars and Some Formcoke	58
REFERENCES	65

LIST OF TABLES

Table	Page
1. Petrographic and Proximate Analyses of the Coals	8
2. Physical Appearance of Formcoke Made With 10 and 20 Per Cent Binder, 3000 and 5000 Psi, and Final Carbonization Heating Rates of 18°C/Min, 12°C/Min and 6°C/Min to 900°C	10
3. Effect of Increase in Binder From 15 to 20 Per Cent on Formcoke Compressive Strength	23
4. Proximate Analyses of Chars	27
5. Proximate Analyses of Some Formcoke	27
6. Compressive Strengths of Formcoke Produced From 18 Mesh 900°C-Lignite Char and 10, 15 or 20 Per Cent Binder Using a Range of Briquetting Pressures	46
7. Compressive Strength of Formcoke Produced From 85 Per Cent of 600°C-Lignite, 900°C-Lignite, 600°C-Subbituminous or 900°C-Subbituminous Char and 15 Per Cent Binder	47
8. Compressive Strength of Formcoke Briquets Made From a Blend of 20 Per Cent Binder and 80 Per Cent-- 18, 35, or 60 Mesh--900°C-Lignite Char	48
9. Percentage Reflectance of 50 Randomly Selected Char Grains From Each of the Chars	59
10. Percentage Reflectance of 50 Randomly Selected Char Grains of Formcoke Representing Each of the Chars	62

LIST OF FIGURES

Figure	Page
1. Effect of Binder Percentage on Average Formcoke Compressive Strength of Briquets Made With 18 Mesh 900°C-Lignite Char	13
2. Effect of Briquetting Pressure on Average Formcoke Briquet Compressive Strength of Briquets Made With 18 Mesh 900°C-Lignite Char	16
3. Effect of Char Grain Size and Initial Charring Temperature on Average Briquet Compressive Strength	19
4. Effect of Binder Percentage on Average Briquet Compressive Strength of Formcoke Made With 18, 35, and 60 Mesh 900°C-Lignite Char	22
5. Formcoke Briquet Made With 20 Per Cent Binder and 60 Mesh Char (left) Exhibiting Devolatilization Cracks. Briquet at Right was Made with 15 Per Cent Binder and 60 Mesh Char Exhibits No Cracking . . .	25
6. A 900°C-Lignite Char Grain (A) Showing a Greater Degree of Alteration Resulting in Greater Porosity Than the 600°C-Lignite Char Grain (B)	33
7. Inertinite Grain Found in a 600°C-Lignite Char Showing Preservation of Cell Wall Structure	36
8. Fusion of Grain to Grain Established by Carbon Residue From Binder (Lighter, Anisotropic)	38
9. Formcoke Made With 18 Mesh 900°C-Lignite Char Showing Poorly Developed Grain to Grain Contact (A). Formcoke Made With 60 Mesh 600°C-Lignite Char Showing Well Developed Grain to Grain Contact (B) . . .	40
10. Cumulative Frequency of Grain Size for 18, 35, and 60 Mesh 600°C-Lignite Char	51
11. Cumulative Frequency of Grain Size for 18, 35, and 60 Mesh 900°C-Lignite Char	53

12.	Cumulative Frequency of Grain Size for 18, 35, and 60 Mesh 600°C-Subbituminous Char	55
13.	Cumulative Frequency of Grain Size for 18, 35, and 60 Mesh 900°C-Subbituminous Char	57

ABSTRACT

Formcoke was produced experimentally from a North Dakota lignite and a Wyoming subbituminous coal. The effect of initial charring temperature (600°C or 900°C), of char grain size (less than 18 mesh, less than 35 mesh, or less than 60 mesh), of briquetting pressure (1000, 2000, 3000, 4000, 5000, 6000, 7000, 8000, 9000 or 10,000 psi) of binder percentage (5, 10, 15, 20 or 25 per cent), and of final carbonization heating rate (18°C/min, 12°C/min or 6°C/min) on formcoke briquet compressive strength was studied.

A final carbonization heating rate of 6°C/min produced uniform shape briquets. Rates of 12°C/min and 18°C/min resulted in cracked and swollen formcoke briquets produced otherwise under the same condition as the briquets carbonized at 6°C/min.

An increase in the briquetting pressure resulted in an increase in formcoke compressive strength.

Formcoke produced from blends of less than 18 mesh-900°C-lignite char and 10, 15, or 20 per cent binder exhibited uniform briquet shape. Blends with 5 per cent binder did not hold together once removed from the mold. Blends with 25 per cent binder produced swollen and cracked formcoke after final carbonization.

The 600°C chars produced stronger formcoke than the 900°C chars. Formcoke compressive strength increased with a decrease in char grain size from less than 18 mesh to less than 35 mesh. Formcoke produced

from less than 35 mesh char exhibited about the same strength as formcoke produced from less than 60 mesh char.

Petrographically, the 900°C char grains appear more porous than the 600°C char grains. Grain to grain contact of the formcoke was better developed in the formcoke made with 600°C chars than formcoke made with 900°C chars. Grain to grain contact was better developed in formcoke made from 60 mesh char than formcoke made from 35 to 18 mesh char.

Formcoke produced from North Dakota 600°C-lignite char exhibited compressive strengths equal to or greater than compressive strengths of formcoke produced from a Wyoming subbituminous coal. The FMC Corporation has been producing formcoke from Wyoming subbituminous coal, and because formcoke produced experimentally from low temperatures (600°C) lignite char was of equal quality to formcoke produced experimentally from Wyoming subbituminous char, large scale formcoke production from North Dakota lignite may be possible.

INTRODUCTION

Statement of the Problem

The use of low-rank coal in the metallurgical industry is becoming increasingly important in the industrialized nations of the world as the sources of coking coals become depleted. This has been particularly true in the Eastern European countries, Russia, and Japan. In these nations the abundance of low-rank, non-caking coals has promoted the development of methods utilizing their abundant, less expensive coals for blast furnace use. In the United States, because of the abundance of good coking coals, little research has been done concerning the use of non-caking coal, especially lignite, in the metallurgical industry.

The purpose of this study was to investigate the various physical and microscopic characteristics of laboratory produced formcoke made from a North Dakota lignite and a Wyoming subbituminous coal. Because the FMC Corporation has been using a Wyoming subbituminous for formcoke production since 1960, and by comparing formcoke produced experimentally from a North Dakota lignite to that produced experimentally from a Wyoming subbituminous coal, I hope to establish the feasibility of production of formcoke from North Dakota lignite. Specifically the effects of initial charring temperature, char grain size, binder percentage, briquetting pressure and final carbonization heating rate of formcoke compressive strength were studied. Proximate analyses and petrographic study of the coal, chars and some of the formcoke, were performed.

Previous Work

In order to use a non-caking coal in blast furnaces, the coal must be converted to a synthetic coke or formcoke. Present day manufacture of formcoke involves three types of processes: (1) blends of caking and non-caking coals, with or without a binder (Yoshida, 1971), (2) blends of semicoke and caking coals, with or without binder (Foch, 1971), (3) a single coal, which can be completely non-caking, and a binder (Joseph, 1973).

First the coal is partially devolatilized or charred. The char or devolatilized coal is blended with a binder, or a binder and a caking coal, or with a caking coal. A pelletizing or briquetting process follows. The pellets or briquets are further devolatilized, or carbonized, yielding a high carbon, low volatile product of suitable strength for blast furnace use.

Presently there are numerous commercial processes used in the production of formcoke. Included among these and encompassing the most common procedures are the hot briquetting of coal blends, the Bergbau-Forschung process, the Nord-Fuvo process and the FMC process.

In Japan formcoke is produced using blends of caking and non-caking coals. The most recent process involves: (1) fluidized pre-heating of the coal, (2) hot briquetting by a double roll press, (3) high temperature carbonization of the briquets. A blend of 0 to 20 per cent strongly caking coal to 80 to 100 per cent non-caking coal has been used to produce good metallurgical formcoke (Yoshida, 1971).

The Bergbau-Forschung process produces formcoke from a single, non-caking coal. The process involves hot briquetting of a brown coal

char and a brown coal soft pitch in a roll press. The briquets are oxidized at 220°C in a 10 to 20 per cent oxygen atmosphere followed by carbonization at high temperatures (Foch, 1971).

The Nord-Fuvo process involves conventional briquetting of a blend of low volatile, non-caking coal with 10 to 15 per cent caking coal. Replacement of the low volatile coal by high volatile, caking coal is possible. The high volatile, caking coal is devolatilized by producing semicoke from it. The semicoke is then blended with the usual 10 to 15 per cent caking coal. Carbonization of the coal follows in a shaft oven (Foch, 1971).

At Kremmer, Wyoming, the FMC Corporation has been producing formcoke since 1960. The non-caking coal is carbonized to produce char and volatile matter. The volatile matter is treated to form a tar which is then used as a binder. Briquetting of the char-binder blend follows in a roll press. The briquets are oxidized on a chain grate at 250°C, then carbonized in a shaft furnace at 850°C to 900°C (Joseph, 1973).

Laboratory Methods

This study involves laboratory produced lignite and subbituminous formcoke. Since the FMC Corporation has been producing formcoke from Wyoming subbituminous since 1960, I tried to duplicate the FMC process as closely as possible with the available materials and equipment.

The formcoke was produced in four steps: (1) initial charring or devolatilization of the coal, (2) blending of char and binder, (3)

briquetting of a char-binder blend, (4) final carbonization of briquets in an oxygen deficient atmosphere.

Charring

The lignite and subbituminous coals were crushed and passed through a 5 mesh (U.S. standard sieve number) screen. The coals were charred in cylindrical retorts which were placed in a preheated 500 lb coke oven which is located at the USEM Coal Research Lab, Grand Forks. The oven was preheated to 600°C (to produce what will hereafter be called 600°C-lignite or subbituminous char) or at 900°C (to produce what will hereafter be called 900°C-lignite or subbituminous char).

Blending of Char and Binder

The 900°C- and 600°C-lignite and subbituminous chars were split into three fractions. Each was further ground, one fraction to less than 18 mesh, one to less than 35 mesh, and one to less than 60 mesh. Blending with a binder followed.

There are numerous binder types used in the production of form-coke. For experimental purposes a petroleum or coal base tar or pitch of low melting point was desired. A roofing tar with a melting point of 75-90°C was selected.

The binder and char were weighed according to desired percentage. A range of 5, 10, 15, 20 and 25 per cent binder was used. The binder and char were placed in a container and heated in an oven until the binder was fluid enough for blending to occur.

Briquetting

The blend was placed in one inch by two and one-fourth inch cylindrical molds. The molds were preheated at the same time and temperature as the char and binder were, thus briquetting occurred in heated molds while the binder was still in a fluid state. A hand operated, Carver Laboratory hydraulic press was used to briquet the blends. Briquetting pressure ranged from 1,000-10,000 psi.

The briquets were removed from the molds after they had cooled. This method produced cylindrical briquets one inch in diameter by about one and three-fourths inches in height. Usually five briquets were produced under identical conditions. Four were tested for compressive strength and one was used for microscopic study.

Final Carbonization

Final carbonization of the briquets took place in a muffle furnace which was also located at the USBM Coal Research Laboratory. The briquets were subjected to a selected heating rate (18°C/min, 12°C/min or 6°C/min) up to 870° to 900°C. The briquets were placed in a tray which allowed carbonization, but not burning to occur. This was accomplished by placing a loose lid on the tray which restricted air circulation but allowed evolving gas to escape, creating an oxygen deficient atmosphere inside the tray.

