Laboratory Study for Comparison of Class C Versus Class F Fly Ash for Concrete Pavement

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16. Abstract

The two objectives of this research were to a) evaluate Class F and Class C fly ash sources for use in paving concrete and b) determine acceptable proportions of each ash type to use in paving mixtures. A combined study was performed involving characterization with respect to AEA adsorption using the foam index test, direct measurement of adsorption, and the iodine number test and a partial factorial experiment was conducted to evaluate concrete mixtures prepared using different fly ash, cement, and aggregate sources. The adsorption testing highlighted known inconsistencies of loss on ignition measurements. Freeze-thaw testing in 4% $CaCl_2$ solutions indicated no statistical difference in performance with or without fly ash but fly ash type was a significant factor in mixtures that failed freeze-thaw testing. Compressive and flexural strength followed the classical behavior expected of fly ash mixtures. Maturity showed the largest impact of fly ash addition. Overall the Class F and Class C ash sources performed well and using either at substitution levels up to 30% is recommended. Additional scrutiny of some Class F sources, with respect to freeze-thaw performance, is warranted. Use at higher levels should be done only after performance testing of the specific combination of materials to be used at the job-mix proportions.

17. Key Words
Fly ash, concrete durability, air entrainment, compressive strength, flexural strength, maturity, direct adsorption isotherm test, coal fly ash iodine number test, foam index test.

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Executive Summary

Currently, Wisconsin Department of Transportation (WisDOT) specifications only permit AASHTO M 295 Class C fly ash for use in portland cement concrete in pavements. Due to concerns regarding air entrainment, AASHTO M 295 Class F fly ash sources were eliminated from WisDOT specifications in the 1990's. With potential changes in the production of fly ash related to the coal sources used, and new unit operations needed to meet changing air quality standards, it is necessary to determine the suitability of current Class C fly ash sources and available Class F sources for use in PCC in pavements. Additionally, it is necessary to evaluate new test methods to help establish the suitability of any fly ash for use in paving concrete.

The two objectives of this research were to:

1) Evaluate several locally available Class F fly ash sources in terms of their potential to impact air entrainment in paving concrete, in comparison with Class C fly ash sources currently in use.
2) Provide mixture design guidance related to acceptable proportions of Class F fly ash that can be used in paving applications without negatively impacting overall performance.

These objectives were met by a combined study where the materials were first characterized with respect to AEA adsorption using the foam index test, direct measurement of adsorption, and the iodine number test. Also as a baseline measurement of fly ash properties, a full ASTM C311 fly ash characterization was performed. Second, a partial factorial experiment was conducted to evaluate mixtures prepared using two (2) different Class C fly ashes, two (2) different Class F fly ash sources, three (3) different portland cement sources, and two (2) different aggregate sources as primary variables. This resulted in a matrix of 42 concrete mixtures with fly ash and six (6) concrete mixtures without fly ash. The concrete mixtures were tested using standard fresh concrete tests (i.e., slump, unit weight, air content) and semi-adiabatic calorimetry, and hardened concrete tests were performed including compressive strength, flexural strength, and freeze-thaw durability (in a $CaCl_2$ solution). Because the experiment was performed as a partial factorial design, the hardened concrete test results were analyzed using a series of statistical analysis procedures.

The adsorption characterization study showed the two Class C and Class F ash sources analyzed had a range of properties. The two Class C ash sources both had a very low loss on ignition (0.2 and 0.3 weight %, respectively) but the ash with the lower loss on ignition actually had the second highest adsorption capacity of the four ash sources tested. For the Class F ashes, which are typically assumed to have a high loss on ignition and adsorption capacity, the loss on ignition values were 0.1 and 2.0 weight %, respectively and their adsorption capacity was in proportion to these values. However, the low loss on ignition Class C ash had an adsorption capacity comparable to the high loss on ignition Class F ash. This shows the need for characterizing fly ash based on adsorption capacity rather than loss on ignition. With respect to impact on air entrainment, the Class F ash sources tested performed in a manner similar to Class C ash currently being used in Wisconsin.

