Advances in Chemical Beneficiation of Fly Ashes Containing Natural Carbon or Powdered Activated Carbon

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ABSTRACT

Due to increased air pollution control regulations throughout North America and other parts of the world, contamination of fly ash with natural carbon or powdered activated carbon (PAC) is becoming a growing threat to the continued use of fly ash in air-entrained concrete applications. This work will provide information on the collaborative efforts between FlyAshDirect and BASF on the development of advanced chemical methods for treating fly ashes affected by various forms of natural carbon or PAC. Results from testing of treated fly ashes in air-entrained concrete evaluations are included, showing that entrained air can be generated and remain stable during concrete agitation. Such stability of entrained air in the presence of chemically-treated fly ash is demonstrated with a mortar screening method and also with laboratory concrete studies. This presentation will further describe a unique method to measure the adsorptive potential of carbon-containing fly ashes in relation to air-entraining admixture performance in concrete. FlyAshDirect's patented CarbonBlocker™ (CB) technology, utilizing BASF chemistry, is shown to provide a commercially-viable method to treat fly ash that is contaminated with even the most aggressive types of PAC, so that the fly ash can be used with predictable performance in air-entrained concrete.

Introduction

Due to various air pollution control standards and legislation, coal-fired power plants throughout the U.S. and other parts of the world have been required and will continue to install air pollution controls (APCs) for the purpose of capturing NOx and mercury emissions. NOx APCs, particularly low-NOx burners, can increase or cause inconsistencies in the level of unburned or "natural" carbon that is measured in fly ash. The challenge of using fly ash containing elevated levels of natural carbon in air-entrained concrete is well known and is attributed to the adsorptive nature of such fly ash carbon to air-entraining admixtures. Furthermore, due to recent U.S. and Canadian air quality standards for mercury emissions, the use of PAC injection systems is rapidly being adopted and further threatens the use of fly ash in concrete. PAC injection, when used ahead of particulate control systems, causes mercury-laden carbon to comingle with the collected fly ash. PAC-contaminated fly ashes present a significantly more challenging situation than natural carbons when used in air-entrained concrete. PACs are specifically designed to have an extremely high surface area to help capture oxidized mercury and hence have an adsorptive potential for air-entraining admixtures that is much greater than natural carbon. Hence, even low concentrations of PAC have been found to render an ash unsuitable for air-entrained concrete applications. Consequently, the implementation of PAC injection has caused large amounts of previously high-quality fly ash in various parts of the U.S. to be sent for disposal. This paper discusses the collaborative efforts between FlyAshDirect, a division of Waste Management (FAD-WM), and BASF in their joint development of chemical formulations used with FAD-WM's patented CarbonBlocker™ (CB) technology to provide a beneficiation solution for natural and PAC contaminated fly ashes. CarbonBlocker™ is a unique approach to apply liquid chemistry to powders in a bulk flow environment. CarbonBlocker™ imparts chemistry on to fly ash in the fluid phase, as opposed to the dense phase, significantly reducing the amount of chemistry needed to effectively beneficiate carbon-containing fly ashes relative to other available chemical beneficiation technologies.

There have been many published studies that have described chemical treatment of fly ash that included fly ash that was contaminated with either natural carbon or PAC[1-8]. This study describes the chemical treatment of industrial fly ashes that have been contaminated with either natural carbon or PAC, and illustrates the impact of the treatment on the stability of entrained air in mortar or concrete. The required level of treatment chemistry can be quantified based on an adsorption saturation dosage measured with a new test developed at BASF. Test data presented in this study specifically demonstrates the ability of CarbonBlocker™ to effectively treat various carbon-tainted fly ashes with BASF chemistry to deliver stable air performance in air-entrained concrete over extended mixing cycles of up to sixty minutes. Natural and PAC-contaminated fly ash samples used in the study were taken from active coal-fired energy generating stations. Such real-life fly ash samples were collected at power stations that have implemented some of the highest PAC injection rates currently used throughout North America, at 5 lb/MMacf (80 mg/acm).

Experimental

Materials and Methods

Mortar and concrete mixes contained Type I/II cement and siliceous fine aggregate. A blend of #57 and #8 limestone was used as the coarse aggregate for concrete batches. Fly ash that was contaminated with either natural carbon or PAC was sourced from multiple locations. Table 1 describes some of the characteristics of the fly ashes used in the various studies.

Table 1. Description of the fly ashes used in the evaluations

Identifier	Fly ash class (ASTM C 618[9])	LOI	Fineness	Foam index	PAC injection rate
Ash A	F	2.87%	12.70%	130	
Ash M	F	5.52%	16.00%	50	
Ash C	C	2.36%	9.81%	130	5 Ib/MMacf (80 mg/acm)
Ash W	C	2.94%	10.60%	210	5 Ib/MMacf (80 mg/acm)
Ash N	C	0.80%	15.27%	135	2.5 Ib/MMacf (40 mg/acm)
Ash J	C	2.70%	14.41%	320	5 Ib/MMacf (80 mg/acm)

Chemical admixtures used for the mortar and concrete testing included natural and synthetic air-entraining admixtures (AEA: BASF Micro Air® air-entraining admixture, BASF MB-VR™ Standard air-entraining admixture, or Euclid Air Mix 200) and water-reducing admixtures (WRA: BASF Pozzolith® 80 water-reducing admixture or Euclid Eucon WR 91).

