Investigation of Biomass Co-Fired Fly Ash Properties: Characterization and Concrete Durability Performance

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ABSTRACT

The co-combustion of biomass with coal can be a sustainable energy source and significantly reduce carbon dioxide emissions. The product of this process, biomass-derived fly ash, is not addressed in US standards for fly ash use in concrete; however, recent European standards have permitted its use with certain restrictions. The engineering properties of co-fired fly ash are relatively unknown compared to coal fly ash. This study characterizes the composition, structure, and performance of co-fired fly ash. Foam index tests show that fly ash replacement of cement increases air-entraining admixture (AEA) demand, likely a result of increased carbon surface area. Furthermore, a carbon distribution study reveals that the finest fraction of carbon has the largest AEA demand. Ultimately, the results indicate that the exclusion of co-fired fly ash from the standards may be inappropriate with respect to chemical, physical, and durability requirements.

INTRODUCTION

Fly ash is a pozzolan that consumes calcium hydroxide and produces strength-providing calcium silicate hydrates, densifies the ITZ (interfacial transition zone) at the cement-aggregate interface, and refines the concrete pore structure [Thomas 2006]. The partial replacement of cement binder with supplementary cementitious materials such as fly ash improves later strength and decreases permeability in concrete. Fly ash typically results from the combustion of a coal fuel source. In 2007, 43% of the 131 million tons of coal combustion products (CCP) generated in the US were used beneficially, with over a quarter of the beneficial CCP usage comprising of fly ash in concrete [American Coal Ash Association 2007]. However, recent increase in use of biofuels, derived from organic materials (e.g., trees, switchgrass), as a combustion fuel source has produced a biomass-derived fly ash. The engineering properties of this co-fired fly ash are relatively unknown compared to that of coal fly ash.

The co-firing of biomass with coal is a sustainable energy source when the consumption of the biomass is less than its rate of growth [Baxter 2005]. Furthermore, co-firing biomass for power generation can be CO_2-neutral when the amount of CO_2 released into the atmosphere is less than or equal to the amount of CO_2 plants intake during their lifetime [Hughes 2000; Maciejewska et al., 2006]. Currently, biomass contributes 16% of the renewable-generated electricity produced in the US [Energy Information Administration 2008]. The use of

biomass as a secondary fuel is typically less than 20% by weight and 10% by energy content in the co-combustion process [Grammelis et al., 2006; Wang et al., 2007]. A by-product of this process, co-fired fly ash, is not addressed in ASTM C 618, which currently defines fly ash as "the finely divided residue that results from the combustion of ground or powdered coal and that is transported by flue gasses". Recent studies [Wang et al., 2007, 2008a, b, c] have indicated that co-fired fly ash performs adequately compared to coal fly ash in concrete strength and durability testing and, thus, its exclusion from the standards may be inappropriate. However, further testing of a variety of co-fired fly ashes is necessary to better characterize their performance in concrete.

Over the past fifteen years research on co-fired ash has been conducted in the Netherlands, the result of an initiative to reduce CO_2 emissions through biomass replacement of coal [Berg 2005]. It was determined that through understanding the impact of co-combustion on the properties and quality of fly ash, the performance of the co-fired ash in concrete could be directly related to its mineralogical and chemical composition [Saraber 2005]. In 2004, CEN members voted to revise the standard to accept fly ash obtained from co-combustion [Berg 2005]. The EN 450-1 permits ashes that are "produced when small quantities of secondary materials are combusted with at least 80% of coal by dry mass in a power station with a maximum ash content of 10% being derived from the secondary material." Allowed co-combustion materials are vegetable materials, green wood and cultivated biomass, animal meal, municipal sewage sludge, paper sludge, petroleum coke, and virtually ash free liquid and gaseous fuels.