Formcoke produced from the 600°C chars exhibited shrinkage up to 1/16 inch in diameter and 1/8 inch in height. Those produced from the 900°C chars showed little shrinkage in diameter, and only 1/6 inch shrinkage in height.

Many manufacturing processes use an oxygen treatment at low temperatures, followed by high temperature carbonization, to keep the briquets from sticking to each other during carbonization. This was not necessary for a small scale laboratory process.

Compressive Strength Testing

The most convenient method for evaluating the formcoke strength was to test the compressive strength of the cylindrical formcoke briquets. The conventional test for coke require more sample than it is feasible to produce with a small scale process. The shatter test requires 50 pounds of sample and the tumbler test 25 pounds (Fieldner and Selvig, 1951). The FMC process also records crushing strength of their pillow shaped formcoke briquet.

The formcoke was tested for crushing strength using a Soil Test hydraulic press. The press is located in the Civil Engineering Laboratory, UND. The crushing strength is defined as the pressure gauge reading at the time breakage first occurs.

Proximate Analyses

Eventually the formcoke must be evaluated in a blast furnace trial run. A proximate analysis, the moisture, ash, fixed carbon, and volatile matter content, of the formcoke is somewhat indicative of how it will perform in a blast furnace. Sulphur content was included because of its importance concerning air pollution and its detrimental effect on the metal being reduced in the blast furnace process.

A proximate analysis of the coal, coal char, some formcoke and binder were performed by the Engineering Experimental Station, UND.

Three runs on each sample were performed. The results were within one per cent of each other before accepted and then averaged.

Petrographic Study

The petrographic study of the coal, char, and formcoke was performed by myself using the UND Geology Department's Leitz ortholux-pol oil immersion reflecting microscope. The samples were prepared by embedding them in epoxy resin. Polishing of the sample followed using 240, 400 and 600 grit paper, then 5 and .05 micron grit on a cloth covered lap wheel. Petrographic composition of each coal was determined using a mechanical stage and counting one thousand points. Reflectance listed is mean maximum reflectance of fifty randomly selected huminite grains.

Materials

The coals used were a North Dakota lignite collected as grab samples from the Indian Head Mine, Zap, North Dakota, and a Wyoming subbituminous collected as-mined from the Armstrong Seam by the Big Horn Coal Company, Sheridan, Wyoming. See Table 1 for petrographic and proximate analyses of the coals.

TABLE 1
 PETROGRAPHIC AND PROXIMATE ANALYSES OF THE COALS

	Petrographic Analysis	
	Huminite	88.3%
	Liptinite	9.4%
North	Inertinite	2.3%
Dakota	Average Reflectance of Huminite (50 points) .24%	
Lignite		
	Proximate Analysis (air dried)	
	Moisture	7.0%
	Ash	8.6%
	Volatile Matter	40.2%
	Fixed Carbon	44.2%
	Sulphur	.9%
	Heat of combustion (Btu) 10,350/lb	
	Petrographic Analysis	
	Huminite	81.9%
	Liptinite	14.4%
	Inertinite	3.7%
Wyoming	Average Reflectance of Huminite (50 points) .42%	
Subbituminous		
	Proximate Analysis (air dried)	
	Moisture	6.3%
	Ash	7.1%
	Volatile Matter	38.4%
	Fixed Carbon	48.2%
	Sulphur	1.1%
	Heat of combustion (Btu) 11,270/lb	

RESULTS

Initial charring temperature, char grain size, binder percentage, briquetting pressure and the final carbonization heating rate affect the compressive strength of the formcoke. These parameters are interrelated and dependent upon each other (and coal type) concerning their effect on formcoke compressive strength. Experiments were carried out to establish a suitable final carbonization heating rate, binder percentage, and briquetting pressure to be used for our laboratory production of formcoke.

Effect of Final Carbonization Heating Rate on Formcoke

The 900°C-lignite char was crushed to less than 18 mesh. Two char-binder blends were made, one using 10 per cent binder and one using 20 per cent binder. The blends were split into two groups each. One group was briquetted at 3000 psi and one group briquetted at 5000 psi. The resulting briquets were further split into three groups and subjected to different final carbonization heating rates. Rates of 18°C/min, 12°C/min and 6°C/min up to 870 to 900°C were used.

Regardless of the binder percentage or briquetting pressure used in making the briquets, the 18°C/min and 12°C/min produced swollen, cracked and friable formcoke. This was due to a too rapid devolatilization of the binder during final carbonization. The 6°C/min rate produced formcoke exhibiting a small amount of shrinkage, but very little distortion of original briquet shape (see Table 2).

TABLE 2

PHYSICAL APPEARANCE OF FORMCOKE MADE WITH 10 AND 20 PER CENT BINDER, 3000 AND 5000 PSI, AND FINAL CARBONIZATION HEATING RATES OF 18°C/MIN, 12°C/MIN AND 6°C/MIN TO 900°C

Binder Percentage	Briquetting Pressure (psi)	Heating Rate (C/min)	Physical Appearance
10	3000	18°	cracked and friable
		12°	cracked and friable
		6°	uniform and undistorted
10	5000	18°	cracked and friable
		12°	cracked and friable
		6°	uniform and undistorted
20	3000	18°	cracked and friable
		12°	cracked and friable
		6°	uniform and undistorted
20	5000	18°	cracked and friable
		12°	cracked and friable
		6°	uniform and undistorted

Effect of Binder Percentage and Briquetting Pressure on Formcoke

Having established a suitable final carbonization heating rate, establishment of a suitable binder percentage and briquetting pressure followed. Desirable on a production basis is a minimal amount of binder and briquetting pressure.

Because a minimal amount of binder and briquetting pressure is desirable, blends were made using 18 mesh 900°C-lignite char and 5, 10, 15, 20, and 25 per cent binder over a range of briquetting pressure. For the 5, 10, 20, and 25 per cent binder char-binder blends, briquetting pressures of 2000, 3000, 4000, 5000, 6000, and 7000 psi were used to produce 4 briquets at each pressure. For the 15 per cent binder char-binder blend, briquetting pressures of 1000, 2000, 3000, 4000, 5000, 6000, 7000, 8000, 9000, and 10000 psi were used to produce four briquets at each pressure.

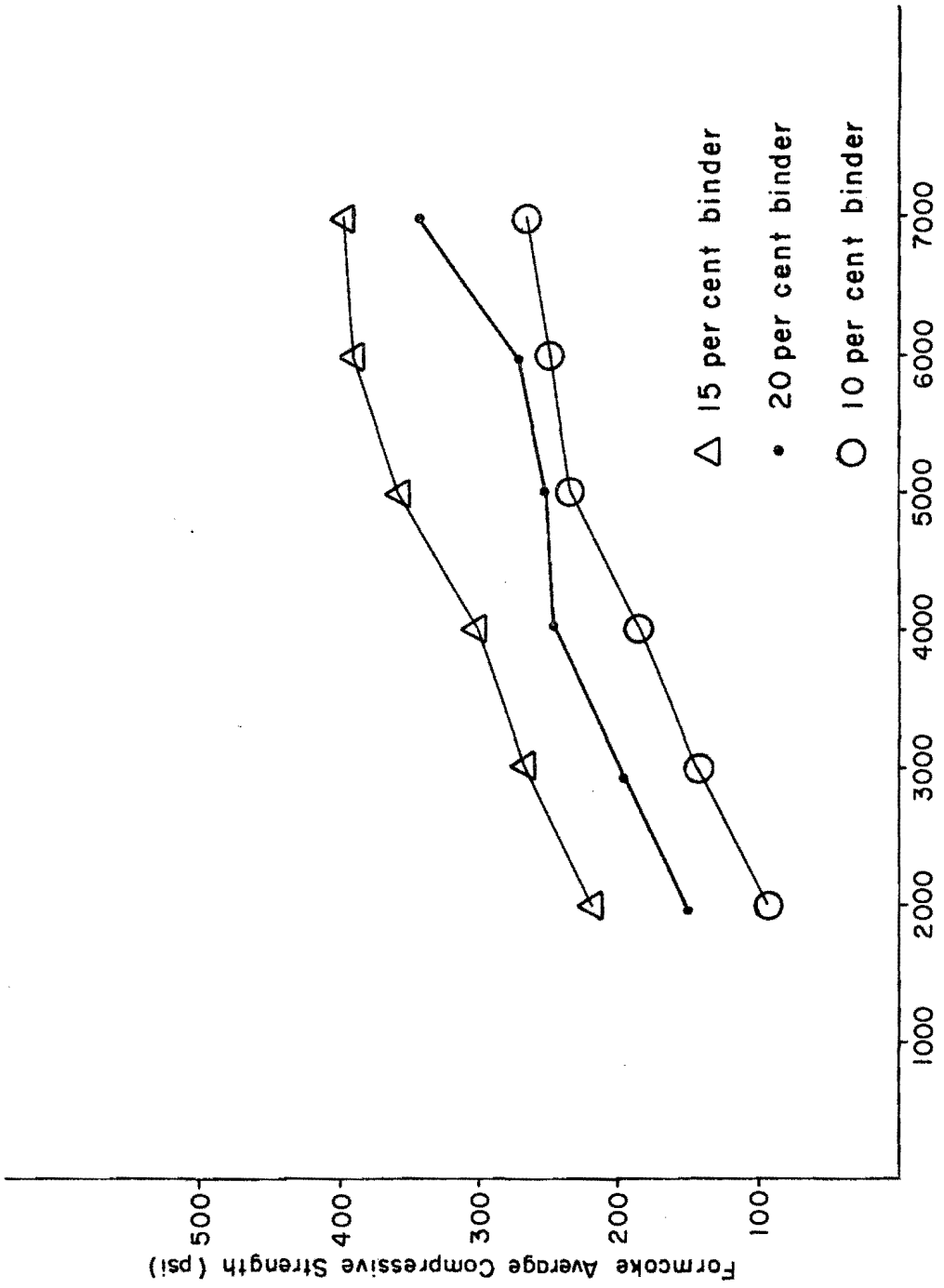
Use of 5 per cent binder in the blend proved to be insufficient; the briquets did not hold together once removed from the mold. Briquets made with 10, 15, and 20 per cent binder in the blend produced formcoke which exhibited uniform undistorted shape. Use of 25 per cent binder in the blend produced formcoke which exhibited excessive swelling and cracking, yielding friable briquets.

The char-binder blends with 15 per cent binder produced formcoke having greater compressive strength than formcoke produced from the 10 or 20 per cent blend (Figure 1).

Formcoke made from the 15 per cent blend showed a 20 to 250 psi greater compressive strength than formcoke produced from the 10 per cent blend briquetted at the same pressure. Formcoke made from the 15 per cent blend showed a 0 to 193 psi greater compressive strength than formcoke produced from the 20 per cent binder char-binder blends briquetted at the same pressure (Appendix A, Table 5). Because of this, 15 per cent binder was used in most of the following char-binder blends.

Regardless of the binder percentage used in the 10, 15, and 20 per cent binder blends, an increase in briquetting pressure resulted

Fig. 1. Effect of Binder Percentage on Average Formcoke Compressive Strength of Briquets Made with 18 Mesh 900°C-Lignite Char.



in an increase in formcoke compressive strength. This held true for each 1000 psi increment in briquetting pressure (Figure 1 and Figure 2).

The optimum briquetting pressure is a function of minimum energy input for the briquetting process resulting in adequate strength formcoke. For our laboratory process, a briquetting pressure of 3000 psi was selected for the following char-binder blends. The briquets were subjected to 3000 psi for 20 seconds.

