

FINAL TECHNICAL REPORT
November 1, 2000, through July 31, 2002

Project Title: **A PROPOSAL TO COMMERCIALIZE A SINTERED
COAL ASH/FLUX MATERIAL FOR BUILDING PANELS**

ICCI Project Number: 00-1/3.1B-1

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ABSTRACT

Illinois coal ash from the power plant at Little Egypt, owned by the Southern Illinois Power Cooperative in Marion, has heavy metals and is considered a difficult waste for disposal. In our process it has tremendous potential because of its high iron and metal content. Iron can provide a variety of colors while the heavy metals can be sequestered inside the panels.

The products are structural panels that are colored, strong, and insulative. By either sintering in an oven at 725°C or by adding a flux and sintering at 525°C we can produce panels that will not leach metals from the ash.

In the work sponsored by the Illinois Clean Coal Institute we have worked to explore production parameters for the panels in both the lab as well as at the industry partner's facility. Fifteen large panels, four feet by eight feet by six inches, were produced at the facility and numerous smaller samples in the lab; these included three one foot by one foot by six-inch blocks. The parameters investigated included refinement of the molds, additive use such as inexpensive fibers to reduce drying shrinkage, and the application of pressure rather than mixing when making the large panels in order to reduce the water usage. Studies of loss on ignition of the various ash mixes used were correlated to strength data, and thermographic analysis were used to investigate heating regimes and to measure interior heat soak rates.

The main findings were:

- Sintering and the addition of boric acid help to sequester metals in the product.
- Large panels without cracks can be fabricated in the factory.
- Use of fiber additives reduces drying shrinkage cracks in the large panels.
- The amount of carbon in the ash affects the strength; more carbon weakens the product and also required more water for mixing.
- Heating to 200°C (to avoid out-gassing) in a moist chamber eliminates spalling.

EXECUTIVE SUMMARY

Illinois coal ash from the power plant at Little Egypt, owned by the Southern Illinois Power Cooperative in Marion, has heavy metals and is considered a difficult waste for disposal. In our process it has tremendous potential because of its high iron and metal content. Iron can provide a variety of colors while the heavy metals can be sequestered inside the panels.

Project Objectives And Technical Approach

The project objective was to develop commercially viable building products using Illinois coal ash. The technical approaches to achieving this objective were:

- to demonstrate the feasibility of sequestering ash metals in a value added product with competitive structural, insulative, density, water absorption and color properties.
- to study the microstructure to optimize the performance parameters of strength, density, water resistance and insulative properties. (Microstructure is the small scale internal makeup of a material which has certain physical properties which affect the larger scale performance characteristics.)
- to optimize the production parameters at the industrial facility to produce these panels.
- to develop a business plan to commercialize the product.

The commercial implementation plan required first developing a viable panel in the laboratory and then proceeding to develop the manufacturing conditions for the industrial partner's plant. The tasks were:

- to demonstrate the feasibility of sequestering ash metals in a value added product with competitive structural, insulative, weight, water absorption and color properties.
- to refine the microstructure in order to optimize the performance parameters of strength, density, water resistance and insulative properties.
- to optimize the production parameters at the industrial facility.

Fifteen large panels, four feet by eight feet by six inches were produced at the industrial facility and numerous smaller samples in the lab; including three 1'x1'x6" blocks. The parameters investigated at the industrial facility included refinement of the molds, addition of inexpensive fibers to reduce drying shrinkage, and pressing the panels, rather than mixing to reduce water use. Lab studies included loss on ignition of the ash correlated with strength data, and thermographic analysis measured interior heat soak rates and heating regimes.

The main findings were:

- Large panels without cracks can be manufactured in the factory.
- Addition of fibers reduces drying shrinkage cracks.

- The amount of carbon in the ash affects the strength - the more carbon, the weaker the product. A higher carbon content in the ash requires more water for mixing, producing a weaker panel.
- There is no problem in heating and sintering all the way through panels which contain metal chases; panels without such chases take longer to heat and sinter through their depth.
- Heating to above the critical 200°C in a moist chamber eliminates surface spalling.

The uncompleted technical goals are:

- To investigate the vacuum sintering of Illinois coal ash for other panels. This was not done because the industrial partner did not have a vacuum furnace available.
- To develop a business plan to commercialize the product.

OBJECTIVES

The project objective was to develop commercially viable building products using Illinois coal ash. The technical approaches to achieving this objective were:

- To demonstrate the feasibility of sequestering ash metals in a value added product with competitive structural, insulative, weight, water absorption and color properties.
- To refine the microstructure in order to optimize the performance parameters of strength, density, water resistance and insulative properties.
- To perfect the production parameters at the industrial facility to produce these panels.
- To investigate the vacuum sintering of Illinois coal ash for other panels.
- To develop a business plan to commercialize the product.

The tasks to accomplish in this project were:

- Preparing laboratory samples of Illinois fly ash materials mixed with acid and sintered in a furnace. Samples were sintered at 500 - 750°C in a ramped furnace.
- Preparing laboratory samples of fly ash materials to sinter. A furnace with a temperature of 500-750°C and a vacuum with a 10^{-4} Tor sintered the fly ash samples. (The temperature is directly related to the size of the vacuum pump used. A sample can be sintered at a lower temperature with a more powerful vacuum pump.) Several test samples were produced at temperatures varying from 500 - 750°C. Initially, the variable parameters were two temperatures and three vacuum pressures.
- Testing the samples for insulation value, water absorption and compressive strength mass using accepted procedures. Samples were prepared from methods described in tasks 1 and 2.
- Refining material composition to improve performance properties of samples.
- Analyzing leachate for heavy metals. This work was done in the Natural Resources Laboratory using the toxic characteristic leaching procedure (TCLP).
- Analyzing the microstructure.
- Fabricating the fly ash mixture as panels.
- Translating the laboratory findings into engineering parameters for use in the factory.
- Fabricating 10-15 full scale panels in the factory.
- Evaluating the processing parameters and costs.
- Testing and evaluating 5 panel types for performance in terms of weight, strength, insulation values, and water intrusion.
- Testing the panels' microstructure and testing impact and fire resistance.
- Assessing the advisability of using Illinois coal as fuel for sintering panels.
- Making recommendations on panel materials and processing.

INTRODUCTION AND BACKGROUND

Illinois coal ash from the power plant at Little Egypt, owned by the Southern Illinois Power Cooperative in Marion, has heavy metals and is considered a difficult waste for disposal. Currently the company mixes it with calcium carbonate scrubber slurry to absorb the heavy metals and it is then placed in abandoned mines as fill. In our process it has tremendous potential because of its high iron and metal content. Iron can provide a variety of colors while the heavy metals can be sequestered inside the panels.

Table 1. Results of leachate of raw ash (row one), leachate from fired ash mixtures (rows 2-5) and fired ash mixtures (rows 6-7). Results are in parts per million, ppm.

	1)Raw 3 Ill. Fly ash-ICP results	2)0% H3BO3, 3hr fire	3)30% H3BO3, 3hr fire	4)0% H3BO3, 3hr vac	5)30% H3BO3, 3hr vac	6)0% H3BO3, no fire	7)30% H3BO3, no fire
B	17.64	14.8	700	17.5	>high	50.51	1075
Be	0.313	0.001	0.004	-0.001	0.002	0.312	0.060
Cd	5.07	0.290	0.429	0.029	0.593	8.46	1.43
Co	0.37	0.006	0.075	<0.003	0.102	0.715	0.111
Cr	5.55	<0.004	<0.004	<0.004	<0.004	0.611	0.100
Cu	8.63	0.109	0.131	<0.003	0.028	1.14	0.293
Mg	76	76.7	56.2	32.0	125	156	25.5
Mn	4.44	1.48	3.13	0.138	5.86	9.65	1.55
Mo	0.87	7.23	0.223	14.6	0.707	<0.006	0.012
Ni	2.16	<0.007	0.106	<0.007	0.226	6.02	0.705
Pb	4.9	<0.028	<0.028	<0.028	0.116	0.128	0.068
V	0.13	0.994	0.025	2.96	0.239	0.023	0.013

Prior Laboratory Tests on Illinois Coal Fly Ash Mixtures for Building Panels

For the laboratory study, Illinois fly ash samples were produced to test the compressive strength, thermal value, water absorption, weight, and leaching of the materials with and without acid, with and without sintering and with and without vacuum sintering. Also, pigmentation was varied by either changes in temperature or amount of boric acid flux. (Flux is an additive that allows the temperature to be lowered for sintering.) (Sintering is heating a material to a high temperature so that the particles fuse into each other.)

