

University of Mississippi

eGrove

Open-File Reports

Mississippi Mineral Resources Institute

1985

Micropulverization and Drying of Lignite

W. Glenn Steele

Follow this and additional works at: https://egrove.olemiss.edu/mmri_ofr

Recommended Citation

Steele, W. Glenn, "Micropulverization and Drying of Lignite" (1985). *Open-File Reports*. 91.
https://egrove.olemiss.edu/mmri_ofr/91

This Report is brought to you for free and open access by the Mississippi Mineral Resources Institute at eGrove. It has been accepted for inclusion in Open-File Reports by an authorized administrator of eGrove. For more information, please contact egrove@olemiss.edu.

Open-File Report 85-2Sc

Micropulverization and Drying of Lignite

W. Glenn Steele and Robert B. Ross

1985

The Mississippi Mineral Resources Institute
University, Mississippi 38677

MICROPULVERIZATION AND DRYING OF LIGNITE COAL

Birs

ENGINEERING AND INDUSTRIAL RESEARCH STATION

Department of Mechanical and Nuclear Engineering

A Technical Report

by

**W. Glenn Steele
and
Robert B. Ross**

Submitted to:

**The Mississippi Mineral Resources Institute
University, MS 38677**

October 1985

**Mississippi State University
Mississippi State, MS 39762**

MSSU-EIRS-M&NE-85-3

Micropulverization and Drying of Lignite Coal

A Technical Report

by

W. Glenn Steele
and
Robert B. Ross

Submitted to:

The Mississippi Mineral Resources Institute
University, MS 38677

Submitted by:

Mechanical and Nuclear Engineering Department
Mississippi State University
Mississippi State, MS 39762

October 1985

MMRI GRANT NO. 85-25
U.S. BUREAU OF MINES GRANT NO. G115^128

MSU Publication Notice

Mississippi State University does not discriminate on the basis of race, color religion, national origin, sex, age, or handicap.

In conformity with Title IX of the Education Amendments of 1972 and Section 504 of the Rehabilitation Act of 1973, Joyce B. Giglioni, Assistant to the President, 610 Allen Hall, P. O. Drawer J, Mississippi State, Mississippi 39762, office telephone number 215~3221, has been designated as the responsible employee to coordinate efforts to carry out responsibilities and make investigation of complaints relating to discrimination.

Micropulverization and Drying of Lignite Coal

by

W. Glenn Steele

and

Robert B. Ross

Abstract

This report describes combined micropulverization and drying of lignite coal. Heated air or steam is used as the grinding medium in a fluid energy mill type pulverizer. Average product sizes are less than 10 microns in diameter. Also described is a hot-water drying technique which results in a very dry, high heating value product.

It has been found that a coal type fuel with a mean particle size of about 10 microns can be burned in a boiler originally designed for oil or natural gas with minimum slagging of the heat transfer surfaces. There is an estimated 35 billion tons of recoverable lignite coal in the Gulf Coast region. This region has many oil and gas boilers which are used in industry and consume a significant portion of the nation's total energy usage. A lignite water slurry is one form being considered as a retrofit fuel.

The report gives the results of combined pulverization and drying and of hot-water drying. Detailed analyses of the products from each drying temperature are included. It is found that the inherent moisture content can be reduced from an initial 40% to less than 15% which is crucial for satisfactory slurry production.

TABLE OF CONTENTS

	Page
Title Page	i
MSU PublicationNotice	ii
Abstract	iii
Table of Contents	iv
List of Figures	v
List of Tables	vi
<u>Chapter</u>	
1 Introduction	1
2 Micropulverization	5
3 Experimental Procedure	9
4 Results of Combined Micropulverization and Drying Tests	14
5 Results of Hot-Water DryingTests	30
6 Summary and Conclusions	44
References	46

LIST OF FIGURES

Figure		Page
1	Equilibrium Moisture vs. Temperature for Steam and Air Micropulverized Lignite	22
2	Dry Basis Ash vs. Temperature for Steam and Air Micropulverized Lignite	23
3	Dry Basis Volatile Matter vs. Temperature for Steam and Air Micropulverized Lignite	24
4	Dry Basis Heating Value vs. Temperature for Steam and Air Micropulverized Lignite	25
5	Porosity vs. Temperature for Steam and Air Micropulverized Lignite	26
6	Particle Size vs. Temperature for Steam and Air Micropulverized Lignite	27 ..
7	Density vs. Temperature for Steam and Air Micropulver ized Lignite	28
8	Electron Microscope Photographs .	29
9	Equilibrium Moisture vs. Temperature for Micropulverized, Hot-Water Dried Lignite	36
10	Dry Basis Ash vs. Temperature for Micropulverized, Hot-Water Dried Lignite	37
11	Dry Basis Volatile Matter vs. Temperature for Micropulverized, Hot-Water Dried Lignite	38
12	Dry Basis Heating Value vs. Temperature for Micropulverized, Hot-Water Dried Lignite	39
13	Porosity vs. Temperature for Micropulver ized, Hot-Water Dried Lignite	40
14	Particle Size vs. Temperature for Micropulverized, Hot-Water Dried Lignite	41
15	Density vs. Temperature for Micropulver ized, Hot-Water Dried Lignite	42
16	Electron Microscope Photographs of Hot-Water Dried Lignite	43

LIST OF TABLES

Table		Page
1	As-Mined Analyses	10
2	Feed-Stock Analyses	15
3	Combined Micropulverization and Drying Test Results	18
4	Dry Basis Proximate Analysis for Micropulverized Test Samples	19
5	Ultimate Analyses for Micropulverized Products ...	20
6	Mineral Analysis of Ash for Micropulverized Test Samples	21
7	Hot-Water Drying Test Results	33
8	Ultimate Analyses for Hot-Water Dried Products ...	34
9	Mineral Analysis of Ash for Hot-Water Dried Test Samples	35

CHAPTER 1

INTRODUCTION

The present instabilities in both costs and supply of oil and gas, coupled with the inevitable increase in costs give strong incentives for the conversion of oil and gas-fired boilers to coal. The five general classifications of coal are: peat, lignite, subbituminous, bituminous, and anthracite. These classifications of coal are arranged in an ascending order of carbon content and age. The United States has an estimated ultimate resource of 2.9 trillion metric tons of coal. Of this ultimate resource 182 billion tons are economically recoverable (1).

During the 1970's the Gulf Coast region (Texas, Louisiana, Arkansas, Tennessee, Mississippi, and Alabama) accounted for approximately 50 percent of the nations natural gas use and 31 percent of the nations total energy use. This region also contains an estimated 35 billion tons of lignite coal in depths of less than 200 feet. Therefore, the use of lignite coal as a retrofit fuel for this region is attractive (2).

The quality of the Gulf Coast lignites can vary significantly even within individual mining areas and seams. However, generalized regional trends increase in quality from east to west. Typical as-mined analyses of Gulf Coast lignites show a range of 30 to 50 percent moisture content and 8 to 21 percent ash content (2).

Deposits mined to date have been ideally suited to surface strip mining since they occur in laterally continuous seams, 5 to 15 feet thick, in shallow overburden. Land reclamation conditions are ideal due to the flat to moderately rolling terrain with high to moderate rainfall (2).

At present the three primary methods of burning coal are pulverized coal firing, cyclone firing, and stoker firing. Pulverized coal furnaces are in widest use among utilities and represent the most modern use of this fuel. Cyclone furnaces were introduced because of their ability to burn coals having low ash fusion temperatures and to recover a high percentage of the coal ash as slag instead of allowing it to escape the combustion section and form deposits on boiler tube surfaces. Stoker firing is generally limited to small applications (2).

There are many oil and gas boilers in use today without the capability to burn conventional pulverized coal because of the problems associated with tube slagging. To resolve these problems would generally not be cost effective because of the major modifications and deratings involved. However, many of the problems of retrofitting boilers for coal usage can be alleviated by using a very finely ground coal (3).

Micropulverized coal is a very finely ground (100* less than 44 microns) coal. Conventional pulverized coal particles, due to their larger size tend to impact and deposit on boiler tubes. However, due to their small size, micropulverized coal particles tend to follow flow

streamlines around the boiler tubes. Therefore, there is less boiler tube slagging associated with the burning of micropulverized coal when compared to that from conventionally ground coal (3).

The special properties of lignite coals influence virtually every aspect of direct combustion. Of primary importance are the high moisture contents, low heating values, and alkaline contents common in lignite coals. High sodium content tends to act as a fluxing agent for ash particles which agglomerate and melt on tube surfaces causing slagging or tube fouling. High moisture content necessitates the use of drying before combustion and can cause feed problems (2).

By permanently reducing the inherent moisture content of lignite, the heating value can be increased significantly yielding a higher grade fuel. Also, if the lignite is ground to an ultrafine powder and made into a coal-oil or water slurry, then the potential exists for using it as a retrofit fuel. This end-point use of the lignite is the reason for investigating the properties of the micropulverized/dried product.

Steam drying has been found to be an effective means of permanently drying lignites. Steam drying by the semi-batch Fleissner process operated commercially in Europe from 1927-1960 (3). Research in the areas of steam and hot-water drying are ongoing at the Grand Forks Energy Technology Center. Hot-water drying is a process for the removal of liquid water from high-moisture coal by heating a coal-water slurry under pressure. The tests conducted at Grand Forks have shown that hot-water drying can produce a product with significantly reduced moisture and sodium content thus yielding a product with a higher heating value and a reduced foulage potential (4).

in the first part of this study, the effect of combined pulverization and drying are investigated. The lignite was ground in a fluid energy attrition pulverizer using either superheated steam or air as the working medium.

Parameters used to describe the test results are the changes that occur in equilibrium moisture, ash, volatile matter, and heating value. In addition, the porosity, particle size, density, and surface characteristics of the pulverized lignite are analyzed.

In the second part of the study the results of hot-water drying tests are given. Lignite which had been ultrafine ground using ambient temperature air was heated under high temperature and pressure in an autoclave. The test results are described in terms of the same set of parameters as indicated above for the combined grinding/drying tests.

CHAPTER 2

MICROPULVERIZATION

Micropulverized coal is characterized as having a maximum size of 44 microns. This product can be produced in most types of grinding mills. However, fluid-energy mills are best suited from a machinery wear point of view. Micropulverized coal has a unique characteristic in that the small particle size provides for rapid combustion and less slagging than conventional pulverized coal when used in an oil or gas boiler (5).

The lignite used was pulverized in cooperation with Ergon, Inc. at their Vicksburg facility. A 100 pound per hour fluid-energy mill was used to grind the lignite. In the mill feed coal is ground by its injection into a vertically flowing vortex. The vortex is generated in a manner which provides differential flow velocities within the vortex and a recirculating flow along the containment walls. The vortex is controlled so that it operates in the central region of the containment thus minimizing abrading action along the walls and allowing recirculation. This abrasion along the containment walls is a major problem with many fluid energy mills. Smaller particles exit through a central outlet at the top of the device while (due to the acceleration effects of the vortex) larger particles are forced into the return flow along the walls. The primary grinding mechanism is the impact of particulate material on itself (6).

The tubes in oil and gas boilers are generally spaced closer than those of conventional coal-fired boilers. Because of this increased surface to flow area ratio, the larger ash particles in conventional

pulverized coal will impact the tubes and cause slagging. However as already noted, the very fine ash particles in micropulverized coal or lignite tend to follow the flow streams around the tubes resulting in less slagging and erosion (7).

In addition to the lignite tests described in this paper, other work has been performed on micropulverized coal. Fly ash and combustion/deposition tests were conducted on three different types of micronized coal by Stone and Webster Engineering Corporation in cooperation with Babcock and Wilcox. The three parent coals were all bituminous coals with ash contents ranging from low to high.

The fly ash tests were performed at the Babcock and Wilcox Alliance Research Center. This is a 200,000 BTU/HR test furnace designed to produce fly ash with properties similar to ash from a large utility boiler. Conventional pulverized and micronized grinds of the three test coals were burned in the test furnace. Ceramic probes were inserted at the furnace exit to obtain indications of deposition on superheater tubes and the ash from each coal was collected and analyzed for particle size.

The size of ash particles from micropulverized coal was smaller than that of conventional pulverized coal. The largest ash particle size from micropulverized coal was 14 microns compared to 32 microns top size from conventional pulverized coal. For a given particle distribution the ash from the micropulverized coal was half the size of conventionally ground coal.

Deposits of fly ash on ceramic probes when burning coal ground to micronized and pulverized grinds demonstrated that micronized coal ash particles were small enough to travel with the flue gas around the tubes, while pulverized coal ash particles did not; they impacted and deposited on the tubes.

With data from the ash tests supporting a preliminary conclusion that conversion to micropulverized coal, instead of pulverized coal, will require less boiler derating, scaled-up tests proceeded at B & W's four million BTU/HR Basic Test Combustion Unit (BCTU). The purpose of the scaled-up tests was to investigate the effects of coal particle size on combustion and deposition (5).

