

## High Volume Utilization of Fly Ash Containing Mercury Impregnated Activated Carbon

Chett Boxley, Akash, Vicki Siegel

Ceramatec, Inc., 2425 South 900 West, Salt Lake City, UT 84119 ([cboxley@ceramatec.com](mailto:cboxley@ceramatec.com))

### Abstract:

Mercury emission control regulations that are set to be enforced in 2010 will cause coal-fired power plants to lose fly ash sales revenue and increase ash disposal costs. This will occur because the most promising mercury control technology employs activated carbon to adsorb the mercury; thus causing the fly ash produced after implementation of this technology to be unusable for current concrete applications. In response to this, we have successfully demonstrated that reaction-bonded fly ash can be used as a potential cement/binder for concrete applications. Our research has demonstrated that various types of fly ashes, either with or without activated carbon, could be made into practically strong, usable parts through careful selection of the activator, the water amount, and the curing temperature. This process not only generated strong bonding materials, but it also encapsulated the mercury and activated carbon within the material matrix. Consequently, this new cement composition is not environmentally hazardous, and it does not interfere with the functionality of other organic additives commonly used in concretes (e.g. air entraining agents). For example, we have demonstrated a dramatic improvement in foam index test for an *activated carbon containing fly ash* (where the number of drops required in the foam index test was reduced from ~500 to ~37 for a 3.4% LOI fly ash and down to 9 drops for a 2.5% LOI fly ash). A compressive strength of as high as 100 MPa (14,500 psi) was also obtained. Due to the similarity of using this new approach compared to using portland cement, it will not be a significant economic burden for concrete manufacturers or society as a whole to adopt this technology. Current work is now focused on scaling up this approach in order to process large volumes of various types of fly ash. Ceramatec is also actively looking to partner with coal companies or cement companies who have an active interest in this area. Our proposed technology will actually bring four-fold benefits: 1) making the carbon injection technology for mercury control more easily acceptable for power plants; 2) removing the burden of power plants to dispose of fly ashes; 3) making the high carbon fly ash acceptable to the cement/construction companies; and 4) maintaining cleaner air by reducing the production of portland cement.

### Introduction

More than half of the electricity generated in the U.S. is produced by coal-fired facilities. These power plants also produce coal combustion products (CCP) including fly ash, the non-combustible mineral portion of coal. The production of CCP has been increasing in step with the increasing demand for electricity, creating a more urgent need to minimize the impact of these waste products on the environment - preferably in a cost-neutral or beneficial manner.

Fly ash can replace a portion of the Portland cement used in concrete, which brings several significant benefits<sup>1</sup>:

---

<sup>1</sup> Qunhui Zhou, Yinzhi Zhang, Sid Nelson Jr., Rafic Minkara, Evaluations of Concretes Containing Mercury Sorbents, Presented at the Combined Power Plant Air Pollutant Control Mega Symposium, Washington, DC, Aug. 30 – Sept. 2, 2004

1. For power plants, it reduces disposal costs while actually increasing revenues through the sale of fly ash;
2. For concrete manufacturers, it can dramatically improve product performance (higher strength, decreased permeability, increased durability, better workability, and chemical resistance) while actually reducing costs, since the fly ash is less costly than Portland cement, and
3. For the benefit of society at large, greenhouse gas emissions are reduced by minimizing the production of Portland cement, while the number of landfills needed for the disposal of fly ash is also reduced.

In March of 2005, the EPA published final regulations to control mercury emissions from coal-fired electric utilities, and national enforcement is expected to begin in 2010. For the majority of coal-fired power plants without scrubbers, the most mature retrofit technology available today is the injection of sorbents such as powdered activated carbon (PAC) into the flue gas upstream of the particle control devices (PCDs). Existing particle control equipment (either an ESP or a fabric filter) collects the mercury-containing sorbent along with the fly ash. Generally speaking, however, the fly ash produced with mercury control technologies has much higher mercury (a few  $\mu\text{g/g}$  to  $\sim 100\mu\text{g/g}$ ) and carbon (up to  $\sim 30\text{wt}\%$ ) content as compared to conventional fly ash.

However, preliminary studies have revealed that the PAC injection technology has two negative impacts on the potential use of fly ash in concrete:

1. Fly ash samples with even low concentrations of carbon are discolored; thus, making the material less marketable than other sources of fly ash without PAC.
2. Fly ash containing PAC (above 1% LOI) failed foam index tests. Failing this test prohibits the plant from selling the fly ash for concrete applications.

If carbon-containing fly ash is not suitable for concrete applications, then there will be significant economic and environmental barriers to implementing the EPA regulations. Hence, there is an urgent need to develop novel, high volume utilization technologies for fly ash containing very high concentrations of either unburned or activated carbon from mercury control technologies.