The results of calorimetry testing showed the ash sources follow the classic behavior expected of both Class C and Class F fly ash. Addition of either type of ash reduces the heat of hydration; the reduction associated with the addition of Class F ash is greater than seen with Class C ash. With Class C ash the set time is slightly delayed and the heat evolution is continued over a longer time period within the first 48 hours. Mixtures with Class F ash do not significantly change the heat evolution curve in the first few days other than to reduce the maximum temperature. This is because Class F ash does not react until later in the hydration process. All effects were larger as ash content was increased, regardless of the ash source used.

The results of the testing showed overall no statistically significant differences between concrete mixtures with fly ash or without fly ash with respect to freeze-thaw performance. Most of the mixtures tested performed well. Only 10 of the 48 mixtures had durability factors less than 80% after completion.

However, of the 10 mixtures falling below 80%, 6 had the high loss on ignition Class F ash in the mixture. Only two mixtures were unable to achieve the full 300 cycles of freeze-thaw testing. Given the harshness of testing in $CaCl_2$ solutions, the mixtures performed well over all. However, additional scrutiny of the high LOI Class F ash is required to determine if the poor performance noted was an anomaly of the test, an interaction with the AEA used in the research, or a property of that fly ash source.

When compared to concrete mixtures without fly ash, the measured compressive strength and flexural strength both showed a decrease at early ages that was more pronounced with Class F ash as compared to Class C ash, and more pronounced with higher ash replacements. Both types of ash showed a decrease in the rate of strength gain but the Class C mixtures were comparable to the cement-only mixtures at 14 days age. The Class F mixtures were comparable to the cement-only mixtures by 90-days age. All effects were larger as ash content was increased, regardless of the ash source used.

The maturity testing showed the largest effect of ash replacement. The maturity level required to achieve a specific strength varied widely but not just with the ash content put also the cement type. The type of ash was not statistically significant with respect to maturity, except at early ages (i.e., 1-day).

The major recommendations from this research are:

- Based on this research, there is strong evidence to support using Class F fly ash in paving concrete. The use of Class C ash should also continue. With the exception of freeze-thaw testing, the performance of the Class F ash sources tested was comparable to the Class C ash sources tested. Replacement levels up to 30% can be used without significant changes in fresh or hardened concrete properties.
- Class F ash source F2 demonstrated freeze-taw performance noticeably worse than the other ash sources. Additional testing should be performed to determine if the performance of ash F2 was indicative of the material performance in general, or possibly an artifact of the freeze-thaw testing regime (i.e., Method A with 4% $CaCl_2$ solution), or an interaction with the AEA used in the study.
- Use of either ash type at replacement levels greater than 30% should be considered but performance testing of those mixtures, prepared with the job-mixture materials and mixture design, must be required.
- Based on the adsorption-based tests for characterizing AEA adsorption by fly ash, the Class C ash C2 and Class F ash F2 demonstrated the most likelihood of disrupting air entrainment, although neither significantly impacted air entrainment in this study.
- The existing LOI specification of 2% should be retained but additional testing should be performed to establish the adsorption capacity of any ash used in paving concrete.
- WisDOT should adopt a standardized version of the foam index test as a QC test for concrete producers or field inspectors. It is necessary that a standardized method be adopted to minimize variability. If the intent is to make it a field test, make optional use of the mechanical shaker and allow the operator to perform the test by shaking by hand.
- WisDOT should adopt on a provisional basis use of the direct adsorption isotherm test and the coal fly ash iodine number test. By adopting, on a provisional basis, materials providers and contractors can begin developing a knowledge base of the tests and WisDOT can begin developing a historical database for establishing specification criteria in the future.
- Any construction project using maturity as a means of predicting concrete strength should require periodic and regular calorimetry testing of the cement and fly ash (e.g., each new delivery) to ensure maturity models developed are appropriate considering changes in construction materials

that can occur over a construction season. This should be required for mixtures with or without fly ash as the portland cement is a major factor in establishing the maturity relationship.

- WisDOT should discontinue freeze-thaw testing using $CaCl_2$ solutions in the test procedure. This results in a harsh testing environment, which extends what is already a harsh test (AASHTO T 161) into a more severe environment. Additionally, $CaCl_2$ solutions are corrosive and result in destruction of freeze-thaw chambers. If these solutions are to be used in future research, it is recommended that a 56-day curing regime be used to produce concrete more suitable for sever exposure, especially when the concrete mixtures being tested contain fly ash. Alternatively, a minimum compressive strength could be established for concrete tested for FT performance.

