OST TECHNICAL PROGRESS REPORT
BY-PRODUCT UTILIZATION TEAM-- FY 2001 RESULTS

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DESCRIPTION: The Solid Waste Management Team addresses problems related to solids that are by-products of processes for the production of energy from fossil fuels. The focus is primarily on the potential environmental consequences of utilization. This work addresses by-product utilization goals in the Environmental Product Line, the Power Systems Advanced Research Product Line, and the Watershed Management Product Line. Currently, the team's projects are focused on coal combustion by-products (CCB), primarily fly ash.

RESEARCH OBJECTIVES: In this fiscal year, the Solid Waste Team began development of a bulk leaching protocol based on the results of previous comprehensive leaching tests to quantify the release of trace elements from CCB. The analysis of leaching data was continued to relate release of metals to acid neutralization of CCB, to mineral speciation, and to distribution in crystalline or amorphous phases. Using the Microtox protocol, work was begun to determine the biological toxicity of CCB. Laboratory studies to test the use of fly ash to stabilize phosphate slurries and poultry litter were conducted to assess the potential for use of the CCB/waste material mixtures as agricultural amendments. The adsorption of metal ions by fly ash was studied to determine the effectiveness of fly ash as a cap for contaminated sediments.

LONG TERM GOALS / RELATIONSHIP TO NETL PRODUCT LINES: The By-Product Utilization Team develops solutions to environmental problems related to wastes generated during the mining, processing, and utilization of coal. Current research activity is focused on CCB, particularly fly ash. Considering all applications, only 25% of CCB are currently utilized (ACAA, 2000). Increased usage of this material is constrained by lack of data with respect to: (a) potential leaching of heavy metals to surface and ground waters; (b) the consistency of the composition of CCB when considered as an industrial raw material; and (c) the limited number of environmentally compatible and economic uses for CCB. The long term goal of the Solid Waste Team is to serve as an unbiased source of environmental data that supports increased utilization of CCB and to develop new applications that promote diversified utilization of CCB, particularly for by-products from new combustion technologies, such as fluidized bed combustion (FBC).

SUMMARY OF ACCOMPLISHMENTS:

Environmental Safety and Effectiveness. Demonstrating the environmental safety and effectiveness of CCB is critical to continued or increased utilization of this material. As was demonstrated with the recent Environmental Protection Agency regulatory determination process (Kim et. al, 2001; USEPA, 2000a), specific data is needed to alleviate public concerns, particularly with respect to use in bulk fill and backfilling.

Batch Characterization of CCB: Based on the results of long-term leaching tests and an extensive literature search, a protocol was developed incorporating strong acid digestion to determine total elemental composition, serial static tests at pH 4 and 8 to estimate availability of ions, and serial leaching with deionized water at various liquid-to-solid (L/S) ratios to estimate the rate of release of various ions from CCB.

Isotopic Tracers: Three sets of water samples were collected from the Omega Mine in WV. There was a significant difference in the strontium concentration in water samples from the grouted and ungrouted portions of the Omega Mine. The $^{86}Sr/^{87}Sr$ ratio in water samples from the ungrouted portion of the mine was between 0.7152 and .7159, while the ratio in water from the grouted portion was 0.1740. The differences in the samples from the grouted and ungrouted portions of the mine indicate that this approach can be used to identify the source of the water in the grouted portion of the mine.

Water Quality Monitoring at CCB Remediated Sites. As part of its effort to increase the utilization of CCB, DOE is cooperating in several projects utilizing CCB in site remediation. Potential impacts on water quality are a major concern in all of these projects. To allay the concerns of environmentalists, it is desirable that an unbiased third party obtains water samples, arrange for analysis, and interpret data. The Office of Project Management at NETL has requested that the By-Product Utilization Team serve in this capacity, based on their technical expertise and their extensive experience dealing with water quality issues related to the use of CCB. The water samples include pre-project background samples and up and down gradient samples collected for at least one year after the completion of the project.