In response to this problem, we have developed the use of a reaction-bonded method for treating the fly ash, in which the resulting materials will encapsulate the contaminants, including mercury and activated carbon. We believe the encapsulation effect will not only further immobilize the mercury, but also solve the interference effects between the carbon and the air entraining agents (AEA). This cement material can then be used in several proposed ways. First, the formed cement slip can be directly added to aggregates to form concrete. Another alternative option is obtaining powder from the dried slip, and then combining the powder with aggregates to form concrete. The advantage here is that the storage and distribution of the treated fly ash are simple.

Reaction-bonding for the “treatment” of fly ash has not been demonstrated to the best of our knowledge. However, in terms of producing treated materials from high carbon/mercury containing fly ash and using them for concrete applications, a few critical developments need to be demonstrated:

1. *Need to demonstrate the treatment process with high concentrations of carbon/mercury containing fly ash;*
2. *Need to demonstrate the encapsulation effect of the mercury in the treated fly ash via leaching tests;*
3. *Need to demonstrate the encapsulation effect of the activated carbon in the treatment process via foam-index tests, and show the feasibility of using a treated fly ash as an additive in concrete applications;*
4. *Need to demonstrate the feasibility to form treated fly ash-based cement using high concentrations of carbon/mercury containing fly ash.*

## **Experimental Procedure**

A total of five fly ash samples were procured during the initial phase of this development project. Table I lists each of these samples and their ID, the procurement source, and the historical information available to Ceramatec. Fly ash samples 1A and 2A are both baseline samples for 1B and 2B, respectively. The baseline samples acquired did not contain excess Hg or C from the PAC injection technology. A baseline sample was not available for the EERC fly ash sample, and the sample provider did not inform us whether the EERC fly ash sample was Class F or Class C. This was determined by Ceramatec according to ASTM C618.<sup>2</sup> Generally speaking the fly ash produced from the burning of lignite or sub-bituminous coal more likely falls into the Class C category.<sup>2</sup>

The procured fly ash samples were characterized for their constituents, crystallinity (phases), surface area, particle size, carbon content, and Hg content. Next, they were treated with different ratios of activators and dried at different curing temperatures. The type and ratio of activator to solids are proprietary and therefore will not be discussed here. A DOE matrix was designed to study the individual effects of these variables on properties like foam index and compressive strength. The overall experimental approach is shown in Figure 1 below.

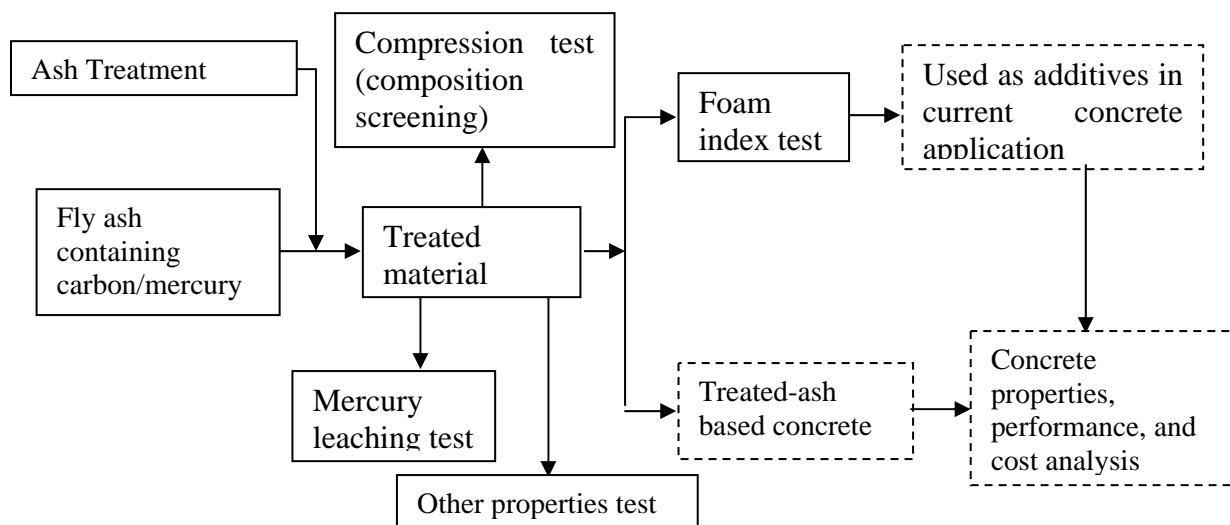
---

<sup>2</sup> <http://www.tfhrc.gov/hnr20/recycle/waste/cfa51.htm>

**Table I. Fly ash samples studied in this project**

Sample ID	Sources <sup>a,b</sup>	Historical information
<b>EERC</b>	Energy and Environmental Research Center (EERC) at University of North Dakota	Fly ash from a power plant burning lignite coal and injecting Norit regular activated carbon for mercury control
<b>1A</b>	ADA-ES, LLC	Fly ash from a power plant burning sub-bituminous coal and equipped with an electrostatic precipitator (ESP) to collect the fly ash
<b>1B</b>	ADA-ES, LLC	Fly ash from the same power plant as 1A, but with bromine-treated activated carbon injection for mercury control
<b>2A</b>	ADA-ES, LLC	Fly ash from a power plant burning sub-bituminous coal and equipped with spray dryer absorbers (SDA) for sulfur emission control and fabric filters (FF) to collect fly ash
<b>2B</b>	ADA-ES, LLC	Fly ash from the same power plant as 2A but with bromine-treated activated carbon injected for mercury control

