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An Investigation into the Effects of Fly Ash on Freeze-Thaw Durability Prediction

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An Investigation into the Effects of Fly Ash on Freeze-Thaw Durability Prediction

A thesis submitted in partial fulfillment
of the requirements for the degree of
Master of Science in Civil Engineering

by

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University of Arkansas
Bachelor of Science in Civil Engineering, 2018

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ABSTRACT

Air is purposefully entrained into concrete primarily to improve resistance to freeze-thaw deterioration while saturated with water. Air entraining admixtures (AEAs) are chemical admixtures designed to entrain air into the concrete to provide adequate resistance to the effects of freezing and thawing. One of the challenges associated with air entrainment in concrete is the interaction of an AEA with supplementary cementitious materials present in the concrete, particularly fly ash. Fly ash is a by-product of the coal fired electrical generation industry, and often contains residual unburned carbon and other components that can increase the AEA demand of a particular concrete mix. Properly estimating the amount of AEA required to reach the specified air content in a concrete containing fly ash is of utmost importance to the ready mix concrete supplier, as an insufficient air content may lead to job site rejection and the resultant monetary losses.

This study aimed to better relate fly ash and concrete properties obtainable prior to final concrete placement, to direct measures of concrete durability obtainable only after the concrete has set and been put into service. A new device known as the Super Air Meter (SAM) was studied concurrently to better examine the relationship between its System Air Metric number (SAM number) and hardened concrete durability properties. Generally, fly ashes with higher foam index values and surface areas required higher dosages of AEAs to reach a specified air content value. No such relationship could be determined with the more commonly available loss on ignition percentage of the fly ash. The SAM number correctly predicted an acceptable spacing factor in 9 out of 12 different concretes tested, although all of the concretes tested displayed poor performance in freeze-thaw durability. These results demonstrate that air content testing alone is not necessarily sufficient to ensure high quality, durable concrete structures.

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INTRODUCTION

A typical concrete may consist of coarse and fine aggregates, cement and supplementary cementitious materials, water, chemical admixtures, and air. For many decades, air has purposefully been entrained within concrete mixtures to, among other things, increase resistance to freezing and thawing action while saturated with water. The entrained air bubbles in the fresh concrete leave room for air voids in the hardened concrete, where forces developed due to the freezing and thawing of water can be dissipated. [1]

One of the many variables that must be considered when determining the correct air entrainment admixture (AEA) dosage for a specific air content in concrete is the interaction of the AEA with other components of the concrete mixture. Fly ash, in particular, can have unpredictable effects on the AEA demand. Several test methods exist to analyze and quantify this demand: some methods examine properties of the fly ash only, and others consider both the interaction of the fly ash and the chemical admixture. [2, 3]

Fly ash is a byproduct of the coal fired electrical generation plants that provide much of the power used in the United States. It's used to improve the strength and durability properties of portland cement concrete, repurposing a waste product that would otherwise end up in a landfill. [4] The unburned carbon portion of the ash is mainly responsible for inconsistencies in properties of concrete made with fly ash, particularly the air content. The capacity for adsorption of an AEA by the fly ash is related to the porous surface area of the unburned carbon component of that fly ash. [5] If a fly ash is adsorbing AEA molecules, then there are less available in the aqueous cement paste solution to form the small air bubbles ideal for providing resistance to freezing and thawing action. A greater AEA dosage is needed as a result. This proportion of high

surface area carbon in the fly ash can vary between different fly ashes, but can also vary for a given fly ash source over time.

As specified in ASTM C618, fly ash is classified into two categories, Class C and Class F. [6] Class C ashes typically originate from the burning of sub-bituminous coal. Greater than 18% of the mass of a Class C ash is comprised of calcium oxide. In contrast, Class F ashes typically originate from the burning of higher quality coal, such as anthracite. They contain a maximum of 18% calcium oxide by mass. [6] Other sources of variability in fly ash include the method of combustion, and the type (if any) of preprocessing performed on the coal. Some coal-fired boilers use pulverized coal to increase efficiency, while others blend multiple sources of coal and fire them together. [7] These factors can cause differences in the chemical composition and physical properties of the fly ashes. Particle size and fineness, important characteristics in evaluating the pozzolanic reactivity of a particular fly ash, can also be affected by production methods. [4]

Entrained air has been recognized for many decades as vital to producing concrete that is durable and resistant to freeze-thaw cycles. [1] Air tests, especially as documented in ASTM C231, seek to quantify the air void system through a single parameter, the air content by total volume. [8] While useful, the air content percentage provides little information regarding the size and distribution of the individual air bubbles comprising the air void system in the cement paste. Air bubble size and distribution have a greater effect on the freeze-thaw resistance of a particular concrete than the actual amount of air present in that concrete. A well distributed air void system consisting of small air bubbles is typically more resistant to freeze-thaw action than a poorly distributed one containing a small number of large air pockets, even if the two systems contain the same total amount of air by volume. [9]

The traditional method for determining information about the air void system beyond the air content of fresh concrete is by determining the spacing factor as specified in ASTM C457.