The testing was performed using one parent coal sample ground to three degrees of fineness: (1) conventional pulverized grind (70* minus 200 mesh); (2) micropulverized, with a top size of 44 microns; and (3) finer micropulverized with a top size of 15 microns. The mass mean diameters were 9.4 and 8.3 microns for the micropulverized and finer micropulverized products respectively.

The initial testing began using conventional pulverized coal. Five parametric tests were planned for this grind, however the deposition test section became clogged with ash before the tests could be completed. The ash buildup occurred within seven hours after start-up.

The micropulverized coal was then tested in the BCTU for 64 hours continuously. The tubes were blown with compressed air every hour, and photographs were taken before and after soot blowing. The hourly soot blowing provided controllable deposition. Occasionally, the coal would hang up in the feed hopper, suddenly free itself, and then start feeding again. When this happened, the coal feed rate would increase

briefly and then level out in a short period of time. This surge in feed rate would increase the tube surface temperature 100-200°F causing a drastic increase in deposition rates. However after the temperatures leveled out, the air soot blowing adequately cleaned the tubes. The base deposits on the tubes did not grow over the extended test.

As expected, the flames of the conventionally pulverized and micropulverized coals were visibly different. Because the particles of the micronized coals are smaller and the surface where combustion takes place is larger than with conventional grinds, the particles burn in a shorter, brighter flame. The increased brightness is due to the more intense combustion than is typically seen with conventional grinds.

The ash furnace tests support the contention that ash deposition rates will be less for micropulverized coal than for conventional pulverized coal. The results, though not completely conclusive, indicate that equipment modifications and or plant derating will be significantly less in converting to coal if micropulverized coal is used instead of pulverized coal. While these tests were performed with bituminous coals, similar trends would be expected with lignites.

CHAPTER 3

EXPERIMENTAL PROCEDURE

The lignite investigated was obtained from a test pit opened by Mississippi State University on land leased by Phillips Coal Company in Panola County, Mississippi. The lignite was from the Claiborne group and was beneath approximately thirteen feet of overburden. The seam was approximately six feet thick. The samples were placed in barrels lined with polyethylene bags prior to pulverization studies at the Ergon laboratory in Vicksburg. The proximate analysis of the as-mined lignite performed by Phillips Coal Company is shown in Table 1. This analysis is shown on an as-received and equilibrium moisture basis.

The equilibrium moisture value is defined as the in-place coal seam moisture value. This moisture value is obtainable in the laboratory by placing a coal sample in a 97% relative humidity environment for a specified period of time. The laboratory results agree well with the actual bed moisture for higher rank coals, however the equilibrium moisture results produced in the laboratory for the lignite investigated are low compared to the actual bed moisture (8). Even though the actual bed moisture is predicted low for this lignite, it is a reproducible property which is used as a basis of comparison.

The lignite was pulverized at the Ergon laboratory with both steam and air used as pulverizing mediums. Temperatures of 250, 325, 400, and 460 °F were used with steam as the pulverizing medium. The maximum steam temperature that could be obtained due to the limitations of the steam superheater was 460 °F. Temperature runs of 250 and 325 °F were planned for the air medium. However, the lignite began to ignite at

325°F and no further testing was done with the air medium at this temperature. Six samples from each temperature run were obtained and stored in one gallon airtight plastic containers.

TABLE 1. AS-MINED ANALYSES

Proximate Analysis (5)

	As Received	Dry	Equilibrium
Moisture	44.25		40.31
Ash	11 .28	20.23	12.08
Volatile Matter	27.22	48.82	29.14
Fixed Carbon	<u>17.25</u>	<u>30.95</u>	<u>18.47</u>
Total	100.00	100.00	100.00
Sulfur	0.38	0.69	0.41
Gross Calorific Value			
BTU/LB	5622.00	10085.00	6020.00
MAFBTU/LB		12634.00	

MAF=Mineral and Ash Free

Sulfur percentage based on ash weight

The samples from these tests were then analyzed to determine the effects of grinding and drying on the products. The tests conducted were: volatile matter, ash, total moisture, equilibrium moisture, heating value, and density. Porosity tests, particle size distribution, ultimate analyses, and electron microscope photographs were also conducted by: University of North Dakota Energy Research Center, Ergon, Inc., Commercial Testing and Engineering Co., and Mississippi State University respectively. The volatile matter, ash, and total moisture tests were conducted according to ASTM procedures D-3175, D-3174, and D-3173 respectively.

II

The gross heating value of the lignite was determined by burning 0.8 to 1.1 grams of lignite in a Parr adiabatic oxygen bomb calorimeter. Corrections were made for the combustion of the fuse wire and temperature gradients before and after combustion according to ASTM procedure D-2015. Corrections for thermochemical contributions to the heating value were neglected.

The equilibrium moisture content of the coal was determined by two methods. The first method consisted of mixing an approximate 2/3 solid to liquid ratio coal-water slurry and agitating it for thirty minutes. The slurry was then placed in a water bath maintained at 30°C for three hours. The excess water was then filtered from the mixture and the solids were placed in a humidity chamber according to ASTM standards. The second method consisted of taking samples directly from the airtight containers and then placing them into the humidity chamber according to ASTM standard D-1412. This technique was used so that the equilibrium moisture value could be approached from above and below thereby increasing the certainty in a final equilibrium moisture value.

Density tests were performed by measuring the liquid displacement of a weighed lignite sample. Lignite samples were completely dried in an oven and weighed before placement into a flask of known volume. Ethanol was then dispensed into the flask from a burette until a known calibrated volume was obtained and all of the sample had been submerged. Ethanol was used because the micropulverized lignite floated in water. The density measurements reported herein are based on eight samples at a given run condition. This density measurement is defined as an apparent density uncorrected for mineral matter.

Steps were taken to insure that accurate data were obtained. Moisture tests were conducted with six samples from each of the six one gallon containers. Therefore, an average moisture recording at a specified run condition is based on 36 samples. Heating value, ash, and volatile matter data were obtained in a similar manner except three samples were taken from each container yielding an average based on 18 samples at a given run condition.

Porosity tests were conducted using a heats of immersion determining calorimeter. The wetting liquid used was methanol. When an outgassed sample is immersed in a liquid which wets its surface, a rise in temperature of the system results. The increase is proportional to the total surface area wetted and the heat released on wetting a unit area of the adsorbent (9,10). The results obtained by this method should be treated with caution. The real or absolute surface area of the lignite is an uncertain quantity, and experimental errors may run as high as $\pm 50\%$ (1). However, the results obtained using the heats of immersion method can be reproduced with an acceptable standard deviation. Therefore, the results should be considered on a relative basis.

The particle size distribution (based on volume) was determined with a Coulter Counter model Ta II particle analyzer. The particle analyzer determines the number and sizes of particles suspended in a conductive liquid by forcing the suspension through a small aperture and monitoring an electrical current which also passed through the aperture. Electrodes are immersed in the conductive fluid on opposite sides of the aperture. As a particle passes through the aperture, it

changes the resistance between the electrodes. This produces a current pulse having a magnitude proportional to the particle volume. These pulses are electronically scaled and counted.

In addition to the particle size tests, mineral ash analyses were performed by Ergon. A mineral ash analysis is comprised of the determination of all the inorganic material found in the coal ash.

Three ultimate analyses were performed in accordance with ASTM standards. An ultimate analysis is the determination of carbon and hydrogen in the material as found in the gaseous products of its-complete combustion, the determination of sulfur, nitrogen and ash in the material as a whole and the estimation of oxygen by difference. The three samples analyzed were feed-stock, 250 °F air-ground and 460°F steam-ground. These samples were obtained by combining an equal amount of sample from each of the gallon containers at a specific run condition. Only three analyses were performed because it was thought that little change would be seen and the processes tested would represent the band in which the rest of the data would fall.

Electron microscope photographs were taken of samples at each run condition. These samples were mixtures of each of the gallon containers at a given condition as mentioned previously for the ultimate analyses. Photographs were taken at magnifications of 2,000, 5,000, 10,000, and 20,000.

CHAPTER 4

RESULTS OF COMBINED MICROPULVERIZATION AND DRYING TESTS

The equilibrium moisture, proximate and ultimate analyses, and heating value of the feed coal are given in Table 2. The results of the steam and air pulverization tests are shown in Figures 1-7. The mean values and the data uncertainty bands are given on each Figure. These uncertainty bands represent the 95% confidence range (± 2 standard deviations of the data). The data plotted in Figures 1-7 are given in Table 3 and a dry basis proximate analysis of the pulverized lignite is given in Table 4. In addition, electron microscope photographs are shown in Figure 3.

Figure 1 shows the equilibrium moisture versus steam pulverizing temperature. The initial moisture of the feed-stock is about 37% and the minimum moisture value of 13% occurs at a steam temperature of 325°F. Note that essentially all moisture reduction has occurred at or before 250°F and little change occurs after this temperature. Air pulverization at 250°F yields a final equilibrium moisture value of 15%.

Figure 2 shows the dry basis ash versus pulverizing temperature for both steam and air grinding mediums. From Figure 2, there is essentially no change in ash percentage with pulverization medium or temperature. The dry basis volatile matter versus temperature is shown in Figure 3 for the steam and air grinding mediums. As with the ash there is very little change in values for either pulverization process.

TABLE 2. FEED-STOCK ANALYSES

Equilibrium Moisture (\$)	37.49	
	As Received	Dry
Moisture	41 .41	
Ash	15.48	26.42
Volatile Matter	27.45	46.85
Fixed Carbon	<u>15.66</u>	<u>26.73</u>
Total	100.00	100.00
Ultimate Analysis (X)		
Moisture	41 .41	
Carbon	30.33	51 .76
Hydrogen	3.04	5.19
Nitrogen	0.40	0.69
Chlorine	0.02	0.04
Sulfur	0.70	1 .19
Ash	15.48	26.42
Oxygen	<u>8.62</u>	<u>14.70</u>
Total	100.00	100.00
Gross Calorific Value		
BTU/LB	5277.34	9007.23

Figure 4 shows the dry basis higher heating value versus temperature for both steam and air pulverization. Figure 4 shows that the feed-stock has a dry basis heating value of about 9007 BTU/LBM. The heating value increases with pulverization temperature for both the steam and air grinding mediums. The maximum heating value for steam pulverization is 9356 BTU/LBM which occurs at a temperature of 460°F. The air pulverization data show that the heating value is increased from the feed-stock value to 9470 BTU/LBM at the 250°F pulverizing temperature. There is no definite explanation for this increase in heating value since the ash and volatile matter are essentially unaffected by grinding medium temperature. Therefore, it is concluded

that there may not be a definite trend in heating value with pulverizing temperature as Figure 4 might suggest. Due to the inhomogeneous nature of the lignite itself the distinct possibility exists that additional testing could reveal a near constant heating value with process temperature.

Figure 5 shows the porosity versus pulverization temperature for both the air and steam grinding mediums. The feed-stock porosity value is 181 square meters per gram (SQ.-M/G). The minimum porosity value of 85 (SQ.-M/G) occurs at a steam temperature of 325°F. It is interesting to note that after this minimum occurs there is an increase in porosity with temperature. This indicates that at temperatures greater than 325°F substances other than water are being driven off and the pore structure is being opened up making the coal more friable (10). The air pulverization yields a porosity value of 121 (SQ.-M/G) at 250 °F.

The particle size (based on volume) versus temperature is shown in Figure 6 for both air and steam pulverization. The largest particle size is about nine microns for the 250°F air pulverization. Little change occurs for the particle size with steam process temperature. The average particle size for steam pulverization is about seven microns.

The apparent density versus temperature is shown in Figure 7. It should be noted that there is an increase in the density value at 250°F compared to the feed-stock value, however there is little change in values beyond the 250°F temperature. These results coincide with the porosity results mentioned earlier.

The ultimate analysis results for the 250°F air ground and 460°F steam ground materials are given in Table 5. As expected there is little difference between the two run conditions. There is also little difference between these values and the feed-stock ultimate analysis given in Table 2. The mineral analyses of ash results are given in Table 6. As with the ultimate analyses there is little change in ash constituents with pulverization condition.

The electron microscope photographs shown in Figure 8 are at a nominal magnification of 10,000. This magnification was determined to show the most descriptive surface characteristics of the lignite particles. The pictures show that the particles are approximately shaped as spheres. The steam pulverized material shows a general trend of some surface smoothing with increasing steam temperature. The air pulverized material appears to have the roughest surface of the samples analyzed.

TABLE 3
Combined Micropulverization and Drying Test Results

Feed-Stock 250°F A.G. 250°F S.G. 325°F S.G. 400°F S.G. 460°F S.G.