Note: a. The sources listed here are the sample provider for Ceramatec, not the fly ash producer.  
 b. The EERC fly ash was generated through the DOE mercury control program supervised by Ms. Lynn Brickett at NETL and the fly ash samples from ADA-ES were generated through the DOE mercury control program supervised by Mr. Andrew O’Palko at NETL.



**Figure 1. The flow chart for the overall planned work**

Treated ash sample casting: The casting slip was prepared by mixing the activator solution with the appropriate amount of water, and then a superplasticizer was added to help the dispersion of the fly ash. To this solution, the fly ash powder was added, which was followed by vigorous shaking to get homogeneous slips. The reaction starts upon mixing of the components, so the viscosity of the slip gradually increases. In some cases the viscosity increased dramatically within minutes, making the casting of the slip impossible.

For the samples investigated in this study, the EERC fly ash had the longest working time (i.e. the viscosity increased the slowest) compared to the 1A and 1B fly ash samples; and in general using different activators provided slightly different working times. After shaking, the slips were cast into cylindrical molds ( $\text{Ø } 0.5''$  H 1.25'') under strong vibration in order to eliminate air bubbles. Five samples were molded from each batch for testing. Each sample was sealed in the mold, and there was a 7-day interval between the sample casting and the compression testing for all samples, this was done to ensure that the samples were cured in a consistent manner.

## **Results & Discussion**

### **Characterization of the procured fly ash samples**

Composition analysis: One of the most important properties for fly ash is its chemical composition. The composition of the fly ash samples is determined by the type of coal burned and the configuration of the power plants, but each sample can be categorized as either Class F or Class C fly ash.<sup>2</sup> Class F fly ash contains a low concentration of CaO (i.e. between 1 and 3% by weight), whereas Class C fly ash contains anywhere from 8 to 13% CaO by weight. A sample of each of the five procured fly ash types was sent to Wyoming Analytical Laboratories, Inc. (Golden, CO) for bulk chemical composition analysis by X-ray fluorescence (XRF) and also loss on ignition (LOI) characterization. The major components of each fly ash sample are listed in Table II.

The compositions of samples 1A and 1B (group "1") were quite similar to each other since these two samples were generated at the same power plant. Table II shows that the group "1" samples contained approximately 37% SiO<sub>2</sub>, 20% Al<sub>2</sub>O<sub>3</sub>, 8% Fe<sub>2</sub>O<sub>3</sub>, 21% CaO, and 1% SO<sub>3</sub>, and this fly ash was determined to be of the Class C type. The group "2" fly ash samples (2A and 2B) contained a greater amount of lime (CaO, 28%) and sulfur (SO<sub>3</sub>, 14%) relative to the group "1" samples. This is because the power plant, from which they were generated, was equipped with a spray dryer absorber (SDA). The SDA intentionally injects lime into the system to trap sulfur; thus, lime and sulfates are collected together with the fly ash down stream. However, despite these differences, according to ASTM C618 it was concluded that the group 2 samples were also of the Class C fly ash type. The EERC sample contained 41% SiO<sub>2</sub>, 27% Al<sub>2</sub>O<sub>3</sub>, 4% Fe<sub>2</sub>O<sub>3</sub>, 16% CaO, and 1% SO<sub>3</sub>, and was also classified as a Class C fly ash.

**Table II. Major components of fly ash samples**

Sample	SiO <sub>2</sub> (wt%)	Al <sub>2</sub> O <sub>3</sub> (wt%)	Fe <sub>2</sub> O <sub>3</sub> (wt%)	CaO (wt%)	SO <sub>3</sub> (wt%)	Class C or F
<b>EERC</b>	40.84	26.60	4.38	16.21	1.19	C
<b>1A</b>	37.09	19.65	9.64	21.99	1.35	C
<b>1B</b>	36.09	20.26	7.05	21.45	1.22	C
<b>2A</b>	25.71	14.68	5.67	27.89	14.53	C
<b>2B</b>	24.04	14.68	4.79	29.28	14.39	C

According to previous studies high carbon content makes fly ash un-suitable for use in concrete mixtures as a replacement for Portland cement, which is currently the largest utilization for fly ash; however, it is believed that the mercury contamination does not interfere in the concrete matrix.<sup>3</sup> Consequently, it is important to know the carbon content for these five fly ash samples. One way to estimate the carbon content is to use the LOI as an approximation, which was performed by Wyoming Analytical Labs. As a confirmation of the carbon content determination by LOI, a fly ash sample of each type was sent to SGS North America, Inc. (Denver, CO) for carbon content analysis. Additionally, since the fly ash power plants were unable to provide us with the Hg content of the fly ash, a sample of each fly ash type was sent to ChemTech Ford, Inc. (Salt Lake City, UT) for Hg content analysis by atomic absorption spectroscopy. Table III summarizes the results of the LOI, carbon content analysis, and mercury content analysis.

