Concretes and Fly Ashes from a Full-Scale, Concrete-Friendly™ C-PAC™ Mercury Control Trial

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Abstract

If power plants across the country adopt traditional powdered activated carbon injection to meet state and federal mercury reduction requirements, vast volumes of quality fly ashes will no longer be able to be sold as a replacement for cement in concretes. Sorbent Technologies has developed a brominated carbon-based mercury sorbent, C-PAC™, which adsorbs very little of the concrete air-entraining admixture (AEA) additives that are the root of the problem. C-PAC™ was recently tested at full-scale at Midwest Generation's Crawford Station in Chicago in a month-long trial. Injected before the plant's undersized ESP at about 4.5 lb/MMacf, it averaged over 80% mercury removal in the long-term testing. The Crawford Station burns a common Powder River Basin coal and sells much of its fly ash as a cement substitute.

This paper presents the results of an examination of the fly ashes from this C-PAC™ trial and resulting concretes made from them. Three areas are discussed:
(1) characterization of the resulting fly ashes from both the front and back hoppers, including their Hg concentrations, carbon contents, LOIs, and particle sizes;
(2) measurement of the adsorption of AEAs by these fly ashes using standardized foam index methods; and (3) the evaluation of the properties of concretes made using these fly ashes, including their air content, air stability, setting time, and compressive strength. Measurements are reported by Sorbent Technologies, as well as two of the largest ash marketing companies in the U.S., Headwaters Resources and Lafarge North America.

1. Introduction

The most important use of fly ash is as a replacement for cement in concrete. Commonly, cement in a concrete mix is replaced by fly ash about 20% by weight [1]. Fly ash enhances the workability, durability, and ultimate strength of concrete. Additionally, substituting one ton of fly ash for Portland cement eliminates about one ton of CO_2

emissions [2]. In 2005, about 71 million tons of fly ash were produced by U.S. coal-fired power plants, of this 41% was utilized, and half of the fly ash used (~15 million tons) replaced cement in concrete [3].

When the Clean Air Mercury Rule (CAMR) is implemented nationwide in 2010 and when state rules in Illinois and elsewhere are implemented earlier, the current utilization levels of fly ash could be difficult to sustain. Duct injection of powdered activated carbon (PAC) based sorbents is the leading technology for power plant mercury emission control. However, the injection of standard PAC sorbent results in a fly ash that can no longer be used in concrete. This is because the standard PAC strongly adsorbs the air-entraining admixture chemicals (AEA) which are added to the concrete slurry to create the air bubbles needed to enhance the freezing-thaw capability of the concrete. The cost of mercury emission control can be two to six times more for plants that can no longer be selling their fly ash for concrete applications [4].

To solve this problem, Sorbent Technologies has developed a new activated-carbon-based mercury sorbent, C-PAC™, that is “concrete-friendly.” This particular mercury sorbent effectively removes mercury from the flue gas, while having minimal adsorption of AEA.

This paper describes full-scale test results performed with C-PAC™ at Midwest Generation’s Crawford power station. It focuses on the characterizations of fly ashes containing the C-PAC™ created during the full-scale mercury control trials and the properties of concretes created with these fly ashes.

2. Experimental Design

2.1 Materials

Concrete-friendly PAC (C-PAC™) mercury sorbent is a brominated powdered activated carbon made by a patented process. It has a BET specific surface area of about 570 m^2/g and a median particle size of about 17 nm.

The concrete materials used in the laboratories of Sorbent Technologies and Headwaters Resources to make test concretes included Portland cement (type I), gravel, sand, and Crawford fly ash with and without C-PAC™. The formula and raw materials used by the two laboratories were different. The ratios of water to cement, W/C, for the 100% Portland cement (No Ash) were 0.58 and 0.52 for the Headwaters and Sorbent Technologies’ concretes, respectively. For the 20 wt% fly ash control (Ash) and the C-PAC-fly ash (C-PAC) concretes which require less water, 0.56 and 0.50 W/C were used for each by the two laboratories. Twenty percent of the cement was replaced by fly ash in ash control and C-PAC concretes for both laboratories. Four commercially-available AEAs were used for foam index measurements and concretes mixes.

2.2 Full-scale test of C-PAC™ sorbent for mercury control

The full-scale C-PAC™ injection for mercury control at the Crawford power plant was conducted in three distinct phases: (1) Baseline testing (three weeks) with continuous mercury emission monitoring while taking fly ash and coal samples; (2) Parametric testing (one week) to determine the sorbent injection rate and operation conditions for the long-term run; and (3) Long-term testing (one-month) to examine the sorbent performance, fly ash effects on concrete utilization, and any balance-of-plant effects during a continuous sorbent injection run with the normal boiler operation. The C-PAC™ sorbent was injected into the flue gas before the cold-side electric precipitator (ESP) where it captured the mercury and was collected by the ESP mixing with the fly ash (Figure 1). At Crawford during the long-term test an average of 81% of gas phase mercury was removed by injecting an average of about 4.5 lb/MMacf of sorbent. Crawford Unit 7, which burns 100% Powder River Basin (PRB) subbituminous coal, has historically sold some of its fly ash for cement substitution.