Mortar Test for Determining Stability of Entrained Air

A screening method was developed to determine the stability of entrained air in the presence of carbon-containing fly ash. The mortar design is based on 600 lb/yd^3 (356 kg/m^3) of cement or a 80%/20% by mass blend of cement and fly ash, 0.45 water/cementitious ratio, graded standard silica sand, and a selected dosage of air-entraining admixture. The details of the method are as follows:

1. Mix mortar in accordance with ASTM C 305[9].
2. Measure air content using the standard gravimetric procedure (400mL brass cup) described in ASTM C 185[9].
3. Mix mortar at slow speed for an additional 10 minutes at slow speed and determine the air content again.
4. Mix mortar at slow speed for an additional 20 minutes, then measure the air content (30-minute reading).
5. Mix mortar at slow speed for an additional 30 minutes, then measure the air content (60-minute reading).

Concrete Mixture Design, Mixing, and Testing

For testing fly ashes that contained natural carbon, laboratory concrete mixtures were designed with a total cementitious content of 564 lb/yd^3 (335 kg/m^3) that included 20% by mass fly ash. For testing with fly ashes containing PAC, laboratory concrete mixtures were designed with a total cementitious content of 600 lb/yd^3 (356 kg/m^3) that included 25% by mass fly ash. Initial slump of concrete was targeted at 5-7” (125-180mm). Air-entraining admixture was dosed to produce an initial air content of 5-7%. Concrete mixing was performed in laboratory-scale rotating drum mixers that were equipped with a controller to adjust the drum speed from 0-20 rpm. The concrete mixing cycle was 3 minutes at 20 rpm followed by 3 minutes rest followed by 2 minutes at 20 rpm. After the initial mixing cycle, the speed of the mixer was reduced to 3 rpm for a 60-minute period to simulate transport of concrete in a ready-mix truck. Slump and air content (pressure method) were measured. Unit weight measurements were performed with each air content measurement. In most cases, slump, air content, and unit weight of the concrete were measured after the initial mixing cycle, again following 30 minutes of slow agitation, and once more following a total of 60 minutes of slow agitation.

Saturation Dosage of Treatment Chemistry

Published studies have described techniques to determine the adsorption potential of carbon-containing fly ashes, based on color change after methylene blue adsorption or UV-Vis absorbance of air-entraining admixture[4,10]. For the fly ashes contaminated with PAC in this study, methylene blue adsorption was found to be very high and not reproducible. New methodology was developed to determine the dosage of treatment chemistry that is required for each natural or PAC-contaminated fly ash. Such dosage is intended to passivate the free surface area of carbon in the fly ash. In brief, the method is as follows:

1. Prepare slurry (1 part fly ash to 2 parts liquid) with a dilute solution of a tracer chemistry.
2. Filter the slurry after 30 minutes of continuous mixing to remove the liquid phase.
3. Use a technique such as Gel Permeation Chromatography (GPC) to determine the amount of tracer chemistry that was removed from solution, after setting a baseline using the dilute solution of tracer chemistry.
4. Calculate the level of treatment chemistry required for the tested fly ash (saturation level).

Saturation dosages for the fly ashes that were used for the mortar and concrete studies are shown in Table 2.

Table 2. Treatment chemistry saturation dosage for fly ashes

Identifier	Fly ash class (ASTM C 618[9])	Treatment chemistry saturation dosage per mass of fly ash fl.oz./ton (mL/kg)
Ash A	F	33 (1.08)
Ash M	F	10 (0.33)
Ash C	C	54 (1.76)
Ash W	C	90 (2.93)
Ash N	C	43 (1.40)
Ash J	C	107 (3.49)

Chemical Treatment of Fly Ash

Treated fly ash that was used for the studies presented here were prepared either with a lab-scale or pilot-scale treatment system, or with the industrial treatment system in place at some power plants. Details of the treated fly ashes are shown in Table 3. Natural carbon Class F (ASTM C 618[9]) fly ashes were usually treated with CB chemistry, while PAC-contaminated Class C (ASTM C 618[9]) fly ashes were treated with ACB chemistry. Ash J was a PAC-contaminated fly ash that was treated with both CB and ACB chemistries.