Specialized Leaching. The By-Product Utilization Team has conducted extensive long term leaching tests on a random population of CCB samples. Efforts are currently focused on analyzing the data generated from these tests and relating leaching results to the chemical and physical properties of the fly ash. Because the current sample set contains 48 samples from 10 states and from both pulverized coal combustors and FBCs, leaching tests will not be continued on additional random samples. Leaching tests are being performed on samples such as the poultry litter/CCB composite and the phosphate slurry/CCB mixtures.

Biotoxicity Test (Microtox) as an Indicator of Potential CCB Toxicity A simple test to determine biological toxicity of CCB in water would provide significant support for increased utilization. Many biological methods are very cumbersome (e.g., using rats), and some tests require a long time for the organism to develop (fathead minnows, trout). Microtox uses a luminescent bacterium that in the presence of a toxicant loses its ability to luminesce. Using the Microtox assay, 20 CCB samples have been screened for their degree of inherent bio-toxicity. This study included inductively coupled plasma (ICP) analysis to determine if elevated concentrations of metals included in the Resource Conservation and Recovery Act list could be correlated to measured toxicity values. The majority of CCB tested exhibited very low toxicity.

By-Product Stabilization Other by-products, particularly phosphate slurries and poultry litter, could be utilized more readily if they are stabilized with CCB.

Remediation of Phosphate Slurry with Fluidized-Bed Combustion By-Products (FBCB): Phosphate slurry (also known as phosphate slime) is a waste product of the phosphate mining industry. It resists

dewatering, causing it to accumulate, consuming land and contributing to ground water pollution. We have determined that phosphate slurry and FBCB mixed together can be easily transported and disposed, and it might also be of use as an agricultural amendment.

Phosphate slurry and FBCB were mixed in ratios ranging from 0% fly ash to 70% fly ash by volume. Fly ash contents of 30% and less did not enhance settling. However, at fly ash contents greater than 30%, a dry, friable material was produced. Slump values and leaching tests indicated that this material, which could be produced by a simple process, could be used as an agricultural soil amendment.

Poultry Litter Stabilization The poultry industry is getting larger, while the geographic area that this industry occupies is getting smaller. Poultry litter contains nitrogen, as ammonia and as nitrate, and phosphorous. Land application of animal manure as a plant nutrient on the Delmarva Peninsula has been a major factor in the region's failure to meet water quality goals in the Chesapeake Bay Watershed (Zynjuk, 1995). The purpose of this experiment was to determine if the addition of fly ash had an effect on the release of phosphorous from chicken waste.

Increased CCB Utilization. The following tasks develop new technologies for increased utilization of CCB from electric utilities.

In-situ Capping of Contaminated Sediments: Capping involves the placement of a cover over an established layer of contaminated sediment, which seals the sediments physically and chemically, preventing pollutants from migrating into the overlying water. *In-situ* caps can serve as diffusive seals preventing upward migration of contaminants or as reactive barriers that encourage precipitation or degradation of toxic compounds within the cap.

Enhanced Leaching of CCB: Metals Recovery from CCB by Enhanced Leaching. In this exploratory project, samples with high concentrations of water-soluble heavy metals, as determined by previous leaching tests (Kazonich and Kim, 1999), were to be tested to determine if a simple technique could be used to accelerate leaching of potential contaminants for both metals recovery and environmental improvement. An acid solution with a pH between 1 and 4 was considered a suitable leachant. Economic projections were made based on the volume and concentration of the leachate solutions assuming the best available commercial extraction technology.

A preliminary cost evaluation of metals extraction from fly ash indicated that the extremely small amount of saleable trace metals in commercial fly ash could not justify capital expenditures for extraction on the basis of metal values (typically a few cents per ton of ash). Avoided disposal costs are also likely to be relatively small and would not be comparable to the extraction and separation costs for metal recovery from CCB. Although Seidel and Zimmels (1998) claimed that economic quantities of iron and aluminum could be recovered from CCB, their leaching experiments showed that, at pH < 4, less than 25% of the metals could be leached. The leaching rate and yield were affected by the presence of calcium, which is the most soluble of the major metals in CCB. Due to the unfavorable economic projection, work on this project was suspended. No additional work is scheduled on this project.

