

**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

Reporting Period Start Date: 21 July, 2004

Reporting Period End Date: 20 January, 2005

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Report Issued: 27 June 2005

DOE Contract No. DE-FC26-03NT41726

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Abstract

Work on the project primarily focused on the design and testing of different hydraulic classifier configurations. A four cell, open channel, cross flow classifier with and without weirs separating the cells was evaluated. Drawbacks to this configuration included thick sediment compression zones and relatively low throughput. The configuration was redesigned with inclined lamellae plates, to increase sedimentation area and decreased sediment compression zone thickness. This configuration resulted in greater throughput for any given product grade and enhanced product recovery. A digital model of a hydraulic classifier was also constructed based upon Stokes law and the configurations of the tests units. When calibrated with the size of the ash used in the tests, it produced a reasonable approximation of the size, yield and recovery of the actual product. The digital model will be useful to generate test data, at least on a relative basis, of conditions that are hard to generate in the laboratory or at larger scale. Test work on the dispersant adsorption capacity, settling tests and leaching test were also conducted on materials collected from the Coleman power station pond.

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Executive Summary

Work primarily focused on configuration of the hydraulic classifier during the reporting period. A laboratory scale cross flow hydraulic classifier was constructed to provide data to develop a digital model of this step in the process. The initial configuration was not unlike that of commercially available apparatus. The unit was feed from one end of the unit with dispersed slurry of fly ash at pulp densities typically in the range of 12 to 15% by weight solids. The thickened coarse sediment was withdrawn from the bottom of the unit in four different underflows and the product collected from a submerged launderer at the end of the classifier. Good product grades were achieved with this configuration with average particle sizes (D_{50}) in the 3 μm to 5 μm range.

Attempts were made to improve the overall efficacy of particle separation by increasing the velocity in the initial cells. This was done by inserting weirs of various heights between the cells. This resulted in thicker sediment compression layers. Two different designs that incorporated inclined lamellae plates were tested. This greatly increased the sedimentation area and decreased the thickness of the sediment compression zone. The lamellae classifiers allowed greater throughput while maintaining similar product grades. For example the initial open channel classifier required 60 to 80 minutes of residence time to produce a product with a D_{50} of 3 μm to 5 μm in diameter, while the lamellae classifier could produce the same grade of product in 20 to 25 minutes of average residence time. Product yields for the lamellae tests were in the 15 to 25% range for the tests.

Hydraulic classification was found to be effective in reducing the LOI of the ash. The carbon in the ash was found to disproportionate to the underflow and the product LOI was lower than the feed LOI by a factor of about two. A digital model of the hydraulic classification system was constructed based upon Stokes law, which states simply:

$$V_p = G (\rho_s - \rho) D^2 / 18\mu$$

where V_p is the rising or settling velocity of the discrete particle, G is the gravity constant, ρ_s is the density of the particle, ρ is the density of the carrier fluid, D is the diameter of the discrete particle and μ is the viscosity of the carrier fluid. From this the settling velocity of the particles can be calculated. Stokes law assumes laminar flow and that the particles do not interact with one another. Comparisons of the grades, yields, recoveries and particle sizes of products calculated from the model were compared with actual results. The parameters were reasonably close, which indicates that the model will be a useful tool in simulating difficult experiments and in scaling up the hydraulic classifier design.

Dispersant adsorption and dispersion tests were performed on samples collected from the ash pond at the Coleman power station with NSF (naphthalenesulfonic formaldehyde polymer condensate sodium salt). Total adsorption capacity was measured at ~0.9 g of NSF/Kg ash and 1.4 g/Kg was required to keep the sediment suspended for the 30 minutes as proscribed in the tests. These results were congruent with previous observations.

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 superpozzolan from both dry ESP ash and pond ash.

Subtask 1.1 Sample Procurement

Status: Essentially Completed.

Subtask 1.2 Sample Characterization

Status: Individual Sample Characterization completed and reported, the characterization of the larger samples for processing is on going.

Subtask 1.3 Evaluation of Dispersion Parameters

This task is further divided into two subtasks.

Status: Subtask 1.3.1 Evaluation of Flotation Parameters. This task is essentially completed (see previous report).

Subtask 1.3.2 Evaluation of Dispersion Parameters

Status: Subtask 1.3.2 Evaluation of Dispersion Parameters. This task is largely completed, some of the results were presented in the previous report, some herein.

SORPTION TESTS WITH DISPERSANTS

Purpose and Procedure. DisalTM is a commercial dispersant consisting of an aqueous solution of naphthalenesulfonic, formaldehyde polymer sodium salt. In this project, laboratory sorption studies evaluated the ability of Kentucky coal ashes to sorb DisalTM in water. With a few exceptions discussed in our previous report (Robl et al. (2004)), sorption tests consisted of 10 grams of ash or another solid sample mixed with approximately 900 grams of distilled and deionized (D&D) water. Because DisalTM sorption is most effective under neutral to alkaline conditions, 0.02 molar (M) sodium carbonate solutions were usually added to any pH < 5 ash-water mixtures to raise the pH above 7 before DisalTM addition. The DisalTM was added to the mixtures in the form of a 1% (10,000 parts per million) stock solution diluted with D&D water. With some

exceptions, the mass of 1% Disal™ added to the samples was approximately the same as the mass of ash or other solids in the mixtures.

After the addition of Disal™, the mixtures were tumbled for at least two hours on an end-over-end stirrer. The samples were then filtered at 0.45 microns. The pH values of the filtrates were measured and recorded. The dissolved Disal™ concentrations in the filtered samples were determined with a Perkin-Elmer Lambda 12 ultraviolet-visible spectrometer using a wavelength of 295.0 nanometers (nm). By knowing the total amount of Disal™ added to an ash-water mixture and the amount remaining dissolved in the filtered sample through spectrometer measurements, the grams of Disal™ sorbed per kilogram of ash (g Disal™/kg ash) can be calculated. In general we have observed that the easily dispersed ashes have relatively low Disal™ sorption values.

Results and Discussion. Additional Disal™ sorption tests were performed on a Coleman pond ash (Sample No. 12-14-04). In the test, 10.0 g of ash were mixed with 931.0 g of D&D water. A total of 10.0 g of 1% Disal™ was added to the mixture. A duplicate with an identical composition was also prepared. Because the Coleman samples were suitably alkaline, no pH adjustment with sodium carbonate was required. The results in Table 1 show that the sorption values of the 12-14-04 Coleman pond ash are significantly lower than most of the values of the other Coleman samples.

Percolating rain water will quickly remove readily water-soluble salts from ash. We have found that a low concentration of the water soluble salts increase the effectiveness of the Disal™. Divalent cations including calcium and magnesium increase the amount of Disal™ absorbed and also the amount required to disperse the ash. We think that the salts bind the negatively charged sulfate sites on the molecule decreasing its charge and increasing the amount of reagent needed to disperse the ash.

Table 1. Disal™ sorption results on a Coleman pond ash (12-14-04). For comparisons, sorption results on other Coleman samples reported previous are listed.		
Plant	Ash Type, CAER Identification No., and Description	g Disal sorbed per kg ash
Coleman	Pond ash 12-14-04	0.84
Coleman	Pond ash 12-14-04 Duplicate	0.90
Coleman	Flyash 92940	3.23
Coleman	Flyash 93030	6.53
Coleman	Flyash 93030, 50 grams ash used	9.96
Coleman	Flyash 93030, 100 grams ash used	9.86
Coleman	Flyash 93030, 200 grams ash used	9.99
Coleman	Flyash 92946	4.56
Coleman	Water washed flyash 92946	2.06
Coleman	Washed carbon residue 92946	2.62
Coleman	Washed carbon residue 92946, Duplicate	2.38

Coleman	Washed carbon-depleted flyash 92946	2.11
Coleman	Pond core sample, Sample #15-1	1.01
Coleman	Pond core sample, Sample #15-1, Duplicate	1.52
Coleman	Pond core sample, Sample #15-1, 25 grams of ash used	1.91
Coleman	Pond core sample, Sample #15-1, 50 grams of ash used	2.42
Coleman	Pond core sample, Sample #15-1, 100 grams of ash used	2.35
Coleman	Pond core sample, Sample #15-1, 200 grams of ash used	2.32
Coleman	Pond core sample, Sample #15-1, 500 grams of ash used	4.70
Coleman	Pond core sample, Sample #15-1, Washed carbon residue	1.76
Coleman	Pond core sample, Sample #15-1, Washed carbon-depleted	0.29

As discussed in our last report, significant correlation (now $R^2 = 0.77$ including the data from Table 1) exists between the amount of Disal™ that a Kentucky ash sorbs in water and the conductivity of its leachate in D&D water. Again, the conductivity of the leachate would be related to its total dissolved solids (TDS) concentration, including dissolved calcium and other major ions.