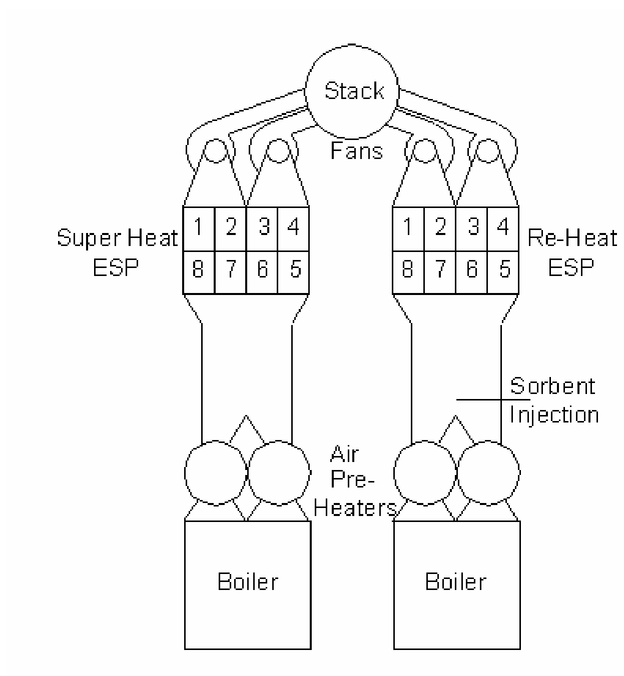

Figure 1. Crawford Unit 7 configuration. Sorbent was injected in the Re-Heat side. Hoppers 5 to 8 are the four front hoppers; 1 to 4 are the back four.

2.3 Fly ash sampling

Baseline fly ash taken before sorbent injection started and long-term testing fly ash taken during the mercury control trial at Crawford Unit 7 were collected using vacuum and thief methods. The testing side ESP (Re-Heat side) has two rows of hoppers, each row with four hoppers (Figure 1). Every day one set of eight samples from each hopper was taken during the three weeks of baseline monitoring. Two sets, one for low load, another for high load, were taken every day during the one-month of full-scale sorbent injection. The mercury concentrations and LOI of each of those samples were analyzed. The front composite samples were a mixture with an equal weight of each of the four front hoppers' samples; the same was done for the back row composite samples. These composite samples were tested for Hg, LOI, carbon, particle size, and foam index. A total of about 600 individual hopper fly ash and 120 composite ash samples were produced. About 200 lbs of front hopper fly ash and 60 lbs of back composite fly ash for concrete testing were collected using the vacuum method during both the baseline and the long-term test. The fly ash used in concrete was made by blending 80% of front ash with 20% of back ash.

2.4 Procedures and methods

The mercury in the fly ash was analyzed using the thermal combustion CVAA method with an Ohio Lumex-RP-915 analyzer. LOI measurement of the fly ash followed the ASTM procedure. Carbon in fly ash was measured with a Leco SC444DR Carbon/Sulfur analyzer. Particle size distribution was determined by Horiba LA 920 laser scattering particle analyser. Foam index, measured by three different laboratories, followed the standard procedure of each laboratory, with the principle being the same: a water suspension of fly ash or the mixture of fly ash with cement was titrated with an AEA solution. At the end point, a layer of foam was formed and stabilized on the surface. The added amount of AEA at the end point determined the foam index (FI). The specific foam index (SFI) of the sorbent was calculated using the difference between samples containing sorbent and the baseline samples and dividing the difference by the sorbent mass in the sample.

Three types of concretes were made in the laboratories following the ASTM C192 procedure [6]. Concrete specimens for unconfined compressive strength (UCS) and petrography testing were cured under the C192 instructions. The air content of fresh concrete was measured using the ASTM pressure method [7]. For air stability testing, the fresh concrete was initially made the same as ASTM C192. After being made, slump and air were measured using time, based on ~30 minute intervals, in static and dynamic states. The static state reflects ready-mix fresh concrete on-site without continuous mixing, while in the dynamic condition, a ready-mix fresh concrete was made with continuously mixing or agitation. UCS was measured following the ASTM C39 procedure [8]. The penetration resistance method [9] was applied to determine the time of setting of the concrete.

3. Characterization of baseline and long-term fly ash

3.1 Mercury, LOI, and carbon captured in fly ash

The high mercury levels measured in the fly ash samples indicated that the C-PAC™ captured mercury from flue gas very efficiently (Figures 2 & 3). The mercury in the long-term fly ash was ten times higher than that of the baseline ashes. Baseline fly ash samples include very low Hg and LOI (Figure 3).

The mercury concentrations from each hopper in the front row were very close. The thirty-day average Hg and distribution in front hopper 5, 6, 7, and 8 were the same. A lower amount of Hg was found in hoppers 4 and 2 than in 3 and 1, which were in the back (Figure 3). A higher amount of LOI was observed in center hoppers 6 and 7, and 3 and 2, than side hoppers 5 and 8, and 4 and 1. This implies that more C-PAC™ was collected by the center part of the ESP than the sides. The sorbent distribution in the duct upon entering the ESP may not have been uniform.

The sorbent injection process effectively transfers mercury from the flue gas to the solid phase (sorbent-fly ash). Measuring the mercury in fly ash can evaluate the performance of the sorbent. If the mercury and ash in the coal, and the mercury in the ash streams can be determined accurately, a mass balance of mercury can be calculated. An average of 89% of coal mercury was captured in sorbent-fly ash during the long-term test, based on a calculation using the following data: 90 ppb Hg and 6.49% ash in the coal, an average 1550 ppb Hg in the fly ash, and 20% of coal ash going to bottom ash without Hg. This 89% Hg removal is higher than the 81% determined by gas phase measurement.

