

**An Integrated Separation Technology for
Processing Coal Combustion By-Products
and Organic Bio-solids**

by

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Senior Mineral Processing Engineer

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EXECUTIVE SUMMARY

An integrated separation process utilizing off-the shelf technology for the treatment of coal combustion by-products and organic sludge is presented in this report. The process provides for the economically viable and environmentally sound utilization of nearly 100% of coal combustion byproducts to produce multiple new valuable products. The major goals of the integrated process were to manage and consume the huge inventories of combustion byproducts generated daily by coal-burning boilers in the state of North Carolina, reducing major organic waste streams such as paper mill biosolids and animal waste byproducts and create new jobs in the economically disadvantaged regions of the state.

A flowsheet for an integrated wet separation pilot plant was designed and operated after bench-scale testing. The purpose of the pilot plant was to confirm the design data and parameters on a continuous basis, to provide reliable operation data for commercial plant design, establish design criteria, operation parameters, process efficiency and preliminary economic and market

feasibility. In addition, the pilot plant produced bulk quantities of the multiple products for testing and market analysis. The integrated process consisted of wet separation technology including size separation to produce bottom ash, and flotation to produce high carbon product and low carbon fly ash. Next the intermediate carbon-containing ash was used in conjunction with organic biosolids to produce lightweight aggregate(LWA) via the patented pyro-process (US Patent No. 5,057,009). The results of the bench-scale testing and the pilot plant testing are presented in this report as well as preliminary economic and marketing initiatives for commercialization.

Results from the bench-scale tests indicated that the integrated process could separate coal combustion ash into bottom ash, high carbon product and low carbon fly ash. The recovery of high carbon product was 8.4% with a corresponding carbon grade of 66.5% LOI. The average yield of low carbon fly ash was 53.6% with a corresponding carbon grade of 1.10 % LOI. These results were confirmed on a continuous pilot plant scale where the high carbon recovery was 3.6% with a corresponding carbon grade of 71.3% LOI. The average yield of low carbon fly ash from the pilot plant was 73.4% with a corresponding carbon grade of 2.50 % LOI. In addition the intermediate carbon residue from the wet process was successfully combined with paper mill sludge to produce synthetic lightweight aggregate (LWA).

Recovery of carbon as a cleaner concentrate from the bench-scale tests ranged from 3.1 to 12.92% whereas the corresponding grade of carbon as % LOI ranged from 61.0 to 72.5%. The yield of low carbon fly ash was in the range of 44.9 to 59.9 % whereas carbon in the fly ash ranged from 0.05 to 4.01% LOI. Approximately 20.0 tons (40,000 lb) of coal ash from Progress Energy Carolinas Skyland Plant was processed through the pilot plant. The throughput for the pilot plant ranged from 542.0 to 602.3 lb/hr with an average of 557.2 lb/hr. Average yields (% weight of feed) of low carbon fly ash, high carbon product, bottom ash and intermediate carbon product were 73.4%, 4.7%, 7.8% and 10.3 % respectively. Percent carbon (measured as % LOI at MRL) ranged from 0.66 to 7.63 % with an average of 2.52% for the low carbon fly ash, 67.16 to 75.33 % with an average of 71.95% for the high carbon product, and 12.67 to 61.56% with an average

of 41.52% for the intermediate carbon product. Calorific value in Btu/lb of the high carbon product ranged from 9,337 to 10,379 with an average of 9,936 Btu/lb.

The difference in the average yield of fly ash between the bench tests and the pilot plant tests was due to the separation screen size for fly ash, 100 mesh (0.0058 in or 149 μm) for the bench-scale tests and 60 mesh (0.0098 in or 250 μm) for the pilot plant tests.

An initial sample of the low carbon fly ash from the pilot plant tests (separated on a 60-mesh screen) that was sent out to a certified commercial laboratory for ASTM C-618 specification passed all the specified tests except % fineness which was 47.6 % compared to the maximum limit of 34%. Another sample of the same ash separated on a 200-mesh sieve (0.0029 in or 75 μm) was sent out for ASTM C-618 testing. This time around, % fineness was 27.3 ± 2.1 which was below the maximum limit.

It was established that grinding of the coal ash material from PEC plant was necessary to produce a fly ash product that would consistently meet the ASTM C-618 specification for % fineness (34% maximum) and further screening out of the coarser fraction would generate a fly ash product with consistent % carbon below 2.0% LOI.

Synthetic lightweight aggregates (LWA) were successfully produced on both bench-scale and pilot scale tests. Pellets of varying shapes, color and sizes were produced from the available stock of byproducts. These pellets were produced on a bench scale by tumbling, extrusion, and hydraulic press. Bulk quantities were produced with flat die pelletizing press and roll mill briquetting machines. Production of these bulk quantities was subcontracted to outside pelletization companies. They were subsequently fired in a laboratory muffle furnace to produce synthetic lightweight aggregates of varying color, size and shape, strength, loose density, specific gravity and gradation. These lightweight aggregates had the following characteristics: loose density of 40.0 - 55.0 lb/cu ft, 0.9 - 1.80 specific gravity, 5.0 - 15.0% water of adsorption depending on the size and shape of the pellets. ASTM C-330 Specification Test was performed

on the synthetic LWA produced from the bench-scale tests. The measured 28-day compressive strength was 3,250 pounds per square inch. ASTM C-330 Specification Test on the bulk LWA sample produced during the pilot plant study is still pending. Based on the physical characteristics of the LWA that have been produced during the pilot plant studies, we expect the compressive strength to be above the 3,250 psi obtained from the bench-scale studies. The results of the ASTM C-131-03 Standard Test for Resistance and Degradation for the LWA from the pilot plant study showed LA Abrasion of 29.0 % loss. The results of the ASTM C641-98 Standard Test for Staining showed no stain. Results of the TCLP test on a sample of the LWA showed that the eight elements evaluated were all below the regulatory levels.

The four major end products from a commercial plant utilizing the integrated process technology would be high carbon product, bottom ash, low carbon fly ash, and lightweight aggregates (LWA). The potential uses for the carbon product include fuel to the power plants, application as direct charge carbon to electric arc furnace and direct iron production for the steel industry. As a fuel, the value could be estimated on the basis of the Btu content and will range from \$45 to \$50 per ton. The demand for the high carbon product for this application will depend on the location and size of the power plants. The carbon for direct charge is high value at \$100 per ton. The final potential use of the high carbon product is the proprietary iron reduction process of Nucor Steel. The value of carbon for this application would be lower around \$50-60 per ton, but the processing cost would also be lower. The demand for the high carbon for the iron reduction application is higher (7,000 tons per year being a low end). Pelletization of the high carbon product to achieve the specified size requirement needed for these applications may be economically feasible.

Low carbon fly ash (Class F) is used in concrete application as replacement for Portland cement. Local producers of cement blocks are reluctant to use low carbon Class F ash since concrete made with Class F fly ash takes longer to develop strength. Therefore, in this local region, concrete block manufacturers were not considered as a potential market for the low carbon fly ash. The other use of Class F ash is for ready mix concrete application. These producers of ready mix

concrete use large quantities of class F ash which they buy from Santee Cooper in South Carolina. One major ready mix producer, Cemex, has a long-term contract with Santee Cooper and they are not interested in switching from them at this time. The other major producer of ready mix, Southern Concrete, could use all the 40,000 tons of ash from the proposed commercial plant and would be glad to put that in a contract. The price for low carbon fly ash in our region is in the range of \$20 to \$30 per ton.

None of the concrete block manufacturers in western North Carolina currently uses bottom ash in their products because the production difficulties associated with the variability in the quality of bottom ash off sets the potential cost savings

The lightweight aggregate (LWA) market in western North Carolina is served by Carolina Stalite that supplies the customers with expanded shale with loose bulk density of 54-56 lb/ft³ at a price of \$25 - 35 per ton depending on the density. The demand for LWA in our region is in the range of 70,000 to 100,000 tons per year.

The main focus for business development was a fully integrated commercial plant producing fly ash, bottom ash, high carbon product and lightweight aggregate. This would potentially utilize all (100%) of the coal ash and eliminate land filling. A commercial plant based on the fully integrated concept and processing 100,000 tons of coal ash per year would require an estimated initial investment of \$10.0 million without land and utility costs. Three other different scenarios for commercial plant development were considered. The return on investment (ROI) as a ratio of yearly income to investment expressed as percent was calculated for the various business scenarios. The return on investment ranged from a low of 10% to a high of 16%.

A detailed account of the economic, marketing and business initiatives for the process technology can be found in an accompanying report “Integrated Plant for processing High Carbon Coal Ash - Economic/Business Feasibility Study” by Ken Butcher

INTRODUCTION AND BACKGROUND

The NC State Minerals Research Laboratory (MRL) and Waste Reduction Partners (WRP) of the Land of Sky Regional Council joined efforts in 2000 to initiate a technically sound and practical program for the management of high-volume coal combustion by-products (CCBs) in North Carolina. The accumulation of CCBs from coal burning boilers throughout the state of North Carolina posed a substantial storage problem, and the area was overripe for an effective management program, which would be environmentally sustainable. In particular, the public utility generators had filled storage ponds on their properties, and the storage overload had grown to critical proportions. Much the same situation existed for paper mill biosolids, which were stored on site in landfill cells along with daily output of CCBs from the mill boilers.

A conversion process via the patented pyro-process (US Patent No. 5,057,009) to produce synthetic light weight aggregates (LWA) from CCBs and paper mill sludge was conceived to process large quantities of both these waste materials. MRL and WRP initiated and formed a consortium that brought together academia, private industry and state agencies to evaluate the production of LWA by the pyro-process.

Participants of the consortium had various interests in eliminating CCB inventories through the creation of useful building products and recovery of high quality carbon. A product, which would also consume paper mill biosolids, was the first research and development target explored. The participants formed a voluntary consortium of academia, private industry, state agencies, and community active groups. The industry partners included representatives from the power companies, industrial ash generators, expeditors for coal ash products, as well as concrete block manufacturers. It was important to welcome interested parties from not only the western North Carolina area and other parts of the state but also from neighboring states. Attendees from Tennessee, South Carolina and Georgia brought new perspectives to be considered and contributed added breadth to the choices of processing methods and the range of ash-derived products.

Membership of this consortium has evolved during the intervening years to include the following participants and supporters: Progress Energy Carolinas; Duke Energy; Santee Cooper; Full Circle Solutions; Land-of-Sky Regional Council; NC Department of Pollution Prevention and Environmental Assistance; Ecusta Business Development Center; Blue Ridge Paper Products; Jackson Paper and Manufacturing Company; Miller Perlite; Appalachian Products; General Shale Brick; Metromont Materials; Small Business Technology Development Center of the University of North Carolina system and North Carolina State University

The initial development program conducted at the Minerals Research Laboratory (MRL) was to formulate an acceptable LWA product, which fulfilled user specifications for light weight concrete block and structural concrete application. Support work and program coordination efforts were furnished by retired technical volunteers in WRP.

The work advanced steadily, using proven technologies well established in the mineral industry, to generate a very satisfactory LWA. The consortium then concluded that a more comprehensive CCB conversion program would have more appeal for full scale manufacture and commercialization, if a variety of products from CCB separation were generated. This product assortment would include bottom ash, low carbon fly ash, recovered carbon of high purity, as well as the synthetic LWA. The bottom ash would serve the concrete block market, the fly ash was in demand for ready-mix concrete, the high carbon product could be pelletized and used as a fuel for reburn or by the steel industry, and the LWA was a sought-after component for light weight concrete applications. After scouting all these potential markets and finding them to be viable, the consortium then inaugurated an integrated pilot plant program for the CCB separation and conversion.

A flow scheme for the separation was designed, which could be scaled up from the laboratory-to-pilot plant-to full commercialization. Techniques and equipment, fundamental to the mining and minerals commercial sectors, were integral parts of the design.

The plan was organized in three (3) parts: (1) Phase I – bench scale evaluation studies (2) Phase II continuous pilot plant evaluation and (3) Phase III – conceptual commercial plant design including marketing efforts and attraction of business entrepreneurs to build a full scale manufacturing plant.

Phase II would serve to validate the process in a continuous mode and produce substantial quantities of products for preliminary marketing efforts and for demonstration-of-use purposes. A marketing consultant with considerable experience and a very progressive plan for this study was selected.

The consortium agreed to the name, CAROLINA ASH PRODUCTS, (CAP), for the integrated pilot plant program. The products generated might be designated with the CAP label as: bottom ash would be named CAP Granules, low carbon fly ash would become CAP Ash, recovered high carbon product would be CAP Carbon, and LWA would have the name CAP Stone. Phase II of the program processed 20 tons of ponded ash from Progress Energy storage with favorable results. This processing campaign proceeded with relative ease at a rate of 600 lbs of raw ash per hour.

Preliminary Studies:

Prior to committing laboratory research and development effort as well as support and coordination activities to this program, it was necessary to determine (1) the level of need for a freshly launched CCB re utilization program in this state and (2) what degree of versatility was necessary for such a process. State agency records and a survey of recent boiler operations indicated that in the state of North Carolina at least 77 facilities manage CCBs from their boiler operations. Despite continuing development efforts in more eastern parts of the state, the use of CCBs for commercial applications has not increased to a level which alleviates the need to store vast quantities in private monofills on the generator's property. More than 1.25 million tons of CCBs are landfilled in NC annually, with 300,000 tons per year generated in western NC alone. Many different boiler types, which span both very old units and those that are modern and efficient, were to be found. These included stoker-fired units, pulverized coal boilers, slag-tap

furnaces, and fluidized bed combustion boilers covering a range of sizes. Any systems designed to separate CCB components from all these sources and convert them to useful secondary products would need broad processing latitude and flexibility. Up to the present there has been a paucity of research done in the western part of the state to find productive alternatives to storage in landfills. With tipping fees in our largest landfill now approaching \$40 per ton and escalating transportation fees (about \$200 per truckload up to 40 tons, traveling a distance of 25 miles), the problem has become urgent both economically and ecologically. A well-designed, integrated wet process for CCB separation into value-added products had much potential to be less expensive than landfilling as well as to provide saleable materials with acceptable profit margins.

These preliminary investigations also revealed that other major waste streams, in particular paper mill sludge and municipal and animal waste biosolids were important candidates for priority attention. Since these biosolids could furnish the organic component of a synthetic LWA product, they could be consumed as part of the same program. This added interest value to a growing list of partners to support and participate in the program.

A lengthy series of bench experiments was carried out to develop a process to yield the products favored by members of the consortium, including the ash suppliers, users of the technology, and the ultimate customers for the products.

Preliminary studies were the most inclusive that could be achieved for screening crude ash mixtures of varying quality and composition. Typical batches of ash, both from daily outputs and storage ponds of at least 15 different boiler locations in the state of NC were examined in bench studies. These covered as many boiler types of different sizes as were practical, including stoker-fired boiler units, pulverized coal boilers, cyclone boilers, slag-tap furnaces, and fluidized bed combustion boilers. Individual components, isolated from the separation sequence, were evaluated for quality as marketable products. Promising results from these evaluations determined the family of products to be pursued, using the integrated process.

The treatment of numerous batches of varying compositions has yielded reproducible separations. The resulting products -- low carbon fly ash, recovered carbon, and bottom ash -- all met the specifications for the intended use. Separation residues, along with additional crude ash mixtures were combined with biosolids and pelletized. Firing the resulting pellets at 2,200° F furnished an attractive LWA product. Scale-up of this process was advanced. A 50 lb-batch of LWA was produced by firing dry pellets in a gas-fired furnace at the Brick Research Center, Clemson University and used for ASTM C-330 Specification testing. Indications were favorable that LWA was a commercially worthwhile candidate to include in the family of ash-derived products from this project.

Among the early considerations in the study was a possible final location of another pilot plant in Phase III of the program. This choice was influenced by a number of community factors. Job creation is now a major thrust in the western part of the state, which has suffered recent effects of plant closings and accompanying rises in unemployment. The ideal area for the pilot plant proved to be a recently designated Brownfields site in Transylvania County. A major specialty paper producer closed its plant on that property over 3 years ago. Ecusta Business Development Center now owns the property and has several vacant production buildings available. This represented an attractive opportunity for community economic revitalization, and the project pulled in added state and community support for that reason. The plan for the pilot plant at Ecusta has been put on hold at this stage due to unexpected circumstances affecting the site.

METHODOLOGY

The principal flowsheet for the wet separation process for coal ash was designed by MRL on the basis of bench-scale testing also performed by MRL. Discussion of the results of the bench-scale testing is included in this report. The purpose of the pilot plant was to confirm the design data and parameters generated by batch tests on a continuous basis, to provide reliable operation data for commercial plant design, establish design criteria, operation parameters, process efficiency and preliminary economic feasibility of the process. In addition, the pilot plant program was to produce bulk quantities of the products for testing and market analysis.

Bench-scale Wet Separation (Froth Flotation) Testing

The bench-scale flotation tests used approximately 500 - 1,000 grams of raw ash as feed. The procedure consisted of removing unwanted trash and oversize cinders by screening on a 6-mesh (0.131 in or 3.33 mm) sieve. The scalped raw ash was subjected to varying methods of size separation to remove the bottom ash before or after flotation. The initial tests involved attrition scrubbing of the scalped ash (minus 6-mesh fraction) at 50% solids in an attrition scrubber. The scrubbed product was screened on a 100-mesh (0.0058 in or 150 μm) sieve to separate it into plus 100-mesh coarse and minus 100-mesh fine fractions. Each fraction was subsequently advanced to the flotation process and treated separately to remove unburned carbon. Another procedure involved attrition scrubbing the raw ash at 50% solids to break all the lumps before removing unwanted waste and cinders on a 6-mesh sieve. Next the scalped product was separated into plus 100-mesh and minus 100-mesh fractions by wet screening and each fraction was processed separately by flotation to recover unburned carbon. The final size separation procedure consisted of screening the raw ash on a 6-mesh sieve to remove unwanted material and cinder, attrition scrubbing of the scalped material at 50% solids and separating the product into plus 30 mesh (0.0234 in or 595 μm) and minus 30-mesh fractions by screening. Each fraction was subsequently floated separately to recover the unburned carbon. The procedure for flotation was to condition the pulp in a beaker at about 35% solids with addition of reagents for about 1.0 minute. Next, the conditioned pulp was transferred into the flotation cell, the pulp density was

adjusted to about 25% solids and a rougher concentrate was floated out (until completion). The rougher tailings were removed from the cell, filtered and dried. Each rougher tailings sample was subjected to dry screening on a 100-mesh (0.0058 in or 150 μm) sieve to separate plus 100-mesh coarse fraction as bottom ash and leaving minus 100-mesh fraction as the low carbon fly ash. The rougher concentrate was transferred into the cell and refloated to produce a cleaner concentrate and cleaner tails. Both cleaner concentrate and cleaner tails were filtered and dried. The only process variable evaluated was reagent addition. Fuel oil and pine oil were used as the collector and frother respectively for the initial tests whereas Cytec S-8259 and Cytec S-4760 collectors and Cytec F-533 frother was used for the subsequent tests. Two tests used coal ash samples from Duke Energy Plant and Ecusta Business Development Plant. One single test evaluated biodiesel from NC State University Solar Center as a collector for carbon flotation. Percent carbon in the flotation products, unburned carbon, low carbon ash and intermediate carbon was determined as % LOI at MRL. A few of the bench-scale test samples were submitted to outside certified commercial laboratories for proximate analysis and mineral analysis.

Loss on Ignition (LOI) Determination.

Loss on ignition (LOI) is considered as a good estimate for percent carbon in coal ash. Loss on ignition is determined as the weight loss of a sample from 110° C to 750° C. It is measured by weighing a ground sample, heating it to 1,000° C for 1.50 hours in a muffle furnace, cooling it to room temperature in a desiccator, and then weighing the sample again to determine the percent weight loss. The loss on ignition was used as the standard quality control parameter for the pilot plant operation.

Bench-scale Tests for Lightweight Aggregate (LWA) Production

Several bench-scale tests were performed during an earlier project funded by Progress Energy Carolinas in 2003. The bench-scale batch studies determined the proper mixture of ash and sludge to produce LWA. Mixture ratios of 80-87% ash and 5 -16% sludge were evaluated. The ash consisted of 90% by weight of Progress Energy's ash and 10% of Ecusta's ash. In some of the tests, fine perlite from Miller Perlite Company, Morganton, NC was used as a substitute for

binder and sludge at the weight ratio of 2.5 - 10.0%. Carboxymethylcellulose (CMC-T) by Hercules, Inc. and bentonite clay were used as binders at the weight addition of 0-2.7%.