1 Introduction

Currently, Wisconsin Department of Transportation (WisDOT) specifications only permit AASHTO M 295 Class C fly ash for use in portland cement concrete (PCC) in pavements. Due to concerns regarding air entrainment, AASHTO M 295 Class F fly ash sources were eliminated from WisDOT specifications in the 1990's. With potential changes in the production of fly ash related to the coal sources used, and new unit operations needed to meet changing air quality standards, it is necessary to determine the suitability of current Class C fly ash sources and available Class F sources for use in PCC in pavements. Additionally, it is necessary to evaluate new test methods to help establish the suitability of any fly ash for use in paving concrete.

1.1 Research Objectives

The two objectives of this research are to:

3) Evaluate locally available Class F fly ash sources in terms of their potential to impact air entrainment in paving concrete, in comparison with Class C fly ash sources currently in use.
4) Provide mixture design guidance related to acceptable proportions of Class F fly ash that can be used in paving applications without negatively impacting overall performance.

These objectives were met by a combined study where the materials were first characterized with respect to AEA adsorption using the foam index test, direct measurement of adsorption, and the iodine number test. Also as a baseline measurement of fly ash properties, a full ASTM C311 fly ash characterization was performed. Second, a partial factorial experiment was conducted to evaluate mixtures prepared using two (2) different Class C fly ashes, two (2) different Class F fly ash sources, three (3) different portland cement sources, and two (2) different aggregate sources as primary variables.

1.2 Benefits

The benefits gained from this research are primarily economic and environmental. First, reducing the portland cement content in paving concrete by continuing or increasing the use of fly ash, can result in a reduction of the price per yard of concrete as portland cement is the most expensive component in PCC. Although these savings could be significant, a much larger economic benefit can result from an increase in durability for PCC pavements if the fly ash is high quality and is used in the proper proportions. Environmental benefits stem from the fact that concrete mixtures containing less portland cement result in a more sustainable construction material. The production of portland cement produces approximately 3-5% of the anthropogenic CO_2 released annually worldwide, and thus reducing the portland cement content in concrete pavement mixtures will have a positive benefit on the overall production of greenhouse gases.

1.3 Report Organization

- Introduction – Project Introduction, Research Objectives, Benefits, and Report Organization
- Background – Background from the literature review to further describe the research need and methods.
- Experimental Methods – Details of the specific experiments conducted.
- Results and Discussion – Experimental results and discussion of those results.
- Conclusions and Recommendations – Conclusions drawn from the research and recommendations to WisDOT based on this research.

2 Background

2.1 Fly Ash Use in Concrete

Coal fly ash has been used in concrete since the 1930's, with the first published results detailing this use appearing in 1937 (Davis et al.). Fly ash is the airborne residue from pulverized coal combustion processes and is typically collected as part of pollution control efforts by a variety of means including venturi scrubbers, fabric filters, and electrostatic precipitators. These combustion units typically burn pulverized coal as a fuel and, with stable operating conditions and fuel sources, produce a reasonably consistent quality of fly ash.

Two separate mechanisms are responsible for the formation of fly ash particles resulting from coal combustion - fragmented char and condensation of volatilized compounds. The larger particles typically result from the fragmentation of char in the later stages of burnout (Malte and Rees, 1979). Inorganic compounds, which are volatilized in the combustion chamber, will condense onto existing particles as well as form primary particles (Helmuth, 1987), resulting in a large range of particle sizes from submicron to over 1 mm. The actual size distribution of a particular fly ash is influenced by the type and efficiency of the collection system, type of coal, and combustor operating conditions. Particles larger than 45 µm (#325 mesh sieve) are typically non-reactive in concrete (Mehta, 1986) and are considered to contribute minimally to hydration products, but potentially act as filler helping to decrease the porosity of the hardened cement paste and in many cases adding to measured strength gains.