The variability of fly ash, due to its production as a by-product material, can present challenges in the prediction and control of concrete behavior. One major issue encountered when utilizing fly ash in concrete occurs when its unburned carbon content interferes with air-entraining admixtures (AEAs). AEAs are used to generate a small and well-distributed air-void system in concrete to prevent damage from freezing-and-thawing cycles and salt scaling [Ley et al., 2008]. AEAs are solutions of surfactants with a hydrophilic nature that orient at the water-air interface to stabilize the air bubbles [Freeman et al., 1997]. The AEAs are adsorbed on the surface of the carbon rendering them ineffective in stabilizing the air bubbles and inhibiting the production of an adequate air void system. The surface area, surface polarity, and other properties of the carbon strongly influence AEA adsorption [Scott et al., 2007]. The use of co-fired fly ash, which could embody high carbon content, must therefore be tested for interaction with AEAs.

The objective of this study is to characterize the composition and structure of the co-fired fly ash and to further explore its influence on the durability of concrete (i.e., its AEA interaction). The foam index test [Harris et al., 2008a, b, c] will be used to provide a rapid relative measure of the AEA demand for both coal and co-fired fly ash mixtures. The foam index values will then be compared to loss on ignition (LOI) for all fly ash samples to assess if a relationship exists between residual carbon content and potential for interaction with AEAs. Furthermore, to determine if physical processing of the fly ash could improve its behavior in concrete, this study will investigate the different size fractions of unburned carbon and their effect on AEA adsorption. Ultimately, the properties of the co-fired ash and its effect (deleterious, beneficial or benign) on AEA demand in comparison to coal fly ash will be examined.

EXPERIMENTAL

Materials

Nine types of fly ash (FA) provided by Jack Watson Electric Generating Plant in Gulfport, MS (WA), Ernest C. Gaston Electric Generating Plant in Wilsonville, AL (EC), and Greene County Electric Generating Plant in Demopolis, AL (GC) were investigated. The processing variables used in producing the co-fired FA are provided in Table 1. The samples were produced by co-firing bituminous coal with a range of wood types varying in weight percentage replacement of coal, energy content, and moisture content. Commercially available ASTM C 150 Type I portland cement was used for all testing. The compositional analysis shown in Table 2 was performed using X-ray fluorescence spectroscopy. Loss on ignition, a common indicator of unburned carbon, was determined by thermogravimetric analysis, which involves exposing the samples to temperatures up to $950^{\circ}C$.

All of the coal and co-fired samples have total calcium contents, expressed as calcium oxide, lower than 10% which categorizes them as Class F FA. The results reveal similar concentrations of the main oxides for both the co-fired and coal FA samples with SiO_2, Al_2O_3, and Fe_2O_3 accounting for over 80%. Therefore, these samples meet the ASTM C 618 stipulation for Class F FA that requires these three main oxides be a minimum of 70% by weight. However, ASTM C 618 also specifies a maximum LOI of 6% for FA, a limit exceeded by some of the samples. This is likely a result of differing combustion conditions. The biomass co-firing resulted in lower LOI compared to the coal FA in five samples indicating no correlation between increased biomass to increased carbon content. Sulfur trioxide levels of the coal and co-fired samples are all below the ASTM C 618 limit of 5%, and also showed no correlation to biomass content. The alkali contents of the samples, expressed as total sodium oxide equivalent, are within the range of typical coal FA values [Malvar and Lenke 2006], however their high alkali content in general could inhibit their use for mitigation of alkali-silica reaction. Even though co-fired FA has been shown to be more alkaline compared to coal FA [Demirbas 2003], no strong correlation exists with these samples.

Table 1. Fly Ash Biomass Properties

		Wood Properties, %			
Material	FA Type	Weight	Energy[a]	Moisture[b]	Wood Type
WA-C	Coal Only	-	-	-	-
WA-B1	Co-Fire	5	1.83	52.97	10 mm Pine Chips
WA-B2	Co-Fire	5	1.91	53.92	10 mm Pine Chips
EC-C	Coal Only	-	-	-	-
EC-B1	Co-Fire	4	-	-	10 mm Pine Chips
EC-B2	Co-Fire	8.2	-	48.06	10 mm Pine Chips
GC-C	Coal Only	-	-	-	-
GC-B1	Co-Fire	5	1.85	59.74	10 mm Clean Chips
GC-B2	Co-Fire	5	1.50	61.69	10 mm Whole Tree Chips