<u>Equilibrium Moisture (?)</u>						
Lower Limit	34.23	12.24	13.05	11 .91	12.80	11.59
Mean	37.49	15.16	16.81	13.52	14.22	13.70
Upper Limit	40.75	18.07	20.56	15.14	15.63	15.80
<u>Dry Basis Heating Value (BTU/LB)</u>						
Lower Limit	8574.16	9166.33	8941 .42	9131.63	9098.29	9065.64
Mean	9007.23	9470.80	9168.26	9275.10	9269.65	9356.29
Upper Limit	9440.30	9775.28	9395.10	9418.56	9441.10	9646.95
<u>Volatile Matter (?)</u>						
Lower Limit	45.32	46.58	45.41	46.20	45.19	45.39
Mean	46.84	47.64	46.63	46.92	45.48	45.75
Upper Limit	48.37	48.71	47.86	47.63	45.76	46.11
<u>Dry Basis Ash (?)</u>						
Lower Limit	24.66	23.53	25.43	25.52	25.81	25.41
Mean	26.63	25.48	26.32	26.38	26.19	25.89
Upper Limit	28.60	27.42	27.22	27.23	26.57	26.37
<u>Porosity (sq-m/g)</u>						
Mean	181.21	121.49	87.93	85.35	110.93	111.63
<u>Particle Size (Microns)</u>						
Mean		8.2	6.38	6.93	7.33	6.79

A.G.=Air Ground
S.G.=Steam Ground

TABLE 4

Dry Basis Proximate (\$)

	250°F A.G.	250°F S.G.	325°F S.G.	400°F S.G.	460°F S.G.
Ash	25.48	26.33	26.48	26.19	25.95
Volatile Matter	47.64	46.73	46.92	45.51	45.75
Fixed Carbon	<u>26.88</u>	<u>26.94</u>	<u>26.60</u>	<u>28.30</u>	<u>28.30</u>
Total	100.00	100.00	100.00	100.00	100.00

Gross Calorific Value

BTU/LB Moisture Free	9470.80	9168.26	9275.10	9269.65	9356.29
BTU/LB MAF	12709.07	12445.04	12615.75	12558.80	12635.10

A.G.=Air Ground

S.G.=Steam Ground

MAF=Moisture and Ash Free

TABLE 5

Ultimate Analysis for Micropul verized Products

	<u>250°F A.G.</u>		<u>460°F S.G.</u>	
	As Received	Dry	As Received	Dry
% Moisture	4.08		4.17	
% Carbon	48.34	50.40	47.82	49.90
% Hydrogen	4.57	4.76	4.27	4.46
% Nitrogen	0.95	0.99	0.66	0.69
% Chlorine	0.02	0.02	0.01	0.01
% Sulfur	0.89	0.93	0.86	0.90
% Ash	25.21	26.28	25.70	26.82
% Oxygen	<u>15.94</u>	<u>16.62</u>	<u>16.51</u>	<u>17.23</u>
Total	100.00	100.00	100.00	100.00

A.G.=Air Ground

S.G.=Steam Ground

TABLE 6

Mineral Analysis of Ash for Micropulver ized Test Samples

Constituent (?)	Feed-Stock	250°F A.G.	250°F S.G.	325°F S.G.	400°F S.G.	460°F S.G.
SiO ₂	50.57	51 .00	51.17	51 .91	51.96	49.42
Al ₂ O ₃	27.00	26.25	27.44	27.02	27.86	26.28
TiO ₂	1 .87	1.93	1.93	1.96	1.98	1.92
Fe ₂ O ₃	5.37	5.60	5.72	5.70	4.90	5.11
CaO	6.32	6.52	6.49	6.49	6.73	6.89
MgO	1 .69	1.72	1.84	1.97	1.67	1.73
K ₂ O	0.53	0.118	0.54	0.52	0.54	0.51
Na ₂ O	0.41	0.34	0.24	0.13	0.38	0.20
SO ₃	3.89	5.82	4.33	4.07	3.68	5.23
P ₂ O ₅	0.09	0.11	0.08	0.09	0.09	0.12
SrO	0.01	0.01	0.01	0.01	0.02	0.01
BaO	0.12	0.16	0.15	0.07	0.13	0.13
Mn ⁺ O ₂	0.06	0.06	0.06	0.06	0.06	0.06
Undetermined	<u>1 .71</u>	<u>0.00</u>	<u>0.00</u>	<u>0.00</u>	<u>0.00</u>	<u>2.39</u>
Total	100.00	100.00	100.00	100.00	100.00	100.00
Base/Acid Ratio	0.18	0.19	0.18	0.18	0.17	0.19

