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DEVELOPMENT OF POTENTIAL USES FOR THE RESIDUE FROM FLUIDIZED BED COMBUSTION PROCESSES

Quarterly Technical Progress Report, June—August 1980

By
L. John Minnick

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Work Performed Under Contract No. AC21-77ET10415

L. John Minnick
Plymouth Meeting, Pennsylvania

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Prepared By
L. John Minnick

For
U.S. DEPARTMENT OF ENERGY
Morgantown Energy Technology Center
Morgantown, WV 26505
Under Contract No. DE-AC21-77ET10415

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OBJECTIVE OF WORK

This agreement between the Prime Contractor, L. John Minnick, and the US Department of Energy (DOE) provides for an extensive research program to evaluate the solid wastes from fluidized bed combustion for potential commercial uses. In addition, the investigation will explore methods by which the material can be safely disposed of should commercial applications be slow in developing, or not of sufficient magnitude to account for utilization of all of the waste materials.

BACKGROUND AND SCOPE OF WORK

Previous research under this contract has clearly shown that spent bed materials from atmospheric fluidized bed (AFB) boilers are potentially useful in a number of commercial applications. Extensive laboratory studies to date have confirmed the fact that AFB residue materials from different sources do contain a substantial component of reactive lime which can, after proper processing, be concentrated and effectively separated from the residue. Furthermore, these spent bed materials also exhibit unusual cementitious properties when used as received or after processing in certain construction-related compositions.

Major emphasis in this study has been directed toward evaluation of a number of different sources of AFB residue in the following areas of research:

- Characterization of AFB by-product materials from several different boilers under various operating conditions.

- Determination of the most effective means of beneficiation or separation of these materials, including preliminary conditioning or hydration as an initial step prior to use.

- Laboratory evaluation of the feasibility for use of AFB residue in such applications as a lime substitute, neutralization of acidic trade wastes, flue gas desulfurization, and the stabilization of highly plastic soils.

- Performance testing of commercial applications involving some cementitious activity, such as in road base compositions, the production of structural materials, placement of structural fill material, and the dewatering and stabilization of sludges and slurries.

- Conduct small-scale field demonstrations to confirm laboratory performance of AFB residues in several of the most promising commercial applications.

The most recent trend of the work has been in the direction of activities related to demonstration projects.

SUMMARY OF ACTIVITIES DURING QUARTER

BENEFICIATION STUDIES

- Several samples of AFB residue have been separated by means of a mineral cyclone.
- Strengths of mixes containing a fine quartz silica are compared with those containing pulverized coal fly ash.

APPLICATION STUDIES

- Road base mixes prepared with currently produced AFB residue are compared with previous mixes.
- More briquetted aggregate has been produced and evaluated.
- Eight thousand pounds of residue are ready for producing masonry units.
- AFB residue is evaluated in an expansive grout.
- A further evaluation has been made of the road base test pads in Ohio.
- New laboratory test pads have been made to evaluate new formulations for dimensional stability.

BENEFICIATION STUDIES

SEPARATION OF AFB RESIDUE BY MINERAL CYCLONE

Several Alexandria AFB residue samples were processed in a Black Clawson 5" Cellu-Clone in an effort to concentrate the lime. This apparatus separates a wet sample of low solids concentration into two fractions on the basis of the specific gravity and particle size of the solids. The following samples were processed:

Sample No.	Sample Description
1190	AFB residue (Alexandria) tailings from Deister table separation
1191	As received AFB residue (Alexandria)
1192	Milled AFB residue (Alexandria)

Two fractions result from each sample. Data from the separation runs are shown in Table I.

Chemical analyses of the separated fractions are currently being performed along with an analysis of an unprocessed sample. These analyses should show if a lime rich fraction is being obtained.

PROPERTIES OF COMPOSITIONS CONTAINING AFB RESIDUE, SILICA, AND AGGREGATE

Compressive strength data were obtained from road base compositions in which a finely ground quartz silica was substituted for pulverized coal fly ash. Table II provides the gradations of the silica and crushed limestone used in the mixes. The silica has trace amounts of impurities and contains no aluminum oxide.

Four compositions were compared. The formulations for the mixes, densities, and compressive strengths are shown in Table III.