[10] This method has its downsides, however. Despite its usefulness, the spacing factor can only be determined once the concrete has hardened. Specialized equipment for polishing and examining concrete specimens is also required, along with experienced and patient personnel.

Seeking to quantify the air void distribution of fresh concrete, researchers at the Oklahoma State University have developed a device called the Super Air Meter (SAM). Similar in form to a standard Type B air meter, it uses a series of pressure steps to determine the System Air Metric number (SAM number) of the concrete in addition to the air content. The SAM number has been shown to empirically correlate to the ASTM C666 durability factor and the ASTM C457 spacing factor. Additionally, the SAM number can be determined while the concrete is still fresh, so modifications to the concrete mixture can be made prior to final set in the event of an unacceptable test result. [11] The durability factor is a direct laboratory measure of the resistance of a concrete to freezing and thawing action. This test method is useful, but time consuming, and not entirely representative of field conditions that a concrete might experience. The spacing factor is a parameter that characterizes the air void system of the hardened cement paste. It is related to the maximum distance any point in the cement paste could be from the edge of an air void. Determination of the spacing factor takes far less time than the durability factor, and is also correlated to concrete freeze-thaw durability. [10]

Previous testing involving the SAM has generally focused on varying the amount of AEA among similar concretes, then comparing the fresh air properties (air content and SAM number) to hardened properties (spacing factor and specific surface typically, along with the durability factor). These concrete mixes typically contained one type of fly ash, with AEA types and

dosages varying. Low slump (less than 3 in.) concrete was tested primarily. [12, 13] This study investigated a wider variety of fly ashes to determine their effect on AEA performance and the SAM number. Additionally, this work investigated concrete with a 4 in. slump to evaluate the SAM's performance in predicting air void system quality for exposed concrete structures other than pavements (made with low slump concrete), such as bridge decks.

In this study, multiple fly ashes were gathered and a range of physical properties pertinent to concrete AEA demand were investigated. Each fly ash was incorporated into batches of Arkansas Department of Transportation Class S(AE) (Structural Air Entrained) concrete, where fresh and hardened air system parameters, along with freeze-thaw durability, were determined. Conclusions were drawn about what fly ash properties predict the AEA dosage, how the SAM performs with higher slump mixtures and a variety of fly ashes, and the effects of the different fly ashes on freeze-thaw performance.

EXPERIMENTAL PROCEDURES

Fly Ash Physical Property Investigations

12 different fly ashes (7 class C and 5 class F) were obtained from ready-mix concrete plants and regional coal fly ash producers in Arkansas and Oklahoma, with additional samples obtained from power plants in Texas, Illinois, Kentucky, Alabama, and Missouri. These are representative of fly ashes used in concrete construction projects throughout the south and central portions of the United States. All fly ashes conformed to ASTM C618 according to the manufacturers, however additional laboratory testing was performed upon arrival. [6] This additional testing consisted of loss on ignition, foam index testing, and Brunauer-Emmett-Teller

(BET) surface area analysis. Table 1 summarizes the results of these test methods to further characterize the fly ashes beyond the standard ASTM C618 classification.

Table 1. Fly Ash Properties and Information.

Fly Ash No.	Source	Class	LOI (%)	BET Surface Area (m ² /g)	Foam Index (mL AEA/g FA)
1	Newark, AR	C	0.376	1.655	0.26
2	Marissa, IL	F	0.569	0.907	0.01
3	Redfield, AR	C	0.619	2.866	0.32
4	Springfield, MO	C	0.549	1.560	0.05
5	Louisville, KY	F	1.559	1.561	0.26
6	Ghent, KY	F	2.993	2.712	0.31
7	Red Rock, OK	C	0.407	0.965	0
8	Gentry, AR	C	0.474	1.082	0.02
9	Oologah, OK	C	0.323	0.909	0.01
10	Sikeston, MO	C	0.592	3.398	0.28
11	Wilsonville, AL	F	3.370	2.001	0.35
12	Franklin, TX	F	0.057	0.641	0

Loss on Ignition

Loss on Ignition (LOI) is primarily an indicator of the unburned carbon residue remaining within a fly ash. Other volatile materials, usually in minor quantities, may be burned off during the ignition process as well. [14] LOI for each of the 12 fly ashes used in this study was determined according to ASTM D7348 Method A. [15] Approximately 1 g of each fly ash was weighed to the nearest 0.1 mg and placed in a heat resistant crucible capable of withstanding temperatures over 750 °C. Crucibles and samples were placed in a muffle furnace and heated up to 750 °C incrementally over the course of 2 hours. After two more hours in the furnace, samples were carefully removed and allowed to cool. They were then weighed to the nearest 0.1 mg and

the LOI value was calculated. Each fly ash was tested twice. Two samples were taken from each source of fly ash with the average reported as the LOI percentage. None of the fly ashes tested exceeded a LOI value of 6%, meeting the requirements of ASTM C618 for both Class F and Class C fly ashes. Fly ash samples were not oven dried prior to ignition, though they were stored in tightly sealed containers in an indoor environment to minimize temperature and humidity effects.

