**Pilot Demonstration of Technology for the Production of High Value Materials
from the Ultra-Fine (PM_{2.5}) Fraction of Coal Combustion Ash**

Semi-Annual Status Report

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Abstract

Work on the project focused on the determination of the hydraulic classification characteristics of the Coleman and Mill Creek ashes. The work utilized the hydraulic classifier developed earlier in the project. Testing included total yield, recovery of $<5\text{ }\mu\text{m}$ ash diameter particles and LOI partitioning as functions of dispersant dosage and type, retention time and superficial velocity. Yields as high as 21% with recoveries of up to 2/3 of the $<5\text{ }\mu\text{m}$ ash fractions were achieved. Mean particle size (D_{50}) of varied from 3.7 to 10 μm . The ashes were tested for there pozzolanic activity in mortars as measured by strength activity index using ASTM criteria. Additional testing included air entrainment reagent demand and water requirements. The classified products all performed well, demonstrating excellent early strength development in the mortars. Some increased air entrainment demand was noted. The conceptual design of a process demonstration unit PDU was also completed. A flexible, trailer-mounted field unit is envisioned.

Executive Summary

Project efforts focused on: the determination of the hydraulic classification characteristics of the Coleman and Mill Creek ashes; the evaluation of the pozzolanic characteristics of the products of hydraulic classification of these ashes, and the conceptual design of the process demonstration unit.

The lamella classifier, developed earlier in the project, was used in the hydraulic testing of the Coleman and Mill Creek ashes. Testing criteria included total yield, recovery of $<5\ \mu\text{m}$ ash diameter particles and LOI partitioning as functions of dispersant dosage and type, retention time and superficial velocity. Dispersants, representative of two chemical families were tested and included a commercially available sulfonated naphthalene formaldehyde condensate and polycarboxylate. Product yields of up to 21% of feed solids were achieved with recoveries of $<5\ \mu\text{m}$ particles as high as 64%. Retention times were varied from 20 to 30 minutes and superficial velocities from 4 to 5.5 cm/min. Mean particle sizes (D_{50}) of the products varied from 3.7 to 10 μm as functions of dispersant dosage and retention time. However, the relative fineness of the ash feed to the classifier also had a major influence on the product size. Loss on ignition (LOI) was found to disproportionate in the hydraulic classifier, being primarily enriched in the first and second underflows.

Testing of the products from hydraulically classifying the ashes was initiated. Pozzolanic activity, as measured by strength activity index (SI) were determined for the ashes in mortars using ASTM procedures. In general the overall strength index for both the Coleman and Mill Creek materials was excellent with SI values of 90% achieved in 3 days and $\sim 100\%$ in 7 days. Water requirements were also well below that of the control. Additional testing included air entrainment reagent demand. Some increased air entrainment demand was noted. The conceptual design of a process demonstration unit PDU was also completed. A flexible, trailer-mounted field unit is envisioned.

The conceptual design of the ultrafine ash Process Demonstration Unit (PDU) was completed. The feed design basis was 2 tons of raw ash feed per hour. This size of circuit will require it to be located at the power plant to keep ash handling logistics reasonable. The test circuit was designed to be flexible and truck-mounted. The circuit will contain a feed system, primary classifier, froth cells, secondary classifier, thickener and filter.

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Results and Discussion

The results and discussion of progress made during this reporting period are summarized in this section, which is organized by Task and Subtask. For clarity, major accomplishments of each Task or Subtask are highlighted with a narrative description of specific activities.

Task 1 – Feedstock Evaluation.

The objectives of this task are to obtain the necessary data to finalize equipment selection for the PDU design and the reagent requirements to produce high quality filler and super pozzolan from both dry ESP ash and pond ash.

Subtask 1.1 Sample Procurement

Status: Essentially Completed.

Subtask 1.2 Sample Characterization

Status: Largely Completed.

Subtask 1.3 Evaluation of Dispersion Parameters

This task is further divided into two subtasks.

Status: Subtask 1.3.1 Evaluation of Flotation Parameters.

Status, task is largely completed.

Subtask 1.3.2 Evaluation of Dispersion Parameters

Status: Subtask 1.3.2 Evaluation of Dispersion Parameters.