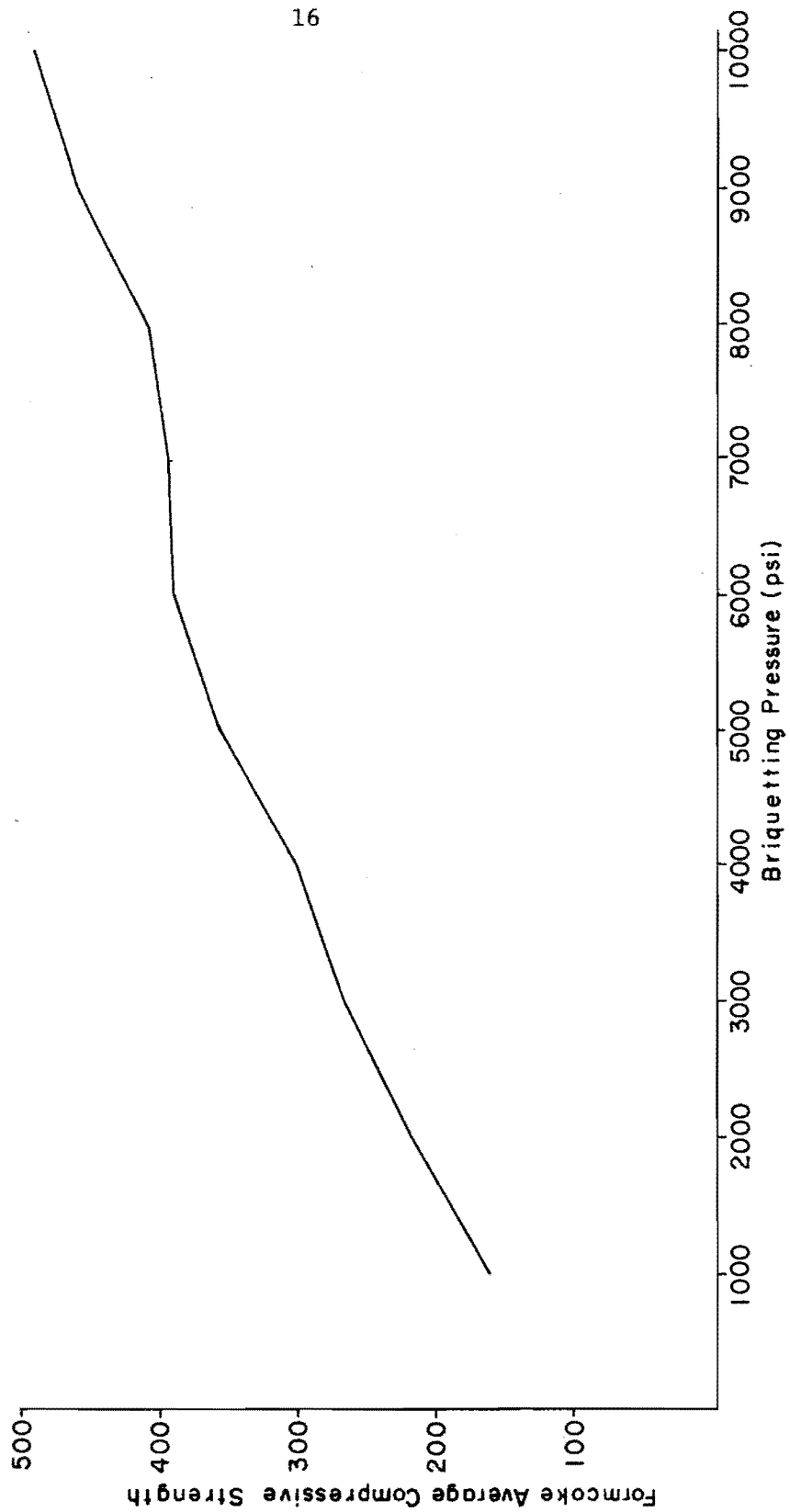
Effect of Initial Charring Temperature on Char Grain Size on Formcoke

For laboratory production purposes, a final carbonization heating rate of 6°C/min, a blending ratio of 15 per cent binder in the char-binder blends, and a briquetting pressure of 3000 psi were selected. The following work concerns the effect of initial charring temperature and char grain size on briquet crushing strength.

The 900°C-lignite, 600°C-lignite, 900°C-subbituminous, and 600°C-subbituminous chars were used in the char-binder blends from which the following formcoke briquets were made. Each char was split into three fractions. One fraction was ground to less than 18 mesh, one to less than 35 mesh, and one to less than 60 mesh. These size fractions will be referred to as 18 mesh, 35 mesh, or 60 mesh char. Refer to Appendix B for cumulative frequency distribution of grain size curves for each size fraction of each char.

Five formcoke briquets were made under each set of conditions. Four were tested for crushing strength and one was prepared for microscopic study.

Fig. 2. Effect of Briquetting Pressure on Average Formcoke Compressive Strength of Briquets Made With 18 Mesh 900°C-Lignite Char.



Effect of Initial Charring Temperature on Formcoke Compressive Strength

Formcoke made with blends of 900°C-lignite char, regardless of grain size, was much lower in compressive strength than formcoke made from blends of 600°C-lignite, 900°C-subbituminous, or 600°C-subbituminous char (Figure 3). The formcoke made with 900°C-lignite char and exhibiting the greatest compressive strength was made with 18 mesh char, and ranged from 388 to 410 psi in compressive strength. This was still several hundred psi lower in compressive strength than formcoke produced from the other chars (Appendix A, Table 7).

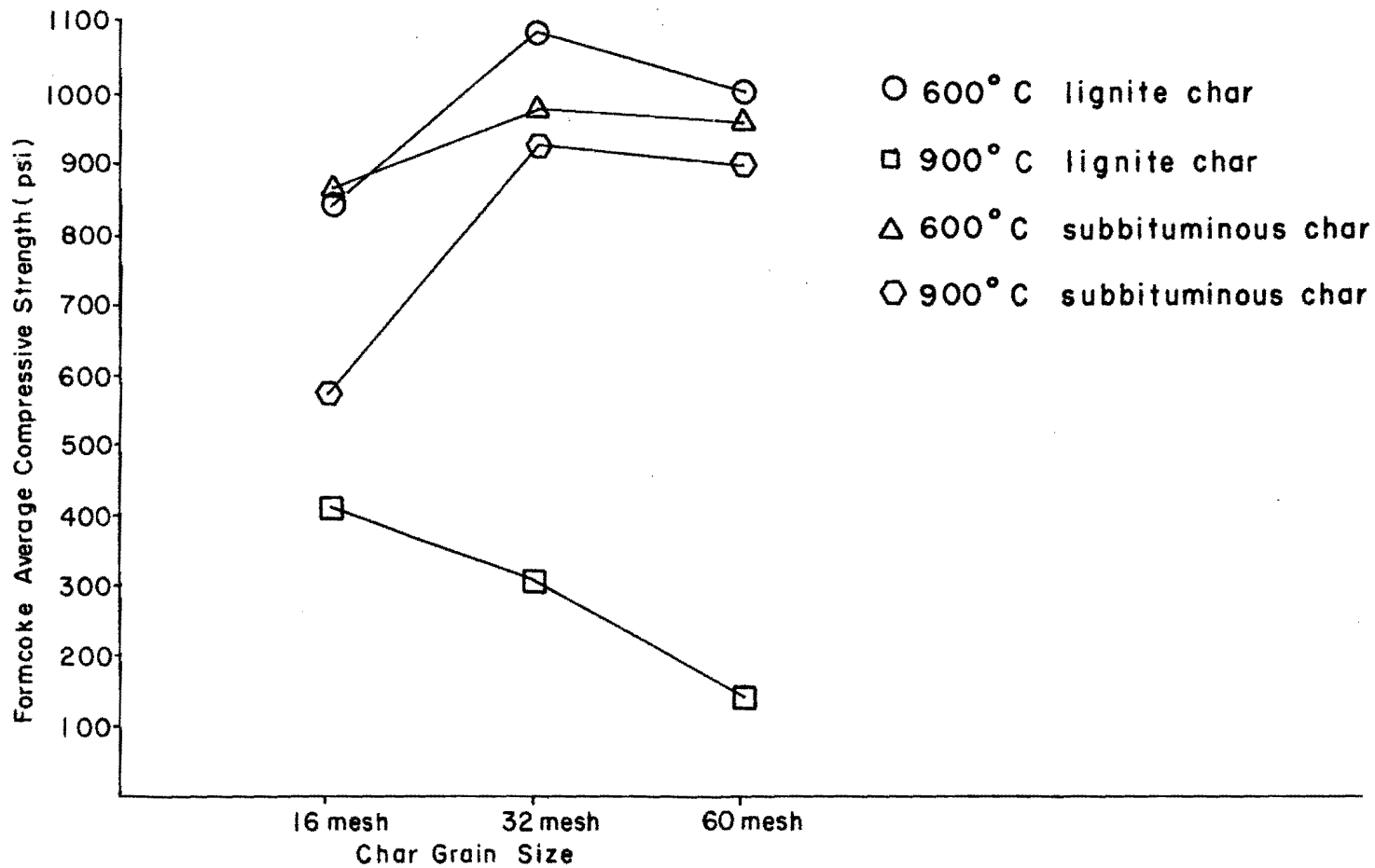
Formcoke produced from blends of 600°C-lignite char ranged higher in compressive strength than formcoke produced from 900°C-subbituminous char or 600°C-subbituminous char. The greatest strength 600°C-lignite formcoke was produced from 35 mesh char and ranged from 1063 to 1152 psi in compressive strength.

The formcoke produced from 35 and 60 mesh-600°C-subbituminous char exhibited greater compressive strength than formcoke produced from 35 and 60 mesh-900°C-subbituminous char. The 35 mesh-600°C-subbituminous char produced formcoke exhibiting 954 to 1053 psi compressive strength. The 35 mesh-900°C-subbituminous char produced formcoke exhibiting 896 to 950 psi compressive strength. The 18 mesh-900°C and 600°C subbituminous chars produced formcoke of about the same strength (Appendix A, Table 7).

Effect of Char Grain Size on Compressive Strength

Formcoke produced from blends of 18 and 35 mesh--600°C-lignite, 900°C-subbituminous and 600°C-subbituminous char--exhibited an increase in compressive strength with a decrease in char grain size (Figure 3).

Fig. 3. Effect of Char Grain Size and Initial Charring Temperature on Average Briquet Compressive Strength.



Formcoke produced from 18 mesh 600°C-lignite char ranged from 112 to 264 psi lower in compressive strength than formcoke produced from 35 mesh 600°C-lignite char. Formcoke produced from 18 mesh 600°C-subbituminous char ranged from 17 to 66 psi lower in compressive strength than formcoke produced from 35 mesh 600°C-subbituminous char. Formcoke produced from 18 mesh 900°C-subbituminous char ranged from 322 to 350 psi lower in crushing strength than formcoke produced from 35 mesh char.

Formcoke produced from blends of 60 mesh--600°C-lignite, 900°C-subbituminous and 600°C-subbituminous--chars were equal or slightly lower in compressive strength than formcoke produced from their respective 35 mesh char (Figure 3).

