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Automated Foam Index Testing: A Quantitative Approach to Measure the Capacity and Dynamics during Air Entraining Agent Uptake

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ABSTRACT

It is shown that quantitative uptake capacities and interaction dynamics of air entraining agents (AEA) with combustion fly ash and cement-ash mixtures are possible to obtain through the automated acquisition of foam index values. Automation includes adding controlled amounts of water and AEA, measuring bubble stability after intense agitation of the mixtures, identifying foam index values, and finally draining and washing the sample cell to make it ready for the next test. Because this automated approach enables the real-time detection of bubbles as they burst, it is also possible to compare bubble stability after titrating AEA into cement-ash mixtures with corresponding changes in surface tension of the liquid covering the mixtures. These tests showed that the generation of bubbles and their stability were associated with changes in liquid layer surface tension. The potential of applying the automated foam index test (AFIT™) to control air content in concrete with and without fly ash is also examined relative to using the C231 ASTM testing procedure on concrete mixes.

1. INTRODUCTION

The dual hydrophilic/hydrophobic nature of surfactants is beneficially exploited in many industrial processes and formulations, and in cleaning and foaming applications. In the US concrete industry, surfactants called air entraining agents (AEA's) are commonly used in concrete mixes to instill small, stable air bubbles that increase workability and resistance of the set material to freeze/thaw cycles [1-3]. Control of the proper dosing of AEA into a concrete mixture starts at the ready mix plant and ends at a construction site where contract specifications are to be met. It is not surprising that manufacturers and suppliers of AEA's do not specify exact quantities of AEA to be used in concrete mixes for meeting contracted air contents because of the multitude of factors affecting its efficacious use [4-6]. Using appropriate amounts of AEA is imperative: too much creates excessive air void volume which can decrease concrete strengths to levels below contract specifications.

The increased use of pozzolanic mineral admixtures like coal combustion fly ashes as mineral admixtures in concrete add to the complexity of properly dosing AEA. This issue is becoming more critical as more combustion fly ash is used in concrete, i.e. over 15 million tons during 2005 in the US [7], where it is well known to beneficially impact the physical, chemical, economic and environmental aspects of concrete [8]. Nevertheless, the constituents within fly ash, particularly the unburned carbon - as usually measured by loss on ignition (LOI) measurements - has been described to absorb AEA more aggressively than do the cement, sand or aggregate constituents [9-13]; thereby, the creation of the required air content when using fly ash may be completely altered unless AEA dosages are changed as compared to when not using fly ash. Furthermore, although ASTM C-618 [14] specifies limits typically applied in the US on the LOI of fly ash to be used as a mineral admixture, AEA demand in concrete mixes may be substantially different when two different fly ashes are admixed into the concrete even though they have identical LOI's.

Some work has been done to develop spectroscopic methods that quantify AEA uptake by fly ash and cement-ash mixtures [11, 15]. In an ultraviolet-visible spectroscopic (UV-Vis) procedure, the latent liquid on an ash or cement-ash mixture is removed after mixing it with a prescribed amount of AEA, and then the liquid is analyzed relative to known AEA-liquid concentrations. Another method that has been used for is the foam index test [16]. As commonly practiced, dilute ash-water mixtures with or without cement are incrementally dosed with AEA, in between which the foam created on the surface of the mixture is observed for stability and/or uniformity. Studied comprehensively with a goal of establishing a standardized approach for foam index applications in the concrete industry [12], significant insight was generated that focused on the influence of carbon physical and chemical properties relating to AEA adsorption.

From a theoretical perspective, the foam index test should be capable of quantifying the capacity of constituents to absorb AEA within cement, ash and mixtures thereof, if subjectivity could be eliminated, because when absorptive sites are saturated with surfactant or other surfactant-uptake reactions are complete [15] any additional adsorption of AEA on these sites should be hindered. Then, AEA available in excess of site saturation is available for lowering the surface tension of the water within the mixture and, upon aggressive agitation, metastable bubbles will begin to form on top of the mixture.

The metastability of the bubbles suggests AEA adsorption onto the solid surfaces is a dynamic process, as has been discussed [15,17], and requires an equilibration time greater than that for decreasing surface tension within the liquid layer. Upon saturation of the AEA absorptive sites, bubble stability on the liquid surface should be dramatically improved because the surfactant is able to maintain a decreased surface tension in the liquid. Although bubble bursting is still observed at these higher AEA dosages, and may be partially a result of liquid drainage and wall thinning, their stability improves to the extent that multiple layers of bubbles begin to be observed on top of ash-cement-water mixtures. The visual foam index test has its origin within these physical/chemical considerations.