Status: this task is largely completed.

Subtask 1.4 Testing of Processing Parameters

This Subtask is divided further into three Subtasks

Subtask 1.4.1 Testing of Dispersion Parameters.

No activity.

Subtask 1.4.2 Testing of Dewatering

Previously Reported.

Subtask 1.4.3 Environmental Testing

Subtask 1.5 Process Simulation.

Processing of Coleman Ponded Ash and Mill Creek Dry Silo Ash. Bulk samples of ash collected from the dry silo at LG&E's Mill Creek power plant and the western-most pond at WKE's Coleman power plant were tested in the lamella classifier. The development and early testing of this classifier was described in our last report.

Approximately 300 pounds of sample was used. The Coleman materials was passed through a 20 mesh screen to remove plant debris and coarse bottom ash that had been deposited in the pond along with the fly ash over time. The ash was added to a 1200 liter tank and stirred with two 12" impellers driven by 1 ½ horsepower motors through a 1:5 reduction gear box. This feed system fully entrains the ash at pulp densities of up to 17% solids by weight and provides a reasonably consistent feed over time.

The ash slurry is pumped from the feed tank with a peristaltic pump, which provides excellent flow control. The feed slurry is pumped into a plenum which is divided into individual feed tubes. Flow is balanced among the individual tubes via constrictions that are provided from a series of gate valves.

The classifier itself is 150 cm long by 37 cm wide and 43 cm high. It has a volume of ~228 liters, as adjusted for the thickness of the lamella plates. The sidewalls are inclined at 45°. The lower part of the classifier is terminated with 4 cones which neck down to a ½ inch connection. The purpose of this zone is to thicken the underflow. Because it is below the lamella it is not included in the volume or cross sectional values used to calculate residence time and superficial velocity.

The coarse underflow is removed from the classifier via four peristaltic pumps which also serve to lock the unit. The overflow of the classifier passes through a launder system and flows to the product tank. The launder is submerged to provide a still zone for the efficient collection of cenospheres and is vented to prevent siphoning.

Test Conditions. Various dispersants were screened for effectiveness according to the methods outlined in the previous report. Two types of commercially available dispersants were used, a naphthalene-sulfonate-formaldehyde condensate (NSF) based product and a commercial polycarboxylate (PC). The dispersants were added as liquids in the concentrations as prepared by the manufactures (~40% for the NSF and ~20% for the PC).

Table 1. Classifier Tests: Dispersants and Slurry Flows.										
Date	Test	Material	Disp.	Con.	Feed	U1	U2	U3	U4	Prod.
				mg/g	Kg/min	Kg/min	Kg/min	Kg/min	Kg/min	Kg/min
06/21/04	1	Ghent	NSF	1.9	9.1	0.59	0.36	0.36	0.36	7.45
06/21/04	2	Ghent	NSF	1.9	9.0	0.60	0.36	0.37	0.37	7.30
01/05/05	1	Coleman	NSF	2.0	7.1	0.57	0.38	0.38	0.37	5.44
01/05/05	2	Coleman	NSF	2.0	7.0	0.56	0.38	0.38	0.38	5.34
01/12/05	1	Coleman	NSF	0.8	7.2	0.58	0.38	0.39	0.38	5.44
01/12/05	2	Coleman	NSF	0.8	7.2	0.58	0.39	0.40	0.39	5.41
01/20/05	1	Coleman	NSF	1.4	7.3	0.59	0.38	0.37	0.38	5.57
01/20/05	2	Coleman	NSF	1.4	7.3	0.60	0.39	0.38	0.37	5.57
03/09/05	1	Mill Creek	PC	3.0	7.3	0.63	0.42	0.40	0.40	5.45
03/16/05	1	Mill Creek	PC	3.0	8.9	0.68	0.45	0.42	0.40	6.92
03/21/05	1	Mill Creek	PC	3.0	9.5	0.42	0.41	0.42	0.41	8.11

The dispersant conditions and flows are presented in Table 1. Data for tests made of the Ghent material is also presented for comparison purposed. All the dispersants were added to the feed tank at the start of the run with the exception of the last Mill Creek test which employed a metering pump and added the dispersant at the appropriate concentration and flow rate to the feed slurry. The overall feed rate for the Coleman ash was held relatively constant throughout the testing and the dosage of the dispersant was varied from 0.8 to 2.0 mg of dispersant per gram of solid. In contrast, the dosage of the dispersant for the Mill Creek ash tests was held constant while the feed rate was varied.