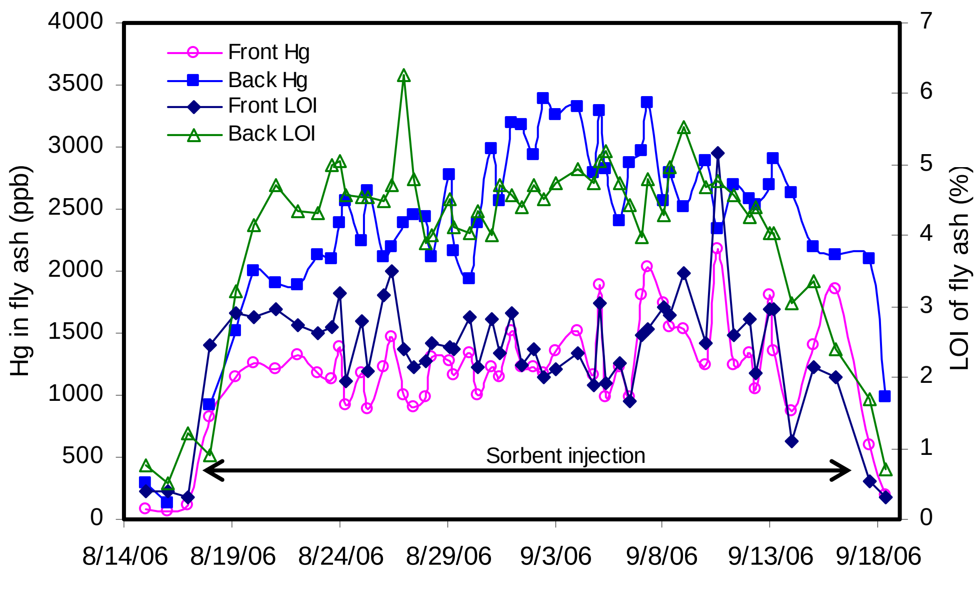

Figure 2. Hg and LOI of composite front and back fly ashes during the long-term trial. From 8/17 to 9/7/2006 injected average 4.5 lb/MMacf C-PAC. 9/7 to 9/16 had injected PAC and B-PAC alternated 2, 4, and 6 lb/MMacf.

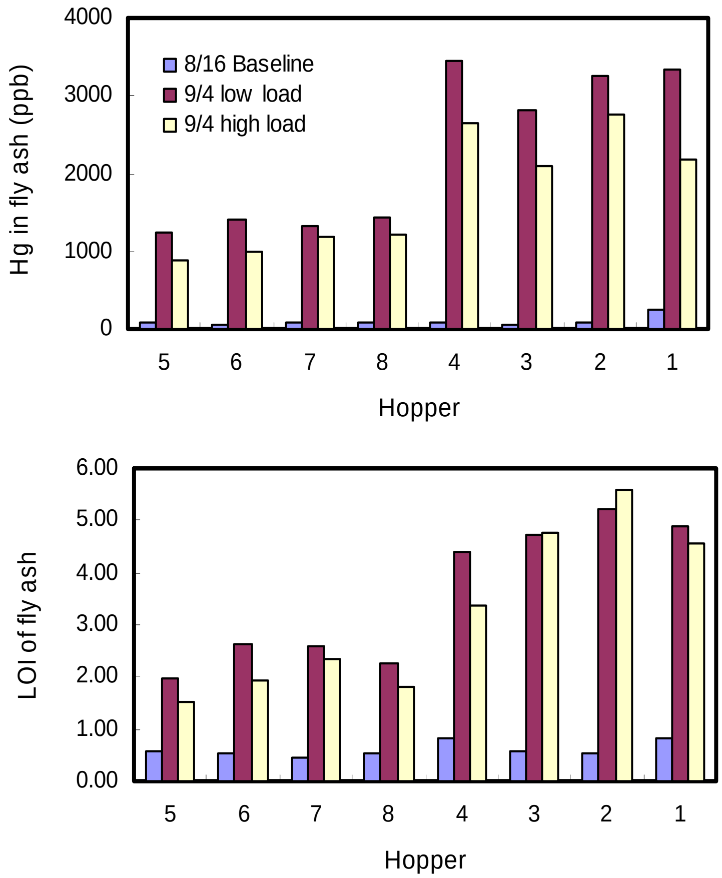

Figure 3. Mercury and LOI distributed in hoppers. Long-term testing on 9/4. 8/16 baseline fly ash was taken at low load. 5 to 8 are front hoppers; 4 to 1 are the rear hoppers.

3.2 Particle size of the fly ash

The fineness of the fly ash is important in cement replacement because the particle size influences the pozzolanic and cementitious properties of fly ash. The pozzolanic activity of fly ash is proportional to the amount of particles under 10 µm, whereas particles larger than 45 µm show little pozzolanic activity [3]. A good quality fly ash concrete utilization is characterized by low carbon and high glass content, with 75% or more of the particles finer than 45 µm. The Crawford fly ash had more than 90% of particles less than 45 µm (Figure 4). The fine particles and low unburned carbon (UBC) make Crawford fly ash an excellent cement material.

The median size of the front and back hopper fly ashes during the long-term testing were 7.46 and 3.51 µm, respectively. All front and back fly ash samples exhibited a bi-modal log size distribution pattern. The bi-modal distribution probably reflects the ESP performance as a function of particle size rather than the actual distribution of the particles. The low frequency of ~ 1 µm particles is likely the result of inefficient ESP collection for this size of particles. Particles of ~1.0 µm are too fine to be removed by electrostatic forces and too large to be removed by Brownian movement. In contrast, a

17 µm median size for C-PAC™ is greater than that of most of the fly ash. The mercury sorbent has a single-peak log distribution. It appeared that more of the finer particles were collected during long-term C-PAC™ injection than in the baseline tests at the same load. This is corroborated by lower opacity readings noted during the sorbent injection tests.