Nevertheless, whether visual foam index testing actually determines the ultimate uptake of AEA on fly ash or cement-ash mixtures has been seriously questioned [11,12]**.** This research, using UV-Vis spectroscopic and surface tension procedures, suggested the time of AEA equilibration with solid adsorption sites was between 15 seconds to greater than 30 minutes. Other research pointed to the possibility that it may be as long as one hour [11]. Such differences may be related to physical and chemical differences of the samples studied because diffusion within particles control AEA equilibration [9-13,18] as does the formation of semi-stable salts during the interaction of AEA with free Ca^{2+} and Mg^{2+} ions [15]. However, UV-Vis and surface tension techniques or the manual foam index tests methods used to date have not dynamically measured AEA equilibration times.

A potential approach to increase the applicability of foam index testing is to develop automated and non-intrusive instrumentation that eliminates visual observation of bubble instability. Fortunately, bursting bubbles are well known to emit sound or acoustic emission (AE). By combining AE hardware with computer controlled agitation, AEA titration and AE data collection, subjectivity could be eliminated and, possibly, quantitative analytical information acquired.

This manuscript describes the development of an automated foam index test (AFIT™) instrument that is based on detecting sound waves from bursting bubbles. It presents a comparison of AFIT-derived foam index data with surface tension data; describes the need to test cement-ash mixtures rather than just ash by itself; discusses the dynamic and analytical capacity of AFIT that provides a measure of AEA saturation of adsorption sites within dilute fly ash and cement-ash mixtures; and elucidates differences in equilibration times and dependencies of foam index values on AEA dilutions used during titration.

2. EXPERIMENTAL

Table 1 lists the LOI's of the some of the Class F combustion fly ashes examined by use of the AFIT™ instrument. All ashes were obtained from commercial sources, had LOI's between 0.7%-6.9%, and were tested by themselves and in cement (Type I/II Portland cement)-ash mixtures. The AEA's used were sodium dodecylbenzenesulfonic acid (SDBS) from Aldrich, sodium lauryl sulfate from Fisher Scientific, vinsol resin (VR) from Master Builders Company, Cleveland, OH, and MicroAir® (MA) also from Master Builders; the AEA dilutions were between 1:1 water:AEA to 200:1 water:AEA.

A picture of the AFIT instrument is presented in Figure 1. A non-intrusive detection configuration was used in which an acoustic sensor (Physical Acoustics Corporation, Princeton Junction, NJ) was placed on the outside of an AFIT test cell; this cell, in which

Table 1. Ash labels, LOI's, ash class and AEA's used during AFIT data acquisition.

Sample label	LOI, %	Class	AEA's used
Ash1	4.5	F	VR & SDBS
Ash2	1.7	F	VR & SDBS
Ash3	2.3	F	VR & SDBS
Ash4	5.2	F	VR & SDBS
Ash9	2.9	F	VR
Ash10	4.7	F	VR
Ash11	3.9	F	VR
Ash12	5.1	F	VR & MA
Ash13	6.9	F	MA
Ash14	5.9	F	SDBS
Ash 15	0.7	C	SLS
Ash 16	2.5	C/with Hg sorbent	SLS

ash, cement, water and AEA are mixed, is contained within a sound-proof enclosure when a cover is closed. The cell has a stir propeller within it that is connected to a motor mounted onto the cell lid; it is also connected to tubing from computer-controlled pumps that enable dosing of water or AEA, and the draining of the ash-water mixtures upon test completion.