[a]As percent of total BTUs produced during co-combustion with coal
[b]Moisture content before co-combustion with coal

Table 2. Chemical and Physical Properties of Fly Ashes and Cement

Component, %	WA-C	WA-B1	WA-B2	EC-C	EC-B1	EC-B2	GC-C	GC-B1	GC-B2	Cement[d]
SiO_2	60.65	62.61	61.64	42.77	42.84	43.95	45.32	47.85	48.03	20.45
Al_2O_3	19.48	19.14	19.58	25.62	26.21	26.45	23.39	24.71	24.41	4.7
Fe_2O_3	7.01	6.13	6.67	14.67	13.87	13.18	14.01	11.73	10.19	3.32
Σ(Oxides)										28.47
CaO	2.85	2.83	2.74	3.83	4.01	3.76	1.13	1.22	1.24	63.06
MgO	1.53	1.39	1.49	1.27	1.25	1.25	1.18	1.20	1.16	3.27
SO_3	0.34	0.29	0.38	0.48	0.44	0.52	0.06	0.10	0.07	2.8
Na_2O	1.916	1.984	1.762	0.507	0.521	0.527	0.677	0.622	0.802	0.067
K_2O	1.514	1.471	1.512	2.126	2.075	2.094	2.416	2.582	2.428	0.532
Na_2O_{eq}										0.42
P_2O_5	0.191	0.190	0.185	0.596	0.651	0.650	0.326	0.370	0.341	0.06
TiO_2	0.830	0.796	0.816	1.264	1.313	1.309	1.074	1.136	1.138	0.25
Mn_2O_3	0.065	0.059	0.066	0.063	0.059	0.060	0.120	0.097	0.088	0.16
Cr_2O_3	0.068	0.059	0.039	0.066	0.064	0.062	0.052	0.053	0.051	0.02
ZnO	0.006	0.004	0.005	0.010	0.008	0.008	0.014	0.011	0.009	0.06
V_2O_5	0.052	0.051	0.050	0.075	0.076	0.074	0.061	0.062	0.060	-
ZrO_2	0.043	0.045	0.041	0.066	0.067	0.065	0.035	0.038	0.041	-
LOI[a]	3.113	2.420	2.752	5.869	5.465	5.225	8.952	7.398	9.434	
LOI-Below[ab]	-	-	-	-	-	-	5.910	4.780	6.020	-
SFI[c]										

[a]Determined by thermogravimetric analysis
[b]Tested on samples collected below the No. 200 sieve (75 μm)
[c]Specific foam index measured by foam index test in units mL AEA/100 kg cm
[d]Bogue Potential Composition: 54.00% C_3S, 15.82% C_2S, 6.84% C_3A, 10.10% C_4AF

Foam Index Test

There is no current standard test to measure the degree of interaction between an AEA and a given FA sample. However, the foam index test is a commonly used field test for determining the acceptability of a particular FA for use in concrete with relation to AEA demand. Typically an AEA solution is incrementally added to a cement, FA, and water slurry in a container and subsequently shaken. The amount of AEA required to produce a stable foam surface is recorded as the foam index. Variables including shaking consistency, slurry hydration, and mixture proportions have motivated Külaots et al., [2003] and Pedersen et al., [2007] to propose a more reproducible standard foam index test.

The procedure for the foam index test used in this experiment was derived from work done by Harris et. al., [2008a, b, c] to develop an ASTM standard test method to predict AEA dosage in concrete containing FA. The commercial AEA (Daravair 1000) used in this experiment is an aqueous solution of neutralized resin acids and rosin acids. In this test, 5 g of FA, 18 g of cement, and 45mL of deionized water (17.1 MΩ-cm) were placed into a

125mL cylindrical capped jar. The mixture was shaken for 30 seconds and 0.06 mL of 11.5 vol. % AEA solution was added. The mixture was then vigorously shaken for 10 seconds and the stability of the foam was observed. The AEA solution was subsequently added one 0.06mL drop at a time until the foam index was reached. The foam index is the minimum amount of AEA solution needed to produce a foam that is stable (bubbles exist over the entire surface) for 20 seconds. In this study specific foam index (SFI) values are utilized, which are expressed as the unit commonly used in industry for admixture dosage: mL AEA/100 kg cementitious material (cm).