Table 2. Classifier Tests: Retention and Velocity Parameters and Selected Results										
Date	Test	Material	Feed	Product	R _t	S _v Avg.	5 µm	Rec.	D ₅₀	D ₅₀
			Kg/min	Yield	Min	cm/min.	Kg/min	%5 µm	Feed	Prod.
06/21/04	1	Ghent	9.1	21.0%	30.60	5.24	0.26	63%	21	3.7
06/21/04	2	Ghent	9.0	21.6%	31.03	5.17	0.25	65%	20	3.8
01/05/05	1	Coleman	7.1	14.1%	40.15	4.04	0.11	40%	29	7.6
01/05/05	2	Coleman	7.0	13.4%	40.80	3.97	0.11	37%	31	7.8
01/12/05	1	Coleman	7.2	14.0%	40.06	4.05	0.13	33%	18	9.0
01/12/05	2	Coleman	7.2	13.4%	40.19	4.04	0.13	30%	17	10.0
01/20/05	1	Coleman	7.3	12.8%	39.70	4.08	0.15	29%	21	7.5
01/20/05	2	Coleman	7.3	12.0%	39.61	4.09	0.16	25%	20	7.8
03/09/05	1	MillCreek	7.3	10.0%	39.93	4.06	0.10	33%	32	7.4
03/16/05	1	MillCreek	8.9	12.4%	32.06	5.03	0.16	43%	28	5.4
03/21/05	1	MillCreek	9.5	14.5%	28.76	5.57	0.13	64%	30	5.9

Selected test results along with process parameters are presented in Table 2. Overall, product yield, on a solids basis, varied from 10% to 20% of the feed. The average retention time (R_t) varied from about 30 to about 40 minutes, while the mean superficial velocity (S_v) varied over a narrower range, from about 4 to 5 cm/minute. Both parameters were adjusted by the underflow rates and are averaged across the four cells.

Figure 1. Plot of Cumulative Particle Diameters for the Feed (F) and Products (P) of Hydraulic Classification for the Coleman (C), Ghent (G) and Mill Creek (MC) ashes.

This coarser nature of the products from the Mill Creek and Coleman ashes compared to that of Ghent is related to the coarse nature of the feed (Figure 1). The feed flow of $<5 \mu\text{m}$ material varied considerably over the test series, from a high of 0.25 kg/min for the Ghent ash to as low as 0.10 kg/min for the Mill Creek ash. Almost 20% of the Ghent feed ash has diameters of $5 \mu\text{m}$ or less compared to 13% of the Coleman and 9% of the Mill Creek. In a perfect system, at constant retention time and superficial velocity, the grade of the products should be relatively constant and the primary variation should be in the overall yield. However, in practice this is not the case as inefficiencies in sorting, etc. do influence the product grade. Actually the data suggests that the overall recovery of particles in the 1 to $2 \mu\text{m}$ range was actually higher for the Mill Creek test run from 3/21/05 than that of Ghent tests even though the Ghent products had a lower D_{50} .

As expected, the underflows from the classifier are greatly depleted in $<5 \mu\text{m}$ material (Figure 2). Overall the first underflow (U1) is typically very coarse followed by U2, U3 and U4 underflows. The last two underflows (U3 and U4) tend to be enriched in particles in the 10 to $20 \mu\text{m}$ size range and, for most tests have a D_{50} that is less than that of the feed. In the example presented in Figure 1, the feed had a D_{50} of $28 \mu\text{m}$, with values of 53, 30, 23 and $22 \mu\text{m}$ for U1 through U4 respectively. The product had a D_{50} of $\sim 6 \mu\text{m}$ for the test.

Figure 2. Plot of Cumulative Particle Diameters for the Mill Creek (MC) ash for Feed (F) Product (P) and Underflows (U1, U2, U3 and U4).