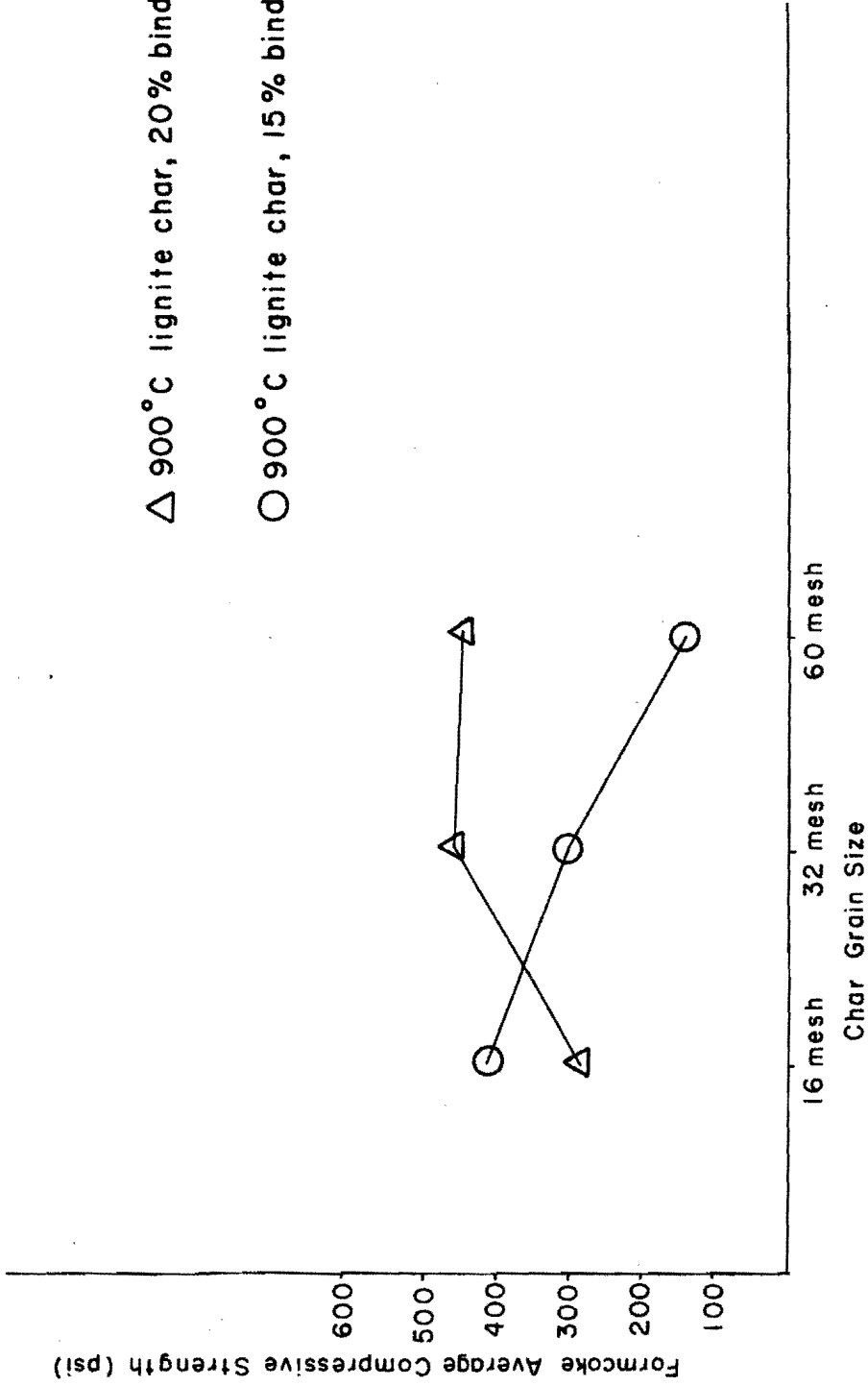
Formcoke produced from the 900°C-lignite char exhibited a decrease in compressive strength with a decrease in grain size. A decrease in grain size results in an increase in total grain surface area. Because of this, the decrease in compressive strength with decreasing size was probably due to a binder deficiency in the blends made from the finer grained 900°C-lignite char.

A batch of formcoke briquets were made from blends of 20 per cent binder and 18, 35, and 60 mesh 900°C-lignite char. The resulting formcoke showed a relationship between char grain size and compressive strength similar to the formcoke produced from 15 per cent binder and 600°C-lignite, 900°C-subbituminous, and 600°C-subbituminous chars. The 35 and 60 mesh chars produced formcoke of higher crushing strength than formcoke produced from the 18 mesh char (Figure 4). The 35 and 60 mesh char produced formcoke of about the same compressive strength.

Fig. 4. Effect of Binder Percentage on Average Briquet Compressive Strength of Formcoke Made With 18, 35, and 60 Mesh 900°C-Lignite Char.

△ 900°C lignite char, 20% binder

○ 900°C lignite char, 15% binder



Perhaps an increase in formcoke compressive strength could be obtained by increasing binder percentage to 20 per cent, for the 60 mesh 600°C-lignite, 900°C-subbituminous, and 600°C-subbituminous char-binder blends. Initial work concerning optimum binder percentage was with blends of 18 mesh char, and, by decreasing char grain size, a binder deficiency may have occurred in briquets made with 15 per cent binder and the finer grain chars.

Unlike the formcoke produced from 900°C-lignite char, increasing the binder percentage to 20 per cent produced no significant change in compressive strength in formcoke produced from 60 mesh--600°C-lignite, 900°C-subbituminous, and 600°C-subbituminous--chars (Table 3).

TABLE 3
EFFECT OF INCREASE IN BINDER FROM 15 TO 20 PER CENT ON FORMCOKE
COMPRESSIVE STRENGTH

Binder Percentage	Char Type	Compressive Strength (psi)	Average Compressive Strength (psi)
15	900°C-lignite	137, 125, 137, 189	147
	600°C-lignite	1010, 990, 976, 1049	1007
	900°C-subbituminous	976, 836, 937, 875	906
	600°C-subbituminous	989, 950, 1001, 937	969
20	900°C-lignite	475, 462, 436, 462	459
	600°C-lignite	927, 1027, 963, 976	973
	900°C-subbituminous	937, 993, 851, 930	937
	600°C-subbituminous	862, 927, 989, 914	923

For these formcoke briquets, whatever may have been gained by increasing the binder percentage may have been lost due to cracking of the formcoke briquets during final carbonization, resulting in a decrease in formcoke strength. Figure 5 shows cracking exhibited in a 60 mesh 600°C-subbituminous char formcoke briquet made with 20 per cent binder in the char-binder blend, and an undistorted formcoke briquet made with 15 per cent binder in the blend.

Proximate Analyses and Sulphur Content of the Chars and Some Formcoke

Proximate analyses and sulphur content of the chars and some of the formcoke produced from the chars were conducted and are listed in Tables 4 and 5.

The coal moisture and volatile matter content decreased greatly while the fixed carbon and ash content increased greatly, after charring had occurred. Sulphur content of the coal increased slightly after charring.

The volatile matter contents of the 900°C chars were about 4 to 7 per cent lower than the volatile matter contents of the 600°C chars made from the same coal. Ash contents of the 900°C chars were about 3.6 per cent higher than the ash contents of the 600°C chars of the same coal. Fixed carbon contents of the 900°C chars were about 6 per cent higher than fixed carbon contents of the 600°C chars of the same coal. The moisture and sulphur contents were about the same for both the 600°C and 900°C chars.

Formcoke volatile matter contents were 7 to 9 per cent lower than the volatile matter contents of the 600°C chars the formcoke was

Fig. 5. Formcoke Briquet Made With 20 Per Cent Binder and 60 Mesh Char (left) Exhibiting Devolatilization Cracks, Briquet at Right was Made With 15 Per Cent Binder and 60 Mesh Char Exhibits No Cracking.

TABLE 4
PROXIMATE ANALYSES OF CHARS

Sample	600°C- Lignite Char	900°C- Lignite Char	900°C- Subbituminous Char	600°C- Subbituminous Char	Binder
Moisture	1.0	1.4	1.4	1.0	-
Ash	12.0	16.4	10.8	12.4	-
Volatile Matter	8.4	4.1	9.0	3.2	78.5
Fixed Carbon	77.2	76.7	77.8	82.0	20.4
Sulphur	1.3	1.4	1.0	1.5	1.4

TABLE 5
PROXIMATE ANALYSES OF SOME FORMCOKE

Sample	Formcoke Made With 600°C- Lignite Char	Formcoke Made With 900°C- Lignite Char	Formcoke Made With 600°C- Subbituminous Char	Formcoke Made With 900°C- Subbituminous Char
Moisture	1.1	.9	.7	.7
Ash	15.3	16.7	11.9	12.6
Volatile Matter	1.0	.7	.7	.8
Fixed Carbon	81.2	80.0	85.7	84.4
Sulphur	1.3	1.5	1.2	1.2

produced from. Formcoke volatile matter contents were about the same as the volatile matter contents of the 900°C chars the formcoke was produced from.

Formcoke fixed carbon contents ranged from 80.1 to 85.7 per cent, and were 4 to 9 per cent higher than the fixed carbon contents of the chars the formcoke were produced from.

Formcoke sulphur and moisture contents were about the same as moisture and sulphur contents of the chars the formcoke was produced from. Moisture contents of the formcoke ranged from .7 to 1.1 per cent. Sulphur contents ranged from 1.2 to 1.5 per cent.

When carbonization occurs, the volatile matter and moisture evolve from the coal, thus the fixed carbon and ash percentage of the coal increases. The degree to which this occurs is dependent upon the temperature the coal is subjected.

Temperatures of 600°C or 900°C are sufficient to drive out any moisture contained in the coal. Moisture present in the chars, or the formcoke, at the time the proximate analyses were performed, was probably moisture absorbed from the air by the chars or the formcoke.

The volatile matter contents should be greater, and the fixed carbon and ash contents should be less, of coals subjected to lower carbonization temperatures than other coals. Thus the 600°C chars should, and do, contain more volatile matter and less fixed carbon and ash than the 900°C chars or any of the formcoke (carbonized at temperatures up to 870-900°C).

However, the fixed carbon, ash and volatile matter contents of the 900°C chars, and formcoke produced from these chars, should be about the same. But, the formcoke volatile matter contents are

slightly less, and the fixed carbon contents slightly more than the volatile matter and fixed carbon contents of the 900°C chars the form-coke was produced from. This is probably due to a greater degree of carbonization occurring during carbonization of the formcoke than occurred during the charring of the 900°C char. Blending with a low melting point binder of low ash content may also have increased the fixed carbon content of the formcoke. Since the melting point of the binder is low (75 to 95°C), at temperatures of 900°C all that is left of the binder is a carbon residue, which may have contributed to the increase in fixed carbon content noticed in the formcoke.

Petrographic Study of the Chars and Some of the Formcoke

The 600°C-lignite, 900°C-lignite, 600°C-subbituminous, and 900°C-subbituminous coal chars were studied microscopically. Reflectance measurements were made of 50 altered huminite grains from each char (Appendix C, Table 9).

Formcoke made from 18, 35, and 60 mesh--600°C-lignite, 900°C-lignite, 600°C-subbituminous, and 900°C-subbituminous--chars were also studied microscopically. Reflectance measurements were made of 50 altered huminite grains from some of the formcoke (Appendix C, Table 10).

The per cent light reflected from a huminite coal particle is a function of fixed carbon content of the coal. Measuring the reflectance of the huminite particles of the chars and formcoke is a method of determining relative degree of carbonization that has occurred during charring of the coals or final carbonization of the formcoke briquets.

Reflectance Measurements of Altered Huminite Grains of the Chars

The average reflectance of the 600°C chars were 1.75 to 1.78 per cent lower than the huminite reflectance of the same coal, 900°C chars.