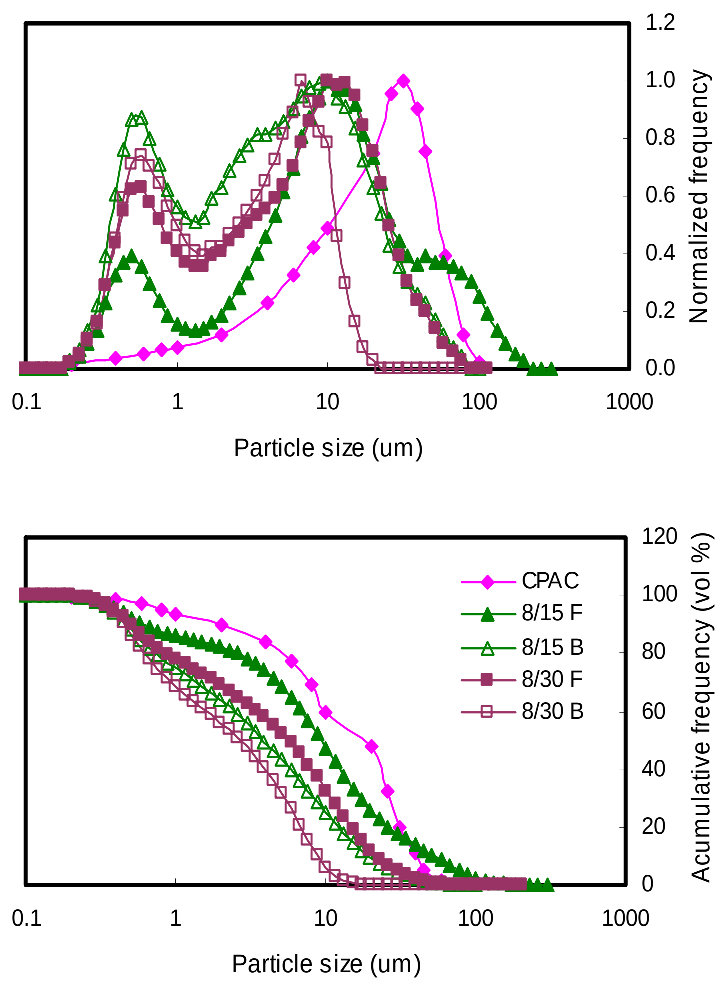

Figure 4. Particle size distributions of C-PAC™ and fly ash measurements by Headwaters.

3.3 Correlation of Hg, LOI, carbon, particle size of fly ash

Mercury, carbon, and LOI values are related to the carbon injection rates, the mercury in the coal, the mercury in the flue gas, and the boiler operation. Mostly they are proportional to the injected mercury sorbent level. Carbon and LOI values display a linear relationship (Figure 5). The UBC in this fly ash was very low. Consequently, the carbon concentration can be used to represent the amount of sorbent injected. In this paper, however, LOI was used as a substitute for carbon as the reference (X-axis in Figure 6) because: (1) the regression of carbon and LOI in fly ash is linear, (2) LOI is more easily determined, and (3) LOI data of all individual hopper fly ash samples was obtained. Mercury in fly ash clearly increased with LOI and carbon content, but the mercury per unit of LOI or carbon is almost identical for front and back fly ashes. Particle size appears to be decreasing as LOI increases (Figure 5).