Table 3. Fly ash treatment

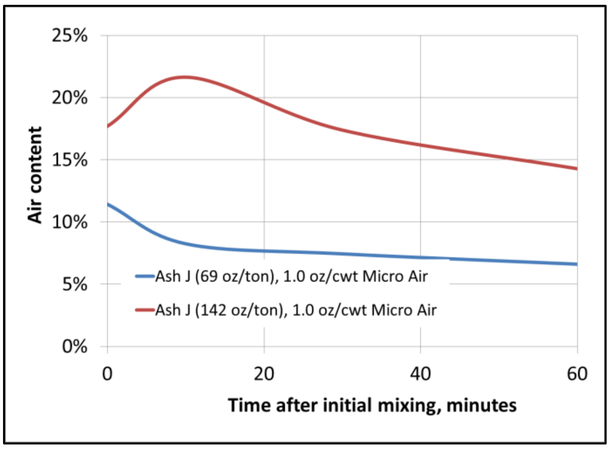
Identifier	Class	Foam index	Treatment chemistry	Treatment chemistry dosage
Ash A	F	120	CB	5 fl.oz./ton (0.16 mL/kg)
Ash A	F	45	CB	11.1 fl.oz./ton (0.36 mL/kg)
Ash M	F	20	CB	5.4 fl.oz./ton (0.18 mL/kg)
Ash M	F	20	CB	10.4 fl.oz./ton (0.34 mL/kg)
Ash M	F	15	CB	20.9 fl.oz./ton (0.68 mL/kg)
Ash C	C	0	ACB	37 fl.oz./ton (1.21 mL/kg)
Ash C	C	0	ACB	47 fl.oz./ton (1.53 mL/kg)
Ash W	C	0	ACB	93 fl.oz./ton (3.03 mL/kg)
Ash N	C	0	ACB	23 fl.oz/ton (0.75 mL/kg)
Ash J	C	80	CB	69 fl.oz/ton (2.25 mL/kg)
Ash J	C	0	CB	142 fl.oz/ton (4.63 mL/kg)
Ash J	C	190	ACB	66 fl.oz/ton (2.15 mL/kg)
Ash J	C	0	ACB	138 fl.oz/ton (4.50 mL/kg)

Lower dosages of treatment chemistry are required with the natural carbon fly ashes, due to the lower adsorption of natural carbon compared to PAC. Fly ash foam index values were reduced as a result of the treatment, and higher treatment dosages usually resulted in a lower fly ash foam index.

Results and Discussion

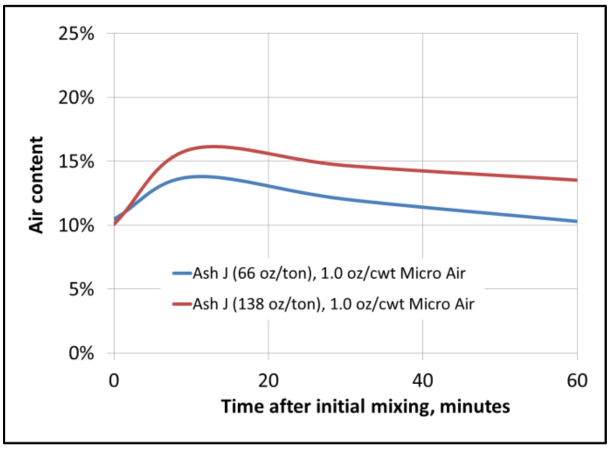
Stability of Entrained Air: Fly Ash Contaminated with PAC

Ash J, a PAC-contaminated Class C fly ash was treated with two dosage levels of either CB or ACB. The treated fly ash was tested for its influence on air stability using the mortar method described in this paper. A synthetic air-entraining admixture was used to generate air in the mortar. The test results for the fly ash treated with CB are shown in Figure 1 and for the fly ash treated with ACB are shown in Figure 2. A comparison of the plots indicates that with the use of treated Ash J, the ACB treatment allows for a more linear response of air content in mortar compared with the CB treatment.



100 oz/ton = 3.26 mL/kg; 1.0 oz/cwt = 0.65 mL/kg

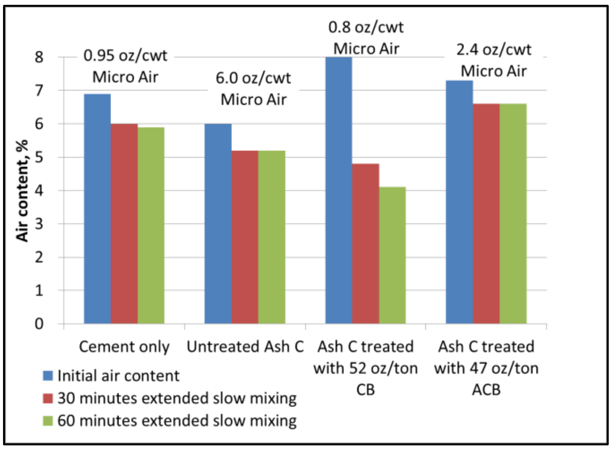
Figure 1. Stability of entrained air in mortar: Ash J treated with CB