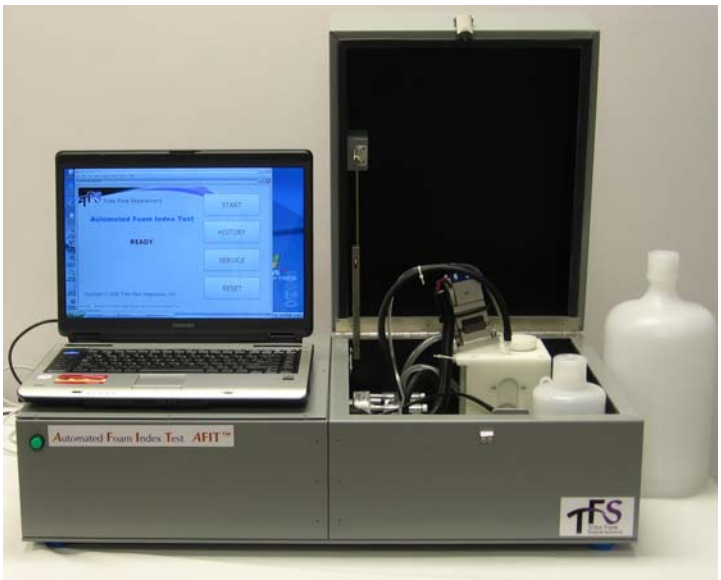

Figure 1. Overall view of the AFIT instrument with labtop computer.

Output of the acoustic sensor is connected through a sound board; software enables the counting of the number of bursting bubbles in the cell - each burst creates an acoustic wave (or Event in the following data) that is detected by the sensor. A plot of Events-versus-ml of added AEA is displayed during each test and upon conclusion of which the foam index value is displayed in user-requested units such as ml AEA-per-kg or drops AEA, etc.

For the tests described herein, the amount of fly ash or cement-fly ash used during each test was 10, 20, 40 or 80g and the amount of water added was 200 ml; these values mean the solids/water mass ratio was between 1/20-to-8/20, i.e. 0.05-to-0.40. After each test, a computer-controlled drain and rise cycle is activated that washes the cell making it ready for the next sample. Computer control allows AEA titration levels to be accurately set or changed and the period of AE data collection after each titration to be precisely set or changed. In the tests performed, it was typical to use AEA-water titration levels between 0.05-5 ml.

2.3 Surface tension tests

Surface tension measurements on water and fly ash-water mixtures were accomplished using a rather simple approach that relied on measuring the force needed to pull an immersed needle [19] from the mixtures as the amount of titrated AEA was increased. Both SDBS and VR AEA’s were titrated into 200 ml of deionized water with or without 80g of fly ash. Prior to testing, the SDBS and VR were diluted 100:1 (water:AEA) to accomplish water surface tension testing whereas the VR was diluted 4:1 (water:VR) to accomplish water-fly ash surface tension testing.

First, the force needed to pull the immersed needle out from either the water or water-fly ash samples without AEA was measured. The AEA was then titrated into the liquid, followed by complete mixing for 1-2 minutes without creating surface bubbles, and then the mixture was allowed to equilibrate for one minute before the force needed to pull the needle out was again measured. Up to 15 titration steps were performed during each test. Because the absolute value of the force for water-fly ash mixtures was affected by ash particles floating on top of the water and which were attached to the needle as it was pulled from the mixtures, reported herein are relative surface tension values, i.e. force after AEA titration divided by the force when no AEA was present.

2.4 ASTM C231 testing

Air content measurements of freshly mixed concrete followed the ASTM C-231 standard [20] while using a Type B concrete air meter (Gilson Company, Inc). Portland cement (Type I/II) and All Purpose Sand and Gravel (meeting ASTM C-33 specifications) were acquired from a local retail establishment. Mixtures of these components with and without combustion fly ash were made while using a ratio (cement plus ash)/water = 1.77. These mixtures were placed in the concrete air meter and air contents measured, establishing average air content values from three individual tests.

3. RESULTS AND DISCUSSION

3.1 Water:AEA dilution and sample mass

The potential of acquiring foam index values that are linearly dependent on AEA dilutions or the mass of the samples tested is an important issue related to data repeatability and the transfer of foam index values from one test site to another. These repeatability questions were addressed by acquiring AFIT data on the ashes shown in Table 1, some plots from which are presented in Figures 2-4.

In Figure 2 are displayed foam index curves for Ash13 when using 2:1 H_2O:MA and 4:1 H_2O:MA; the dashed vertical lines within the plots signify the peak of the foam index Events. For 2:1 dilution the foam index was 27.5 ml/20g; for 4:1 dilution the foam index was 45 ml/20g. The ratio of the diluted AEA between these two tests was 2:1/4:1 = 3/5 = 0.6; the ratio of the foam index values from Figure 2 was 0.61.