The top priority issue for use of this ash is leaching. (Leaching is extraction of a chemical such as metals by water action over time.) Standard leaching and nuclear resonance tests for some 30 chemicals was done by the Illinois Water Survey on all sample types. For the leachate tests, the samples were first placed in distilled water that has a pH of about 5.6 to simulate the typical pH of rain in Illinois. The samples were submerged in water for approximately 4 days before testing. The results showed that heavy metals were well sequestered, as seen in Table 1.

Sintering this ash with water or with a flux, boric acid, at a temperature which melts the ash, sequesters the metals. Most ash melts at about 700°C but the addition of the boric acid acts as a flux allowing melting of the ash at about 525°C. Ash is made up of some floaters or air filled spheres which makes this material light weight, useable for insulation and structures. The high iron content allows for coloration; the color changes from dark rust to tan to orange to violet.

Summary of Findings from Prior Work on Illinois Coal Fly Ash

- The strongest samples were those sintered at 725°C without boric acid or those with acid but sintered at the lower 525°C temperature. This probably means that at 700°C the glass transition state is reached without the acid flux. The boric acid at 525°C acts as a flux to reach the glassy melted state.
- The most insulative material is that which is vacuumed at 727°C without boric acid.
- More boric acid (a flux) or higher temperature gives the most intense color because the valence of the iron shifts as the glass transition temperature is reached.
- The leaching tests showed that the non-fired samples did not sequester the chemicals nearly as well as those that were fired. The boric acid had no effect at the 725°C temperature. It is expected that the boric acid would help to sequester the metals at 525°C.
- The water absorption tests showed that samples without boric acid dissolved at 525°C, while samples treated with boric acid showed little dissolving even at 725°C.

The conclusion was that panels with a choice of price (based on acid cost or sintering temperature), color, insulation value, strength and associated weight, non-leaching nor water absorbing can be produced.

EXPERIMENTAL APPROACH

For the laboratory portion of this study, Illinois fly ash samples were produced to refine and the compressive strength and the thermal value, water absorption, and leaching of the material with and without vacuum sintering. Samples were heated either at 525°C or 725°C. For the scale up portion of the project we fabricated new molds at 4'x8'x6". Translation of the laboratory findings into engineering parameters for use in the factory was done. At the factory we made 10 full-scale panels. During this we evaluated the processing parameters and costs. The panels were tested for performance in terms of weight, strength, insulation values, and water intrusion.

RESULTS AND DISCUSSION

Material Characterization and Testing in the Laboratory:

Task 1) sinter using Illinois coal ash, Task 2) refine microstructure, Task 3) make all sample materials and test performance parameters, Task 6) refine material based on test results.

Fly ash bricks and cubes were made to optimize the sintering behavior, to reduce shrinkage cracking, to enhance insulative and mechanical properties, especially compressive strength (Table 2). All samples began with a 0.15 water/solids ratio and increased until a pourable consistency was found. All bricks measured 2" x 4" x 6" unless otherwise noted, and all cubes measured 2" x 2" x 2". All samples consisted of fly ash and water, and any other additives are noted in Table 2:

Table 2. Preliminary lab samples testing different compositions and firing temperatures for strength and insulative properties.

Additives	Water/Solids Ratio (w/s)	Sintering Temp. (°C)	Comments
2% ADVA Flow	0.23	None	Drying shrinkage cracks
10% Water Reducer	0.27	None	
15.5% Mighty	0.23	None	
3% Lignosulfanate	0.20	105°	Dark brown, large cracks
12% Darvan C	0.27	700°	Surface yellow, flaky above orange/brown, soft
Control (no additives)	0.18	105°	Remained dark brown shrinkage cracks appeared.
Control	0.18	700°	Turned red/orange, hard, good shape, small cracks on edge
Control	0.23	None	Remained dark brown, no cracks. 8% water absorption.
Control	0.23	105°	Turned lighter brown, no cracks. Broke down in water.
Control	0.23	200°	Turned brown, exploded in oven. Broke down in water.
Control	0.23	700°	Turned orange, exploded in oven. 15% water absorption.
White fibers (threads)	0.23	700°	Turned orange brown, few white clusters, cracks side and bottom
Micro-porous hollow fibers	0.23	700°	Turned red/brown, white clusters, hard and good shape, large cracks on bottom
3% Silica Sand	0.23	700°	Turned orange-red, very small cracks.
Straw	0.23	700°	Turned orange-red, small cracks.
10% Borax	0.33	None	Very large drying shrinkage cracks.
20% Boric acid	0.20	None	
10% Boric acid	0.23	700°	Turned orange red, some small cracks.
10% Boric acid, 3% silica sand	0.23	700°	Turned red-brown with large amount of white clusters, severe cracking
10% Boric acid, straw	0.23	700°	Turned red/brown, many white clusters, no cracks

Compressive Testing of Ash Mixtures for Compressive Strength Properties

Compressive tests were run on fly ash cubes measuring 2"x 2"x 2". Results of compressive tests of fly ash cubes are found in Table 3. Samples made with boric acid and fired at 700°C are the strongest. The addition of sand to the boric acid samples results in significantly stronger samples. Firing temperature is directly related to compressive strength; samples fired at 700°C, both with boric acid and without boric acid, will not dissolve in water.

Table 3. Average compressive strengths of UIUC lab fly ash samples.

Cure	Sample	Max load (KN)	Max load (lb)	Ave. Load (lb)	Av. Compressive Strength, (psi)
1 day no boric acid	1	0.98	219.	222.	55.
	2	0.99	223.		
	3	0.99	223.		
4 day no boric acid	1	1.72	388.	369.	92.
	2	1.67	376.		
	3	1.53	345.		
105 deg no boric acid	1	17.77	4000.	2996.	749.
	2	11.06	2489.		
	3	11.11	2500.		
200 deg no boric acid	1	8.61	1937.	1694.	423.
	2	5.72	1287.		
	3	8.26	1858.		
700 deg No boric acid	1	20.96	4718.	4241.	1060.
	2	12.95	2915.		
	3	22.61	5089.		
700 deg With boric acid	1	50.99	11478.	11674.	2918.
	2	52.73	11870.		
700 deg with Boric acid and sand	1	156.91	35321.	30455.	7613.
	2	106.16	23897.		
	3	142.81	32147.		
700 deg with Sand and no Boric acid	1	34.37	7737.	7923.	1980.
	2	36.17	8142.		
	3	35.05	7890.		

Task 4) Develop vacuum sintered fly ash samples

A preliminary set of vacuum sintered test samples were made to record any dimensional changes that occur when fly ash samples are fired in a vacuum. First, very small fly ash samples were fired to 500°C in air. At this point, the vacuum pump was turned on and firing continued as it reached approximately 800°C. The temperature was held here for 1 hour, and then the temperature was decreased under 500°C, where the vacuum pump was turned off. This test resulted in a 3% dimensional decrease after firing in the sample with 10% boric acid, and a 1.7% dimensional decrease in the sample without boric acid. Because of a thermocouple malfunction, the furnace probably was heated to 700°C instead of 800°C. Although it was hypothesized that firing the samples in the presence of a vacuum would expand the samples, the samples instead decreased in size. A possible

explanation is that some larger glass bubbles were vacuumed open and voided of air, and the melt state allowed for overall shrinkage.

Task 5) Analyze leachate results

Table 4 shows the results of leachate testing of fly ash samples with boric acid or without boric acid (as control), and either air-cured or sintered at 700°C.

Table 4. Leachate results.

	fly ash control, air-cured	fly ash control, sintered at 700°C.	fly ash with boric acid, air-cured.	fly ash with boric acid, sintered at 700°C.
Al	2.84 %	1.29%	2.48%	2.48%
As	230	270	220	270
B	657	275	1.42%	1.19%
Ba	299	295	268	292
Be	114	5.9	10.0	8.7
Ca	2.05%	2.39%	1.89%	2.54%
Cd	76	84	65	57
Co	30	21	27	25
Cr	214	138	213	184
Cu	194	114	177	155
Fe	9.08%	50.46%	7.14%	7.63%
K	1.35%	0.94%	1.27%	1.38%
Li	80	42	64	53
Mg	3480	4020	2890	3280
Mn	224	265	191	231
Mo	266	233	241	271
Na	2580	1320	2100	1810
Ni	138	80	124	123
Pb	1520	1140	1390	1110
S	2.60%	268()	2.30%	2.16%
Sb	0.42	0.42	<45	<48
Sc	13.1	10.2	11.8	140
Se	0.42	0.42	<45	<48
Si	0.16%	0.08%	0.12%	0.08%
Sr	298	363	268	378
Ti	2380	1440	2150	1530
Tl	48	<42	<45	<48
V	264	154	235	280
Zn	6820	2010	5820	2810

The results in Table 4 show that for nearly all of the chemical constituents of the ash, sintering sequesters (binds) and allows the release of fewer chemicals than the non-sintered sample. The tests also show that the non-sintered sample containing boric acid also sequesters chemicals better than the non-sintered sample without boric acid. The most successful sequestering of most chemicals occurs in the sintered sample with boric acid. The conclusion is that both sintering and the boric acid are necessary to sequester the chemicals in the ash material.