100 oz/ton = 3.26 mL/kg; 1.0 oz/cwt = 0.65 mL/kg

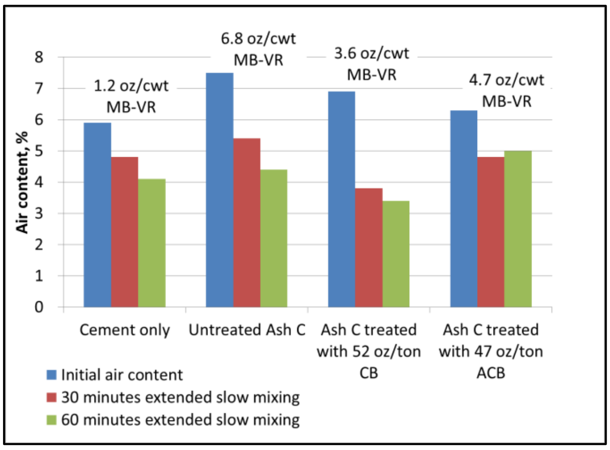
Figure 2. Stability of entrained air in mortar: Ash J treated with ACB

Ash C, a PAC-contaminated Class C fly ash, was treated with 52 oz/ton (1.70 mL/kg) CB or 47 oz/ton (1.53 mL/kg) ACB. Laboratory concrete mixes were prepared using these two treated fly ashes, along with a batch prepared with untreated fly ash and a batch prepared without fly ash for comparison. Either a synthetic or natural air-entraining admixture was used to generate air in the concrete. The air content measured over the full mixing cycle is shown in Figure 3 for the concrete that used Micro Air® air-entraining admixture, while the air content measured over the full mixing cycle is shown in Figure 4 for the concrete that used MB-VR™ Standard air-entraining admixture. It is clear from these two plots that the concrete that contained fly ash treated with ACB showed a much more stable air content than the concrete that contained fly ash treated with CB, independent of the type of air-entraining admixture.



100 oz/ton = 3.26 mL/kg; 1.0 oz/cwt = 0.65 mL/kg

Figure 3. Stability of entrained air in concrete: Ash C and Micro Air® air-entraining admixture



100 oz/ton = 3.26 mL/kg; 1.0 oz/cwt = 0.65 mL/kg

Figure 4. Stability of entrained air in concrete: Ash C and MB-VR™ Standard air-entraining admixture

Petrographic analysis of concrete specimens was completed using methods described in ASTM C 457[9]. Concrete slabs that were cut and polished for petrographic analysis were obtained from concrete cylinders that were cast after the initial mixing cycle or after the 60-minute agitation cycle. The two specimens from each mix were compared to investigate the change in the air void system of the concrete with extended mixing of concrete. Values that are typically associated with concrete that will have adequate freeze/thaw durability are air content ≥4.5%, specific surface area ≥600 in^2/in^3 (24 mm^2/mm^3), and spacing factor ≤0.008 in (0.20 mm). The concrete that was used for the air void analysis results shown in Table 3 is the same concrete that produced the concrete air content results in Figure 3. The results in Table 3 show that treatment of Ash C with CB causes the air void system to become unstable with extended mixing of the concrete. The treatment of Ash C with ACB causes the air void system to improve (higher specific surface and lower spacing factor) with extended mixing of the concrete.

Table 3. Petrographic Parameters of Hardened Concrete (Concrete Mixes from Fig. 3)

		After initial mixing			After 60 minutes agitation		
Concrete description	Micro Air dosage, oz/cwt (mL/kg)	Air content, %	Specific surface area, in^2/in^3 (mm^2/mm^3)	Spacing factor, in. (mm)	Air content, %	Specific surface area, in^2/in^3 (mm^2/mm^3)	Spacing factor, in. (mm)
Cement only	0.95 (0.62)	7.4	447 (18)	0.008 (0.20)	6.9	460 (18)	0.008 (0.20)
25% Ash C, untreated	6.0 (3.9)	7.5	614 (24)	0.006 (0.15)	4.2	625 (25)	0.008 (0.20)
25% Ash C, 52 oz/ton (1.7 mL/kg) CB	0.8 (0.5)	7.7	559 (22)	0.006 (0.15)	6.1	335 (13)	0.012 (0.30)
25% Ash C, 47 oz/ton (1.53 mL/kg) ACB	2.4 (1.6)	8.7	499 (20)	0.006 (0.15)	7.4	663 (26)	0.005 (0.13)

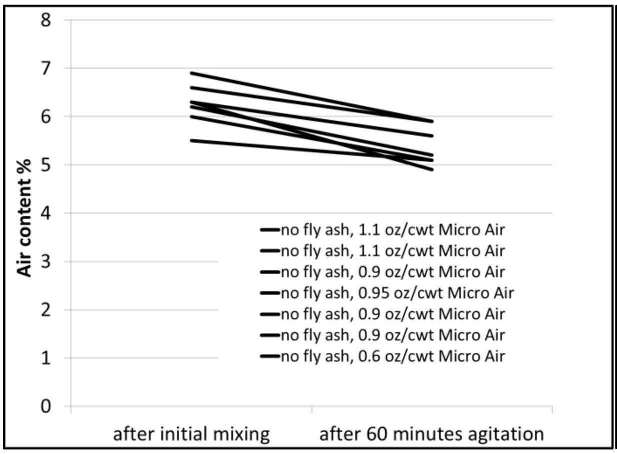
The concrete that was used to produce the air void analysis results shown in Table 4 is the same concrete that produced the concrete air content results in Figure 4. The results in Table 4 show that treatment of Ash C with ACB causes the air void system to be maintained or improve with extended mixing of the concrete.