Foam Index

The foam index (FI) method seeks to quantify the ability of a given combination of fly ash, portland cement, and AEA to maintain a stable amount of entrained air. [16] Many different test procedures for determining the FI exist today. [2, 5, 17, 18] Recently, an ASTM standard test procedure for foam index testing was approved under the designation ASTM C1827, however, this standard was published after the conclusion of FI testing performed here. [18] The procedure used in this study was similar to one recommended by GCP Technologies [19], and conformed to ASTM C1827, except horizontal agitation of the sample was used instead of vertical agitation.

The procedure used in this work is as follows: a base FI was first determined using only portland cement. 10 g of cement was placed into a 60 mL cylindrical jar along with 25 mL of distilled water. The jar was then capped and agitated for 60 s using an automatic shaker table set to 280 oscillations per min. The jar was uncapped and a single drop (0.02 mL) of an aqueous solution of 5% AEA was added using a pipette gun. A standard pipette could also be used; however, the pipette gun was much more reliable in terms of drop size and drop stability. Regular pipettes were difficult to manipulate and resulted in inaccurate dosages of AEA.

The jar was recapped and placed on the shaker table for 15 s, where it was then removed and inspected for foam presence and stability. A foam was deemed stable when the top surface of the liquid within the jar was covered in a stable layer of air bubbles that remained for at least 45 s. Air bubbles tended to form along the sides of the jar at first. As more AEA solution drops were added, the entire surface of the liquid became covered with air bubbles. Representative comparisons of various foam stages can be found in Figure 1. The number of drops added to the slurry solution was recorded as the FI value. FIs can be standardized so comparisons can be made across different drop sizes and AEA solution concentrations. This is done by determining the amount of pure AEA added and dividing it by the total mass of fly ash or reactive constituents present in the slurry. Typical values in tests performed ranged from 0.02 to 0.16 mL AEA / g FA depending on the fly ash (FA) used. These results are consistent with other combinations of AEA and fly ash in the literature. [5, 16] FI values are variable due to the inherent variability of fly ash and the subjectivity associated with determining the correct endpoint of the test.



Figure 1. Comparison of Foam Index test jars at different AEA dosages. The two jars on the left have just reached the FI, while the far-right jar has greatly exceeded it, resulting in a large foam buildup.

BET Surface Area

BET nitrogen gas adsorption testing was performed to determine the specific surface area of each of the 12 fly ash samples. This step was performed because it was assumed that the surface area of the fly ashes may have some correlation to their absorptivity. [20] Using nitrogen gas, fly ashes were degassed at 200 °C for approximately 24 hr prior to testing. Weights taken before and after degassing ensured that only water vapor and the atmospheric gases within the glass container were removed. Care was taken so that elutriation did not occur as the nitrogen gas passed through the sample container.

Following degassing, samples were weighed carefully to determine the mass of fly ash to the nearest 0.1 mg. It is important to limit the amount of atmospheric gases that enter the sample container during this step, especially if the BET instrument has two separate stations for

degassing and adsorption testing. Each sample was kept at 77 K for the duration of the test using liquid nitrogen. While some fly ashes returned adequate results (a C constant less than 1000 and a positive y-intercept on the BET linear plot) in the typical BET relative pressure range of 0.05 to 0.3, others did not and required modification to the relative pressure values. These modifications placed the relative pressure as low as 0.0075. BET surface area values can be found in Table 1. BET surface area values of North American coal fly ashes are typically less than 10 m²/g [20, 21], although higher values have been reported. [5]

Concrete Batch Preparation

The mix design for the concrete used in this study is given in Table 2. The mixtures were proportioned to conform to Arkansas Department of Transportation concrete specifications. [22] The total cementitious materials content was 611 lb/yd³, with a water to cement ratio (w/c) of 0.42 and a fly ash replacement percentage of 20. Coarse aggregate was crushed limestone with a nominal maximum aggregate size of 3/4 in., while the fine aggregate was Arkansas River sand (fineness modulus 2.6). Commercially available Type I/II portland cement was used. A polycarboxylate based liquid high range water reducing admixture (HRWR) was used to bring all concrete mixes up to the desired slump of 4±1 in. A commercially available saponified rosin based liquid air entraining admixture was used as the AEA in all concrete mixes.

Table 2. Concrete Mix Proportions. *Dosage is given in units of fl. oz. / 100 lb. total cementitious materials (cement + fly ash).

Cement	489	lb/yd ³
Fly Ash	122	lb/yd ³
Fine Aggregate	1211	lb/yd ³
Coarse Aggregate	1691	lb/yd ³
Water	257	lb/yd ³
HRWR	0.5	fl. oz./100 lb. cm.*

Concrete batches of 1.5 ft³ were prepared indoors (ambient temperature of 72 °F, concrete temperature of 68 °F) using a 3 ft³ drum mixer rotating at approximately 26 rpm. Aggregates, cement, fly ash, admixtures and mixing water were placed in an indoor mixing room overnight to ensure all materials were conditioned to the same temperature. Aggregates were oven-dried for approximately 24 hr and allowed to cool to room temperature before batching. This was done to ensure a consistent moisture content between batches. Additional mixing water was always added to compensate for the absorption capacity of the aggregates.