Task 7) fabricate panels, Task 8) test panels, Task 9) translate lab findings to engineering at factory, 10) build large molds, 11) evaluate equipment and processing needs in the factory, 12) fabricate panels in factory, 13) evaluate processing and costs of fabrication, 14) do lab tests on panels for strength, weight, insulation.

46" x 94" x 6" building panels were poured and fired at Magneco Metrel in Addison, IL (Figure 1, 2). The water ratio, mixing time and flowability were monitored, and an attempt was made to keep the flowability constant. Material from Panel 5 was taken for insulation testing. Panels were made with a variety of components. Panel 3 had cardboard chases and panel 5 had metal chases as seen in figure 2.



Figure 1. Full-scale panel being poured at Magneco Metrel.

Figure 2. Mold for Panel 5 with metal chases.

The composition of panels 1-5 is given in Table 5. After mixing and placement in the molds, all panels were covered with plastic to slow water evaporation. Water did bleed out from the molds soon after pouring. The issues addressed by the variables were overall weight of the panels, bending strength, and drying shrinkage/removal of water needed to cast the panels. Three days after the pour, Magneco Metrel reported that panel 2 had several very large cracks and that panel 3 had small cracks over the cardboard tubes. Strength development in the samples taken showed that they were developing adequate strength for this age. The plant manager worried about moving them into the oven while still wet and thought the panels were prone to cracking due to possible flexure of the molds. He heated the panels in place to dry them further before moving the panels into the drier.

Table 5. Full-scale panel compositions 1-5.

Panel	Water Ratio	Gallons of water used	Flow Rate	Additives
1	33%	44	148	None
2	31%	39	120	Silica sand (9% by weight)
3	30%	43.7	120	10% boric acid, ten 3" diameter x 4 ft. long cardboard tubes spaced 9" on center
4	30%	43.9	120	10% boric acid
5	33%	48	120	10% boric acid, 1.5 pounds of polypropylene fibers .75" long, eleven 1.25" diameter x 4 ft. long metal tubes spaced 9" on center

After a week, panels 1, 2, and 3 had cracked seriously and were broken beyond use. Panels 4 and 5 showed no cracking and little shrinkage. The conclusion is that the presence of the boric acid in the mixture eliminates the cracking and shrinkage during drying. Although panel 3 also contained boric acid, the large size of the cardboard tubes played a role in that panel's later crack formation. After firing the panels, panels 4 and 5 demonstrated the least cracking and shrinkage. Panels 1, 2, and 3 cracked severely. Panel 1 was broken up and the interior of the sample studied. It showed that the panel's interior did not turn orange like the exterior, which meant that a new firing regime (time of sintering, temperature) had to be investigated in order to achieve uniform sintering throughout the panel (Figure 3).

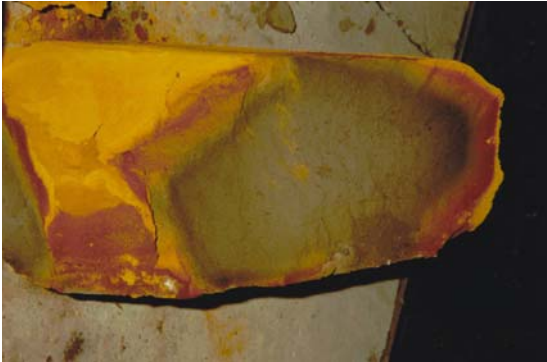


Figure 3. Interior of Panel 1 showing depth of sintering.

Based on these results, before the next pour, new stiffer molds were made and fibers were obtained to reduce cracking of the specimens. In addition we investigated superplasticizers that could lower the pH of the mixture while reducing the water needed for mixing. Most of the superplasticizers used in test samples were made for mixtures of higher pH values. That may be the reason they did not work in the fly ash mixture. Commercially produced ash aggregate was used in the panel mixtures for the next pour. The aggregate is made from fly ash, sewage sludge, and paper mill sludge. The gradation was 1/2" x 4 (100% passing 3/4" sieve). Five more 46" x 94" x 6" fly ash panels were poured at Magneco Metrel. Based upon results from the previous pour and on lab work, the panels were made with varying components as seen in Table 6. Fibers were added, including straw, to reduce cracking and a superplasticizer was added to reduce the amount of water used. Samples of each mixture were taken for testing of compression, insulation, and water permeability. Flow rate was not tested in this pour.

Table 6. Full-scale panel compositions 6-10.

Panel	Fly Ash	Water/Solids Ratio	Gallons of water used	Additives
6	~700 lbs	23%	30	320 lbs lightweight aggregate ½ bag Nyad G (wollastonite) 1 gallon ADVA Flow 66.6 lbs boric acid
7	~1100 lbs	30%	43	½ bag Nyad G 1 gallon ADVA Flow 100 lbs boric acid
8	~1100 lbs	28%	40	1 bag Nyad G Special 2 gallons ADVA Flow 100 lbs boric acid
9	~1100 lbs	27%	40	12 lbs metal fibers (equal volume to polypropylene fibers used in panel 5) ½ gallon ADVA Flow 100 lbs boric acid
10	~1100 lbs	26%	37	Straw (equal volume to metal and polypropylene fibers) ½ gallon ADVA Flow 100 lbs boric acid

Results of Compressive Strength Tests of Panels 6-10

Cubes measuring 2" x 2" x 2" were cast from the material of each of the large panels during the second pour. The cubes were cured, dried, and fired at the University of Illinois and then tested for compressive strength. The results follow in Table 7. The compression tests were done and one sample tested to show the influence of sintering temperature. As expected the higher temperature produced a stronger material. The material tends to have strength in the range of concrete masonry units (blocks), approximately 1,000 psi.

Observations and Tests of Samples from Panels 6-10

After firing of several samples in the second pour, removal from the molds was difficult, and keeping the samples intact was impossible. Samples were taken from panels 8, 9, and 10 since these panels had been fired the best (at approximately 1500 °F) and were still in their molds. Three other samples had been dumped by Magneco Metrel. These samples were not useable due to the poor firing and storage conditions. The forklift was used to drop the panel repeatedly until it broke apart enough to come out of the mold. This was a form of impact test. Some shaking was required but eventually all three panels were completely removed from their molds and each provided us with a large chunk with full thickness to be tested. Observations are noted in Table 8.

Table 7. Average compressive strengths of samples from full-scale fly ash panels produced at Magneco Metrel.

Panel	Additives	Water /Solids Ratio	Firing Temp. (°C)	Ave. Max Load (kN)	Ave. Max Load (lbs)	Ave. Max Compressive Strength (MPa)	Ave. Max Compressive Strength (psi)
6	320 lbs aggregate ½ bag Nyad G (wollastonite) 1 gallon ADVA Flow 66.6 lbs boric	23%	700°	2.40	539	0.834	121
7	½ bag Nyad G 1 gallon ADVA Flow 100 lbs boric acid	30%	500°	3.33	748	1.13	163
			700°	8.66	1946	3.21	465
			900°	25.0	5618	10.2	1478
8	1 bag Nyad G Special 2 gallons ADVA Flow 100 lbs boric	28%	700°	23.02	5173	8.96	1299
9	12 lbs metal fibers (equal volume to polypropylene fibers used in panel 5) ½ gallon ADVA Flow 100 lbs boric	27%	700°	18.3	4112	6.85	993
10	Straw (equal volume to metal and polypropylene fibers) ½ gallon ADVA Flow 100 lbs boric	26%	700°	18.63	4187	7.80	1130

Table 8. Observations on Sintered Panels.

Panel	Weight, lbs.	Cracking	Depth of Sintering	Comments
8	1303	Major crack at surface; covered w/ small cracks and a few deeply penetrating large cracks	Red/orange crust at surface (~1/8"-1/4" thick); red/orange and more porous (1" thick); black, very porous, soft interior	Interior showed signs of heating but not sintering (typical in all)
9	1201	Surface covered w/ small cracks	Red/orange crust at surface (~1/8"-1/4" thick); red/orange and more porous (1" thick); black, very porous, soft interior	Contained metal fibers which held pieces together better
10	1420	Surface smooth w/ few small cracks	Red/orange crust at surface (~1/8"-1/4" thick); red/orange and more porous (1" thick); black, very porous, soft interior	Contained straw as additive

Characteristics of Panels

All three panels came from the second pour at Magneco Metrel. They were all fired at 1500 °F for 5 hours in a large kiln and then stored outside.