**Table III. LOI and carbon content analysis for fly ash samples**

Sample	LOI (wt%)	TOC (wt%)	Mercury content (µg/g)
<b>EERC</b>	3.37	2.89	17.5
<b>1A</b>	1.67	1.93	0.5
<b>1B</b>	2.5	2.4	1.12
<b>2A</b>	3.18	0.26	0.18
<b>2B</b>	3.81	0.77	1.4

Table III shows that the carbon content values recorded for each sample are smaller than the LOI values except for sample 1A. This means the measurements recorded for sample 1A may have some errors, and should be reproduced. Only one measurement was made per sample, so no statistics were generated for these samples. Both methods show an increase in carbon content between samples 1A and 1B, which is due to the activated carbon introduced by the sorbent injection technology. A similar observation was made for samples 2A and 2B. It is interesting to note that the carbon content for the different types/sources of fly ash samples varied significantly. All LOI values determined were within the limit for Class F or Class C fly ash (<6 wt%) as specified by ASTM C618-3. However, the fly ash generated by the power plants with sorbent injection cannot be used for concrete applications because the activated carbon interferes with the air entraining agent (AEA) used in concretes. Also, Table III shows that there is a large difference between the experimentally determined carbon content and the LOI values for samples 2A and 2B. It is believed that because samples 2A and 2B had significantly higher lime

<sup>3</sup> Constance Senior, C. Jean Bustard, Kenneth Baldrey, Travis Starns, Michael Durham, Characterization of fly ash from full-scale demonstration of sorbent injection for mercury control on coal-fired power plants, presented at the 28<sup>th</sup> International Technical Conference on Coal Utilization & Fuel Systems, Clearwater, FL, March 10-13, 2003.

and sulfur concentrations that these constituents may contribute significantly to the total LOI values, as shown in the phase identification section of this report.

The mercury content of the fly ash was rudimentarily characterized by atomic absorption spectroscopy (AA). The results in Table III show that the Hg content of sample 1B was more than doubled compared to sample 1A, which was anticipated since 1B employed a Hg control technology. Similar results were obtained for samples 2A and 2B, inasmuch as 2B showed an 8-fold increase in the Hg concentration. The highest Hg concentration was found in the EERC samples; however, recall that we were unable to obtain a baseline sample that did not employ a Hg control technology from this plant. As mentioned previously, it is believed that the mercury will not directly affect the fly ash utilization in concrete, and the environmental hazard effect from “high” mercury containing fly ash has been previously shown to be negligible.<sup>4</sup> Three leaching protocols, the toxicity characteristic leaching procedure (TCLP), the synthetic groundwater leaching procedure (SGLP), and the Synthetic Precipitation Leaching Procedure (SPLP) were used to show the encapsulation of the mercury in the treated fly ash material.