Table 2 presents foam index values for cement-Ash1 mixtures when using different water:VR dilutions. By plotting the foam index values for 4:1 and 2:1 dilutions relative to the 1:1 values, as displayed in Figure 3 for Ash1, it was possible to determine whether diluting the AEA affected the absolute foam index values based on ml of pure AEA. In both figures the abscissa represents the foam index for 1:1 VR and the ordinate represents the foam index in ml of solution for more highly diluted VR.

The linear fits to data in Figure 4 have slopes of 1.57 for 2:1 H_2O:VR and 2.42 for 4:1 H_2O:VR; if foam index values were independent of the dilution the theoretical slope values would be 1.5 and 2.5, respectively. In other words, foam index values calculated on the basis of the amount of pure AEA were independent of the dilution of the AEA that was used during titration.

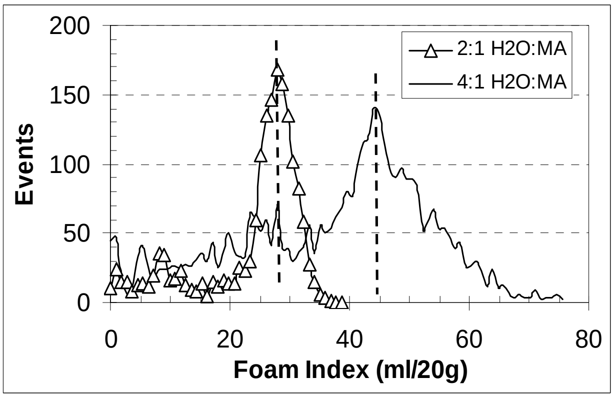

Figure 2. Comparison of foam index values values for Ash13 when different AEA dilutions were used.

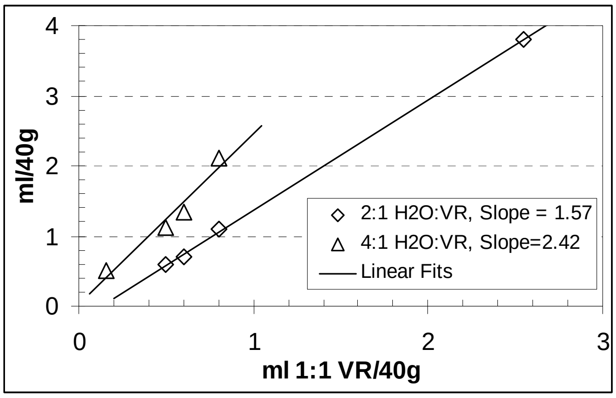

Figure 3. Comparison of foam index for cement-Ash1 mixtures for 1:1 H_2O:VR relative to 2:1 and 4:1 dilutions.

AFIT acquired foam index values were acquired using Ash16 with 0.2% SLS as the AEA and the sample mass for the tests was changed between 10-80g. As displayed in Figure 4, a linear fit proportional to sample mass represented the increasing foam index values to a very high degree.

Table 2. Foam index values (ml AEA + water) per 40 g of sample for cement-Ash1 mixtures using different dilutions of water:VR.

	VR dilution		
% Ash1	4:1	2:1	1:1
100		3.80	2.55
80	2.10	1.10	0.80
50	1.13	0.70	0.60
30	0.70	0.60	0.50
20			0.30
0	0.5		0.16

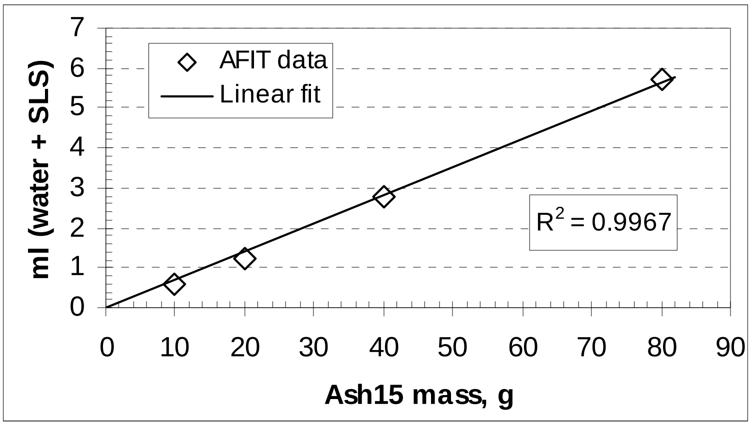

Figure 4. Foam index values increase linearly with sample mass for Ash15 using SLS AEA.