The current AASHTO standard for specifying fly ash for use in concrete is AASHTO M 295 *Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete*. The equivalent ASTM standard is ASTM C618. AASHTO M 295 uses two categories to classify fly ash produced from coal combustion - Class F and Class C. While placing limits on a number of chemical and physical parameters, the singular distinction between the two classes is the combined content of the silica, alumina, and iron oxides, with Class F required to have a minimum 70 wt. % and Class C a minimum 50 wt. %. Historically, this “sum of the oxides” classification would result in a division of fly ashes based on the type of coal combusted, with anthracite and bituminous coals associated with Class F fly ash and sub-bituminous and lignite coals associated with Class C fly ash. In modern power plants, coal fuels are blended and it is difficult to assign a fly ash class to a coal type. Class F fly ash is considered to be pozzolanic whereas Class C fly ash, primarily due to the presence of calcium phases, has cementitious properties in addition to being pozzolanic.

The inorganic (i.e., non-carbon) portion of fly ash is a mixture of crystalline and glass phases whose abundance and composition is greatly influenced by the coal characteristics and the combustion process. Although both phase types are important in determining fly ash reactivity, it is the glass phase that is of importance for determining pozzolanic and hydraulic reactivity (Mehta, 1989). Fly ash is composed of 60% to 90% glass (Roy et al., 1984), but more important than the amount is the structure of the glass. The variation in glass structure is evident between low-calcium and high-calcium fly ash. Low-calcium (i.e., pozzolanic only) fly ash has a highly bonded alumino-silicate matrix that does not react as quickly in concrete. Typically, the pozzolanic reaction for low-calcium fly ash starts after 7 days of hydration (Mehta and Montiero, 2006). Typically strength contributions from Class F ash occur after 28 days of hydration. High-calcium fly ash has a glass with a calcium-modified alumino-silicate structure that starts reacting with the cement pore solution earlier than a low-calcium fly ash. The nature of the crystalline compounds present also varies considerably between Class F and Class C fly ash. Class F fly ash typically contains between 10 wt. % and 30 wt. % crystalline phases composed primarily of non-reactive mullite, sillimanite, and quartz (Mehta and Montiero, 2006). Class C fly ash, on the other hand, typically contains a higher proportion of reactive crystalline components including free lime, anhydrite, tricalcium aluminate, and calcium sulfoaluminate (Mehta and Montiero, 2006). Quartz is the primary non-reactive crystalline phase present in high-calcium fly ash and is present in similar proportions in the low-calcium fly ash.

Another important characteristic of pulverized coal combustion fly ash is the presence of various forms of carbon intermixed with the fly ash. The carbon is present in different forms, broadly classified as either char particles that are typically 5 to 50 μm, or soot and carbon black particles that are typically a micrometer or less in diameter. The combustion of coal is a two-step process where initially gases and highly volatile components are driven out of the coal structure leaving behind a carbon char that burns as a second step. These chars either burn in the combustor, or are carried out of the furnace as un-burnt carbon intermixed in the fly ash (Bailey, 1990). These char particles can take on a wide range of morphologies depending on the coal maceral from which they originated. The texture, porosity and specific surface area of these chars vary with changes in particle morphology. That is, although two different ash sources have the same abundance of carbon, the physical nature of the carbon can be significantly different and the associated properties of that carbon (e.g., adsorption) can be significantly different. The other class of carbon, soot and carbon black, condenses out of the exhaust stream of the combustor as the stream cools in the exhaust path. As a result, process conditions that affect fly ash cooling rate can also affect the physical properties of the ash carbon fraction.

The general concern with carbon in fly ash is two fold. The lesser problem is one of color, which affects architectural applications but is generally not a concern for transportation structures. The second, more serious concern is that carbon can readily adsorb organic chemicals onto its surface. The adsorption potential of carbon is a function of the porosity, surface texture, surface chemistry, and specific surface area of the carbon particle. Specific surface area is inversely proportional to the particle diameter and therefore adsorption by carbon increases with decreasing carbon particle size. Gao (1996) documented the strong correlation between decreasing particle size and increasing adsorption.

The abundance of carbon in fly ash is determined by means of a loss on ignition (LOI) test and there are specified maximum limits for LOI in fly ash. In AASHTO M 295, the limit is 5 %wt. LOI for both Class C and Class F ash. WisDOT specifications limit the LOI to 2 %wt. Although these existing specifications do limit the total LOI in fly ash, as a surrogate for identifying carbon, they do so without consideration for the type of carbon.