Materials were batched in the following order: water (with HRWR), cementitious materials, half of the coarse aggregate, half of the fine aggregate, the remainder of the coarse, and the remainder of the fine aggregate. Aggregates were split up in this way to avoid clumps on the sides of the mixer. Before the addition of any aggregates into the mixer, the mixer was turned on to distribute the aggregates evenly in the water and cement slurry. The mixer ran for 3 min, was paused for 3 min, then ran for 2 additional min to fully incorporate the materials together.

Determining AEA Dosages for 6% Air

For each fly ash, a different AEA dosage was required to reach 6% air content in the concrete. 6% was chosen as the target air content largely because it is specified by numerous state and federal agencies, including the Arkansas Department of Transportation, which specifies that Class S(AE) concrete shall contain 6±2% air. [22] The range of acceptable air values in this study was restricted to 6±1% to reduce variability between mixtures.

By assuming a linear relationship between the concrete unit weight and its air content, the theoretical unit weight of the concrete at 6% air can be determined if an initial (prior to addition of AEA) unit weight and air content are known. Immediately after the conclusion of mixing, and

before any AEA was added to the concrete mix, a unit weight test was performed. This was followed by an air test using the Type B air test functionality of the SAM. From these values, a unit weight at 6% air could be estimated, and AEA could be added until this unit weight was achieved.

Typically, the initial air content and unit weight were determined, then a dosage was prescribed, based largely on trial and error, that would reach 6% air in the concrete. AEA was added to the mixer and the concrete batch was mixed for approximately 2 min to fully incorporate the admixture. After the 2 min mixing period, another unit weight test was performed. If this unit weight was within 2 lb/ft³ of the theoretical unit weight at 6% air, a Type B air test was again performed to confirm the fresh air content. An aggregate correction factor was not used to best represent the actual state of practice. [23]

If an air content within the 6±1% range was obtained, a SAM test was performed immediately following the air test to determine the SAM number in accordance with AASHTO TP-118. [11] The SAM number has been correlated to indicators of concrete durability such as the spacing factor and durability factor. [12, 13] While the SAM test was performed, several prisms were made to further investigate the concrete air void system after the concrete had hardened. Two 3 in. x 3 in. x 11.25 in. prisms were cast for hardened air content and spacing factor testing and three 3 in. x 4 in. x 16 in. prisms were cast for freeze-thaw testing.

Hardened Air Void Investigations

The 3 in. x 3 in. x 11.25 in. prisms were cured in a lime water solution at 70 °F inside an environmental chamber for at least 2 weeks before further processing. A 1 in. thick slice was sawn from the center of each prism by clamping the prism onto a concrete saw and making one

cut down the center. This resulted in two halves of approximately 5.625 in. in length. The half with the least amount of chipping damage around the edges was selected and from it, a 1 in. section was cut. Careful attention was paid to reducing chipping, mainly by reducing the feed rate of the saw as the blade approached the final edge of the concrete.

One surface of the resulting 1 in. thick section was then polished using a series of abrasive sanding disks (80, 140, 200, and 270 grit) on a flat lapidary polishing wheel. An apparatus was set up to continuously rotate the samples while the abrasive disk spun on the sample face to avoid any potential wear marks or streaks on the finished surface. Every sample was coated in a mixture consisting of 20% lacquer and 80% acetone before each new grit to protect smaller voids on the surface. The lacquer/acetone mixture was allowed to dry to the touch before moving onto polishing. Each sample was polished for approximately 10 min before moving onto a finer grit of sanding disk. Red construction crayon marks were drawn on the samples before each run on the polisher in a 0.25 in. grid to ensure that all areas of the concrete were polished sufficiently.

Once the 270 grit was finished, and both the coarse aggregates and the cement paste were polished adequately, the samples were placed in 100% acetone for 20 min to remove any remaining lacquer and construction crayon. Black felt tip markers were used to paint the polished surface black. Two layers of black ink were applied in this manner, with the ink allowed to dry for over 6 hr in between coats. Acrylic ink was used instead of black marker on half of the samples. This produced a much darker, matte surface without any streaks, and is recommended for any future investigations. Two applications of the acrylic ink were used. Finely powdered wollastonite was used to fill in the voids on the blackened surface by pouring it on then wiping off the excess with the palm of the hand. Samples were examined under magnification to ensure

all voids were filled. At this time, any voids lying within aggregates, either coarse or fine, were colored in using a black marker. Only the voids in the paste were relevant to this analysis.

Digital scans of each prepared sample were made in 8-bit grayscale at 3200 dpi, and run through a MATLAB analysis script by Fonseca and Sherer. [24] Threshold values distinguishing between black and white on the scanned image, indicating what is an air void and what isn't, averaged out at 165 for the samples prepared using black marker, and 100 for the samples prepared using an acrylic ink. Threshold values were determined for each sample by comparing the diameter of any given air void from the grayscale scanned image to the diameter of the same air void on the fully black and white processed image. Once the two diameters were approximately equal, the proper threshold value was known. [24] After processing, hardened air void parameters of interest, such as the spacing factor and hardened air content, are determined using the MATLAB script in accordance with ASTM C457 Procedure C. [10] Results are shown in Table 3.