A.G.=Air Ground

S.G.=Steam Ground

Figure 1. EQUILIBRIUM MOISTURE VS. TEMPERATURE FOR
STEAM AND AIR MICROPULVERIZED LIGNITE

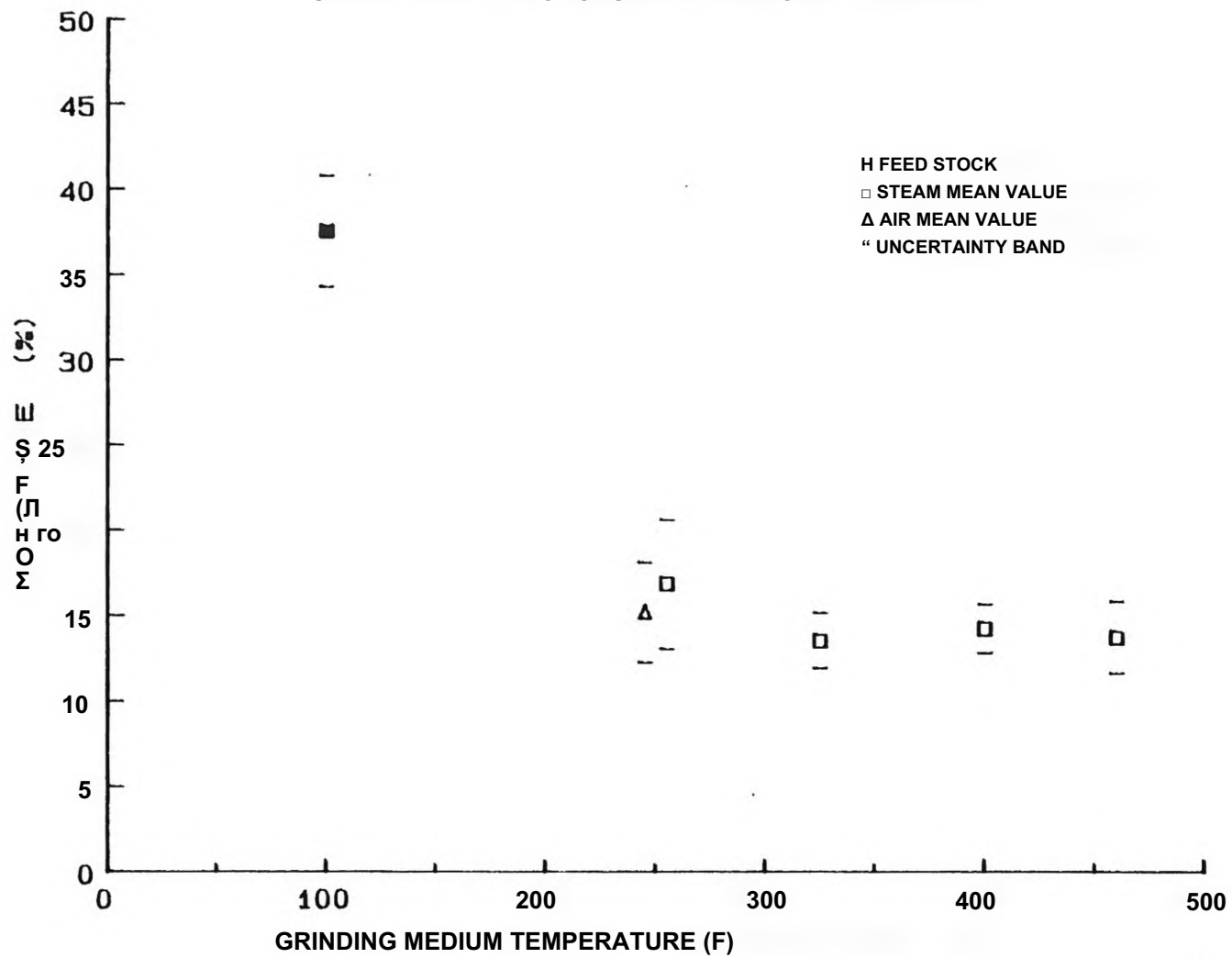


Figure 2. DRY BASIS ASH VS. TEMPERATURE FOR
STEAM AND AIR MICROPULVERIZED LIGNITE

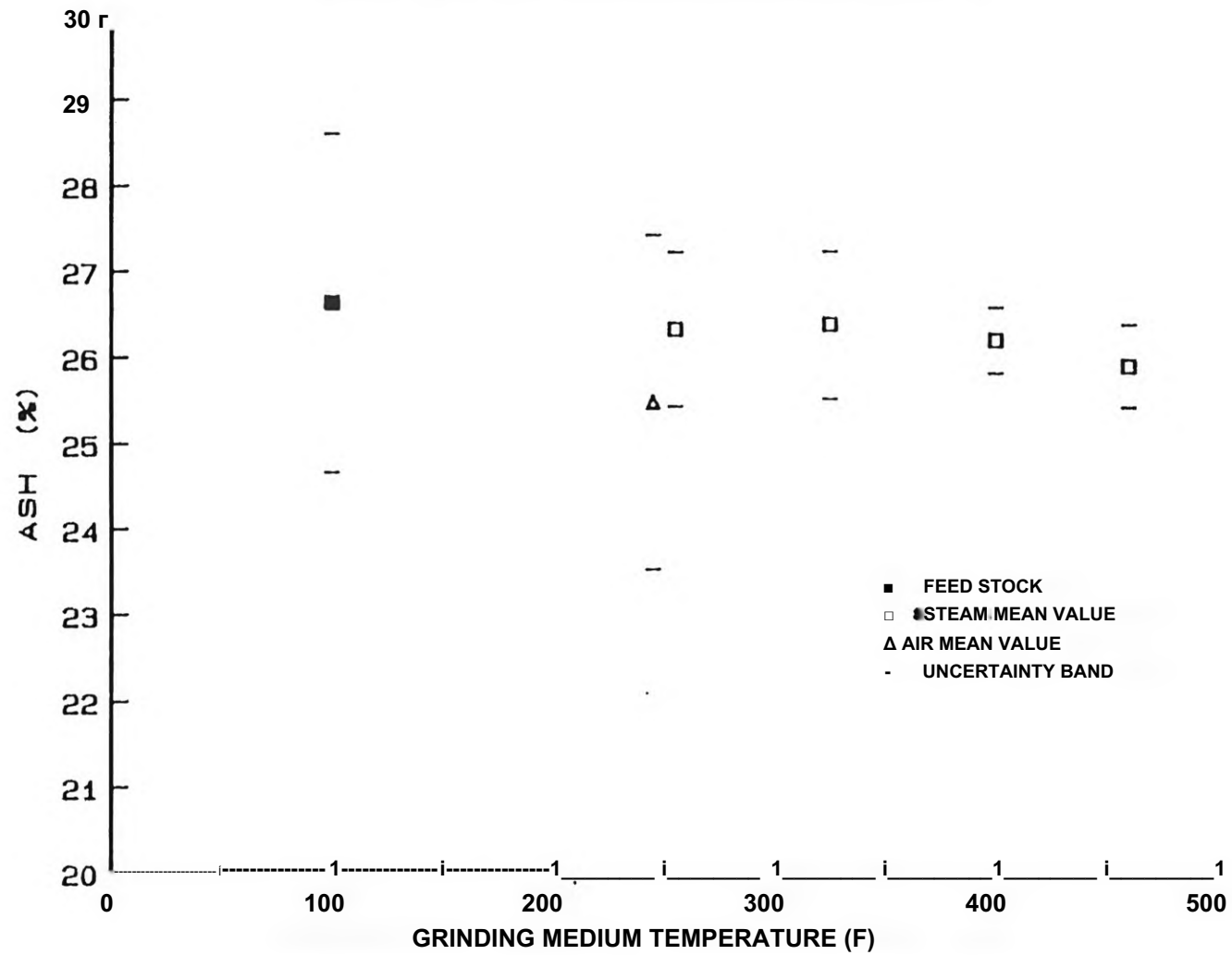


Figure 3. DRY BASIS VOLATILE MATTER VS. TEMPERATURE
FOR STEAM AND AIR MICROPULVERIZED LIGNITE

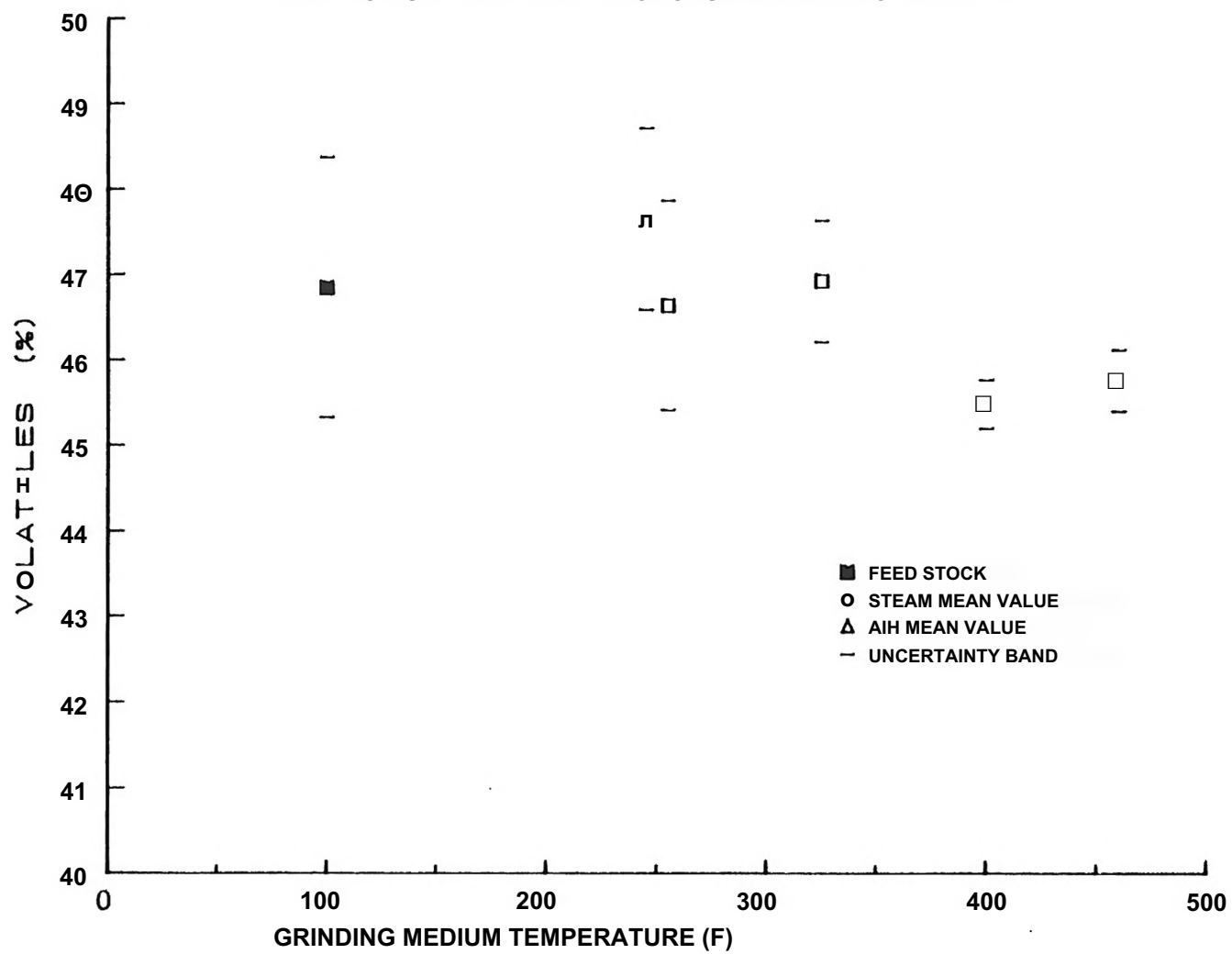


Figure 4. DRY BASIS HEATING VALUE VS. TEMPERATURE FOR
STEAM AND AIR MICROPULVERIZED LIGNITE

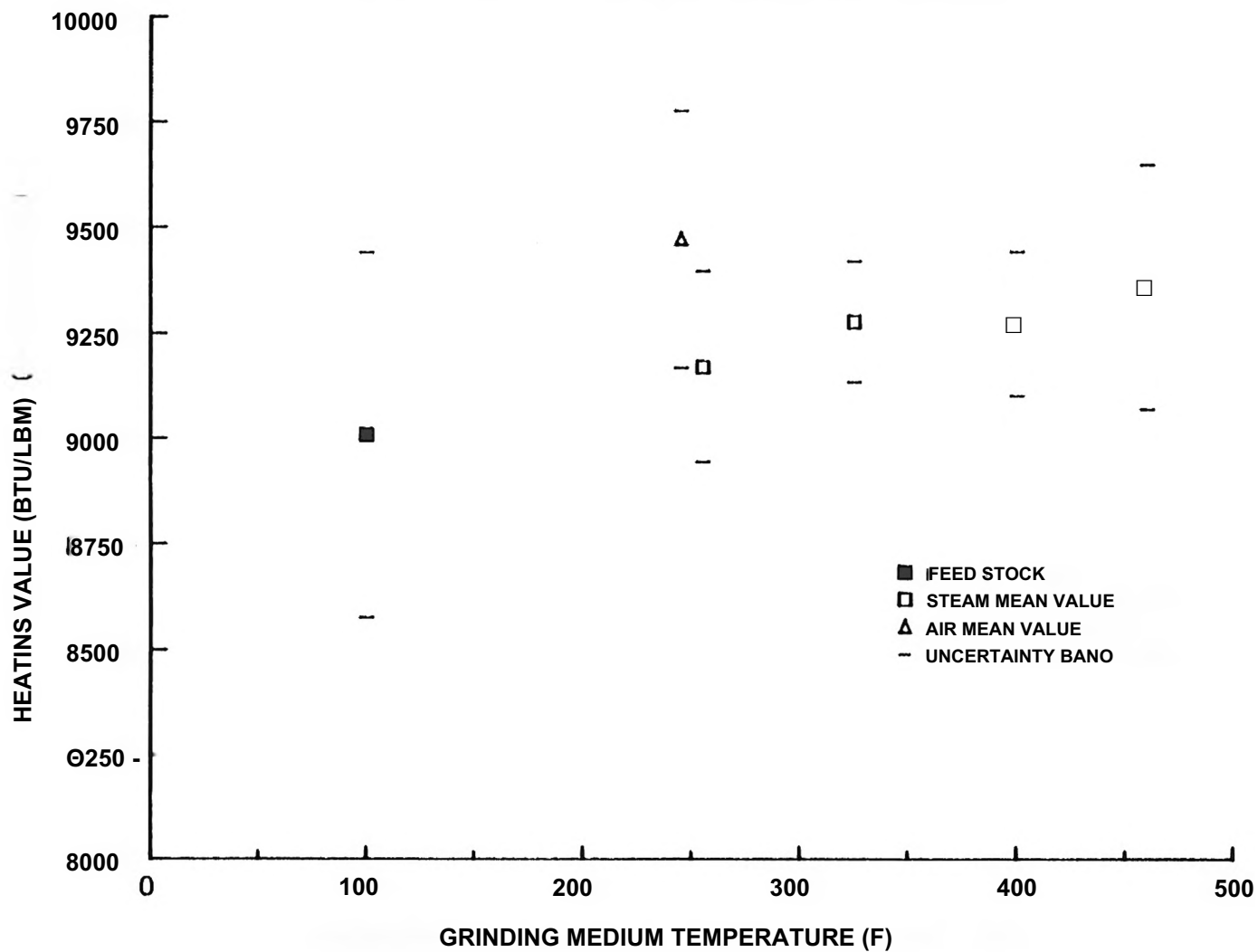


Figure 5.

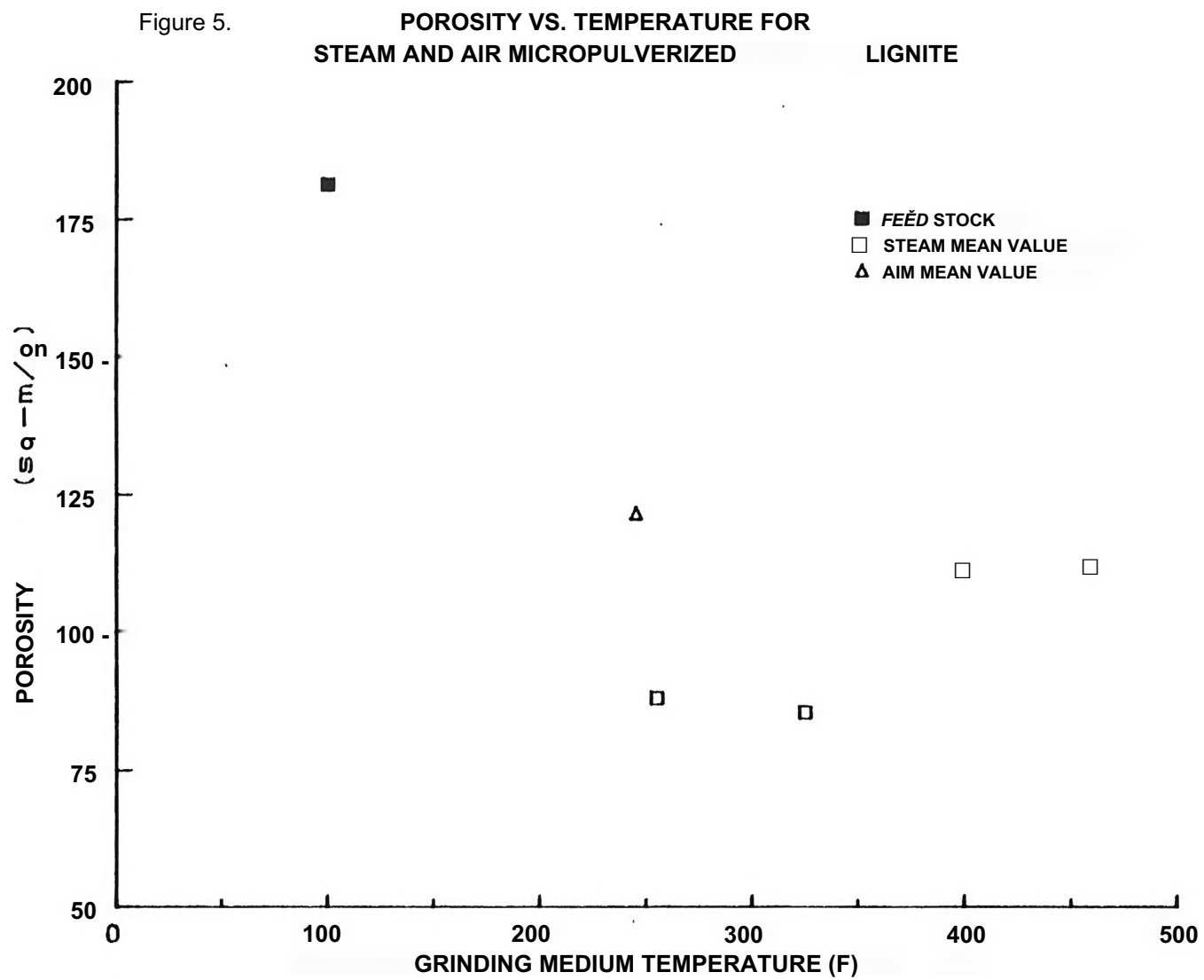
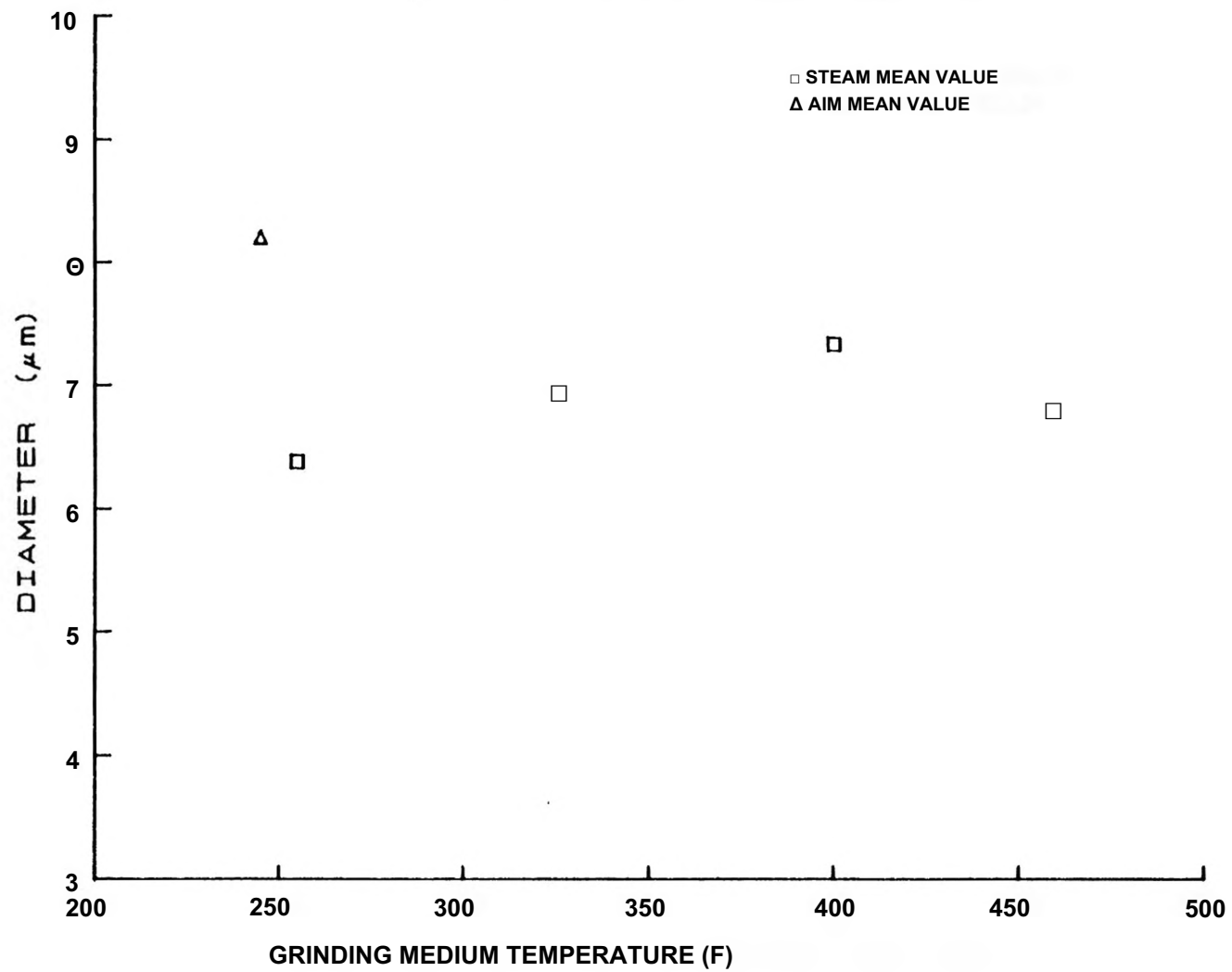