2.2 Fly Ash Characteristics Related to Concrete Strength

Fly ash has been used in concrete for many years, but it was not until late in the 1940s that fly ash was widely accepted. Benefits from the use of fly ash include; improved workability, decreased heat of hydration, lower cost concrete, potential increased sulfate resistance and alkali-silica reaction (ASR) mitigation, increased late strength, along with decreased shrinkage and permeability (Schlorholtz, 2006). However, problems can occur when fly ash is used in concrete. The problems include AEA adsorption by fly ash in carbon, ASR accentuated at pessimum replacement levels, slow initial strength gain, and overall variability with fly ash.

The pozzolanic reaction common with all fly ash produces hydration products similar to those of cement but requires a hydroxyl ion source for the reaction to proceed. The basic pozzolanic reaction is shown below in equation 2.1

$$\text{hydroxyl ion source} + \text{pozzolan (silica source)} + \text{water} \rightarrow \text{calcium silicate hydrate} \qquad (2.1)$$

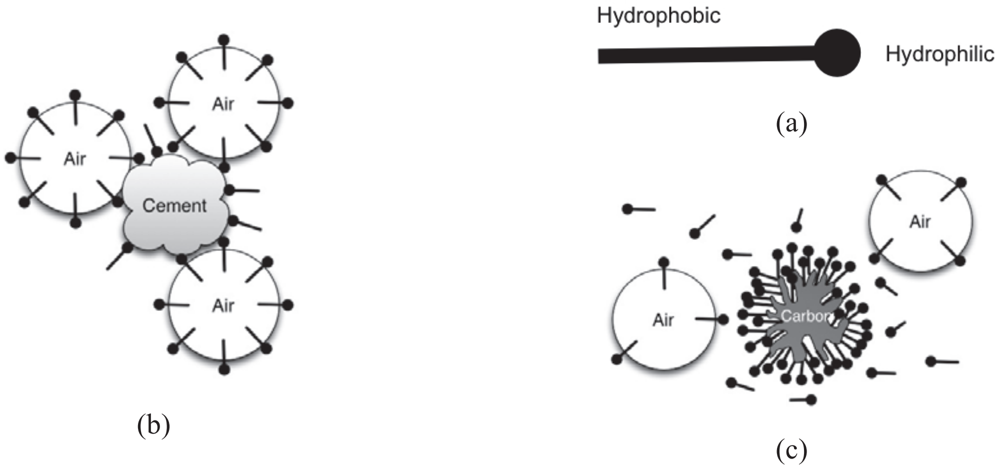
It is typically assumed that calcium hydroxide produced as part of the cement hydraulic reaction acts as the source for the hydroxyl ions. Therefore, cement hydration and the pozzolanic reaction of fly ash are jointly connected. The fly ash can provide several beneficial changes to hardened cement paste: it can react to contribute to the concrete hydration products, it can consume calcium hydroxide during the pozzolanic fly ash reactions, and it can act as a filler decreasing the concrete porosity by occupying capillary pore space. In concrete using fly ash as a partial cement replacement, typically the initial strength gain is delayed while the final strengths are increased. This is more common with Class F fly ash as compared to Class C fly ash. The slow increase of initial strength is believed to be due to chemisorption of calcium ions on the fly ash particles resulting in less initial calcium silicate hydrate formation. The final strength increases are believed to be due to a refinement of the capillary pore spaces and a reduction in calcium hydroxide. Therefore, when comparing Class C and Class F fly ashes, it is important to assess the rate and degree of strength gain and these characteristics are dependent on the fly ash, the cement, and the degree of fly ash replacement in the concrete mixture.

2.3 Fly Ash Characteristics Related to Air Entrainment

Air is entrained in concrete to create a protective network of closely spaced air voids in the hardened cement paste. These voids are spherical in shape and range in diameter from less than $4x10^{-5}$ to 0.05 inch (< 0.01 to 1.25 mm). Air entrainment is accomplished through the use of air entraining admixtures (AEAs) added during concrete mixing. In addition to protecting the hardened cement paste from freeze-thaw attack, entrained air also improves the workability of the fresh concrete, significantly reducing segregation and bleeding.