The procedure included tests that produced disc pellets using a laboratory hydraulic press, pseudo-spherical pellets by tumbling in a rotating cylinder and extruded pellets using an extrusion machine. Initially mixing of the raw byproduct materials was done manually with a spatula in a stainless steel pan. Finally, a laboratory Muller Mill was used for mixing and the spherical pellets were made by tumbling the mixed components in a laboratory rod mill without the rods. The green pellets were dried in a laboratory oven to remove moisture and also to increase the strength of the pellets. The dry pellets were heated to the firing temperature in a bench top muffle furnace to produce small quantities (about 1.0 lb at a time) of fired pellets. Prior to the current pilot plant studies, several batch tests were performed to produce LWA using the procedures described above. These bench tests used the designed mixture ratio of 85-90% coal ash to 10-15% sludge and 0.5% organic binder, CMC-T. The standard temperature for firing the pellets was nominal 2,200° F, although some of the tests evaluated lower temperatures as well as varying the dwell time for firing (15.0 to 60.0 minutes). Results of the current bench tests are included in the appendix and discussed in this report.

After the design parameters for production of the LWA had been established by the bench-scale tests, it was decided to contract the production of bulk quantities of pellets to an outside commercial laboratory. Preliminary evaluation of the pelletization was contracted to LCI Corporation of Charlotte, North Carolina, to produce approximately 50.0 lb of pellets. LCI Pelleting Corporation used a laboratory Pellet Press Model 14-175 to pelletize a mixture of fly ash and paper mill sludge into large dust-free, free-flowing pellets to be dried and fired into lightweight aggregate. Approximately fifty pounds of a mixture consisting of a finely blend of fly ash, paper sludge and organic binder with a moisture content of 10% was prepared at MRL facility and shipped to LCI facility in Charlotte. Four samples were provided composing of the same moisture level but prepared with different sources of paper mill sludge and labeled Batches #1 through #4.

The procedure for preparing the mixture involved drying the moist coal ash and raw paper sludge separately in the laboratory oven at 200° F overnight to remove moisture. Next, the coal ash was screened on an 8-mesh (0.093 in or 2.38 mm) sieve to remove coarse particles and trash. The scalped ash and dry paper sludge were then mixed in varying proportions in a Muller Mill to produce the finely blended mixture at about 10-15% moisture content. Batch #1 contained Progress Energy Carolinas' (PEC) coal ash and P.F. Glatfelter (PFG) paper (flax) sludge. Batch # 2 contained PEC ash and P. F Glatfelter landfill (PFG LF) paper sludge. Batch #3 contained PEC coal ash and Ecusta flax pulp sludge. Batch #4 contained PEC ash and Blue Ridge Paper Products' (BRPP) sludge. Eight tests were performed at LCI to produce eight batches of pellets. The test conditions for preparing the pellets at LCI with the LCI/Kahl 14-175 pellet press operating at 100 rpm are listed in Table 1 below:

Table 1: Test Conditions for Batch Pelleting with LCI/Kahl Pellet Press

| Run # | Mixture Description and Composition | | | | Die Dimension, mm | | Rate kg/hr |
|-------|-------------------------------------|----------|--------|-------------|-------------------|---------------|---------------|
| | Batch # | Coal Ash | Sludge | Moisture, % | Diameter | Pressway s | |
| 1 | 1 | PEC | PFG | 10 | 6.0 | 18.0 | N/A |
| 2 | 1 | PEC | PFG | 10 | 6.0 | 12.0 | N/A |
| 3 | 2 | PEC | PFG LF | 20 | 6.0 | 12.0 | N/A |
| 4 | 2 | PEC | PFG LF | 20 | 6.0 | 24.0 | N/A |
| 5 | 2 | PEC | PFG LF | 20 | 6.0 | 33.0 | N/A |
| 6 | 3 | PEC | Ecusta | 20 | 6.0 | 33.0 | 168 |
| 7 | 4 | PEC | BRPP | 20 | 6.0 | 33.0 | 168 |
| 8 | 4 | PEC | BRPP | 20 | 8.0 | 40.0 | 168 |

Run #1 and #2 used Batch #1 mixture. In the first run (Run 1) the mixture from Batch #1 was manually fed to an LCI/Kahl 14-175 pellet press operating at 100 rpm with a die having 6.0 mm

diameter holes and 18.0 mm pressway length (die # 63142). The operation of the unit in this run was erratic, exhibiting spikes in current draws (5-8 amps) and significant vibration. Warm pellets were formed with a lot of powder and fines. Run 2 was made with the same Batch #1 material but used a die having 6.0 mm diameter holes and 12.0 mm pressway length (# 63171). The Pellet Press operated better, but discharged more powder and fines and made softer pellets. In the subsequent runs, the moisture content of the mixture was increased to 20% by addition of water. Run 3 used Batch # 2 material at 20% moisture that was fed to the Pellet Press with a die having 6.0 mm diameter holes and 12.0 mm pressway length. Soft, well-formed pellets were produced with fewer fines. Some of the Batch #2 material was run with a die having 6.0 mm diameter holes and 24.0 mm pressway length (#63184) in Run 4. Well-formed pellets were produced at 30° C and 4-5 amps with negligible fines. Run 5 was with same Batch #2 material with a die having 6.0 diameter holes and 33.0 mm pressway length (# 61415). Good-looking pellets with no fines were formed at 4.5 amps and a temperature of 30° C. Run 6 utilized Batch #3 material and was run with same dies as Run 5 to gather rate data. Again good pellets were formed at the rate of 168 wet kg/hr (134 kg/hr dry). The pellets were produced at 44° C and drew an average of 6.0 amps. Runs 7 and 8 used Batch #4 material. A small amount of material was run in Run 7 to provide a direct comparison with the pellets made in Run 6. Run 8 was made with a larger die having 8.0 mm diameter holes and 40.0 mm pressway length (# 63298). Good-looking pellets were formed at 5.0 amps. The pellets produced from all runs except Run 1 and Run 3 were retained and shipped to MRL for further evaluation.

Pilot Plant Operation of the Wet Separation (Flotation) Process

The procedure for the wet process for coal ash separation pilot plant consisted of delivery of the raw coal ash from Progress Energy's Skyland Plant to MRL facility in Asheville. The raw ash was scalped on a 6-mesh screen to remove coarse cinders and trash. Next the screened product was fed at specific flow rates into an attrition scrubber. From the scrubber, the product was screened at 30-mesh to remove the 6 x 30 mesh coarse fraction as bottom ash product. The undersize minus 30 mesh fraction was advanced to a conditioner where the appropriate reagents were added. The carbon was floated as a rougher flotation concentrate followed by a cleaner

flotation of the rougher concentrate to produce a cleaner concentrate. The concentrate and tailing products were filtered, and subsequently dried. Approximately 20 tons of coal ash from Progress Energy's Skyland plant was processed through the pilot plant. The actual pilot plant operation lasted for a period of twelve (12) days including the shake down runs, but the whole project took about three months to complete. Evaluation of the process variable was performed during this period.

Construction of Wet Separation Pilot Plant and Shake-Down

The flowsheet for the wet separation process for coal ash is shown in Figure 1. MRL provided all the equipment defined in the flowsheet for the exclusive use of the project. MRL personnel performed the normal plumbing within the plant including connection of pipes and tubing to individual equipment. Also, normal electrical connections from the individual equipment to the main power source in the building were made by MRL personnel. After construction, the pilot plant underwent a shakedown to evaluate operational characteristics of the plant, including running of water through the pilot plant to test for leaks, flow rates, hydrodynamics, smooth operation and mechanical operation of each individual equipment. This was followed by running at least 4,000 lb of coal ash through the pilot plant. This was to validate the pilot plant for mechanical equipment durability, operational characteristics and functionality of the flowsheet.

a. Sample Description and Preparation.

Progress Energy Carolinas supplied approximately 20 tons (40,000 lb) of coal ash from their Skyland, NC plant. The raw coal ash from the utility company was delivered at MRL facility as moist material at 70- 80% solids. It was air-dried on a concrete floor to remove some of the moisture before screening on a 6-mesh sieve to remove an unwanted trash and coarse cinder product. The scalped coal ash (minus 6 mesh fraction) was stored in 2,000-lb polyolefin supersacks at 5 -10% moisture.

b. Methodology for Pilot Plant Operation.

The process and equipment configuration was designed by MRL and approved by CAP

consortium. The following is the general description of the pilot plant operation.

The pilot plant was operated by MRL technicians. Their duties included adjusting process parameters, sample collection and preparation, data recording and other minor adjustments in the performance of the equipment.

All process parameters and equipment changes during actual plant operation was established by MRL. Optimization of the process and evaluation of the pilot plant performance was undertaken by MRL and approved by sponsors.

The operation of the pilot plant was suspended at appropriate times (recommended by stakeholders), in order to evaluate the results of the process.

The pilot plant was designed to treat between 600 and 700 lb/hr of the scalped coal ash. Minus 6-mesh scalped ash was fed to the pilot plant in 2,000 lb polyolefin supersacks via a Tecweigh FC20 belt feeder with a hopper. The feed was discharged into a three-pot Wemco attrition scrubber where the ore was scrubbed at 50-60% solids by weight. The retention time in the attrition scrubber was about ten (10) minutes. The first addition of water to this wet system commenced from this stage of the process. After scrubbing, the pulp was screened on a Derrick horizontal vibratory screen with 30-mesh (0.0234 in or 0.595 mm) screen size to remove the 6 x 30 mesh oversize as bottom ash product. The undersize minus 30 mesh fraction flowed down by gravity into the carbon flotation conditioner. Flotation reagents, promoter and frother were added to the conditioner. Retention time in the conditioner was between one (1) to three (3) minutes. The conditioned pulp discharged into six (6) banks of #7 Denver Sub A flotation cells where the pulp density was adjusted to below 25% solids with addition of water. Unburned carbon in the coal ash was floated off and collected as rougher float concentrate whereas the low LOI ash was retained in the flotation cell as rougher tails. The rougher float concentrate was cleaned in another two (2)-banks of #7 Denver Sub A flotation cells to produce a cleaner flotation concentrate (high carbon product) and cleaner flotation tails. Both cleaner concentrate and

cleaner tails were stored in steel drums. They were subsequently filtered on the pan filter, dried in the laboratory dryers and saved in 55-gallon drums. The rougher flotation tails were advanced to a Sweco vibratory screen with 60-mesh (0.0098 in or 250 μm) sieve to remove the coarse plus 60-mesh oversize as the second 30 x 60 mesh bottom ash product. All bottom ash products were saved wet in 55-gallon steel drums. The Sweco screen underflow (-60 mesh fraction) was sent to the settling tank (thickener) to remove most of the entrained water. Sulfuric acid and Cytec Superfloc A-100 flocculant were added to the screen undersize slurry to improve the settling of the solids and production of clear supernatant in the thickener. The thickener overflow was discharged into the wastewater treatment system. Underflow from the thickener, at more than 45% solids, was periodically pumped into 55-gallon drums and later filtered on the pan filter. The filter cake from the pan filter was also collected in 55-gallon drums and dried in the laboratory dryers as needed. During operation of the pilot plant, periodic samples were taken of the various unit operations for control purposes and to obtain design data. Toward the end of each pilot plant run, usually after the system has attained steady state conditions, samples were collected from the various circuits. Flow rates through the pilot plant were also measured periodically and also during the collection of the process samples. These samples were processed to provide the needed design parameters

Production of Bulk Quantities of Green Pellets from Coal Ash and Sludge

Production of lightweight aggregates (LWA) by the pyro-process consisted of batch mixing of bulk quantities of coal ash (a combination of processed ash from the pilot plant and unprocessed ash from the utility plant) and paper mill sludge, making pellets from the mixture, drying the green pellets and firing in a rotary kiln to produce the LWA. Approximately 4,000 pounds of green pellets were produced from coal ash and sludge to be used for subsequent firing to manufacture synthetic lightweight aggregates (LWA). Production of this bulk quantity of green pellets was contracted to a commercial facility, Carolina Pelleting & Extrusion in Newton, North Carolina. Two tons of coal ash (raw ash from Progress Energy and processed ash from the pilot plant) and 600 pounds of sludge from P.F. Glatfelter landfill were shipped to Carolina Pelleting facility in Newton, NC, for making the green pellets. The procedure consisted of mixing in

batches moist coal ash (processed and raw) and wet sludge at a weight ratio of 86.5% to 13% with about 0.5% binder in a mixture. Two batches each containing 2,000 lb of coal ash, 300 lb sludge and 10.0 lb of organic binder, sodium carboxymethylcellulose, per batch were prepared and used for the pressed pelletization. Carolina Pelleting and Extrusion used the pelleting process with flat die pelleting press. Their equipment is a commercial version of the LCI/Kahl press pelletizer used for the batch test. The process consisted of proportioning of the composite materials, mixing them in the right proportions in a mixer, feeding the mixture into the pelleting press to make the pellets, drying and cooling of pellets and finally screening to remove the unwanted size fractions. The green pellets were dried in a vibratory fluidized bed dryer attached to the pelletizer. Two sizes of pellets were produced, 5.0 mm and 12.0 mm diameter. Moisture content of the mixture was studied as the variable during the pelletization tests. Next the pellets were brought back to MRL and advanced to the next phase of the program. At MRL, the dry green pellets were screened to remove the fine powders generated by moderate mechanical handling during transportation. The 5.0 mm diameter pellets were screened on a 12-mesh sieve whereas the 12.0 mm diameter pellets were screened on a 6-mesh sieve. The dry pellets were fired in the rotary kiln at 2,200° F for a specified time depending on the feeding rate into the kiln. The fired pellets from the kiln were cooled quickly in air to produce the lightweight aggregates.

Preliminary Marketing, Economic, and Business Feasibility Study

A consultant was contracted to study the preliminary marketing, economic and commercial aspects of the concept. The main objectives were: (i) to identify specific customers for the end products, (ii) to identify specific technical and business issues of the potential customers of these end products, (iii) to estimate the cost of plant equipment and construction, (iv) to estimate the cost of manufacturing the end products, (v) to design a profit model for plant operation and derive a return on investment (vi) to explore the possible business structure for a commercial plant construction and operation, (vii) to identify specific environmental regulations and procedures required for planning commercial construction and operation. Funding for this study was provided by the North Carolina Rural Economic Grant through the Land of Sky Regional Council, Asheville, NC. The consultant contacted and visited several ash generators, potential

customers for the end products, equipment manufacturers, federal and state regulators, and community interest groups to collect reliable marketing, economic and business data for this study. Some of these stakeholders, especially the coal ash and bio-solid generators, were partners of the CAP consortium and they included, Progress Energy Carolinas, Duke Energy, and Blue Ridge Paper Company. The potential customers of the end products included Nucor Steel Company, Charlotte, for high carbon product, Cemex (formerly Metromont) and Southern Concrete, Asheville, for the Class F (low carbon) fly ash. General Shale and Carolina Stalite were the last two potential customers of bottom ash and light weight aggregates that were contacted by the consultant. Information and data collected included current and future uses and prices of the end products as well as current and future marketing trends (demand and supply) in the region. Equipment manufacturers provided the estimated capital and operational cost of plant and equipment. The first step in estimating the plant cost was to determine the production rates for each piece of equipment. This type of information was provided by the material balance flow sheet generated as a spread sheet from the pilot plant operation data. This material balance spread sheet is included in Appendix D. A partial list of equipment for the plant could also be prepared from the pilot plant data and the material balance sheet. Next, cost estimates were obtained for each piece of equipment from suppliers and manufacturers. In most cases, the manufacturers suggested that more testing on the actual material would be required to make a firm recommendation and quote. This was especially true of filters and dryers where transport of water and heat through a bed of material is highly dependent on particle size and packing density. A simple model MS Excel spread sheet for plant capital cost estimates, profit and loss summary and return on investment (ROI) for varying business scenarios was designed by the consultant. An accompanying report entitled “Integrated Plant for processing High Carbon Coal Ash - Economic/Business Feasibility Study” provides in-depth account of the marketing, economic and business initiatives for this project.

DISCUSSION OF RESULTS

The lists of Tables, Figures and Photographs for this report are enclosed in Appendix A, Appendix B and Appendix C respectively, whereas raw operation data sheets are enclosed in Appendix D. Operation data generated from the pilot plant runs are tabulated in Table 3 through Table 7. Percent carbon in the products was determined as % LOI at MRL for quality control purposes. Some of the same samples were sent out to a certified chemical laboratory for determination of % carbon. Selected samples of the high carbon product samples were sent out to an outside certified laboratory for proximate analysis and some of the low carbon fly ash samples were submitted to an outside laboratory for mineral analysis. Results of these analyses are listed in Tables 12 and Table 13. The data were used to establish correlations between the % LOI determined at MRL and % carbon determined by a certified laboratory. The correlation between % LOI at MRL and Btu/lb determined by certified laboratory was also established. These correlation curves are shown in Figure 4a through Figure 4c and their corresponding data are listed in Table 8. There were good correlations among the variables with correlation coefficients (R^2) in the 0.995 range. Percent carbon and Btu/lb values were calculated from the measured % LOI at MRL and are listed in Table 9.

Bench-scale Flotation Tests

A summary of the results of the bench-scale tests is tabulated in Table 2 and individual data sheets for each test are included in the appendix. As expected, there was good correlation between the recovery (% yield) and grade of carbon (% LOI). Figures 5a and 5b show the correlation between the grade (% LOI) and recovery (% yield) for the high carbon concentrate. The higher the recovery of carbon as cleaner concentrate, the lower the grade of carbon as % LOI. Recovery of carbon as a cleaner concentrate ranged from 3.1 to 12.92% whereas the corresponding grade of carbon (% LOI) ranged from 72.5 to 61.0%. The yield of low carbon fly ash was in the range of 44.9 to 59.9 % whereas % carbon in the fly ash ranged from 0.05 to 4.01% LOI. When the feed material was split into coarse and finer fractions and floated separately, the results showed that higher grade carbon was recovered from the coarser fractions. The influence

of reagent addition (type and amount) on recovery and grade of carbon in the high carbon and low carbon ash products was not significant. Variation in the results of these tests was within the experimental accuracy. The results of a single comparison test between biodiesel and Cytec S-8259 as a promoter indicated that biodiesel achieved lower carbon recovery (% yield) and correspondingly higher % carbon in the high carbon concentrate. Recovery of low carbon ash (% yield) was significantly higher with biodiesel, 66.5% for biodiesel versus 57.8% for Cytec S-8259 but % carbon in the fly ash was lower.

Continuous Pilot Plant Flotation Tests

The mass flow rates in lb/hr, percent yield, pulp density as % solids are tabulated in Table 3, Table 4, and Table 5 respectively. The throughput for the pilot plant ranged from 542.0 to 602.3 lb/hr with an average of 557.2 lb/hr. Average yields (% weight of feed) of low carbon fly ash, high carbon product, bottom ash and an intermediate carbon product were 73.4%, 4.7%, 7.8% and 10.1 % respectively. Percent carbon (measured as % LOI at MRL) was 0.66 to 7.63 % with an average of 2.52% for the low carbon fly ash, 67.2 to 75.3 % with an average of 71.95% for the high carbon product, and 12.67 to 61.56% with an average of 41.52% for the intermediate carbon product. Based on the correlation curves generated from the raw data, average % carbon in the fly ash, high carbon product and intermediate carbon product was 1.96, 69.0, and 39.5% respectively. Calorific value in Btu/lb of the high carbon product ranged from 9,337 to 10,379 with an average of 9,936 Btu/lb. Results of proximate analysis for some of the high carbon samples are listed in Table 12. The ash ranged from 22.10 to 31.09%, fixed carbon ranged from 65.04 to 73.08%, the calorific value ranged from 9,439 to 10,828 Btu/lb, sulfur was in the range of 0.34 to 0.44% and the volatile organic matter ranged from 0.0 to 2.62%. Only one sample of the coal ash feed to the pilot plant was submitted for proximate analysis. The results were as follows: ash 72.11%, fixed carbon, 26.53%; volatile matter 1.07%; calorific value, 1,195 Btu/lb and sulfur, 0.04%. The proximate values for the raw coal supplied to the power plant were 12,000 Btu/lb, 10.0 % ash, 32.0 % volatile matter and 0.98% sulfur. Sulfur and volatile organic matter were concentrated in the high carbon product with sulfur increasing from 0.04% in the feed to 0.44% in the carbon. The concentration of sulfur in the high carbon product may be

problematic. However, it is important to note that sulfuric acid was used as pH adjuster for flocculation of the carbon product prior to filtration. The contribution of sulfur from sulfuric acid is not known but sulfuric acid additions for pH adjustment were substantial. Washing of the carbon product after filtration with water might reduce the level of sulfur in the final carbon product.