Increasing the feed rate for the Mill Creek ash increased the yield of the material by almost 50%. The recovery of the 5 μm , a more important measure here, almost doubled from 33% to 64% of feed. The grade of the product, as expressed as the mean particle diameter on a volumetric basis (D_{50}) did not change in a concomitant fashion, likely due to the very coarse feed in the first test. Varying the dispersant concentration in the Coleman ash test series did not result in a significant difference in yield, which varied from about 12% to 14%. There was a difference in the average grade of the particles which improved with dispersant dosage.

Table 3. Classifier Tests: Results for LOI.								
Date	Test	Material	Yield	LOI Feed Rate	LOI Product Rate	LOI Recovery	LOI Feed	LOI Product
			%					
06/21/04	1	Ghent	21%	0.044	0.005	12%	3.4%	2.0%
06/21/04	2	Ghent	22%	0.048	0.005	11%	3.9%	2.0%
01/05/05	1	Coleman	14%	0.073	0.006	8%	8.9%	5.3%
01/05/05	2	Coleman	13%	0.073	0.006	8%	9.0%	5.1%
01/12/05	1	Coleman	14%	0.066	0.005	7%	8.0%	4.1%
01/12/05	2	Coleman	13%	0.065	0.005	7%	7.9%	4.2%
01/20/05	1	Coleman	13%	0.066	0.005	7%	7.1%	4.0%
01/20/05	2	Coleman	12%	0.064	0.004	7%	6.9%	4.0%
03/09/05	1	Mill Creek	10%	0.021	0.003	14%	2.2%	3.0%
03/16/05	1	Mill Creek	12%	0.026	0.005	19%	2.1%	3.3%
03/21/05	1	Mill Creek	15%	0.030	0.007	23%	2.3%	3.6%

Loss on ignition (LOI) is commonly used as a surrogate for carbon, as it is a reasonably approximation for most Class F ash. The LOI was found to disproportionate in the classification tests (Table 3). For the Ghent and Coleman materials, most of the

LOI went with underflow and the product was substantially lower in LOI than the feed material. This was not the case for the Mill Creek ash, where it tended to have a small disproportionation to the product, although the bulk of the LOI (~80%) went with the underflow. This resulted in an increase in LOI in the product compared to the feed. The causes for this are uncertain; presumably the carbon is finer than typical in this ash. Alternatively, the carbon in the Mill Creek ash may have a relatively stronger affinity for the dispersant compared to the other ashes, which may have resulted in a higher charge and a tendency to resist settling.

Task 2. Pilot/Demonstration Plant Final Design.

Subtask 2.1 PDU Conceptual Design of PDU Facility.

Processing Strategy. The processing strategy that was used in the PDU conceptual design is based on experience obtained while conducting other ash beneficiation projects. The first project entitled “Technology for the Recovery of Fuel and Adsorbent Carbons from Coal Burning Utility Ash Ponds and Landfills” focused on recovering unburned carbon while the second project entitled “Advanced Multi-Product Coal Utilization By-Product Processing Plant” was primarily concerned with recovering additional construction materials. Both of these projects involved excavation of combustion ash from a utility ash pond and processing the excavated ash to recover the specific components. Many of the material handling issues such as feed preparation and rejection of oversize particles will also be encountered when attempting to recover ultra-fine ash (UFA) a.k.a. filler. Similar unit process will be involved in the present flowsheet and the PDU design incorporates strategies and techniques that will hopefully avoid difficulties encountered during the previous projects. Each of the unit processes are described in detail in the following narrative.

Flowsheet Description. The flowsheet that will be utilized for the PDU is shown in Figure 3. The flowsheet has a feed design basis of 2 tph dry solids excavated ash and incorporates the unit processes necessary to prepare the feed as a slurry for primary classification to reject coarse (+100 mesh) material. The -100 mesh fines report to froth flotation, a process which is used to remove fine carbon. Flotation can be by-passed if there is insufficient fine carbon present in the selected substrate. These unit processes (i.e. primary classification and froth flotation) are necessary to prepare the feed for secondary classification in a lamella classifier, a unit process that selectively recovers the ultra-fine ash (UFA) which will be evaluated for use as filler.