AEAs contain surface-active agents (surfactants) that concentrate at the air-water interface, and lower the surface tension of the water. Consequently, they reduce the energy required to form the bubbles and break them down into smaller bubbles. The natural tendency of small air bubbles in the absence of surfactants is to coalesce to form larger bubbles, because the surface energy of the former is higher, and any system seeks its lowest surface energy. The surfactants form an elastic film around the air bubbles that reduces the risk of coalescence when collisions occur during mixing (Pigeon, 1995; Mindess et al., 2003).

AEAs are composed of molecules that have a negatively charged hydrophilic (water-loving) end, often referred to as the head, and a hydrophobic (water-hating) end often referred to as the tail. This is shown schematically in Figure 2.1a. In the concrete mixture, these molecules tend to align at the air-water interface with their hydrophilic groups in the water (adsorbed to cement grains) and the hydrophobic portion in the air, thereby effectively binding the air voids to the cement grains (Pigeon, 1995, St. John, 1998, Mindess et al., 2003). This is shown schematically in Figure 2.1b.



(a)

(b)

(c)

Figure 2.1. Schematic drawings of a) an AEA molecule, b) the mechanism of air-void stabilization in concrete, and c) the mechanism of AEA adsorption on carbon (Sutter et al., 2013a). Used by permission of the National Academies.

While in aqueous solution, AEAs may adsorb to carbon, affecting the AEAs ability to stabilize air voids in concrete (Freeman, 1996). This is shown schematically in Figure 2.1c. If a significant amount of AEA is adsorbed to the carbon, the AEA aqueous concentration is reduced to a point where the AEA is no longer able to stabilize the air voids, reducing the amount of air entrained and altering the air-void size distribution as bubbles coalesce. The AEA needs to reside at the interface of air bubbles and water in the plastic concrete mixture to stabilize the bubbles and form air entrained concrete. The result is that carbon can interfere with the air entrainment process in concrete and lead to freeze-thaw durability problems.

When AEA is adsorbed on carbon in fly ash, a higher quantity of AEA needs to be added to a concrete mixture to ensure the desired air-void system formation. It should be noted that carbon can occur in fly ash not only from combustion, as previously described, but carbon is also added as powdered activated carbon (PAC) for pollution control measure.

2.4 Tests to Asses Fly Ash Characteristics Related to Concrete Strength

Strength tests have always played a role in the acceptance of fly ash for use in concrete. To evaluate and classify an ash, strength testing is conducted on mortar cubes containing specific amounts of portland cement and fly ash and is referred to as the strength activity index (SAI). This test has been criticized because at the existing specification limit of 75%, inert materials have been shown to pass the test (Sutter et al., 2013). Additionally, the test evaluates the fly ash at only one replacement level (i.e., 20 %wt.) and therefore does not provide guidance on mixture design.

Ultimately, mixture designs using the job mixture of cement and fly ash need to be evaluated for compressive or flexural strength. Because of the variability that exists, evaluating strength as a function of time is also necessary if early age predictions of strength are necessary for construction (e.g., form removal, construction traffic).

Calorimetry is increasingly being applied to concrete and concrete materials as a test to monitor and assess the hydration process. Calorimetry monitors a concrete mixture's temperature and a heat evolution curve can be developed indicating the extent of the hydration reaction at a given point in time. Three principal approaches to calorimetry are isothermal conduction, adiabatic, and semi-adiabatic calorimetry. In isothermal conduction calorimetry, the heat of hydration is directly measured by monitoring the heat flow from the specimen with both the specimen and surrounding environment maintained at approximately constant temperature. In adiabatic calorimetry the specimen chamber is well insulated in an attempt to maintain no heat loss from the chamber during curing. The temperature increase of the specimen results from the heat produced during hydration. In the semi-adiabatic method, some heat loss is tolerated and the heat flow from the reaction chamber is monitored. The sum of the heat loss and the temperature increase of the specimen are taken as the measure of heat generated in the hydration reaction. Obviously, final strength and the rate of strength gain are determined by the degree and rate of cement and fly ash hydration.