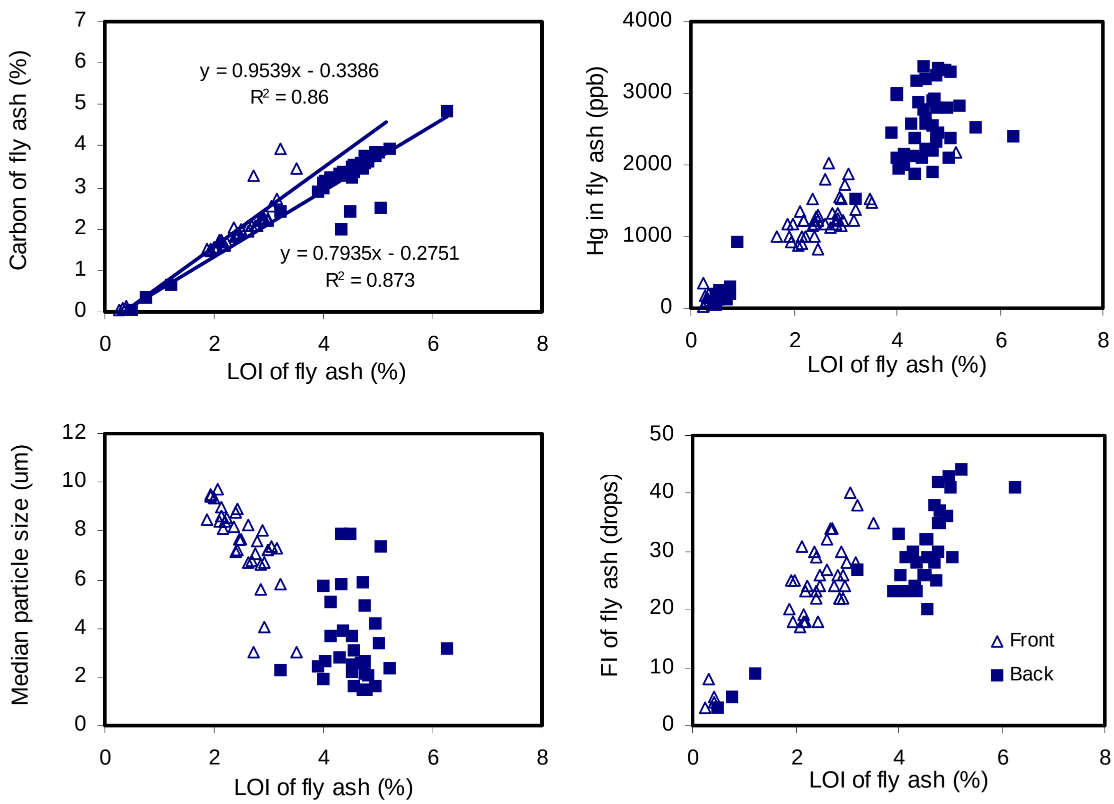

Figure 5. Correlations of mercury, carbon, LOI, particle size, and MB-VR FI of fly ash. All samples are composite.

4. Foam indexes of fly ashes

The foam index (FI) is a routine method used by the industry to quantify the relative capacity of a fly ash to adsorb AEAs. Because there are no standard FI methods, each organization uses their own standard to determine the suitability of a particular power plant's fly ash. According to the Crawford ash utilization contract, using Lafarge's method and the AEA Vinsol, if the foam index is less than 40 drops, Crawford's fly ash is acceptable for cement replacement in premium concrete. If the value of FI is greater than 40 but less than 100, it still can be used in standard concrete. Based on this contract specification, the front fly ash of Crawford including C-PAC still can be used in the premium concrete, while the back ash only can be used in standard concrete. All three AEAs (Darex II data from Sorbent Technologies, Vinsol from Sorbent Technologies and Lafarge, and MB-VR from Headwaters) showed a higher FI in back ash than in front ash. This may be due to the higher carbon or LOI of the back ash than the front (Figure 2&3). The FI of both front and back ash increased with LOI or carbon (Figure 5), but the FI per unit of LOI or carbon was found to be higher in front hoppers than in the back.

The 40-drop cut off for premium concrete is a somewhat arbitrary value which is based on the specific form index (SFI) of the unburned carbon (UBC). It is the SFI of the carbon, its AEA adsorption per unit of carbon (drops/gram or ml/gram), combined with the expected degree of variation in the number of carbon units per ash unit, that determines what is acceptable. There will be natural process variations in the amount of unburned carbon (or amount of PAC) in each truck load of fly ash going to the ready-mix plant. These plants generally add a constant amount of AEA per batch—they cannot statistically sample each ash shipment. If the UBC or PAC has a high SFI, then small variations will lead to large differences in the effective amount of unadsorbed AEA available to create bubbles. With too little unadsorbed AEA, there is not enough void space and the concrete will crack upon freezing. With less than expected carbon and too many bubbles, the concrete will have insufficient strength. Because the SFI of C-PAC is only a fraction of that of UBC (Table 1), variations in its incorporation rate in the fly ash result in little variation in air entrained. In fact, it can have a dampening effect on UBC-caused air entrainment variations.

Table 1. The average specific foam index of UBC of ash control and C-PAC

AEA	Specific Foam Index (SFI)		
	Ash UBC	C-PAC	Ratio of UBC to C-PAC
Darex II	3.6 ml/g	0.68 ml/g	5.3
Vinsol	5714 drops/g	1682 drops/g	3.4
MB-VR	3644 drops/g	949 drops/g	3.8

While the value of the FI is an important parameter, the variation of FI is even more important. For example, the long-term fly ash, including an average of ~2.5 wt% of C-PAC™, had an average FI of 44 drops using Vinsol. This can still be a good cement substitute, because the FI did not vary more than the baseline (Figure 6C). The standard deviation (SD) of the C-PAC ash was only 4 drops, while the SD of a month of baseline control ash was 5 drops (Figure 6C).

