UTILIZATION OF COAL ASSOCIATED MINERALS

QUARTERLY REPORT No. 11

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OBJECTIVE AND SCOPE

The purpose of this research program is to examine the effects of coal mineral materials on coal waste by-product utilization and to investigate new and improved methods for the utilization of waste by-products from the cleaning, combustion and conversion processing of coal. The intermediate objectives include: (1) the examination of the effects of cleaning, gasification and combustion on coal mineral materials; and (2) the changes which occur in the coal wastes as a result of both form and distribution of mineral materials in feed coals in conjunction with the coal treatment effects resulting from coal cleaning or either gasification or combustion.

SUMMARY OF PROGRESS TO DATE

Work has been concentrated in the following areas of research: the effect of carbon in flyash upon the compressive strength of hydraulic cement, lightweight aggregate, mineral wool, strip mine soil revegetation and utilization of fluidized bed bottom ash for structural materials.

Reports pertaining to the above areas of research are included in this quarterly report.

A financial report and milestone chart are presented in Appendix A.

FLUIDIZED BED FLYASH IN PORTLAND CEMENT

A promising approach to the area of sulfur dioxide emissions control at electric utility generating plants and in industrial boilers is the fluidized bed combustion process. A large scale unit is operating at the Rivesville Plant of the Monongahela Power Company near Morgantown, West Virginia.

In this process, limestone is introduced in the fluidized bed to serve as a sulfur dioxide absorbing agent.

Waste by-products from this process are spent limestone bed ash and also flyash.

This paper discusses the effect of certain minerals in the flyash upon its utilization potential, particularly as a partial replacement for portland cement in concrete.

A sample of flyash from the carbon burn-off cell was obtained from the fluidized bed combustion facility at Rivesville, West Virginia. The sample was taken during a steady state run and to insure representativeness, the sample was taken at intervals over a four hour period. During this steady state run, the feed coal size was $-1/2$ inch $+1/4$ inch and the feed limestone size was -6 $+16$ mesh. It is thought that a representative sample of the flyash was obtained during this steady state run. However, different operating conditions may produce flyash having slightly different characteristics.

The chemical analyses of the flyash is presented in Table 1.

From Table 1 it can be noted that the carbon content is 30.24 percent which is larger than the carbon content of a typical flyash. A typical flyash will have a carbon content ranging from 0 to 2 percent.

This flyash was calcined in the laboratory at 800°C in an oxidizing atmosphere in order to reduce the carbon content by oxidation. The carbon content of the calcined flyash was 0.70 percent.

Cubes were made of various mixtures of portland cement and flyash in order to study the effect of the carbon in the flyash upon compressive strength when the flyash is used as a partial replacement for portland cement.

The compressive strength specimens were made in accordance with the American Society for Testing and Materials Designation C109 "Compressive Strength of Hydraulic Cement Mortars". This designation covers determination of the compressive strength of hydraulic cement mortars, using 2-inch cube specimens.

Table 2 presents the composition and compressive strength of the specimens.

Flyash from the fluidized bed combustion process was used as a replacement for portland cement at levels of 10, 20 and 30 percent.

The flyash was used in both the uncalcined state (containing carbon) and the calcined state in which the carbon had been removed by oxidation.

Two different types of portland cement were used. Type I cement is a regular portland cement and Type III cement is a high early strength portland cement.

The specimens were formed and cured in accordance with ASTM methods and compressive strength was tested at times of 24 hours, 3 days, 7 days and 28 days.

The compressive strength data is shown in Table 2 and is also shown graphically in Figures 1 and 2.

Figure 1 shows the data when using Type I cement and Figure 2 shows the data when using Type III cement.

Figure 1 indicates that when using Type I cement stronger compressive strengths are achieved when using calcined flyash than when using uncalcined flyash.

Figure 2 indicates that when using Type III cement the compressive strength data is inconclusive.

There are indications that the carbon in the uncalcined flyash decreases compressive strength. The flyash can be utilized by calcining it in order to remove the carbon by oxidation.