CCB & Road Salt. The objective of this task was to determine if a combination of CCB and road salt would decrease environmental problems, particularly plant toxicity, associated with the use of road salt

for ice control during the winter. In FY01, a comprehensive literature search was conducted on the potential to use CCB as an additive to road salt.

Careful management can solve environmental and corrosion problems, according to the Salt Institute (2001). Salt is available, is nontoxic, is cheap, and is easy to store and spread. Highway departments use a combination of sand and salt to melt snow and ice; sand is frequently used as an additive to keep road salt from forming large aggregate particles. When this project was initiated, it was assumed that CCB (fly ash or bottom ash) could be a beneficial and inexpensive replacement for sand when combined with road salt. By increasing the proportion of bottom ash, and using less salt, environmental problems associated with excessive salt might be prevented. It was decided to also test fly ash as the sand replacement, since fly ash is routinely used as a sand replacement in other applications.

Only one study on the use of fly ash or bottom ash as a road traction agent was located (Wiscomb, 1997). Air quality was compared after using bottom ash, scoria, and sand (with added salt) as traction materials in Gillette, Wyoming. Bottom ash was considered as an inexpensive replacement for sand, which is not locally available. Data from these tests suggested that bottom ash had fewer impacts on air quality. Based on research of salt and abrasives, it was determined that bottom ash and cinders are inexpensive, but will not melt ice and can be covered with snow. Bottom ash is not uniform in size and might require other expensive equipment to crush it to fit into the salt sprayers. While fly ash is finer than sand, it has been used successfully as a sand replacement in other applications. However, in-depth analysis of the problem indicated that fly ash could contribute to fugitive dust problems, and that substituting CCB for sand was unlikely to reduce the amount of salt used. Therefore, work on this project was discontinued.

RESULTS:

Batch Characterization of CCB. Metal solubility from CCB can be influenced by pH, complexation by inorganic species or dissolved organic matter, and the reducing properties of the waste. The total concentration of contaminants in a waste material does not correlate with release to the environment. The availability for leaching is a more relevant parameter for environmental purposes than the total concentration. To determine this, an availability test based on the extraction of fine-grained material at a high L/S ratio and at a controlled pH of 4, assumed to be a lower pH limit found in natural environments, was used. A second test at a pH of 8 or above is recommended to determine leachability of oxyanionic species. In addition, the acid consumption can be used to estimate the neutralization capacity of the material.

Changes in the leachability of materials and the total amount of material that can be leached as a function of time can be assessed by testing at different L/S values. The rate of release of material from the waste that can be expected under normal environmental conditions over various periods of time is acquired by simulating natural conditions using various L/S ratios of leachant, normally deionized water, to waste solid. The L/S ratio is related to a time scale through the cumulative infiltration rate. A batch extraction with deionized water at an L/S ratio of 1 can be used to assess the leached quantities during a short term (<10 years). A batch extraction with deionized water at an L/S ratio of 10 can be used to assess the leached quantities at medium term leaching (<50 years). This L/S ratio has also been found to approximate the results from column leaching at an L/S ration of 10.

The alkalinity of a stabilized product proves to be a significant factor in controlling leachability. The leachability of metals is drastically reduced at high pH due to the formation of insoluble hydroxides or adsorption. The leachability of oxyanions is at a maximum at neutral pH and in most cases decreases at high pH.