Utilizing the data in Table 1 and the results for the 26 ashes tested previously an R^2 of 0.74 is calculated between the Disal™ sorption values of the Kentucky ashes and the dissolved calcium concentrations (Ca^{2+}) of their leachates (Figure 1). The water-soluble calcium may react with Disal™, and again, any chemical interactions between Disal™ and dissolved calcium diminishes the dispersive effectiveness of the Disal™.

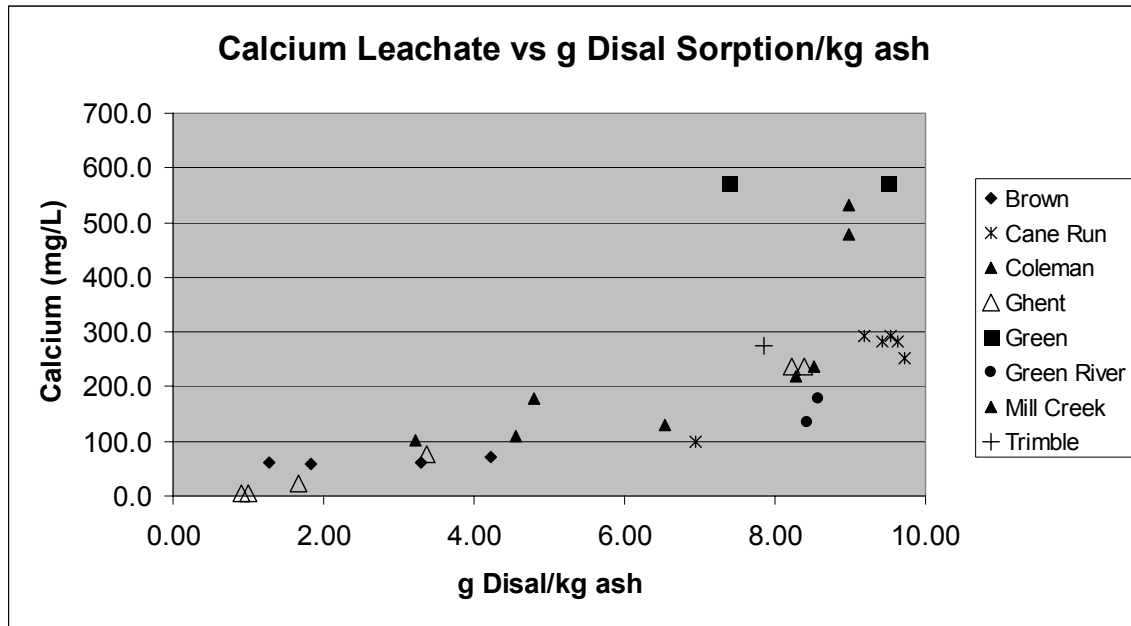


Figure 1: Relationship between the amount of Disal™ sorbed (g/kg ash) in aqueous suspensions of Kentucky coal ashes and the calcium concentration (mg/L) of their distilled and deionized water leachates. The four data points in the upper right corner of Figure 1 probably represent gypsum saturation.

SETTLING TESTS WITH DISPERSANTS

Purpose and Procedure. The ability of Disal™ and other products to disperse ash particles in water was estimated by monitoring the specific gravities of ash-water suspensions over time. The useful residence time of ash in the hydraulic classifier is probably not greater than about 30 minutes, ash dispersion should be maintained for at least that amount of time.

The laboratory settling experiments consisted of placing 100 grams of an ash into a one-liter glass cylinder. Because the pilot scale tests utilize Lexington, Kentucky municipal tap water, the laboratory settling studies included tests with the tap water as well as distilled and deionized (D&D) water. Disal™ or other commercial dispersants were then added to the aqueous mixtures either directly or as a 1% aqueous solution. The total mass of liquids in each mixture was approximately 900 grams. After the samples were shaken by hand, a stop watch was immediately started and a Fisher-brand hydrometer (which measures specific gravity) was placed into the suspension. As the ash settled in the suspension, specific gravity declined. For consistency, "zero time" was defined as when the specific gravity of the suspension reached 1.040. As the specific gravity of each suspension reached 1.035, 1.030, 1.025, 1.020, 1.015, and 1.010, the times were recorded. Dispersant dosages were adjusted until the specific gravity of the suspensions remained between 1.010 and 1.040 for approximately 30 minutes.

Results and Discussion. During this reporting period, settling tests were performed on the Coleman (12-14-04) pond ash using Disal™. The tests indicate that 1.4 g of Disal™/kg of ash is required to maintain a specific gravity between 1.010 and 1.040 for 30 minutes (Figure 2). Table 2 compares the Coleman pond ash results to the Disal™ results of several other ashes. Like the Mill Creek pond ash, the results for the Coleman are fairly favorable and indicate that the ash may be adequately dispersed with relatively low dosages of Disal™. The settling results of the Coleman pond ash are also consistent with the Disal™ sorption values in Table 2, which predicted that this ash lacks Disal™-sorbing calcium salts probably because of weathering conditions in the pond.

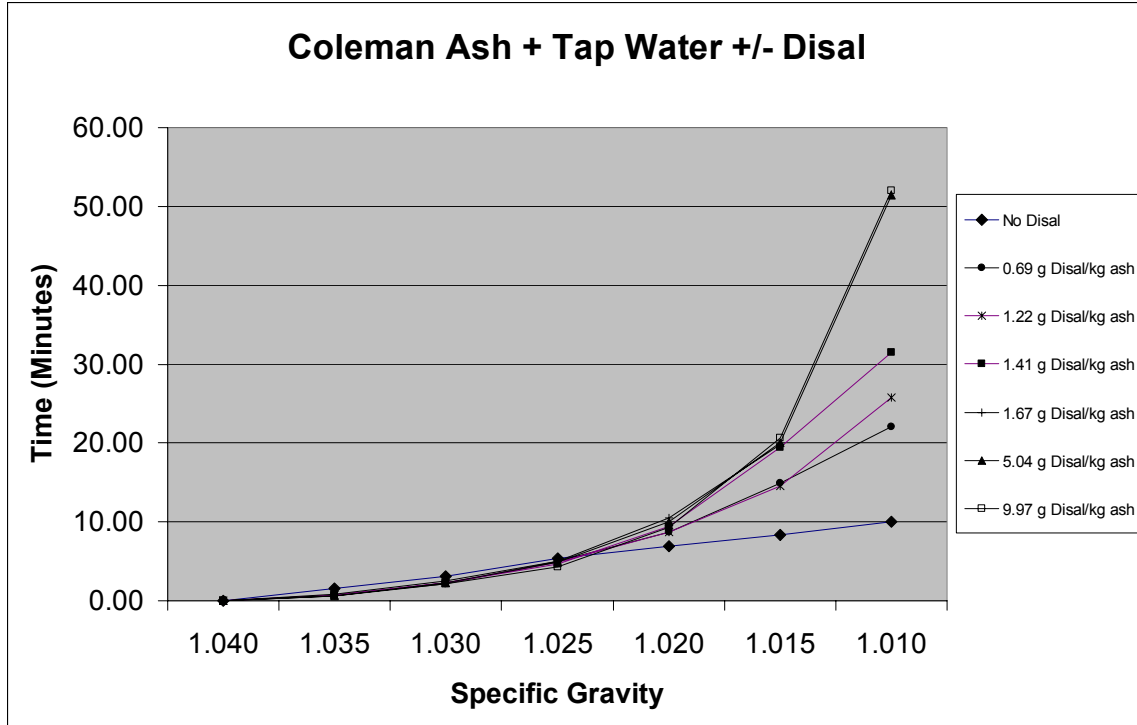


Figure 2. A settling test plot of time versus specific gravity for a Coleman pond ash (12-14-04) in Lexington, KY, tap water. The plot indicates that approximately 1.4 g of Disal™/kg ash are required to maintain a specific gravity between 1.010 and 1.040 for 30 minutes.