Table 1

Chemical Analysis of Flyash From Fluidized
Bed Combustion Process

SiO_2	30.10
Al_2O_3	13.40
Fe_2O_3	9.07
TiO_2	0.46
CaO	38.70
MgO	1.24
Na	0.23
K	0.90
C	30.24
S	3.85

Table 2

Composition and Compressive Strength of Specimens

<u>Specimen</u>	<u>Portland Cement Type</u>	<u>Flyash</u>	<u>24 Hour</u>	<u>3 Day</u>	<u>7 Day</u>	<u>28 Day</u>
1	I	No	2160	2540	2700	3560
2	I	10% Uncalcined	1150	2100	2680	2800
3	I	20% Uncalcined	930	1090	1830	2150
4	I	30% Uncalcined	380	860	1090	1670
5	I	10% Calcined	1130	1920	2700	3400
6	I	20% Calcined	780	1410	1900	2640
7	I	30% Calcined	340	670	800	1970
8	III	No	2500	3660	3850	4460
9	III	10% Uncalcined	2400	2970	3770	4320
10	III	20% Uncalcined	1460	1910	2160	3050
11	III	30% Uncalcined	840	1270	1490	2290
12	III	10% Calcined	1890	3110	3150	3630
13	III	20% Calcined	1160	1510	1580	3880
14	III	30% Calcined	920	1320	1650	3330

FIGURE I
COMPRESSIVE STRENGTH OF SPECIMENS
TYPE I CEMENT

FIGURE 2
COMPRESSIVE STRENGTH OF SPECIMENS
TYPE III CEMENT

LIGHTWEIGHT AGGREGATE

During the previous quarter, laboratory tests were conducted using coal refuse to produce a lightweight aggregate (LWA) for concrete mixture. The aggregate was made by grinding refuse to -100 mesh and pressing the powder into a 1-inch diameter cylinder, approximately 1-inch high. The cylinders were then fired in an electric furnace and crushed in a jaw crusher to 5/8-inch x 0-inch.

To determine the optimum conditions necessary to form a functional LWA, tests were conducted varying three of the factors in the production procedures. Three different raw materials were selected. Refuse from preparation plants were collected from operations mining the Illinois #6 seam, the Pocahontas #3 seam and the Pittsburgh #8 seam. Four test pellets were formed at a pressure of 4,000 psi and 8,000 psi for each material. Two pellets from each material and pressure were placed in an electric furnace and fired at 1000°F while the other half of the test pellets were fired at 2000°F.

Compressive strength tests were performed on all the samples to determine which material would make the strongest LWA and which factors had the most effect on the performance of the materials. The results of the tests are shown in Table 3.

From the test results, the Illinois #6 formed at 8,000 psi and fired at 2,000°F was found to have the strongest compressive strength. In the following report, tests will be run making comparisons of compressive strength between concrete mixes made with Illinois #6 LWA and concrete mixes using limestone aggregate as a standard. Chemical and elemental analysis were performed during the quarter. The results of these tests will be in the next report.

Firing Temperature	Forming Pressure	Samp. No.	Illinois # 6	Pocahontas #3	Pittsburgh #8
1000°F	4000 PSI	#1	425	75	165
		#2	415	80	230
		Ave	420	78	198
	8000 PSI	#1	450	85	280
		#2	565	80	315
		Ave	508	83	298
2000°F	4000 PSI	#1	1200	295	695
		#2	1735	210	705
		Ave	1468	253	700
	8000 PSI	#1	1695	375	1000
		#2	2520	370	990
		Ave	2108	373	995

Table 3 Comparison of Compressive Strength (PSI) Tests Between Coal Refuse Pellets Formed Under Varying Pressures and Fired Under Varying Temperatures.

MINERAL WOOL

Increased interest in energy conservation in the United States has led to an increased demand for insulating material. One common insulating material in use today is mineral wool produced from blast furnace slag or siliceous limestone (wool rock). The Coal Research Bureau has been involved in research in past years designed to evaluate the use of coal ash from conventional power generation stations as the source material for mineral wool production. These studies have indicated that many coal ashes can be successfully converted into mineral wool of a quality comparable with commercial wool. Based on these studies, it was determined that flyash from a fluidized bed coal combustor had potential as a source material for mineral wool if the carbon content of the ash could be greatly reduced.