Sieving and TOC readings

An approach similar to Külaots et al., [2004] was utilized to study the size distribution of unburned carbon and its effect on AEA adsorption in coal and co-fired FA. The three high LOI samples (GC) were mechanically dry-sieved in 250g portions. ASTM E 11 standard US testing sieves (80, 100, 200, 325, 400, and 500, with nominal openings of 180, 150, 75, 45, 38, and 25 μm, respectively) were utilized. The samples were placed on a mechanical sieve shaker for 10 minutes. Retained FA on each fraction was weighed. Furthermore, TOC measurements of each size fraction were taken on a Shimadzu Total Organic Carbon Analyzer (TOC-V cpn) with a Solid Sample Module (SSM-5000A). The sample is burned in the SSM at 900° C, at which point the organic carbon components of the sample are converted to carbon dioxide. The CO_2 from the sample is then detected by the analyzer, and total organic carbon is calculated based on the amount of CO_2 produced.

To explore a possible mitigation technique to decrease the LOI of the GC samples below the 6% ASTM C 618 limit, 100g of each GC sample was placed on the No. 200 sieve and mechanically vibrated for 10 minutes. X-ray fluorescence spectroscopy and thermogravimetric analysis were conducted on the fraction above and below the sieve. Results for unburned carbon content were different by a maximum of 13% (magnitude of 1.1% difference) between TOC and thermogravimetric measurements for the three unsieved FA samples. This is predominantly a result of different measurement techniques (i.e., the TOC machine measures carbon dioxide released during combustion while thermogravimetric analysis measures total mass loss).

RESULTS AND DISCUSSION

AEA Interaction

A comparison is made in Figure 1 between LOI values and specific foam index (SFI) values for nine different FA samples. There appears to be some correlation that indicates increased LOI results in an increased SFI, and thus increased AEA demand. This finding conflicts with some literature that found no unique correlation between LOI and AEA demand in coal-derived FA [Freeman et al., 1997; Hill et al., 1997; Ley et al., 2008], while it is similar to others [Gebler et al., 1983; Scott et al., 2007]. Criticism of LOI has mainly stemmed from its lack of ability to identify the form and properties of the carbon, which affect AEA adsorption [Freeman 1997]. There appears to be no trend with regard to AEA demand between co-fired FA and coal FA, either between samples derived from the same facility or among the data overall. In both the WA and EC samples, co-fired FA exhibited SFI values below the companion coal sample. However, one co-fired GC sample had a higher SFI compared to the companion coal sample. Both of these phenomena appear to be attributable to LOI differences.
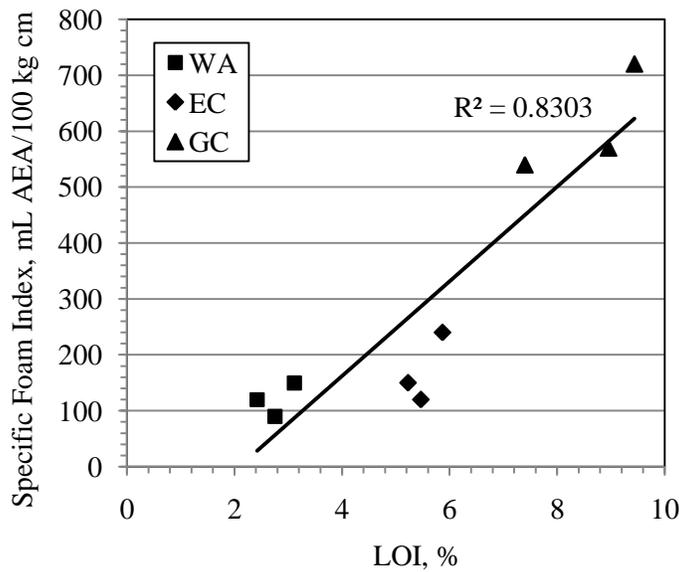
Fig. 1. LOI of Fly Ash vs. Specific Foam Index