Table 3. Air Void System and Durability Parameters. The acceptable range for fresh air contents was $6\pm 1\%$. *This value does not meet the requirements for a valid SAM number in AASHTO TP – 118.

Fly Ash No.	SAM Number (psi)	Fresh Air (%)	Hardened Air (%)	Spacing Factor (in.)	Durability Factor
1	0.25	6.0	5.8	0.009	42
2	0.61	5.1	4.0	0.013	38
3	0.22	6.6	5.0	0.009	28
4	0.48	5.6	4.7	0.010	32
5	0.23	5.7	3.9	0.011	46
6	0.36	6.8	5.0	0.009	42
7	0.22	6.0	4.7	0.009	48
8	0.18	6.5	5.3	0.008	47
9	0.22	6.7	6.3	0.007	29
10	0.25	7.0	4.8	0.011	39
11	0.40	6.8	5.8	0.008	17
12	*0.06	6.2	5.4	0.010	50

Freeze-Thaw Durability Investigations

During the concrete batching process, samples were made for freeze-thaw investigation via ASTM C666. [25] Each sample was cured in limewater at 70 °F for at least 3 weeks prior to testing. Some samples were up to 6 weeks old at the start of testing.

A standard freeze-thaw chamber conforming to ASTM C666 Procedure A was used, capable of testing up to 17 individual concrete prisms, with one control sample. [25] Each sample's cross-sectional dimensions, length, weight, and fundamental transverse frequency were measured before the start of testing. [26] 30 freezing and thawing cycles were performed with the samples in a submerged condition. Each cycle lasted approximately 4 hr, alternating between 0 °F and 40 °F. The chamber was checked daily to ensure the concrete prisms remained submerged and that there weren't any major temperature variations from one side to the other.

Once a sample's relative dynamic modulus of elasticity had fallen below 60% of its initial value, it was removed and a spare prism of the same nominal dimensions and density was used to fill that gap in the chamber. It was hoped that each concrete specimen would reach the desired 300 cycles without falling below the 60% threshold, however, no specimen made it past 240 cycles. Final durability factors are shown in Table 3.

RESULTS

Indicators of Required AEA Dosage

Loss on Ignition

LOI is a relatively simple and straight forward test to perform, and is often done by fly ash manufacturers as a quality assurance test, with results made available to concrete producers. Past studies have suggested a relationship between LOI and the amount of activated carbon components in the fly ash and hence the dosage of AEA required to reach a given air content in concrete. [16] More recent studies have demonstrated that there is no significant relationship between LOI and AEA dosage in concrete. [21, 27] The results of this study similarly show no relationship between LOI and AEA dosage, as shown in Figure 2. Further, these results indicate that the portion of the fly ash burned off at 750 °C has minimal effect on the AEA demand.

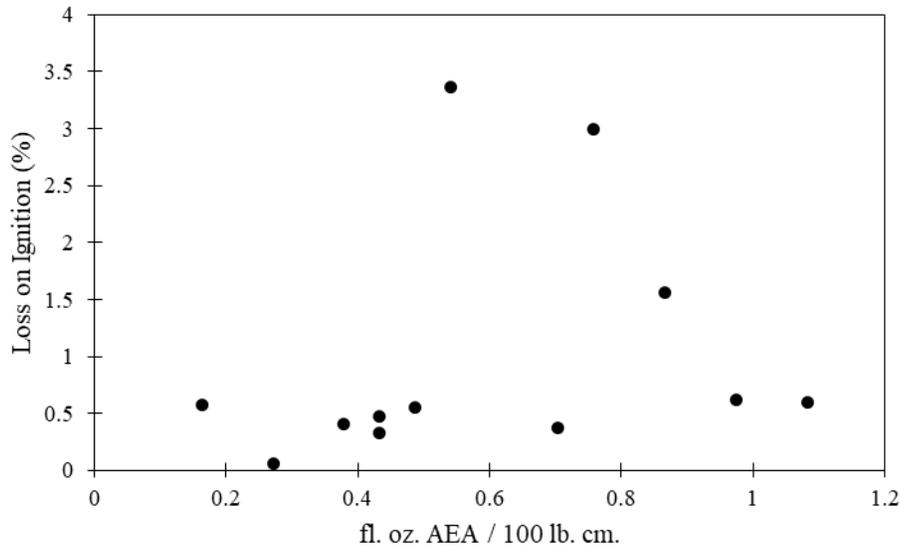


Figure 2. Comparison of Loss on Ignition and AEA dosage requirements.

Foam Index

The FI is unique among fly ash characterization techniques performed here in that it includes the combined effects of the fly ash, cement, and AEA that is to be used in the concrete. When compared to AEA dosage, there was good correlation using the methods prescribed in this study, as shown in Figure 3. The FI test has limitations however, due partially to the subjectivity involved in determining the endpoint of the test. It isn't always obvious when the foam layer on the surface of the cement slurry solution has stabilized. Because of its simplicity, operators of the FI test may not receive adequate instruction or guidance on what constitutes a stable foam. The recent publication of ASTM C1827 should help fill this knowledge gap. In the meantime, the FI test can provide useful information within individual laboratories and concrete ready-mix producers. It can be used to make comparisons for guiding trial batches as part of a more robust concrete mix design evaluation program.