Figure 6. PARTICLE SIZE VS. TEMPERATURE FOR
STEAM AND AIR MICROPULVERIZED LIGNITE



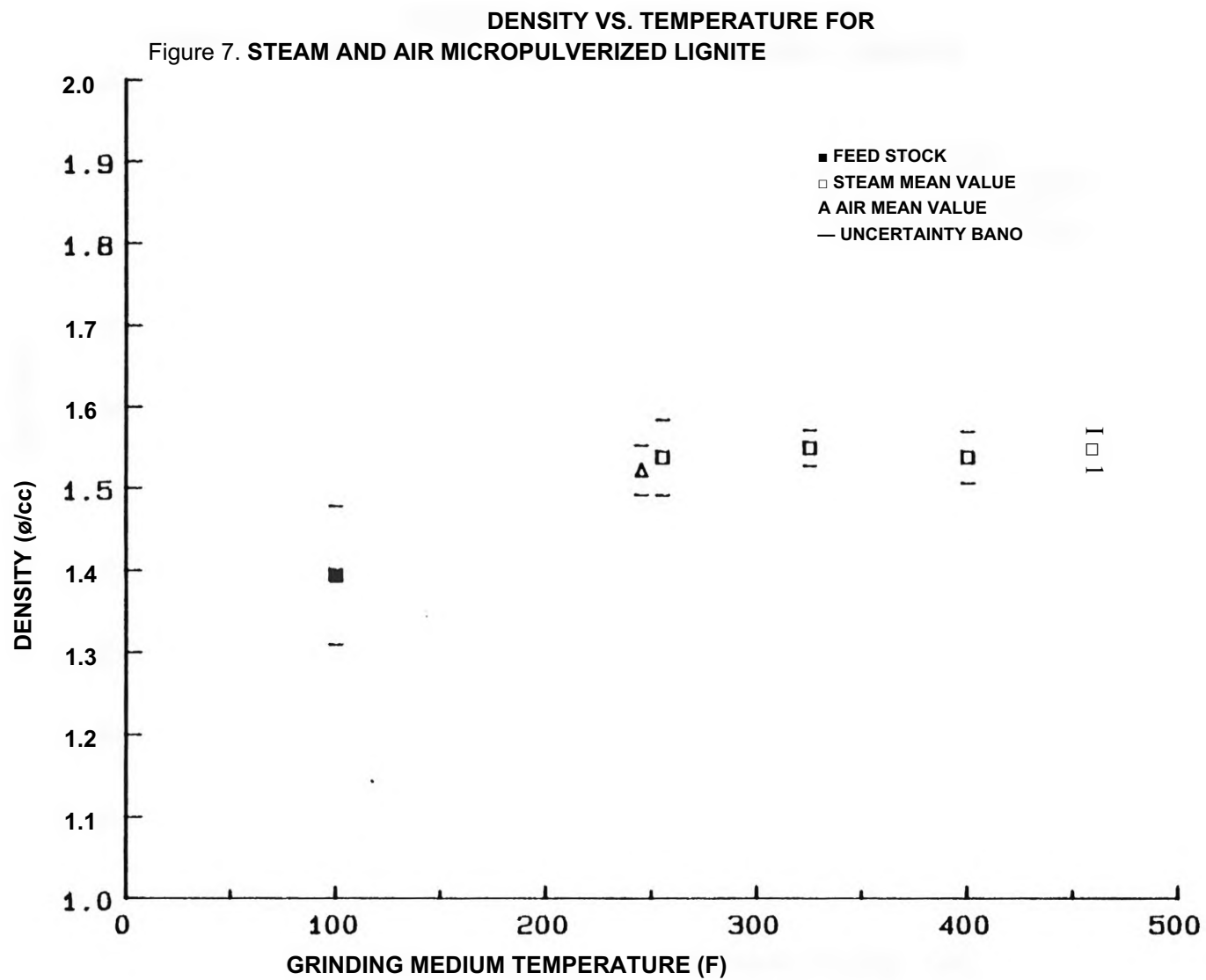
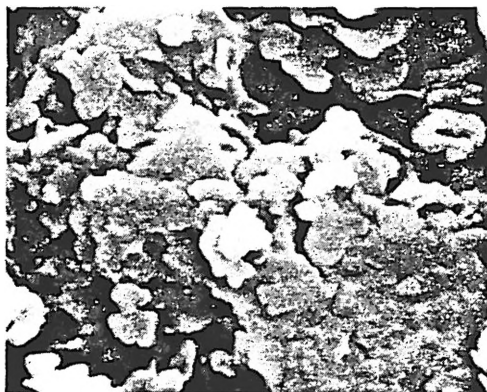
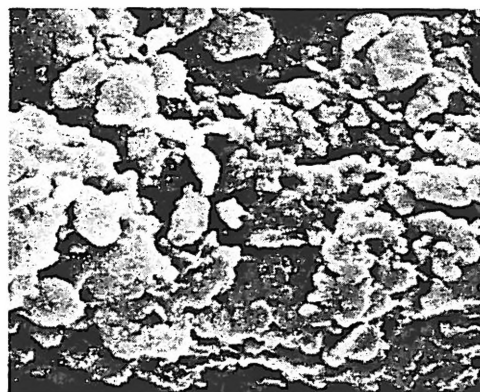


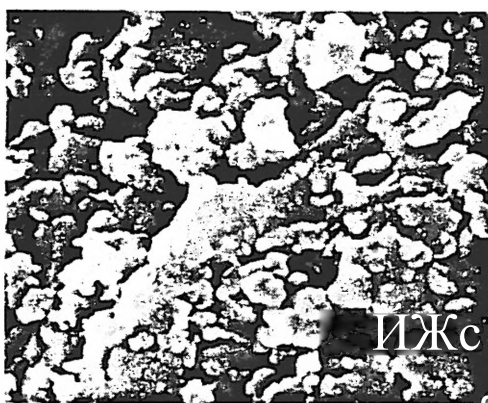
Figure 8. ELECTRON MICROSCOPE PHOTOGRAPHS



250°F Steam Pulverized



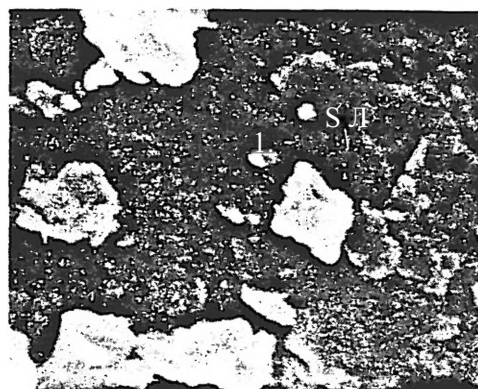
325°F Steam Pulverized



250°F Air Pulverized



400°F Steam Pulverized



460°F Steam Pulverized

CHAPTER 5

RESULTS OF HOT-WATER DRYING TESTS

The lignite used in the hot-water drying tests was from the same area in Panola County, Mississippi as the material used in the combined micropulverization/drying tests. The material was taken from a test pit about 20 feet away from the previous test pit. The lignite was micropulverized by Ergon, Inc. at their Vicksburg, MS fluid energy mill plant. They used ambient temperature air as the grinding medium.

In the hot-water drying process, a 50/50 (by mass) lignite water slurry is heated in an autoclave. Since this is a closed system, the pressure will be at the saturation pressure for the mixture temperature. The slurry is produced with the micropulverized lignite by adding distilled water and agitating. A 150 mi slurry is then poured into the 300 mi autoclave which is then heated to a specified temperature and maintained at that temperature for ten minutes.

Hot-water drying tests were run at 392°F, 464°F, 518°F, 572°F, and 644 °F. The results of the analyses of the products of these tests are shown in Figures 9-15. As in the previous figures, the mean values and the data uncertainty bands are given on each figure. The data plotted in Figures 9-15 are given in Table 7. The ultimate analyses of the products are given in Table 8, and mineral analyses of the ash in the products are given in Table 9. The properties of the feed stock (ambient air ground) material are given in all figures and tables.

Figure 9 shows the equilibrium moisture versus hot-water drying temperature. The initial moisture of the feed-stock is about 30% and the minimum moisture value of 4% occurs at 644°F. It is believed that this permanent moisture reduction results from surface chemistry

modifications and from pore collapse. The temperature breaks down carboxyl groups causing the expulsion of carbon dioxide from the pores. This discharge pushes the water out of the lignite pores (^ and 11).

Figures 10 and 11 show respectively the ash and volatile matter variation versus temperature. There is a decrease in the ash percentage and a slight decrease in volatile percentage.

The dramatic effect of hot-water drying is shown in Figure 12, the heating value change with temperature. The dry basis heating value increases from 10,280 BTU/lb for the feed-stock to 11,501 BTU/lb for the material processed at 64°F. This rise can be attributed to the removal of carboxyl groups which do not add to the heating value. These groups are mixed with the slurry water which is vacuumed out leaving a richer final product with a higher heating value.

Figures 13, 14, and 15 show some unusual trends in this Panola Co., MS lignite. The porosity decreases with drying temperature as is expected. However, with the decarboxylation and pore shrinkage, it would be expected that the particle size would decrease and the density would increase. For this lignite, these two properties show the reverse trends.

Some of this same lignite has been tested at the University of North Dakota Energy Research Center (11). Their tests showed that Panola Co. lignite contains higher quantities than normal of long chain waxes and alkanes. These are apparently being pushed out of the particles during the drying process and then condensing on the particle surfaces during the cooling process. This would decrease the porosity of the particles while increasing their size. The density of the product is also affected by this wax coating over the particles.

Electron microscope photographs of the hot-water dried products are shown in Figure 16. These are at a nominal magnification of 10,000. These pictures show the smoothing effect of the increase in temperature until the highest temperature (644⁰F) is reached. At this temperature the wax on the surface of the particles forms in an irregularly shaped pattern.