Carbon distribution

The distribution of the GC FA particle sizes determined through dry sieving can be seen in Figure 2. It should be noted that each size fraction does not contain particle sizes only within its limits (i.e., finer particles were retained on coarser sieves). This phenomenon may be explained by the coarse particles trapping the finer particles or by the agglomeration of finer particles. Both inorganic and carbon particles were retained on all mesh sizes. From visual inspection it was observed that fractions retained on increasingly finer sieves appeared to be increasingly lighter in color. This trend is reflected in Figure 3, which shows the TOC of each particle size fraction seen in Figure 2. The two largest particle fractions all have unburned carbon contents greater than 40%. Even though these size fractions appear to be pure carbon, more than half of the particles by mass in all but one fraction are actually inorganic. A large reduction in carbon content occurs below the No. 100 sieve (< 150 μm) and continues to decrease in a linear fashion. The coarse particles retained above the No. 80 sieve (>180 μm) of the coal sample have an unburned carbon content more than 10% higher compared to both co-fired samples. This indicates that co-combustion may result in finer carbon particles, but not necessarily a lower amount of unburned carbon.

The amount that unburned carbon retained on each size fraction contributes to the total unburned carbon in a given FA sample is given in Figure 4. There is some correlation between this distribution and the particle size distribution. However, the largest portion of the carbon resides between the 45-150 μm particle range while the largest particle size fraction is less than 45 μm, a result similar to previous studies [Kulaots 2003]. Furthermore, despite the high unburned carbon contents of the coarser fractions (seen in Figure 3) the larger particles have low mass contribution to the FA and, consequently, a lower unburned carbon contribution to the FA when compared to the smaller particles. The carbon retained on the coarsest sieve from the coal FA contributed more to the sample's total carbon than for the co-fired ashes, whereas the carbon retained in the 45-150 μm particle range had a higher contribution for the co-fired ashes than for the coal FA.
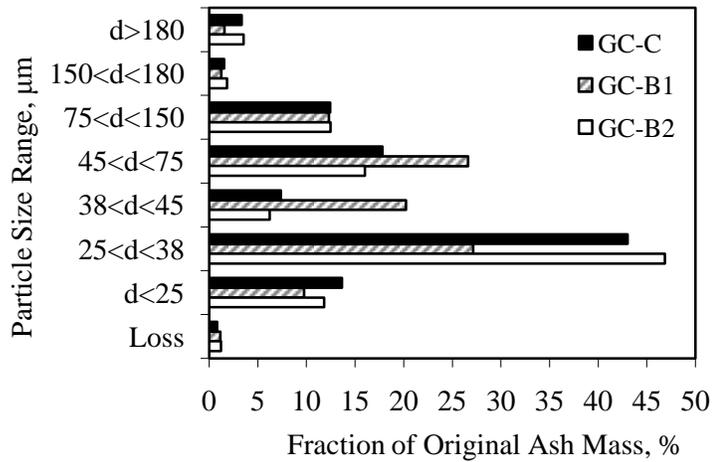
Fig. 2. Particle Size Distributions for GC Samples. Loss Can be Attributed to Particles Lost During the Sieving Process
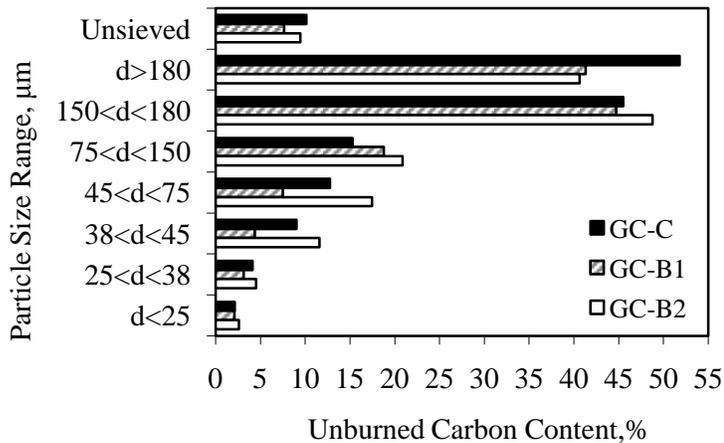
**Fig. 3. Unburned Carbon Content (TOC) of Different Size Fractions**
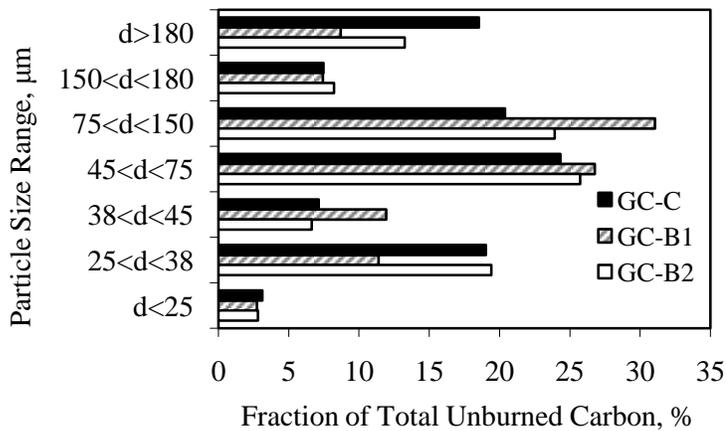
Fig. 4. Contribution of Each Fraction to Total Unburned Carbon (TOC)
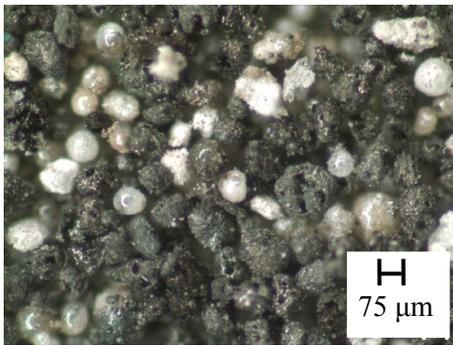
Mitigation investigation