It is anticipated that the UFA will have an average particle size of less than 5 μm and a top size of less than 20 μm . The recovered UFA will be in a dilute slurry (<4% solids w/w) so it will be necessary to thicken and filter the slurry to remove most of the water. Dryer design will be based on the moisture content and handling characteristics of the UFA filter cake.

Figure 3. Schematic Diagram of PDU Design for Filler Production.

Flowsheet Considerations. In order to operate the PDU, it will be necessary to conduct testing at the utility site. There are several reasons for this decision, most notably the amount of feed material required. With an operating basis of 2 tph dry feed solids and a design UFA production rate of 0.38 tph dry solids, the PDU will reject over 1.5 tph of ash. Based upon the quantity of feed material needed and the quantity of ash that will be rejected, the only feasible location to conduct testing at this scale is at the utility site where there is more than sufficient feed ash available and the existing ash pond can be use for storage of reject ash.

Conducting testing at the utility site warrants that the PDU must be self-contained and self sufficient since ash ponds are typically located in remote area where electrical power is not available. The PDU will be constructed on two mobile trailers owned by CAER. The first trailer, a 48' flatbed will contain most of the processing equipment and serve as a working platform. The second trailer, a 53' box trailer will contain the froth flotation cells, a workshop and storage for parts. The entire PDU will be assembled at CAER to ensure there is adequate wiring, plumbing and clearance available for all components. All connections will then be labeled, disassembled and packed for transport in accordance with Kentucky highway regulations.

Electrical service will be provided by a diesel generator with capabilities of supplying 440V, 220V and 110V service, all of which will be required to run the PDU equipment. Process water will be pumped from a suitable source using a portable gasoline water pump and stored in a process water sump, which will be filled as needed.

Feed System. The PDU will be fed with moisturized ash recovered from the ash pond. The feed will be pre-screened to reduce the size and remove misplaced bottom ash and

vegetation that can cause valve plugging. The pre-screening will be accomplished using the coarse dewatering screen mounted on the flatbed trailer to remove +3/8 inch (~9.5 mm) material. The coarse dewatering screen will be fed with Bobcat loader onto a 1'X10' conveyor belt and -3/8 inch material will be stockpiled with a 1.5'X26' conveyor. Oversize will be collected and removed using wheel-barrows. In this manner, a sufficient amount of feed material will be generated to conduct all of the anticipated testing.

During PDU operation, the stockpiled feed ash (-3/8 inch) will be fed onto the 26' conveyor with the Bobcat loader. Feed ash will be conveyed into a sloped bottom primary mix tank (500 gallon) along with sufficient water to produce a slurry with the desired pulp density (10-20% solids). The primary mix tank will be equipped with a mechanical agitator (1.5 hp) to keep all solids in suspension. The slurry will also be mixed with a centrifugal pump (120 gpm) withdrawing slurry from the bottom of the tank and re-circulating it to the top of the tank.

The slurry will then be transferred to a second sloped bottom feed tank (500 gallon) by using a by-pass valve on the recirculation line, thus avoiding the need for an additional transfer pump. Before entering the second mix tank, the slurry will be pumped over a coarse screen (6 mesh) to remove any coarse material present. Past experience has shown that the quantity of +6 mesh is almost insignificant (<1% of the feed), but even this small amount can present valve and pump plugging problems. The second mix tank will be agitated with a mechanical stirrer (1.5 hp) and re-circulated with a centrifugal pump (120 gpm) arrangement identical to the primary mix tank. Once all of the feed slurry is transferred into the second tank, the first will be filled, prepared and transferred to the second as necessary.