Reflectance of the huminite grains of the 600°C-lignite char ranged from 3.30 to 4.35 per cent, and averaged 3.75 per cent. Reflectance of the huminite grains of the 900°C-lignite char ranged from 4.88 to 6.10 per cent and averaged 5.50 per cent. Reflectance measurements of the 600°C-subbituminous char ranged from 2.96 to 3.98 and averaged 3.6 per cent. Reflectance measurements of the 900°C-subbituminous char ranged from 5.10 to 6.24 per cent and averaged 5.38 per cent.

An increase in huminite reflectance is expected after such particles have been subjected to temperatures higher than those temperatures which were involved in determining the rank of the coal. For low-rank lignite and subbituminous coals, charring temperatures of 600°C or 900°C would certainly produce an increase in reflectance of the huminite grains. The increase is, and should be, greater for the 900°C chars than the 600°C chars.

Reflectance Measurements of Altered Huminites of Some of the Formcokes

Huminite particles of formcoke produced from the 600°C-lignite char ranged in reflectance from 5.17 to 6.33 per cent and averaged 5.70 per cent; from the 900°C-lignite char ranged from 5.15 to 6.24 per cent and averaged 5.59 per cent; from the 600°C-subbituminous char ranged from 5.60 to 6.40 per cent and averaged 5.94 per cent; and from the 900°C-subbituminous char ranged from 5.36 to 6.14 per cent and averaged 5.77 per cent.

The ranges of altered huminite reflectance of the 600°C chars were about 2.00 per cent lower than the ranges of altered huminite reflectance of formcoke made from these chars. This is expected since the formcoke briquets made with 600°C chars were carbonized at 870 to 900°C to produce the formcoke, thus undergoing a greater degree of carbonization than was involved in producing the 600°C chars.

The ranges of the altered huminite reflectance of the formcoke made with 900°C chars were slightly higher than the ranges of altered huminite reflectance of the 900°C chars. This indicates that a greater degree of carbonization occurred during final carbonization of the formcoke than occurred during the 900°C charring of the coals.

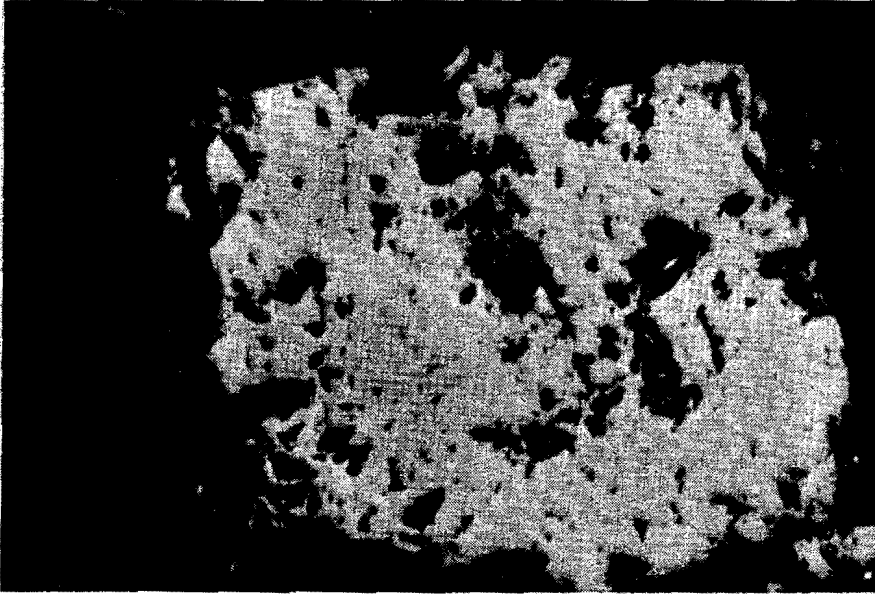
Petrographic Examination of the Chars

The liptinite fraction, in the chars and in the formcoke, appears to have been completely devolatilized or altered beyond recognition. I could not find any recognizable liptinite in either the 600°C char, 900°C char, or formcoke produced from these chars.

The huminite fractions of the lignite and subbituminous coals were severely altered after charring. The alteration--a fusing of the huminite and liptinite fractions into a homogenous appearing grains, lacking any structure except for devolatilization holes, and giving a semifusinite appearance to the grain--occurred to a greater degree in the 900°C chars than the 600°C chars (Figure 6).

The 900°C char grains are generally more porous, and appear weaker, than the 600°C char grains. The holes appearing in the grains probably arose from evolution of volatile matter contained in the huminite or the liptinite fractions.

Fig. 6. A 900°C-Lignite Char Grain (A) Showing a Greater Degree of Alteration Resulting in Greater Porosity Than the 600°C-Lignite Char Grain (B). (525X)



A



B

The inertinite fraction of the original coals was 2.3 to 3.7 per cent, and few inertinite grains were found in petrographic samples made from the chars or formcoke. Inertinite grains located in the samples exhibited slightly higher reflectivity than the altered huminites of the sample. The original inertinite structure did not appear to be damaged (Figure 7).

Petrographic Examination of Some of the Formcokes

Fusion of grain to grain is what lends strength to the formcoke. This seems to have been accomplished by the binder rather than interaction between grains of char (Figure 8). Since the binder has a low melting point (75 to 95°C) and is 78 per cent volatile matter, material remaining after carbonization at 870 to 900°C is mainly anisotropic carbon residue. This carbon residue is responsible for fusing the grains together.

Grain to grain contact is better developed in formcoke made with the 60 mesh chars than the formcoke made with 35 mesh char. Grain to grain contact is poor in formcoke made with 18 mesh char. There are more "floating" grains (grains which appear unattached to any others in polished section) in formcoke made with the coarser 18 and 35 mesh chars.

The 600°C chars produced formcoke exhibiting better grain to grain contact than formcoke produced from the 900°C chars. Grain to grain contact appeared to be best developed in formcoke made with 35 and 60 mesh 600°C-lignite chars, and poorest in formcoke made with 18 and 35 mesh 900°C-lignite chars (Figure 9).

Fig. 7. Inertinite Grain Found in a 600°C-Lignite Char Showing Preservation of Cell Wall Structure. (1100X)

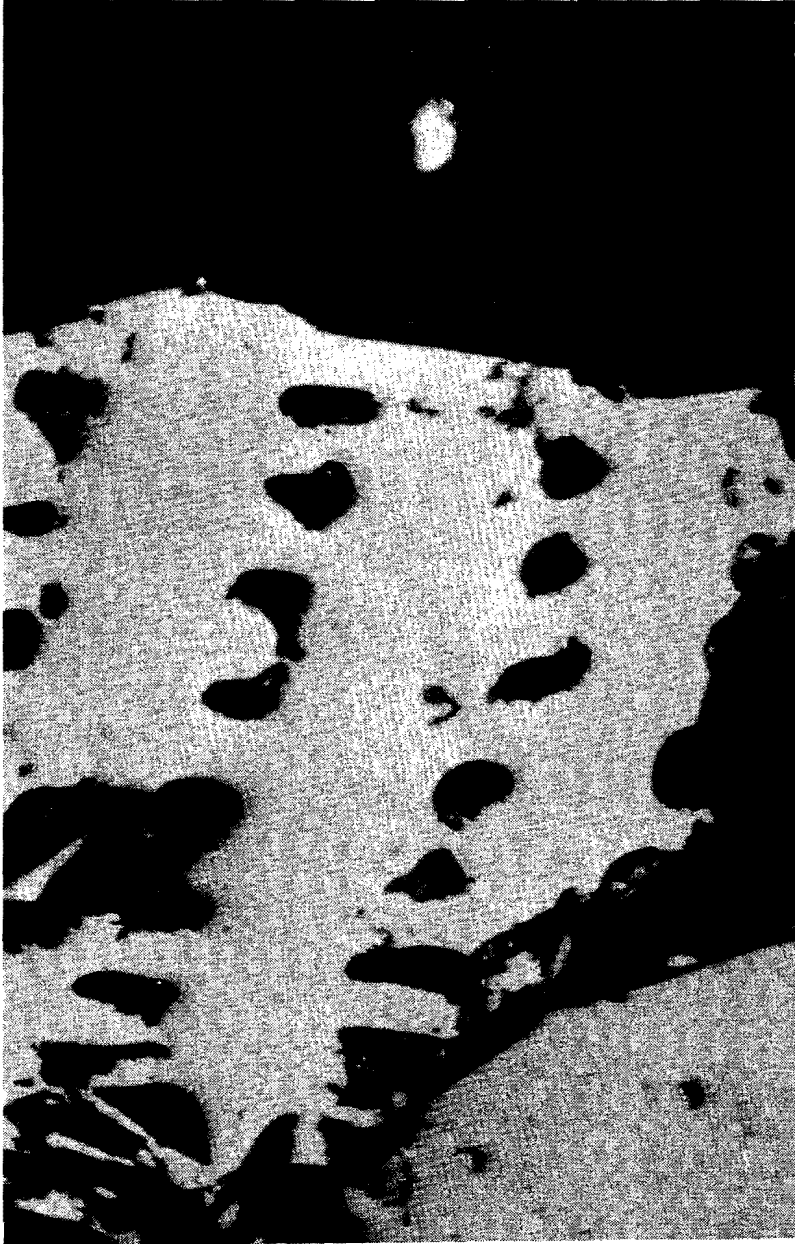


Fig. 8. Fusion of Grain to Grain Established by Carbon Residue From Binder (Lighter Anisotropic). (1100X)