Mixes A, B, and D were prepared with AFB residue from the same source. The residue used in Mix A was wet slurried for 30 days prior to use. The AFB residue in Mixes B and D was hydrated just prior to use. Mix C was prepared to evaluate the potential for the silica to react with a commercially hydrated lime.

The results indicate that the quartz silica is pozzolanic in that it reacts with lime to form cementitious products. Mix A, however, failed to develop sufficient mechanical stability to permit handling during the 56 day moist cure at 73°F.

Mixes B and D provided for a direct comparison of fly ash and quartz mixes and both developed similar compressive strength.

APPLICATION STUDIES

REPRODUCIBILITY OF AFB RESIDUE COMPOSITIONS

Concern over the potential variability of the properties of AFB residues as a function of the conditions under which they are produced led to a limited program to check the reproducibility of previously obtained data. The compressive strengths of road base compositions utilizing AFB residue that is currently being produced were compared with those obtained in previous evaluations. Table IV provides the mix formulations and compressive strength results. The AFB residue was hydrated prior to use.

In May 1977, 27 mixes were evaluated. The compressive strengths of comparable formulations that were similarly cured are summarized in Table V.

TABLE 1

DATA FROM SEPARATION OF AFB RESIDUE

IN 5" CELLU-CLONE CLEANER

Run No.	Description of AFB Residue Test Sample	Total Weight of Test Sample (lbs)	Weight of Separated Fractions (lbs)	
			Coarse	Fine
1190	Tailings from Deister table processing	6	2.8	3.2
1191	As received	6	4.1	1.9
1192	Milled	6	4.1	1.9

TABLE II

GRADATION OF LIMESTONE AND FINELY GROUND QUARTZ SILICA

Sieve Size	Percent Passing (By Weight) Limestone	Silica*
3/4"	100	
3/8"	56.8	
# 4 (4.76 mm)	26.0	
# 8 (2.38 mm)	16.1	
# 16 (1.18 mm)	10.4	
#100 (0.15 mm)	4.1	
#200 (75 μm)		100
#275 (53 μm)		97
#325 (45 μm)		93

*Known as Newport 325 mesh silica, a product of Pennsylvania Glass Sand Corporation, Pittsburgh, Pennsylvania

TABLE III

MIX FORMULAS AND PROPERTIES OF COMPOSITIONS CONTAINING SILICA IN PLACE OF PULVERIZED COAL FLY ASH

Mix Designation	Mix Formulas (% by Weight)					Compressive Strength[1] (psi)			Dry Density[2] (pcf)	Moisture Content[2] (%)
	Limestone	AFB Residue	Silica	Lime	Fly Ash	7 Day	28 Day	56 Day		
A[3]	60	30	10	-	-	(Failed in handling)			128.5	12.4
B[4]	60	30	10	-	-	210	530	690	130.3	7.9
C	84	-	12	4	-	250	300	-	146.1	7.1
D[4]	60	30	-	-	10	235	520	-	124.9	9.3

[1]All specimens were cured in moist condition at 73°F except for Mix C (7 day) which was at 100°F.

[2]For specimens at time of molding.

[3]AFB residue for this mix was wet slurried for 30 days prior to use.

[4]AFB residue for these mixes was hydrated just prior to use.

TABLE IV

COMPRESSIVE STRENGTHS OF AFB ROAD BASE COMPOSITIONS

(For Check on Reproducibility)

Mix Designation	Mix Formulas (% by Weight)				Ratio Residue/ Fly Ash	Compressive Strength (psi)		Dry Density[2] (pcf)	Moisture Content[2] (%)
	Alliance AFB Residue	Fly Ash[1]	Concrete Sand	Limestone		7 Day (73°F	28 Day Cure)		
1	30	10	60	0	3:1	180	893	119.2	9.6
2	12.5	12.5	75	0	1:1	172	850	122.9	6.9
3	10	30	60	0	1/3:1	188	860	118.2	7.2
4	30	10	0	60	3:1	235	520	124.9	9.3
5	12.5	12.5	0	75	1:1	216	990	132.1	9.8
6	10	30	0	60	1/3:1	540	1540	125.6	8.5

[1]Pulverized coal fly ash from National Minerals Corporation.