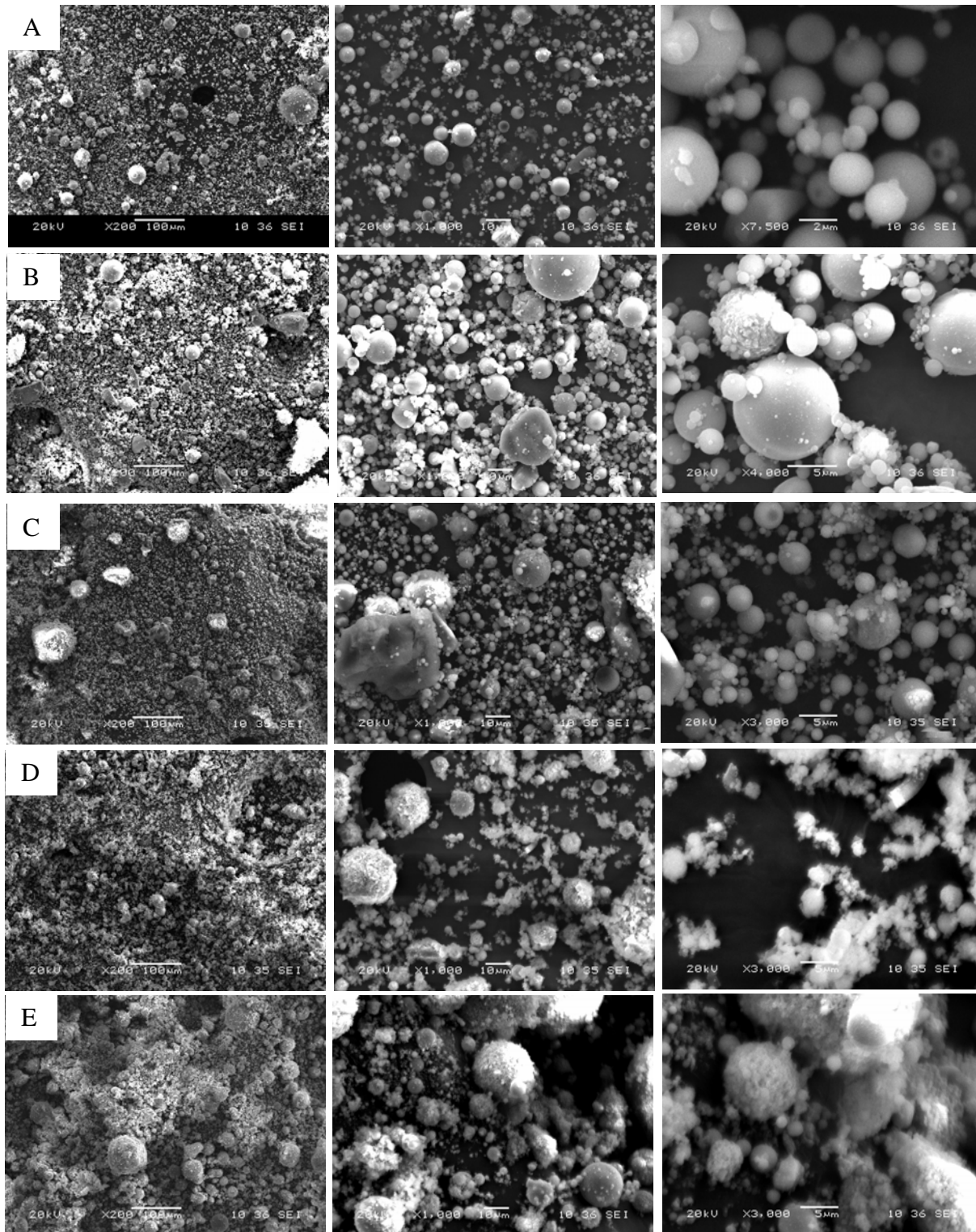
Surface area & particle size: The surface area and particle size of the fly ash were measured by the BET method and by laser scattering at Ceramatec, and the results are shown in Table IV. As expected, each sample had a broad particle size distribution, which is a common characteristic for fly ash samples. Furthermore, the EERC sample had a much finer particle size compared to the other samples. The EERC fly ash sample also had the greatest surface area, which may be partially due to the finer particle size and/or its high carbon content (~3 wt%), since usually the activated carbon used for sorbent injection has a very high surface area (~600 m<sup>2</sup>/g).<sup>5</sup> This phenomenon can be seen clearly by comparing the surface area values between samples 1A and 1B or samples 2A and 2B. As discussed before, the major difference between 1A and 1B (2A and 2B) is the carbon content, therefore we conclude that the surface area difference between these samples is likely due to the activated carbon used in the sorbent injection technology.

Particle morphology: SEM was used to characterize the morphology of the different fly ash powders. Figure 2 shows SEM images recorded at three successive magnifications for each fly ash sample type. Figure 2 shows that the bulk of the particles are spherical in shape. The particle size varied from <1 μm to 100 μm in diameter. This data is consistent with the data reported in the literature. The highest magnification SEM images shows that the surface of the EERC, 1A, and 1B particles are smoother than that of the particles in samples 2A and 2B.

---

<sup>4</sup> National Risk Management, Research and Development: Control of mercury emission from coal-fired electric utility boilers: interim report, EPA-600/R-01-109, April 2002

<sup>5</sup> Technical datasheet for Norit Darco FGD activated carbon from Norit Americas Inc.



**Figure 2. SEM images for (A) EERC, (B) 1A, (C) 1B, (D) 2A, and (E) 2B fly ash samples (the magnification increases from left to right.)**

**Table IV. Surface area and particle size of fly ash samples**

	<b>EERC</b>	<b>1A</b>	<b>1B</b>	<b>2A</b>	<b>2B</b>
<b>Specific surface area (m<sup>2</sup>/g)</b>	14.64	4.52	10.66	5.49	7.74
<b>Mean particle size (μm)</b>	9.40	24.43	18.41	15.12	17.89
<b>D10 (μm)</b>	0.34	0.54	0.48	0.42	0.43
<b>D50 (μm)</b>	3.49	11.27	7.97	6.46	7.70
<b>D90 (μm)</b>	22.94	66.36	52.20	41.91	50.38