Sample	Disal™ + Lexington, KY Tap Water
Coleman Pond Ash (12-14-04)	1.4
E.W. Brown (0406-23)	3.7
Water Washed E.W. Brown (0406-23)	0.9
E.W. Brown Pond Ash (040720)	>>100
-100 Mesh E.W. Brown Pond Ash (040720)	>>100
Cane Run flyash (0406-30)	>>100
Water Washed Cane Run (0406-30)	>100
Mill Creek, Unit 1, Nov. 2000	11
Water Washed Mill Creek, Unit 1, Nov. 2000	5
Mill Creek 2003 "Standard" Ash	7
Water Washed Mill Creek "Standard" Ash	12
Mill Creek Pond Ash	0

Discussion. Test results from the Cane Run and Coleman ashes are consistent with our previously reported work. Batch leaching and sorption studies on the Cane Run flyash further support a correlation between the ability of an ash to adsorb Disal™ in water and the conductivity and calcium concentration of a D&D leachate of the ash. Sorption studies predicted relatively low sorption of Disal on the Coleman pond ash. The settling results were consistent with the sorption studies and indicate that only about 1.4 g Disal/kg ash are required to suspend the Coleman ash in Lexington KY tap water for 30 minutes.

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

Work is ongoing and will be reported in the next period.

Subtask 1.4.3 Environmental Testing

BATCH LEACHING TESTS

Purpose and Procedure. The purpose of the aqueous batch leaching tests is to determine whether any of the ashes contain significant concentrations of water-soluble species, the identity of those species, and whether they could possibly interfere with dispersion agents. In this project, the leaching tests consisted of placing 48 grams of an ash into a borosilicate glass jar with approximately 900 grams of distilled and deionized (D&D) water (>17 Mohm.cm). The samples were agitated for at least 35 minutes on an end-over-end tumbler. The leachates were then filtered at 0.45 microns and analyzed for pH, conductivity, major anions (at least chloride, fluoride, and sulfate), and major cations (at least aluminum, calcium, magnesium, potassium, and sodium). The pH measurements were determined with a ThermoOrion model 410+ Meter using pH 4.00 +/- 0.01, 7.00 +/- 0.01 and 10.00 +/- 0.02 Fisher Scientific, Inc. buffers. A YSI Inc. Model 32 meter provided the conductivity measurements, which are related to the total dissolved solids concentrations of the samples. Anion concentrations were measured on a Dionex LC-20 ion chromatograph using EPA Method 300.0 (US EPA, 1993). The cations were analyzed with a Spectrometrics Inc. Inductively Coupled Argon Plasma-Atomic Absorption Spectrometer (ICP-AA) using EPA Method 200.7 (US EPA, 1994).

Results and Discussion. Previously, batch leaching tests were performed on 26 Kentucky pond and fly ashes. Since the last reporting period, chemical analyses were completed on a batch leachate of Cane Run (040630) fly ash or sample #10 in Tables 4-6

of Robl et al. (2004). The data are shown in Table 3. The leachate of this Cane Run sample has a significantly lower conductivity and less sulfate, aluminum, and calcium than the leachates of the other Cane Run flyashes as previously reported. In contrast, the Cane Run (040630) leachate contains significantly more chloride and magnesium than the other Cane Run samples.

As discussed previously and elsewhere in this report, significant proportional relationships exist between the ability of an ash to sorb Disal™ and the calcium concentration or conductivity of its distilled and deionized water leachates. The Cane Run results in Table 3 are consistent with this relationship. The relatively low calcium and conductivity values of the Cane Run (040630) leachate may explain why the Disal™ sorption value of the ash is only 6.94 g Disal™/kg ash when compared with 9.19 – 9.71 g/kg for the other unwashed Cane Run fly ashes previously reported.

Table 3. Distilled and deionized water leaching results on Cane Run flyash (040630).	
Parameter	Value
g of ash leached	48.0
g of D&D water in leachate	904.0
Liquid to solid ratio of leachate	18.8
pH	10.81
Conductivity (ohms/cm)	638
Sulfate (mg/L)	227
Chloride (mg/L)	4.83
Fluoride (mg/L)	0.28
Aluminum (mg/L)	3.70
Calcium (mg/L)	99.0
Magnesium (mg/L)	5.44
Potassium (mg/L)	29.7
Sodium (mg/L)	15.5

Subtask 1.5 Process Simulation.

Introduction. The development of the process circuit will require the integration of a number of unit processes. Their integration will be greatly facilitated by the ability to conduct computer simulation of the overall process in advance of actual construction. Also, process simulation is of value in optimizing the operation of the final circuitry.

A reconnaissance of the available commercial software was conducted. We found that probably the leading software for simulation of mineral processing circuitry is that of the Julius Kruttschnitt Mineral Resources Center. Their software, JKSimMet V5.1 offers the ability to digitally model and integrate a variety of unit processes including spiral

concentrators, hydrocyclones, screen decks, and various mills. Good piping circuitry and ancillaries are also present. CadChem, a chemical engineering software modeling program, has similar features and abilities at a lower cost.

What we failed to find was any software that modeled hydraulic classifiers or froth flotation. The latter is under development in the JKSimMet program. These operations are of key importance to the process. Because of the lack of availability, we began developing our own model during the research period. Because we think that the standard release analysis that we use for froth flotation will generate adequate data for process modeling, we focused on hydraulic classification. The development of the model required the construction of a small pilot classifier to generate data.

HYDRAULIC CLASSIFICATION AND CONCENTRATION OF FINE PARTICLES.

Background Discussion. The sorting of very fine particles in hydraulic systems is a dictated by Stokes Law, which states simply:

$$V_p = G (\rho_s - \rho) D^2 / 18\mu$$

Where V_p is the rising or settling velocity of the discrete particle, G is the gravity constant, ρ_s is the density of the particle, ρ is the density of the carrier fluid, D is the diameter of the discrete particle and μ is the viscosity of the carrier fluid. From this the settling velocity of the particles can be calculated. Stokes law assumes laminar flow and that the particles do not interact with one another.

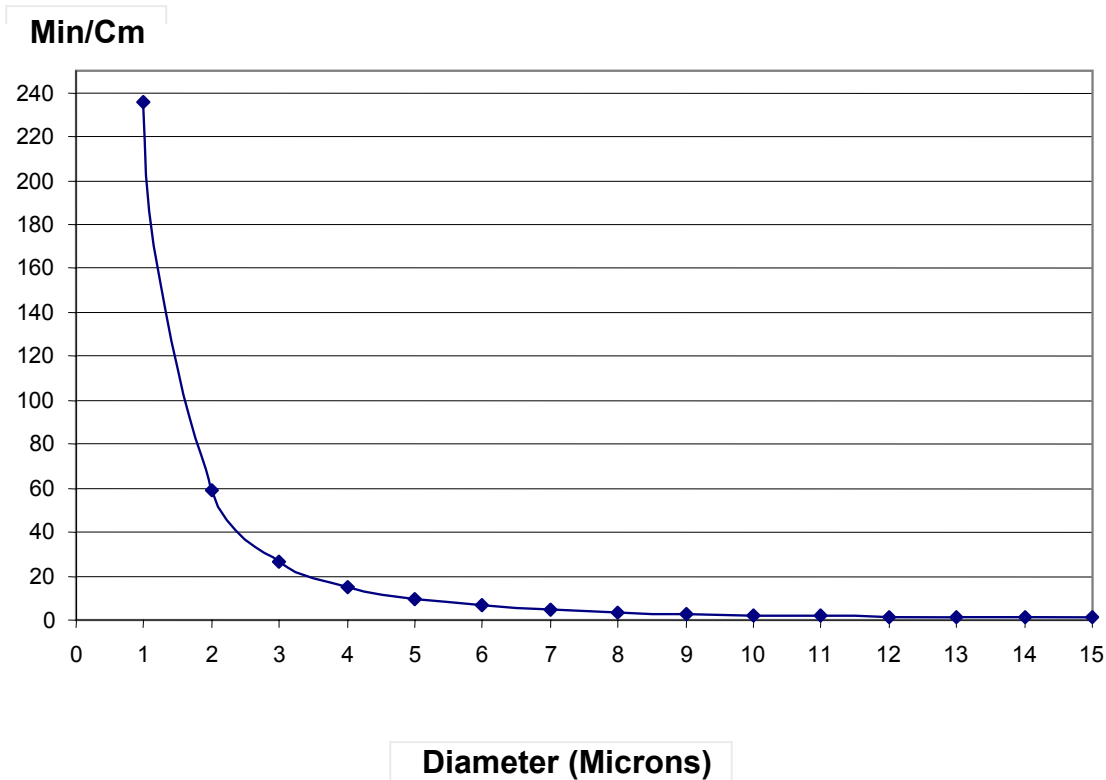


Figure 3. Plot of Stokes Law Settling Velocity vs. Particle Diameter at 20 °C.