[2]For specimens at time of molding.

NOTE: All compressive strengths are the average of three tests.

TABLE V

SUMMARY OF COMPRESSIVE STRENGTHS OF ROAD BASE COMPOSITIONS OBTAINED IN 1977

Mix Formulas (% by Weight)			Ratio	Compressive Strength (psi)	
AFB Residue	Fly Ash[1]	Aggregate	Residue/ Fly Ash	7 Day (73°F Cure)	28 Day (73°F Cure)
44	12	44	3.7:1	310	2040
24	15	61	1.6:1	80	450
24	15	61	1.6:1	140	-
20	15	65	1.3:1	180	550
16	15	69	1.1:1	120	830
12	15	73	0.8:1	250	2060

[1]Pulverized coal fly ash from National Minerals Corporation.

A comparison of the compressive strength results is shown in Table VI. While the comparison shows that the strengths obtained were of similar magnitude, the range of compressive strength is quite broad (although the current tests show less spread after 28 day curing). More data will be required before arriving at a statistical analysis of the variation in the uniformity existing between samples of the material and effect of mix proportions in the test specimens.

PRODUCTION AND EVALUATION OF SYNTHETIC AGGREGATE PRODUCED BY BRIQUETTING

Following preliminary trials with briquetting of various AFB residue compositions (reported in the March-May 1980 Quarterly Report), an additional 850 pounds of briquettes were made. These briquettes were produced in a laboratory scale, roll briquetting machine at the laboratories of K. R. Komarck, Inc., Elk Grove Village, Illinois. There were 18 different mixes consisting of combinations of AFB residue, pulverized coal fly ash, sulfate waste from three sources, and two chemical additives. The mix compositions are given in Table VII along with comments on the quality of the briquettes immediately after molding.

The briquettes are being aged in four different environments:

- in a moist room at 73°F.
- sealed in plastic bags; at room temperature.
- outdoors in open, drained containers.
- in a moist condition at 55°F.

Periodic evaluations of bulk specific gravity, absorption, and scratch hardness were made. Eighty groups of briquettes representing various curing conditions and ages were tested for specific gravity and absorption. Each group consisted of at least three briquettes. The average bulk specific gravity was 1.69 with a range from 1.43 to 2.04. The absorption averaged 15.0 percent with a range from 6.5 to 22.1. The data did not seem to show any relationship between these two parameters and the age or curing conditions of the briquettes. Table VIII tabulates the results of an improvised scratch hardness test.

PREPARATION FOR PRODUCTION OF MASONRY UNITS

Approximately 8000 pounds of AFB residue from Alexandria, Virginia were conditioned at the Allied Concrete Plant in Dresher, Pennsylvania. Sufficient water to hydrate the calcium oxide was added and mixed with the residue in a ready-mix concrete truck. The conditioned material was placed in 55 gallon drums and is being stored in preparation for making masonry units.

TABLE VI

COMPARISON OF CURRENT AND PREVIOUS COMPRESSIVE STRENGTHS OF AFB COMPOSITIONS

	Compressive Strength (psi)			
	7 Day Cure		28 Day Cure	
	Range	Average	Range	Average
Current Data	172 - 540	255	520 - 1540	942
Previous Data (1977)	80 - 310	180	450 - 2060	1186

TABLE VII

MIX FORMULAS AND PRELIMINARY EVALUATION OF BRIQUETTES

Mix Designation	Mix Formulas (% by Weight)						
			Sulfate Wastes[3]				
	AFB Residue[1]	Fly Ash[2]	Glidden	Univ. of TN	Rockwell	% Chemical Additive	Visual Observation of "Green Strength"[4]
1	30	70					Easily handled immediately after molding. (Good)
2	30	70				1% Ethylsilicate 40	Set up within 1 hr. (Excellent)
3	5	45	50				Fair to good.
4	15	35	50				Better than Mix#3. (Good)
5	15	35	50			1% Ethylsilicate 40	Too wet to mold
6	30	20	50				Excellent
7	30	20	50			1% Ethylsilicate 40	Good to excellent
8	70	30					Good
9	70	30				1% Ethylsilicate 40	Excellent
10	30	70				10% $MgCO_3$	Excellent
11	5			95			Poor
12	30			70			Poor
13				100			Poor
14					100		Poor
14-A					100		Fair
15	5				95		Poor to fair
16	30				70		Fair to good
17	30			70			No briquettes - insufficient material
18	5	95					Fair
19	47.5	47.5				5% $MgCO_3$	Good

[1]Alexandria AFB residue.