TABLE 7
Hot-Water Drying Test Results

	Feed-stock	392°F	464°F	518°F	572°F	644°F
<u>Equilibrium Moisture (\$)</u>						
Lower Limit	27.96	15.79	17.89	4.88	4.30	3.31
Mean	29.99	17.95	19.04	8.09	4.91	3.92
Upper Limit	32.02	20.12	20.19	11 .30	5.52	4.54
<u>Dry Basis Heating Value (BTU/LB)</u>						
Lower Limit	9628	9890	9587	10128	10466	10553
Mean	10280	10355	10524	10754	11083	11501
Upper Limit	10931	10820	11 460	11379	11 700	12449
<u>Volatile Matter (%)</u>						
Lower Limit	50.85	50.72	51.97	48.96	50.69	48.78
Mean	52.27	53.01	52.96	51.69	51.67	50.32
Upper Limit	53.68	55.30	53.95	54.42	52.66	51.86
<u>Dry Basis Ash (%)</u>						
Lower Limit	23.51	18.21	20.77	16.88	18.43	21.34
Mean	24.21	20.88	21 .91	21 .85	21.20	23.69
Upper Limit	24.91	23.56	23.05	26.83	23.96	26.04
<u>Porosity (sq-m/g)</u>						
Mean	130.8	74.4	51.3	41.8	25.9	11.9
<u>Particle Size (Microns)</u>						
Mean	10.4	43.1	43.4	35.3	36.4	81.2
<u>Density (g/cc)</u>						
Lower Limit	1.448	1.470	1.434	1.455	1.397	1.340
Mean	1.428	1.419	1.427	1.406	1.357	1.314
Upper Limit	1.409	1.368	1.421	1.357	1 .317	1.289

TABLE 8
Ultimate Analyses for Hot-Water Dried Products
(All values are on a dry basis)

	Feed-Stock	392°F	644°F
% Carbon	55.21	57.36	60.16
% Hydrogen	5.04	4.77	4.88
% Nitrogen	0.56	0.66	0.67
% Chlorine	0.04	0.04	0.03
% Sulfur	1.15	1.10	1.26
% Ash	23.79	21.43	24.80
% Oxygen (diff.)	<u>14.21</u>	<u>14.64</u>	<u>8.20</u>
Total	100.00	100.00	100.00

TABLE 9
Mineral Analysis of Ash for the Hot-Water Dried Test Samples

Constituent (%)	Feed-Stock	392°F	518°F	644°F
SiO ₂	61.91	62.37	60.64	62.97
Al ₂ O ₃	13.83	13.91	15.20	14.47
TiO ₂	1.46	1.37	1.53	1.49
Fe ₂ O ₃	7.07	7.06	7.42	7.27
CaO	6.67	5.91	6.31	5.40
MgO	1.26	0.95	0.91	1.13
K ₂ O	0.17	0.16	0.18	0.16
Na ₂ O	0.25	0.23	0.08	0.37
SO ₃	7.16	7.86	7.50	6.52
P ₂ O ₅	0.06	0.03	0.06	0.06
SrO	0.00	0.01	0.03	0.02
BaO	0.10	0.10	0.08	0.08
Mn, Pb, Cu	0.06	0.04	0.06	0.06
Undetermined	<u>0.00</u>	<u>0.00</u>	<u>0.00</u>	<u>0.00</u>
Total	100.00	100.00	100.00	100.00