The sinking velocity of the particles are found to vary widely for the sizes of particle of interest in this study. Figure 3 presents a plot of the inverse of settling velocity for particles in water as a function of diameter. Stokes law dictates that a one micron particle will take approximately 4 hours to drop 1 centimeter. A 5 micron particle will take approximately 10 minutes to fall 1 centimeter.

The above calculations assume an ideal case, that the particles do not interact and that there are no other forces involved. This, of course is not the case. The amount of movement caused by simple Brownian motion for a 1 micron particle is almost as great as that of gravity. Brownian motion affects are much stronger than gravity for particles much smaller than 1 micron. These are sometimes referred to as “turbidity solids” and do not generally settle unless they are forced to form flocs.

Other factors affecting particle separation is the tendency of the fly ash to naturally flocculate. We have found the ash flocs to be surprisingly stable, even over a wide pH range. Deflocculating the ash slurries, cost effectively, is an important part of this study. Another factor that works against efficient separation is the tendency of the larger particles to interfere (hinder) the movement and settling of the smaller particles. For example a 100 μm particle has a settling velocity of 65 cm/min which is about 10,000 faster than a 1 μm particle. More significantly, the 100 μm particle has a million times the mass of the 1 μm particle.

CLASSIFIER MODELING.

Definition of Terms. The discussion of the performance of the various classifiers and the results of the modeling requires some definition of terms. The *product*, which we are targeting in this work, are the smallest particles of ash, those that are less than $5\ \mu\text{m}$ in diameter. The product *grade* is the average particle diameter size, on a volume basis, stated as the D_{50} . Note, that this is different from the X_{50} that is used to define efficiency of separation, which is the average diameter whereby the particle has a 50% probability of partitioning to the product or overflow and the reject or underflow. The product *recovery* is then the amount of particles that are smaller than $5\ \mu\text{m}$ in the product as a percentage of all of the $<5\ \mu\text{m}$ particles that were available in the feed. The product *yield* is the total mass of the product as a percentage of the feed

Horizontal Open Channel Classifier with Weirs. As a digital model of the hydraulic classifier was not available, we decided to construct one and generate the needed data. A simple cross current horizontal unit was constructed from Plexiglas as shown in Figure 4. The unit consisted of four cells with the overall dimensions of 39cm x 37cm x 35cm (width x length x depth). The total volume of each cell was 51 liters, or about 200 liters for the whole unit. The classifier was fed from one end and the product collected from the other. The coarse ash underflow was pumped from the bottom of the classifier. Each chamber was open to the second or, in some tests, separated from the next by means of a weir. The removal of slurry from the bottom of each cell also increases the relative residence time of the product stream by decreasing the overall discharge rate from one cell to the next.



Figure 4. Photo of open channel hydraulic classifier with weirs.

The test runs performed during the hydraulic classification evaluation work required several tons of ash. Ash from the pond at Ghent was used for the calibration as there was a multi-ton sample available at the CAER. Mean particle diameter (D_{50}) of the product ranged from 3 to 5.3 μm with recovery of the 5 μm particles as high as 70%.

The use of weirs between the cells did not seem to improve performance and had several disadvantages. The weirs created a visible “mud line” which was thicker in the cells with the highest weirs, i.e. upstream cell (Figure 4). The mud line defined a sediment compression zone, within which particle sorting is inefficient. With this configuration the highest weir controlled the overall cross sectional area of flow for the device and created a thick compression zone. One way around this would be to vary the width of the device while keeping the depth constant. This kind of configuration has some practical design considerations that would present some problems in scale-up.

The approach that we chose, however, was to divide the channel into layers through the addition of lamella (Figures 5 and 6). This creates, in effect, a number of thin channels for sedimentation to take place on, the lamella minimize the thickness of the mud line or compression zone.

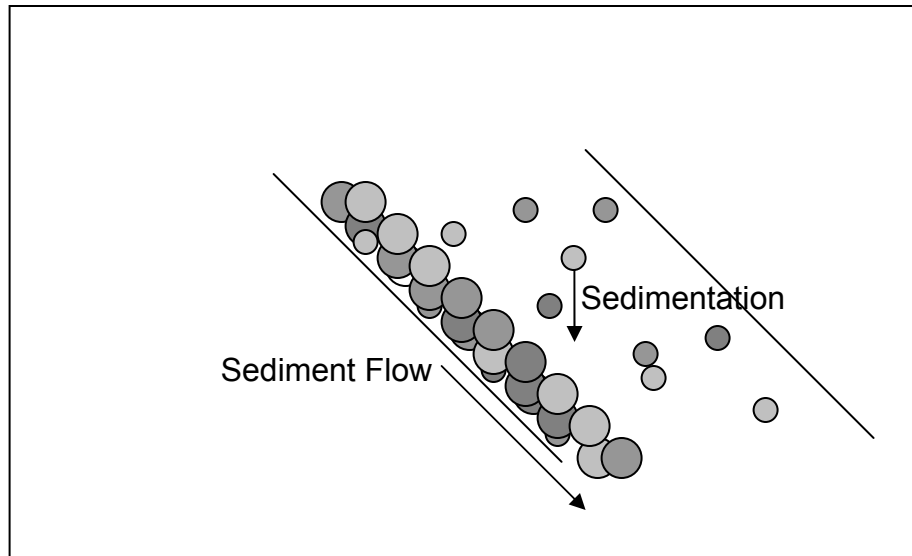


Figure 5. Schematic of Affect of Lamella on Particle Separation.

The lamella, in effect, increases the width of the channel while keeping the depth constant. This is done by “folding” the channel and putting it in a box. There were two different designs for this. Their cross sections are illustrated in Figure 4. The first was designed to fit the preexisting shell of the weir classifier and was used as an expedient to test the idea. The second was developed and built from the ground up. Photos of both types are shown in Figures 4 and 7.

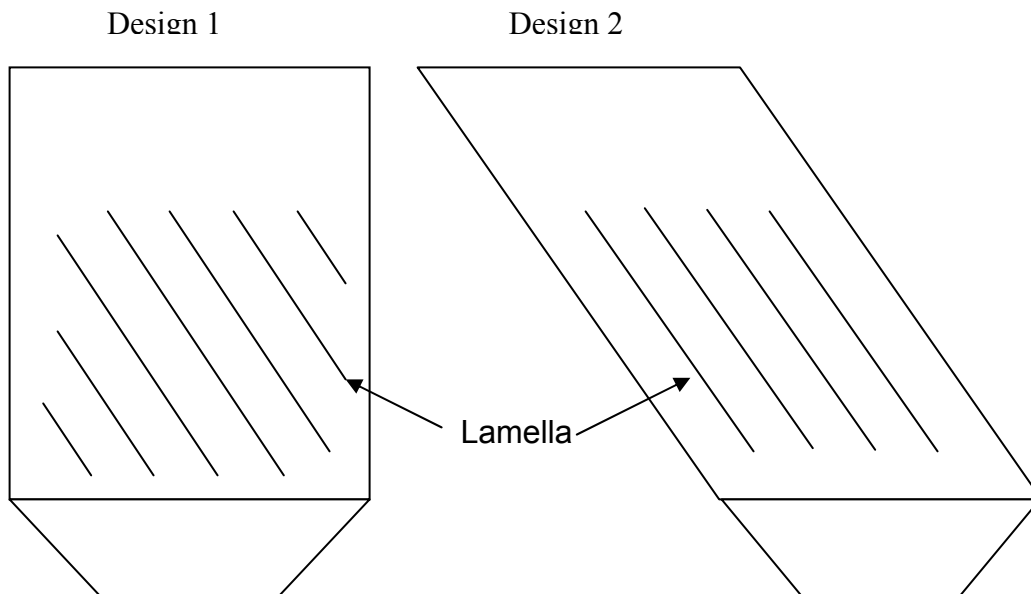


Figure 6. Lamella Design 1 and Design 2 Comparison.