The fluidized bed process flyash previously collected on this project has a carbon content of 32 percent. Coal ashes with such a high carbon content are impossible to melt in the required reducing atmosphere at any reasonable temperature. A series of tests were conducted to determine a reasonable method of oxidizing the carbon in the flyash using available laboratory and pilot scale equipment. The details of this series of tests are not presented here because no attempt was made to optimize variables, the goal being only to greatly reduce the carbon content of the ash in a convenient manner using available equipment. It was determined that placing the ash in a cast iron container and exposing it to a temperature of 800°C for 24 hours under oxidizing conditions would produce an ash with a carbon content of 0.2 percent. This ash was used in subsequent testing. Figure 3

is a comparison of the flyash both before and after oxidation. The high carbon content of the flyash before oxidation is obvious from the much darker color of the untreated ash.

Mineral wool is made commercially by heating the source material until it becomes molten and sufficiently fluid for further processing. The choice of melting equipment used is determined in part by the size and nature of the raw materials and by the type of energy source of fuel to be used. In industry, the most common furnace used in mineral wool production is the water-jacketed cupola type. Some manufacturers have used oil or gas reverberatory furnaces or submerged arc resistance furnaces. In the current testing program, a Detroit 5 pound capacity, 17 KVA, carbon arc electric furnace (Figure 4) capable of being tilted around its horizontal axis was used. This type of furnace was utilized because the molten flyash can be poured directly from the hot furnace chamber as is done in industrial practice. During the heating of the flyash, the furnace was gently oscillated to aid in the formation of a more homogeneous melt by mixing the various molten components. Oscillation also increased the efficiency of the furnace as the heat energy was more evenly distributed among the particles in the chamber. Temperature measurements were made using an optical pyrometer.

When the molten flyash became sufficiently fluid, it was poured in a narrow stream (1/4 to 1/2 inch in diameter) into the path of a "blowing-gas" stream. The gas was directed through a nozzle system located at approximately a right angle to the stream. In commercial practice superheated steam is often used as the blowing gas in order to extend the time period during which the molten particles cool after pouring. This practice can result in greater fiber production as compared to the amount formed

when ambient temperature compressed air is used. In the testing program, however, compressed air was used as there would be fewer safety hazards present for the operator who had to work in close proximity to the blowing apparatus. In industrial practice, no workers are near the blowing apparatus.

The nozzle used in these tests was a flat-spray blow-off nozzle with a slot width of 0.023 inches. At 95 psig it delivered approximately 10 standard cubic feet per minute of compressed air. It was noted that this nozzle design is not as efficient for the production of mineral wool fibers as many currently used in the industry, but nozzle design efficiency was not considered within the scope of the testing program and nozzle choice was restricted to commercial off-the-shelf designs. The nozzle was hand held to maintain proper spatial alignment (air-jet perpendicular or at an upward acute angle) to the slag stream with the nozzle orifice approximately 1/4 inch from the stream as shown in Figure 5.

The compressed air or blowing-gas has a shearing action upon the slag stream, breaking it into individual drops and propelling the drops into a collection area. As the drops of molten slag are propelled through the air, friction causes them to develop long "tails". These "tails" are the insulating fibers and any remnants of the original drops are called "shot". If the pouring and blowing conditions have been properly controlled, almost all of each individual drop will be consumed in the formation of the fibers. If conditions are not optimum, shorter, coarser and more brittle fibers will be formed. In addition to these less desirable fibers, large shot particles will occur. Figure 6 is a photograph taken during a blowing operation using molten fluidized bed process flyash. In the left portion of the picture wool fibers may be seen floating to the floor.