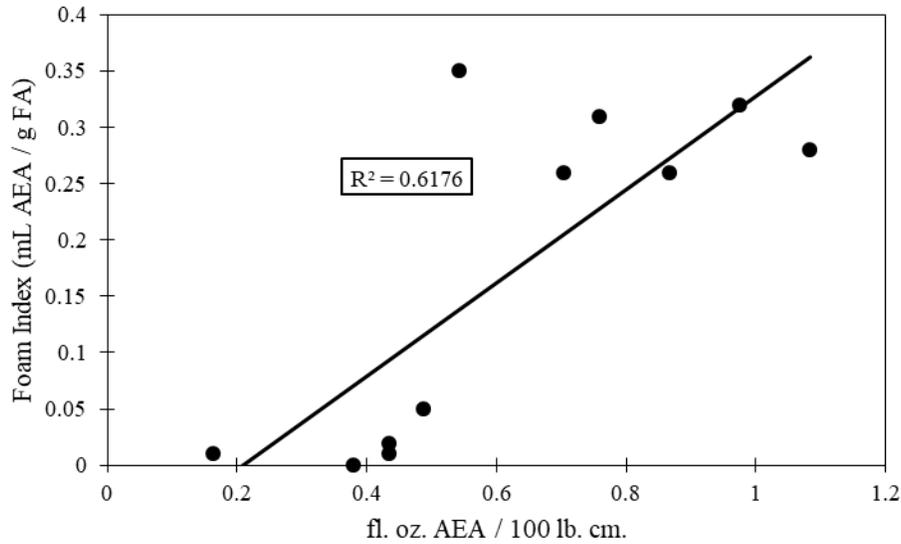


Figure 3. Comparison of Foam Index values and AEA dosages to reach 6% air.

BET Surface Area

Specific surface area provided the best correlation with AEA dosage of the fly ash compared to the other testing performed in this study. As measured using BET nitrogen gas adsorption, an increase in surface area results in a greater AEA demand and consequently, a higher dosage of AEA to achieve a 6% air content. Figure 4 displays the results of the BET analysis compared to AEA demand measured in concrete trial batches. Higher surface areas provide more locations for AEA molecules to interact with the fly ash. This in turn makes less of the AEA available for interaction with the cement paste and water, limiting the quantity of small air bubbles that are desirable for mitigating freeze-thaw deterioration.

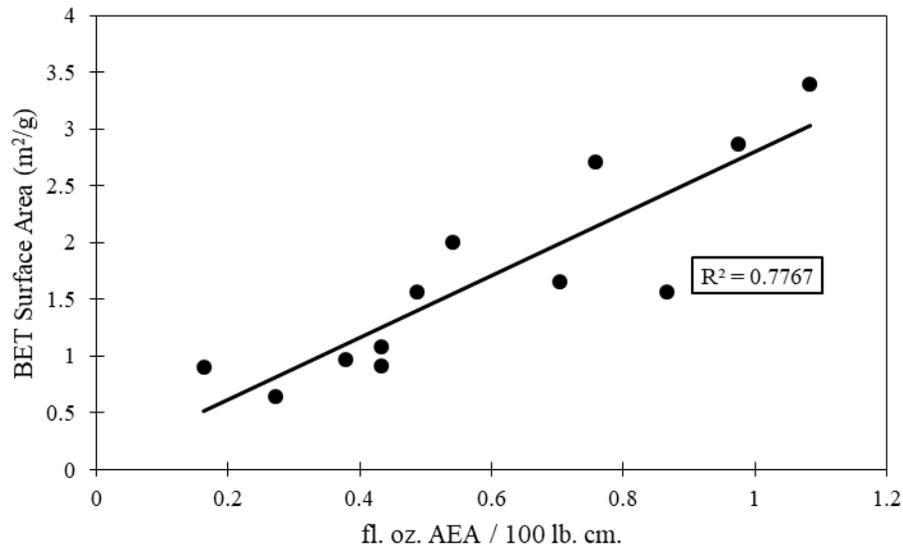


Figure 4. Comparison of AEA dosage to reach 6% air in concrete to fly ash BET surface area.

Results here appear to confirm earlier work done by Ley et al., suggesting a relationship between fly ash surface area and AEA demand in fresh concrete. [27] The higher the surface area per mass unit, the higher the AEA requirements to reach 6% entrained air in the concrete. Rather than the unburned carbon remaining in the fly ash adsorbing AEA, other high surface area components of the fly ash are responsible for increasing the AEA demand. This is why the LOI may not correlate as well with required AEA dosages. The higher the surface area, the more potential there is for AEA molecules to adsorb and the less AEA available in the cement paste to form air bubbles. While BET analyses are likely too complex for a typical ready-mix concrete plant, it can be performed by laboratories to characterize the physical properties of fly ash more effectively than other test methods.