Crystalline phases: The crystalline phases of each fly ash sample were identified by XRD. For the EERC fly ash sample the major crystalline phases were identified as quartz (SiO<sub>2</sub>, peaks labeled Q) and mullite (3Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>, peaks labeled M). The large broad peak located between 20 and 35 degrees indicates that there is a significant amount of an amorphous phase present in the sample as well. There were no apparent peaks related to the Ca containing phases; therefore, Ca may exist inside the amorphous phase. For the fly ash samples 1A and 1B the *major* crystalline phases were quartz and mullite, and the *minor* crystalline phases were lime (CaO, peaks labeled L), calcium sulfate (CaSO<sub>4</sub>, labeled as CS), gypsum (CaSO<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>, labeled as G), magnetite (Fe<sub>3</sub>O<sub>4</sub>, labeled as F), and sodium calcium silicate (Na<sub>2</sub>CaSiO<sub>4</sub>, labeled as SCS). From this data we conclude that the extra Ca content observed in the 1A and 1B samples, relative to the EERC fly ash, forms the lime and calcium sulfate related phases observed. For fly ash samples 2A and 2B, the amounts of calcium sulfate (CaSO<sub>4</sub>), gypsum (CaSO<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>) and calcium sulfate hydrate (CaSO<sub>3</sub>(H<sub>2</sub>O)<sub>0.5</sub>, labeled as CSH) were significantly increased, and were much larger on a relative wt% basis than those in the group “1” samples and the EERC sample. This observation is not surprising based on the large amounts of Ca and S compositions observed for the group “2” samples identified by XRF shown in Table 1. Because the group 2 samples consist of fly ash, gypsum, calcium sulfate hydrate, and calcium sulfate in high concentrations it was decided that these samples were out of the scope of the proposed plan.

To summarize, sample 1A is a typical fly ash sample generated by “current” power plants that burn sub-bituminous coal. Whereas samples EERC and 1B are typical fly ash samples generated by “future” power plants that adopt sorbent injection technology. Samples 2A and 2B are actually mixtures of fly ash, gypsum, calcium sulfate hydrate, and calcium sulfate, which were determined to be out of the scope of this research project. Consequently, the rest of this study will focus on EERC, 1A, and 1B samples only.

## Compressive Strength Data on Experimental Matrix

The compressive strength results for various experimental matrices are shown below.

**Table V: Compressive strength data on DOE: DOE #1**

Trial #	Compressive strength 1 (MPa)	Compressive strength 2 (MPa)	Compressive strength 3 (MPa)	Ave. strength (MPa)	Std. Dev. (MPa)
1	26.92	23.88	21.59	24.13	2.67
2	27.59	23.23	21.41	24.08	3.18
3	22.08	21.28	20.52	21.29	0.78
4	27.25	26.77	24.01	26.01	1.75
5	33.22	21.16	20.00	24.79	7.32
<b>6</b>	<b>48.00</b>	<b>32.62</b>	<b>32.22</b>	<b>37.61</b>	<b>9.00</b>
7	20.23	18.44	16.49	18.39	1.87
8	26.93	26.57	26.02	26.51	0.46

Based on the first round of testing, the DOE was able to help highlight the optimum values for the respective variables. Following this round, a second set of fly ash samples were tested. These were two additional fly ash samples – a sample from the EERC and one obtained from ADA-ES (the power plant names are unknown to us). These two additional fly ash samples have different levels of activated carbon: EERC = ~3.4% LOI, and ADA-1B = ~2.5% LOI. These samples (referred hereafter as “EERC 12/05” and “ADA-1B” respectively) were also tested using the Taguchi DOE method and their results are shown in Table VI below.

**Table VI. Compression Test Results for DOE #2 (EERC 12/05; 3.4% LOI) and DOE #3 (ADA-1B; 2.5% LOI)**

DOE #2 EERC 12/05		DOE #3 ADA-1B			
Exp #	Compressive Strength	Std. Dev.	Exp #	Compressive Strength	Std. Dev.
1	2.3	0.5	1	2.1	0.3
2	126.3	38.3	2	6.5	0.3
3	223.4	132.6	3	59.0	17.5
4	102.9	39.4	4	86.3	13.3
5	4.8	0.4	5	9.3	0.6
6	34.5	2.4	6	30.6	2.4
7	87.1	22.7	7	22.9	1.3
8	3.3	0.5	8	29.1	2.7
9	4.7	0.7	9	12.0	1.0

It is clear that reasonably high strength can be achieved using our proposed process. The above results are comparable to ultra-high strength concrete. By removing the entrapped air bubbles, it will be possible to dramatically reduce the standard deviation.

### Foam Index Test of Treated Fly Ash Samples

In addition to the leaching tests, the results of which essentially show the encapsulation effect of the treatment process for mercury, we investigated the encapsulation effect of the activated carbon. One of the critical reasons that fly ash containing activated carbon is not suitable for concrete applications is because the activated carbon adsorbs the AEA. The AEA is added during concrete mixing to improve the freeze/thaw resistance of the concrete, and is of critical importance to the industry.

The foam index test was designed to check whether this adsorption effect is within an acceptable limit or not. Although it is an important field test for QC of fly ash, it is conducted differently from one power plant to another. A typical procedure is described below.