A total of 17 tests were made which represented some 31 separate determinations (Table 4). Seven were made under the open flow design, test 4 and 6 had weirs between the individual cells. Four tests were run with the first lamella design and seven were run with the second lamella design. Most of the work utilized NSF (Naphthalene Sulfonate Formaldehyde condensate) as a dispersant. Tests 16 and 17 utilized a polycarboxylate dispersant (Grace's ADVA 100 and ADVA flow). The ash was kept constant during the testing to keep a consistent basis of comparison.

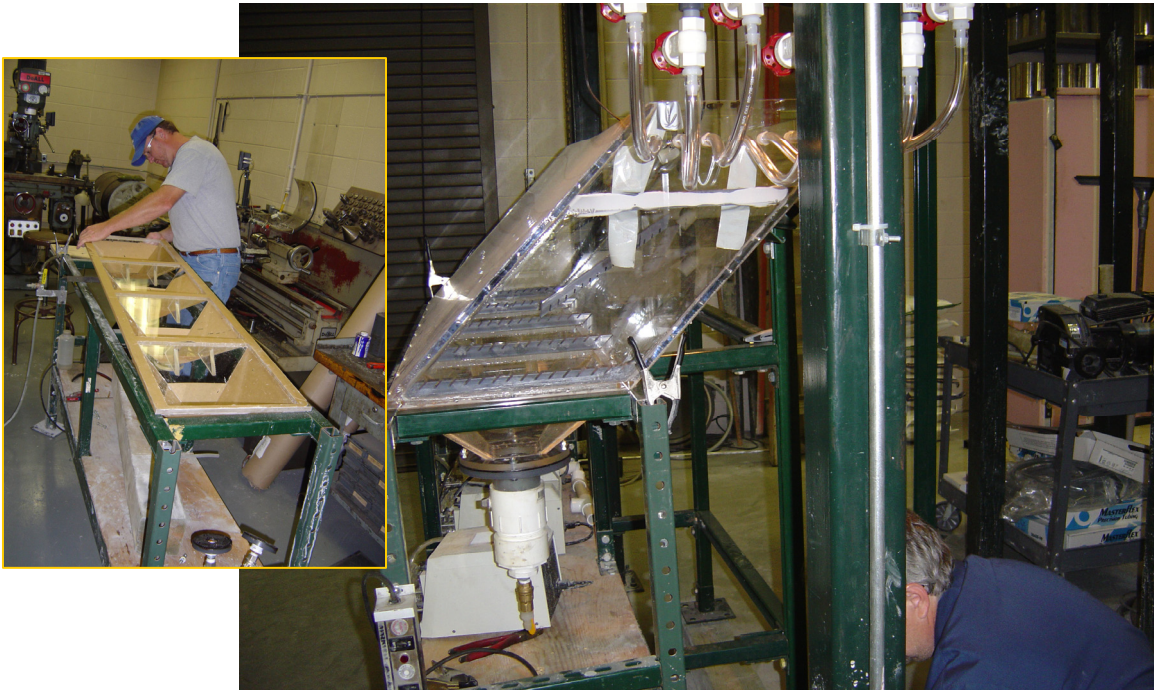


Figure 7. Fabrication of the second lamella cross flow hydraulic classifier.

During the test series the feed rate, and indirectly the residence time of the solids was the primary variable. The mean residence time (MRT), was calculated from the average particle residence time as:

$$\text{MRT} = (Q_f + Q_1)/2V + (Q_1 + Q_2)/2V + (Q_2 + Q_3)/2V + (Q_3 + Q_o)/2V$$

Where, Q_f is the primary flow into the classifier, Q_1 is the flow into the second cell of the classifier ($Q_1 = Q_o - U_1$, where U_1 is the discharge to the underflow from cell one, etc). Q_2 and Q_3 , are the flows into the third and fourth chambers and Q_o , the out flow of the product discharge. The V is the volume of each of the cells, approximately 51 liters. The volume of the cone shaped funnel at the bottom of the classifier was omitted as we considered this to be still zone and part of the collection system. The MRT varied widely, from almost an hour and half to as short as 25 minutes (Table 4).

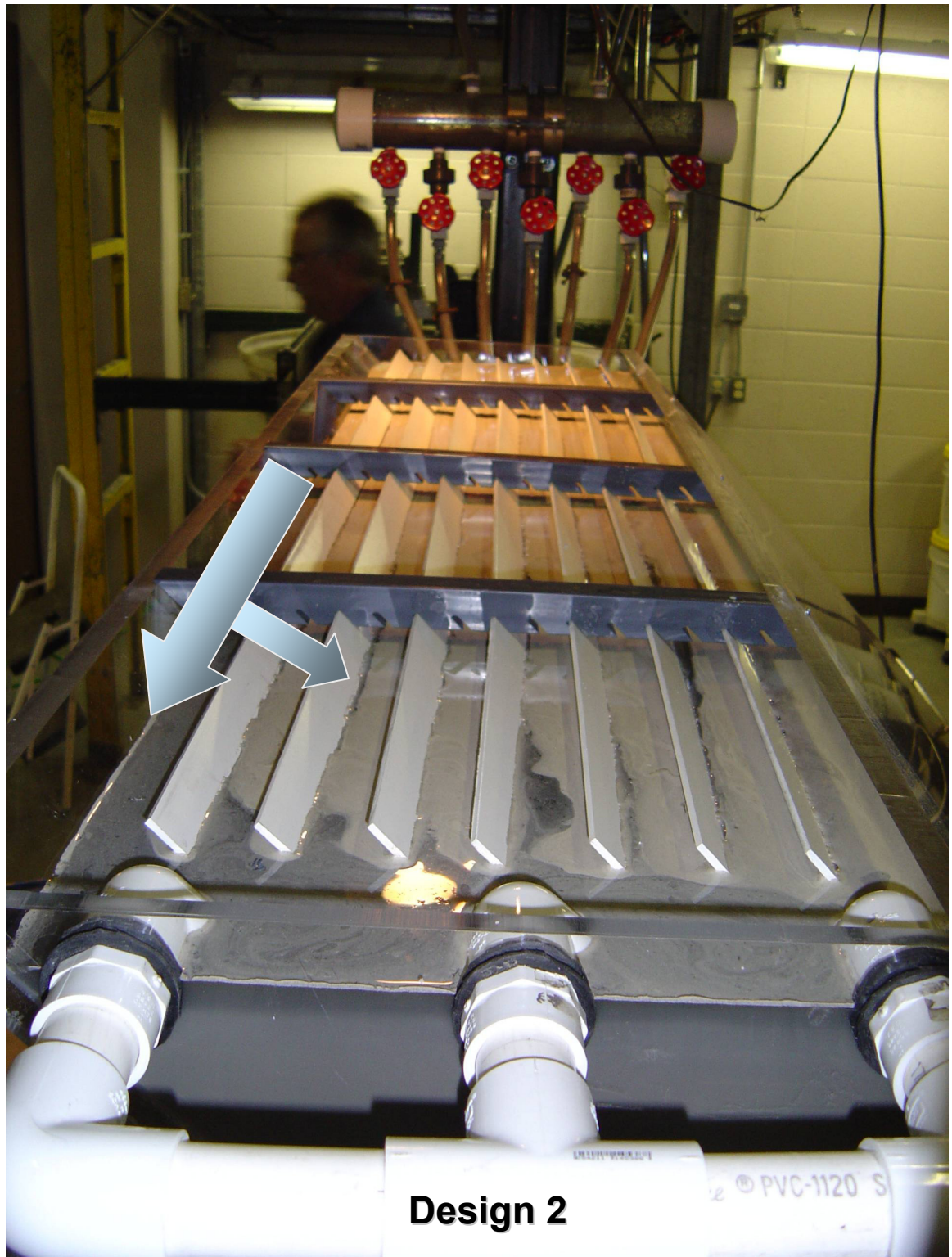


Figure 8. Photo of the secondary classifier in operation. The blue arrow shows the direction of flow and the direction of sedimentation. The foreground is the submerged collection launder which greatly facilitates cenosphere collection.