Figure 9. EQUILIBRIUM MOISTURE VS. TEMPERATURE FOR
MICROPULVERIZED. HOT-WATER DRIED LIGNITE

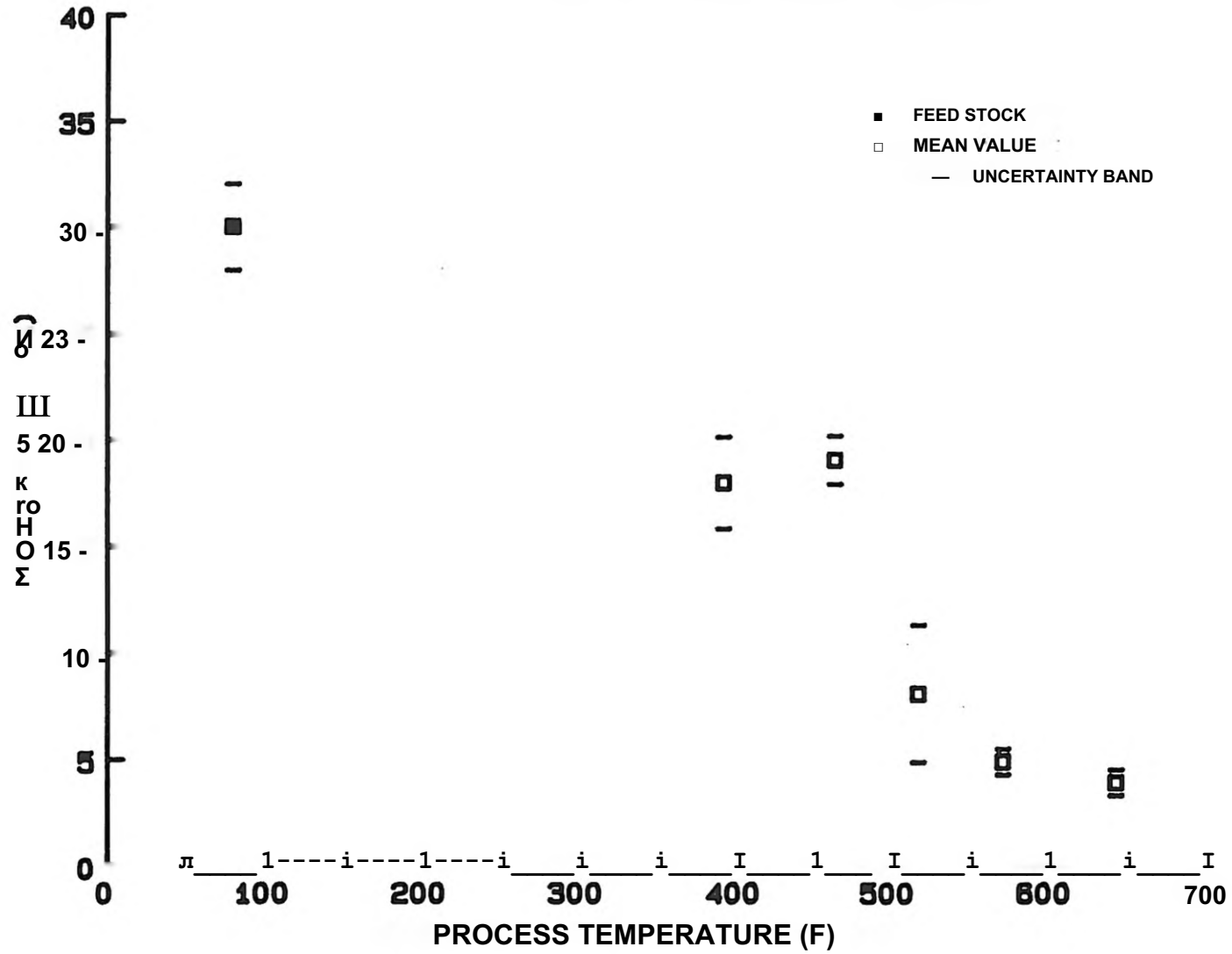


Figure 10. DRY BASIS ASH VS. TEMPERATURE FOR
MICROPULVERIZED, HOT-WATER DRIED LIGNITE

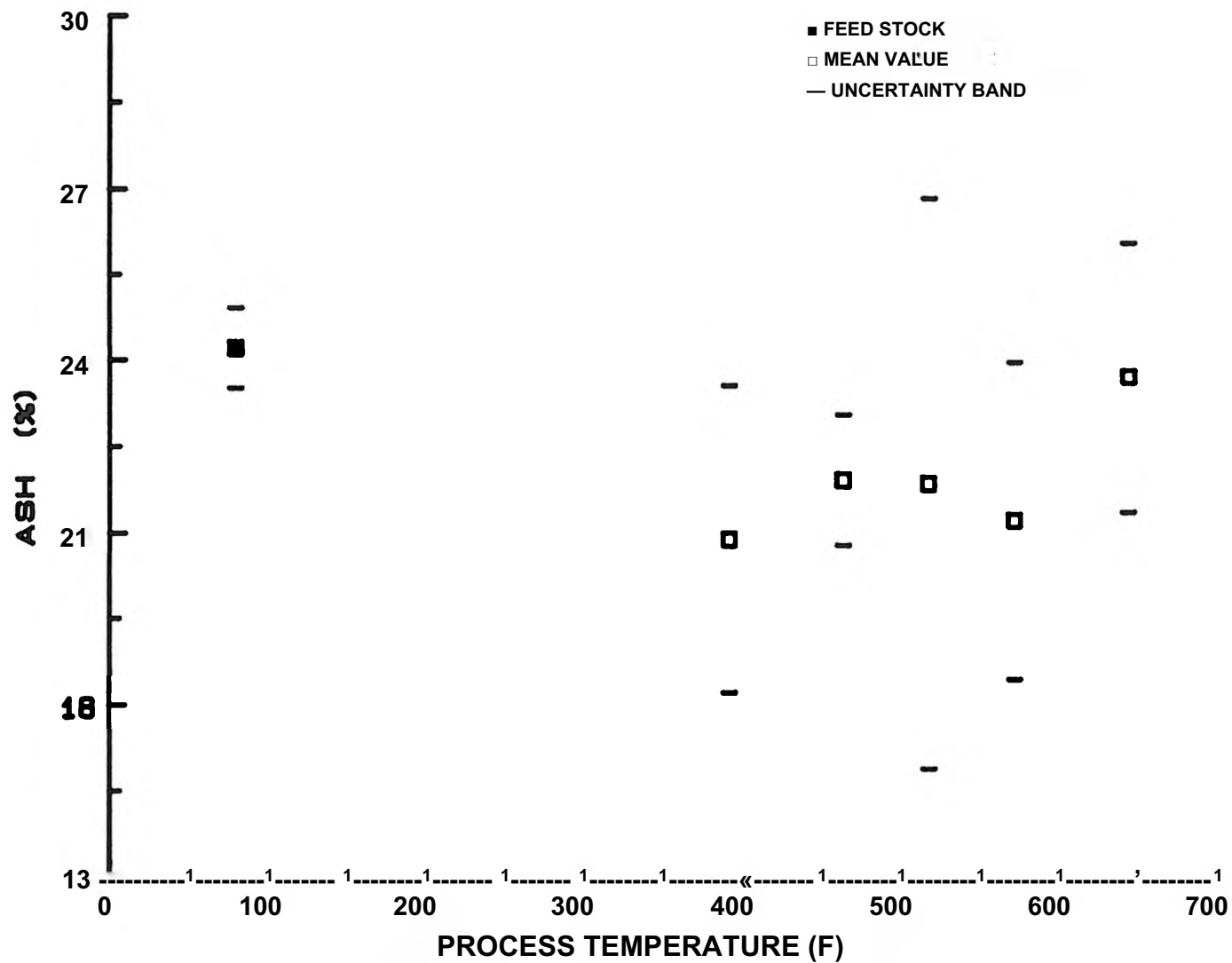


Figure 11. DRY BASIS VOLATILE MATTER VS. TEMPERATURE
FOR MICROPULVERIZED, HOT-WATER DRIED LIGNITE

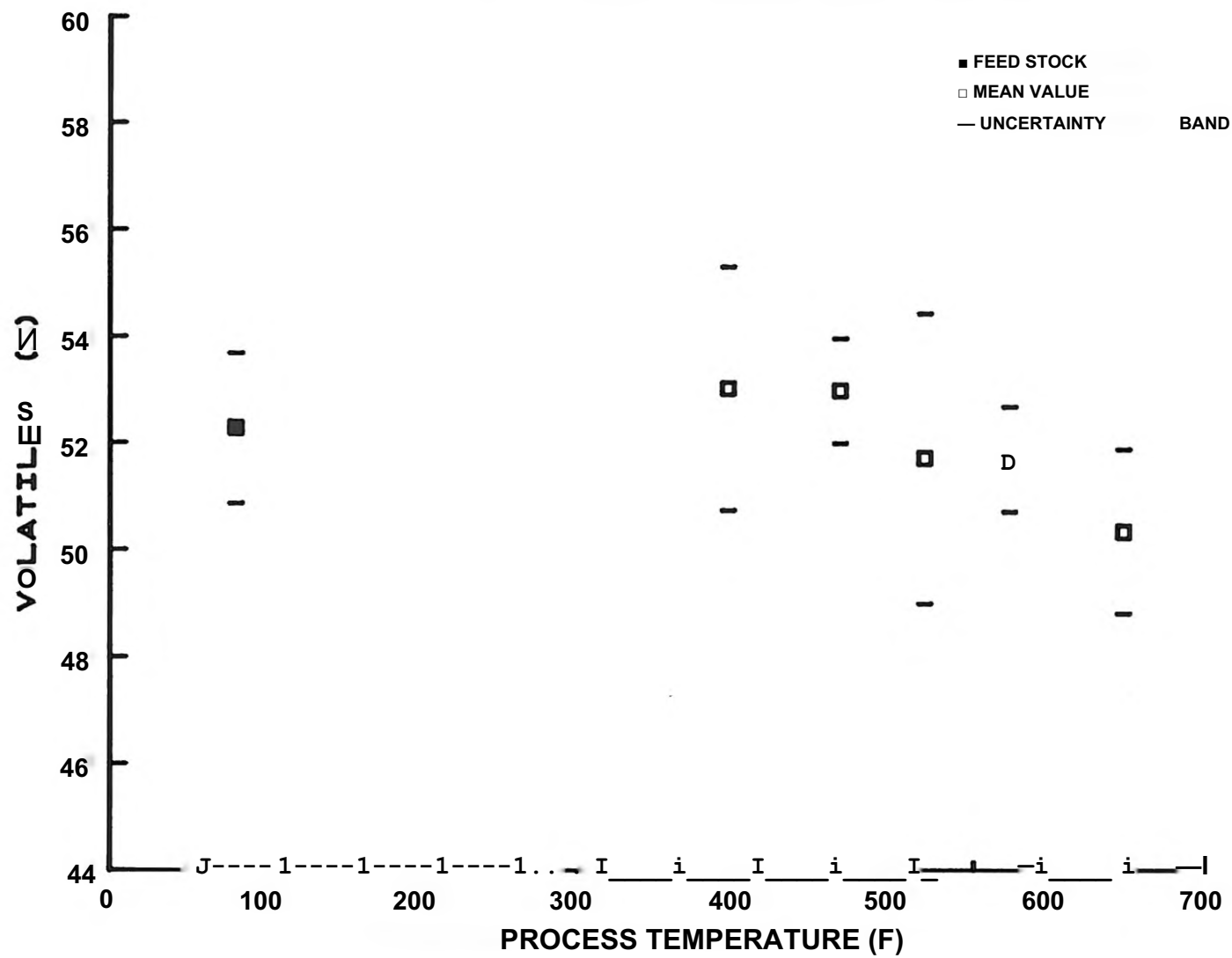


Figure 12. DRY BASIS HEATING VALUE VS. TEMPERATURE FOR
MICROPULVERIZED, HOT-WATER DRIED LIGNITE

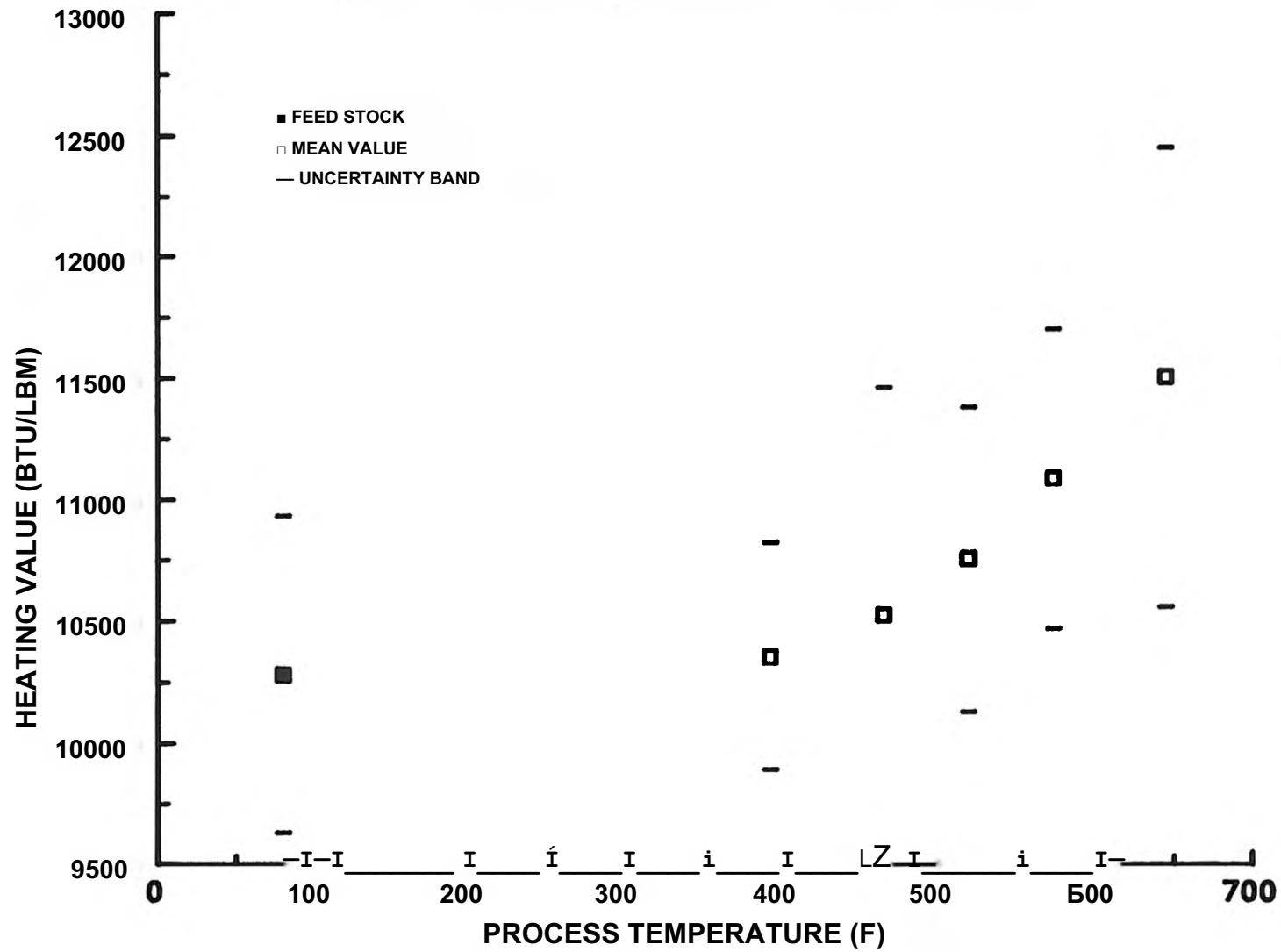


Figure 13.

**POROSITY VS. TEMPERATURE FOR
MICROPULVERIZED HOT-WATER DRIED LIGNITE**

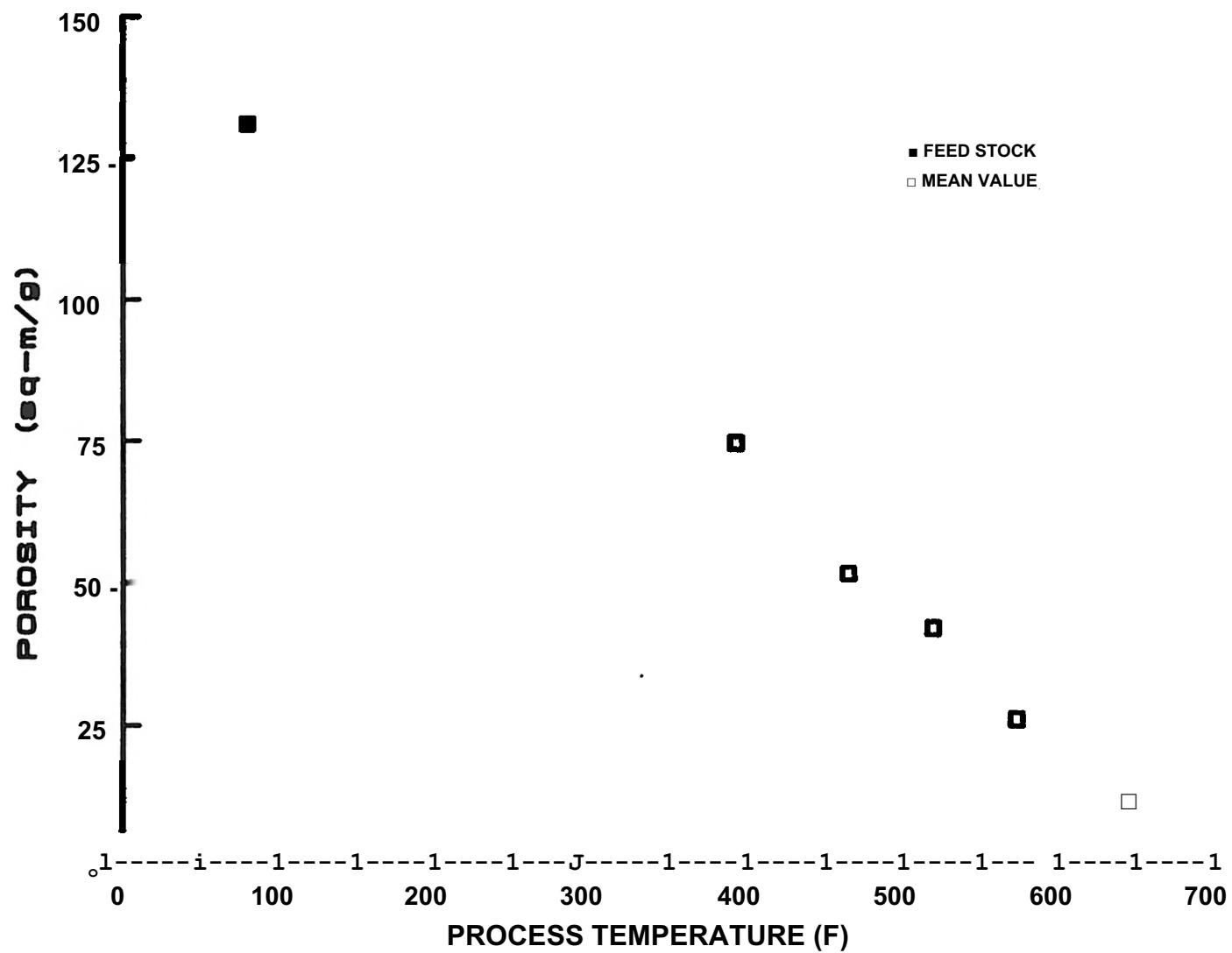


Figure 14.

**PARTICLE SIZE VS. TEMPERATURE FOR
MICROPULVERIZED, HOT-WATER DRIED LIGNITE**

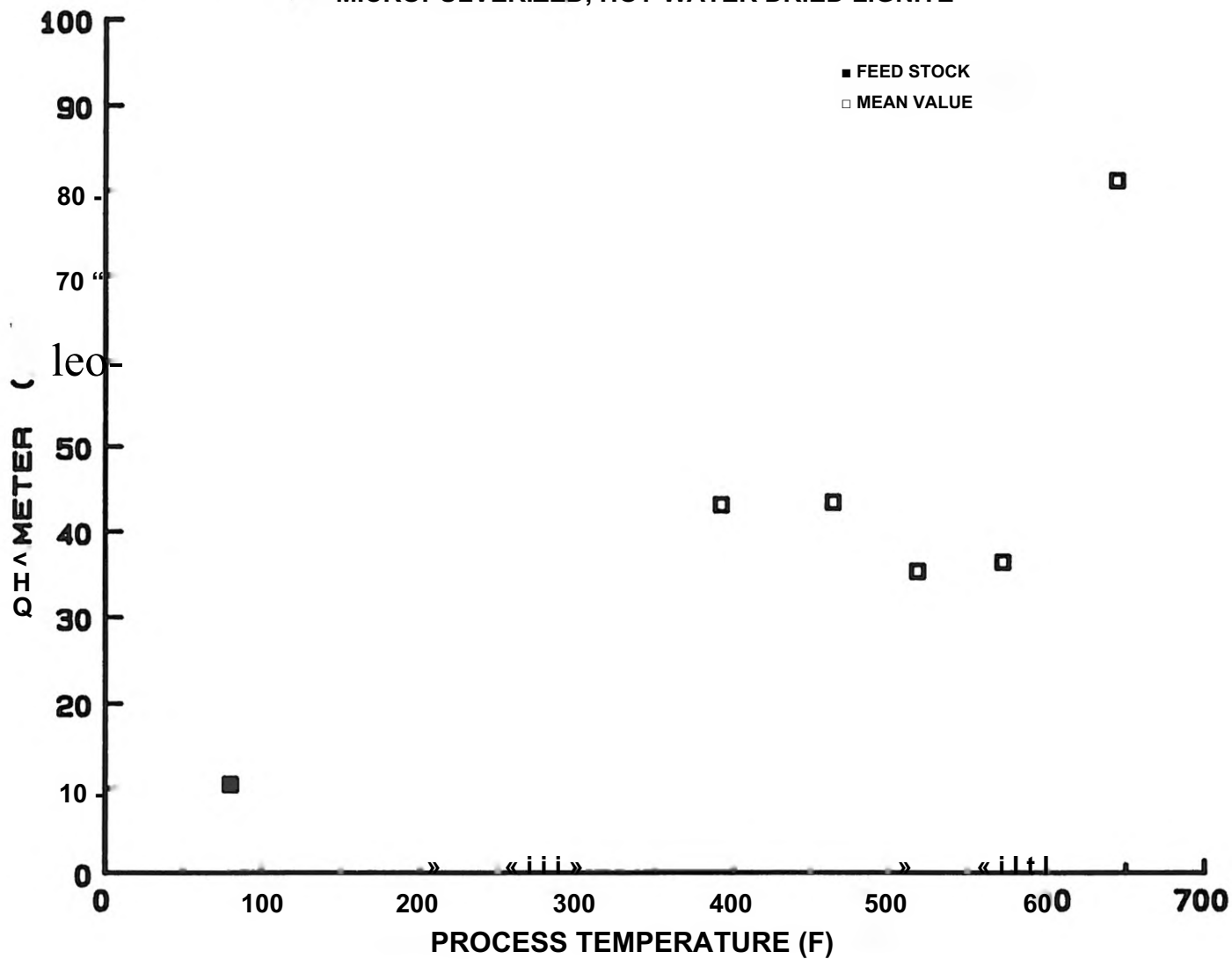


Figure 15.