1. Place 4g of fly ash and 16g portland cement into a 4-ounce jar, and hand-shake to mix them thoroughly
2. Add 50 mL distilled water and hand-shake for 1 min to mix the water and powder well
3. Add 6 drops (~0.2 mL) of diluted AEA solution drop wise into the water/fly ash mixture (the AEA was Darex II from W. R. Grace, and it was diluted with distilled water 1:20)
4. Shake the jar vigorously for 15 seconds, lay it on the table, wait for 5 seconds, and then watch for any bubble breaking for another 15 seconds.
5. If any bubble breaks occur during the 15-second period, then repeat step 3 and 4 till no bubble breaks are observed
6. Record the total number of drops of AEA used to form a stable film, this value is the Foam Index (FI) value for the tested fly ash

In order to quantify the encapsulation effect of our process on the activated carbon, foam index tests were conducted on both un-reacted fly ash powders and treated fly ash samples. The treated fly ash samples were milled into powder form after curing for 7 days at the desired temperature. Table VI shows the results of the foam index tests recorded for the un-reacted fly ash powders and the treated fly ash samples. For the *un-reacted* powders, samples 1B and EERC required significantly more AEA (180 and 500 drops, respectively) when compared to sample 1A (48 drops). These results are consistent with expectations in as much as these samples contained excess activated carbon from the sorbent injection. However, after treatment all three samples required only a small amount of AEA (~12 - 15 drops) to form a stable foam layer, and there was a negligible difference between these three samples, regardless of the activated carbon content.

**Table VI. Foam index test results for un-reacted and treated fly ash samples**

Ash type	Original powder # of Drops of AEA	Treated Sample # of Drops of AEA
<b>1A</b> (no excess C content)	48 drops	12 drops
<b>1B</b> (with excess C content)	180 drops	15 drops
<b>EERC</b> (with excess C content)	500 drops	12 drops

A second set of foam index experiments were conducted with the same two fly ash samples described above – EERC 12/05 and ADA-1B in order to generate more statistics. Nine experimental variations of the treatment process were performed. These results are shown below.

**Table VII. Foam Index Results for DOE #2 (EERC 12/05)**

Exp #	Foam Index Test (as received, baseline = 500 drops)				
	Test 1	Test 2	Test 3	Foam Avg.	Foam Std Dev
1	55	59	53	56	3
2	45	41	35	40	5
3	62	63	58	61	3
4	81	75	70	75	6
5	67	60	58	62	5
6	45	48	47	47	2
7	60	60	69	63	5
8	48	45	45	46	2
9	31	30	32	31	1

**Table VIII. Foam Index Results for DOE #3 (ADA-1B)**

Exp #	Foam Index Test (as received, baseline = 180 drops)				
	Test 1	Test 2	Test 3	Foam Avg.	Foam Std Dev
1	29	30	27	29	2
2	15	18	18	17	2
3	56	59	61	59	3
4	28	31	30	30	2
5	13	12	12	12	1
6	13	13	12	13	1
7	17	16	15	16	1
8	24	23	21	23	2
9	9	8	10	9	1

*DOE #2 Foam Index Results:* Each sample was tested in triplicate to determine an average and standard deviation for the sample foam index. Each sample showed roughly a 10-fold decrease in the foam index results when compared to the baseline sample (EERC = ~500 drops). The best

sample only required an average of 31 drops to pass the foam index test, whereas the worst sample required 75 drops. All of these results are very encouraging, and demonstrate that despite the high activated carbon content in the fly ash, the treatment mitigates the effects of the C on the AEA for use in concrete.

*DOE #3 Foam Index Results:* For this data set, the worst performing sample, by foam index standards was sample #3, which had an average foam index score of 59. On the other hand sample #9 performed well having a foam index score of 9. All samples showed a significant reduction in the required amount of drops required to pass the foam index test.

Figure 3 below plots the foam index results for various types of fly ash as a function of the measured LOI and surface area. It is interesting that the foam index has a stronger correlation with surface area, compared to LOI. This is expected because surface area is more dramatically affected by the amount and type of carbon (two different carbon sources can have the same LOI but completely different surface area). The greater the surface area (more carbon) the greater is the foam index.