Table 4. Mean Residence time and weight percent solids in feedstock underflow and solids from the hydraulic classification tests.							
Test		% Solid					
	MRT	Feed	U1	U2	U3	U4	Product
1a	54.9	14.0	30.5	17.1	15.7	14.2	5.1
1b	55.2	13.7	29.3	16.2	15.5	13.9	4.8
2a	63.9	12.5	38.7	18.3	17.2	15.5	4.4
2b	65.8	12.3	38.2	18.0	17.4	15.4	4.4
3a	89.8	13.7	44.3	25.4	23.2	22.0	2.3
4a	73.7	13.0	39.1	23.0	17.1	14.5	4.0
4b	66.5	13.0	38.6	21.6	16.5	13.2	4.5
5a	68.6	11.8	29.1	27.6	22.4	20.6	2.7
5b	71.0	11.6	27.3	27.0	22.2	20.5	2.7
6a	78.9	15.0	41.7	38.1	21.0	17.0	5.6
6b	70.2	14.9	41.7	27.6	20.7	17.3	5.5
7a	69.2	12.9	19.7	46.2	32.8	22.1	2.6
7b	68.8	13.0	19.9	48.2	33.4	22.3	2.5
8a	29.0	11.6	28.6	50.4	48.0	33.4	5.2
8b	28.3	11.6	29.4	52.0	49.2	33.8	5.2
9a	41.7	10.5	22.4	41.8	37.5	25.3	3.6
9b	42.2	10.4	22.9	42.4	37.7	25.3	3.7
10a	45.2	12.9	26.8	47.1	41.8	27.9	4.4
10b	46.9	12.8	26.7	46.7	46.7	27.6	4.3
11a	42.9	13.3	34.9	47.1	43.8	42.0	2.6
11b	45.5	13.4	35.4	48.8	43.1	42.2	2.4
12a	35.0	13.7	40.9	51.9	46.3	45.0	3.0
12b	34.3	13.5	41.2	54.6	48.4	45.9	2.8
13a	30.6	14.2	42.5	53.6	50.3	49.1	3.6
14a	26.4	14.1	49.6	56.6	54.5	52.4	3.6
14b	26.8	13.8	51.4	53.9	55.0	54.4	3.7
15a	33.0	12.0	37.8	48.1	41.3	39.9	2.1
16a	34.0	14.5	52.8	53.6	49.7	47.0	2.4
16b	32.0	14.7	50.6	56.2	53.0	48.8	2.8
17a	25.0	12.6	47.5	54.1	50.6	48.1	2.9
17b	25.1	12.7	46.9	54.4	52.3	49.5	2.7

The feed rate was varied until the D_{50} particle size of the product was within a target range of between ~ 3 to ~ 5 μm . The mass flow rate of the product, underflow and feed was measured, as was the percentage of solids and the loss on ignition. The particle sizes of the feed, underflow and product was measured with a Cilas laser diffraction particle size analyzer. The product yield and recovery of the 5 μm ash was calculated as percentages of the feed. Typically two sample collections and determinations were made per test, but several times equipment malfunctions, most typically ruptured or plugged pumps, resulted in early termination of tests.

Table 5. Data from hydraulic classifier test runs. Yield (Yld), and solids balance (bal) are as percent of feed. Recovery (Rec) is as percent of the less than 5 μm particle size material. D_{50} is the mean particle diameter as volume percent. Loss on ignition (LOI) is as weight percent of feed and product (Pro).

Test	Solids Flow Rate Kg/min						Solids Bal	D50	Yld	Rec.	Feed	Pro
	Feed	U1	U2	U3	U4	Prod		Mm	%	%	LOI	LOI
1a	0.81	0.40	0.10	0.08	0.07	0.15	102%	4.8	18%	49%	4.71%	2.68%
1b	0.80	0.38	0.10	0.09	0.08	0.13	102%	5.0	16%	44%	4.76%	2.55%
2a	0.55	0.25	0.07	0.06	0.06	0.12	100%	4.6	21%	59%	4.40%	2.28%
2b	0.52	0.24	0.06	0.06	0.06	0.11	97%	4.3	21%	64%	4.05%	2.22%
3a	0.38	0.15	0.05	0.04	0.04	0.04	117%	3.5	11%	34%	3.14%	2.15%
4a	0.50	0.23	0.07	0.06	0.04	0.09	100%	4.3	18%	49%	4.62%	2.52%
4b	0.54	0.23	0.07	0.06	0.04	0.12	106%	4.3	22%	69%	4.79%	2.47%
5a	0.47	0.16	0.09	0.08	0.07	0.07	103%	3.1	14%	36%	4.79%	2.34%
5b	0.45	0.15	0.09	0.07	0.07	0.06	102%	3.0	14%	35%	4.51%	2.13%
6a	0.54	0.26	0.09	0.07	0.06	0.15	86%	4.6	28%	68%	4.79%	2.61%
6b	0.60	0.26	0.09	0.06	0.06	0.13	99%	5.2	22%	51%	4.19%	2.47%
7a	0.52	0.13	0.13	0.12	0.07	0.06	103%	3.2	12%	50%	4.72%	2.79%
7b	0.53	0.13	0.14	0.12	0.07	0.06	101%	3.1	12%	39%	4.57%	2.61%
8a	0.96	0.16	0.18	0.15	0.10	0.35	102%	6.0	37%	79%	5.44%	2.64%
8b	0.98	0.16	0.18	0.16	0.11	0.36	101%	5.5	37%	80%	4.21%	2.77%
9a	0.62	0.12	0.13	0.12	0.07	0.16	104%	5.0	26%	68%	4.33%	2.82%
9b	0.61	0.12	0.14	0.12	0.07	0.16	100%	4.8	27%	70%	4.19%	3.01%
10a	0.72	0.14	0.16	0.13	0.09	0.18	103%	4.5	25%	71%	4.57%	2.87%
10b	0.70	0.14	0.16	0.16	0.08	0.17	98%	4.1	24%	66%	4.75%	2.85%
11a	0.78	0.20	0.16	0.15	0.14	0.11	103%	3.1	15%	47%	4.60%	3.16%
11b	0.75	0.21	0.17	0.15	0.14	0.10	100%	2.9	13%	44%	5.24%	3.15%
12a	0.97	0.23	0.18	0.17	0.16	0.16	107%	3.1	17%	44%	4.60%	3.16%
12b	0.97	0.24	0.19	0.17	0.16	0.16	106%	2.9	16%	55%	5.24%	3.15%
13a	1.14	0.27	0.21	0.19	0.18	0.22	106%	3.4	20%	65%	5.30%	2.97%
14a	1.29	0.29	0.21	0.20	0.19	0.27	112%	3.7	21%	63%	4.71%	2.72%
14b	1.24	0.31	0.19	0.20	0.20	0.27	106%	3.8	22%	65%	4.21%	2.89%
15a	0.89	0.23	0.17	0.15	0.14	0.12	109%	3.6	14%	53%	2.92%	2.05%
16a	1.01	0.29	0.21	0.19	0.18	0.13	102%	3.3	13%	41%	4.36%	2.48%
16b	1.07	0.27	0.22	0.20	0.19	0.16	104%	3.2	15%	51%	4.18%	2.78%
17a	1.16	0.30	0.22	0.20	0.18	0.21	105%	3.7	18%	60%	5.07%	2.58%
17b	1.17	0.30	0.22	0.20	0.19	0.20	106%	3.7	17%	60%	4.79%	2.53%

The mass balance was based on the mass flow of the feed solids divided by the sum of the mass flow of the solids from the underflow and product. We consider a balance within 5% of 100% to be good, and 6% or 7% to be acceptable. The solids balance typically tended to be positive (Table 5). This is most likely due to the feed rate, which was the most difficult of the mass flows to measure accurately, as it involved valving which was turned to collect the sample, this affected the head the peristaltic pump was pushing against. Ideally the flow rate of a perfectly sealed peristaltic pump is not affected by pressure head. However, small affects are undoubtedly present, which in this case, would tend to overstate the feed rate. The filtration of the fine materials was

also a source of some small error as well. The fine nature of the solids resulted is some material not being trapped by the sharkskin filters used, and an underestimate of the product rate would result.

The variation of the performance of the three styles of hydraulic classifiers tested was large. Figure 9 presents a plot of means residence time versus the D_{50} or the average product diameter. The relative improvement in efficiency from the first test of the open channel cross flow classifier to the lamella designs is significant. Slow feed rates and resulting MRT's that exceeded an hour in most cases were required to produce a product with a D_{50} in the 3 to 4 μm range. The second lamellae design readily produced products in this size range with residence times as short as 24 minutes. Short residence times are needed if the scaled up technology is to be a reasonable size.