The initial pilot plant runs used No. 2 fuel oil as the collector for the carbon at 1.96 lb/ton whereas the rest of the runs used Cytec S-8259 as the collector at 1.9 lb/ton. The influence of reagent addition on the grade and recovery of carbon was insignificant.

The results from the pilot plant runs confirmed the results of the bench-scale tests on a continuous basis. Figure 2 and Figure 3 show the graphical representation of the results of the bench-scale tests and the pilot plant tests respectively. These curves clearly show the similarity between the results of the bench-scale tests and the pilot plant test. The results from the continuous pilot plant tests were in agreement with the results of the bench-scale tests within experimental errors. The only exception was the yield of ash which showed significant difference between the bench-scale tests and the pilot plant tests. The % yield of ash from the pilot plant tests was 74% compared to the % yield of 55% from the bench-scale tests. This was expected because the separation size for fly ash was 60 mesh for the pilot plant tests and 100 mesh for the bench-scale tests.

Solid-liquid separation of the low carbon fly ash was achieved through settling, filtration and drying. Settling of the low carbon fly ash was very effective in a settling tank after the addition of sulfuric acid for pH adjustment and Superfloc A-100 (Cytec Industries, Stamford, CT) as a flocculating agent. The pulp density of the thickener underflow was about 55% solids. This underflow filtered well on a pan filter to produce a final filter cake with 25% moisture. The filter cake was dried in laboratory drying ovens to remove the remaining moisture.

The influence of recovery (% yield) on grade (% LOI) for the high carbon product is shown in Figures 5 and Figure 6 for the batch and pilot plant tests respectively. There was a good

correlation between the weight percent recovery and the grade (measured as % LOI) for the high carbon product. Percent carbon (as % LOI) increased with decreasing recovery (% yield). Also, this relationship was observed in the bench-scale tests as shown in the Figure 5a and Figure 5b, and is consistent with typical flotation processes where the grade of concentrate typically increases with decreasing recovery. Based on the test data, % carbon (% LOI) was correlated to recovery (% yield) by an inverse power relation. The empirical correlation equation is represented as:

$y = 80.75x^{-0.0878}$ for the bench-scale tests and $y = 77.73x^{-0.0608}$ for the pilot plant tests; where $y =$ % carbon (% LOI) and $x =$ recovery (% yield) of high carbon product. Therefore, the empirical maximum % carbon (% LOI) in the high carbon product was 80.75% and 77.73% for the bench-scale tests and pilot plant tests respectively.

Flotation recovery on the basis of two-product formula is represented as:

$$\text{Recovery, \% (R)} = \frac{c(f - t)}{f(c - t)} \times 100$$

where

| | | |
|-----------|---|--|
| C_{max} | = | maximum assay (%LOI) of carbon in concentrate (rougher conc) |
| c | = | assay (% LOI) of carbon in concentrate (rougher conc) |
| f | = | assay (% LOI) of carbon in feed |
| t | = | assay (% LOI) of carbon in tailing (rougher tails) |

The performance of the flotation process is evaluated by calculating the selectivity as flotation separation efficiency using the two-product equation by Joy and Watson as:

$$\text{Flotation Efficiency (FE)} = \frac{(c - f)}{(C_{max} - f)} \times \frac{c(f - t)}{f(c - t)}$$

Using the median values obtained from the pilot plant runs: $c = 55.2\%$; $f = 13.9\%$; $t = 3.48\%$; and $c_{\max} = 60.0\%$; the flotation recovery of carbon was found to be 80.0% and the flotation efficiency was 71.7%.

A sample of the low carbon fly ash was sent out to a certified commercial laboratory for Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for use in Concrete (ASTM C-618) tests and the results are listed in Table 14

The sample passed marginally all the specified tests except % fineness which was 47.6 % compared to the maximum limit of 34%. (Screen analysis of the same sample at MRL resulted in lower % fineness, % retained on 325-mesh, value of 42%). This was expected since the fly ash was separated at a sieve size of 60 mesh during the pilot plant runs. The designed flowsheet for the pilot plant had a separation size for fly ash at 100 mesh, but due to the high throughput for the operation of the pilot plant, we could not use a 100-mesh sieve without blinding the sieve and had to change to a 60-mesh screen.

It was first thought that if the fly ash had been separated at a finer cut size the % fineness would meet the maximum limit and potentially increase the reactivity index which was also at borderline. However, further investigation of the problem revealed that it was more complex than the size of separation of the fly ash. Particle size analyses of several grab samples of fly ash listed in Table 15 through Table 18 clearly showed that significant amount of material was retained on the 100 mesh by 325 mesh fractions. Moreover, the weight percent of fines passing 325-mesh (44 microns) were consistently less than 70% with an average of 57%. Obviously, the sample of coal ash used for the pilot plant was of coarser size distribution than expected. Therefore, using a sieve size of between 100-mesh and 325-mesh for separating the fly ash had only marginal effect on reducing the % fineness of the fly ash. It would appear that the fly ash from PEC Skyland plant would meet the ASTM C-618 specification of 34% maximum fineness consistently only if it was separated on a 200-mesh sieve. This would reduce the yield of low carbon fly ash from 73% (with 60-mesh cut) to less than 55% and require additional cost for the

more expensive fines screening circuits. In order to confirm this assumption, another sample of fly ash that was separated on a 200-mesh sieve was submitted for ASTM C-618 testing. The results of this second ASTM specification tests clearly showed that the fly ash met the specifications for % fineness. Percent fineness for the four samples ranged from 24.6 to 29.5 with an average value of 27.3%.

Meanwhile, additional tests were performed to evaluate other process options to produce fly ash that will consistently meet the ASTM C-618 specified fineness of 34% maximum. These included size separation of the bottom ash from the raw coal ash at a finer size (finer than 60 mesh) and grinding before flotation to separate the carbon. Alternatively, the raw coal ash after scalping on 6-mesh could be ground before separating the bottom ash and advancing the fly ash to the flotation process to remove unburned carbon.

The effect of grinding on the % fineness of the fly ash product was evaluated by these tests. They involved grinding a sample of the pilot plant scrubber discharge and fly ash in a laboratory ball mill and determining the % fineness of the fly ash as weight percent retained on a 325-mesh sieve. The results of these tests are listed in Table 20 through Table 22. It is important to note that the % fineness value obtained at MRL was 5% lower than the value obtained from the outside certified laboratory and the results should be interpreted accordingly. A sample of fly ash from the pilot plant runs on November 22, 2005 was ground in a ball mill for 10.0 minutes, the product was screened on a 200-mesh sieve and the oversize (+ 200 mesh) fraction was reground for 3.5 minutes. The % fineness from this test was reduced from 22.6 % to 17.7% after the grind. When the same sample was ground in the ball mill for 20.0 minutes, the % fineness went down to 10.2% when separating the ash on a 200-mesh sieve. A sample of the attrition scrub discharge from the pilot plant test of November 22, 2005 was also ground in a ball mill for 10.0 minutes, screened on a 200-mesh sieve and the oversize fraction was reground for additional 5.0 minutes. Finally a sample of the same scrubber discharge was screened at 70 mesh to remove the bottom ash and the undersize (-70 mesh) fraction was ground for 10.0 minutes in the ball mill. The % fineness at the separation size of 200 mesh was 17.9% and 19.9% for the 10.0 minute grind plus 5.0

minute regrind and the 10.0-minute grind respectively.

The results of the grindability tests on the scalped coal ash tabulated in Table 23 clearly showed that a 10-minute grind was sufficient to produce a fly ash that would meet the ASTM C-618 specification of 34% maximum fineness. However, this grind also resulted in converting the bottom ash into fine coal ash. Less than 1.0 % by weight of the raw feed remained as bottom ash (plus 100 mesh fraction) after the grind.

Another important feature of these tests, shown in Table 24, is the % carbon (LOI) distribution in the screen fractions of the separated fly ash. It is clearly shown that the coarser particles of the fly ash contained the higher carbon values. Screening out the plus 140-mesh fraction from the fly ash from pilot plant run of September 27, 2005 reduced the LOI from 4.6 to 2.79%. Similar screening of the fly ash from November 22, 2005-pilot plant run on a 100-mesh sieve reduced the LOI from 2.08% to 1.65%. Therefore, removing these coarser particles by screening would decrease significantly the % carbon in the fly ash. This finding is in agreement with our observation from previous tests that size separation of the flotation tailings, after removal of carbon, would reduce further the carbon content and produce consistently lower % LOI fly ash.

These tests have confirmed that moderate grinding of the coal ash material from PEC plant was essential to produce a fly ash product that will consistently meet the ASTM C-618 specifications for % fineness, and screening out of the coarser fraction would generate a fly ash product with consistent % carbon content below 2.0% LOI. However, the moderate grinding (less than 7.0 minutes rod mill grind) would reduce the bottom ash product from 16% to less than 2.0% by weight of the feed.

The conceptual flowsheet for the wet separation process would incorporate moderate grinding of scalped coal ash, attrition scrubbing, screening on a coarse sieve (50 mesh) to remove bottom ash, flotation to recover high carbon product, and screening of flotation tailings on a finer sieve (100 mesh) to produce fly ash with consistent low % LOI.

Production of Bulk Quantities of Green Pellets from Coal Ash and Sludge Mixture

Approximately 4,000 lb of pellets, were made from PEC ash and Ecusta sludge mixture. Four batches of pellets were produced at Carolina Pelleting and Extrusion and shipped to MRL facility in Asheville. The first batch of 5.0 mm diameter pellets with 19.6% moisture weighed 1,155 lb. The second batch also of 5.0 mm diameter pellets with 18.4% weighed 600 lb whereas the third batch of 12.0 mm diameter pellets with 18.4% moisture weighed 900 lb. The fourth batch of 12.0 mm diameter pellets with 17.7% moisture weighed 1,105 lb.

The pellets from all batches were well formed as they discharged from the pellet press but were not strong enough to withstand moderate mechanical handling during drying in the vibrating fluid bed dryer. Also the fall from the screening device to the product container was sufficient to break the pellets and some attrition was observed just from the screening operation itself. Decreasing moisture content of the mixture from 20% to 17.7% resulted in sturdier pellets. Pellets with 15% moisture were sufficiently hard to withstand moderate mechanical handling, but the die plugged at this moisture content. Mechanical handling during the shipment of the dry pellets from Carolina Pelleting facility in Newton to MRL facility in Asheville resulted in additional fines by attrition. Approximately 43% by weight of fines (minus 12-mesh, 0.0065 in, 1.65 mm) was separated from the 5.0 mm diameter pellets and 31.7 % by weight of fines (minus 6 mesh/0.131 in/3.33 mm) were separated from the 12.0 mm diameter pellets by screening. The actual pellet formation was quite easy and the green wet pellets looked acceptable as far as length and smoothness. A high pelleting rate per horsepower was achievable from this mixture. However, the pelleting rate would decrease as the moisture content decreases. The type of binder used might have contributed to the lower strength of the pellets. Aqualon 7LT, carboxymethylcellulose, is a low viscosity cellulose type. A higher viscosity type cellulose binder such as Aqualon 7H4 might be better and result in stronger pellets. In addition these types of organic binders are very expensive and might not be economical for the overall process. Other less expensive binders such as modified starches, liginosulfonate such as Norlig A could be used in larger quantities to improve the strength of the pellets. Evaluation of these other binders in future tests is recommended.

These pellets were test fired in the laboratory muffle furnace to produce lightweight aggregates. Batches of 100 - 120 gram pellets were fired in each batch. Firing temperature was set at nominal 2,200° F and the soaking (dwell) time at this firing temperature was 15 - 30 minutes.

Synthetic LWA of varying size and shapes, color, strength, loose density, specific gravity and gradation sizes were produced after firing in the laboratory muffle furnace. Several batches of dry pellets were fired to produce lightweight aggregates. These pellets were fired at a standard 2,200° F firing temperatures without varying the firing conditions. Photographs of some of the green pellets before firing and fired pellets produced during the batch firing campaign are included in the appendix. These lightweight aggregates had the following characteristics as shown in Table 27: loose density of 40.0 - 55.0 lb/cu ft, 0.9 - 1.80 specific gravity, 5.0 - 20.0 % water of adsorption depending on the size and shape of the pellets. ASTM C-330 Standard Specification for LWA for Structural Concrete was performed on the synthetic LWA from the batch-scale tests. This LWA was produced from pellets that were formed by tumbling in a laboratory rod mill and fired at 2200° F in a Swindel-Dressler periodic gas-fired furnace at the Brick Research Center, Clemson University, South Carolina. The procedure for firing large quantities of pellets (> 25.0 lb) in batches was not the most efficient and the strength of the fired pellets was less than desirable. Consequently, the measured 28-day compressive strength was 3,250 pounds per square inch, less than the expected strength of 5,000 pounds per square inch.

According to the recommendations of engineers from Carolina Stalite Company of Gold Hill, North Carolina, some of the batch-produced LWA was subjected to a quick and dirty soluble leaching tests to simulate the Toxicity Characteristic Leaching Procedure (TCLP). The purpose of these tests was to evaluate the potential for the stabilized fired pellets to release soluble contaminants to the environment. The procedure was to immerse about 100 grams of the fired aggregates in about 250 milliliters of deionized water in a beaker. Allow the aggregate to stay in the deionized water for 12 - 24 hours, decant the clear water from the aggregates and evaporate the water at 64° C to dryness. Observe the presence or absence of precipitate or crystal formation after evaporation. The presence of precipitate or crystal formation indicates leachable salts from

the aggregates. The results of these tests showed that aggregates made from PEC ash and Ecusta sludge exhibited a definite case of leachable salts whereas the results from aggregates made from PEC ash and Blue Ridge Paper Products' sludge were not definitive. It was established by the TCLP test results ((described below) that the LWA generated from a mixture of PEC fly ash and Ecusta landfill sludge would not release soluble contaminants to the environment.

Since, the quick and dirty soluble leachate test indicated that the LWA produced from PEC ash and Ecusta sludge could have potential leachable salt problems, Carolina Stalite recommended that we produce other bulk quantities of pellets using a mixture from PEC ash and Blue Ridge Paper Products' sludge. Larger pellets (briquettes) were recommended and made from the mixture. Pillow shaped briquettes (20 cubic centimeters in volume) were made with Koppert briquette machine at Midrex Technologies, Inc., Pineville, NC. It was recommended to make these larger size briquettes to be fired in the commercial kilns at Stalite. These fired briquettes would subsequently be crushed to produce the specific size gradation of LWA.

Bulk quantities of lightweight aggregates (LWA) were produced by firing dry pellets produced by Carolina Pelleting and Extrusion in the rotary kiln. These pellets were made from PEC ash and Ecusta landfill sludge. The kiln was run at a nominal temperature of 2200° F at a feed rate of 50 lb/hr resulting in the residence time of 20-25 minutes in the kiln. Representative samples of the LWA produced in the rotary kiln were submitted for the ASTM C-131-03 Standard Test for Resistance to Degradation (LA Abrasion), ASTM C-641-98 Standard Test for Staining Materials, and Toxicity Characteristic Leach Procedure (TCLP) test. The results, tabulated in Table 25, showed that the (ASTM C-131-03) LA Abrasion loss was 29% and there was no stain (ASTM C-641-98). The results of the TCLP test, tabulated in Table 26 showed that eight elements (As, Ba, Cd, Cr, Pb, Hg, Se, Ag) evaluated were all below the regulatory levels. Therefore, the LWA produced from a mixture of PEC ash and Ecusta landfill sludge did not have the potential of releasing contaminants into the environment.

A sample of the same bulk LWA produced during the pilot plant study (in the rotary kiln) will

be submitted for the Standard Specification for LWA for Structural Concrete (ASTM C-330) tests and the results will be reported in due course. It is anticipated that the measured 28-day compressive strength would be higher than the strength of 3,250 pounds per square inch that was obtained from the bench-scale studies.

Preliminary Marketing, Economic, and Business Feasibility Study

A detailed account of the economic, marketing and business initiatives for the process technology can be found in an accompanying report “Integrated Plant for processing High Carbon Coal Ash - Economic/Business Feasibility Study” by Ken Butcher.

Marketing

The four major end products from the integrated process technology were high carbon product, bottom ash, low carbon fly ash, and lightweight aggregates (LWA).

High Carbon Product

The high carbon product would contain 65-75% carbon with a heat content of 10,000 - 11,000 Btu/lb. One potential use for this carbon product would be as fuel to the power plants. As a fuel, the value could be estimated on the basis of the Btu content. Since the price of virgin coal averages about \$55.00 per ton at 12,000 Btu/lb, the value of the high carbon would be \$45 - 50 per ton. There may be obvious penalties for the high ash content and the fine particulate size of the carbon. Pelletization of the high carbon product may be necessary to satisfy the current size requirement for feeding conventional types of burners in the power plants. The demand for the high carbon product for this application will depend on the location and size of the power plants.

The other potential use for the carbon is for steel industry application as direct charge carbon to electric arc furnaces or direct iron production (Nucor Steel proprietary new iron reduction process). In the direct charge application, carbon is charged into an electric arc furnace along with oxygen to provide additional energy savings on electric power. For this application the carbon material should have low volatile fraction, low sulfur and low ash content. The ash is not

especially harmful in this application, it just brings no value. The high carbon product generated from this technology has low volatile matter, low sulfur, but high ash content. Therefore, there may be some penalty for the high ash content. Another issue with the high carbon product from this technology is the fine particulate size. The feeding system for the electric arc furnace needs particles of about 1/8 - 1/2 inch. Since the carbon for direct charge is high value at \$100 per ton, pelletization of the high carbon product to achieve the necessary particle size requirement may be economically feasible.

The final potential use of the high carbon product is the proprietary iron reduction process of Nucor Steel. For this application, the product could be used as-is, even without drying because the process incorporates a drying step for other materials. The value for this application would be lower around \$50-60 per ton, but the processing cost would be lower. The demand for the high carbon for the iron reduction application is high, 7,000 tons per year being a low end for Nucor's demand. The potential customer for the high carbon in our region, Nucor Steel Company, would require about two (2) tons of bulk product for commercial trials.

Low Carbon Class F Fly Ash

Local producers of cement blocks are reluctant to use low carbon Class F ash since concrete made with Class F fly ash takes longer to develop strength. Also, the large concrete block producers in our area are owned by Portland cement manufacturers, and replacement of cement, that sells for \$90 per ton (an outlet for their primary product) with fly ash, that sells for \$20 per ton, is not a favorable proposition. Therefore, in this local region, concrete block manufacturers were not considered as a potential market for the low carbon fly ash.

The other use of Class F ash is for ready mix concrete application. In western North Carolina, the two major concrete mix producers are Cemex (formerly Metromont) and Southern Concrete. These producers use large quantities of class F ash which they buy from Santee Cooper in South Carolina. Cemex has long-term contracts with Santee Cooper, they are happy with Santee Cooper and they are not interested in switching from them at this time. Southern Concrete was much

more receptive to the proposed project because they do not have a consistent supply of Class F ash in Asheville at this time. Their main concern was quality issues especially the carbon content of the ash. The lower the better. They were also concerned about variability of the fly ash. It is not enough for the ash to be within specification, it is also important that it be consistent. This consistency requirement suggests that the process technology should include extensive quality control of the products. Southern Concrete could use all the 40,000 tons of ash from the proposed commercial plant and would be glad to put that in a contract.

Bottom Ash

None of the concrete block manufacturers in western North Carolina currently uses bottom ash in their products. The production difficulties associated with the variability in the quality of bottom ash off sets the potential cost savings. General Shale, from nearby eastern Tennessee uses considerable amount of bottom ash in their plant near Johnson City, TN. The amount of 20,000 tons per year from the proposed commercial plant is small compared to their annual usage and they could adsorb the bottom ash from the plant. However, the cost of transportation would become a major issue and could make the economics unfavorable.

Lightweight Aggregate (LWA)

Lightweight aggregate (LWA) market in western North Carolina is served by Carolina Stalite that supplies the customers with expanded shale with loose bulk density of 54-56 lb/ft³ at a price of \$25 - 35 per ton depending on the density. Carolina Stalite has a very strong hold on the market with several long-term contracts with these concrete manufacturers. They are happy with the products as well as the service from Stalite, and they do not intend to switch. Quality and consistency of the product are also important issue with users of LWA. Carolina Stalite has invested considerable consumer engineering service to overcome these issues. The current demand for various types of lightweight aggregate in western North Carolina is between 70,000 and 100,000 tons per year.