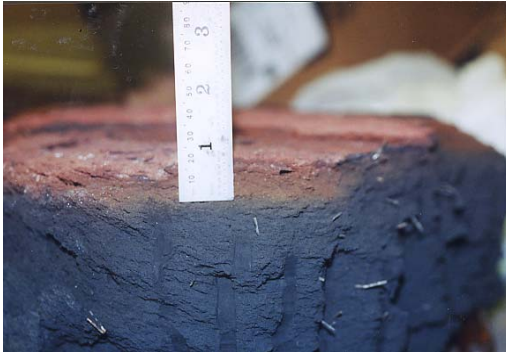


Figure 4: This tested depth of sintering of panel 9 from the second pour; the other panels from this pour had similar results.

Figure 5: Cores taken from panels 8, 9, & 10. The ends are capped with plaster to provide a smooth and even loading surface

Testing of Samples from Large Panels

A core drill was used to take six inches deep by three and three-quarter inches diameter cylinders for compressive testing (figure 5). Each core was capped to provide smooth and level testing surfaces using lime cement. All the samples were very similar in compressive strength. In a core, the top inch of the panel exhibited ceramic properties compared to a piece taken from the middle of a core that had the least sintering (figure 4). The top one-inch crust was nearly seven times stronger than the middle ash. This shows that the ash will gain a great deal of compressive strength when it is fired thoroughly. Results from the samples taken from the panels and laboratory samples made at the same time are very similar as seen in Table 9. Panel 8 showed the highest compressive strength while panels 9 & 10 were similar in their compressive strength. The results from the laboratory samples are in the following section.

Table 9. Results from compressive tests on samples from panels 6-10 from 2nd pour.

Panel		Max Load (lbs)	Max Load (kN)	Max Stress (psi)	Max Stress (Mpa)
8	Cast	2299.5	10.21	208.3	1.42
9	Cast	1748.5	7.76	158.4	1.08
10	Cast	1916.0	8.51	173.6	1.18
9 crust	Cast	14030.0	62.29	1270.8	8.64
9 middle	Cast	2318.0	10.29	209.96	1.43

Testing of Small-scale Fly Ash Cubes Made After the Pouring of Panels 6-10

Several different compositions of fly ash specimens were investigated in experiments involving the mixing and sintering of 2" cubes and the subsequent compressive testing of the cubes. These samples, along with the blocks to test for depth of sintering, were made after the second Magneco Metrel pour to investigate which compositions would be the most successful and feasible for the third panel pour. All of the cubes were mixed using

the same batch of ash as used for the third panel pour at Magneco Metrel. This ash was labeled MM2 ash. The cubes' compositions varied to order to study the effects of sintering temperature and duration, glass powder versus boric acid, calcined ash versus non-calcined ash, cast samples versus compacted samples, and the effect of ADVA Flow in the compositions. Among the properties noted during these investigations were the varying water requirements between specimens and the quality of sample formation.

Table 10 presents composition and sintering information of each type of sample along with the compressive strength test results and percent water absorption.

Table 10. Results of compressive tests of laboratory samples.

Sample Composition	Water Ratio	Sintering Conditions	Ave. Max Load, KN	Ave. Max Load, lbs.	Ave. Max Stress, MPa	Ave. Max Stress, psi	% Water Absorption
10% boric acid	.26	700°C 1 day	15.61	3509	6.06	878	27.6%
10% boric acid	.26	800°C 1 day	26.71	6003	11.9	1718	17.5%
10% boric acid	.26	900°C 1 day	9.61	2161	4.29	621	16.3%
10% glass powder	.25	700°C 1 day	1.403	315.4	0.54	78.9	
10% glass powder	.35	700°C 1 day	0.595	133.6	0.235	34.1	28.3%
10% glass powder	.35	800°C 1 day	4.33	973.7	1.73	251	25.1%
5% glass powder	.35	700°C 1 day	0.646	145.2	0.257	37.2	27.1%
5% glass powder	.35	700°C 3 days	0.807	181.3	0.317	45.9	24.6%
10% boric acid 10% glass powder	.25	700°C 1 day	13.9	3124	5.53	801	22.5%
10% boric acid 10% glass powder	.25	700°C 3 days	10.2	2285	4.00	580.	22.9%
Compacted 10% boric acid	.10	700°C 1 day	11.0	2470.	4.28	620.	26.0%
Compacted 10% boric acid	.10	700°C 3 days	10.9	2458	4.37	633	25.8%
Calcined ash 10% boric acid	.22	700°C 1 day	48.9	10,998	18.2	2642	15.9%
Calcined ash 10% boric acid	.22	700°C 3 days	50.3	11,299	19.1	2770	14.9%
Calcined ash 10% glass powder	.21	700°C 1 day	4.90	1102	1.87	271	16.2%
Calcined ash 10% glass powder	.21	700°C 3 days	5.51	1239	2.11	306	16.2%

Calcined ash, 10% boric acid, 5% glass powder No ADVA Flow	.23	700°C 1 day	10.9	2458	3.99	578	15.3%
Calcined ash, 10% boric acid, 10% glass powder, No ADVA Flow	.22	700°C 1 day	6.69	1503	2.55	370.	15.0%
Compacted, Calcined ash, 10% boric acid, 2% carbowax, No ADVA Flow	.10	700°C 1 day	8.06	1811	2.64	383	20.5%
Compacted, Calcined ash, 10% boric acid, 10% glass powder, 2% carbowax, No ADVA Flow	.10	700°C 1 day	14.9	3339	5.86	849	19.0%
Compacted, Calcined ash, 10% boric acid, 1% ball clay No ADVA Flow	.10	700°C 1 day	31.4	7061	11.9	1725	20.9%
Compacted, Calcined ash, 10% boric acid, 10% glass powder, 1% ball clay No ADVA Flow	.10	700°C 1 day	28.6	6425	11.0	1594	17.6%
25% lithium silicate solution	.33	700°C 1 day	3.5	787	1.38	199	31.0%
40% lithium silicate solution	.57	700°C 1 day	0.437	98.2	0.171	24.8	33.0%

- All samples were mixed with non-calcined ash and cast unless noted otherwise. As with the blocks, all cubes included straw in the composition (see full-scale panel 10) and ADVA Flow, unless otherwise noted. The quantity of solid additives used in a composition (boric acid, glass powder) is a percentage of the mass of ash used (10% boric acid equals 1 part boric acid for 10 parts ash).
- For samples made with glass (5% and 10% samples) as replacement of boric acid and mixed with non-calcined ash, a water ratio of 0.35 was used for most. These cubes were made before we found a water ratio of 0.25 to be adequate for casting.
- Water absorption was recorded based upon 24-hour immersion of pieces of compression cubes in water. The percent water absorption is the weight of retained water (weight gain after immersion) compared to the overall weight after immersion.
- Compacted cubes with calcined ash were made using ball clay or carbowax (PEG 8000) as a binder.

Water Absorption Tests

Water absorption tests were also run on pieces of samples from large-scale panels 6-10 and depth of sintering blocks 1-5. The results of these follow in Table 11.

Table 11. Results of water absorption tests performed on samples from second pour and the 5 large blocks.

Sample	% Water Absorption
Panel 6	31.9
Panel 7	30.8
Panel 8	22.3
Panel 9	21.3
Panel 10	28.6
Block 1 (10% boric acid cast)	33.3
Block 2 (10% boric acid cast with chases)	30.9
Block 3 (10% boric acid pressed)	33.8
Block 4 (10% glass powder cast)	23.6
Block 5 (10% boric acid, calcined ash, cast)	11.6

Analysis of Results

The samples that recorded the highest compressive strength were those that were cast with calcined ash and 10% boric acid, with a three day sintering regime at 700°C resulting in a strength of 2770 psi. A one day sintering regime at 700°C resulted in the second highest recorded strength of 2642 psi. The next strongest composition was recorded to be a compacted calcined ash sample with 10% boric acid and clay sintered at 700°C for 1 day, with a strength of 1725 psi. This was followed in strength by a cast sample with non-calcined ash and 10% boric acid sintered at 800°C for 1 day (1718 psi) and a compacted calcined ash sample with 10% boric acid, 10% glass powder, and clay sintered at 700°C for 1 day (1594 psi). The weakest samples were samples made with the glass powder. Of samples that recorded an ultimate compressive stress of less than 400 psi, 7 out of 10 contained glass in the composition. The last sample was a compacted 10% boric acid sample with carbowax. The sample made with 10% glass powder and a .25 water ratio was significantly stronger than the 10% glass powder sample with a .35 water ratio. Even with the strength increase the glass powder samples was much weaker than the samples made with boric acid. The immediate conclusion is that calcining the ash prior to mixing with boric acid samples results in the strongest compositions and using glass in place of or with boric acid decreases the strength significantly in several cases.