Isotopic Tracers: Strontium isotopes have been used as naturally occurring tracers for geologic processes (Capo et al., 1998; Stewart et al., 1998). Strontium has four naturally occurring stable isotopes and a radiogenic isotope formed by the decay of rubidium. In shales, sandstones, and carbonates, the concentration of strontium may be between 20 and 600 ppm. Relative to other materials, such as soil, the strontium content of fly ash is highly enriched (Straughan et al., 1981). In water in contact with sedimentary rocks, such as coal and shales, the ratio of strontium isotopes would be related to that of the parent material. Water from different sources, such as a grouted area of a mine, would have a characteristic isotopic signature. Assuming that the resulting mixtures are not modified by chemical reactions or physical changes, the isotope ratio of the final mixture can be related to the source by simple mixing models.

In the case of the Omega Mine, the flux of strontium out of the mine would also be influenced by the volume of water in contact with the grouted area and the rate of strontium leaching from the grout material. There are two horizontal borehole discharges from the grouted and ungrouted portions of the mine and 12 other sampling points around the down-dip perimeter of the mine.

Samples are being collected at the site location in acid-washed high density polyethylene bottles, filtered through 0.45 μm filters, and then acidified with 2% Ultrapure HCl. Duplicate acidified (but unfiltered) samples are analyzed by ICP to determine the concentration of Sr. The filtered samples are dried completely in acid-washed Teflon® beakers. A 0.5-ml aliquot is centrifuged to remove any remaining solids and then put through columns containing Sr-SPEC resin. A series of rinses flushes everything except the Sr, and a final rinse releases the Sr from the resin. The Sr rinse is dried down completely again in acid-washed Teflon®, then the Sr residue is dissolved in 10 ml of 3N Ultrapure HNO3. The results that are obtained after numerical analyses are in the form of a ratio of the isotopes of ^{87}Sr to ^{86}Sr. The ^{86}Sr/^{87}Sr ratio of water samples from the ungrouted portion of the mine was between 0.7152 and .7159, while the ratio in water from the grouted portion was 0.1740. The differences in the samples from the grouted and ungrouted portions of the mine indicate that this approach can be used to locate the source of the water in the grouted portion of the mine.

Water Quality Monitoring at CCB Remediated Sites. Five sets of pre-injection and 3 sets of post-injection samples have been collected from 6 sampling points around the perimeter of the injection area at the Rostraver Airport. Average values indicate post-injection increases in the concentration of calcium and sodium, but there was no significant change in the concentration of other elements. Samples collected from 12 sampling points at nearby residences also indicate no change attributable to the placement of the CCB.

Specialized Leaching. In some preliminary tests, samples of FBC ash were mixed with water or 0.1 normal acid and allowed to solidify. After a few weeks, the solids attained the hardness of chalk or wallboard. This amount of confined strength would be adequate to prevent mine subsidence but it could still be crushed easily by hand for leach testing. Experience gained from previous leaching indicates that the solid would only have to be crushed to minus ¼-inch. Crushing to smaller sizes might be worthwhile in lab work because it would take little additional effort, but would speed up leaching because of increased surface area. The NETL By-products Utilization Team has previously leach-tested 5 FBC ash samples. Three of those samples solidified, which stopped the test after only a few weeks duration. The other two samples experienced a decrease in the lixiviant flow for a short time, but later returned to normal flow (an indication of the formation and subsequent dissolution of solids). The release of metals observed in tests of non-solidifying FBC ash samples indicates that the solidifying ash samples should be retested as a granulated material.

Biotoxicity Test (Microtox) as an Indicator of Potential CCB Toxicity: Using the Microtox assay, 20 CCB have been screened for their degree of inherent bio-toxicity. This study included an ICP analysis to determine if elevated concentrations of RCRA metals could be correlated to measured toxicity values. For each Microtox test, a 1-g sample of CCB was added to a flask with 100 ml of H_2O and shaken continuously at 140 rpm for one week in an attempt to regulate the pH. The bacterial population is most stable in the pH range 6 to 8. When the pH is above or below this range, the concentration of H^+ or OH^- ions may affect the bacteria, thus hindering observations of biotoxicity by other elements. Under the normal protocol, CCB samples generally exhibited no toxicity. To induce a toxic response, the samples were heated to increase the dissolution of metal ions or were treated with dilute acetic acid daily (minimum 100-μl and maximum 1-ml treatments per day).