Economics

The main focus for business development was a fully integrated commercial plant producing fly ash, bottom ash, high carbon and lightweight aggregate. This would potentially utilize all (100%) of the coal ash and eliminate land filling. A commercial plant based on the fully integrated concept and processing 100,000 tons of coal ash per year would require an estimated initial investment of \$10.0 million without land and utility costs. Three other different scenarios for commercial plant development were considered. They were: (a) production of only low carbon fly ash and high carbon product. Although this reduces the initial investment and operating costs, it does not eliminate land filling; (b) Using all (100%) the coal ash to produce lightweight aggregate. This would eliminate the separation of the other products and eliminate land filling. However, the process requires high initial investment and operating costs with an unpredictable market for the product. (c) Process to produce bottom ash, fly ash and high carbon, while combining the intermediate carbon product with bio-solids to produce green pellets. These pellets will then be sold to Carolina Stalite who will fire them to produce synthetic LWA that will be marketed as a niche product. This would reduce the initial investment and operating cost and more importantly use the expertise and marketing strength of Carolina Stalite.

The return on investment (ROI) as a ratio of yearly income to investment expressed as percent was calculated for the various business scenarios. The return on investment ranged from a low of 10% to a high of 16%. Although, these ratios are positive and encouraging, they fall short of making a compelling argument to invest in a commercial plant without other incentives. A detailed discussion of the estimated capital and operating costs of a commercial plant could be found in the accompanying report entitled “Integrated Plant for processing High Carbon Coal Ash - Economic/Business Feasibility Study”

CONCLUSION

Both bench-scale studies and continuous pilot plant operation have indicated that the integrated process could separate coal combustion ash into bottom ash, high carbon product and low carbon fly ash. In addition the intermediate carbon residue from the wet process was successfully combined with paper mill sludge to produce synthetic lightweight aggregates (LWA).

The yield (% weight of feed) of low carbon (% LOI) fly ash from the land-filled coal combustion ash (Progress Energy, Skyland Plant) was 45 -55%. Mild grinding of the coal ash was necessary to produce carbon fly ash with consistent fineness below 34% and a carbon grade of less than 2.0% LOI. Removal of coarser particles from the processed fly ash resulted in reduction of the % LOI of the final product. This low carbon fly ash met the Standard Specifications for Coal Fly Ash and Raw or Calcined Natural Pozzolan for use in Concrete (ASTM C-618).

The yield of high carbon product from the same ash was 2.5 - 7.5% and the corresponding grade was 67.0 - 75.5% carbon. Calorific value of the high carbon ranged from 9,337 to 10,379 Btu/lb.

A conceptual flowsheet for the wet separation process would incorporate moderate grinding of scalped coal ash, attrition scrubbing, screening on a coarse sieve (50 mesh) to remove bottom ash, flotation to recover the high carbon product, and screening of flotation tailings on a finer sieve (100 mesh) to produce fly ash with consistent low % LOI.

Synthetic lightweight aggregate (LWA) of varying size and shapes, color, strength, loose density, specific gravity and gradation sizes were produced by the pyro-process. These lightweight aggregates were successfully tested for their application for structural concrete via the relevant ASTM Testing Methods (ASTM C-131-03, ASTM C-330 and ASTM C-641) and TCLP.

The main focus for business development was a fully integrated commercial plant producing fly ash, bottom ash, high carbon and lightweight aggregate (LWA) The potential market for the

carbon product includes as fuel to the power plants, application as direct charge carbon to electric arc furnace and direct iron production for the steel industry. Low carbon fly ash (Class F) could be marketed as replacement for Portland cement in the concrete industry. The market for low carbon fly ash, bottom ash and lightweight aggregate is currently unpredictable in the local region.

A commercial plant based on the fully integrated concept and processing 100,000 tons of coal ash per year would require an estimated initial investment of \$10.0 million without land and utility costs. The return on investment (ROI) as a ratio of yearly income to investment expressed as percent would range from a low of 10% to a high of 16%.

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Table 2: Batch-scale Carbon Separation Tests- Summary of Results

| Test No. | Coal Ash Generator | Promoter | | Frother | | High Carbon | | High Carbon | | High Carbon | | Bottom Ash | | | Low LOI Ash | |
|----------|--------------------|-----------|--------|----------|--------|-----------------|-------|------------------|-------|-------------|-------|------------|---------|-------|-------------|------|
| | | Type | lb/ton | Type | lb/ton | +100 M Fraction | | - 100 M Fraction | | Total | | +30 M | + 100 M | Total | Yield | LOI |
| | | | | | | Yield | % LOI | Yield | % LOI | Yield | % LOI | | | | | |
| T-1 | PEC | Fuel Oil | 0.63 | Pine Oil | 0.72 | 2.3 | 77.96 | 6.1 | 63.26 | 8.4 | 67.30 | | | 29.1 | 51.0 | 4.01 |
| T-2 | PEC | Fuel Oil | 0.65 | Pine Oil | 0.73 | 2.7 | 68.23 | 6.4 | 58.47 | 9.1 | 61.40 | | | 28.0 | 44.9 | 1.63 |
| T-3 | PEC | S-8259 | 0.36 | F-533 | 0.92 | 2.1 | 76.30 | 7.1 | 65.66 | 9.2 | 68.10 | | | 39.9 | 51.1 | 0.10 |
| T-6 | PEC | S-8259 | 0.21 | F-533 | 0.59 | | | | | 3.1 | 72.79 | 6.9 | 22.7 | 29.6 | 51.8 | 1.41 |
| T-7 | PEC | S-8259 | 0.20 | F-533 | 0.71 | | | | | 7.0 | 69.24 | 10.2 | 18.0 | 28.2 | 53.6 | 2.34 |
| T-8 | PEC | S-8259 | 0.49 | F-533 | 1.41 | | | | | 11.7 | 65.00 | 10.1 | 9.7 | 19.8 | 57.4 | 0.11 |
| T-9 | PEC | Fuel Oil | 0.75 | F-589 | 1.19 | | | | | 6.8 | 68.24 | 9.7 | 22.3 | 32.0 | 46.6 | 1.39 |
| T-10 | PEC | S-8259 | 0.24 | F-533 | 0.76 | | | | | 7.7 | 66.46 | 8.8 | 9.0 | 17.8 | 57.2 | 0.52 |
| T-11 | PEC | S-8259 | 0.25 | F-533 | 0.83 | | | | | 10.2 | 64.25 | 0.0 | 17.3 | 17.3 | 58.4 | 0.25 |
| T-15 | PEC | S-4760 | 1.54 | F-533 | 1.81 | | | | | 7.9 | 66.41 | 9.7 | 12.0 | 21.7 | 58.1 | 0.12 |
| T-16 | PEC | S-8259 | 0.80 | F-533 | 2.34 | | | | | 12.5 | 61.47 | 7.8 | 12.1 | 19.9 | 56.2 | 0.05 |
| T-12 | DE/P | S-8259 | 0.63 | F-533 | 1.18 | | | | | 3.6 | 61.10 | 0.0 | 0.0 | 0.0 | 83.9 | 1.75 |
| T-13 | EBDC | S-8259 | 0.61 | F-533 | 1.46 | | | | | 9.8 | 60.98 | 0.0 | 6.1 | 6.1 | 69.6 | 7.03 |
| T-14 | EBDC | S-8259 | 0.82 | F-533 | 2.40 | | | | | 5.2 | 66.42 | 0.0 | 2.5 | 2.5 | 68.7 | 2.10 |
| T-15 | PEC | S-4760 | 1.54 | F-533 | 1.81 | | | | | 7.9 | 66.41 | 9.7 | 12.0 | 19.7 | 58.1 | 0.12 |
| T-16 | PEC | S-8259 | 0.80 | F-533 | 2.34 | | | | | 12.5 | 61.47 | 7.8 | 12.1 | 19.9 | 56.2 | 0.05 |
| T-17 | PEC | S-8259 | 0.59 | F-533 | 1.73 | | | | | 10.3 | 63.91 | 10.6 | 11.8 | 22.4 | 56.1 | 0.05 |
| T-18 | PEC | Biodiesel | 0.74 | F-533 | 2.17 | | | | | 7.5 | 66.47 | 4.9 | 9.2 | 14.1 | 65.8 | 1.38 |
| T-19 | PEC | S-8259 | 0.74 | F-533 | 2.16 | | | | | 12.9 | 57.79 | 4.2 | 6.6 | 10.8 | 59.9 | 0.70 |

PEC Progress Energy Carolinas, Inc.
 DE/P Duke Energy Corporation
 EBDC Ecusta Business Development Corporation

Table 3: Pilot Plant Operation Data: Mass Flowrates, lb/hour

| Product | Date of Operation | | | | | | | | Average | Median |
|----------------|-------------------|----------|----------|----------|---------|---------|---------|---------|---------|--------|
| | 11/22/05 | 11/18/05 | 11/16/05 | 11/14/05 | 9/26/05 | 9/23/05 | 9/21/05 | 9/19/05 | | |
| Feed | 550.5 | 543.4 | 525.5 | 559.0 | 602.3 | 550.9 | 542.4 | 583.7 | 557.2 | 550.7 |
| Scrubber Disch | 530.0 | 529.5 | 502.6 | 546.4 | 581.3 | 534.5 | 535.2 | 561.6 | 540.1 | 534.9 |
| Derrick O/S | 34.3 | 15.2 | 36.0 | 36.2 | 17.8 | 19.4 | 15.2 | 28.8 | 25.4 | 24.1 |
| Derrick U/S | 495.7 | 489.7 | 466.6 | 510.2 | 563.5 | 515.1 | 520.0 | 532.8 | 511.7 | 512.7 |
| Ro Conc | 92.4 | 61.3 | 61.4 | 85.7 | 107.2 | 62.6 | 147.5 | 56.6 | 84.3 | 74.2 |
| Ro Tails | 403.0 | 428.4 | 405.2 | 424.5 | 456.3 | 452.5 | 372.5 | 476.2 | 427.3 | 426.5 |
| Clnr Conc | 29.0 | 36.1 | 13.0 | 12.7 | 23.0 | 12.3 | 61.7 | 19.2 | 25.9 | 21.1 |
| Clnr Tails | 62.0 | 46.1 | 45.2 | 73.0 | 78.4 | 45.8 | 64.5 | 37.4 | 56.6 | 54.1 |
| Sweco O/S | 23.0 | 27.0 | 21.2 | 21.5 | 12.7 | 10.1 | 10.1 | 17.6 | 17.9 | 19.4 |
| Sweco U/S | 380.0 | 401.9 | 384.0 | 403.0 | 443.6 | 442.5 | 362.4 | 458.6 | 409.5 | 402.5 |

Derrick O/S Bottom Ash-1 (6 x 30 mesh Coarse Fraction)

Clnr Conc High Carbon Product

Clnr Tails Intermediate Carbon Product

Sweco O/S Bottom Ash-2 (30 x 60 mesh Fine Fraction)

Sweco U/S Low LOI Fly Ash

Table 4: Pilot Plant Operation Data: % Yield

| Product | Date of Operation | | | | | | | | Average | Median |
|----------------|-------------------|----------|----------|----------|---------|---------|---------|---------|---------|--------|
| | 11/22/05 | 11/18/05 | 11/16/05 | 11/14/05 | 9/26/05 | 9/23/05 | 9/21/05 | 9/19/05 | | |
| Scrubber Disch | 96.3 | 97.4 | 95.6 | 97.7 | 96.5 | 97.0 | 98.7 | 96.2 | 96.9 | 96.8 |
| Derrick O/S | 6.2 | 2.8 | 6.9 | 6.5 | 3.0 | 3.5 | 2.8 | 4.9 | 4.6 | 4.2 |
| Derrick U/S | 90.0 | 90.1 | 88.8 | 91.3 | 93.6 | 93.5 | 95.9 | 91.3 | 91.8 | 91.3 |
| Ro Conc | 16.8 | 11.3 | 11.7 | 15.3 | 17.8 | 11.4 | 27.2 | 9.7 | 15.1 | 13.5 |
| Ro Tails | 73.2 | 78.8 | 77.1 | 75.9 | 75.8 | 82.1 | 68.7 | 81.6 | 76.7 | 76.5 |
| Clnr Conc | 5.3 | 6.6 | 2.5 | 2.3 | 3.8 | 2.2 | 11.4 | 3.3 | 4.7 | 3.6 |
| Clnr Tails | 11.3 | 8.5 | 8.6 | 13.1 | 13.0 | 8.3 | 11.9 | 6.4 | 10.1 | 9.9 |
| Sweco O/S | 4.2 | 5.0 | 4.0 | 3.8 | 2.1 | 1.8 | 1.9 | 3.0 | 3.2 | 3.4 |
| Sweco U/S | 69.0 | 74.0 | 73.1 | 72.1 | 73.7 | 80.3 | 66.8 | 78.6 | 73.4 | 73.4 |

Table 5: Pilot Plant Operation Data: Pulp Density, % Solids

| Product | Date of Operation | | | | | | | | Average | Median |
|----------------|-------------------|----------|----------|----------|---------|---------|---------|---------|---------|--------|
| | 11/22/05 | 11/18/05 | 11/16/05 | 11/14/05 | 9/26/05 | 9/23/05 | 9/21/05 | 9/19/05 | | |
| Feed | 85.3 | 80.7 | 86.1 | 85.7 | 84.1 | 82.7 | 83.0 | 90.7 | 83.9 | 84.9 |
| Scrubber Disch | 49.4 | 51.8 | 53.7 | | 42.7 | 41.4 | 38.1 | 36.8 | 46.2 | 42.7 |
| Derrick O/S | | | | | 11.0 | 11.5 | 9.6 | 16.0 | 10.7 | 11.3 |
| Derrick U/S | 17.8 | 19.3 | 16.5 | 35.3 | 42.8 | 44.8 | 35.1 | 35.4 | 30.2 | 35.2 |
| Ro Conc | 8.6 | 8.8 | 9.6 | 5.2 | 11.3 | 5.3 | 6.9 | | 8.0 | 8.6 |
| Ro Tails | 6.0 | 10.3 | 9.1 | 26.9 | 10.2 | 11.2 | 7.9 | 16.9 | 11.7 | 10.8 |
| Clnr Conc | 5.6 | 5.8 | 2.4 | | 4.4 | 2.9 | 8.1 | 2.4 | 4.9 | 4.4 |
| Clnr Tails | 6.0 | 6.2 | 8.4 | 6.2 | 8.6 | 4.6 | 5.5 | 3.2 | 6.5 | 6.2 |
| Sweco O/S | 42.6 | | | | 38.9 | 39.8 | 38.7 | 37.9 | 40.0 | 38.9 |
| Sweco U/S | 7.9 | 7.5 | 7.4 | | 10.1 | 10.9 | 5.4 | 13.7 | 8.2 | 7.9 |

Table 6: Pilot Plant Operation Data: % Carbon as % LOI (MRL)

| Product | Date of Operation | | | | | | | | Average | Median |
|----------------|-------------------|----------|----------|----------|---------|---------|--------------------|---------|---------|--------|
| | 11/22/05 | 11/18/05 | 11/16/05 | 11/14/05 | 9/26/05 | 9/23/05 | 9/21/05 | 9/19/05 | | |
| Feed | 13.48 | | | | | 14.31 | | | 13.90 | 13.90 |
| Scrubber Disch | 10.19 | 10.74 | 10.17 | | 13.00 | 13.66 | 13.51 | 14.09 | 12.19 | 13.00 |
| Derrick O/S | 1.52 | | 1.64 | | 8.05 | 13.47 | 9.05 | 2.94 | 6.11 | 5.50 |
| Derrick U/S | 11.18 | 10.53 | 10.65 | | 14.58 | 13.85 | 13.86 | 13.30 | 12.56 | 13.30 |
| Ro Conc | 48.38 | 55.22 | 58.09 | | 58.06 | 65.05 | 39.53 | 30.08 | 50.63 | 55.22 |
| Ro Tails | 2.57 | 3.66 | 3.30 | 6.78 | 3.83 | 2.75 | 2.99 | 9.13 | 4.38 | 3.48 |
| Clnr Conc | 70.00 | 69.10 | 73.10 | 74.19 | 70.95 | 75.33 | 67.16 ¹ | 70.98 | 71.95 | 70.98 |
| Clnr Tails | 34.20 | 39.18 | 47.72 | 52.85 | 46.21 | 61.56 | 12.67 | 37.79 | 41.52 | 42.70 |
| Sweco O/S | 10.81 | | 19.97 | | 27.99 | | 24.92 | 30.47 | 22.83 | 24.92 |
| Sweco U/S | 0.66 | 1.10 | 2.12 | 3.91 | 2.46 | 1.21 | 1.03 | 7.63 | 2.52 | 1.67 |

Note 1: Average of four samples (62.3, 60.0, 74.7, 71.7)

Table 7: Pilot Plant Operation Data: Reagent Addition, lb/ton

| Reagent | Date of Operation | | | | | | | | Average | Median |
|--------------|-------------------|----------|----------|----------|---------|---------|---------|---------|---------|--------|
| | 11/22/05 | 11/18/05 | 11/16/05 | 11/14/05 | 9/26/05 | 9/23/05 | 9/21/05 | 9/19/05 | | |
| Fuel Oil | | | | | | | 1.96 | 1.96 | 2.0 | 2.0 |
| Cytec S-8259 | 2.16 | 1.79 | 1.62 | 1.85 | 1.97 | 1.97 | | | 1.9 | 1.9 |
| Cytec F-533 | 1.15 | 1.23 | 1.20 | 1.58 | 1.54 | 1.54 | 1.47 | 1.47 | 1.4 | 1.5 |

Table 8: Correlation Data Points for the Integrated Pilot Plant Process for CCB's

| Low LOI Fly Ash | | High Carbon | | Processed Ash Products | |
|-----------------|------------------|-------------|------------------|------------------------|----------------|
| % LOI (MRL) | % Carbon (Lab 1) | % LOI (MRL) | % Carbon (Lab 2) | % LOI (MRL) | Btu/lb (Lab 2) |
| 0.70 | 0.64 | 47.30 | 43.97 | 2.94 | 984 |
| 2.40 | 1.13 | 68.70 | 65.04 | 47.30 | 6,390 |
| 2.70 | 2.17 | 70.90 | 69.30 | 68.70 | 9,439 |
| 4.80 | 4.51 | 73.10 | 69.96 | 70.90 | 9,814 |
| 11.80 | 9.66 | 75.30 | 73.88 | 73.10 | 10,306 |
| 14.30 | 12.10 | | | 75.30 | 10,828 |

Lab 1 = ALS Chemex, Sparks NV

Lab 2 = SGS, Norfolk, VA

Table 9: Calculated Data Based on the Correlation Curves

| Date | Low LOI Fly Ash | | High Unburned Carbon | | |
|----------|-----------------|-----------------------|----------------------|-----------------------|---------------------|
| | % LOI (MRL) | % Carbon (Calculated) | % LOI (MRL) | % Carbon (Calculated) | Btu/lb (Calculated) |
| 9/19/05 | 7.60 | 6.30 | 71.0 | 68.5 | 9,337 |
| 9/21/05 | 1.00 | 0.67 | 67.2 | 64.6 | 9,390 |
| 9/23/05 | 1.20 | 0.84 | 75.3 | 73.0 | 10,556 |
| 9/26/05 | 2.50 | 1.95 | 70.9 | 68.4 | 9,923 |
| 11/14/05 | 3.90 | 3.14 | 74.2 | 71.9 | 10,397 |
| 11/16/05 | 2.10 | 1.61 | 73.1 | 70.7 | 10,239 |
| 11/18/05 | 1.10 | 0.76 | 69.1 | 66.6 | 9,664 |
| 11/22/05 | 0.70 | 0.42 | 71.3 | 68.9 | 9,980 |
| Average | 2.51 | 1.96 | 71.4 | 69.0 | 9,936 |
| Std. Dev | 2.31 | 1.97 | 2.7 | 2.7 | 451 |
| Median | 1.65 | 1.23 | 71.2 | 68.7 | 9,952 |

Table 10: Recovery (% Yield) and % Carbon for Fly Ash and High Carbon Products from Pilot Plant Tests

| Date | Low LOI Fly Ash | | | High Carbon | | | |
|----------|-----------------|-------------|-----------------------|-------------|-------------|-----------------------|---------------------|
| | % Yield | % LOI (MRL) | % Carbon ¹ | % Yield | % LOI (MRL) | % Carbon ¹ | Btu/lb ¹ |
| 9/19/05 | 78.60 | 7.60 | 6.30 | 3.50 | 71.0 | 68.5 | 9,337 |
| 9/21/05 | 66.80 | 1.00 | 0.67 | 11.38 | 67.2 | 64.6 | 9,390 |
| 9/23/05 | 80.30 | 1.20 | 0.84 | 2.40 | 75.3 | 73.0 | 10,556 |
| 9/26/05 | 73.70 | 2.50 | 1.95 | 4.38 | 70.0 | 68.4 | 9,923 |
| 11/14/05 | 72.10 | 3.90 | 3.14 | 2.27 | 74.2 | 71.9 | 10,397 |
| 11/16/05 | 73.10 | 2.10 | 1.61 | 2.33 | 73.1 | 70.0 | 10,239 |
| 11/18/05 | 74.00 | 1.10 | 0.76 | 6.64 | 69.1 | 66.6 | 9,664 |
| 11/22/05 | 69.00 | 0.70 | 0.42 | 4.77 | 71.3 | 68.9 | 9,980 |
| Average | 73.45 | 2.51 | 1.96 | 4.71 | 71.4 | 69.0 | 9,936 |
| Std. Dev | 4.47 | 2.31 | 1.97 | 3.09 | 2.7 | 2.7 | 451 |
| Median | 73.40 | 1.65 | 1.23 | 3.94 | 71.2 | 68.7 | 9,952 |