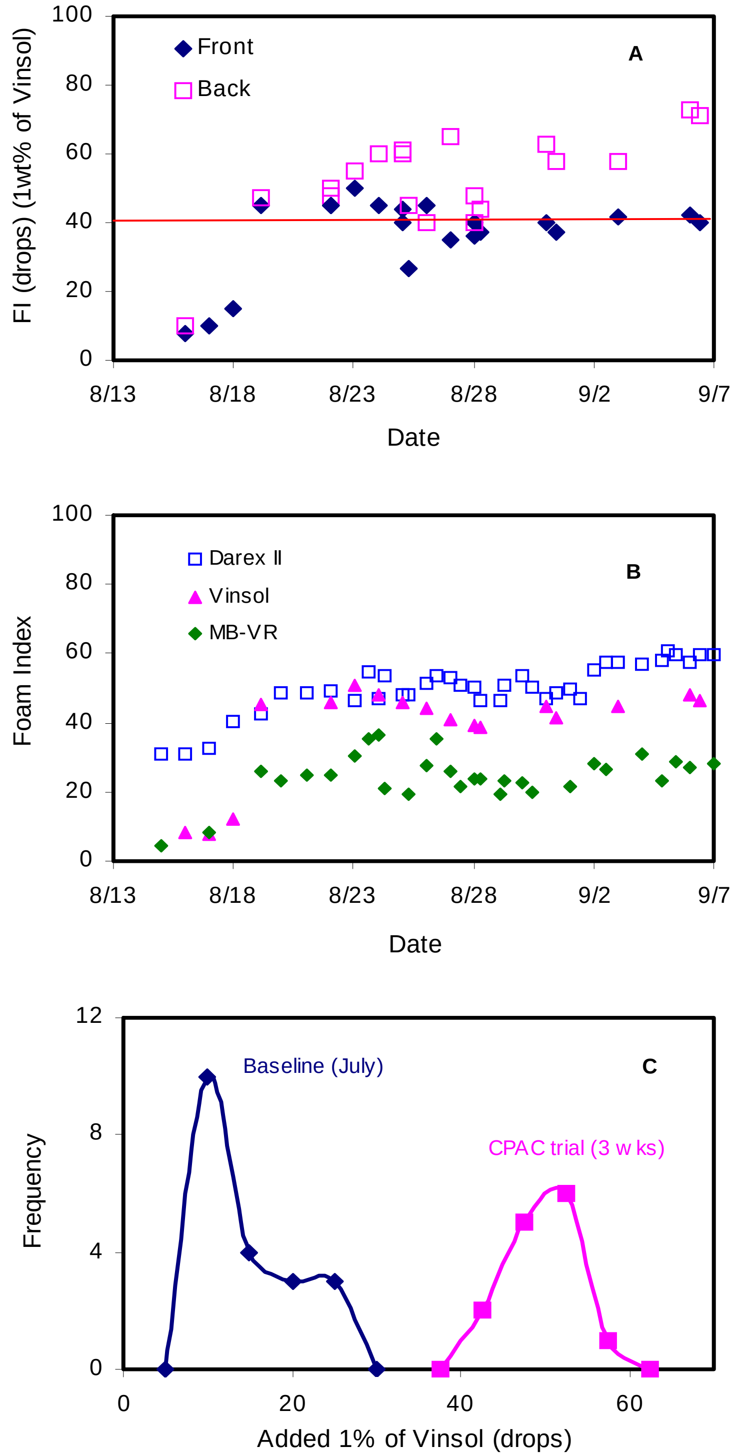

Figure 6. Foam index of composite fly ash samples for the first three weeks of full-scale C-PAC testing.
A: Vinsol FI of fly ash from front and back hoppers, from Lafarge and Sorbent Technologies.
B: Calculated foam indexes based on 80% of front composite plus 20% of back composite data. Darex II (Sorbent Technologies), Vinsol (Lafarge & Sorbent Technologies), and MB-VR (Headwaters). C: Foam index distribution of baseline and long-term fly ash. The average ± SD for the baseline and long-term ash were 14 ± 5 and 45 ± 4 drops, respectively.

5. Properties of concrete

Although strong adsorption of AEAs is the major issue for PAC-based mercury sorbents, the other properties of concrete that contain C-PAC™ fly ash also have to be similar to those without C-PAC™. The most important of these concrete properties are air content and bubble stability, slump, setting of time, and the strength of hardened concrete. Concrete slump is a measure of the material's consistency. A 6±1 inch slump test was performed on all batches of concretes. Maintaining a constant slump eliminates the complication of slump effects in the interpretation of the air-entrained data.

5.1 Air content of fresh concrete

The most important factor for evaluating whether C-PAC™-fly ash can be used in concrete is the relative sensitivity of air entrainment with a constant AEA dose routine to variations in the fly ash. Foam index is a quick method of obtaining the relative AEA adsorption of fly ash or sorbents, but it cannot provide an absolute value for the amount of AEA that has to be added to the concrete. However, the AEA needed to reach the target air and slump values is proportional to the FI (Figure 7A, Table 2). The specific foam index, adsorbed AEA per gram of sorbent, is much higher than that calculated based on dosage of AEA added to concrete to reach the target air volume. Cations like Ca^{2+} and Mg^{2+} dissolve from the ash into the FI testing solution and can precipitate out some AEAs [10]. In addition, the forming of a stable layer of foam on the container surface seems to need more AEA than in regular concrete to form 6% air voids. The air content of fresh concrete had a linear relationship with the added AEA in concrete (Figure 7). A demand dosage of AEA can be easily calculated based on this linear relation. Table 2 summarizes the FI, slump, air, and AEA dosage of No Ash, Ash, C-PAC, and PAC concretes (the "PAC" sample had an equal weight of regular PAC mixed with baseline fly ash) made by Sorbent Technologies. The dosage of AEA in the table was used for concretes made for UCS and air stabilization tests.

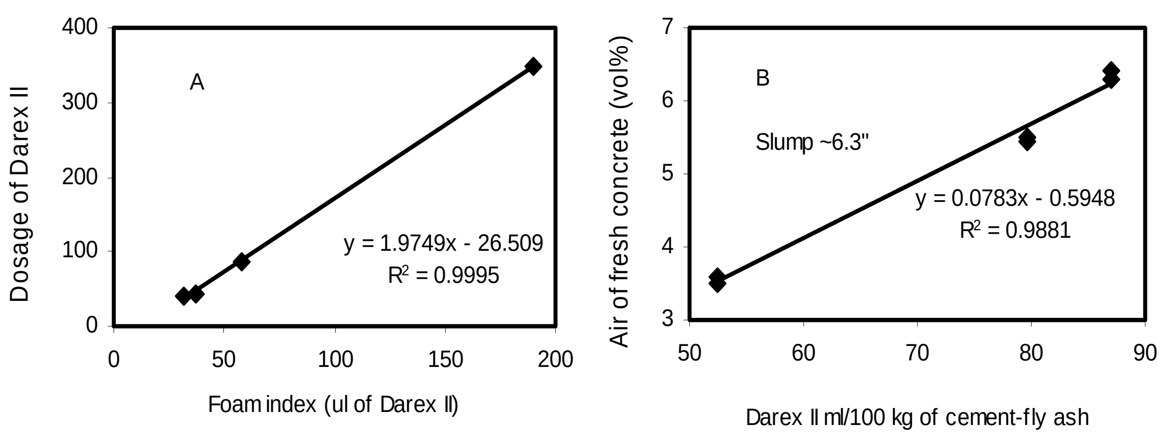

Figure 7 (A) AEA used in concrete vs. FI. (B) Air content vs. AEA added in concrete with C-PAC-fly ash.