The value EC50, as defined by Microtox, is an indirect measure of the test organism's condition. An EC50 value indicates that 50% or more of the bacterial population is healthy, and the decrease in luminosity cannot be attributed to the contaminant. If the EC50 value is less than 50, then the contaminant is assumed to have affected some proportion of the population. The meaning of the EC50 values is given in the following table. The measured EC50 of the CCB samples tested were generally in the non-toxic range, and for many of the samples that showed moderate toxicity, the effect could be related to pH.

The Microtox data was compiled and analyzed to determine if any toxicity trends could be observed. The overall conclusion is that the ashes tested to date appear to show very little or no toxicity on the bioluminescent organism. Where toxicity was observed, an attempt was made to determine if an elevated metal concentration was the likely mechanism. However, in some cases, extremely high levels of soluble metals did not appear to result in toxicity, and in other cases of toxicity, one would not predict it based on the metal analysis.

By-Product Stabilization. Other by-products, particularly phosphate slurries and poultry litter, could be utilized more readily if they are stabilized with CCB.

Phosphate Slurry Remediation. Because of its high calcium content and pozzolonic properties, fly ash from FBC units can be mixed with phosphate slimes to form a stable material. A phosphate/CCB material is high in phosphorous and calcium and could be processed for direct use in agricultural applications, thereby converting two waste materials into a useful product. A process to produce

phosphate/CCB material has been designed.

Phosphatic slurries and fly ash were mixed in ratios ranging from 0% fly ash to 70% fly ash by volume. Fly ash contents of 30% and less did not enhance settling. However, at fly ash contents greater than 30%, a dry, friable material was produced.

Slump analysis was also performed on select fly ash and phosphatic clay mixtures. Slump is a physical test that defines how stiff or how resistant a mixture is to deformation. The mixture of 30% by volume (24.9% by wt.) remained a liquid throughout the sampling period and would not have given reportable results had it been evaluated in the slump cone. The 45% by volume (38.5% by wt.) sample took several days to report a slump result. The 65% by volume (55.2% by wt.) sample reported zero slump after only the second day.

Analyses, including orthophosphate content, the release of phosphorous and other minor fertilizer nutrients (K, N), nitrates, and ammonia are currently in progress. Leaching tests such as TCLP and multiple extraction procedure (MEP) are also being conducted to determine the feasibility of using the mixtures as soil amendments in the agriculture industry. A process design to produce CCB-phosphate material utilizes standard mixing equipment.

Small column leaching tests using 100 g of the fly ash and slurry mixture and sequential additions of 100 ml of synthetic precipitation (SP) has been initiated. Samples taken daily are analyzed for total phosphorous, hydrolyzable phosphorous, and nitrate.

Poultry Litter Stabilization. The poultry industry is getting larger, while the geographical area that this industry occupies is getting smaller. Poultry litter contains nitrogen, as ammonia and as nitrate, and phosphorous. If litter is applied to fields as a plant nutrient to supply enough nitrogen to plants, excess phosphorous is also applied. Phosphorous and nitrogen in the soil are washed off by rain and enter streams. Agricultural runoff is the largest source of non-point source pollution. Land application of animal manure on the Delmarva Peninsula has a significant impact on the region's failure to meet water quality goals in the Chesapeake Bay Watershed (Zynjuk, 1995).