These outcomes, i.e. linearity in the foam index values with change in sample mass and change in AEA dilution, provides a quantitative assurance about AFIT measurements, enables the transfer of foam index values to sites other than the one at which samples were tested, and provides greater flexibility in acquiring foam index values especially for samples that have either very low propensities to absorb AEA, like cement or some pozzolans, or very high propensities to absorb AEA, like ashes with high LOI's. With low AEA absorbing materials, the establishment of high-quality foam index data can be accomplished by using high AEA dilutions or large sample sizes. For high AEA absorbing materials, the time required to acquire AFIT results can be minimized by using less dilute AEA and/or smaller samples sizes.

3.2 Surface tension

The surface tension testing was accomplished using water by itself and ash:water mixtures. In general, surface tension showed an initial rapid decline followed by a plateau in which very small decreases occurred. For water by itself, a value of 0.1 ml SDBS or VR was the estimate for achieving a critical micelle concentration (CMC) which gives the CMC as 0.47 g/l in water; previous data for SDBS has determined CMC values between 0.24-1.82 g/l [17].

Figure 5 compares AFIT AE data with relative surface tension data when VR was titrated into a water-Ash1 mixture. In agreement with surface tension and foam index data for water only: the number of AE Events began to increase as the liquid surface tension began to decrease; the number of Events went through a maximum as the surface tension was decreasing; the number of Events decreased to lower values and then leveled concomitant with a leveling in the relative surface tension. Therefore, the rise and decrease in AE Events from water and water-ash mixtures with AEA titration are associated with the decrease in surface tension of the liquid and the eventual attainment of CMC levels.

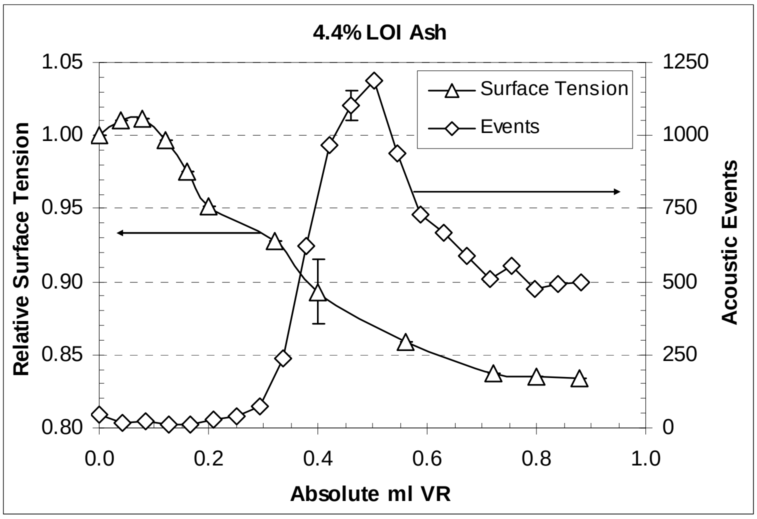

Figure 5. Relative surface tension of and AE Events from water-fly ash mixtures.

3.3 AFIT data of cement, fly ashes and cement-ash mixtures

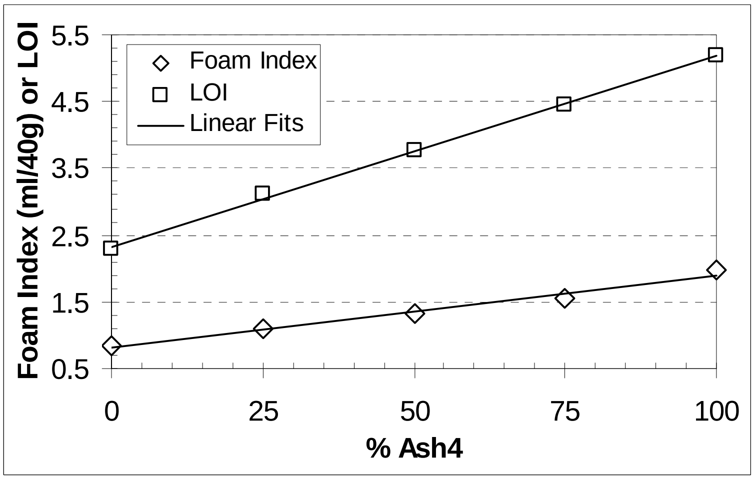

Figure 6. Foam index values from AFIT and the LOI's of Ash3-Ash4 mixtures.