Table 2. Air properties of concretes (Sorbent Technologies data).

Concrete	AEA (ml/100 kg Cement-ash)*	Slump (inch)	Air (vol%)	FI (µl)
No Ash	41	6.0	6.7	32
Ash	43	6.1	6.0	37
C-PAC™	87	6.3	6.4	58
PAC**	349	6.0	5.5	190

* A dosage of 30 to 320 ml/100 kg of cement material is mentioned in the MSDS of the AEA.
** An equal weight of brominated PAC (Norit Darco Hg-LH) mixing with baseline fly ash. An extra 40 ml/100 kg of Darex II was required to increase air to 6%.

In comparison to the ash control, to make a 6 vol% of air in concrete about double the amount of AEA was required, while approximately ten times the amount was needed for a standard PAC (Norit Darco Hg-LH). Even though more AEA was used in C-PAC concrete than in the control to achieve the same air, the dosage still satisfies contractor specifications, while the standard PAC ash was out of the range. Headwaters also indicated that 2 to 2.5 times more AEA Micro-Air™ AEA was needed for C-PAC concrete to achieve the same air as the control samples. The amount of extra AEA needed when using ash varies with the LOI of the fly ash (baseline). Up to 5 times as much may be needed [11]. It is only after the carbon in the fly ash is saturated by AEA that the added reagent begins to function as intended. The BET surface area of the C-PAC™ sorbent, ~570 m^2/g, is essentially the same as the commercial PAC sorbent. The lower AEA adsorption of C-PAC™ is related to its special properties rather than its surface area.

5.2 Air stabilization

As discussed above, the same air content (6% vol) can be entrained in concretes with C-PAC containing fly ash as with the control ash concrete. However, it is necessary not only to achieve an equivalent initial air amount, but to also keep the air bubbles stable. In many cases, when large amounts of AEA are added to obtain the initial air content, the air content may not be stable over time. Figure 8 indicates the air stability of ash and C-PAC concretes in both the static and dynamic cases. In these tests of the static case, the initial air in the fresh ash and C-PAC concretes were 6.8 and 5.5 vol%, respectively. The air in both was very stable over the 70 minutes. The static case reflects ready-mix fresh concrete setting on the jobsite without mixing or agitation. In the dynamic case, the initial air of ash and C-PAC concretes were 6 and 5 vol% respectively. Some air was gradually lost with time. About 30% of the initial air was lost after 90 minutes, following the production and mixing of the concrete. The dynamic measurement simulated ready-mix concrete delivery to a site with continuous mixing or agitating. The stability of air in both ash control and C-PAC concretes were precisely the same. This suggests that the C-PAC mercury sorbent in concrete does not influence the air lost at this dosage.

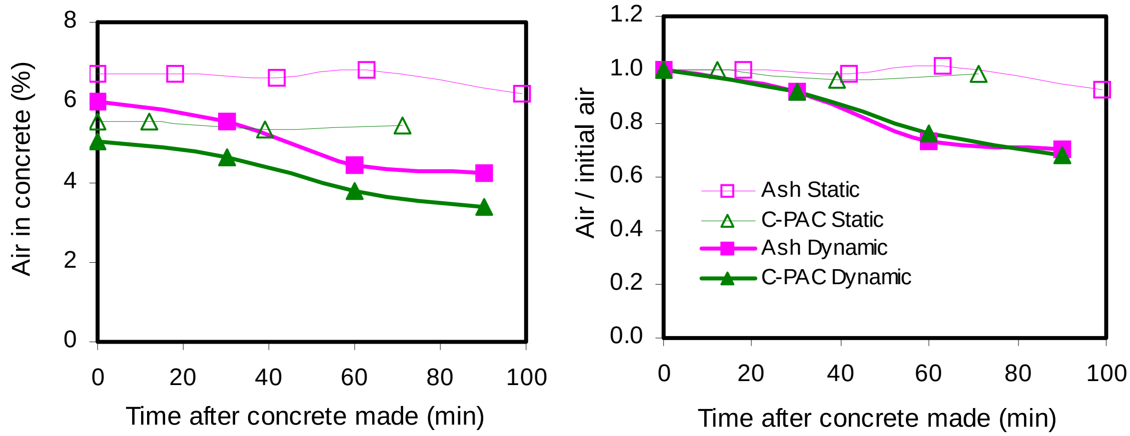

Figure 8. Air stabilization with time of fresh concrete incorporated with baseline fly ash (AC) and long-term C-PAC Hg sorbent test fly ash (C-PAC). Open and full symbols represent static and dynamic case, respectively. Static case measurements are from Sorbent Technologies; dynamic case results are from Headwaters.