Table 4. Petrographic Parameters of Hardened Concrete (Concrete Mixes from Fig. 4)

		After initial mixing			After 60 minutes agitation		
Concrete description	MB-VR dosage, oz/cwt (mL/kg)	Air content, %	Specific surface area, in^2/in^3 (mm^2/mm^3)	Spacing factor, in. (mm)	Air content, %	Specific surface area, in^2/in^3 (mm^2/mm^3)	Spacing factor, in. (mm)
Cement only	1.2 (0.8)	5.9	553 (22)	0.008 (0.20)	5.7	497 (20)	0.008 (0.20)
25% Ash C, untreated	6.8 (4.4)	8.6	595 (23)	0.005 (0.13)	5.7	521 (21)	0.009 (0.23)
25% Ash C, 52 oz/ton (1.7 mL/kg) CB	3.6 (2.3)	10.3	362 (14)	0.007 (0.18)	No test	No test	No test
25% Ash C, 47 oz/ton (1.53 mL/kg) ACB	4.7 (3.1)	7.8	446 (18)	0.006 (0.15)	8.0	508 (20)	0.006 (0.15)

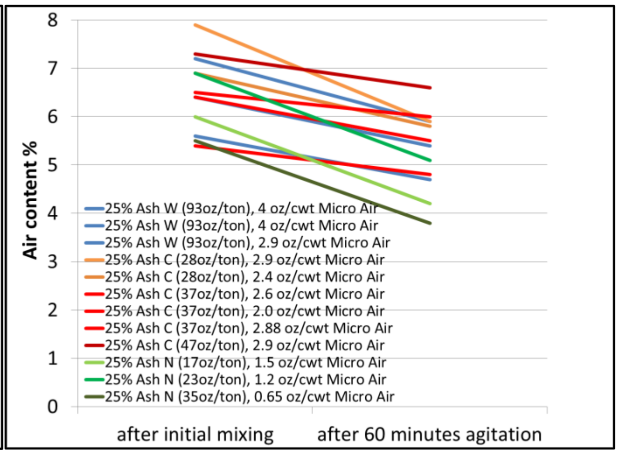
Based on these results, ACB was chosen as the treatment chemistry for PAC-contaminated fly ash. Also, the results from the mortar screening test for air stability correlated well with results from the air contents measured during the extended mixing cycle in concrete, which was considered to be a validation of the mortar screening method.

The stability of entrained air in concrete in the absence or presence of different sources of treated fly ash and fly ashes treated with different levels of ACB and a synthetic air-entraining admixture is shown in Figures 5 and 6. The general trend in both plots is towards a decrease in air content with agitation. Some of the air loss is related to a reduction of the slump of the concrete during mixing, which occurs to some extent in all of the concrete mixes that were tested. A comparison of the plots suggests that the air content reduction is no greater when treated fly ash is used as a concrete ingredient. Treatment dosage changes for a particular fly ash seem not to change the degree of entrained air loss. In general, for each fly ash, a lower treatment dosage causes the dosage of air entraining admixture, which is required to achieve a target air content, to rise.



1.0 oz/cwt = 0.65 mL/kg

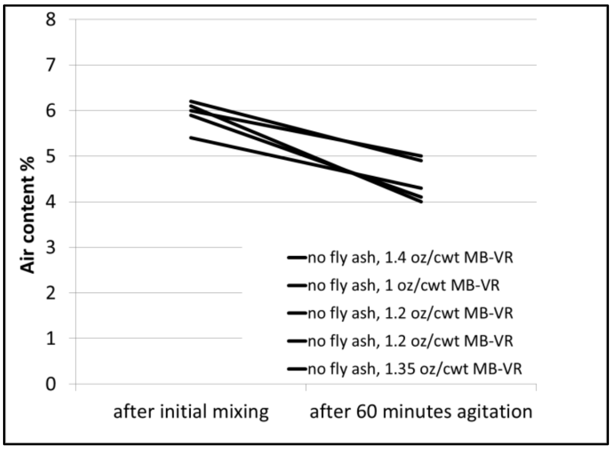
Figure 5. Stability of entrained air in concrete: cement only case, synthetic AEA



100 oz/ton = 3.26 mL/kg; 1.0 oz/cwt = 0.65 mL/kg

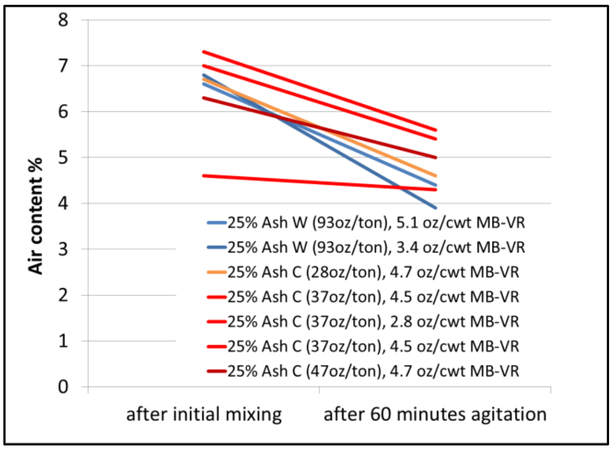
Figure 6. Stability of entrained air in concrete: treated fly ashes, synthetic AEA