Figure 6 shows that foam index values of 100% Ash3, 100% Ash4 and mixtures of Ash3 and Ash4 samples are linearly related, and linearly increase with increasing LOI contents as the percentage of Ash4 attained the 100% level. Linearity in foam index values with increasing LOI values suggests that the carbon content in the Ash3-Ash4 mixtures controlled their foam index values and that chemical interactions other than carbon with the AEA did not control AEA uptake. To examine the potential of measuring other chemical interactions, the AFIT instrument was used to test ash-cement mixtures.

Figure 7 displays AFIT foam index curves for 100% Ash 11, 100% Portland cement and mixtures of Ash11-cement. Portland cement absorbed considerably less AEA than did Ash 11. if the concentration of unburned carbon was the only factor affecting these AFIT-measured foam index values, as was observed for ash-ash mixtures in Figure 6, then the foam index values of a 70% cement:30% Ash11 mixture and a 20% cement:80% Ash11 mixture should have been linearly related to the LOI contents of the 100% cement and 100% Ash11 samples. However, as seen in Table 3, if the foam index values were calculated from a linear extrapolation between 100% cement-to-100% ash, significantly greater values values would have been obtained than were measured by using the AFIT instrument. Because the amount and type of carbon increased proportionally with the percentage of ash in these mixtures whereas the AFIT foam index values did not increase in the same proportion, it is proposed that components other than carbon and interactions other than with carbon [10-12, 15] also influence the foam index values in cement-ash mixtures.

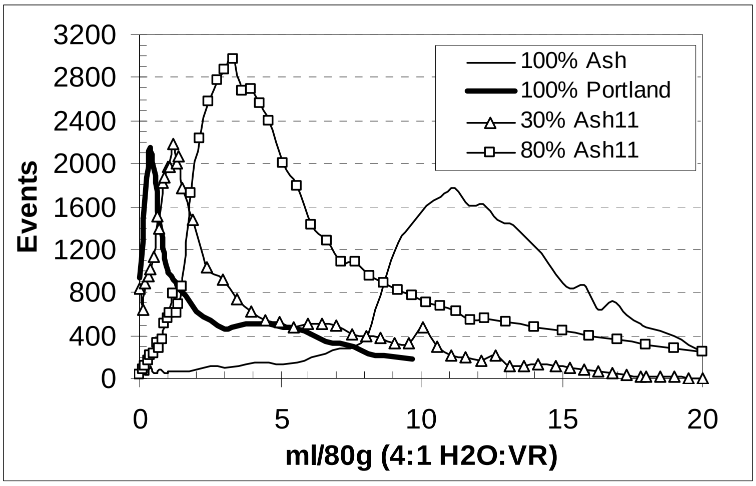

Figure 7. Foam index curves for cement-Ash11 mixtures.

Table 3. Measured and calculated foam index values for cement-ash mixtures: calculated values assume that carbon contents would control AEA uptake.

Sample	LOI Content %	AFIT Foam Index, ml/80g	Foam Index, ml/80g, extrapolate from LOI
100% Cement	0.10	0.6	
70% Cement:30% Ash11	1.17	1.5	3.6
20% Cement:80% Ash11	3.12	4.1	9.6
100% Ash	3.90	12.1	

Although the presence of free, finely-divided Ca^{2+} and Mg^{2+} specie have been proposed to diminish AEA foaming in ash-cement mixtures [15], their contribution would be primarily dependent on the cement content in the mixtures examined. Because the foam index value of 100% cement was more than 20 times less than of 100% Ash11, it is expected that the influence of Ca^{2+} or Mg^{2+} species was relatively small relative to the influence of carbon in this sample. Nevertheless, some form of cement-ash interaction had to occur to diminish the effect of carbon on AEA uptake in the cement-ash mixtures.

The overall practical effect of these AFIT data confirms the need to use cement-ash mixtures rather than just ash by itself during foam index testing if proper AEA dosages for concrete mixes were to be predicted.