Maturity testing combines physical testing (i.e., compression testing) with measurement of heat evolution in the concrete mixture. Maturity accounts for the combined effects of temperature and time on the strength development of PCC (Carino, 2001). The underlying assumption is that a given concrete mixture achieves approximately the same strength at the same maturity level, expressed in units of temperature-time, for any combination of temperature and time that constitute that value of maturity (Carino, 2001). The standard practice of determining maturity is ASTM C1074 *Standard Practice for Estimating Concrete Strength by the Maturity Method*. The method describes two ways to express maturity, the time-temperature factor and the equivalent age. The time-temperature factor assumes strength gain is a linear function of temperature while the equivalent age approach assumes that maturity is a non-linear function that follows the Arrhenius equation for chemical reactions (Carino, 2001). A strength-maturity relationship for a given mixture is established by making concrete cylinders of the mixture and embedding temperature sensors in at least two cylinders. The monitored cylinders and additional cylinders are cured under the same conditions and the temperature of the monitored cylinders is recorded over 28 days. At time intervals of 1, 3, 7, 14, and 28 days, the additional cylinders are tested for strength. The relationship between strength and maturity is plotted providing a means of estimating strength of the mixture at any given maturity value. Often this process is simplified by plotting the strength versus the log maturity, which yields a straight line that can easily be fit by linear regression. The practical application of maturity is to monitor the temperature of field-placed concrete over time, calculate the maturity value and from that, estimate the in-place strength without need of performing destructive strength tests. With respect to PCC mixtures containing fly ash, the maturity function is important to understand for each specific combination of cementitious materials as each combination of fly ash and cement may yield different strength maturity relationships, even when the different fly ash and cements are proportioned in the same ratios.

2.5 Tests to Assess Fly Ash Characteristics Related to Air Entrainment

One test used to determine the performance of a fly ash with regards to air entrainment, is the foam index test. While not yet standardized, various versions of the foam index test have been used to test performance of a given fly ash with a combination of air entrainer and cement. In one version, 20 g of cement is placed in a 125 mL glass jar. 50 mL of water is added to the jar, which is then capped and shaken for 1 minute. Next, diluted AEA solution is added in small increments of 2 to 5 drops at a time. After each addition, the jar is re-capped and shaken for 15 seconds. The stability of the foam is observed. The minimum amount of diluted AEA needed to produce foam that is stable (i.e., bubbles exist over the entire surface) for 45 seconds is the foam index of the cement-only slurry. Then, repeat steps 1 through 4 using 16 g of cement and 4 g of fly ash to develop the foam index of the cement and fly ash mixture.

The foam-index test is a quick test to show possible changes in the amount of air-entraining agent required when using fly ash in the concrete. Since it only tests a single cement and a single source of fly ash at a time, the results apply only to that combination (Schlorholtz, 2006). One problem with the test is the adsorbate (i.e., AEA solution) and the adsorbent (i.e., cement or cement/fly ash) are not in contact long enough to achieve equilibrium. Plus, the solution concentration is constantly being changed which also affects equilibrium conditions. These factors contribute to the subjectivity of the test. Additionally, the test does not provide a quantitative assessment of the increased AEA dosage required when fly ash is substituted for portland cement in concrete.

A version of the foam index test, proposed for standardization, was recently published (Kueber-Watkins, 2013; Sutter et al., 2013). This test is based on the test published by Harris et. al (Harris, 2008; Harris, 2008a; Harris, 2008b) but is modified to use different strength solutions, depending on the adsorption potential of the ash, to achieve a foam index result in 15 ± 3 minutes. Although the adsorbent and adsorbate are likely still not in equilibrium in this time frame, it is a consistent time between tests and thereby reduces the subjectivity of the test. The other modification of the Harris test was to use a mechanical agitator to shake the sample thereby eliminating another source of subjectivity.

Recently two new tests were developed to characterize fly ash based on adsorption. The first is the coal fly ash iodine number test (Ahmed, 2013; Sutter et al., 2013), which is based on ASTM D4607-94(2006) *Standard Test Method for Determination of Iodine Number of Activated Carbon*. In the coal fly ash iodine number test, the mass of iodine adsorbed from an aqueous solution by a specific mass of fly ash is determined. In the published form of the test (Sutter et al., 2013), a number of iodine adsorption measurements are made with fly ash samples of different mass, resulting in an isotherm relating the mass of iodine adsorbed per gram of fly ash. For the research conducted here, a different version of the iodine number approach was developed based on ASTM D1510 *Standard Test Method for Carbon Black-Iodine Adsorption Number.* In this version, only a single sample of fly ash is reacted with an iodine solution, resulting in a one point isotherm, but a shorter test. Although iodine-based tests determine iodine adsorption, rather than AEA adsorption, the tests represent the adsorption potential of the ash with a standard adsorbate and therefore can be used to compare ash adsorption properties on a uniform basis.