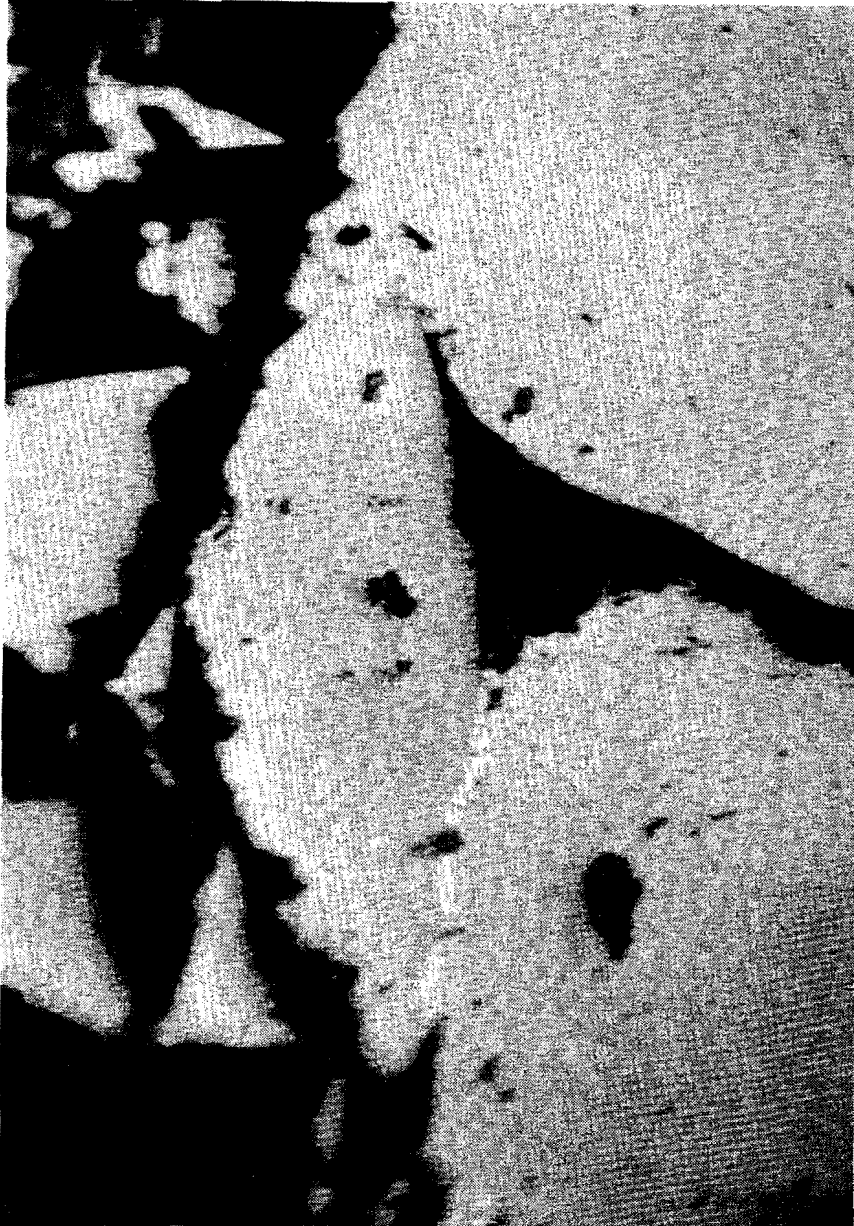
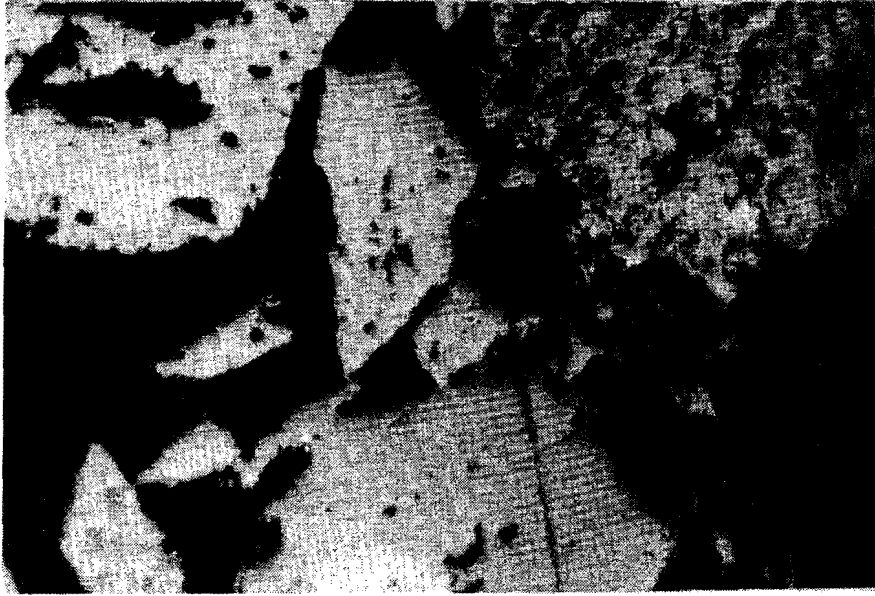
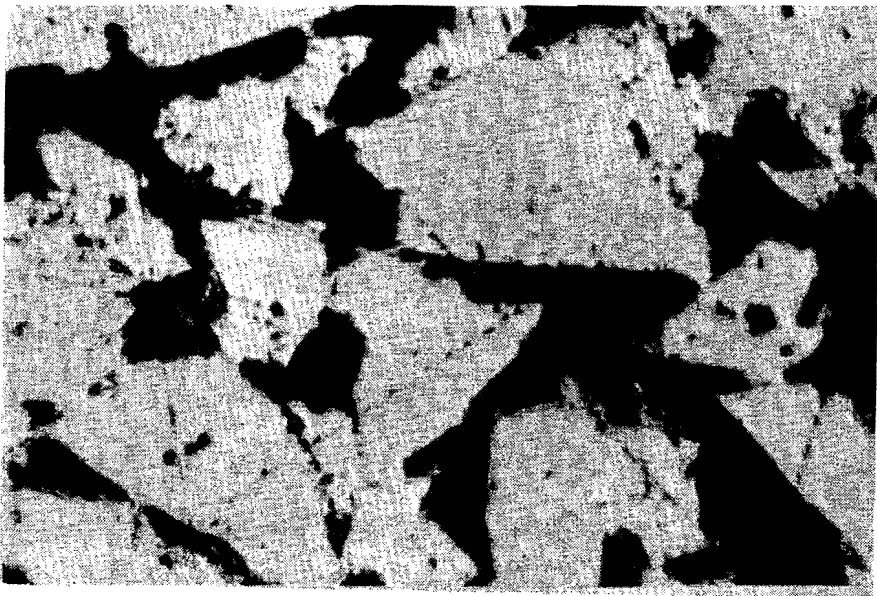


Fig. 9. Formcoke Made with 18 Mesh 900°C-Lignite Char Showing Poorly Developed Grain to Grain Contact (A). Formcoke Made with 60 Mesh 600°C-Lignite Char Showing Well Developed Grain to Grain Contact (B). (525X)



A



B

CONCLUSIONS AND DISCUSSION

Initial charring temperature, char grain size, binder percentage, briquetting pressure and final carbonization heating rate affected the compressive strengths of laboratory produced formcoke.

An increase in briquetting pressure resulted in an increase in formcoke compressive strength. A briquetting pressure of 3000 psi was selected for laboratory purposes. It does not necessarily represent the optimum briquetting pressure, and can not be compared to briquetting pressures of a roll press.

Binder percentage could be varied from greater than 5 per cent to less than 25 per cent. Fifteen per cent binder was optimum when blended with 18 mesh 900°C-lignite char. Since the finer grain chars have a greater total surface area than the 18 mesh chars, 15 per cent binder may not be the optimum amount when blended with 35 or 60 mesh chars. However, compressive strengths of formcoke produced with blends of 60 mesh--600°C-lignite, 900°C-subbituminous, and 600°C-subbituminous--chars and 20 per cent binder varied little from compressive strengths of formcoke produced from 60 mesh char and 15 per cent binder.

The formcoke made with 20 per cent binder and 60 mesh--600°C-lignite, 900°C-subbituminous, and 600°C-subbituminous--char exhibited shrinkage cracks after final carbonization. The cracks, due to too much binder or too rapid a devolatilization of the binder, probably weakened the formcoke briquet. The final heating rate of 6°C/min (selected for

a 18 mesh and 15 per cent char binder blend) may have been too rapid for the 60 mesh char and 20 per cent binder blends.

The final carbonization heating rate could vary on a production basis depending upon the nature of the coal char, binder percentage, and possibly the briquetting pressure applied to the char-binder blends. A rate of 6°C/min was selected as adequate for laboratory production of formcoke, and produced uniform briquets exhibiting no devolatilization cracks when used on briquets made with 15 per cent binder.

The 35 mesh and 60 mesh chars produced formcoke exhibiting greater compressive strength than formcoke produced from the 18 mesh char. Formcoke produced from the 35 mesh char was of about the same compressive strength as formcoke produced from the 60 mesh char. Microscopically, formcoke produced from 60 mesh char seemed to show better grain to grain contact than formcoke produced from 35 or 18 mesh char, and formcoke produced from the 35 mesh char exhibited better grain to grain contact than formcoke produced from 18 mesh char.

Initial charring temperature had a significant effect of formcoke compressive strength. The 900°C chars produced lower compressive strength formcoke than the 600°C chars.

Binder residue seems to be responsible for fusing the grains together. However, since the 600°C chars produce formcoke exhibiting greater compressive strength than the 900°C chars, grain reactivity of the chars probably effects the compressive strength of the formcoke.

Microscopically, the 600°C char seemed to produce formcoke exhibiting better developed grain to grain contact than the 900°C

rs. This may be due to shrinkage of the briquets during final carbonization (due to devolatilization of the binder and further devolatilization of the chars, especially the 600°C chars). Formcoke produced from the 600°C chars exhibit shrinkage up to 1/8 inch in height and 1/16 inch in diameter. Formcoke produced from the 900°C chars produced briquets exhibiting little or no shrinkage in diameter, and up to 1/16 inch in height. This may result in a density difference in the formcoke produced from the two char types, resulting in more dense and stronger formcoke being produced from the 600°C chars.

Another factor that may have affected the formcoke compressive strength is char porosity resulting from evolution of volatile matter, retained in the grains, during charring. The 900°C chars appear more porous than the 600°C chars. Char porosity also appears greater in formcoke made with 900°C char than formcoke made with 600°C char, in spite of the fact that all the briquets were charred at the same temperature (870 to 900°C). If porosity results in a lower strength grain, it would also result in lower strength formcoke produced from those grains. This detrimental effect on formcoke strength would be greater in the formcoke made from 900°C char than formcoke made with 600°C char.

In conclusion, formcoke produced from 600°C-lignite char had equal or better compressive strength as formcoke produced from 600°C-bituminous char or 900°C-subbituminous chars under the same laboratory conditions. Because of this, and since a Wyoming subbituminous coal has been used to produce formcoke on a commercial basis, I feel that commercial production of formcoke using North Dakota lignite is possible.

To produce the greatest strength formcoke, the coal should be
dried at a low temperature (600°C or possibly less) and ground to at
least 35 mesh. If formcoke of greater density is desired, the char
should be ground finer than 35 mesh. The optimum binder percentage,
briquetting pressure, or final carbonization heating rate are depen-
dent upon the blending, briquetting, and carbonization processes used
in a large scale production process. For laboratory purposes, a bri-
quetting pressure of 3000 psi, binder percentage of 15 per cent, and
final carbonization heating rate of 6°C/min, produced uniform shape
formcoke exhibiting compressive strength up to 1152 psi.

APPENDIX A

COMPRESSIVE STRENGTHS OF FORMCOKE

TABLE 6

COMPRESSIVE STRENGTHS OF FORMCOKE PRODUCED FROM 18 MESH 900°C-
LIGNITE CHAR AND 10, 15 OR 20 PER CENT BINDER USING A RANGE
OF BRIQUETTING PRESSURES

Binder Percentage	Briquetting Pressure (psi)	Compressive Strengths (psi)	Average Compressive Strengths (psi)
10	7,000	228, 276, 302, 268	268
	6,000	197, 236, 249, 249	249
	5,000	170, 249, 249, 236	236
	4,000	170, 173, 210, 183	184
	3,000	131, 147, 163, 144	145
	2,000	71, 105, 92, 111	95
	10,000	446, 485, 525, 485	485
	9,000	412, 446, 420, 485	466
	8,000	328, 472, 446, 407	413
	7,000	354, 407, 380, 446	396
15	6,000	351, 367, 436, 420	395
	5,000	328, 375, 38, 367	361
	4,000	328, 315, 302, 263	302
	3,000	263, 263, 302, 249	369
	2,000	183, 236, 201, 214	210
	1,000	183, 144, 157, 162	160
	7,000	341, 249, 407, 394	348
	6,000	236, 253, 289, 304	275
20	5,000	276, 289, 196, 249	253
	4,000	249, 259, 232, 253	248
	3,000	157, 209, 237, 197	200
	2,000	136, 170, 157, 144	152

TABLE 7

COMPRESSIVE STRENGTH OF FORMCOKE PRODUCED FROM 85 PER CENT OF 600°C-LIGNITE, 900°C-LIGNITE, 600°C-SUBBITUMINOUS OR 900°C-SUBBITUMINOUS CHAR AND 15 PER CENT BINDER

Char Type	Char Grain Size	Compressive Strength (psi)	Average Compressive Strength (psi)
900°C-Lignite	18 mesh	937, 836, 810, 875	842
	35	1049, 1104, 1152, 1063	1092
	60	1010, 990, 976, 1049	1007
600°C-Lignite	18	338, 423, 475, 410	589
	35	312, 286, 312, 335	931
	60	137, 125, 137, 187	906
900°C-Subbituminous	18	810, 862, 849, 937	864
	35	1001, 1003, 954, 987	989
	60	989, 950, 1001, 937	969
600°C-Subbituminous	18	569, 578, 546, 564	589
	35	939, 940, 950, 986	931
	60	976, 836, 937, 875	906

TABLE 8

COMPRESSIVE STRENGTH OF FORMCOKE BRIQUETS MADE FROM A BLEND OF 20 PER CENT BINDER AND 80 PER CENT--18, 35, OR 60 MESH--900°C LIGNITE CHAR

Grain Size	Compressive Strength (psi)	Average Compressive Strength (psi)
3 Mesh	299, 322, 238, 286	284
5 Mesh	488, 475, 436, 462	465
10 Mesh	475, 462, 436, 462	459

APPENDIX B

CUMULATIVE FREQUENCY OF CHAR GRAIN SIZE

Fig. 10. Cumulative Frequency of Grain Size for 18, 35, and 60 Mesh 600°C-Lignite Char.