[2]Pulverized coal fly ash from Wagner Station, Baltimore Gas and Electric Co.

[3]Properties of sulfate wastes were identified in July 1980 report.

[4]"Green" strength is the ability of the briquettes to hold together immediately after molding. (See following page for meaning of poor to excellent.)

RATINGS OF BRIQUETTE "GREEN STRENGTH" BASED ON

VISUAL OBSERVATIONS IMMEDIATELY AFTER PRODUCTION

Poor - Must be careful handling briquettes or they will fall apart. Slightest finger pressure will break briquette.

Fair - Can handle briquettes as they come out of mold and place in container without them falling apart.

Good - Can be dropped on floor and will not fall apart.

Excellent - Hard immediately after molding; difficult to break with finger pressure; they feel like hard rubber.

TABLE VIII

HARDNESS OF BRIQUETTES AS EVALUATED BY AN IMPROVISED SCRATCH TEST[1]

	Cure (8 Days)	
Mix No.	Moist Room at 73°F	Outdoors in Open Container
1	S	S
2	S	S
3	VS	VS
4	MH	-
6	D	D
7	S	S
8	H	H
9	S	MH
10	H	H
11	MH	S-MH
12	MH	MH
13	D	D
14	D	D
14-A	MH	S
15	MH	S
16	VS	VS
18	S	S
19	MH	H

[1]Scratch test consisted of scratching the briquette with a steel knife blade.

H - Hard; a light scratch appears with moderate pressure; surface cannot be pared.
MH - Medium hard; larger scratch; some powder; surface pares with difficulty.
S - Soft; deep wide scratch easily made; pares easily.
VS - Very soft; crumbles easily; scratch is indistinct.
D - Disintegrates in storage or in handling.

USE OF AFB RESIDUE AS AN EXPANSIVE GROUT

Compositions consisting of combinations of AFB residue, AFB fly ash, and pulverized coal fly ash have been prepared in slurry form and molded into cylindrical (3.42" diameter x 3.31" high) test specimens. All materials were mixed dry for ten minutes. Forty percent waste was then added to bring the mixture to a "flowable" consistency. Test specimens were molded by pouring the resulting slurry into molds a third at a time and rodding each lift 25 times. After molding, the specimens were cured for 24 hours under ambient conditions and then placed in their respective curing chambers (55°F or 73°F) for 7 or 28 days. The specimens were evaluated for compressive strength and permeability. The compressive strength and permeability results, along with the mix compositions, are presented in Table IX. Additional test specimens are being prepared for dimensional stability analyses and degree of expansion.

OHIO ROAD BASE DEMONSTRATION

The road base test sections placed in a parking lot in Canton, Ohio in December 1979 have been monitored three times: May 2nd, July 9th, and August 27th, 1980. The layout of five test strips, formulations of four different mixes, and the locations of control points are shown in Figure 1. The AFB residue was hydrated prior to use at a fly ash conditioning plant in the Chicago area.

Monitoring of these sections consisted of:

- Visual inspection of the surface.

- Measurement between all adjacent control points.

- Evaluation of the relative hardness or strength development by a modified penetration resistance measurement.

- Periodic photographing.