Note 1: calculated values from correlation curves

Table 11: Recovery (% Yield) and Carbon Grade (as % LOI) in High Carbon Product for Batch and Pilot Plant Tests

| Batch Tests | | Pilot Plant Tests | |
|-------------|---------------------|-------------------|---------------------|
| % Yield | % Carbon (as % LOI) | % Yield | % Carbon (as % LOI) |
| 9.20 | 68.10 | 5.30 | 71.30 |
| 3.10 | 72.80 | 6.60 | 69.10 |
| 7.00 | 69.20 | 2.50 | 73.10 |
| 11.70 | 65.00 | 2.30 | 74.20 |
| 6.80 | 68.20 | 3.80 | 70.90 |
| 7.70 | 66.50 | 2.20 | 75.30 |
| 10.20 | 64.30 | 11.40 | 67.20 |
| | | 3.30 | 71.00 |

Table 12: Results of Proximate Analysis

| Sample ID | Description | % Moisture | % Ash | % Volatile | % Fixed Carbon | Btu/lb | % Sulfur |
|-------------------|-------------|------------|-------|------------|----------------|--------|----------|
| 11/22-1 | Head Feed | 0.29 | 72.11 | 1.07 | 26.53 | 1,195 | 0.04 |
| 11-22-5 | Ro Conc | 1.24 | 53.12 | 1.67 | 43.97 | 6,390 | 0.30 |
| 11-22-7 | Clnr Conc | 1.58 | 31.09 | 2.29 | 65.04 | 9,439 | 0.44 |
| 11-16-7 | Clnr Conc | 0.85 | 26.69 | 2.50 | 69.69 | 10,306 | 0.39 |
| 09-26-7 | Clnr Conc | 1.10 | 29.59 | 0.00 | 69.31 | 9,814 | 0.34 |
| 09-23-7 | Clnr Conc | 1.40 | 22.10 | 2.62 | 73.88 | 10,828 | 0.40 |
| Average Clnr Conc | | 1.23 | 27.37 | 1.85 | 69.48 | 10,097 | 0.39 |
| STDEV Clnr Conc | | 0.32 | 3.96 | 1.24 | 3.61 | 603 | 0.04 |
| Median Clnr Conc | | 1.25 | 28.14 | 2.40 | 69.50 | 10,060 | 0.40 |

Table 13: Chemistry (Mineral Analysis)

| Date | Low LOI Fly Ash | | | | Raw Ash |
|--|-----------------|----------|----------|----------|----------|
| | 09-23-05 | 09-26-05 | 11-16-05 | 11-22-05 | 09-23-05 |
| Silica (SiO ₂) | 54.77 | 56.25 | 57.58 | 58.23 | 48.17 |
| Alumina (Al ₂ O ₃) | 24.85 | 25.73 | 24.17 | 24.66 | 25.65 |
| Iron (Fe ₂ O ₃) | 7.79 | 7.98 | 9.25 | 9.07 | 6.39 |
| Combined (SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃) | 87.41 | 89.96 | 91.00 | 91.96 | 80.21 |
| Lime (CaO) | 0.78 | 0.77 | 0.66 | 0.64 | 0.73 |
| Magnesium (MgO) | 1.23 | 1.26 | 1.19 | 1.15 | 1.13 |
| Loss-on-ignition (LOI) | 5.28 | 2.38 | 1.97 | 0.95 | 13.05 |

Table 14: Results of ASTM C-618 Specification Testing on Fly Ash from Pilot Plant

| Test | Results | | ASTM C 618 Specifications (Class F) |
|--|---------|------------------------------|-------------------------------------|
| | 70 mesh | 200 mesh | |
| Density, Variation from Average, % | 0 | 0 | 5 % max. |
| Fineness, % | 47.6 | 29.7 28.3 24.8 26.7 | 34 % max. |
| Strength Activity Index (% of Control @ 28 days) | 76.8 | 77.0 | 75 % min. |
| Water requirement, % of Control | 103 | 102 | 105 % max. |
| Soundness, % Contraction | -0.004 | -0.002 | 0.8 % max. |

Table 15: Particle Size Analysis of Fly Ash from Bench-scale Tests

100-Mesh Fly Ash Separation

| Particle size | | Test T-12 (Duke Energy) | | | | Test T-14 (Ecusta) | | | | Test T-7 (PEC) | | | |
|----------------------------|---------|-------------------------|-------|--------------|---------|--------------------|-------|--------------|---------|----------------|-------|--------------|---------|
| | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | Weight | | Cum Weight % | |
| US Mesh | Microns | g | % | Retained | Passing | g | % | Retained | Passing | g | % | Retained | Passing |
| 30 | 600 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 |
| 50 | 300 | 0.9 | 0.8 | 0.8 | 99.2 | 0.0 | 0.0 | 0.0 | 100.0 | 0.1 | 0.1 | 0.1 | 99.9 |
| 70 | 212 | 1.8 | 1.6 | 2.4 | 97.6 | 1.3 | 1.2 | 1.2 | 98.8 | 0.1 | 0.1 | 0.2 | 99.8 |
| 100 | 149 | 2.6 | 2.3 | 4.6 | 95.4 | 6.0 | 5.5 | 6.7 | 93.3 | 0.5 | 0.5 | 0.6 | 99.4 |
| 140 | 102 | 3.5 | 3.1 | 7.7 | 92.3 | 4.4 | 4.0 | 10.7 | 89.3 | 3.9 | 3.5 | 4.2 | 95.8 |
| 325 | 44 | 20.2 | 17.6 | 25.3 | 74.7 | 25.2 | 23.0 | 33.7 | 66.3 | 33.7 | 30.6 | 34.8 | 65.2 |
| Pan | -44 | 85.6 | 74.7 | 100.0 | | 72.7 | 66.3 | 100.0 | | 71.8 | 65.2 | 100.0 | |
| Total | | 114.6 | 100.0 | | | 109.6 | 100.0 | | | 110.1 | 100.0 | | |
| % Fineness at 70 mesh Cut | | 23.5 | | | | 32.9 | | | | 34.7 | | | |
| % Fineness at 100 mesh Cut | | 21.7 | | | | 28.9 | | | | 34.4 | | | |
| % Fineness at 140 mesh Cut | | 19.1 | | | | 25.7 | | | | 31.9 | | | |

Table 16: Particle Size Analysis of Grab Samples of Fly Ash from Pilot Plant - Nov 22, 2005
60-Mesh Fly Ash Separation

| Particle size | | Ash with Medium Carbon | | | | Ash with Light Carbon -I | | | | Ash with Light Carbon -II | | | | Ash with Light Carbon -III | | | | | |
|----------------------------|---------|------------------------|-------|--------------|---------|--------------------------|-------|--------------|---------|---------------------------|-------|--------------|---------|----------------------------|-------|--------------|---------|--|--|
| | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | | |
| US Mesh | Microns | g | % | Retained | Passing | g | % | Retained | Passing | g | % | Retained | Passing | g | % | Retained | Passing | | |
| 50 | 300 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 | | |
| 70 | 212 | 0.6 | 0.7 | 0.7 | 99.3 | 0.9 | 0.7 | 0.7 | 99.3 | 0.8 | 0.9 | 0.9 | 99.1 | 2.8 | 2.2 | 2.2 | 97.8 | | |
| 100 | 149 | 2.3 | 2.6 | 3.3 | 96.7 | 3.2 | 2.6 | 3.3 | 96.7 | 3.0 | 3.2 | 4.1 | 95.9 | 9.3 | 7.3 | 9.5 | 90.5 | | |
| 140 | 102 | 4.4 | 5.0 | 8.4 | 91.6 | 5.4 | 4.4 | 7.7 | 92.3 | 5.7 | 6.1 | 10.2 | 89.8 | 13.4 | 10.5 | 19.9 | 80.1 | | |
| 200 | 75 | 10.5 | 12.0 | 20.4 | 79.6 | 12.6 | 10.2 | 18.0 | 82.0 | 12.5 | 13.4 | 23.6 | 76.4 | 18.4 | 14.4 | 34.3 | 65.7 | | |
| 325 | 44 | 17.4 | 20.0 | 40.4 | 59.6 | 21.1 | 17.2 | 35.1 | 64.9 | 18.5 | 19.8 | 43.5 | 56.5 | 22.5 | 17.6 | 51.9 | 48.1 | | |
| Pan | -44 | 52.0 | 59.6 | 100.0 | | 79.8 | 64.9 | 100.0 | | 52.7 | 56.5 | 100.0 | | 61.6 | 48.1 | 100.0 | | | |
| Total | | 87.2 | 100.0 | | | 123.0 | 100.0 | | | 93.2 | 100.0 | | | 128.0 | 100.0 | | | | |
| % Fineness at 70 mesh Cut | | | | 40.0 | | | | 34.6 | | | | 43.0 | | | | 50.8 | | | |
| % Fineness at 100 mesh Cut | | | | 38.3 | | | | 32.9 | | | | 41.1 | | | | 46.9 | | | |
| % Fineness at 140 mesh Cut | | | | 34.9 | | | | 29.7 | | | | 37.0 | | | | 39.9 | | | |
| % Fineness at 200 mesh Cut | | | | 25.1 | | | | 20.9 | | | | 26.0 | | | | 26.8 | | | |

| Composite Fly Ash | | | | | | Ash with Medium Carbon, Scrubbed | | | | | | Raw PEC Ash | | | | | | | | | | | |
|----------------------------|---------|--------|------|--------------|---------|----------------------------------|---------|----------------------------|------|--------------|---------|---------------|---------|--------|------|----------------------------|---------|--|--|------|--|--|--|
| Particle size | | Weight | | Cum Weight % | | Particle size | | Weight | | Cum Weight % | | Particle size | | Weight | | Cum Weight % | | | | | | | |
| US Mesh | Microns | g | % | Retained | Passing | US Mesh | Microns | g | % | Retained | Passing | US Mesh | Microns | g | % | Retained | Passing | | | | | | |
| 50 | 300 | 0.0 | 0.0 | 0.0 | 100.0 | 50 | 300 | 0.0 | 0.0 | 0.0 | 100.0 | 50 | 300 | 15.6 | 7.9 | 7.9 | 92.1 | | | | | | |
| 70 | 212 | 5.1 | 1.2 | 1.2 | 98.8 | 70 | 212 | 1.0 | 0.6 | 0.6 | 99.4 | 70 | 212 | 6.5 | 3.3 | 11.2 | 88.8 | | | | | | |
| 100 | 149 | 17.8 | 4.1 | 5.3 | 94.7 | 100 | 149 | 4.2 | 2.5 | 3.1 | 96.9 | 100 | 149 | 9.7 | 4.9 | 16.1 | 83.9 | | | | | | |
| 140 | 102 | 28.9 | 6.7 | 12.0 | 88.0 | 140 | 102 | 9.4 | 5.6 | 8.7 | 91.3 | 140 | 102 | 14.5 | 7.3 | 23.4 | 76.6 | | | | | | |
| 200 | 75 | 54.0 | 12.5 | 24.5 | 75.5 | 200 | 75 | 21.0 | 12.5 | 21.2 | 78.8 | 200 | 75 | 24.6 | 12.4 | 35.8 | 64.2 | | | | | | |
| 325 | 44 | 79.5 | 18.4 | 43.0 | 57.0 | 325 | 44 | 35.4 | 21.1 | 42.3 | 57.7 | 325 | 44 | 34.4 | 17.4 | 53.2 | 46.8 | | | | | | |
| Pan | -44 | 246.1 | 57.0 | 100.0 | | Pan | -44 | 97.0 | 57.7 | 100.0 | | Pan | -44 | 92.8 | 46.8 | 100.0 | | | | | | | |
| Total | | 431.4 | | | | Total | | 168.0 | | | | Total | | 198.1 | | | | | | | | | |
| % Fineness at 70 mesh Cut | | | | 42.3 | | | | % Fineness at 70 mesh Cut | | | | 41.9 | | | | % Fineness at 70 mesh Cut | | | | 47.3 | | | |
| % Fineness at 100 mesh Cut | | | | 39.8 | | | | % Fineness at 100 mesh Cut | | | | 40.4 | | | | % Fineness at 100 mesh Cut | | | | 44.2 | | | |
| % Fineness at 140 mesh Cut | | | | 35.2 | | | | % Fineness at 140 mesh Cut | | | | 36.8 | | | | % Fineness at 140 mesh Cut | | | | 38.9 | | | |
| % Fineness at 200 mesh Cut | | | | 24.4 | | | | % Fineness at 200 mesh Cut | | | | 26.7 | | | | % Fineness at 200 mesh Cut | | | | 27.0 | | | |

Table 17: Particle Size Analysis of Grab Samples of Fly Ash from Pilot Plant - Sep 27, 2005
60-Mesh Fly Ash Separation

| | | Sample 1 | | | | Sample 2 | | | | Sample 3 | | | | Composite | | | |
|----------------------------|---------|----------|-------|--------------|---------|----------|-------|--------------|---------|----------|-------|--------------|---------|-----------|-------|--------------|---------|
| Particle size | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | Weight | | Cum Weight % | |
| US Mesh | Microns | g | % | Retained | Passing | g | % | Retained | Passing | g | % | Retained | Passing | g | % | Retained | Passing |
| 30 | 600 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 |
| 50 | 300 | 0.3 | 0.2 | 0.2 | 99.8 | 0.2 | 0.1 | 0.1 | 99.9 | 0.2 | 0.2 | 0.2 | 99.8 | 0.7 | 0.2 | 0.2 | 99.8 |
| 70 | 212 | 2.2 | 1.1 | 1.3 | 98.7 | 1.7 | 1.1 | 1.3 | 98.7 | 1.2 | 1.2 | 1.4 | 98.6 | 5.1 | 1.1 | 1.3 | 98.7 |
| 100 | 149 | 8.0 | 4.0 | 5.3 | 94.7 | 6.1 | 4.1 | 5.4 | 94.6 | 4.2 | 4.2 | 5.6 | 94.4 | 18.3 | 4.1 | 5.4 | 94.6 |
| 140 | 102 | 13.7 | 6.9 | 12.1 | 87.9 | 9.8 | 6.6 | 11.9 | 88.1 | 6.3 | 6.3 | 11.9 | 88.1 | 29.8 | 6.6 | 12.0 | 88.0 |
| 325 | 44 | 61.6 | 30.9 | 43.1 | 56.9 | 46.2 | 30.9 | 42.8 | 57.2 | 29.9 | 29.9 | 41.8 | 58.2 | 137.7 | 30.7 | 42.7 | 57.3 |
| Pan | -44 | 113.5 | 56.9 | 100.0 | | 85.5 | 57.2 | 100.0 | | 58.1 | 58.2 | 100.0 | | 257.1 | 57.3 | 100.0 | |
| Total | | 199.3 | 100.0 | | | 149.5 | 100.0 | | | 99.9 | 100.0 | | | 448.7 | 100.0 | | |
| % Fineness at 70 mesh Cut | | 42.3 | | | | 42.1 | | | | 41.0 | | | | 42.0 | | | |
| % Fineness at 100 mesh Cut | | 39.9 | | | | 39.6 | | | | 38.4 | | | | 39.4 | | | |
| % Fineness at 140 mesh Cut | | 35.2 | | | | 35.1 | | | | 34.0 | | | | 34.9 | | | |

**Table 18: Particle Size Analysis of Fly Ash from Pilot Plant - Multiple Grab Samples from Storage Steel Drums
140-Mesh and 200-Mesh Fly Ash Separation**

| Particle size | | Fly Ash #4 200 Mesh Separation | | | | Fly Ash # 3 200 Mesh Separation | | | | Fly Ash #4 140 Mesh Separation | | | | Fly Ash #3 140 Mesh Separation | | | |
|----------------------------|---------|--------------------------------|-------|--------------|---------|---------------------------------|-------|--------------|---------|--------------------------------|-------|--------------|---------|--------------------------------|-------|--------------|---------|
| | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | Weight | | Cum Weight % | |
| US Mesh | Microns | g | % | Retained | Passing | g | % | Retained | Passing | g | % | Retained | Passing | g | % | Retained | Passing |
| 50 | 300 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 |
| 70 | 212 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 | 0.1 | 0.1 | 0.1 | 99.9 | 0.2 | 0.2 | 0.2 | 99.8 |
| 100 | 149 | 0.4 | 0.4 | 0.4 | 99.6 | 0.8 | 0.7 | 0.7 | 99.3 | 0.4 | 0.4 | 0.5 | 99.5 | 0.7 | 0.7 | 0.8 | 99.2 |
| 140 | 102 | 0.1 | 0.1 | 0.5 | 99.5 | 0.3 | 0.3 | 1.0 | 99.0 | 0.3 | 0.3 | 0.8 | 99.2 | 0.8 | 0.8 | 1.6 | 98.4 |
| 200 | 75 | 1.1 | 1.0 | 1.5 | 98.5 | 1.8 | 1.7 | 2.7 | 97.3 | 12.6 | 12.0 | 12.7 | 87.3 | 10.3 | 9.7 | 11.3 | 88.7 |
| 325 | 44 | 26.2 | 24.5 | 26.0 | 74.0 | 21.4 | 19.8 | 22.5 | 77.5 | 23.1 | 22.0 | 34.7 | 65.3 | 19.3 | 18.1 | 29.4 | 70.6 |
| Pan | -44 | 79.2 | 74.0 | 100.0 | | 83.6 | 77.5 | 100.0 | | 68.6 | 65.3 | 100.0 | | 75.1 | 70.6 | 100.0 | |
| Total | | 107.0 | 100.0 | | | 107.9 | 100.0 | | | 105.1 | 100.0 | | | 106.4 | 100.0 | | |
| % Fineness at 70 mesh Cut | | 26.0 | | | | 22.5 | | | | 34.7 | | | | 29.3 | | | |
| % Fineness at 100 mesh Cut | | 25.7 | | | | 21.9 | | | | 34.4 | | | | 28.8 | | | |
| % Fineness at 140 mesh Cut | | 25.6 | | | | 21.7 | | | | 34.2 | | | | 28.3 | | | |
| % Fineness at 200 mesh Cut | | 24.9 | | | | 20.4 | | | | 25.2 | | | | 20.4 | | | |

| Particle size | | Fly Ash Comp. 200 Mesh Separation | | | |
|----------------------------|---------|-----------------------------------|------|--------------|---------|
| | | Weight | | Cum Weight % | |
| US Mesh | Microns | g | % | Retained | Passing |
| 50 | 300 | 0.0 | 0.0 | 0.0 | 100.0 |
| 70 | 212 | 0.0 | 0.0 | 0.0 | 100.0 |
| 100 | 149 | 1.2 | 0.6 | 0.6 | 99.4 |
| 140 | 102 | 0.4 | 0.2 | 0.7 | 99.3 |
| 200 | 75 | 2.9 | 1.3 | 2.1 | 97.9 |
| 325 | 44 | 47.6 | 22.1 | 24.2 | 75.8 |
| Pan | -44 | 162.8 | 75.8 | 100.0 | |
| Total | | 214.9 | | | |
| % Fineness at 70 mesh Cut | | 24.2 | | | |
| % Fineness at 100 mesh Cut | | 23.8 | | | |
| % Fineness at 140 mesh Cut | | 23.7 | | | |
| % Fineness at 200 mesh Cut | | 22.6 | | | |

| Particle size | | Fly Ash Comp. 140 Mesh Separation | | | |
|--------------------------|---------|-----------------------------------|------|--------------|---------|
| | | Weight | | Cum Weight % | |
| US Mesh | Microns | g | % | Retained | Passing |
| 50 | 300 | 0.0 | 0.0 | 0.0 | 100.0 |
| 70 | 212 | 0.3 | 0.1 | 0.1 | 99.9 |
| 100 | 149 | 1.1 | 0.5 | 0.7 | 99.3 |
| 140 | 102 | 1.1 | 0.5 | 1.2 | 98.8 |
| 200 | 75 | 22.9 | 10.8 | 12.0 | 88.0 |
| 325 | 44 | 42.4 | 20.0 | 32.1 | 67.9 |
| Pan | -44 | 143.7 | 67.9 | 100.0 | |
| Total | | 211.5 | | | |
| Fineness at 70 mesh Cut | | 32.0 | | | |
| Fineness at 100 mesh Cut | | 31.6 | | | |
| Fineness at 140 mesh Cut | | 31.2 | | | |
| Fineness at 200 mesh Cut | | 22.8 | | | |