Primary Classification. The purpose of primary classification is to narrow the size distribution of the ash slurry by efficiently removing essentially all of the coarse (+100 mesh) ash and carbon present. To accomplish this, feed slurry from the second mix tank will be diverted into the primary classifier using a by-pass valve in the recirculation line. The primary classifier is approximately 10' tall and 4 ft X 4 ft at the top. Feed flow rate will be monitored with an ultrasonic flowmeter. The primary classifier will be fed at a rate of 30 to 50 gpm (~110 to 190 l/min) into a feed box located above the classifier. The feed slurry will then flow by gravity at a velocity of 80 to 136 ft/min, depending on the feed rate, and impinge against a diverter plate which forces the slurry upwards into the flared classifier chamber. The flared chamber slows the velocity significantly and particles too coarse to remain in suspension (i.e. +100 mesh) settle and fall behind the impingement plate while fine particles (-100 mesh) remain in suspension and overflow the top of the device via a weir and are collected in a launder. The coarse settled particles are removed from the bottom of the classifier as thickened slurry with a variable speed pump. Coarse ash and/or coarse carbon can be recovered if desired with the coarse dewatering screen.

If the feed slurry contains sufficient middling material (~+80 mesh), it may be necessary to remove accumulated middlings from the flared classification chamber. This is accomplished by withdrawing slurry with variable speed pumps through simple slotted

pipes located in the chamber itself. Any coarse carbon present in the middlings can also be recovered with the coarse dewatering screen.

Froth Flotation. Froth flotation will be used to remove fine (-100 mesh) carbon from the ash slurry. Carbon particles in this size range are too small and have too low a density to be removed by high capacity primary classification, thus it is necessary to use a surface modification technique such as froth flotation. The primary classifier overflow slurry (-100 mesh) will flow into a bank of froth flotation cells. The flotation circuit is equipped with 6 individual flotation cells (8 ft³ each) that operate in series. Each cell is equipped with mechanical level controls to individually control froth depth in each cell. Appropriate flotation collector will be added to render the carbon particle surfaces hydrophobic and frother will be added to produce a consistent froth structure. These reagents will be metered into the air intake of the first flotation cell. Froth is removed from each cell by mechanical scrapes and drops into a common launder while the low-carbon tailings slurry flow from the exit port in the last cell. It is anticipated that only 4 cells will be necessary to provide adequate retention time (4-6 minutes), thus the level controls for the last two cells in the series will be opened and slurry will simply pass through to the exit port. The froth slurry will be returned to the pond for storage while the low-carbon tailings will be collected in a flotation tailings sump.

The flotation circuit is equipped with a by-pass in order to evaluate the circuit without flotation. This may be necessary for a substrate that contains insufficient fine carbon to perform froth flotation efficiently. It may be desirable to by-pass flotation and reject fine carbon during secondary classification in order to avoid the cost of the equipment and reagents used in the flotation circuit.

Secondary Classification. The purpose of secondary classification is to efficiently recover UFA and reject coarse (+20 µm) ash and/or carbon. This will be accomplished in a lamella classifier. Feed for the lamella classifier will be flotation tailings or in the case of by-passing flotation, primary classifier overflow (-100 mesh). In either case, the secondary classifier feed will be pumped from the agitated flotation tailings sump (300 gallons). Dispersant will be metered into the sump and flow rate will be monitored with an ultrasonic flowmeter.

The secondary classifier consists of a rectangular cell (4' X 4' X 8' long) with two pyramid-shaped hoppers below each cell. The rectangular cell contains a series of 30 polyethylene plates inclined at a 45° angle, arranged parallel to the flow (see Figure 4). Feed slurry enters the secondary classifier through a manifold to distribute the feed slurry across the device. The slurry flows in a direction parallel to the lamella plates at a velocity of 10 to 15 cm/sec. Coarse particles (+20 µm) settle while fine particles remain in suspension. The purpose of the lamella plates is to provide a settling surface for coarse particles. The coarse particles settle onto the inclined plates, slide along the plates and are deposited in the pyramid-shaped collection hoppers where they consolidate into a high solids slurry (50% solids w/w) and are removed by variable speed pumps. Without the lamella plates, it would be necessary for +20 µm particles to settle as far as 4 ft to be removed from the suspension. This inordinate settling distance creates turbulence and is

a particularly inefficient method of removing these particles. Laboratory studies have shown that using lamella plates inclined at an angle higher than the angle of repose is a much more efficient means of removing the coarse particles.