Figure 7 is a photograph of a sample of the wool made from fluidized bed process flyash. This wool was produced at a temperature of 1375°C without the addition of any fluxing agents. In appearance, the wool is light grey in color, fine textured and with a low attached shot content. This material appears to be very similar to commercially produced mineral wool. During the last quarter of this project, mineral wool made from fluidized bed process flyash will be more closely compared to commercially produced mineral wool to determine if the wool made from flyash could serve as a direct substitute for the commercial product.

FIGURE 3

FIGURE 4

FIGURE 5

FIGURE 6

FIGURE 7

STRIP MINE SOIL REVEGETATION

Limestone fluidized bed bottom ash (LFBA) would appear to have some potential as a strip mine soil amendment agent because of its high calcium content (37 percent as CaO in the sample collected on this project) and because of its relatively fine particle size. Also LFBA contains various plant nutrients such as Mg, Na and K which could be of importance in the revegetation of strip mine soils.

A thirty gallon sample of strip mine soil was collected from an old abandoned strip mine in northern West Virginia. Although the mine has been inactive for over 10 years, there was little vegetation on the site. The soil sample was collected from an area of the mine in which no vegetation was growing. The pH of the soil as collected was determined to be 3.4. A test of the ability of LFBA to neutralize the acidity of this soil was made (Figure 8). The results of this test indicated that approximately 10 percent by weight LFBA could produce a soil with a pH of 7 (neutral). Complete neutralization of the acidity of strip mine soil is of course not necessary, or in many cases even desirable, for revegetation. Based on these tests, a plant growth experiment was devised.

Fourteen clay flower pots of approximately 1 quart capacity were purchased. These pots were rinsed in distilled water, then soaked in distilled water for 48 hours and finally rinsed again in distilled water to leach out any water soluble materials within the clay. The pots were then allowed to air dry. Kentucky Fescue 31 and Medium Red Clover were selected as the test plants and seeds acquired. These plants were used in this study because both have application in strip mine revegetation,

the size of the mature plant is proportionate to the size of the flower pots and the seeds were readily available locally. The strip mine soil and the sample of LFBA were riffled and mixed so as to produce two samples each of the following proportions of LFBA to strip soil (by weight): 1 percent LFBA, 5 percent LFBA, 10 percent LFBA, and 20 percent LFBA. These samples and control samples of 100 percent LFBA, 100 percent strip mine soil, and 100 percent potting soil were placed in the clay pots so as to produce two series of seven pots with each pair of pots containing the same material. Accepted mixing and sample splitting techniques were used to assure that all pots contained representative aliquots of the parent sample. Each pot was planted with 25 seeds on a grid pattern to avoid variability in growth due to crowding. All pots were placed on a table on the roof of White Hall, W.V.U. as shown in Figure 9. The plants were watered with distilled water during dry periods and the test was monitored via a plant count as shown in Table 4. Basically, most pots containing LFBA showed some improvement over the no growth condition of the strip soil, but fell much below the high growth rate of the potting soil. After approximately one month, plants began to die in all pots apparently due to nutrient deficiency. The test was terminated at 35 days and a new test series began under the same parameters except for the addition of a fertilizer.

Work on this phase of the subject grant during the next quarter will include the monitoring of the second set of plant growth tests and a series of water and acid extraction tests to evaluate potential problems of ground water pollution created by the use of LFBA as a soil amendment agent.

Table 4

Strip Mine Soil Revegetation Test

Days Since Planting	PLANT COUNT													
	Strip Soil		1 Percent LFBA		5 Percent LFBA		10 Percent LFBA		20 Percent LFBA		100 Percent LFBA		Potting Soil	
	C	F	C	F	C	F	C	F	C	F	C	F	C	F
1	0	0	0	0	0	0	0	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0	0	0	0	0	0	8	0
3	0	0	0	0	0	0	0	0	0	0	0	0	21	0
5	0	0	0	0	2	0	0	0	0	0	0	0	21	6
6	0	0	0	0	4	1	0	0	0	0	0	0	21	9
7	0	0	0	0	4	2	0	0	0	0	0	0	21	11
14	0	0	0	9	1	8	0	10	1	3	6	6	21	19
21	0	0	0	5	0	9	0	8	0	3	3	7	20	19
28	0	0	0	3	0	6	0	5	0	0	1	7	17	18
35	0	0	0	2	0	5	0	4	0	0	1	4	14	11