Investigation of Sintering Conditions

In regards to sintering temperature, increasing the temperature to 800°C was found to result in strength gain, where strength nearly doubled for cast samples with 10% boric acid and was over 7 times higher for samples cast with 10% glass. However, increasing the temperature further to 900°C decreased the strength in boric acid samples. These 900°C cubes showed significant deformation and bubbling upon firing, and some surfaces appeared glassy. Because of the limits of sintering large-scale panels in the steel

molds in which they are cast, 700°C was chosen as the typical sintering temperature for most samples.

Increasing the duration of sintering was also found to increase the ultimate compressive strength of samples, except for one case, although to a lesser degree than increasing sintering temperature to 800°C. A 23% increase was recorded for 5% glass samples when duration of sintering was increased from 1 day to 3 days. A 13% increase under the same conditions was seen in samples with calcined ash and 10% glass. Increases in strength were also recorded in calcined ash with 10% boric acid samples (5%) and compacted samples with 10% boric acid (2%).

Investigation of Glass vs. Boric Acid as a Flux

Because a water ratio of 0.35 was initially used for samples with glass powder in place of boric acid, a direct correlation between 10% boric acid and 10% glass cannot be made, because a larger water ratio has been shown previously to yield less strength. However, trends can be seen with the use of glass in the composition. Glass cubes made with calcined ash demonstrated much lower strength than the boric acid cubes made with calcined ash. In particular, cubes cast with calcined ash and 10% boric acid were almost 9 times stronger than 10% glass composition cubes with calcined ash mixed and sintered under the same conditions. Therefore, because glass cubes made with non-calcined ash were drastically lower in compressive strength when compared to boric acid cubes made with non-calcined ash, it can be assumed that a glass cube of water ratio 0.25 would still yield a much lower compressive strength than a boric acid cube (as corroborated by test results of block depth of sintering tests 1 and 4). After testing a sample made with 10% glass powder at a 0.25 water ratio it can be seen that decreasing the water ratio does increase strength, but not to the levels that are attained by the samples using boric acid.

Investigation of Calcined vs. Non-calcined Ash

In cast samples with 10% boric acid, calcined ash samples were 3 times stronger than non-calcined ash samples. An increase in compressive strength was also seen in 10% glass samples from non-calcined ash to calcined ash. When comparing the strongest non-calcined 10% glass sample with the strongest calcined 10% glass sample we see that the calcined sample is nearly 4 times stronger than the non-calcined sample. However, in samples made with 10% boric acid and 10% glass, a decrease in strength was found when calcined ash was used instead of regular ash. A decrease in strength was also found in compacted samples with 10% boric acid and carbowax, although compacted samples with the same compositions but clay instead of carbowax more than doubled their strength when calcined ash was used.

Investigation of Cast vs. Compacted Panels

In samples with 10% boric acid, the compressive strength decreased by 29% when the sample was compacted instead of cast. For samples composed of calcined ash, 10% boric acid, and 10% glass, the compressive strength increased for both compacted samples that included clay or carbowax. Both compositions showed a significant increase (130% increase for carbowax and 330% increase for clay). In comparisons between compacted cubes with clay and carbowax, an increase of 350% was found in samples with boric acid

and an 88% increase was recorded for samples with boric acid and glass when clay was used as a binder instead of carbowax.

Investigation of the Effects of Superplasticizer

The effect that the superplasticizer ADVA Flow has on decreasing the composition's water ratio is undetermined. In investigations casting without ADVA Flow, samples were found to be castable at approximately the same water ratio as samples cast with ADVA Flow. Therefore, the role of ADVA Flow may be minimal. Further investigation is needed.

Investigation of Water Absorption

Of the samples with water absorption measured at less than 20%, 9 of the 11 compositions were made with calcined ash, with Block 5 (10% boric acid, calcined ash) demonstrating the least amount of water absorption. Because the carbon is burned out prior to mixing, the porosity of the samples is decreased and a smoother cross section is achieved, allowing for less water absorption. The other two samples of the 11 were cast boric acid samples that were sintered at 800°C and 900°C. Increasing the firing temperature increased the degree of sintering, and as the samples approached the glass transition state, the interiors became less porous and less susceptible to water absorption. The boric acid acted as a flux and allowed the sample to approach this state at lower temperatures than comparable samples with glass did. Consequently, cast samples with glass instead of boric acid recorded higher water ratios than those with boric acid.

Conclusions

The specimen quality was found to be higher in nearly all samples with glass powder in the composition. Specimens with glass were more easily cast and removed from molds, the surfaces were smooth, and the edges were crisp. Samples with boric acid had less uniform edges and surfaces and greater difficulty was experienced removing them from the molds. Cast calcined ash samples, particularly those with boric acid, were found to be the lowest quality in terms of surface uniformity and degree of difficulty in the de-molding process. For compacted samples, those made with clay as a binder showed much smoother surfaces and crisper edges in addition to being more easily de-molded when compared to samples made with carbowax as a binder.

These investigations found that calcining ash typically increases the compressive strength while also providing the lowest water absorption and the lowest water/solids ratios necessary for casting. The presence of boric acid in the composition results in stronger samples than glass powder compositions, although the presence of glass provides increases in the quality of cast samples, including smoother surfaces, sharper edges, and less difficulty in removal from molds. Compacted samples with clay as a binder prove that high strength can be achieved through compaction. Increasing the sintering duration and temperature increases compressive strength and decreases the water absorption, although 800°C appears to be the upper limit before strengths begin to decrease with increasing temperature.

Investigation into Insulative Properties

Bricks measuring 4”x 6”x 2” were made for use in hotbox insulation tests. The bricks were dried in the same manner as compression cubes and sintered at 700°C for 1 day. Table 12 lists the bricks and their respective R-values for thermal resistance. Bricks were made with non-calcined ash, straw, and ADVA Flow unless otherwise noted. The pressed brick was formed by filling a wooden mold that had removable, and tall, sides with an amount of mixture that would make a brick. The material was then compacted using a hydraulic concrete cylinder compressor that loaded a wooden flange with flat sides into the mold to press the sample in one lift.

Table 12. Results of thermal tests on samples made after second pour.

Sample	R-Value (F.ft ² .h/Btu)	K-Value (W/m.K)
10% glass powder, 0.25 water/solids, cast	7.626	0.0386
10% boric acid, 2% carbowax, 0.10 water/solids, compacted	7.209	0.0408

Fabrication of Panels 11-15

After completing the testing and analysis of the cubes, the composition of the next five 46” x 94” x 6” fly ash the panels for the third pour of large-scale panels at Magneco Metrel were determined. Although calcining the ash prior to mixing proved to reduce the water requirement while providing the highest strength, it was determined that calcining on a large scale was not economically feasible. It seems this process is very costly and results in some problems, including clumping of the ash due to calcining. In addition to this, although calcined samples proved strong, they still demonstrated significant problems with shrinkage cracking. Therefore, it was decided that the third pour would concentrate on comparing glass powder in place of the boric acid and cast panels compared to compacted panels. Samples of each mixture were taken for testing of compression, insulation, bending, and water permeability. Flow rate was not tested for in this pour. Compositions are recorded in Table 13.

Table 13. Composition of panels from the 3rd pour at Magneco Metrel.

Panel	Fly Ash	Water/Solids Ratio	Gallons of water used	Additives	
11	Cast	~825 lbs	24%	26.5	80 lbs boric acid 2 gallons ADVA Flow, Straw
12	Cast	~870 lbs	24%	27.5	80 lbs glass powder 2 gallons ADVA Flow, Straw
13	Cast	~1100 lbs	14%	27.5	40 lbs glass powder 480 lbs lightweight aggregate 50 lbs ball clay 2 gallons ADVA Flow, Straw
14	Compacted	~1100 lbs	9%	13	80 lbs boric acid 25 lbs ball clay 2 gallons ADVA Flow, straw
15	Compacted	~1100 lbs	10%	14	80 lbs glass powder 25 lbs ball clay 2 gallons ADVA Flow, straw

Testing of Samples Taken from Panels 11-15

Investigation into Firing Time & Temperature

The lab director of Magneco Metrel thought it would be best if the panels from the third pour were fired at 1500 °F for a period of 10 hours at temperature (Figure 6). The small samples from the third pour were fired at approximately the same temperature (700° C) for 10 hours at temperature so that we could make a good comparison between the large-scale panels and the smaller lab samples. After firing, the lab samples all seemed to be well fired and sintering. There was some degree of carbon still present in the compression cubes, but for the most part sintering had occurred throughout the cubes. The large 10” diameter discs made using a hammer press (Figure 7) were fired through as well.



Figure 6: Panels 11-13 from 3rd pour.

Figure 7: Compression disk made from panel 14 and 15 materials.