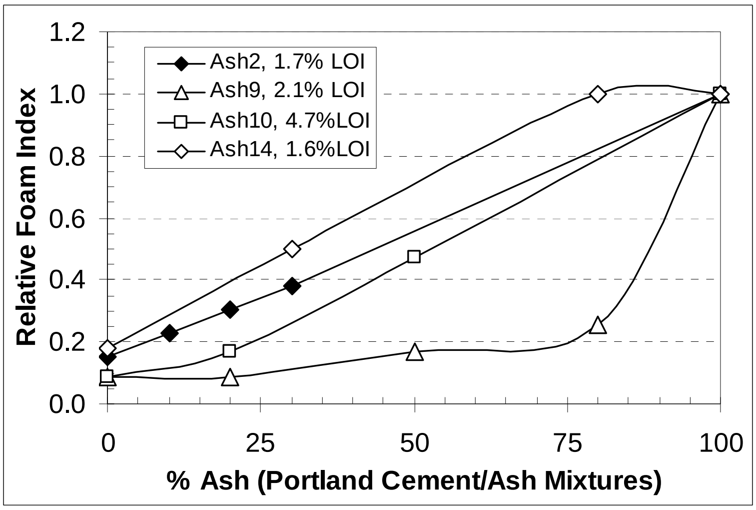

Figure 8. Relative foam index values of four different cement-ash mixtures.

The potential of chemical interactions changing the value at which stable bubbles form can be readily seen by the foam index data in Figure 8, where relative foam index values are presented - the foam index value for each mixture divided by the foam index value at 100% ash - because the absolute foam index values were widely different for the four mixtures. Only cement-Ash2 mixtures had a linear change in AEA uptake with increasing ash content; for the other cement-ash mixtures no linear relationship between ash content or mixture LOI was possible. In other words, bubble stability was established at AEA contents either well-below or slightly greater than would have been predicted by LOI contents.

All of the data for the Class F ash-cement mixtures in Figure 8 had foam index values that were greater than the foam index of the Portland cement. However, as shown in Figure 9, in which are plotted foam index values of two Class C ash-cement mixtures, it is also possible to decrease the foam index below that of the Portland cement. This possibility occurs even if the ash by itself had a foam index greater than the cement. In other words, the amount of AEA required to create stable bubbles, i.e. to instill the proper air content, in cement-ash mixtures is often rather complex and not readily understood by examining the ash LOI or even the foam index values of the ash by itself.

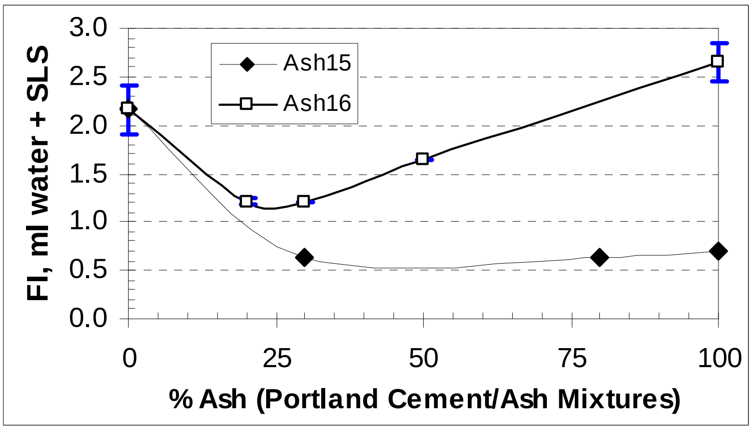

Figure 9. Foam index values for Class C-cement mixtures.

3.4 AFIT foam index values and C231 data

As noted by Baltrus, et al. [15], it may be possible to lower the adsorption capacity of cement for an AEA by substituting an ash that replaces or interacts with free Ca^{2+} and Mg^{2+} from the cement. This statement was based on visual foam index and UV-Vis data that concluded the presence of these free ions was more important in AEA uptake than the presence of unburned carbon, whereas other publications have focused on the role of unburned carbon [10-13]. From the data in Figures 7-9 it is obvious that foam index values of cement-ash mixtures do not always increase linearly with increasing fly ash content and that cement-ash interactions may influence the absorption of AEA by the unburned carbon.