C - Medium Red Clover

F - Kentucky Fescue 31

FIGURE 8

STRIP SOIL pH VERSES LFBA CONTENT

FIGURE 9

UTILIZATION OF FLUIDIZED BED BOTTOM-ASH FOR STRUCTURAL MATERIALS

In the past quarter, bench-scale work was initiated to determine the potential of the fluidized bed bottom ash for utilization in structural products. The initial tests performed during this period were both empirical and systematic in order to optimize parameters prior to standardized procedures.

The parameters investigated included percent bottom ash material, percent aggregate (silica sand), percent water, bulk density of the final product, and compressive strength of the final product. Two additional variables were also examined during this preliminary research: effects of the relative coarseness of the bottom ash on the final product's compressive strength, and the effects of leaching the bottom-ash material on the final product's compressive strength.

The preliminary bench-scale work involved mixing variable proportions of bottom ash, aggregate, and water, forming the wet mix into a solid rectangular product using a mold, and allowing the material to moist-cure for a 7-day period. The solid rectangular product measuring 102 mm x 51 mm x 40 mm was then weighed to calculate bulk density and finally subjected to controlled breakage to determine compressive strength. In Table 5, the parameters and results of each preliminary test are delineated.

As can be seen clearly in the results of the compressive strength tests listed in the table, the sample block made from pulverized, non-leached bottom ash was superior to all others. Based on these preliminary results, tests in the next quarter of work will utilize mixture No. 6 to examine the effects of prolonged curing and the incorporation of flyash as an aggregate.

Table 5

Results of Preliminary Tests

Utilization of Fluidized Bed Bottom Ash in Structural Materials

Test Block #	Composition ¹			Bottom Ash Treatment			Results	
	% Bottom Ash	% Aggregate	% Water	Coarse ²	Fine ³	Leached ⁴	Bulk Density (grams/Cm ³)	Compressive Strength ₂ (pounds/in ²)
1	100.0	0	20.0	yes	no	yes	1.79	352
2	100.0	0	20.0	yes	no	yes	1.71	228
3	100.0	0	25.0	yes	no	yes ⁵	1.60	366
4	75.0	25.0	25.0	no	yes	yes ⁵	1.69	796
5	50.0	50.0 ⁶	25.0	yes	yes	yes	1.76	454
6	75.0	25.0	25.0	no	yes	no	1.75	1501

1. Weight basis
2. Coarse Ground to - 14 mesh
3. Pulverized to - 60 mesh
4. Bottom ash leached in H₂O at 100°
5. Sample oven-dried at 80°C for 24 hrs.
6. Aggregate Composition = 50% Silica + 50% Coarse Bottom Ash

UTILIZATION OF COAL ASSOCIATED MINERALS

FINANCIAL REPORT

11TH QUARTER, APRIL 1, 1980 - JUNE 30, 1980

Expenditures This Quarter

Personal Services	\$17,759.53
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Equipment, R & A	1.76
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Current Expense

Benefits	3,756.20
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Overhead	9,994.18
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Supplies	4,463.79
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Travel	1,405.11
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TOTAL EXPENDITURES 11TH QUARTER	\$37,380.62
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TOTAL EXPENDITURES TO DATE	\$334,762.61
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TOTAL CONTRACT AWARD TO 9/24/80	378,000.00
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CONTRACT BALANCE	\$43,237.39
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UTILIZATION OF COAL ASSOCIATED MINERALS

West Virginia University

Facet	Task	<u>FIRST YEAR</u> (quarters)				<u>SECOND YEAR</u> (quarters)				<u>THIRD YEAR</u> (quarters)			
		1st	2nd	3rd	4th	1st	2nd	3rd	4th	1st	2nd	3rd	4th
I	1												
	2												
	3												
II	4												
	5												
	6												
	Final Report Preparation												

Work Schedule