The "modified penetration resistance measurement" was made by driving a No. 4 reinforcing bar into three randomly selected locations in each test strip with a sledge hammer. The number of blows required to drive the rod for each inch of a four inch penetration were recorded. Table X shows the changes in length (in percent) that have taken place between control points and Table XI shows the results of the penetration resistance measurements. Two of the four mixes appear to be dimensionally stable (1 and 3); Mix 4-A seems marginal; and Mix 2 (2-A is same composition) shows relatively high expansion. The penetration resistances are increasing indicating that the test strips are gaining strength. It was also evident that the bottom two inches of the strips had greater penetration resistance than the upper two inches. There was no cracking in any of the strips; however, all the exposed surfaces were uneven. In general, the mixtures were performing reasonably well, recognizing that the sections were placed under winter conditions and no surface

TABLE IX

COMPRESSIVE STRENGTH AND PERMEABILITY OF EXPANSIVE GROUTS

Mix Formulas (% by Weight)			Compressive Strength (psi) — Curing Conditions					Previously Obtained Compressive Strength[4] (psi)-73°F Cure	
			7 Days		28 Days				
AFB Residue[1]	Fly Ash[2]	AFB Fly Ash[1]	55°F	73°F	55°F	73°F	Permeability[3] (cm/sec)	7 Days	28 Days
80	20	0	229	245	386	433	1.31×10^{-5}	140	530
40	60	0	231	220	297	498	1.16×10^{-5}	140	610
40	20	40	90	101					
30	40	30	106	128					
20	60	20	136	267					
10	80	10	103	264					
20	0	80	193	193	350	371	1.22×10^{-5}		
10	0	90	250	286	407	432	7.14×10^{-6}		

[1] Alliance AFB residue that was hydrated prior to use. The AFB fly ash was also from Alliance.

[2] Pulverized coal fly ash from National Minerals Corporation.

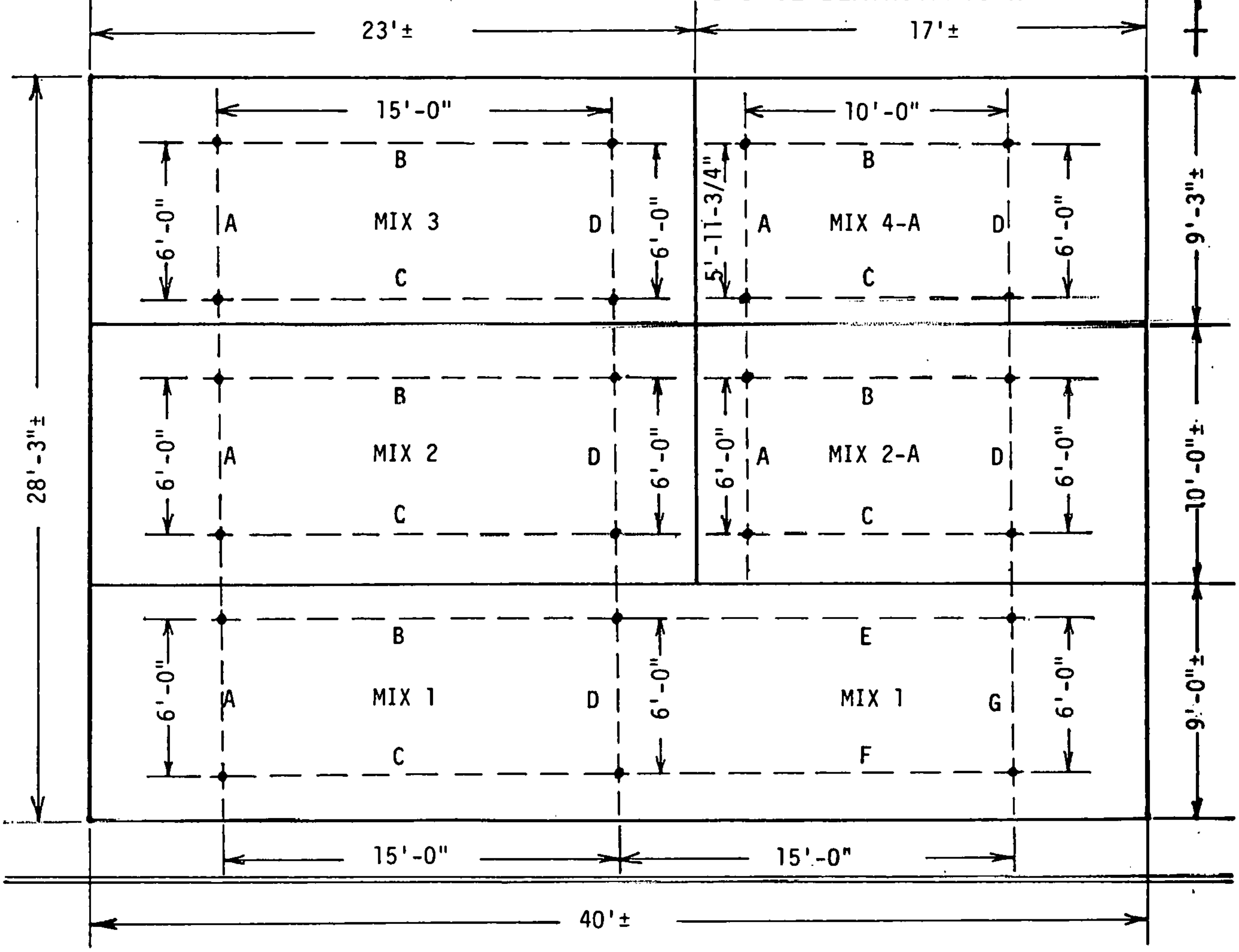
[3] Permeability test specimens were cured for 7 days at 73°F prior to testing.