Compression Tests on Samples from Magneco Metrel 3rd Pour, Panels 11-15

Investigation into Boric Acid vs. Glass Powder as a Flux

After testing the samples made from the third pour at Magneco Metrel, it is still evident that samples made using boric acid as a flux rather than glass are much stronger in compression. Cubes from panel 11 performed the best with a maximum load 6.3 times stronger than the next strongest sample and 22.1 times stronger than the weakest sample. Samples 11 and 14 outperformed their glass counterparts significantly.

Investigation into Cast vs. Compacted Fly Ash

By comparing the cast boric acid sample with the compacted boric acid sample and the cast glass powder sample with the compacted glass powder sample it can be seen that the cast samples are stronger than the compacted samples. This could be in part due to the method of compaction since all compaction for the compression cube was done by hand in the molds. It is also very evident that compacted samples are much more fragile even after being fired and are quite dusty. This is probably due to the lower water ratio in the mix. Compressive results are shown in Table 14.

Table 14. Results of compressive tests and water absorption tests on samples made during the 3rd pour.

Sample Composition	Water Ratio	Sintering Conditions	Ave. Max. Load, KN	Ave. Max Load, lbs.	Ave. Max Stress, MPa	Ave. Max Stress, psi	% Water Absorption
Cube 11 10% Boric Acid	0.24	700°C 10 hours	27.76	6240.7	10.8	1560.2	24.1%
Cube 12 10% Glass Powder	0.24	700°C 10 hours	1.369	307.8	0.53	76.9	23.3%
Cube 13 Glass, LWA, Clay	0.14	700°C 10 hours	4.425	994.8	1.7	248.7	18.3%
Cube 14 Boric Acid, Clay Compacted	0.09	700°C 10 hours	2.635	592.4	1.3	182.8	34.6%
Cube 15 Glass Powder, Clay Compacted	0.10	700°C 10 hours	1.255	282.1	0.51	74.2	28.8%

Thermal Tests on Insulation Bricks of Panels 11-15

According to insulation test results recorded in Table 15, the samples have a relatively low R-value, and samples made with glass powder exhibit a higher R-value than those made using boric acid. Again there is the exception of Sample 13, which seems that have the lowest R-value of all, this may be due to the light weight aggregate since that it the main difference from the other samples. It would also appear that compacted samples, which were made with a lower water to solids ratio thus making them less porous, have a higher average R-value than cast samples. As is, the fly ash panels offer very little insulation value, but they do allow for a long thermal lag between the different temperature zones, which, if used correctly, could be useful for getting higher energy efficiency within a temperature controlled structure. This is the same principle employed with adobe brick, clay brick, or other high mass earthen building materials.

Table 15. Results of thermal tests performed on samples from panels 11-15.

Sample	R-Value (F.ft ² .h/Btu)	K-Value (W/m.K)
Sample 11 10% Boric Acid	5.934	0.0496
Sample 12 10% Glass Powder	7.449	0.0395
Sample 13 Glass, LWA, Clay	5.707	0.0516
Sample 14 Boric Acid, Clay Compacted	6.919	0.0426
Sample 15 Glass Powder, Clay Compacted	7.626	0.0347

Water Absorption Tests on Samples from Panels 11-15

Since the samples were fired at 700° C, a temperature that was shown in earlier tests to cause the fly ash to take on ceramic properties, none of the intact samples dissolved in water. (Samples from panel 14 did not de-mold intact.). Results are shown in Table 16.

Table 16. Results of water absorption test on samples made during the 3rd pour.

Sample	% Water Absorption
Panel 11	24.1%
Panel 12	23.3%
Panel 13	18.3%
Panel 14	34.6%
Panel 15	28.8%

Investigation into Cast vs. Compacted Panels

It appears that the compacted samples (figure 9) had higher water absorption than the cast samples. This could have to do with the lower water ratio of the mix and the much more powdery nature of the compacted samples after firing. Again the exception to this was sample 13, which had much lower water absorption than all other samples. This could be the result of having the highest clay content and also containing less ash to absorb water because of the volume taken up by the lightweight aggregate.

Investigation into Boric Acid vs. Glass Powder as a Flux

Without good results from sample 14 it is hard to say whether or not the boric acid samples absorbed more or less water than the glass samples. Using the available results from 14 and 15, and comparing them to the results of 11 and 12, it would appear that the boric acid samples absorb less water after firing than the glass powder samples.

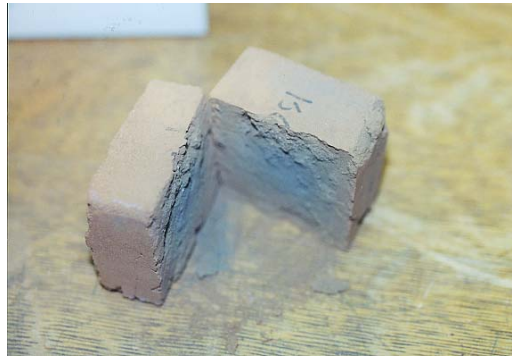


Figure 9: Compression cube from panel 15 came apart along grain boundary formed when sample was compacted.

Bending Tests

Tests performed on the bending bars made from the same ash mixtures used for the third pour at Magneco Metrel and for all the samples from the third pour were tested using a three kip load cell and a three point bending frame. Each stick was supported three inches from center on either side and loaded in the center. After making the bending sticks from the ash used in the third pour we found that sticks made from batches 14 and 15 were too fragile to remove from the molds in any form other than dust. We did obtain three good samples from batches 11, 12, & 13 as well as samples made from a compressed disk made on a hammer press at Magneco Metrel. This disk was made from ash from pours

14 and 15 and should at the least give some idea as to the bending strength of compacted samples. Results are shown in Table 17.

Table 17. Results of bending tests performed on samples made during the 3rd pour.

Specimen	Mechanical test	Max. Force (lbs.)	Max. Stress (psi)	Displacement at failure (in)
11	Bending	59.051	55.58	.024
12	Bending	3.459	2.91	.015
13	Bending	9.049	8.04	.009
Average of all samples from compressed disk	Bending	13.438	7.24	--

Investigation into Boric Acid vs. Glass Powder as Flux

Using the first three samples (11, 12, & 13) again we see that samples made using boric acid as a flux creates much stronger samples than those that use glass powder. Sample 11 was the only boric acid sample among the three and it ended up taking a max load 6.5 times stronger than 13, the next strongest glass sample, and 17 times stronger than 12, the weakest glass sample. Samples 14 & 15 were not used for this comparison since it can not be determined what they contained since they were made from a mixture of the ash from both panel 14 and 15.

Investigation into Cast vs. Compacted Panels

Whether the sample is cast or compacted does not seem to have a great affect on the bending strength of the samples. Aside from the major outlying bending stress obtained by the cast boric acid sample all the rest of the samples, cast and compacted, are relatively similar. It would seem that since the material has such a low bending strength anyway that being cast or compacted has little difference on the strength. It is much more evident that the addition of boric acid instead of glass powder has a much more definite effect on the strength.

Samples from Panels 11 – 15

The final set of panels produced at Magneco Metrel was fired at approximately 1500 °F for 10 hours. These samples turned out somewhat different than the previous panels. They seemed to exhibit much better sintering, most likely due to the longer firing time. They lost a considerable amount of size during firing, but were very dense and quite heavy. This is strange since these panels contained less water than previous samples. Noticeable shrinking did not occur in samples fired in the lab, which were fired for the same amount of time and at approximately the same temperature. This shrinkage made it much easier to remove the panels from the mold and we did not have to resort to the banging and shaking that we did on the previous attempt to remove panels from their molds. The only other major difference between these panels and previous panels was the addition of more ADVA flow. The high firing temperature and time caused the molds to deform a lot. This caused major cracking and breaking of the panels. Without this deformation of the steel molds it appeared that at least some of the samples would have come free of the molds completely intact. If the reason for the shrinkage could be found and controlled it could prove useful in production of full size panels (a method similar to

terra cotta perhaps). As for the deformation of the steel molds switching to a ceramic mold or firing shelf that can take temperatures up to 800°C should solve this problem.

Investigation into Cast vs. Compacted Panels

There was a definite difference between the cast and compacted panel samples after firing. The cast samples were mostly intact except for major cracking caused by the aforementioned deformation of the steel molds. The compacted samples on the other hand were in very bad shape even where serious deformation of the molds was not present. Panel 14 was very flaky and came apart in layers. Panel 15 was in the worst condition, when we attempted to dump it from the mold it fell apart into small pieces (see figure 10). There were only a few sections that were larger than an inch or so in all dimensions. This was probably due to the method of compaction used. The soil tamper did not provide a uniform compaction and did not provide adequate compaction to keep the samples together. After firing and testing a sample made with a hammer press at Magneco Metrel it was seen that compacted samples could yield strength and stability if compacted enough. At this point it seems that cast samples work the best, but if more sufficient methods of compaction on a large scale could be employed then compaction may prove to be viable.