The SAM Number and Measures of Concrete Durability

Spacing Factor

A spacing factor of 0.008 in. or less is correlated with good concrete freeze-thaw performance. [28] One of the purported benefits to performing a SAM test rather than a typical Type B air pressure test is that the SAM number correlates to the ASTM C457 spacing factor. [12] The recommended SAM value correlated to a spacing factor less than 0.008 in. is 0.20 psi according to AASHTO. [11] The relationship between spacing factor and the SAM number was examined in this study, with the results shown in Figure 5. Superimposed on this figure are lines representing a 0.008 in. spacing factor and a 0.20 psi SAM number. Most of the concrete in this study fell within the “bad” quadrant of the graph (top right). These are concretes without sufficiently low (<0.008 in.) spacing factors. The SAM number successfully predicted spacing factors greater than 0.008 in. for all but three of the concrete mixtures tested in this study. For two samples, the SAM test resulted in a false negative, predicting a spacing factor greater than 0.008 in., when the actual spacing factor was greater than 0.008 in. (bottom right of graph). In one case, the SAM test resulted in a false positive (top left of graph) indicating an unacceptable spacing factor when the actual spacing factor was less than 0.008 in.

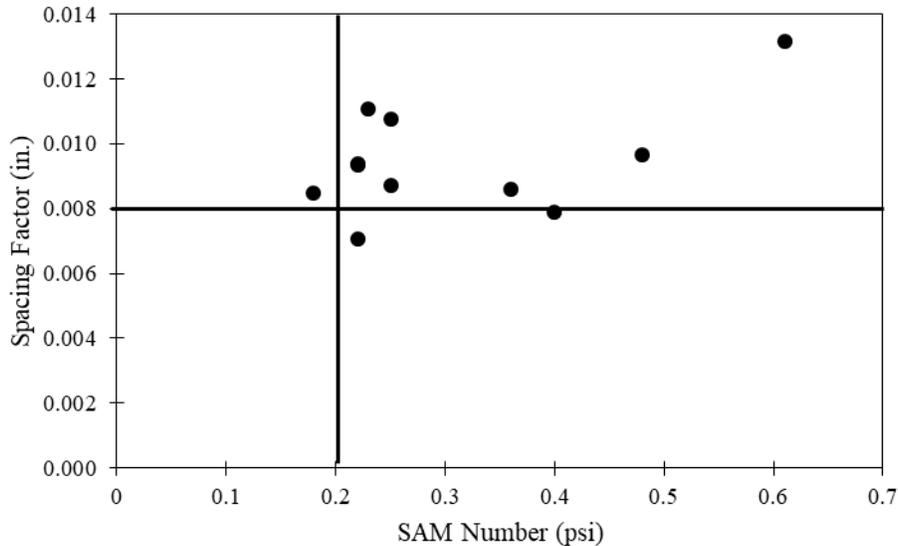


Figure 5. Comparison of SAM Number and Spacing Factor, with AASHTO recommended limitations superimposed at a SAM number of 0.20 psi and a spacing factor of 0.008 in.

The concrete mixtures tested in this study all contained different fly ashes and all contained fresh air contents that would be acceptable for most specifiers. All mixtures failed either the spacing factor limit of 0.008 in. or the SAM number limit of 0.20 psi. This highlights a potential difficulty in specifying a SAM number as part of a performance based specification. Concrete producers and contractors are likely to find existing mixtures with unacceptable SAM numbers despite having adequate total air content.

Durability Factor

The relationship between SAM number and durability factor generated in this study is shown in Figure 6, along with the AASHTO recommended limitations for a durable concrete (a SAM number of 0.32 psi and a durability factor of 70). Despite the inclusion of fly ash, and meeting typical fresh air content limits, the concrete produced in this study provided less than

satisfactory results during freeze-thaw testing. This is attributed to the quality of coarse aggregates used to make the concrete. Numerous pits and voids were discovered in the aggregates during the polishing process for determining the spacing factor. While these voids are not considered when determining the spacing factor, they may contribute to the poor performance of the specimens during freeze-thaw testing. Further evaluation of these aggregates is ongoing. A lightweight chert portion of the aggregate appears to be a contributing factor to poor freeze-thaw performance.