[4] For reproducibility comparison only.

NOTE: All compressive strength samples were vacuum saturated for ½ hour and soaked at atmospheric pressure for 1 hour prior to testing. Those mixes containing all three of the materials (AFB residue, AFB fly ash, and pulverized coal fly ash) were very fragile and testing was discontinued on these specimens.

FIGURE I

DIMENSIONS, MIX PROPORTIONS, AND LOCATIONS OF
CONTROL POINTS FOR CANTON ROAD BASE DEMONSTRATION



Mix	AFB Residue Preparation	Percent Aggregate[1]	Percent AFB Residue[2]	Percent Fly Ash[3]
1	As Received	50	15	35
2	As Received	70	10	20
2-A	Pre-crushed	70	10	20
3	Pre-crushed	75	5	20
4-A	Pre-crushed	80	20	-

[1]Sand and gravel, local to Canton.
[2]From Alliance, Ohio.
[3]A bituminous coal fly ash from western Pennsylvania.

TABLE X

DIMENSIONAL STABILITY MEASUREMENTS

CANTON, OHIO TEST STRIPS

Mix No.	Measurement No.	Original Length (ft)	Monitoring Date 5/2/80	7/9/80	8/27/80
			Change in Original Length (%)*		
1	1	6.0	+0.17	+0.17	+0.34
	2	15.0	-0.14	-0.14	-0.14
	3	15.0	-0.07	-0.07	-0.07
	4	6.0	+0.17	+0.34	+0.34
	5	15.0	+0.07	+0.07	+0.07
	6	15.0	-0.07	0	-0.07
	7	6.0	+0.52	+0.69	+0.52
2	1	6.0	+0.52	+0.78	+0.69
	2	15.0	-0.49	-0.42	-0.42
	3	15.0	-0.83	-0.70	-0.70
	4	6.0	+3.13	+3.30	+3.30
2-A	1	6.0	+1.22	+1.40	+1.22
	2	10.0	+0.10	+0.21	+0.21
	3	10.0	+0.31	+0.42	+0.31
	4	6.0	+1.04	+1.22	+1.22
3	1	6.0	-0.17	0	+0.34
	2	15.0	-0.21	0	-0.07
	3	15.0	-0.14	-0.14	0
	4	6.0	0	+0.17	0
4-A	1	5.98	+0.35	+0.70	+0.70
	2	10.0	+0.21	+0.31	+0.42
	3	10.0	0	+0.21	+0.10
	4	6.0	0	+0.35	+0.34

*Plus (+) means increase in length; minus (-) means decrease.

TABLE XI

MODIFIED PENETRATION RESISTANCE MEASUREMENTS

CANTON, OHIO TEST STRIPS

	Monitoring Date		
	5/2/80	7/9/80	8/27/80
Mix No.	Average Number of Blows for 4" Penetration*		
1	23.3	18.7	32.0
2	24.0	23.0	33.7
2-A	21.7	29.7	26.0
3	22.0	18.3	29.3
4-A	21.3	16.7	19.7

*The total number of blows required for 4" penetration were recorded at each of 3 locations in each test strip. These totals were averaged.

treatment was applied during construction.