The purpose of this experiment was to determine if the addition of fly ash had an effect on the release of phosphorous from chicken waste. Shake flask studies were conducted in which the material was placed in an Erlenmeyer flask with a leaching solution and placed on a laboratory shaker for a given length of time. The leaching solution was sampled at the end of the period and analyzed for phosphorous, nitrate, and ammonia. For this experiment, one chicken waste and 4 different fly ashes were used. The fly ashes were selected from an inventory of 50 ash samples based on their performance in previous studies on sequestering metals. The experimental design was based on the WET, the California Waste Extraction Test, using a 50-g sample and 500 ml leachant solution. The samples were a chicken manure control and mixtures of fly ash and chicken manure, 50% each by weight. The manure was homogenized and sieved to -50 mesh. The leachant solution was a synthetic precipitation, prepared according to EPA Method 1312 Synthetic Precipitation Leaching Procedure (deionized water, adjusted with a mixture of 60/40 sulfuric and nitric acids to a final pH of 4.2).

The flasks containing the sample and leachant solution were placed in a laboratory rotary shaker @ 25 ^{o}C and 125 rpm for 7 days. Samples of the solution were taken at the end of the experimental

period and analyzed by inductively coupled plasma-atomic emission spectroscopy (ICP-AES) for phosphate ion and by ion chromatography for total phosphorous, nitrate, and ammonia. Mixtures of chicken manure and fly ash had at least 40% less phosphorous and phosphate in solution than chicken waste alone.

There was no reduction in ammonia concentration with the addition of fly ash to the manure samples, although studies by others have shown that ammonia is adsorbed by CCB. Nitrate was not detected in the control (manure-only) solutions. It was apparently released from the CCB. Currently, manure is land-applied based on the release of nitrogen. The results of this test indicate that the addition of fly ash has no appreciable effect on the availability of nitrogen, but does reduce the release of phosphorous.

Increased CCB Utilization. The following tasks develop new technologies for increased utilization of CCB from electric utilities.

In-Situ Capping Material of Contaminated Sediments. Capping involves the placement of a cover over an established layer of contaminated sediment, which seals the sediments physically and chemically, preventing pollutants from migrating into the overlying water (Apak et al., 1998; Palermo et al., 1998; Wang et al., 1991). *In-situ* caps can serve as diffusive seals, preventing upward migration of contaminants, or as reactive barriers that encourage precipitation or degradation of toxic compounds within the cap (Jacobs et al., 1999; Ricou et al., 1998). In this fiscal year, laboratory tests were conducted using a sediment that was collected from a contaminated site, a zinc smelter. The sediment was placed in laboratory mesocosms with and without a CCB cap. A more conventional cap (soil) was used as a positive control. Water was collected and analyzed by ICP to determine if the CCB caps prevented the diffusion of metals into the overlying water.

Two hundred ml of sifted soil from a zinc smelter was placed in each of 4 (1-L) beakers, and 80 ml of de-ionized water was added to saturate the soil. Three different caps were added to the soil, and one beaker was used as a control. The first cap was topsoil, the second was sand, and the third was fly ash. Fifty ml of the three materials was placed on top of the soil. Three hundred ml of de-ionized water was carefully added to the soils so that the materials would not be disturbed.

Ten ml of water were drawn off the surface water daily, the water was filtered, acidified, and analyzed for metals by ICP. The pH and temperature were taken daily. After 10 days, samples of the soil were taken. Three small core samples were taken from each beaker. Approximately 1-cm slices of the core were placed in scintillation vials. The cap was also sliced and placed in a scintillation vial. These samples were then digested by adding 5ml of HCl and 5 ml of HNO3, heated for approximately 30 minutes, and left to sit over night. They were then filtered and sent to the lab for analysis.

Grab samples of the sediment were put into centrifuge tubes to collect the pore water. Tubes were placed into the centrifuge for 30 minutes, and then they were acidified with HCl and sent to the lab for analysis. The zinc concentration in the water over the uncapped soil contained approximately 1000 mg/l of zinc. The sand and FA# 44 were relatively ineffective at preventing the migration of zinc from the contaminated soil. However, the other fly ash samples were as effective or more effective than the topsoil in sequestering the metals below the cap.

FY01 Publications

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