**Table 19: Particle Size Analysis of Raw Ash from Pilot Plant after Grinding
Scrubber Discharge from 11/18/05 and 11/22/05**

| | | 10-min Grind: -200 mesh Fraction | | | | 5.0-min Re grind of + 200 mesh Oversize | | | | Grind and Re grind Combined Product | | | |
|----------------------------|---------|----------------------------------|-------|--------------|---------|---|-------|--------------|---------|-------------------------------------|-------|--------------|---------|
| Particle size | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | Weight | | Cum Weight % | |
| US Mesh | Microns | g | % | Retained | Passing | g | % | Retained | Passing | g | % | Retained | Passing |
| 50 | 300 | 0.0 | 0.0 | 0.0 | 100.0 | 0.1 | 0.1 | 0.1 | 99.9 | 0.0 | 0.0 | 0.0 | 100.0 |
| 70 | 212 | 0.0 | 0.0 | 0.0 | 100.0 | 0.1 | 0.1 | 0.2 | 99.8 | 0.0 | 0.0 | 0.1 | 99.9 |
| 100 | 149 | 0.1 | 0.1 | 0.1 | 99.9 | 0.2 | 0.2 | 0.4 | 99.6 | 0.2 | 0.2 | 0.3 | 99.7 |
| 140 | 102 | 0.1 | 0.1 | 0.3 | 99.7 | 1.4 | 1.4 | 1.8 | 98.2 | 0.7 | 0.8 | 1.1 | 98.9 |
| 200 | 75 | 0.0 | 0.0 | 0.3 | 99.7 | 7.4 | 7.2 | 9.0 | 91.0 | 3.0 | 3.5 | 4.6 | 95.4 |
| 325 | 44 | 4.0 | 5.5 | 5.7 | 94.3 | 30.2 | 29.5 | 38.5 | 61.5 | 14.5 | 17.1 | 21.6 | 78.4 |
| Pan | -44 | 69.0 | 94.3 | 100.0 | | 62.9 | 61.5 | 100.0 | | 66.6 | 78.4 | 100.0 | |
| Total | | 73.2 | 100.0 | | | 102.3 | 100.0 | | | 84.9 | 100.0 | | |
| % Fineness at 70 mesh Cut | | 5.7 | | | | 38.4 | | | | 21.5 | | | |
| % Fineness at 100 mesh Cut | | 5.6 | | | | 38.3 | | | | 21.4 | | | |
| % Fineness at 140 mesh Cut | | 5.5 | | | | 37.4 | | | | 20.8 | | | |
| % Fineness at 200 mesh Cut | | 5.5 | | | | 32.4 | | | | 17.9 | | | |

40.0% Weight Retained on 200 Mesh after 10.0 minutes Grind

**Table 20: Particle Size Analysis of Fly Ash from Pilot Plant after Grinding
Fly Ash Sample #4 from 11/22/05**

| | | 10-min Grind: - 200 mesh Fraction | | | | 3.5-min Re grind of + 200 mesh Oversize | | | | Grind and Re grind Combined Product | | | |
|----------------------------|---------|-----------------------------------|-------|--------------|---------|---|-------|--------------|---------|-------------------------------------|-------|--------------|---------|
| Particle size | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | Weight | | Cum Weight % | |
| US Mesh | Microns | g | % | Retained | Passing | g | % | Retained | Passing | g | % | Retained | Passing |
| 50 | 300 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 |
| 70 | 212 | 0.1 | 0.1 | 0.1 | 99.9 | 0.1 | 0.1 | 0.1 | 99.9 | 0.1 | 0.1 | 0.1 | 99.9 |
| 100 | 149 | 0.1 | 0.1 | 0.2 | 99.8 | 0.3 | 0.2 | 0.3 | 99.7 | 0.2 | 0.2 | 0.3 | 99.7 |
| 140 | 102 | 0.1 | 0.1 | 0.4 | 99.6 | 1.5 | 1.0 | 1.3 | 98.7 | 0.5 | 0.5 | 0.8 | 99.2 |
| 200 | 75 | 0.1 | 0.1 | 0.5 | 99.5 | 11.7 | 8.0 | 9.3 | 90.7 | 3.5 | 3.5 | 4.3 | 95.7 |
| 325 | 44 | 7.1 | 8.6 | 9.1 | 90.9 | 41.0 | 28.1 | 37.4 | 62.6 | 17.1 | 16.9 | 21.2 | 78.8 |
| Pan | -44 | 74.8 | 90.9 | 100.0 | | 91.2 | 62.6 | 100.0 | | 79.6 | 78.8 | 100.0 | |
| Total | | 82.3 | 100.0 | | | 145.8 | 100.0 | | | 101.1 | 100.0 | | |
| % Fineness at 70 mesh Cut | | 9.0 | | | | 37.4 | | | | 21.1 | | | |
| % Fineness at 100 mesh Cut | | 8.9 | | | | 37.3 | | | | 21.0 | | | |
| % Fineness at 140 mesh Cut | | 8.8 | | | | 36.6 | | | | 20.6 | | | |
| % Fineness at 200 mesh Cut | | 8.7 | | | | 31.0 | | | | 17.7 | | | |

29.5 % Weight retained on 200 mesh after 10.0 minutes grind

**Table 21: Particle Size Analysis of Products from Pilot Plant after Grinding
Scrubber Discharge and Fly Ash Samples from 11/22/05**

| Particle size | | Scrubber Discharge | | | | Fly Ash | | | |
|----------------------------|---------|------------------------------------|-------|--------------|---------|------------------|-------|--------------|---------|
| | | 10-min. grind of -70 mesh Fraction | | | | 20-minutes Grind | | | |
| | | Weight | | Cum Weight % | | Weight | | Cum Weight % | |
| US Mesh | Microns | g | %, | Retained | Passing | g | %, | Retained | Passing |
| 50 | 300 | 0.0 | 0.0 | 0.0 | 100.0 | 0.0 | 0.0 | 0.0 | 100.0 |
| 70 | 212 | 0.1 | 0.1 | 0.1 | 99.9 | 0.1 | 0.1 | 0.1 | 99.9 |
| 100 | 149 | 0.4 | 0.4 | 0.4 | 99.6 | 0.1 | 0.1 | 0.2 | 99.8 |
| 140 | 102 | 1.9 | 1.7 | 2.1 | 97.9 | 0.2 | 0.2 | 0.3 | 99.7 |
| 200 | 75 | 7.3 | 6.4 | 8.6 | 91.4 | 0.4 | 0.3 | 0.7 | 99.3 |
| 325 | 44 | 20.6 | 18.2 | 26.7 | 73.3 | 12.2 | 10.1 | 10.8 | 89.2 |
| Pan | -44 | 83.0 | 73.3 | 100.0 | | 107.8 | 89.2 | 100.0 | |
| Total | | 113.3 | 100.0 | | | 120.8 | 100.0 | | |
| % Fineness at 70 mesh Cut | | 26.7 | | | | 10.7 | | | |
| % Fineness at 100 mesh Cut | | 26.4 | | | | 10.6 | | | |
| % Fineness at 140 mesh Cut | | 25.2 | | | | 10.5 | | | |
| % Fineness at 200 mesh Cut | | 19.9 | | | | 10.2 | | | |

87.1 % by weight of scrubber discharge reported in -70 mesh fraction

Table 22: Summary of Results of % Fineness for Fly Ash for Varying Separation Sizes

| Ash Source | Sample Description | Separation Size | Time, min. | | % Fineness |
|------------|----------------------------------|-----------------|------------|---------|------------|
| | | Mesh | Grind | Regrind | |
| PEC | Bench Tests Fly Ash | 100 | 0.0 | 0.0 | 34.4 |
| Duke | Bench Tests Fly Ash | 100 | 0.0 | 0.0 | 21.7 |
| Ecusta | Bench tests Fly Ash | 100 | 0.0 | 0.0 | 28.9 |
| PEC | Pilot Tests Fly Ash 9/27/05 | 70 | 0.0 | 0.0 | 42.0 |
| PEC | Pilot Tests Fly Ash 11/22/05 | 70 | 0.0 | 0.0 | 42.3 |
| PEC | Pilot Test Scrubbed Ash 11/22/05 | 70 | 0.0 | 0.0 | 41.9 |
| PEC | Pilot Test Scrubbed Ash 11/22/05 | 140 | 0.0 | 0.0 | 31.2 |
| PEC | Pilot Test Fly Ash 11/22/05 | 200 | 0.0 | 0.0 | 22.6 |
| PEC | Pilot Test Scrub Discharge | 200 | 10.0 | 5.0 | 17.9 |
| PEC | Pilot Test Fly Ash 11/22/05 | 200 | 10.0 | 3.5 | 17.7 |
| PEC | Pilot Test Scrubber Discharge | 70 | 10.0 | 0.0 | 26.7 |
| PEC | Pilot Tests Fly Ash 11/22/05 | 200 | 20.0 | 0.0 | 10.2 |

PEC Progress Energy Carolinas

Duke Duke Energy Corporation

Ecusta Ecusta Business Development Corporation

Table 23: Particle Size Analysis of Coal Ash from Pilot Plant after Grinding

Head Feed from the Pilot Plant

| | | Test # 147 T-0 (No grind) | | | | Test # 147 T-1 (10.0 min grind) | | | | Test # 147 T-2 (15.0 min grind) | | | | Test # 147 T-3 (5.0 min grind) | | | |
|----------------------------|---------|---------------------------|----------|--------------|---------|---------------------------------|-----------|--------------|---------|---------------------------------|-----------|--------------|---------|---------------------------------|-----------|--------------|---------|
| Particle size | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | Weight | | Cum Weight % | | Weight | | Cum Weight % | |
| US Mesh | Microns | g | No grind | No grind | Passing | g | min grind | min grind | Passing | g | min grind | min grind | Passing | g | min grind | min grind | Passing |
| 50 | 300 | 15.6 | 7.9 | 7.9 | 92.1 | 0.1 | 0.1 | 0.1 | 99.9 | 0.1 | 0.1 | 0.1 | 99.9 | 0.3 | 0.3 | 0.3 | 99.7 |
| 70 | 212 | 6.5 | 3.3 | 11.2 | 88.8 | 0.2 | 0.2 | 0.3 | 99.7 | 0.2 | 0.1 | 0.2 | 99.8 | 0.7 | 0.6 | 0.8 | 99.2 |
| 100 | 149 | 9.7 | 4.9 | 16.1 | 83.9 | 0.9 | 0.8 | 1.0 | 99.0 | 0.4 | 0.2 | 0.4 | 99.6 | 2.1 | 1.8 | 2.6 | 97.4 |
| 140 | 102 | 14.5 | 7.3 | 23.4 | 76.6 | 2.6 | 2.2 | 3.3 | 96.7 | 1.7 | 0.9 | 1.3 | 98.7 | 5.1 | 4.3 | 6.9 | 93.1 |
| 200 | 75 | 24.6 | 12.4 | 35.8 | 64.2 | 8.2 | 7.1 | 10.4 | 89.6 | 7.3 | 4.0 | 5.4 | 94.6 | 11.2 | 9.4 | 16.2 | 83.8 |
| 325 | 44 | 34.4 | 17.4 | 53.2 | 46.8 | 21.6 | 18.7 | 29.0 | 71.0 | 30.7 | 17.0 | 22.4 | 77.6 | 23.2 | 19.4 | 35.7 | 64.3 |
| Pan | -44 | 92.8 | 46.8 | 100.0 | | 82.2 | 71.0 | 100.0 | | 139.9 | 77.6 | 100.0 | | 76.8 | 64.3 | 100.0 | |
| Total | | 198.1 | 100.0 | | | 115.8 | 100.0 | | | 180.3 | 100.0 | | | 119.4 | 100.0 | | |
| % Fineness at 70 mesh Cut | | 47.3 | | | | 28.8 | | | | 22.3 | | | | 35.1 | | | |
| % Fineness at 100 mesh Cut | | 44.2 | | | | 28.3 | | | | 22.1 | | | | 34.0 | | | |
| % Fineness at 140 mesh Cut | | 38.9 | | | | 26.6 | | | | 21.4 | | | | 30.9 | | | |
| % Fineness at 200 mesh Cut | | 27.0 | | | | 20.8 | | | | 18.0 | | | | 23.2 | | | |

Table 24: % LOI Distribution in Screen Fractions of Fly Ash

Pilot Plant run 09/27/05

| Sample | Weight | | Cum Weight % | | LOI | Cum % LOI | |
|-----------|--------|-------|--------------|---------|-------|-----------|---------|
| | g | % | Retained | Passing | % | Retained | Passing |
| Feed | | | | | 4.64 | | |
| 70 Mesh | 5.8 | 1.3 | 1.3 | 98.7 | 18.46 | 18.46 | 4.06 |
| 140 Mesh | 48.1 | 10.7 | 12.0 | 88.0 | 14.48 | 14.91 | 2.79 |
| 325 Mesh | 137.7 | 30.7 | 42.7 | 57.3 | 4.63 | 7.52 | 1.81 |
| Pan | 257.1 | 57.3 | 100.0 | | 1.81 | 4.25 | |
| Calc Head | 448.7 | 100.0 | | | 4.25 | | |

Pilot Plant run 11/22/05

| Sample | Weight | | Cum Weight % | | LOI | Cum % LOI | |
|-----------|--------|-------|--------------|---------|-------|-----------|---------|
| | g | % | Retained | Passing | % | Retained | Passing |
| Feed | | | | | | | |
| 70 Mesh | 5.1 | 1.2 | 1.2 | 98.8 | 15.21 | 15.21 | 1.92 |
| 100 Mesh | 17.8 | 4.1 | 5.3 | 94.7 | 8.04 | 9.64 | 1.65 |
| 140 Mesh | 28.9 | 6.7 | 12.0 | 88.0 | 4.98 | 7.04 | 1.40 |
| 200 Mesh | 54.0 | 12.5 | 24.5 | 75.5 | 2.62 | 4.78 | 1.19 |
| 325 Mesh | 79.5 | 18.4 | 43.0 | 57.0 | 1.52 | 3.38 | 1.09 |
| Pan | 246.1 | 57.0 | 100.0 | | 1.09 | 2.08 | |
| Calc Head | 431.4 | 100.0 | | | 2.08 | | |

Table 25: Results of ASTM C131-03 (LA abrasion) and ASTM C641-98 (Staining) Specification Testing on LWA

| Test | Specification | Results |
|---------------------|---------------|----------|
| LA Abrasion, % Loss | ASTM C131-03 | 29 |
| Staining | ASTM C641-98 | No Stain |

Table 26: Toxicity Characteristic Leaching Procedure (TCLP) Extraction for LWA

| TCLP Metals | Extracted Results mg/L (ppm) | Quantitation Limit mg/L (ppm) | Regulatory Level mg/L (ppm) |
|---------------|---------------------------------|----------------------------------|--------------------------------|
| Arsenic (As) | 0.02 | 0.01 | 5.0 |
| Barium (Ba) | 0.33 | 0.01 | 100.0 |
| Cadmium (Cd) | BQL | 0.01 | 1.0 |
| Chromium (Cr) | BQL | 0.01 | 5.0 |
| Lead (Pb) | 0.27 | 0.01 | 5.0 |
| Selenium (Se) | BQL | 0.01 | 1.0 |
| Silver (Ag) | BQL | 0.02 | 5.0 |
| Mercury (Hg) | BQL | 0.001 | 0.2 |

Notes:

BQL = Below the Quantitation Limit

TCLP Method Used = SW846/6010 for all elements except Hg that used SW846/7470 A

Table 27: Physical Properties of Lightweight Aggregate (LWA)

| Sample (LWA) Description | | | | Loose Density lb/cu ft | Specific Gravity | Water Absorption % |
|--------------------------|------------|-----------------|---------------|---------------------------|------------------|--------------------|
| Ash | Sludge | Pelletizer Type | Particle Size | | | |
| PEC | Ecusta | Rotating Drum | + 4 mesh | 46.6 | 1.432 | 11.45 |
| PEC | Ecusta | Rotating Drum | 4 x 6 mesh | 40.4 | 1.205 | 11.64 |
| PEC | Ecusta | Rotating Drum | ½ x 3/8 | 47.7 | 1.337 | 9.55 |
| PEC | Ecusta | Rotating Drum | + ½ | 48.1 | 1.363 | 7.82 |
| PEC | BRPP | Lab Die Press | + ¼ | 37.8 | 1.432 | 20.70 |
| PEC | Glatfelter | Lab Die press | + ¼ | 34.0 | 1.337 | 18.60 |
| PEC | BRPP | Briquette | 5/16 x 8 mesh | 51.7 | 1.641 | 12.74 |
| PEC | BRPP | Briquette | 5/16 x 8 mesh | 50.0 | 1.534 | 6.46 |
| PEC | BRPP | Briquette | 5/16 x 8 mesh | 57.1 | 1.800 | 11.28 |
| PEC | BRPP | Briquette | 5/16 x 8 mesh | 52.1 | 1.560 | 6.58 |
| PEC | Ecusta | Com. Die Press | + 8 mesh | 49.1 | 1.567 | NA |
| PEC | Ecusta | Com. Die Press | 8 x 12 mesh | 43.6 | 1.314 | NA |

Lab Die Press = LCI/Khal laboratory pellet press
 Com. Die Press = Commercial flat die pelleting press
 NA = Not Available