The size and carbon distribution results were then utilized to assess whether sieving to reduce the unburned carbon content of the GC samples could be used to reduce their LOI and to improve the foam index results. Trends in the data were observed to determine the size of the sieve to use for separation. Passing the FA through the No. 200 sieve (75 μm) results in a 35% average reduction of unburned carbon content while only retaining less than 20% of the FA. Instead, passing the FA through the No. 325 sieve (45 μm) reduces the unburned carbon content by an average of 55% from the original amount, but also retains nearly 40% of the FA. The unburned carbon content refers to the mass of the carbon passed through the sieve divided the mass of the FA passed through the sieve. Therefore a reduction in carbon mass is offset by the subsequent reduction in overall mass and a limit is reached where reduction in unburned carbon content is less than the reduction in mass passing each consecutively smaller sieve. For the purposes of this study the No. 200 sieve was chosen to utilize the most FA with the greatest reduction in unburned carbon content.

The LOI of the three GC samples were reduced by an average of 35.2% (with a standard deviation of 1.1%) after processing through the No. 200 sieve. Two samples, GC-C and GC-B1, were reduced to below the 6% ASTM C 618 limit. Oxide analysis on the processed FA revealed that the main oxide concentrations increased by over 4% on average, the calcium content increased by over 11% on average, and the alkali content increased by over 9% on average when compared to the unprocessed FA. This indicates that the finer FA particles contain more of these chemical components than the coarser particles. ASTM C 618 does not mention if processed FA is acceptable for concrete use, yet the standard only places a maximum limit on particle size and, consequently, making a FA that currently meets this limit finer is acceptable. On the other hand, EN-450 does permit the usage of processed FA (e.g., through sieving, blending, etc.) in concrete.