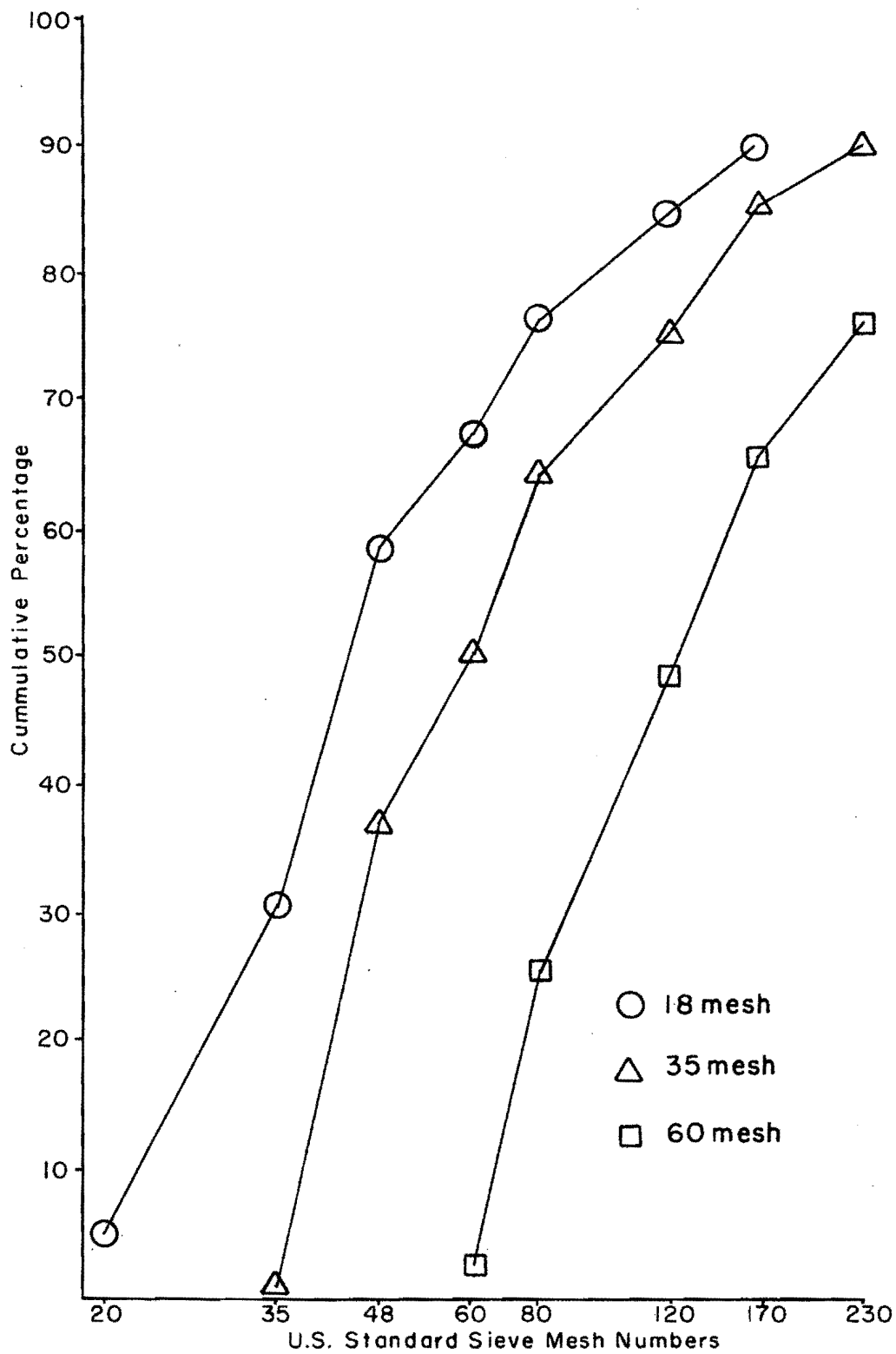


Fig. 11. Cumulative Frequency of Grain Size for 18, 35, and 60 Mesh 900°C-Lignite Char.

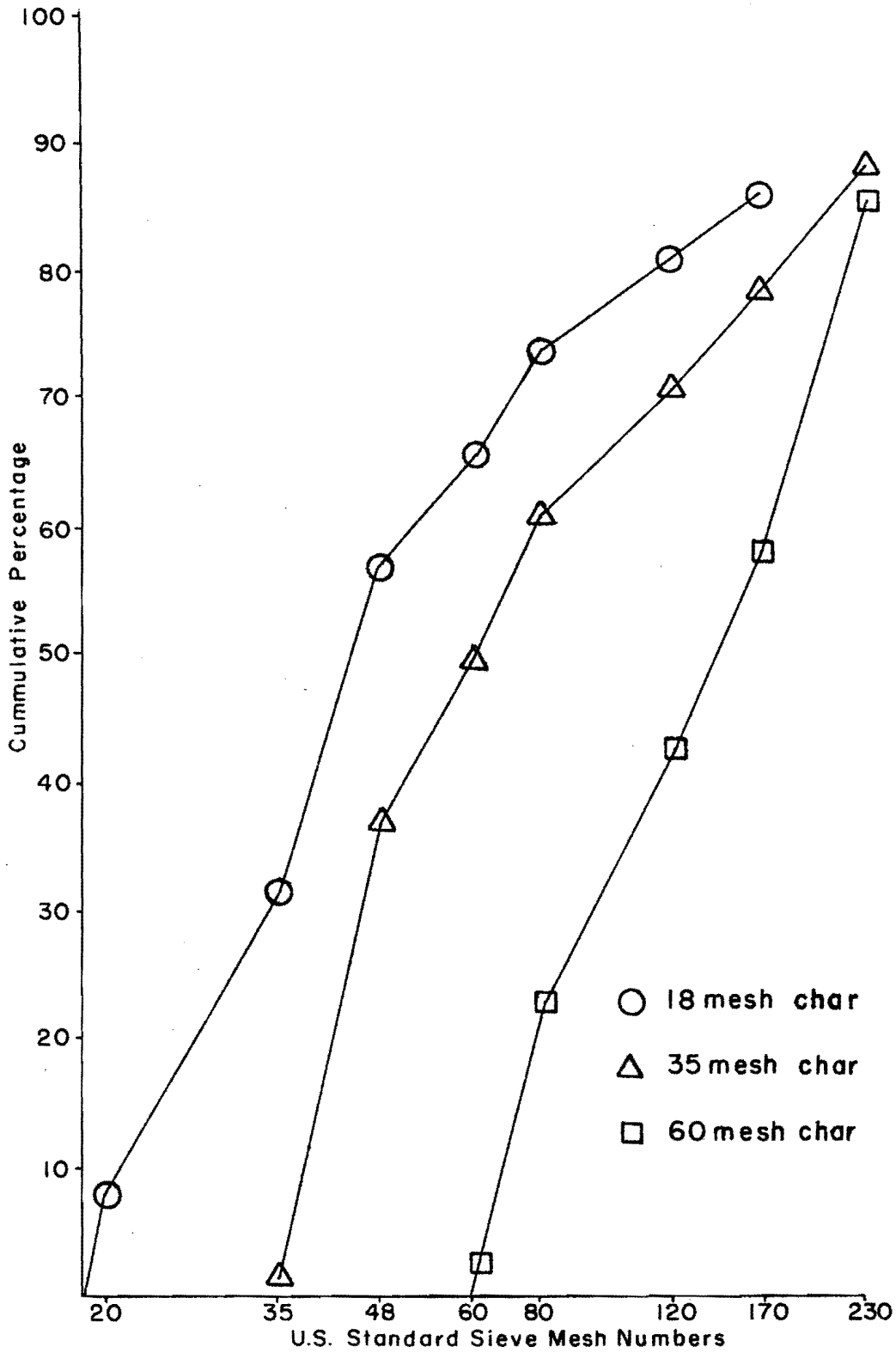


Fig. 12. Cumulative Frequency of Grain Size for 18, 35, and 60 Mesh 600°C-Subbituminous Char.