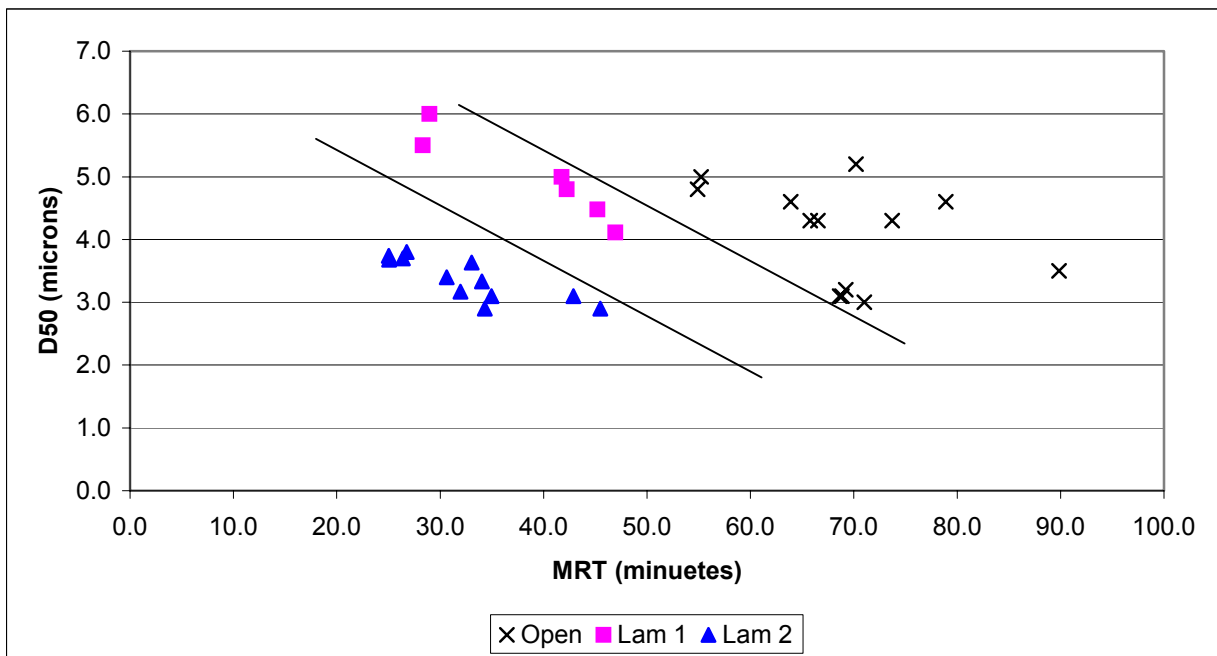


Figure 9. Plot of Mean Residence Time (MRT) versus D_{50} of product size for the hydraulic classifier configurations tested.

Another way to examine performance is to investigate product size as a function of feed rate. For example, the average solids feed rate for the open flow classifier tests was 0.55 kg/min, this produced an average product D_{50} of 4.1 μm . The average solid feed rate for the second classifier tests was 1.0 kg/min, which produced an average product D_{50} of 3.4 μm . Thus the lamellae configuration was able to perform at a much higher rate of feed while delivering a better grade of product.

The relationship between grade and yield is also of significance. It is a measure of the efficiency of the technology. A plot of grade versus yield is displayed in Figure 10. The improved efficiency of the lamellae classifiers, as indicated by a steeper yield-grade curve, is clearly evident from the plot.

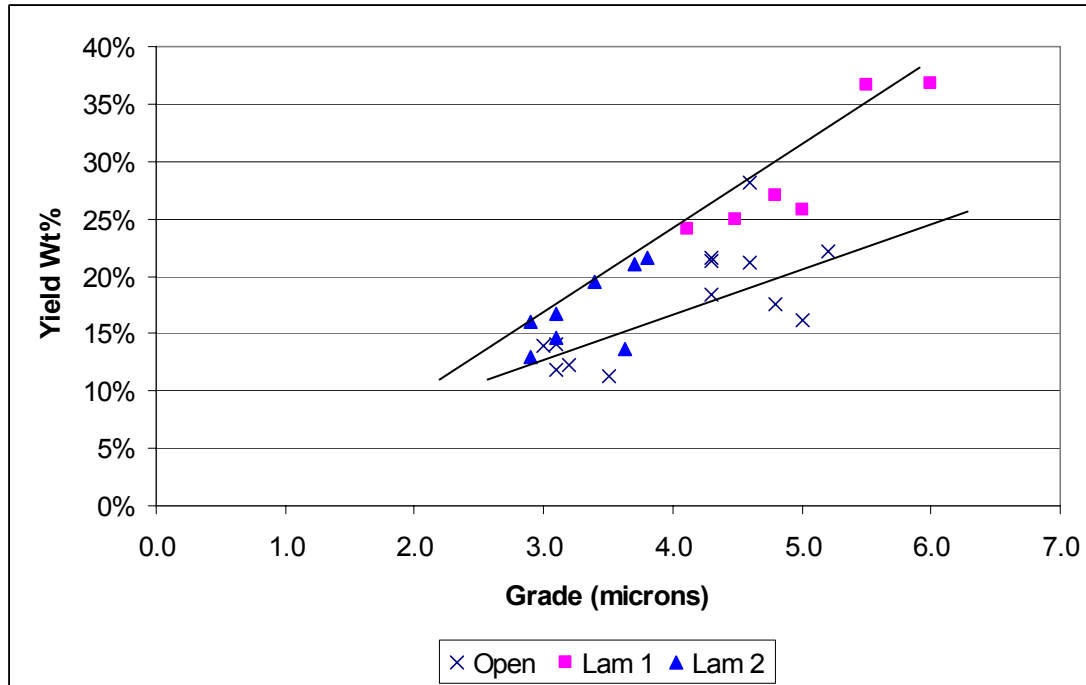


Figure 10. Plot of yield versus grade for the types of classifiers tested.

Generation of a Model of the Lamellae Hydraulic Classifier. A digital model of the final lamellae classifier was constructed using an Excel spreadsheet. The model assumes that the particles obey Stokes Law perfectly with no affect of shape, particle-particle interaction resulting in hindered movement or settling, no Brownian motion, etc. It is also assumed that the particles are of uniform density throughout their size range.

The inputs to the model include cell width, cell depth, cell length, feed rate, withdrawal rate (underflow rate), size distribution of feed material and the vertical cord length between the lamellae or “D”. In a system that has more than one underflow, the model adjusts for residence time variation from multiple underflows. The flow to the next cell based on the feed rate and the withdrawal or underflow rate from the previous cell. Up to four cells are incorporated in the model. Finally, the model assumes that the particles partition to the underflow when they are calculated to have dropped by “D” i.e., the distance between the lamellae. The distance the particle drops is calculated from Stokes law and from residence time.

Obviously there are particle-particle interactions, the particles do vary in density and Brownian motion effects are of major significance for the smaller particles. The model presents what is in essence a perfect system. It is of value for comparing actual test data with theoretical results. In addition the relative affects of various parameters that are difficult to incorporate into lab test can be examined.

The size of the feed and the resulting product from a test of the second lamella classifier are presented in Figure 11. In addition to the actual size distribution of the product, the size distribution that was calculated from the model for the product is also

plotted. A D_{50} of 2.5 μm is calculated from the model for the conditions of the test and the feed size distribution, a D_{50} of 3.7 μm was actually measured. The product yield predicted from the model is 22% and the total recovery of the $<5 \mu\text{m}$ material is 89%. The measured yield was 21% and the recovery of $<5 \mu\text{m}$ particles were 63%. If the model is assumed to represent the maximum theoretical recovery, it can serve as a measure of the efficiency of the device. For example, the ratio between the actual recovery and the theoretical recovery in the example was 71%.