FURTHER EVALUATION OF DIMENSIONAL STABILITY OF ROAD BASE TEST PADS

Ten new test pads, 4' long, 6" wide, and 6" thick have been made from ten combinations of Georgetown AFB residue and AFB fly ash with pulverized coal fly ash and sand aggregate. The compositions of the test pads are shown in Table XII. The pads were molded in wooden forms and remained covered and in the forms for 28 days at which time the cover and forms were removed. The pads are now exposed to the weather. Dimensional stability measurements are to be made periodically over a 20" gage length. These will be reported as trends develop. Companion laboratory specimens will also be tested for compressive strength and dimensional stability.

ENGINEERING PROPERTIES OF AFB FLY ASH

The particle size distribution, moisture-density relationships, shear strength characteristics, and permeabilities of compacted samples have been determined for two AFB fly ashes - Alliance and Georgetown. The results are given in Figure 2 and Table XIII.

CONCLUSIONS

- The road base mixes in which quartz silica was substituted for the pulverized coal fly ash gave compressive strengths similar to those using the pulverized coal fly ash.

- The compressive strengths of road base mixes using recently produced AFB residue were of the same order of magnitude as those obtained in 1977, although the data covers a broad range of test results.

- Briquettes, produced from a number of trial mixes, show promise as a synthetic aggregate.

- Expansive grout mixes of slurry consistency achieve satisfactory compressive strengths in seven days.

- Sections of the test strips in Canton, Ohio show minimal expansion.

ACTIVITIES DURING NEXT QUARTER

During the next quarter, the following work will be performed:

- Masonry units (8" x 8" x 16" hollow block) containing AFB residue, pulverized coal fly ash, and aggregate will be manufactured.

TABLE XII

COMPOSITIONS OF 4′ x 6″ x 6″ TEST PADS

	Mix Formulas (% by Weight)			
Mix No.	Sand Aggregate[1]	AFB Residue[2]	AFB Fly Ash[2]	Pulverized Coal Fly Ash[3]
1	60	10	-	30
2	60	10	10	20
3	70	10	-	20
4	70	5	5	20
5	70	10	10	10
6	80	5	-	15
7	80	10	10	-
8	80	5	6	10
9	80	10	-	10
10	80	20	-	-

[1]Lockhart sand.

[2]AFB residue and fly ash from Georgetown University, Washington, DC.

[3]Pulverized coal fly ash from National Minerals Corporation.