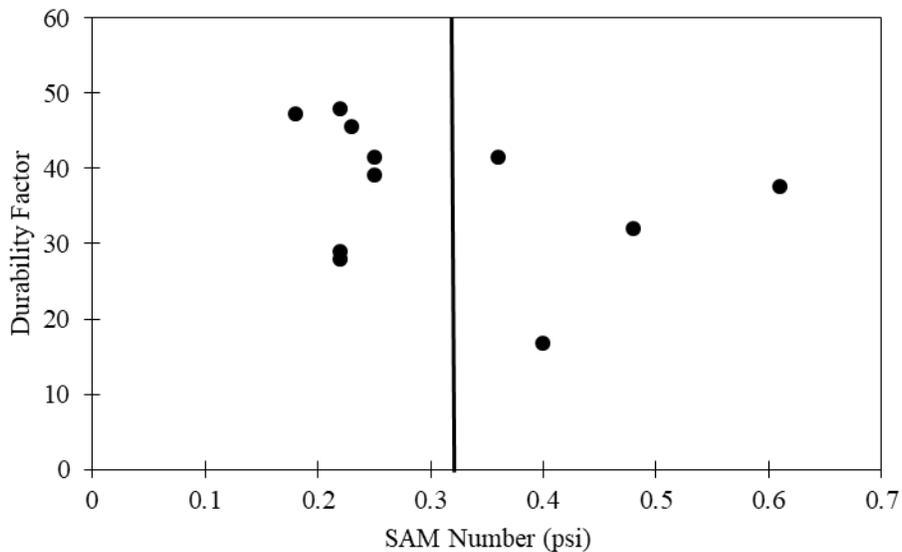


Figure 6. Comparison of SAM Number and Durability Factor, with AASHTO recommended limitations at a SAM number of 0.32 psi and a durability factor of 70 (not shown). No concretes exceeded the durability factor limit.

It should be noted that much of the data forming the relationship between durability factor and SAM number was collected using ASTM C666 Procedure B. [13] The durability factors in this study were determined using Procedure A because the freeze-thaw chamber used in this study was only outfitted to perform Procedure A. Procedure A specifies that prisms are kept saturated with moisture throughout the entire testing process, while Procedure B specifies

alternating between wet and dry conditions. Procedure A is typically considered harsher and more destructive to concrete samples than Procedure B is. [29]

The results of this study demonstrate the complex nature of ensuring adequate freeze-thaw performance in concrete. Fresh air content testing alone does not necessarily guarantee successful resistance to freeze-thaw deterioration, but it is still the most widely specified test method. The SAM number or spacing factor were insufficient to ensure freeze-thaw durability for the samples tested in this study. Fly ash had a significant effect on the AEA dosage required to reach 6% total air content and despite this threshold being reached in all mixtures, differing spacing factors and SAM numbers resulted. Based on this work, it may be challenging for some combinations of fly ash and portland cement to achieve adequate SAM numbers. This work only included one AEA, but included a range of fly ashes on various States' Department of Transportation qualified product lists in the central and southern United States.

CONCLUSION

In this study, concrete was made with 12 fly ashes (both C and F) and the resulting air content (fresh and hardened), spacing factor, SAM number, and freeze-thaw durability performance were measured. Additionally, physical characterization was performed on the fly ashes using LOI, FI testing, and surface area via BET nitrogen gas adsorption. The goal was to better understand the effects of different fly ashes on AEA dosage, determine what physical characteristics of fly ash can be related to their propensity to adsorb AEA, and to examine the freeze-thaw durability of concrete made with this combination of fly ash and AEA. Conclusions are as follows:

1. Loss on ignition (LOI) does not correlate with the propensity of a particular fly ash to adsorb AEA. Unburned carbon content is not the only factor contributing to AEA adsorption by a fly ash.
2. BET nitrogen gas adsorption is an effective method to determine the surface area of coal fly ashes. This surface area correlates well with the amount of AEA that may be adsorbed by a fly ash within a fresh concrete mixture. Future standardized test methods to estimate the adsorption of AEA by fly ash should be compared with BET surface areas.
3. The foam index (FI) is effective at correlating to the amount of AEA adsorbed by a fly ash in concrete. This correlation is less strong compared to the BET method, however, despite the specificity of FIs to both the fly ash and AEA. For a typical concrete supplier, the FI method used in this study may be realistic for regular use if the shaking operation is standardized with an oscillating table and if the amounts of AEA added are measured with a pipette gun or similar device capable dispensing drops accurately and consistently. Training and guidance should be provided from experienced individuals on determining the endpoint of the test as well. The new standard ASTM C1827 test procedure should be incorporated into quality control procedures at ready mix concrete plants. It can be performed quickly and requires little specialized equipment.
4. The SAM number provided a reasonable cutoff beyond which it is likely that the spacing factor of hardened concrete will exceed 0.008 in. This study however included concrete with spacing factors and SAM numbers that were close to the recommended cutoff values (0.20 psi and 0.008 in.). It is not clear how to improve the spacing factor, though in this study there was a relationship between the spacing factor and the total hardened air content. As hardened air content increased, the spacing factor decreased. This could be an effective way to increase

the spacing factor and SAM number of a mixture to acceptable values, but may run into the upper limits on entrained air present in many specifications.

5. None of the concrete mixtures in this study had acceptable freeze-thaw performance by ASTM C666 Procedure A despite the spacing factors and SAM numbers being close to the recommended limits and despite all mixtures having close to 6% entrained air. However, this poor performance may be attributable to the aggregates used, rather than the hardened cement paste in which the air void system resides.
6. The SAM number resulted in two false negatives and one false positive in terms of predicting the spacing factor in the hardened cement paste. Concrete tested in this study had SAM numbers ranging from 0.18 psi to 0.61 psi and spacing factors between 0.007 in. and 0.011 in. despite having similar fresh air content.
7. The fresh air content was 1.2% greater than the hardened air content on average. The hardened air was consistently lower than the fresh air content. Once the aggregate correction factor of 1.2% (measured with two separate air meters) is applied to the fresh air results, this difference disappears, although concretes containing aggregates with such high correction factors are beyond the scope of an air pressure test.

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