Figure 1: FLOWSHEET FOR A PILOT PLANT TO RECOVER UNBURNED CARBON, BOTTOM ASH AND LOW LOI ASH

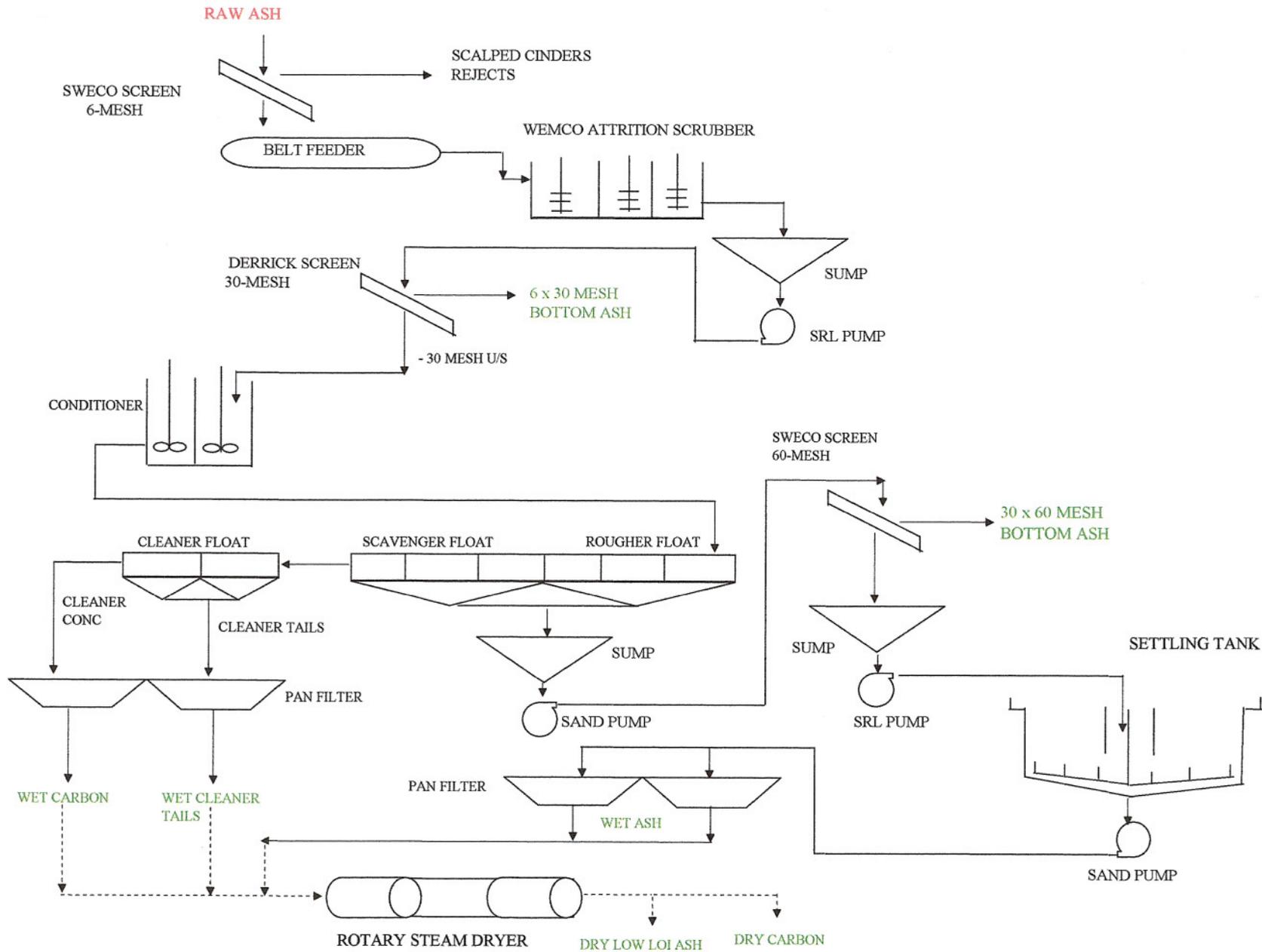
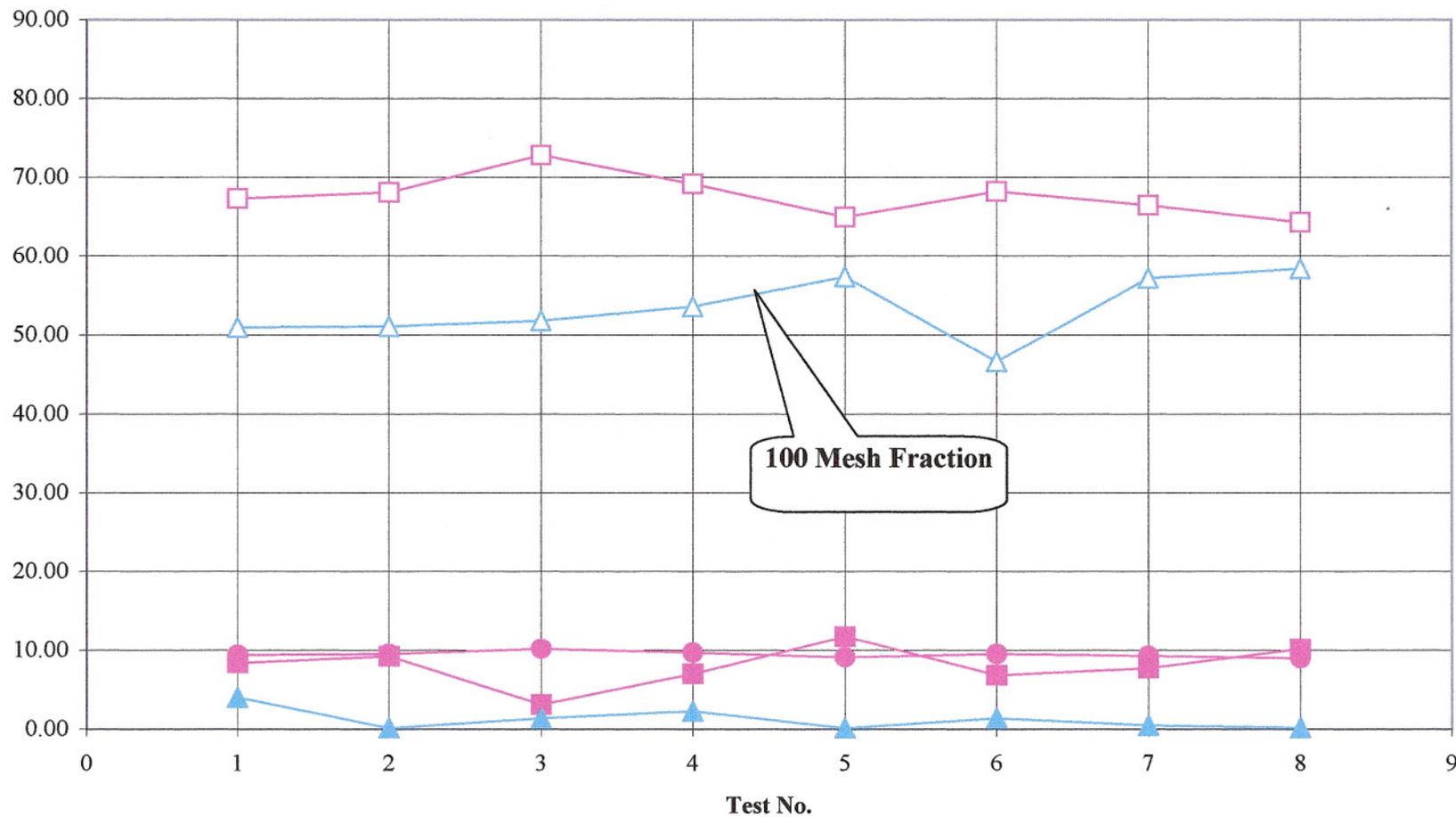
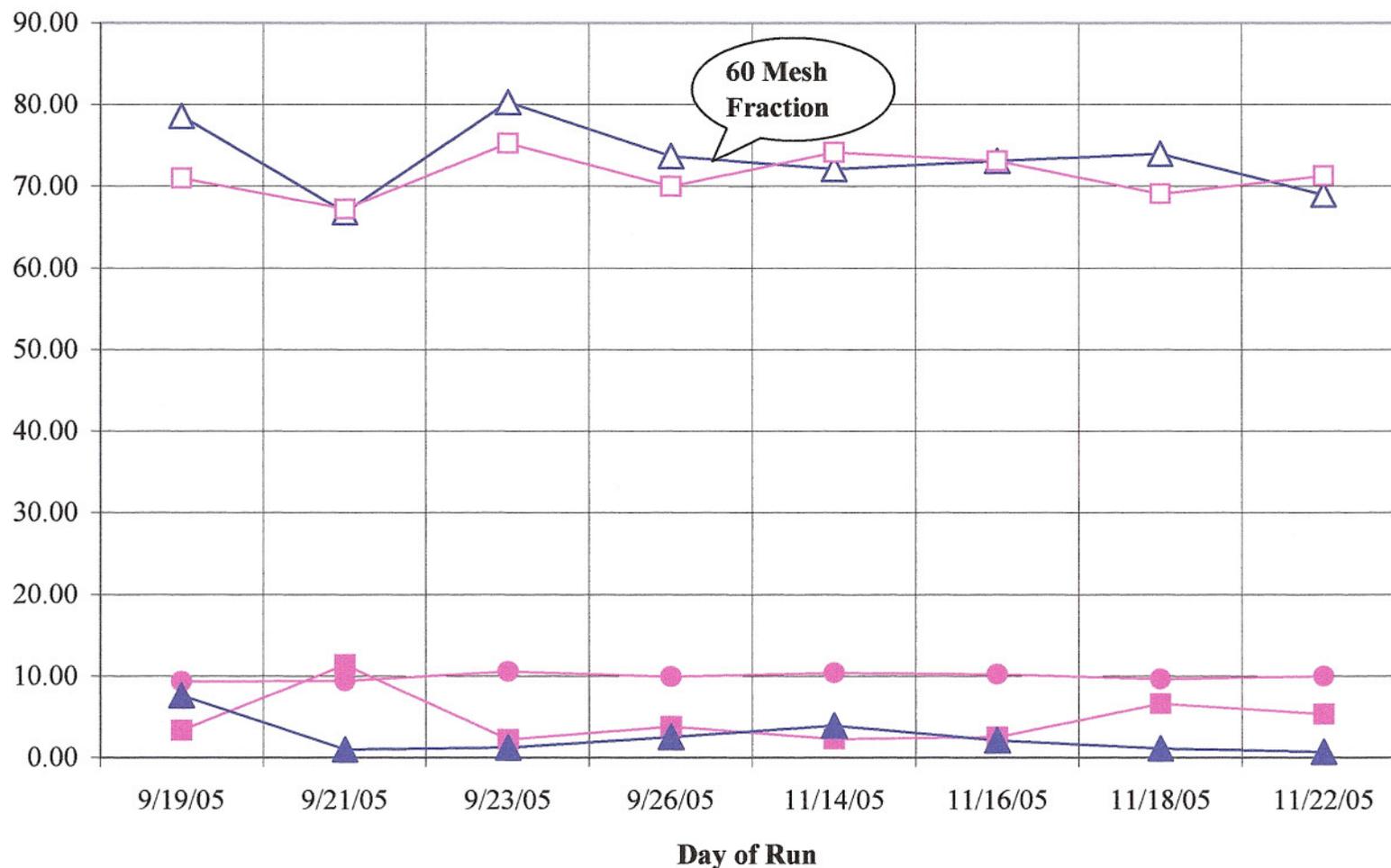


Figure 2: Bench-scale Wet Processing of Coal Combustion Ash: Carbon Separation Tests Data



■ % Yield, Carbon
 □ % LOI(MRL), Carbon
 ● 1000 Btu/lb, Carbon
 △ % Yield, Ash
 ▲ % LOI (MRL), Ash

Figure 3: High Carbon and Low LOI Fly Ash Pilot Plant Separation Tests Data



—△ % Ash Yield
 —■ % Carbon Yield
 —● 1000 Btu/lb, Carbon
 —▲ % LOI (MRL), Ash
 —□ % LOI (MRL), Carbon

Figure 4a: Correlation between % LOI (MRL) and % Carbon in Fly Ash

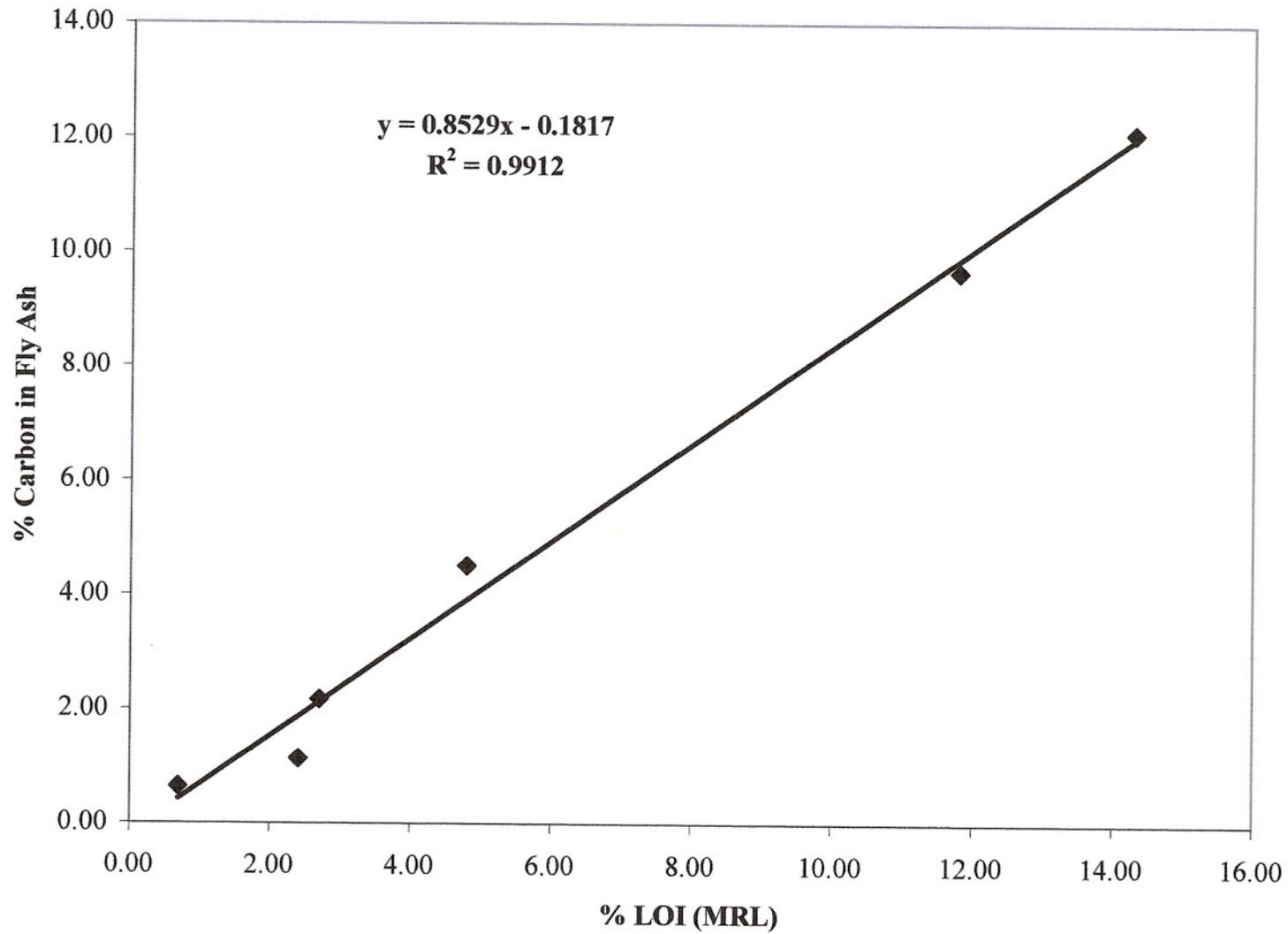


Figure 4b: Correlation between %LOI (MRL) and % Carbon for High Carbon Product

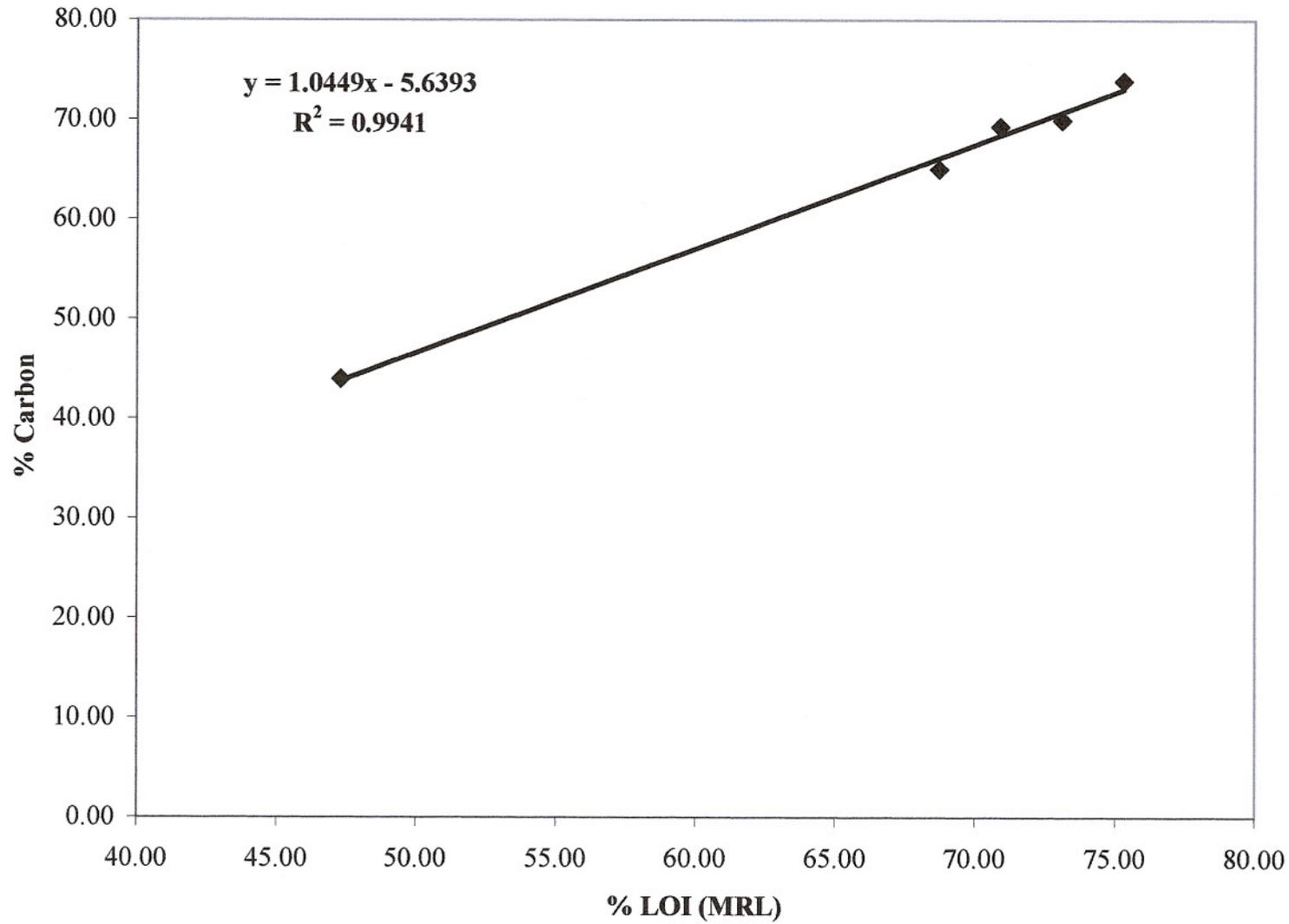
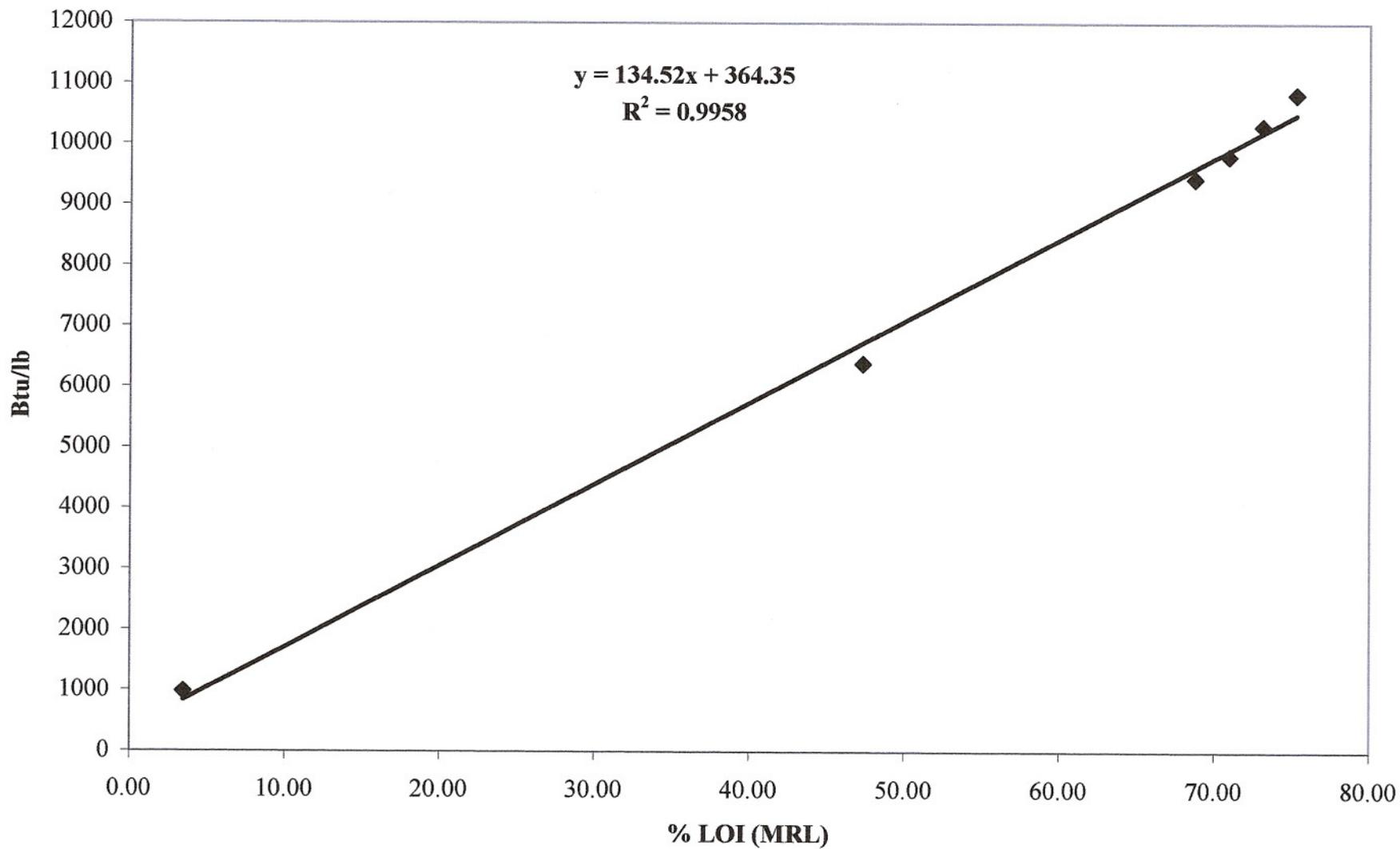
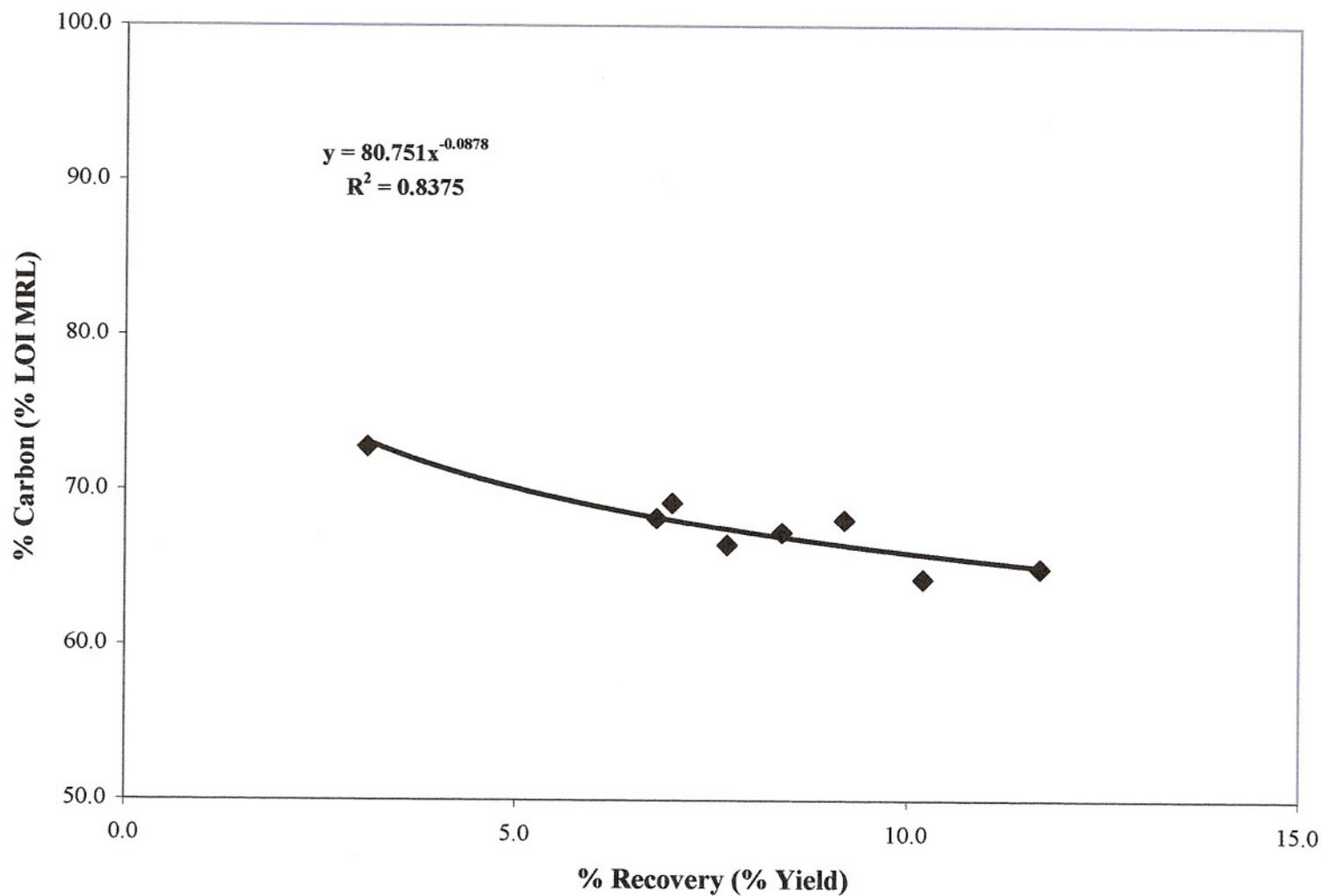