DENSITY VS. TEMPERATURE FOR MICROPULVERIZED, HOT-WATER DRIED LIGNITE

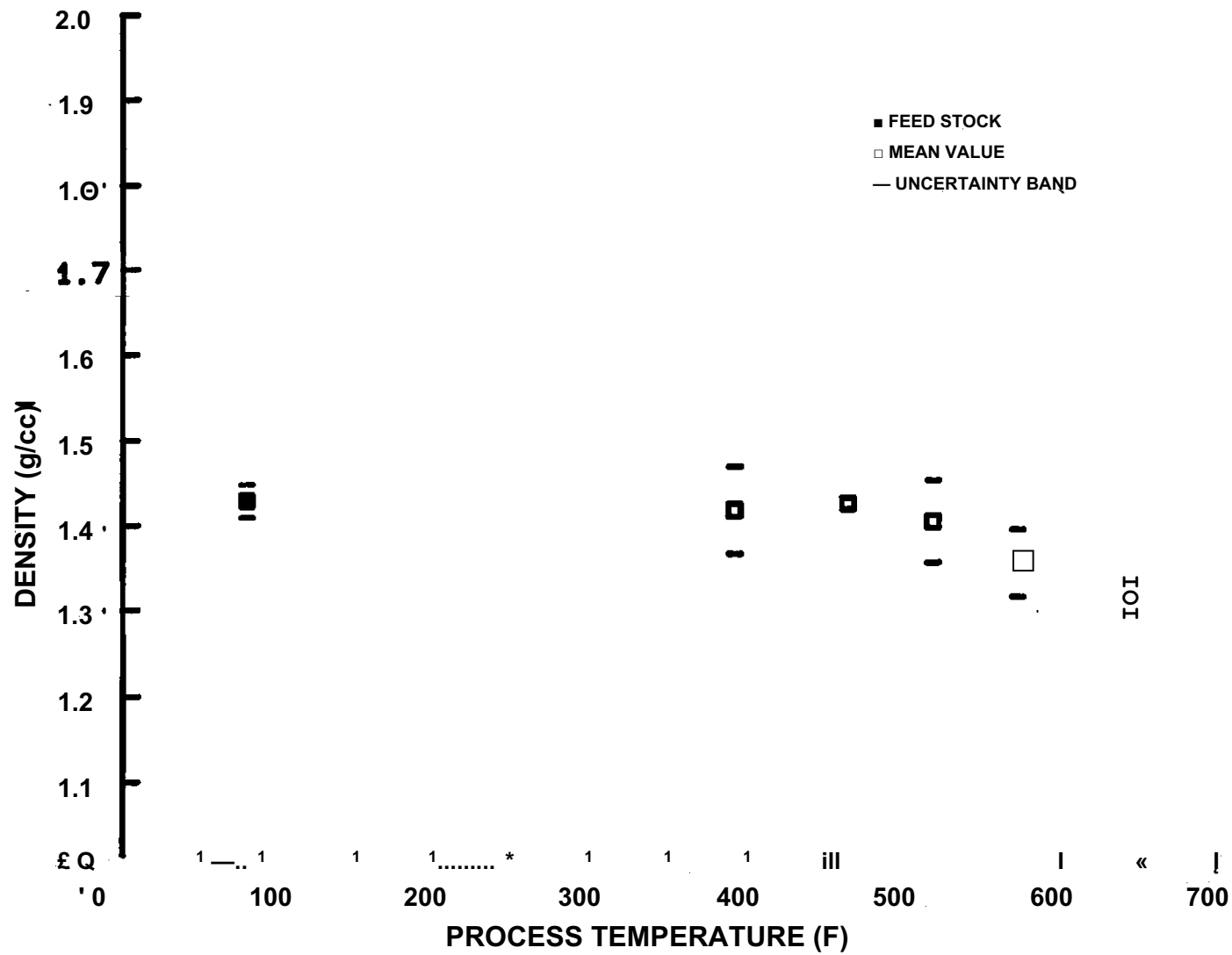
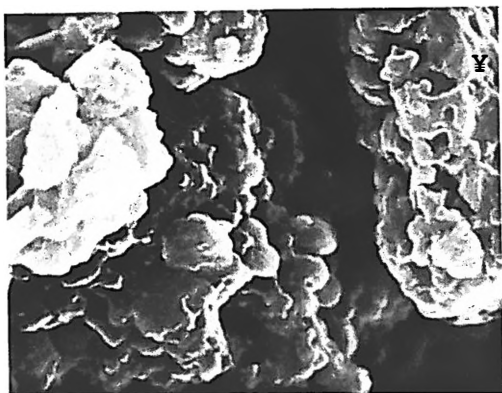
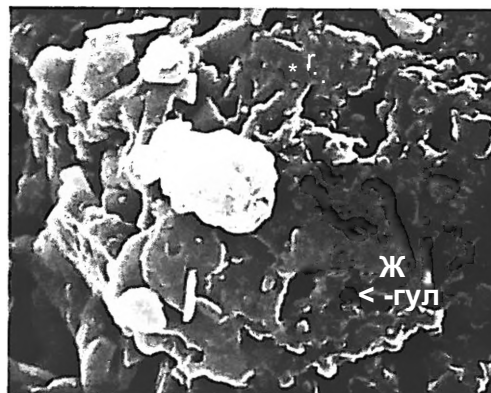


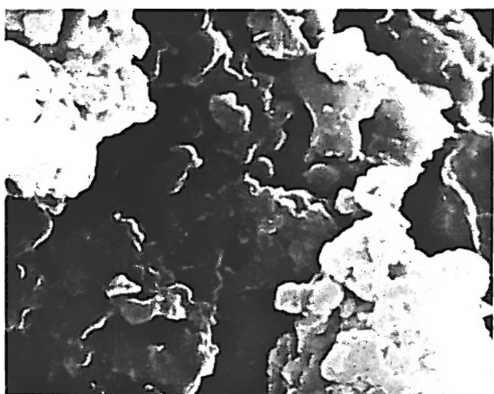
Figure 16. ELECTRON MICROSCOPE PHOTOGRAPHS OF HOT-WATER
DRIED LIGNITE.



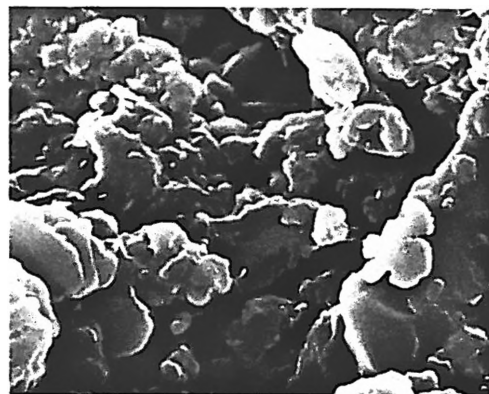
Air-Ground



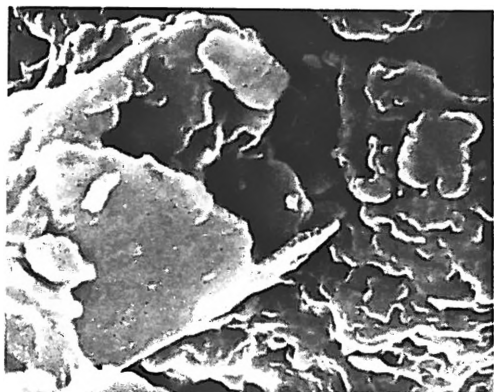
392°F



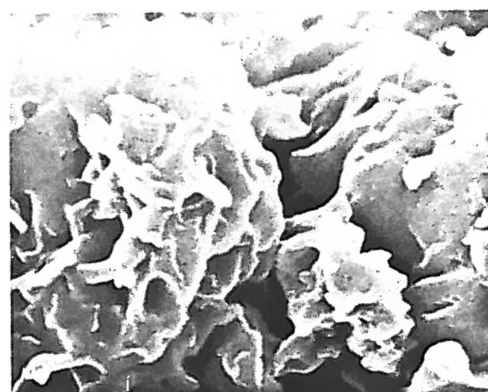
464°F



518°F



572°F



644°F

CHAPTER 6

SUMMARY AND CONCLUSIONS

With 35 billion tons of lignite deposits in the Gulf Coast region coupled with the energy requirements of this area an effective means of burning lignite is needed. Micropulverization of lignite decreases the problems of slagging associated with the burning of conventional grinds in boilers. Micropulverization with a heated grinding medium such as air at 250°F yields a permanently dried product with an equilibrium moisture of 15% or less.

The permanent reduction of equilibrium moisture is of paramount importance in order for lignite to be a successful retrofit fuel for oil and gas boilers. A coal-water slurry is one of the principal methods of transportation and fuel forms under investigation at present. There is interest in slurries because of the benefits in both transportation costs and fuel handling.

The costs involved with transportation would include train shipping charges, special handling procedures, and or special cars for shipping the lignite. Shipping lignite in an as-mined state can cause problems with spontaneous ignition which can be alleviated somewhat by controlling the moisture loss and access to oxygen. There are also problems associated with the shipping of an ultrafine powder as well. Shipping coal with particle sizes as small as discussed in this paper without proper coverings could be disastrous with respect to load loss.

Transportation by slurry pipelines reduce some of the problems associated with solids shipping.. Slurry pipelines eliminate the problems with spontaneous ignition and load loss completely. In

addition to the reduction in fuel handling problems, preliminary work in cooperation with Ergon, Inc. and Phillips Coal Co., has shown that a lignite slurry fuel could be delivered at a cost of about \$3.64 per million BTU. This cost is competitive with current natural gas costs of \$3.00 to \$5.00 per million BTU and fuel oil costs of \$4.00 to \$6.00 per million BTU. Although results are inconclusive, slurry work at Ergon has shown that the micropulverized lignite can form a stable slurry with 55% solids loading.

Combined pulverization and drying of lignite as described in this report yields a product with a permanent reduction in moisture, and a mean particle size (based on volume) of less than ten microns in diameter. Both of these results are essential and significant in the pursuit of a retrofit fuel for gas and oil boilers. In addition, preliminary results show that a slurry fuel can be competitive with natural gas and fuel oil prices.

Hot-water drying results show that a significant reduction in equilibrium moisture and increase in heating value can be accomplished with this technique. The resulting slurry product can potentially be used directly as a fuel after some dewatering. More work is needed in this area to determine the suitability of the slurries formed and to find ways to recover the energy used in the drying process.

Further work in the areas of the cost effectiveness of the micropulverization process, slurry concentration, drying processes, and fuel handling problems is needed.

REFERENCES

1. Berkowitz, N., An Introduction to Coal Technology, Academic Press, New York, 1979.
2. White, D. M., Kaiser, W. R. and Groat, C. G., "Status of Gulf Coast Lignite Activity," Proceedings of the Eleventh Biennial Lignite Symposium, GFETC/IC-82-1, NTIS, San Antonio, TX, June 15-17, 1981.
3. Stanmore, B., Baria, D. N. and Paulson, L. E., "Steam Drying of Lignite: A Review of Processes and Performance," Report No., DOE/GFETC/RI-82/1, Grand Forks Energy Technology Center, Grand Forks, ND, 1982.
4. Baria, D. N., Maas, D. J. and Paulson, L. E., "Steam/Hot-Water Drying of Lignite-Part 1: Activities at the University of North Dakota Hot-Water Drying of Lignite to Produce Slurries," presented at the Twelfth Biennial Lignite Symposium, Grand Forks, ND, May 18-19, 1983.
5. Hartness, J. L. and Koeroghlian, "Dry Micronized Coal Applications: Progress Report on a Utility Station Gas-fired Boiler," presented at the International Coal and Lignite Conference and Exhibition, Houston, TX, November 13-15, 1984.
6. Taylor, D. W., "Comminution of Pulverent Material by Fluid Energy," U. S. Patent No. 4,219,164, August 26, 1980.
7. Bouchillon, C. W., Steele, W. G., Clippard, J. A. and Burnett, J. D., "Evaluation of the Micropulverization, Drying and Beneficiation of Lignite," presented at the Sixth International Coal Utilization Exhibition and Conference, Houston, TX, November 15-17, 1983.
8. Luppens, J. A., "Experience with the Application of the Equilibrium Moisture Test to Lignite," Proceedings of the 2nd Coal Testing Conference, Lexington, KY, February 16-17, 1982.
9. Karr, Clarence, Jr., Analytical Methods for Coal and Coal Products, Volume I, Academic Press, New York, 1978.
10. Tye, Charles, Test Results, Energy Research Center, University of North Dakota, Grand Forks, ND, January 9, 1985.
11. Sears, R. E., Baker, G. G., Maas, D. J., Potas, T. A. and Patel, R., "Production and Combustion of Hot-Water Dried Low-Rank Coal Slurries," presented at the Thirteenth Biennial Lignite Symposium, Bismarck, ND, May 21-23, 1985.