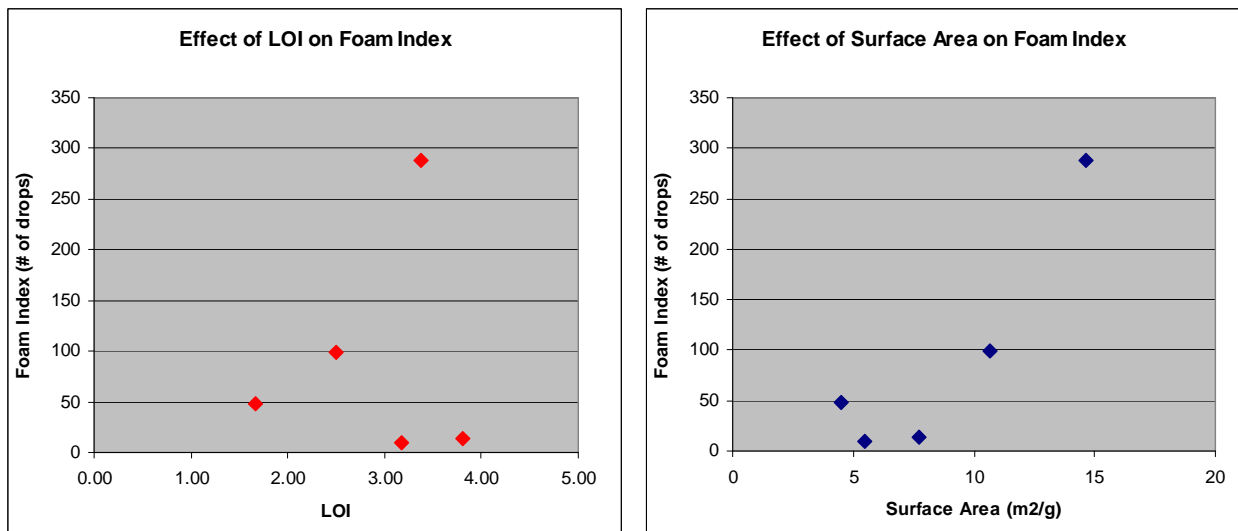


Figure 3: Foam Index results plotted against LOI and surface area

### Mercury Leaching Tests of Treated Fly Ash Samples

Six unique samples were sent to Rutgers University to perform the three unique mercury leaching tests. Table IX shows a list of the samples that were sent to Rutgers for testing. These samples were tested for leachable amounts of mercury. Three leaching test methods were employed to simulate the leaching process; the tests were 1) Toxicity Characteristic Leaching Procedure (TCLP), 2) Synthetic Precipitation Leaching Procedure (SPLP), and 3) Synthetic Groundwater Leaching Procedure (SGLP). These three test procedures are very similar; however, the primary difference between the three tests is in the leaching media used. The acetic acid used in the TCLP test is to simulate the major component found in leachate at any typical municipal waste landfill. The leaching media used in SPLP is to simulate acid rain, i.e., a mixture of sulfuric acid and nitric acid. The leaching media used in SGLP is deionized water. The SGLP was designed primarily for use with materials, such as low-rank coal ash, that undergo hydration reactions upon contact with water.

**Table IX. Description of Samples Sent to Rutgers for Mercury Leaching Tests (A1 and A2 are the two different activator types tried in this test)**

Sample Name	Fly Ash Sample	Description
Baseline EERC	EERC	Non-treated baseline sample
EERC-A1-RT	EERC	Treated with A1 and cured at RT
EERC-A2-75	EERC	Treated with A2 and cured at 75°C
Baseline 1B	1B	Non-treated baseline sample
1B-A1-RT	1B	Treated with A1 and cured at RT
1B-A2-75	1B	Treated with A2 and cured at 75°C

The highest leachate concentration for the TCLP test among the six types of solid samples was 0.00010 mg/L, which was significantly less than the TCLP regulatory level of 0.2 mg/L. The highest leachate concentration obtained for the SPLP test was 0.00033 mg/L, which was much lower than the national primary drinking water limit for mercury of 0.002 mg/L. Finally, the highest SGLP leachate concentration was 0.00039 mg/L. We can conclude that these six types of solid samples have much lower mercury concentration as compared to the regulatory levels (0.2 mg/L).

## Conclusions

Different types of treated fly ash samples were tested for leachable amounts of mercury, foam index, and compressive strength. The primary variables were presence of alkali and test methods.

Three leaching test methods, namely: Toxicity Characteristic Leaching Procedure (TCLP), Synthetic Precipitation Leaching Procedure (SPLP), and Synthetic Groundwater Leaching Procedure (SGLP), were employed to simulate the leaching process. The highest leachate concentration for the TCLP test among the six types of solid samples is 0.00010 mg/L, which is greatly less than the TCLP regulatory level of 0.2 mg/L. The highest leachate concentration for SPLP test is 0.00033 mg/L, which is much lower than the national primary drinking water limit for mercury of 0.002 mg/L. Finally, the highest SGLP leachate concentration is 0.00039 mg/L. We can conclude that these six types of solid samples have much lower mercury concentration as compared to the regulatory levels.

Further, treatment of high carbon fly ash can be very effective in neutralizing the effect of high carbon content. By a proprietary treatment process, high compressive strength (> 100 MPa, 14,500 psi) samples were successfully produced. A concomitant improvement in foam index test was also observed (where the number of drops required was reduced from 500 to 37 for a heavily loaded activated carbon containing fly ash. Other samples were reduced from 180 drops down to 9 drops for an activated carbon containing fly ash). It was also proposed that surface area (m<sup>2</sup>/g) may be a better predictor of foam index drops than % LOI. Further testing must be performed to verify this observation.

## Acknowledgements

The authors would like to thank the Department of Energy (contract # DE-FG02-05ER84197) for their funding of this research through the SBIR program. The authors would also like to thank their Ceramatec colleagues for their continued support for this project.