Figure 4c: Correlation Between % LOI (MRL) and Btu/lb for Coal Combustion Products



**Figure 5a: Recovery (% Yield) versus % Carbon (% LOI MRL) for High Carbon Product
Bench-scale Tests: All reagents**



**Figure 5b: Recovery (% Yield) versus % Carbon (% LOI MRL) for High Carbon Product
Bench-scale Tests with Cytec Reagents**

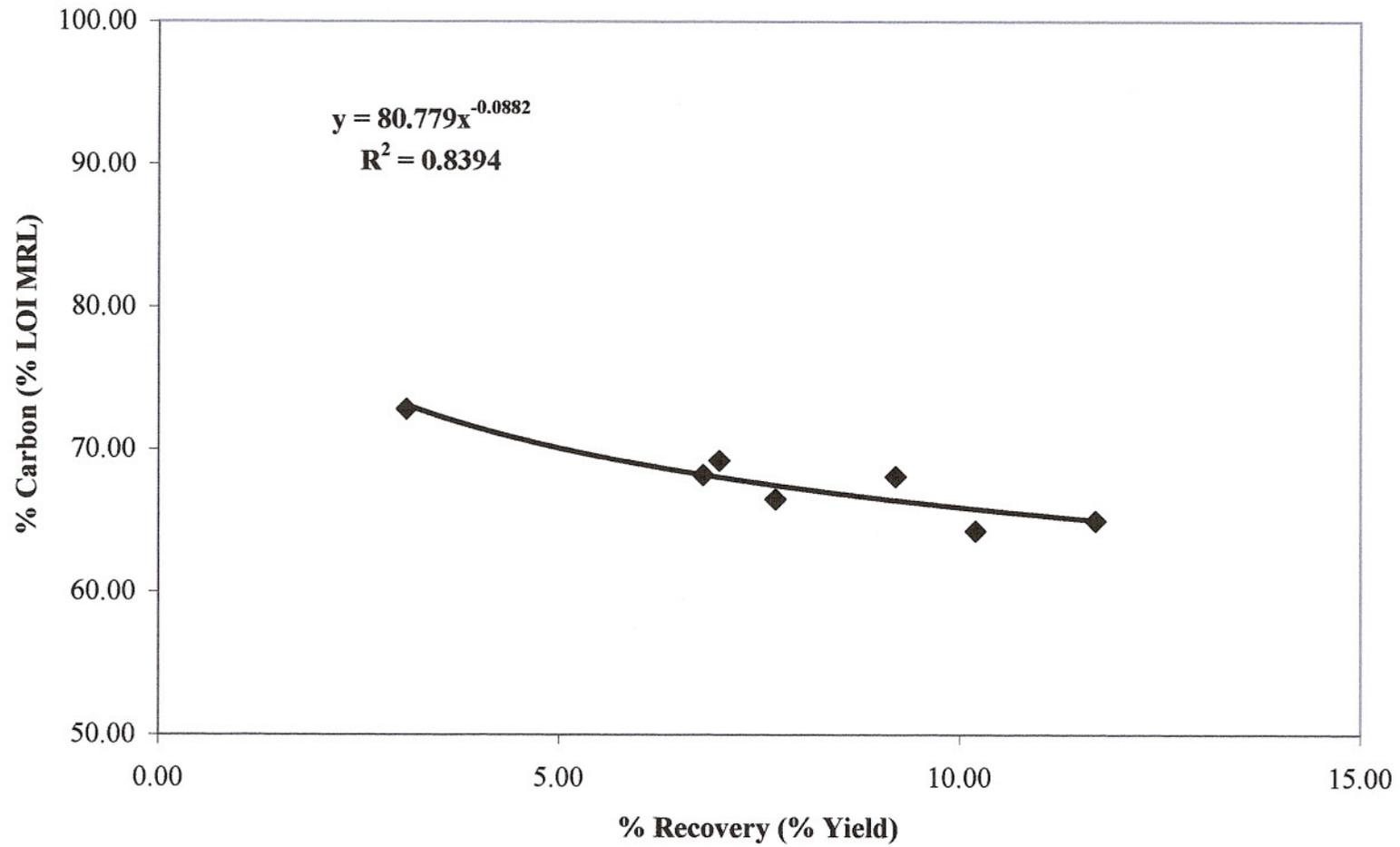
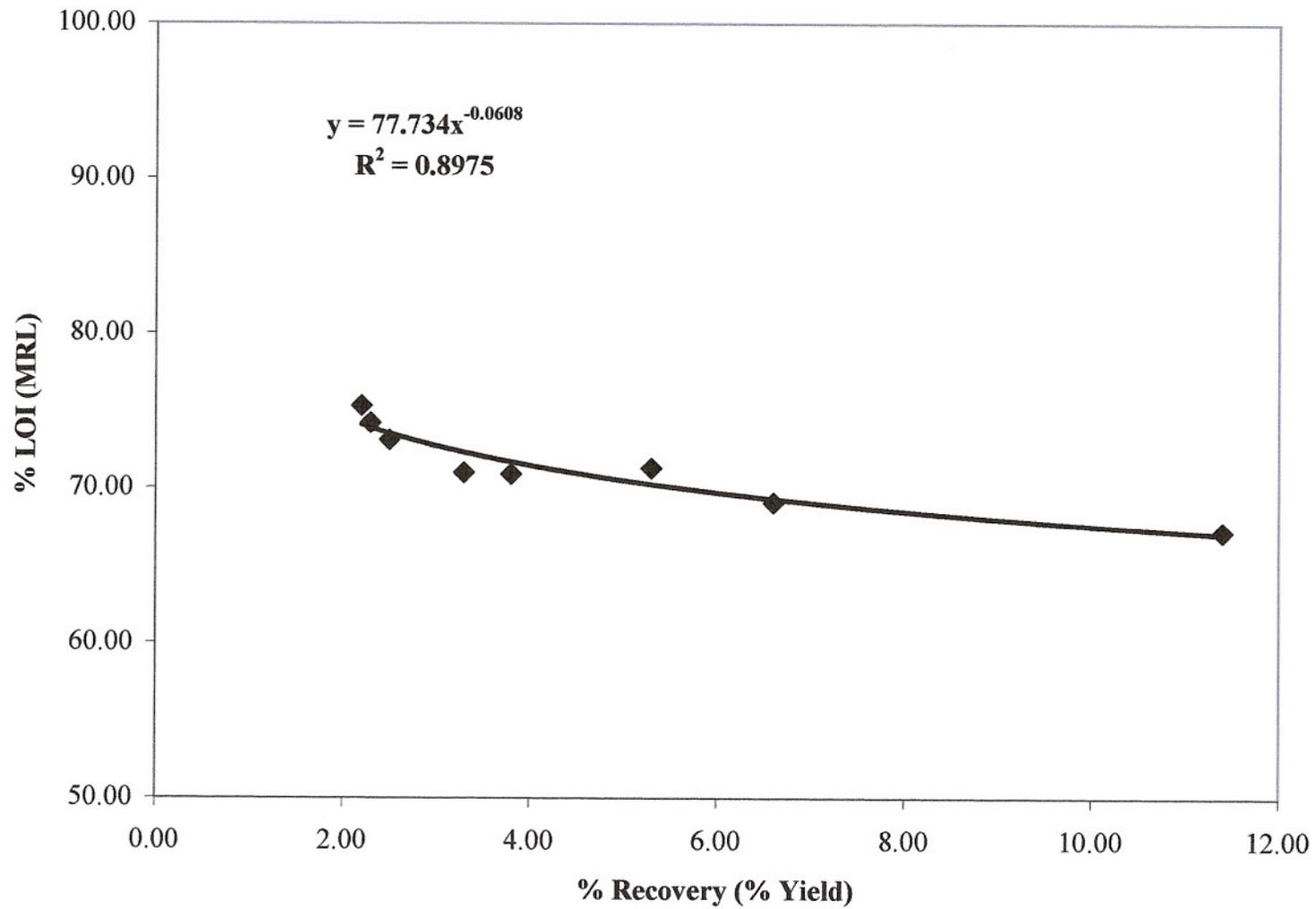
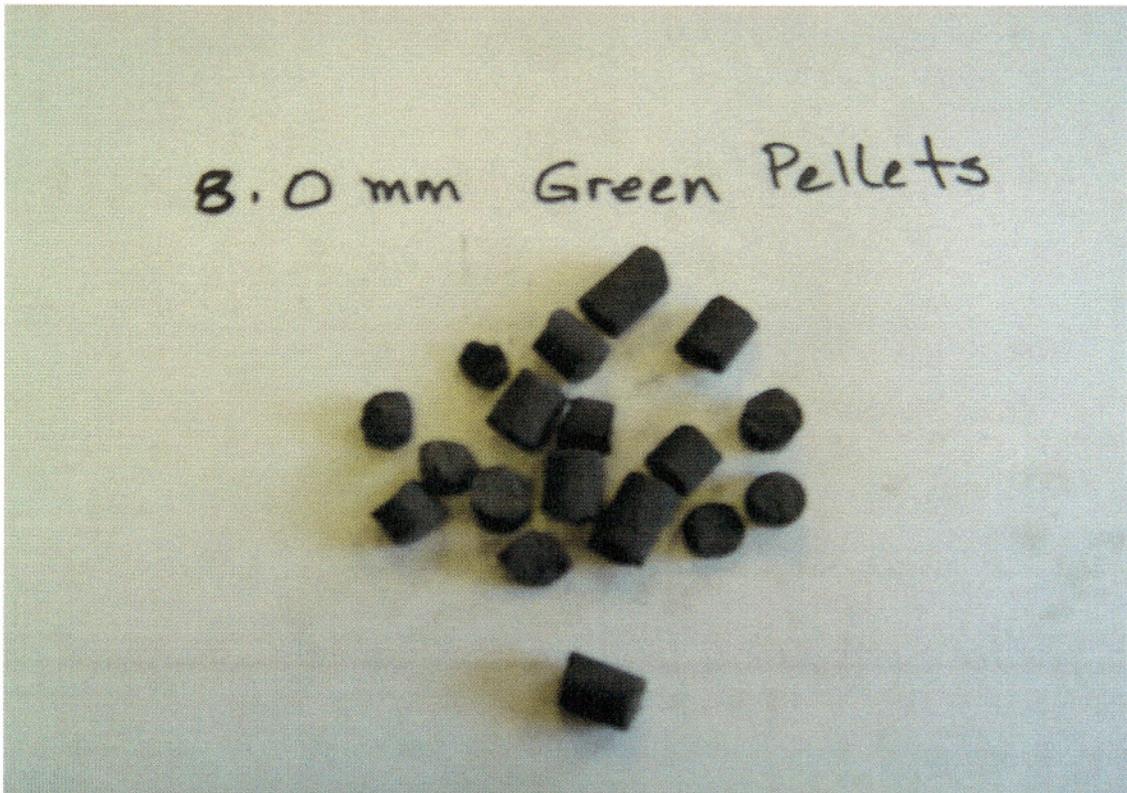


Figure 6: Recovery (% Yield) versus % Carbon (% LOI MRL) for High Carbon Product Continuous Pilot Plant Tests

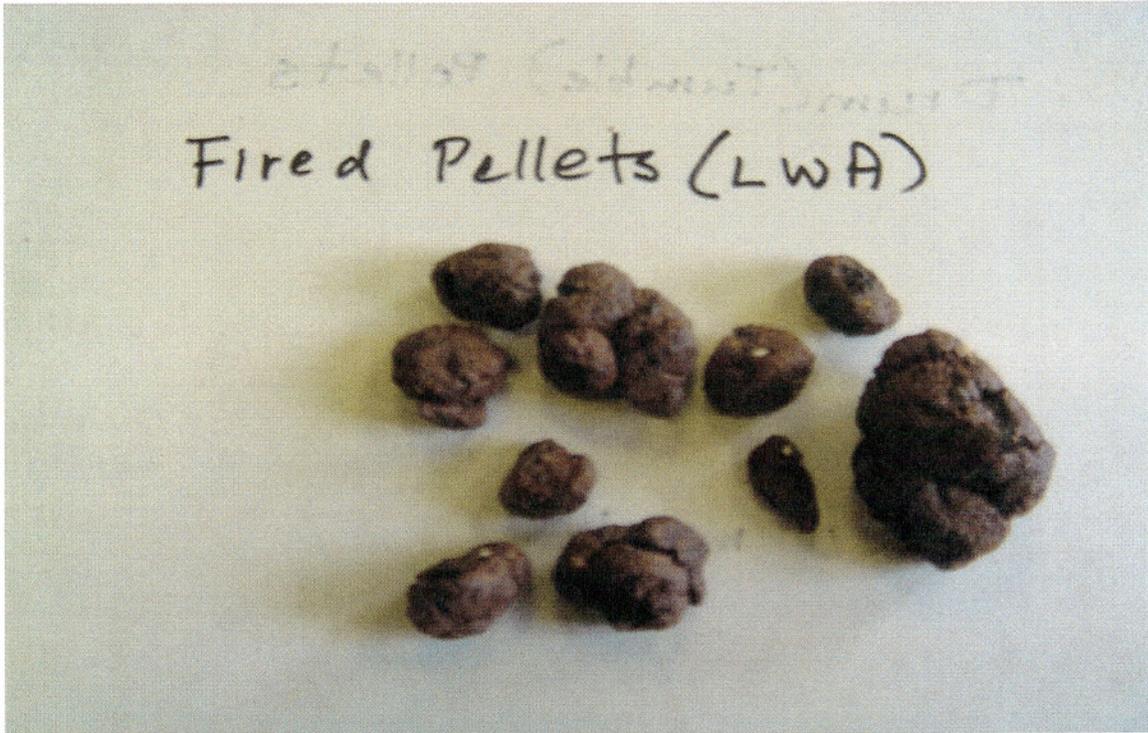




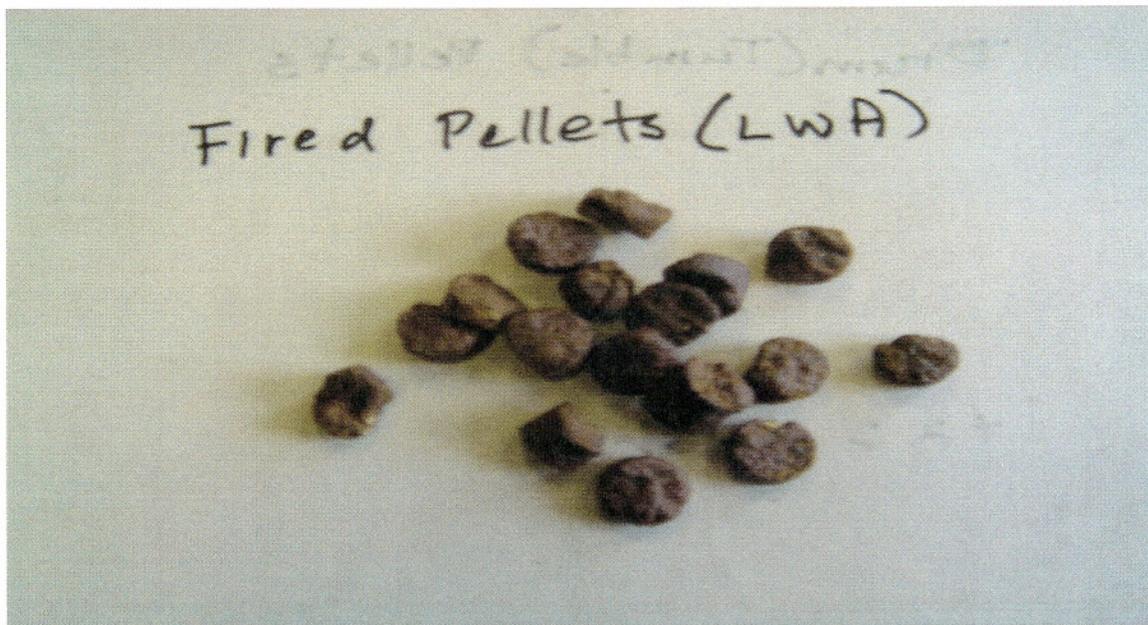
Photograph 1: 12mm diameter Green Pellets (Commercial Flat Die Pelleting Press)



Photograph 2: 8 mm diameter Green Pellets (Laboratory Flat Die Pelleting Press)



Photograph 7: 9.5 mm Pseudo-spherical Fired Pellets (LWA) (Laboratory Rotating/tumbling Drum)



Photograph 8: 12 mm diameter Fired Pellets (LWA) (Commercial Flat Die Pelleting Press)



Photograph 17: Bloated 20 cc Briquettes after Firing at 2200° F



Photograph 18: 20 cc Pillow-shaped Green Briquettes



Photograph 15: LWA Produced from fired briquettes after crushing and sizing



Photograph 16: LWA Produced from fired briquettes after crushing and sizing