Figures 7 and 8 show the same type of data that is shown in Figures 5 and 6. The difference is that a natural air-entraining admixture was used. These results show that the same trends that were described for Figures 5 and 6 appear in these figures as well, suggesting that the effect of the treatment is independent of the character of the air-entraining admixture.



1.0 oz/cwt = 0.65 mL/kg

Figure 7. Stability of entrained air in concrete: cement only case, natural AEA

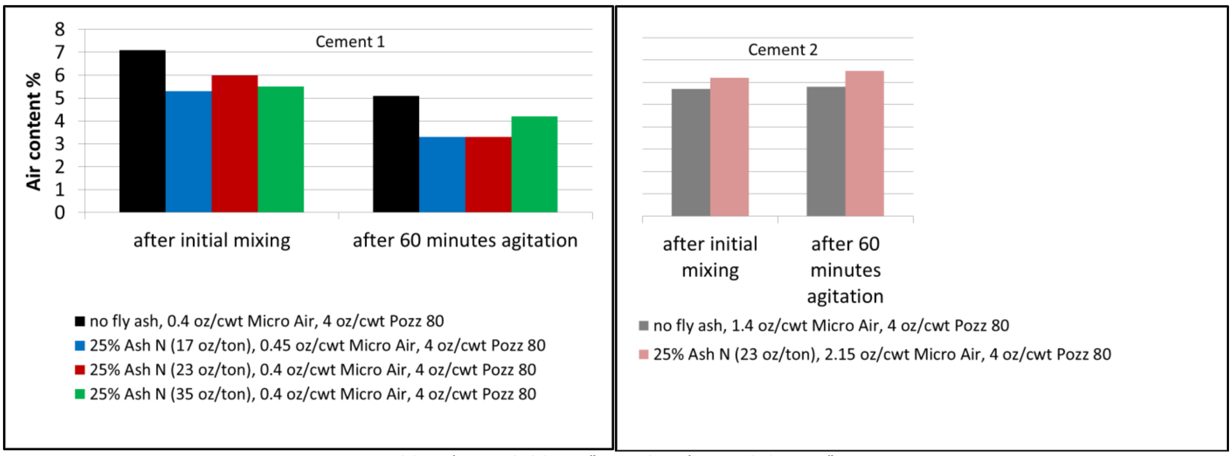


100 oz/ton = 3.26 mL/kg; 1.0 oz/cwt = 0.65 mL/kg

Figure 8. Stability of entrained air in concrete: treated fly ashes, natural AEA

Influence of Water-Reducing Admixture

Figure 9 shows the stability of entrained air in the absence and presence of treated Ash N, a synthetic air-entraining admixture, and Pozzolith® 80 water-reducing admixture (ASTM C 494[9]). Ash N was treated with three different dosages of ACB. The concrete was produced with two different brands of portland cement. The two sets of columns on the right, one representing a mix without fly ash and the other representing a mix with treated fly ash, show a similar upward trend. Both of these concrete mixes required higher dosages of AEA compared to the concrete mixes depicted in the two sets of columns on the left (due to the characteristics of the cements). The two sets of columns to the left all trend down to lower entrained air contents in similar increments. This data shows that addition of a water-reducing admixture doesn't change the beneficial effect of the chemical treatment of the fly ash with ACB.