To further examine the practical effects of such behavior, AFIT derived foam index values in cement-ash mixtures were used to determine the amount of AEA to be added to concrete mixes, and then these mixes were subjected to measurement by using the ASTM C231 air content method [20]. Shown in Figure 10, the C231-determined air contents for cement-Ash1 mixtures using 0%, 20% and 30% fly ash were approximately constant at 7.4%, 7.2% and 6.9, respectively; also shown is the air content of a concrete mix without AEA added. If the amount of AEA added had been estimated to be linearly dependent on Ash1 content instead of from measured foam index values, the amount of AEA used for the 20% and 30% admixtures would have been ~2 times larger than the values actually used, leading to significantly greater concrete mix air contents. For cement-Ash2 mixtures (Figure 11), the amount of AEA to be added by AFIT measurements was determined to increase linearly with ash concentration. Consequently, the concomitant C231 air contents were found to be approximately constant at 7.4%, 8.4% and 8.4%, respectively. Therefore, by measuring the foam

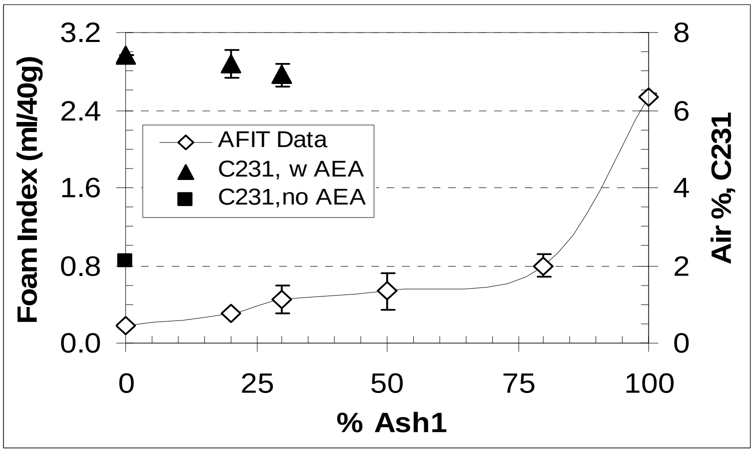

Figure 10. Foam index values and C231 air contents of cement-Ash1 mixtures.

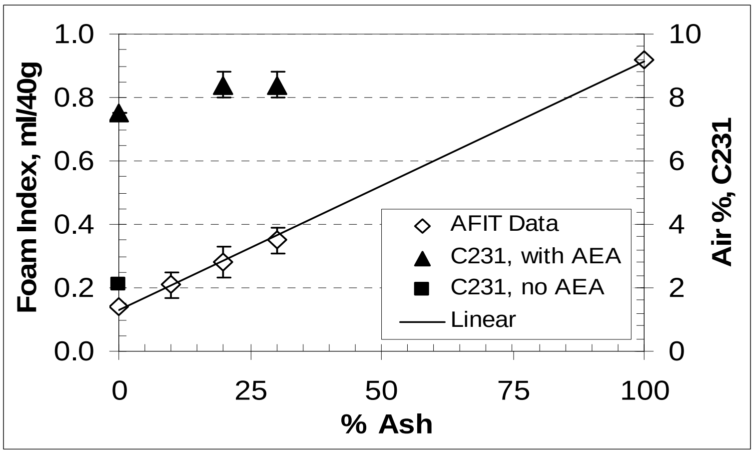

Figure 11. Foam index values and C231 air content of cement-Ash2 mixtures.

index values of cement and cement-ash mixtures using the AFIT method it was possible to instill constant C231 air contents when ashes were used as mineral admixtures in concrete mixes.

4. CONCLUSIONS

Automation of the foam index test was accomplished by detecting acoustic waves emanating from a container in which cement-ash mixtures were placed and into which water was added, AEA was dosed and sample agitation was performed using precise computer control. The real-time nature of the acoustic emission from this AFIT instrument provided dynamic assessments of foam stabilities for ash and cement-ash mixtures even with different AEA's. Shapes of the AFIT curves as a function of increasing AEA dosages were experimentally correlated with changes in liquid surface tensions. These interactions were independent of the AEA dilution used during the AFIT testing as long as AEA equilibration with solid adsorption sites was met. It was also possible to measure dynamic adsorption and equilibration of AEA onto the cement and ash surfaces. These data suggest previous discussions about the relative importance of unburned carbon versus free Ca^{2+} and Mg^{2+} surface sites on AEA uptake is ash dependent and not exclusive to either carbon or free ions independent of each other. Nevertheless, it was possible to instill constant wet concrete air contents when substituting up to 30% ash for cement in concrete mixes if the AFIT determined foam index values were used as the measure for the amount of AEA to be added to the concrete mixes. Overall, AFIT operation instilled quantitative foam index measurements independent of the type of AEA or the types of ashes used during testing.

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