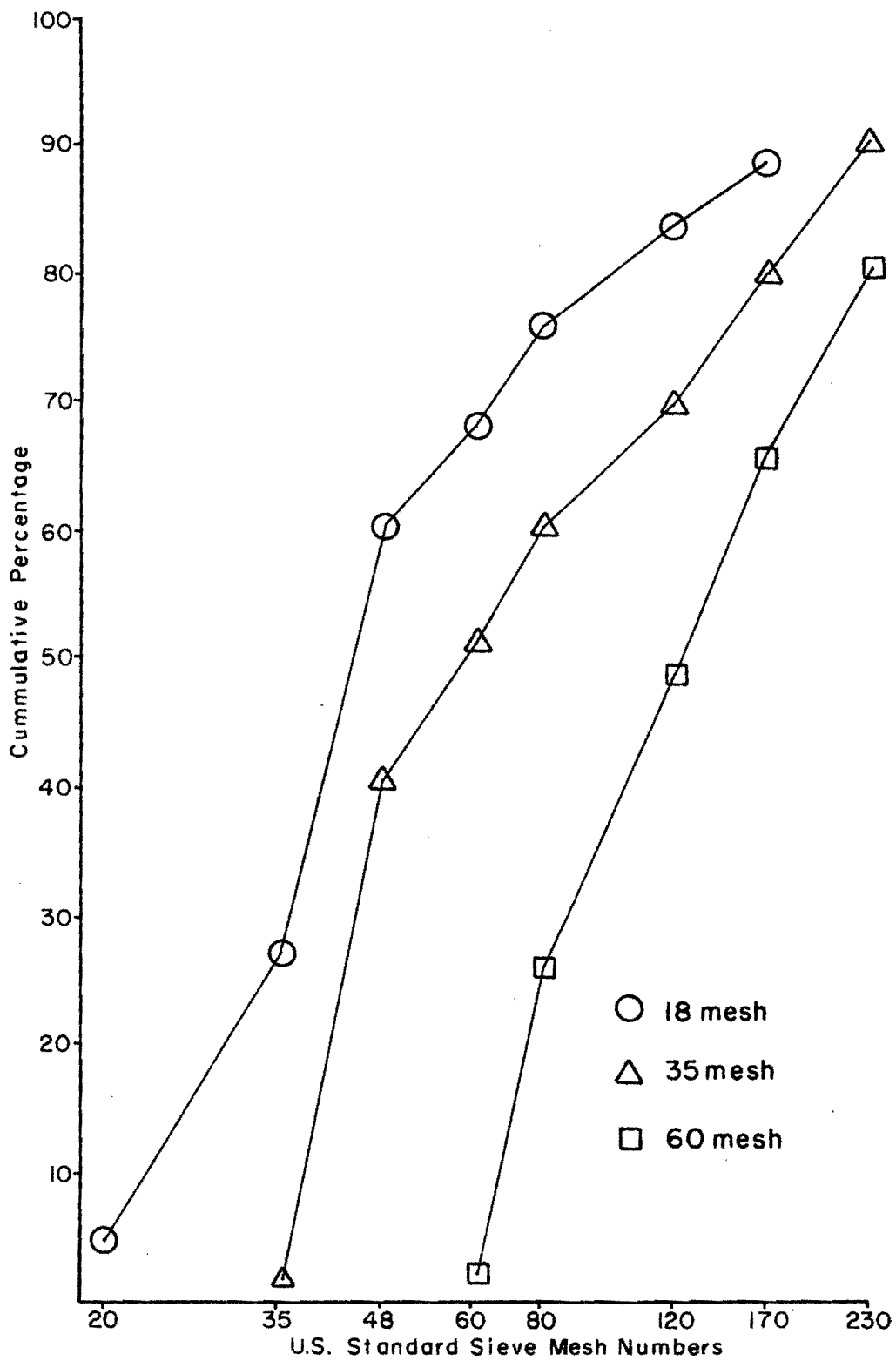
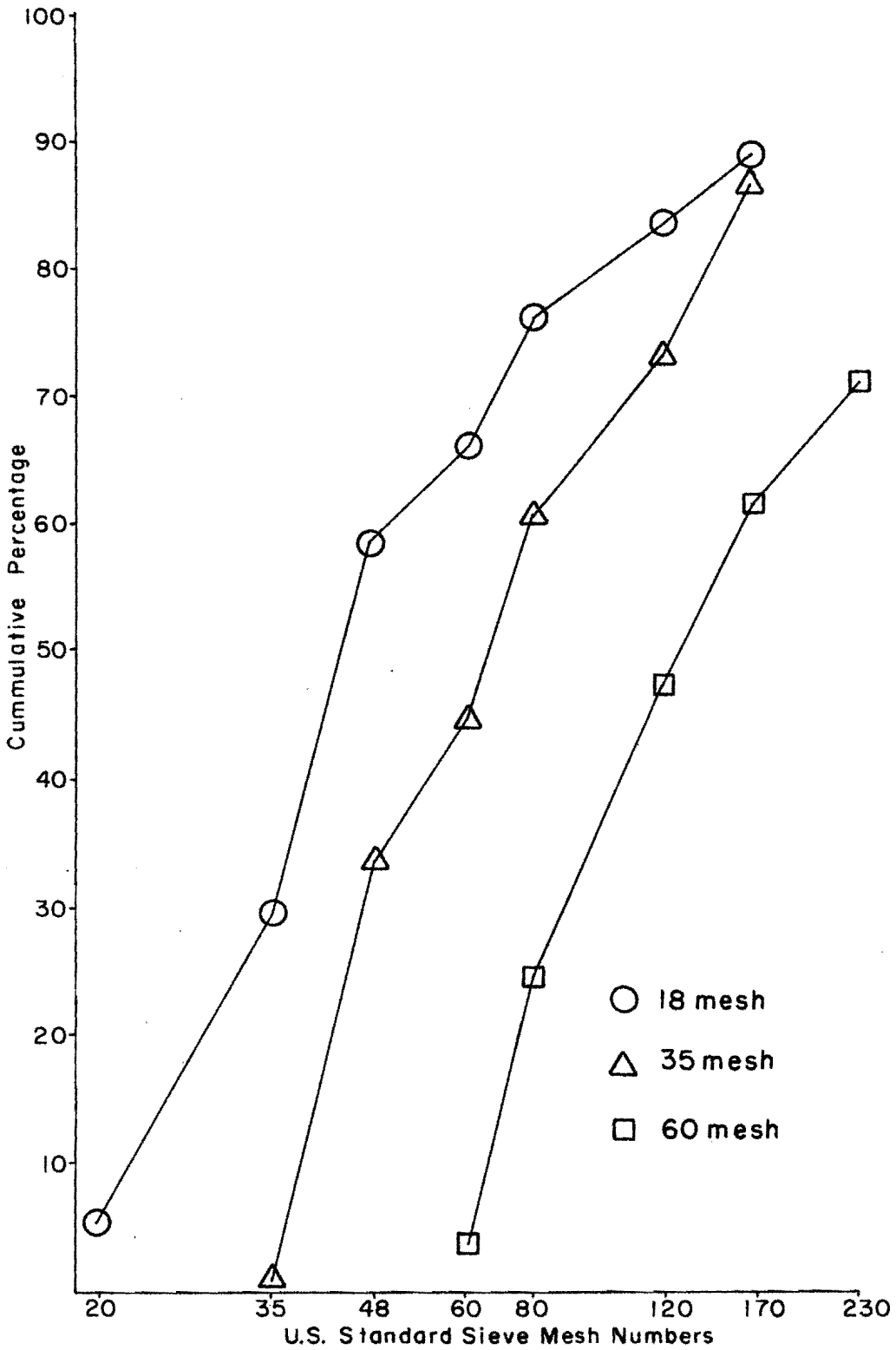


Fig. 13. Cumulative Frequency of Grain Size for 18, 35, and 60 Mesh 900°C-Subbituminous Char.



APPENDIX C

PERCENTAGE REFLECTANCE OF HUMINITE GRAINS
OF THE CHARS AND SOME FORMCOKE

TABLE 9

PERCENTAGE REFLECTANCE OF 50 RANDOMLY SELECTED CHAR GRAINS FROM EACH OF THE CHARs

500°C- Lignite Char	600°C- Lignite Char	Char Type 900°C- Subbituminous Char	600°C- Subbituminous Char
Percentage Reflectance			
4.88	3.30	5.10	3.10
4.90	3.35	5.14	3.28
4.95	3.47	5.16	3.29
5.06	3.48	5.19	3.30
5.06	3.54	5.20	3.34
5.19	3.60	5.34	3.37
5.20	3.64	5.41	3.38
5.24	3.66	5.44	3.42
5.28	3.67	5.46	3.45
5.29	3.70	5.48	3.45
5.30	3.70	5.50	3.51
5.33	3.72	5.53	3.56
5.34	3.77	5.53	3.57
5.36	3.80	5.54	3.59
5.36	3.81	5.54	3.59
5.37	3.81	5.55	3.60
5.39	3.82	5.55	3.61
5.41	3.88	5.56	3.62
5.42	3.89	5.56	3.63

TABLE 9--Continued

°C- nite ar	600°C- Lignite Char	Char Type	
		900°C- Subbituminous Char	600°C- Subbituminous Char
Percentage Reflectance			
.48	3.89	5.56	3.66
.50	3.90	5.57	3.69
.50	3.91	5.60	3.70
5.51	3.92	5.60	3.74
5.51	3.92	5.60	3.77
5.53	3.95	5.66	3.81
5.54	3.96	5.66	3.82
5.57	3.96	5.68	3.83
5.57	3.97	5.68	3.87
5.58	3.98	5.68	3.88
5.59	3.98	5.68	3.89
5.60	3.99	5.69	3.89
5.62	3.99	5.70	3.89
5.62	4.00	5.70	3.90
5.63	4.01	5.70	3.94
5.63	4.02	5.71	3.95
5.65	4.03	5.73	3.96
5.66	4.05	5.76	4.01
5.67	4.06	5.76	4.03
5.69	4.06	5.78	4.04

TABLE 9--Continued

°C- Lignite Char	600°C- Lignite Char	Char Type	
		900°C- Subbituminous Char	600°C- Subbituminous Char
Percentage Reflectance			
70	4.10	5.79	4.06
73	4.14	5.80	4.08
75	4.15	5.84	4.10
78	4.16	5.85	4.11
82	4.17	5.90	4.13
83	4.20	5.92	4.14
84	4.21	5.96	4.18
87	4.30	5.97	4.19
89	4.34	6.20	4.20
90	4.35	6.25	4.24
10	3.95	5.66	3.79
Average			
50	3.75	5.38	3.60

TABLE 10

PERCENTAGE REFLECTANCE OF 50 RANDOMLY SELECTED CHAR GRAINS OF
FORMCOKE REPRESENTING EACH OF THE CHARS

Formcoke From 900°C- Lignite Char	<u>Formcoke Type</u>		Formcoke From 600°C- Subbituminous Char
	Formcoke From 600°C- Lignite Char	Formcoke From 900°C- Subbituminous Char	
Percentage Reflectance			
5.15	5.17	5.36	5.60
5.18	5.20	5.44	5.68
5.20	5.35	5.47	5.70
5.23	5.42	5.50	5.70
5.25	5.46	5.51	5.72
5.26	5.49	5.52	5.74
5.27	5.50	5.53	5.77
5.27	5.50	5.54	5.79
5.28	5.52	5.56	5.78
5.30	5.53	5.56	5.80
5.31	5.56	5.58	5.82
5.34	5.57	5.61	5.83
5.36	5.58	5.63	5.83
5.36	5.59	5.65	5.84
5.38	5.60	5.66	5.86
5.39	5.60	5.68	5.87
5.41	5.61	5.68	5.87
5.42	5.61	5.69	5.88

TABLE 10--Continued

Formcoke From 900°C- Lignite Char	<u>Formcoke Type</u>		
	Formcoke From 600°C- Lignite Char	Formcoke From 900°C- Subbituminous Char	Formcoke From 600°C- Subbituminous Char
Percentage Reflectance			
5.45	5.61	5.70	5.90
5.46	5.62	5.71	5.92
5.48	5.63	5.72	5.93
5.49	5.63	5.72	5.93
5.50	5.64	5.74	5.93
5.50	5.64	5.76	5.95
5.51	5.64	5.77	5.96
5.51	5.67	5.79	5.96
5.55	5.68	5.80	5.96
5.56	5.68	5.81	5.98
5.59	5.69	5.81	5.98
5.60	5.70	5.82	5.98
5.63	5.70	5.84	5.99
5.66	5.74	5.84	6.00
5.69	5.77	5.85	6.00
5.70	5.79	5.86	6.01
5.72	5.80	5.88	6.02
5.77	5.80	5.90	6.02
5.78	5.80	5.93	6.02
5.80	5.83	5.94	6.02

TABLE 10--Continued

Formcoke From 900°C-- lignite Char	<u>Formcoke Type</u>		
	Formcoke From 600°C-- Lignite Char	Formcoke From 900°C-- Subbituminous Char	Formcoke From 600°C-- Subbituminous Char
Percentage Reflectance			
5.80	5.84	5.95	6.03
5.81	5.85	5.98	6.07
5.84	5.85	6.04	6.09
5.90	5.90	6.05	6.09
5.97	5.94	6.07	6.10
5.99	5.95	6.08	6.11
6.04	5.97	5.85	6.14
6.06	6.00	5.98	6.17
6.07	6.05	6.09	6.23
6.11	6.20	6.09	6.30
6.17	6.10	5.77	5.98
6.24	6.33	6.14	6.40
Average			
5.59	5.70	5.77	5.94

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