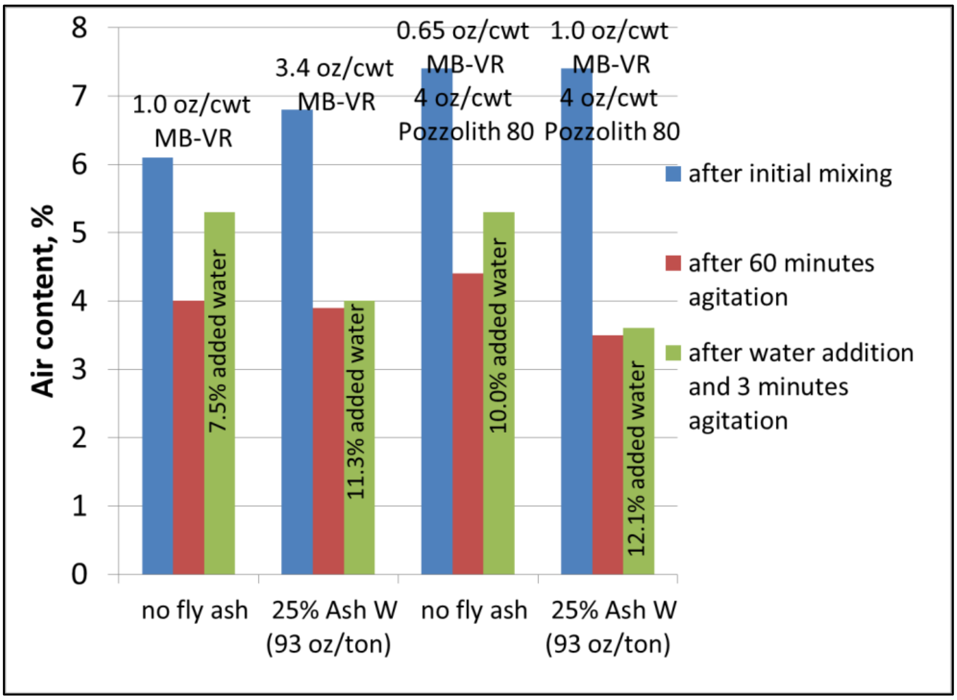


100 oz/ton = 3.26 mL/kg; 1.0 oz/cwt = 0.65 mL/kg

Fig. 9. Stability of entrained air in concrete: synthetic AEA and Type A WRA

Influence of Water Addition for Slump Increase

Figure 10 shows the results from a study that included a natural air-entraining admixture, concrete mixes without fly ash or Ash W treated with ACB, and concrete mixes with or without Pozzolith® 80 water-reducing admixture. In this study, the concrete mixing cycle was extended for three additional minutes at mixing speed. During this additional mixing cycle, water was added to the concrete to return the slump from 2.5 ± 0.5 inches (65 ± 10 mm), which is where it had decreased to from the original measurement after the initial mixing cycle, to 5.25 ± 0.5 inches (130 ± 10 mm). The air content results show that in the presence of the treated fly ash, the air content is unaffected by the water addition with mixing. In the absence of fly ash, the air content tends to rise with the water addition during mixing. This suggests that the concrete entrained air content is more stable when treated fly ash is included in the mix composition.



100 oz/ton = 3.26 mL/kg; 1.0 oz/cwt = 0.65 mL/kg

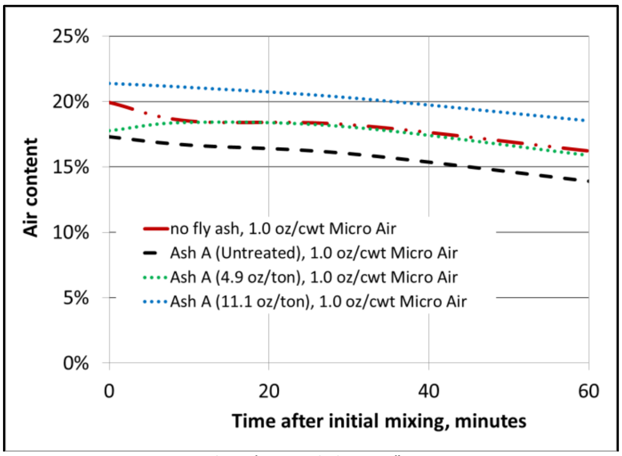
Figure 10. Effect of addition of retempering water on the entrained air in concrete

Effect of Treatment Chemistry on Mortar and Concrete Performance: Fly Ash Containing Natural Carbon

Figure 11 shows the results from a study that included no fly ash, untreated Ash A and Ash A treated with CB. The fly ash was tested for its influence on air generation and air stability using the mortar method described in this paper. A synthetic air-entraining admixture was used to generate air. The air content results show that in the presence of the treated fly ash, the air content is greater than that of untreated Ash A. The air content of the mix containing Ash A treated with 4.9 oz/ton (0.16 mL/kg) of CB was very similar to the mix containing no fly ash. In the concrete mix containing Ash A treated with 11.1 oz/ton (0.36 mL/kg) of CB, more entrained air was generated and the trend of air over time was similar to the mix that contained no fly ash.

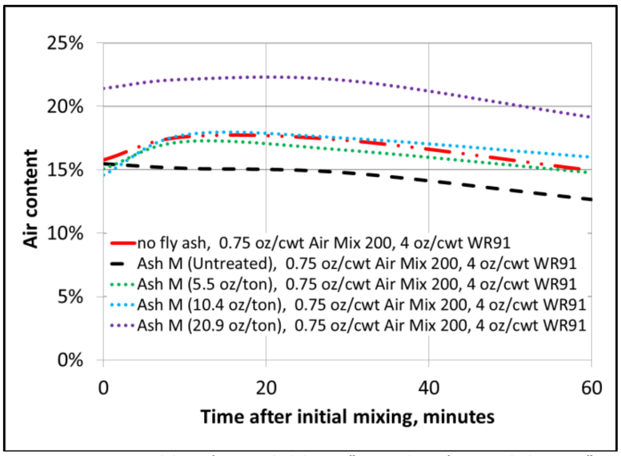
Figure 12 shows the results from a study similar to the one represented in Figure 11, this time utilizing Ash M, a natural air-entraining admixture, and a different Type A water-reducing admixture. The air content results show that in the presence of the treated fly ash, the air content is greater than that of untreated Ash M. The air content of the mix containing Ash M treated with 5.5 oz/ton (0.18 mL/kg) or 10.4 oz/ton (0.34 mL/kg) of CB was very similar to the mix containing no fly ash. In the concrete containing Ash A treated with 20.9 oz/ton (0.68 mL/kg) of CB, more entrained was generated and the trend of air over time was similar to the mix that contained no fly ash.