5.3 Setting times

The setting times of concrete including fly ash with C-PAC™ (C-PAC) and without C-PAC™ (Ash) were almost identical (Figure 9). For both the C-PAC and ash control concretes, the calculated time at initial set, 500 psi, and final set, 4000 psi, were 315 and 466 minutes, respectively. This suggests that C-PAC™ does not affect the setting of concrete in comparison with the ash control. The setting of concrete without any fly ash was faster than fly ash concrete.

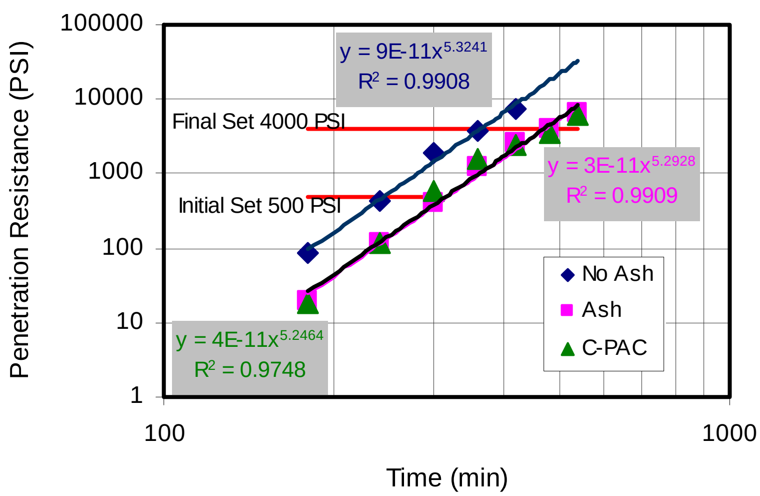

Figure 9 Time of setting of concretes

5.4 Unconfined compressive strength

It is well established that fly ash reduces early strength, but enhances its ultimate strength.   Indeed, the non-fly ash concretes had a higher UCS than the fly ash concretes, both ash and C-PAC, at 3 and 7 days (Figure 10).   It appeared that the concrete-friendly mercury sorbent improved the early strength by 10 to 15% compared to the ash control concrete.

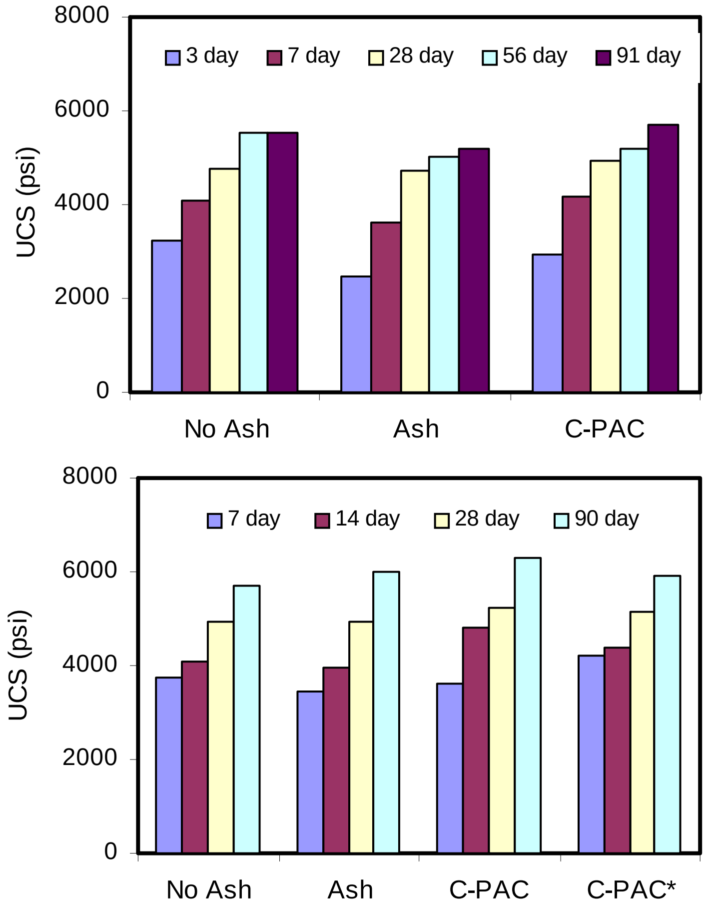

Figure 10. Unconfined compressive strength of concretes made by Headwaters (top) and Sorbent Technologies (bottom). For the top set of concretes made by Headwaters: the slump of No Ash, Ash Control, and C-PAC were 6.0, 6.5, and 6.5 inches and air was 4.2, 5.7, and 5.8 vol% after adding AEA 7, 10, and 22 ml/100 kg of cement-ash in each. Sorbent Technologies' parameters are listed in Table 1. CPAC* is a synthetic mixture with baseline fly ash, in contrast to C-PAC, which went through the ESP.

6. Conclusions

- The increased fly ash mercury concentrations with C-PAC injection confirm high 80% to 90% mercury removal rates at reasonable injection rates. Mercury, LOI, and carbon in fly ash were proportional to the sorbent injected into the duct. Mercury levels correlated well with LOI and carbon in the fly ash.
- Compared to ash-containing control concrete, concrete with C-PAC-fly ash was shown to exhibit the same air content, slump, and setting time with only a relatively higher dosage of AEA. Importantly, the standard deviations of foam index values of fly ashes with C-PAC were no higher than those without.
- C-PAC mercury sorbent does not appear to affect air stability with mixing and produces concretes at least as strong as fly-ash-containing concrete without it.
- C-PAC appears to both achieve high mercury reductions and preserve continued fly ash sales for concrete.

Acknowledgement

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