Figure 5 shows the specific foam indices of the GC samples before and after the partial removal of unburned carbon particles. Processing results in an average reduction of 13.6% in AEA demand, a value less than half the corresponding reduction in unburned carbon content. Furthermore, sample GC-C after processing has a specific foam index four times that of EC-C despite having comparable LOI values. This demonstrates that processing coal or co-fired FA through removing large carbon particles may not be a productive technique in reducing AEA demand even though standard LOI limits are met. This result is similar to those found in previous studies for coal FA [Kulaots 2004; Sporel 2007]. Freeman et al., [1997] proposes that the external particle surface area is most accessible and, consequently, AEA adsorption will likely increase with decreasing particle size and increase with increasing macro-porosity. The majority of carbon removed during the sieving process was large char particles seen in the optical microscope image in Figure 6 mixed in with glassy inorganic particles. However, much of the fine carbon may be in the form of soot, which has been shown to adsorb AEA 2-5 times more than the char particles on a mass basis [Gao 1997]. The structure and surface chemistry of the unburned carbon, even more than surface area and particle size, can control its reactivity towards AEA [Hill 1997]. Therefore, alternative mitigation techniques should be examined.
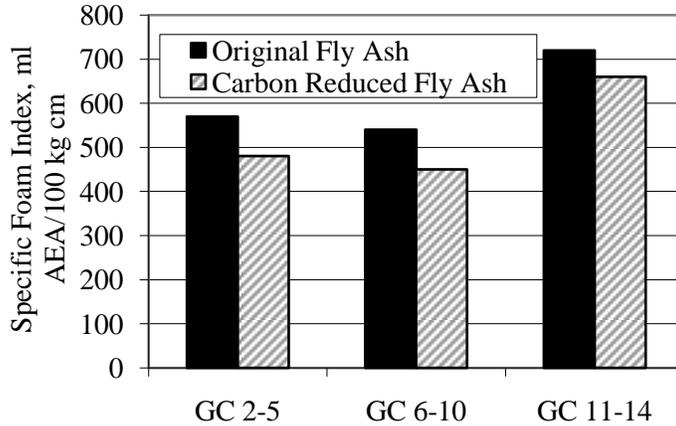
Fig. 5. Change in the Foam Index After Partial Removal of Unburned Carbon Particles Retained on No. 200 Sieve

Fig. 6. Optical Microscope Image of GC-B1 Fraction Retained on No. 200 Sieve

CONCLUSIONS

This study investigated differences between coal FA and FA derived from the co-combustion of coal with biomass (wood). No correlation between biomass content and increased sulfur trioxide levels, increased LOI, or increased alkali content was found. All FA mixes increase by 200-2300% AEA demand, as measured by the foam index test, as compared to the pure cement mix. The AEA demand appears to be correlated to higher LOI for these samples and does not appear to be effected by biomass content. The high LOI coal and co-fired FA samples exhibit similar size and carbon distributions. However, co-combustion may result in finer carbon particles possibly residing in the 45-150 μm particle range. Likewise, the largest portion of the carbon in all samples is located between 45-150 μm in size. The low mass contribution of the largest size fraction restrains its overall carbon contribution despite having the highest unburned carbon content. A mitigation technique to lower the unburned carbon content of high LOI samples through dry sieving reduced LOI within standard limits. However, foam index testing revealed that processing coal or co-fired FA through removing large carbon particles may not be a productive technique in reducing AEA demand. This is likely the result of only removing the large unburned carbon char particles found in both co-fired and coal ash while leaving the finest fraction of carbon (predominantly in the form of soot), which is known to possess the largest AEA demand. Overall, the conclusions made in this study demonstrate that the co-firing of wood

with coal to produce co-fired FA results in minor differences compared to coal FA with respect to AEA demand. Furthermore, this study showed that the exclusion of co-fired fly ash from the US standards may be inappropriate with respect to chemical, physical, and durability requirements.

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