Both studies indicate that stable and predictable entrained air is generated in the presence of ashes treated with CB.



1.0 oz/cwt = 0.65 mL/kg

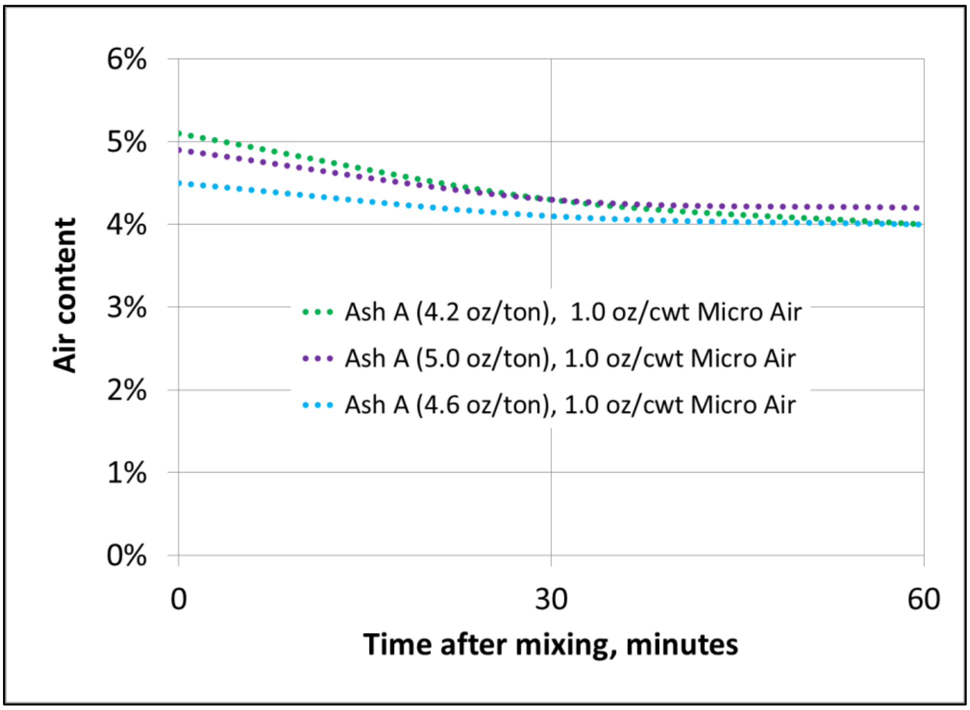
Figure 11. Stability of entrained air in mortar: Ash A and Micro Air® air-entraining admixture



100 oz/ton = 3.26 mL/kg; 1.0 oz/cwt = 0.65 mL/kg

Figure 12. Stability of entrained air in mortar: Ash M, Air Mix 200, Eucon WR 91

Figure 13 shows the results of a concrete study that includes three samples of Ash A treated at the power plant during commercial loading on different days. The samples were treated with either 4.2 oz/ton (0.14 mL/kg), 5.0 oz/ton (0.16 mL/kg), or 4.6 oz/ton (0.15 mL/kg) of CB. A synthetic air-entraining admixture was used for this study. The results indicate that all three samples generated similar air results and stability.



100 oz/ton = 3.26 mL/kg; 1.0 oz/cwt = 0.65 mL/kg

Figure 13. Stability of entrained air in concrete: Ash A and Micro Air® air-entraining admixture

Conclusions

Stable entrained air in concrete produced with fly ash that is contaminated with PAC or natural carbon may be difficult to achieve. This work has shown that the beneficiation of such fly ash with CarbonBlocker™ technology allows the fly ash to be used effectively in air-entrained concrete. The appropriate chemistry is chosen based on the character of the fly ash, and the treatment dosage can be determined using an analytical method. Concrete made with PAC-contaminated fly ash that has been treated with ACB has been shown to have relatively stable entrained air with acceptable petrographic parameters. Treatment with CB of fly ash that contains natural carbon has been shown to produce stable entrained air in mortar and concrete. A mortar method for screening air stability has been shown to be an effective predictor of entrained air stability in concrete.

CarbonBlocker™ has provided a commercially viable means to treat fly ashes containing natural carbon with six sites currently in operation since 2005. This technology has been recently improved through the development of two new chemistries to provide a technically and economically-viable fly ash beneficiation technology for fly ashes containing either natural carbon or PAC.

Additional work is ongoing to characterize the freeze/thaw durability of air-entrained concrete made with PAC-contaminated fly ash that has been treated with ACB.

Acknowledgements

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