FIGURE 2

PARTICLE SIZE DISTRIBUTION OF AFB FLY ASHES

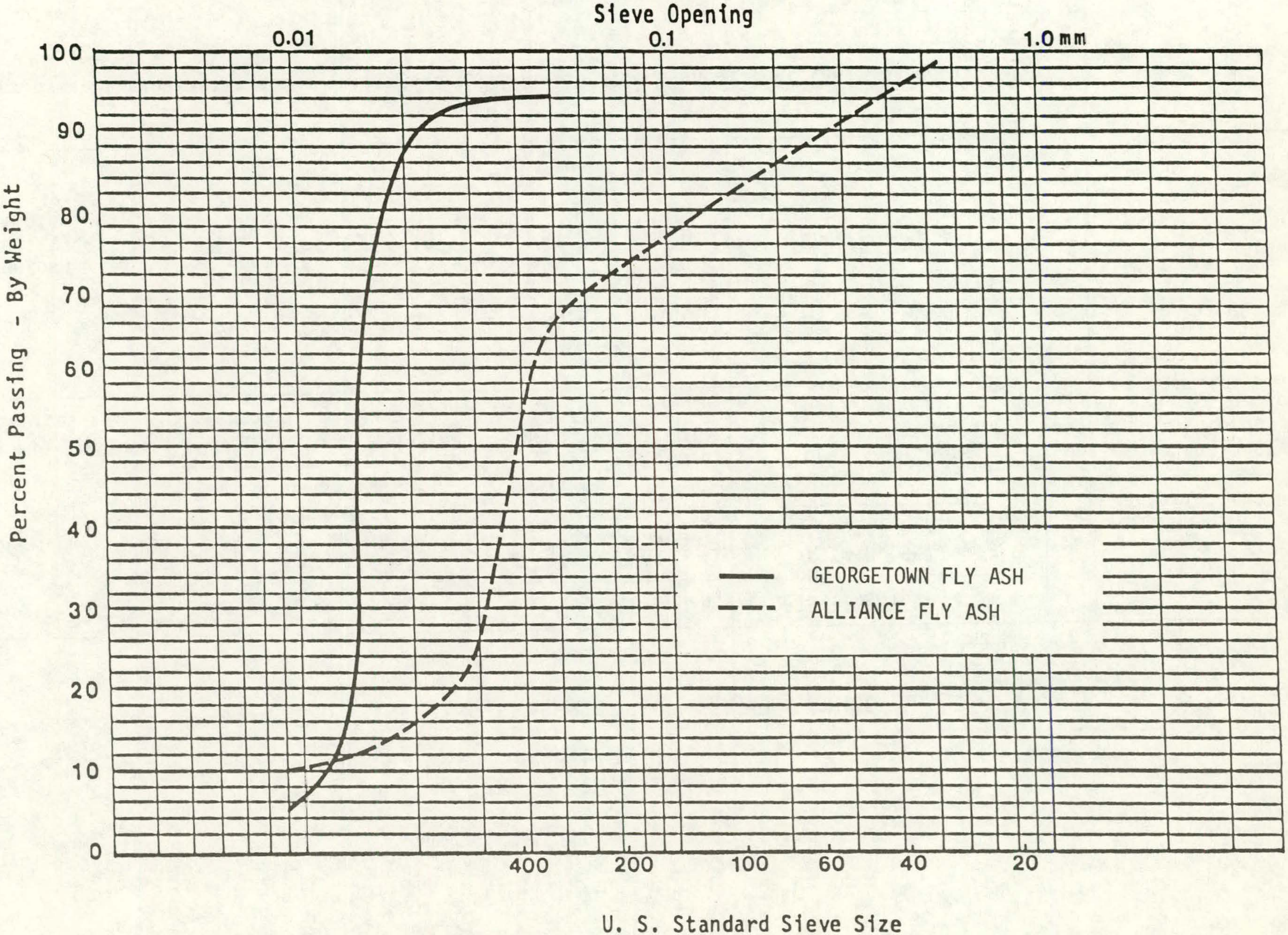


TABLE XIII

ENGINEERING PROPERTIES OF AFB FLY ASHES

AFB Fly Ash Source	Coefficient of Permeability (cm/sec)		Moisture-Density Relationships				Shear Strength Characteristics*	
			ASTM D 698 (Standard)		ASTM D 1557 (Modified)			
	At Standard Density	At Modified Density	Maximum Dry Density (pcf)	Optimum Moisture (%)	Maximum Dry Density (pcf)	Optimum Moisture (%)	Unit Cohesion (pcf)	Angle of Internal Friction (ϕ)
Alliance, OH	1.3×10^{-4}	5.5×10^{-5}	80.5	30.0	83.3	24.0	0	33.9°
Georgetown University, Washington, DC	1.1×10^{-3}	1.1×10^{-5}	61.2	43.0	69.8	36.8	0	34.6°

*Shear strength characteristics determined by means of direct shear test (ASTM D 3080).

- Two masonry walls, approximately 25' long and 4' high, will be constructed to evaluate the weathering characteristics of the masonry units.

- Investigation of combinations of techniques for conditioning, beneficiation, or otherwise separating AFB by-products will be continued - this will include chemical and physical analysis of the resulting fractions.

- Studies on the most effective means of eliminating or controlling expansion will be continued.

- Evaluation of briquetted aggregates will be continued and additional briquettes will be produced of those types showing satisfactory properties.

- Methods for predicting the amount of volume change of expansive grouts will be explored.

- Definitive discussions will be underway with the Ohio Department of Transportation and the Appalachian Regional Commission relative to a road base demonstration scheduled for the 1981 construction season.

- Monitoring of the road base test strips in Canton, Ohio will be continued.

- There will be a continuing study to determine the nature of the chemical reactions that occur in the compositions containing AFB residue and pulverized coal fly ash.

- Efforts will be continued to develop a demonstration of the effectiveness of AFB residue in treating acidic trade wastes.

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