The fine particles ($-20\text{ }\mu\text{m}$) remain in suspension and overflow the end of the classifier through a submerged orifice that is vented to prevent siphoning. The purpose of submerging the launder is to prevent cenospheres from reporting with the UFA product. Cenospheres are hollow ash spheres that are generally much coarser than the UFA. Since they are hollow, they have a low density ($<1.0\text{ g/cm}^3$) and readily float and accumulate on the UFA slurry. By removing suspended fines below the surface, cenospheres remain above the UFA collection point where they can be separately removed.

Figure 4. Simplified Diagram of Secondary (Lamella) Classifier.

UFA Dewatering. The UFA slurry is recovered from the secondary classifier as a dilute slurry (<5% solids w/w), thus it is imperative to have efficient solid/liquid separation. The UFA slurry will be flocculated using a high molecular weight polyethylene oxide polymer (PEO). Laboratory evaluations showed that using a dosage of 2.5 ppm Ucarfloc 302 (Dow Chemical) provided adequate settling rate and clarity for efficient solid/liquid separation. The flocculated slurry will be pumped into a 1500 gallon cylindrical tank with a 45° sloped bottom which will serve as a thickener. The flocculated slurry will be distributed through a submerged feed manifold and solids will settle to the base of the

tank where they will be withdrawn as thickened slurry (30% solids w/w). Clarified water will overflow the thickener and be recycled as process water.

The thickened solids will then be dewatered on a rotary vacuum drum filter. The vacuum drum (1.5' diameter X 1' face) will be covered with a commercial monofilament filter cloth (M901) which has been shown in laboratory evaluations to provide acceptable cake moisture (30% solids w/w) and solids capture (>80%). The filtrate will be recycled back to the thickener in order to collect as much of the UFA as possible. The dewatered UFA filter cake will be collected for drying evaluations by dryer manufacturers.

Subtask 2.2 PDU Construction of PDU Facility.

No activity this period.

Task 3. Pilot/Demonstration Plant Operation.

No activity this period.

Task 4. Product Evaluations and Final Economic Evaluation

Subtask 4.1 Commercial Scale Final Design

No Activity this period.

Subtask 4.2 Product Testing.

Subtask 4.2.1 Superpozzolan Testing.

Summary. The overarching objective of this task in the project is to substantially decrease the amount of Portland cement that is utilized in the formulation of concrete while maintaining or increasing the strength and durability of the concrete. Research to-date has focused on the pozzolanic reactivity of ultrafine fly ash in concrete, as a partial replacement for Portland cement. It is our goal that the research will contribute to the production of concrete materials containing high fly ash contents, while exhibiting high performance and durability characteristics.

Previous research at the CAER has indicated that ultrafine ponded fly ash can be very reactive in mortar and concrete and rapidly forms cementitious material that significantly increases the compressive strength of these materials. The fine size and spherical nature of the ash also provides water reduction in mortar and concrete, which contributes to higher strengths. Reported herein are results of testing to evaluate the pozzolanic reactivity and performance of classified fly ash in portland cement-based mortars (a pozzolan is defined as “a siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties”).

Experimental Methods. Ultra fine fly ash filler from the classifying process has been evaluated for its performance in mortar, both in the fresh and hardened state. All testing has been completed in accordance with ASTM C 311 guidelines. Mortar was used to test for water demand, air content, and compressive strength. The only significant deviation from ASTM protocol involved the mortar air test in which Vinsol resin was not used as the air entraining admixture (AEA). The AEAs used were: abietic acid-sodium salt (5.9% solution), dodecyl (laural) sulfate (5% solution), Master Builders MicroAir, and Grace Darex II. Compressive strengths of fly ash mortars are presented as a percent of control mortar, which contains only Portland cement as the cementitious component (i.e. no replacement with fly ash). The fly ash mortars contain a 20% replacement by weight of fly ash for Portland cement.

Results: Water Reduction. Addition of fly ash, as expected, resulted in a reduction in the quantity of water required to achieve constant flow (i.e. workability). The addition of Coleman classified fly ash gave a higher water reduction than the standard Class F Trimble fly ash (Table 4), while the Mill Creek product was similar to the Trimble fly ash. This “filler effect” is primarily caused by the small size and spherical shape of the particles (Dodson, 1990), with a smaller median size generally causing a higher water reduction. Although the Mill Creek product is considerably finer than the Trimble fly ash there was some difficulty in dispersing it within the mortar mix, thus imparting a more “sandy” rheology to the mortar. This is possibly caused by agglomerates of fly ash particles that were dispersed ultrasonically within the particle size analyzer, but were not properly dispersed in the mortar. This problem was not observed for the other fly ashes, and will be investigated by collecting another sample of ash for classification.