Figure 10: Panel 13 just before being dumped on left, note deformation in steel mold and loss of weld integrity; right: Panel 15 as it is being dumped off of the mold. This panel shattered into small pieces.

Testing of Core Samples Taken from Panels 11 & 12 from the 3rd Pour.

Useable samples were obtained only from two of the 5 panels from the third pour. While 11, 12, & 13 held up well after firing, only samples from 11 & 12 could be drilled or cut because the aggregate in panel 13 caused it to break apart and crumble when we attempted to cut or drill it. Samples from panels 14 & 15 were unobtainable due to their very flaky and brittle condition. They seemed to have very little strength and could be crushed or pulled apart with bare hands. After testing, (Table 18), it was evident that the samples from the 3rd pour were considerably stronger than the samples taken from the 2nd pour. The boric acid samples from panel 11 were over 28 times stronger than the panels from the 2nd pour. The glass powder samples were also stronger than the samples from the 2nd they were nearly 2 times stronger. This shows that an increase of firing time can contribute to a significant increase in strength since the panels from the 3rd pour were fired twice as long (10 hours) as the panels from the 2nd pour (5 hours). The lower water content upon casting could also account for the increase in strength.

Investigation into Boric Acid vs. Glass Powder

Since we were able to obtain samples from both panels 11 and 12 we can compare a boric acid panel with a glass powder panel. As with all other tests the boric acid samples significantly outperformed the glass powder samples. The boric acid samples were over 14 times stronger than the glass powder samples. They also exhibited a much more solid interior with better sintering. The interior of the boric acid samples was hard and solid, while the interior of the glass powder sample was still soft for most of the sample. This was strange since the glass powder samples showed a much more significant color change throughout the sample than the boric acid did. The boric acid sample had a very dark red/orange crust that only penetrated about 1/8 of an inch from the surface. The crust was better intact than those of the samples from the second pour, there were no signs of the crust separating from the rest of the sample and the entire sample seemed to be a similar hardness. There was also a difference in the coloration of the interior (middle) of the two panels. The boric acid panel was very dark black all the way through, while the glass powder samples had a rusty color penetrating from the top and to some degree from the bottom and a light gray middle section. Another note is that while capping the samples with plaster the glass powder samples absorbed most of the water from the plaster. This was not noticed in the boric acid samples.

Table 18. Results of compressive tests on core samples taken from panels made during the 3rd pour.

Specimen	Core	Max. Load (lbs.)	Average Max Load	Max. Stress (psi)	Average Max Stress
Panel 11	1	63419	55713.33	5742.39	5044.67
	2	50486		4571.35	
	3	53235		4820.26	
Panel 12	1	4152.8	3810.03	376.02	345.02
	2	3579.3		324.21	
	3	3698.0		334.84	

Tasks Added to the Proposal After Project Started; Loss on ignition studies to correlate carbon content with strength and studies on depth of sintering

Loss on Ignition Study

Test cylinders were made from three different sets of fly ash used at different times throughout the life of the project. The color of these ashes varied greatly and so therefore the chemical contents would vary including the amount of carbon. It was important to know the influence of the amount of carbon on the strength properties. The first set of fly ash came from preliminary tests done in the summer of 2000 before ICCI funding. The second set of fly ash was used in pre-panel investigations done at the Materials Research Lab at the University of Illinois Urbana-Champaign. The third set of ash was used in the production of building panels 1-10 created at Magneco Metrel. The cylinders were made according to common water/solids ratios and a percentage of boric acid used in preliminary investigations and in panels. Therefore, the only variable between sets was

the type of fly ash used. The cylinders were then tested for compressive strength, (see Table 19), in an effort to correlate strength to loss on ignition, which is a function of unburned carbon present in the ash. (Loss on ignition indicates the weight loss of a sample during firing of the sample. In fly ash it is an indication of the amount of carbon that was present and was burned off.) The cylinders used for this test measured 1” diameter x 2”.

Table 19. Results of compressive strength tests on cylinders.

Fly ash type	Water/solids	Boric acid	Ave. Max Stress (MPa)
Summer 2000 (Old)	23%	10%	13.8
Summer 2000 (Old)	28%	10%	16.3
Spring 2001 (Red)	23%	10%	21.0
Spring 2001 (Red)	28%	10%	14.0
Magneco Metrel (MM)	23%	10%	1.57
Magneco Metrel (MM)	28%	10%	1.33

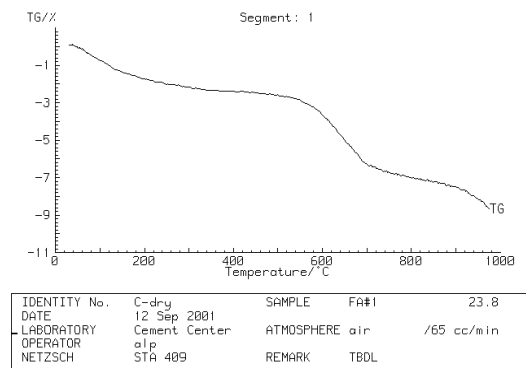
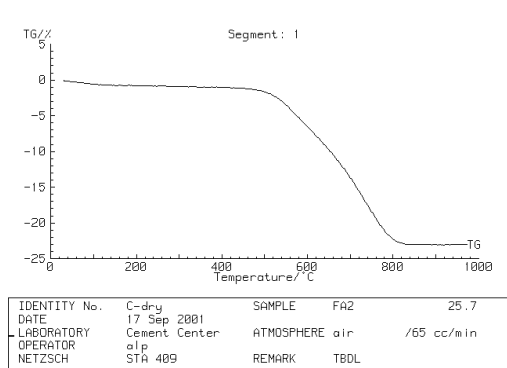


Figure 11: Loss on ignition of “MM” ash, FA2, which had high carbon content.

Figure 12: Loss on ignition of the “red” fly ash (FA1), which had low carbon content less than MM ash.

From the LOI data in Figure 11, the Magneco Metrel fly ash (labeled FA2 in LOI graphs) had a high loss on ignition of about 1.5% at 550°C and a total of 23.0% at 900°C. This fly ash was visibly darker and grainier than the previous fly ash used, due to the increased carbon. The other two sets of fly ash were similar in texture and color (red-brown) while also being less dense and finer than the Magneco Metrel fly ash; their LOI data are also similar and therefore had similar amounts of carbon.

Table 20. Loss on ignition for three types of ash.

Type of Fly Ash	Loss on Ignition at 550°C, %	Loss on Ignition at 900°C, %
“Old “ ash, FA3	4.5	8.4
“Red ash” FA1	2.75	7.5
Magneco Metrel ash, FA2	1.5	23.0

The Magneco Metrel ash had a very high LOI loss on ignition, indicating that it had lots of unburned carbon. It formed an extremely weak composition compared to the other two sets of ash. Based on these results, the conclusion can be made that the high LOI results in a lower strength composition. The high carbon content also made this ash grainier, darker, and denser, and it required more water to reach a flowable composition because it was more water retentive. The other ash was finer, redder, and less dense, and it required less water to achieve a flowable mixture, all due to the reduced presence of carbon. This variability in carbon content accounts for the discrepancies in water ratios in the lab samples and the panels. The panels with a higher content of carbon required higher water ratios than determined in preliminary lab samples.

Experimentation to Determine Depth of Sintering in Blocks

Fly ash blocks measuring 12" x 12" x 6" were made at the University to test for depth of sintering. These larger scale samples were considered representative of the full-scale panels being produced at Magneco Metrel, with the panel and sample thickness being equal. The blocks were made in a wooden mold with removable sides, and the material was cast or compacted around a small tube to allow for a thermocouple to be placed inside the center of the sample. The blocks were air cured, dried at 100°F and 200°F, and fired in a furnace with a ramping rate of 6°/min for approximately 72 hours at ~700°C. Measures were taken to prevent surface flaking (spalling) during the drying process. Temperatures from the inside of the furnace and from inside of the sample were recorded from the time the furnace was started until the time the inside sample temperature equaled the inside furnace temperature. This data was used to produce a heat transfer rate function in order to determine the rate of the depth of sintering. This information was crucial in determining the sintering regime of the full-scale panels. Information on each block is in Table 21.

Table 21. Composition and Comments on Depth of Sintering Test Blocks.

Block	Water/Solids	Additives	Comments	Post-Sintering
1	0.32	10% boric acid ADVA Flow Straw	Cast Non-calcined ash No tubes	Surface flaking (due to drying regime); Red-brown color
2	0.37	10% boric acid ADVA Flow Straw	Cast Non-calcined ash 2 steel tubes	Surface cracks above tubes; Red-brown color
3	0.15	10% boric acid ADVA Flow Straw	Compacted Non-calcined ash No tubes	Small surface cracks; Orange-brown color
4	0.25	10% glass powder ADVA Flow Straw	Cast Non-calcined ash No tubes	Surface clean, smooth, and hard; Violet color
5	0.22	10% boric acid ADVA Flow Straw	Cast Calcined ash No tubes	Severe surface cracking, although hard; Orange color



Figure 13: Block 1 fired at 700 °C (shown with thermocouples).