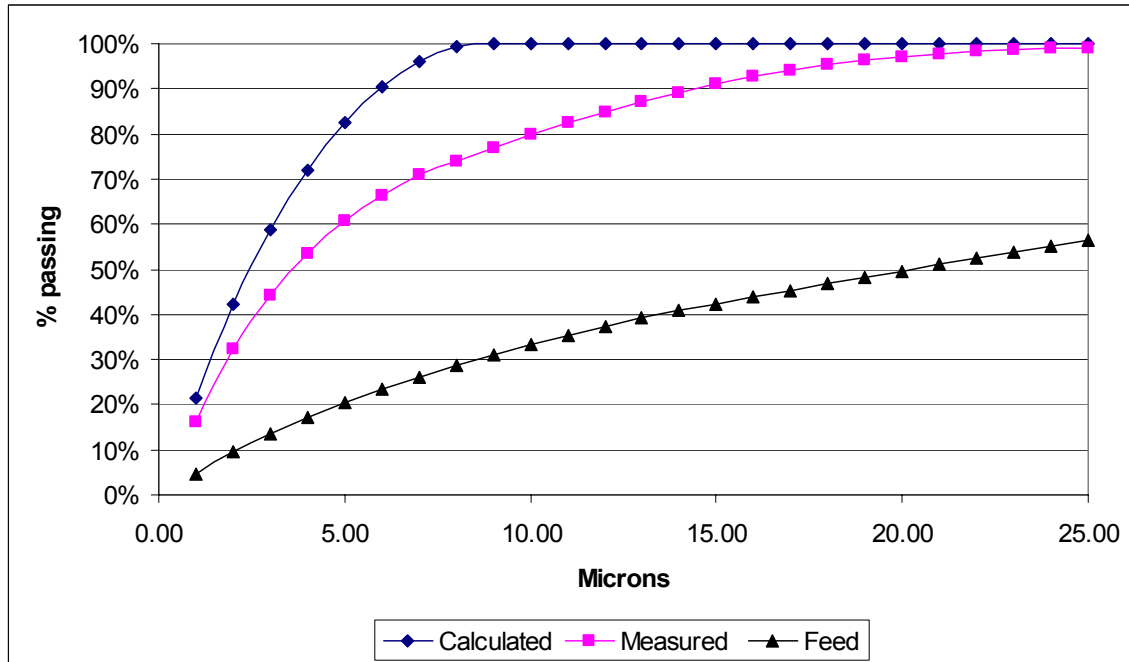


Figure 11. Plot of calculated versus measured product as a function of feed sized distribution for second generation lamellae classifier.

The model provides a reasonable indication of how the classifier will behave and is useful in generating the relative affect of parameters such as temperature. For example, using the feed size distribution as illustrated in Figure 11, 64% of the product is calculated to be less than 3 μm in diameter at 20 $^{\circ}\text{C}$. At 4 $^{\circ}\text{C}$, 55% of the product is calculated to be less than 3 μm in diameter. The absolute numbers are not precise but the relative difference on particle size is of value. The impact of going from summer to winter on average particle size is about the same as increasing the distance “D” between the plates by 3 cm, or changing the residence time by 20% to 25%.

The Affect of Hydraulic Classification on Loss on Ignition. Generally, we have found that the average particle sizes of the residual carbon are larger than that of the fly ash. The bulk density of the carbon is less, about 1.7 to 1.9 g/cm^3 versus about 2.3 g/cm^3 for the siliceous materials. This would tend to make the carbon particles partition with correspondingly smaller siliceous particles. For example, a 10 μm diameter carbon particle has the same settling velocity as a 7.9 μm siliceous particle. For the most part however, the difference in particle sizes is found to have a larger affect.

Figure 12 illustrates the partitioning of the LOI from a test run of the lamellae classifier and compares it with the overall ash balance. The carbon is found to disproportionate from the siliceous part of the ash as the product yield was 22%, the product contained only 13% of the LOI. The bulk of the LOI is found to partition to the first two underflows of the classifier.

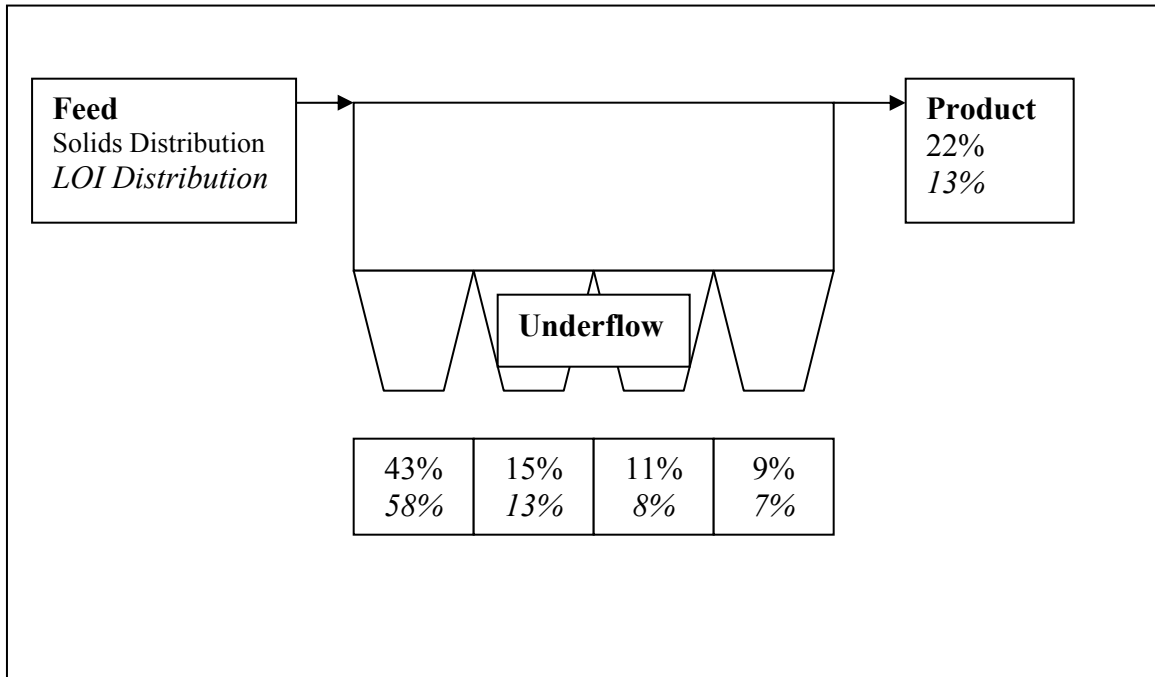


Figure 12. Diagram illustrating the partitioning of total solids and LOI during a typical hydraulic classifier test.

It is often assumed that, for a Class F ash, LOI is a good proxy for carbon concentration. This is not strictly true however. The LOI and carbon data is presented in Table 6 below for the calibration tests of the hydraulic classifier. The reduction in LOI during the tests was in the 40 to 50% range. The carbon concentration was lower than the measured LOI buy approximately 0.8%. TGA-mass spectrometry tests were made on the recovered products. It was found that the non-carbon LOI was primarily due to small amounts of carbonate and sulfates with possible some tightly bound water.

The improvement in LOI achieved by hydraulic classification is significant, particularly for concrete and mortar applications. The LOI and carbon levels found in the feed materials were high and near the ASTM C-618 limit (6%). The product LOI and carbon level is indicative of a high quality material.

Test	Feed %LOI	Product %LOI	Product %C
1a	4.71%	2.68%	2.03%
1b	4.76%	2.55%	2.21%
2a	4.40%	2.28%	2.11%
2b	4.05%	2.22%	1.84%
3a	3.14%	2.15%	0.99%
4a	4.62%	2.52%	2.10%
4b	4.79%	2.47%	2.31%
5a	4.79%	2.34%	2.45%
5b	4.51%	2.13%	2.38%
6a	4.79%	2.61%	2.18%
6b	4.19%	2.47%	1.73%
7a	4.72%	2.79%	1.94%
7b	4.57%	2.61%	1.96%
8a	5.44%	2.64%	2.80%
8b	4.21%	2.77%	1.43%
9a	4.33%	2.82%	1.51%
9b	4.19%	3.01%	1.18%
10a	4.57%	2.87%	1.70%
10b	4.75%	2.85%	1.90%
11a	4.60%	3.16%	1.44%
11b	5.24%	3.15%	2.09%
12a	4.60%	3.16%	1.44%
12b	5.24%	3.15%	2.09%
13a	5.30%	2.97%	2.32%
14a	4.71%	2.72%	1.99%

Task 2 - Filler and Superpozzolan Testing

No activity.

Task 3 - Finalization of Flowsheet Design

No activity.

Task 4 - Construction of PDU Facility.

No activity.

Task 5 - Operation of PDU

No activity.

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