Table 4. Water Demand in Mortar to Achieve Constant Flow		
	Median Diameter (microns)	Water Demand (%)
Control	18	100
Trimble Fly Ash	20	97.0
Mill Creek Product	10	97.5
Coleman Product	8	94.0
Ghent Product	4	91.1

Figure 5. Strength activity index of mortar prepared using Mill Creek CP as a partial replacement for Portland cement.

Compressive Strength. Compared to a “standard” quality Class F fly ash, which typically achieves control strength after about 1 month curing, the classified products performed well. Figure 5 provides the results for the Mill Creek classified product (CP) and indicates that control strength was achieved after approximately 10 days curing. The Coleman CP exhibited a higher performance in mortar, achieving control strength after 7 days curing. This is caused by the smaller particle size of the fly ash and the greater water reduction in the mortar; the latter results in a lower water content, which is well known to increase compressive strength of mortar and concrete.

Figure 6. Strength activity index of mortar prepared using Coleman CP as a partial replacement for Portland cement.

Data for a classified product (CP) produced from Ghent ponded fly ash are provided in Figure 7. This material has the smallest median particle size and the greatest water reduction. It can be seen that, because of the water reduction, control strength was achieved within 1 day curing, decreased slightly at 3 days, then increased substantially thereafter. The data are graphically summarized in Figure 8 to elucidate the effect of particle size on compressive strength.

Mortar Air. The ultrafine classified ash products required substantially greater dosages of air entraining admixture (AEA) to achieve 18% air than did the control mortar or even mortar prepared with “standard” fly ash: approximately twice as much AEA was required for the Ghent CP and Coleman CP ash mortars than for the control or standard fly ash mortars (Figures 9 & 10). The higher demand for AEA to achieve a constant air content is typical when fly ash is used as a cement replacement. This is generally believed to be a result of adsorption of the AEA from solution by unburned carbon in the fly ash. To test this, the Coleman CP and Ghent CP fly ashes were ignited in air at 750°C to remove the carbon, and the mortar air test conducted on the ignited product. The results shown in Figure 9 indicate that for the Ghent CP, removing the carbon had no impact on the air entraining properties. This suggests that the high degree of fineness probably caused the lower entrained air content. However, for the Coleman product, removal of the carbon caused a significant increase in entrained air (Figure 10). The reason for the different behavior is not yet known, but will be investigated during the second project year.

Figure 7. Strength activity index of mortar prepared using Ghent CP as a partial repl

Figure 8. Median diameter of classified fly ash products vs. strength activity index, shown for different curing durations.

Figure 9. Air content of mortar prepared with Ghent CP ash. Micro Air is a commercial air entraining admixture (AEA).

Figure 10. Air content of mortar with Coleman CP ash and Micro Air AEA.

Conclusions. Based on the mortar testing and analysis of calcium hydroxide concentration, it is concluded that the ultrafine fly ash obtained by classification of ponded fly ash is a very reactive pozzolan, especially when compared to standard Class F fly ash. In mortar the ultrafine fly ash not only produces high long-term strengths but also achieves control strength within 1 day of curing. However, use of the ultrafine ash in concrete results in a relatively slow strength gain (compared with mortar) that is not understood at this time. The disparity between mortar and concrete results will be investigated during the second year.

Another issue that needs further study is the significantly higher dosage of AEA required to achieve a specified air content. A higher dosage of AEA is not, in and of itself, of great concern as long as it is consistent from one concrete batch to the next. However, if the AEA demand fluctuates greatly from one Ready Mix batch to the next, then that would inhibit the acceptance of the classified ultrafine ash by the industry. Of particular interest here is the lack of dependence of entrained mortar air content on the carbon content of Ghent ultrafine fly ash, whilst removal of carbon in the Coleman product greatly increased the mortar air content. The difference in carbon properties between these two products will also be investigated further.

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