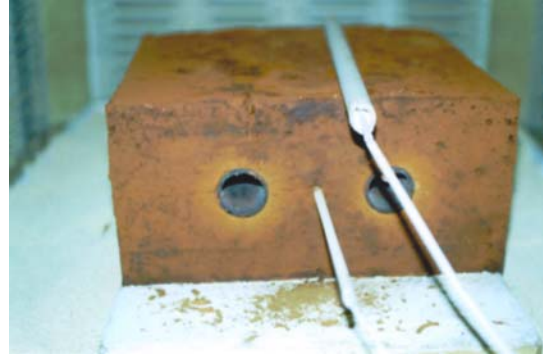


Figure 14: Block 2 sintered at 700 °C with cast-in chases.

Graphs were made from the data of internal block temperature, furnace temperature, and time in order to analyze the depth of sintering of the fly ash samples (figure 15). The time it took the internal temperature of Block 1 (without metal chases) (Figure 13) to reach the inside furnace temperature of 700°C was longer than the time it took the internal temperature of Block 2 (with metal chases) (figure 14) to reach the same final temperature. Therefore, the presence of the metal chases allows the sample to be fired for a shorter period of time, although cracking above the chases occurred, possibly due to differential heating and drying of the metal and the fly ash. A block with non-metal chases might prevent this cracking. It can be seen from the charts that the interior temperatures of the blocks do reach the maximum furnace firing temperature. It takes longer in the block without chases. However the ones with chases may have differential heating and cooling rates between metal as ash material hence the cracking over the metal chases.

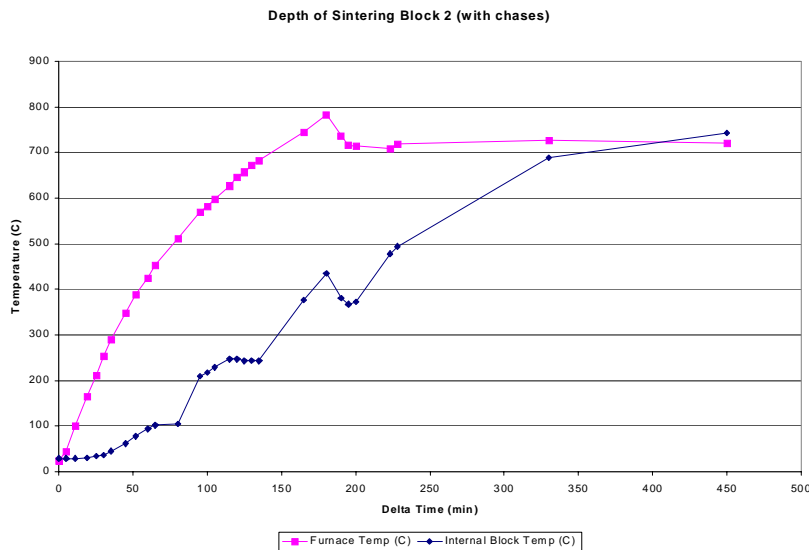


Figure 15: Graph comparing the furnace temperature with the internal temperature of block 2, over time.

Mechanical Testing of Fly Ash Blocks for Depth of Sintering Experimentation

Each of the fly ash blocks was tested for bending strength and compressive strength after sintering. Displacement at failure was also recorded during the bending strength tests. The results are in Table 22.

Table 22. Results of compressive and bending tests performed on large blocks.

Specimen	Mechanical test	Max. Force (lbs.)	Max. Stress (psi)	Displacement at failure (in)
Block 1- Cast, no chases	Compression	9,330	259	---
	Bending	2,340	32.5	0.276
Block 2- Cast, with chases	Compression	2,930	81.4	---
	Bending	1,330	18.5	0.162
Block 3- Pressed, no chases	Compression	4,850	190	---
	Bending	862	12.0	0.197
Block 4- Cast, 10% glass	Compression	6,611	195.6	---
	Bending	166.2	4.71	0.0163
Block 5- Cast, calcined ash	Compression	19,501	866	---
	Bending	2,097	45.1	0.150

Analysis of Results

Upon breaking the blocks through mechanical testing, it was noted that the orange coloration due to sintering only appeared within approximately 1-2 inches from the surface. The interior of the blocks remained gray and dry and somewhat powdery, especially in cast Blocks 1 and 2. Block 3 exhibited better interior consolidation (and consequently the best microstructure of the blocks) of the fly ash, due probably to pressing the specimen instead of casting. However, this block sheared into layers, with each layer being the thickness of one pressed lift. Although it was first believed that the depth of sintering problem was due to the interior of the fly ash specimens not reaching the desired sintering temperature, the results of the depth of sintering temperature analysis on the blocks and the subsequent observations of the interiors upon breaking have shown otherwise. The interior of the blocks did reach the desired sintering temperature of the furnace (and actually exceeded it, which will be discussed later) and was sintered at that state for several hours, although the orange coloration and hardening of the fly ash occurred only at the surface to approximately 1-2 inches from the surface. It was believed that the strength of the ash in the gray and orange areas was equal. A possible scenario for the occurrence of the gray ash was that oxygen was not reaching the interior of the blocks and the carbon was not burned out of the fly ash. The presence of boric acid in the samples may have sintered the surface of the blocks to the glass transition state, sealing off pores that might otherwise allow oxygen to reach the interior of the blocks. As shown previously with the LOI tests, fly ash higher in remaining carbon has less strength and requires more water than does fly ash with less remaining carbon. Therefore, a possible solution for this situation would be to calcine the ash alone before mixing into the composition. Calcining involves sintering the ash in its raw state in an oven to ensure that the remaining carbon is burned out.

Before testing, it was hypothesized that Block 4, with no surface cracking and significant surface hardness, would be stronger than Block 5, with severe surface cracking. Despite the severe surface cracking of Block 5, the calcined block demonstrated the greatest bending and compressive strengths of all of the previous blocks. Furthermore, the interior of the block exhibited entirely uniform sintering throughout, the first block to do so. The large cracks at the surface were found not to continue through the block, resulting in a strong, uniform specimen beyond the large surface cracks. Consequently, Block 5 demonstrated that calcining the raw ash prior to making the sample results not only in a uniform interior cross-section after sintering, but also results in a sample with considerable strength. However, the severe surface cracking remains a critical issue. Block 4, on the other hand, showed no cracking on its smooth surface and appeared to be a strong, solid specimen. Upon testing, however, Block 4 was found to be only moderately strong in compression when compared with the other blocks and the weakest of all blocks in bending. The cross-section still showed a depth of sintering ring as seen in Blocks 1, 2, and 3. Based upon the results of these tests, the initial intuition that Block 4 was to be stronger than Block 5 was proven wrong. These results, however, are corroborated with compressive tests of cubes using the same compositions. Based upon the results of these blocks, the preliminary conclusions made are that calcining ash prior to mixing results in a strong sample with uniform sintering; and using glass in place of boric acid results in a well-formed, smoothed surface sample, although quite weak.

CONCLUSIONS AND RECOMMENDATIONS

Ten large panels, four feet by eight feet by six inches were produced at an industrial facility and numerous smaller samples were made in the lab; these included three one foot by one foot by six inch blocks. The parameters investigated at the partner's industrial facility included refinement of the molds, additive use such as inexpensive fibers to reduce drying shrinkage, and pressure rather than mixing to reduce water use. In addition, lab studies included loss on ignition of the various ash mixes correlated with strength data, thermographic analysis to measure interior heat soak rates and studies of heating regimes.

The main findings were:

- Sintering and the addition of boric acid both help sequester metals in the ash product.
- Large panels without cracks can be fabricated in the factory.
- Use of fiber additives reduces drying shrinkage cracks to manageable limits.
- The amount of carbon in the ash affects the strength; more carbon weakens the product and requires more water for mixing.
- There is no problem in heating and sintering all the way through panels.
- Heating to above 200°C (to avoid out-gassing) in a moist chamber eliminates surface spalling.
- changed economic state of the U.S. will impact that effort.

DISCLAIMER STATEMENT

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FINAL EQUIPMENT INVENTORY REPORT
November 1, 2000, through July31, 2001

Project Title: **A PROPOSAL TO COMMERCIALIZE A SINTERED
COAL ASH/FLUX MATERIAL FOR BUILDING PANELS**

ICCI Project Number: 00-1/3.1B-1

Principal Investigator: Carolyn Dry, University of Illinois at Urbana Champaign

Project Manager: Francois Botha, ICCI

LIST OF EQUIPMENT PURCHASED

None

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