The second newly developed test is the direct adsorption isotherm test based on ASTM D3860-98(2008) *Standard Practice for Determination of Adsorptive Capacity of Activated Carbon by Aqueous Phase Isotherm Technique*. This test also determines the adsorption isotherm of the ash but does so with an AEA solution. The amount of adsorption is dependent on both the adsorbent (i.e., ash) and the adsorbate (i.e., AEA) and therefore, this test provides the most robust measure of AEA adsorption by a given fly ash, using a specific AEA. This test determines the volume of AEA that will be adsorbed per gram of ash and thereby provides a means of estimating the affect of the ash on AEA dosage. It can be used to compare different ash sources, or it can be used to evaluate different AEAs with a specific ash.

3 Experimental Methods

The research approach used was broken down into five (5) tasks listed below. Each task will be further detailed in the following sections

Task 1 - Raw Material Selection and Procurement
Task 2 - Raw Material Characterization
Task 3 - Preparation of Concrete Mixtures
Task 4 - Testing of Hardened Concrete Mixtures
Task 5 - Data Analysis

3.1 Task 1 - Raw Material Selection

The materials used in this project were as follows:

Portland Cement - Three (3) sources of AASHTO M 85 Type I/II portland cement were used. They are designated PC1, PC2, and PC3, respectively.

Coal Fly Ash – A total of four (4) different AASHTO M 295 coal fly ash sources were used. Two (2) Class C fly ash sources, designated C1 and C2, and two (2) Class F fly ash sources designated F1 and F2.

Aggregate - Two (2) coarse aggregate sources were used in this study. One was a crushed glacial gravel and the other a quarried limestone. Both were 100% WisDOT #1 stone (AASHTO #67) grading. Natural fine aggregate meeting WisDOT portland cement concrete paving specifications were obtained from the same supplier providing the coarse aggregate. Throughout this document, the following abbreviations will be used to refer to the various aggregate sources: G – glacial gravel, L – quarried carbonate.

Admixtures – The admixtures used for this study were a vinsol resin air entraining agent designated AEA1 and a low range water reducer designated WR1.

All materials were shipped directly to American Engineering Testing (AET) where the concrete mixtures were prepared except the aggregate, which was first shipped to Michigan Tech where the coarse aggregate was size classified. The aggregate were then shipped to AET for mixture preparation.

3.2 Task 2 - Raw Material Characterization

3.2.1 Aggregate Properties

The measured properties of the aggregate sources used in this study are provided in Table 3.1.

Table 3.1. Aggregate specific gravity and absorption values

Aggregate Source	Specific Gravity		Absorption (weight %)	
	Coarse	Fine	Coarse	Fine
G	2.74	2.67	0.99	0.95
L	2.65	2.67	2.75	0.54

3.2.2 Size Classification of the Aggregate Sources

Past experience conducting research for WisDOT, where Wisconsin aggregate sources are used, has shown that size classification of the aggregate sources (i.e., particularly the glacial gravel sources) is required. The sources of glacial gravel are typically low in 3/8 inch sized material and to obtain a true #67 grading, and to maintain uniformity between mixtures for comparison purposes, it is necessary to size the as-received material and recombine the sized products to achieve the required grading of the aggregates. The as-received material was sized at Michigan Tech using a Gilson Model GX-4A1 (24-inch by 30-inch) sieve separator. Both sources were size separated and a grading meeting the AASHTO #67 grading specification was prepared for both aggregate sources. The as-received quarried carbonate source met the required grading. For the as-received glacial gravel source, it was necessary to crush approximately 1000 pounds of -3/4 +1/2 inch material to produce -3/8 inch size material. The final grading for the glacial gravel source is shown in Figure 3.1 and the final grading for the quarried carbonate source is shown in Figure 3.2. The gradings shown in Figure 3.1 and Figure 3.2 are the same used for the mixture designs. The mixture design gradings are also summarized in Table 3.2.

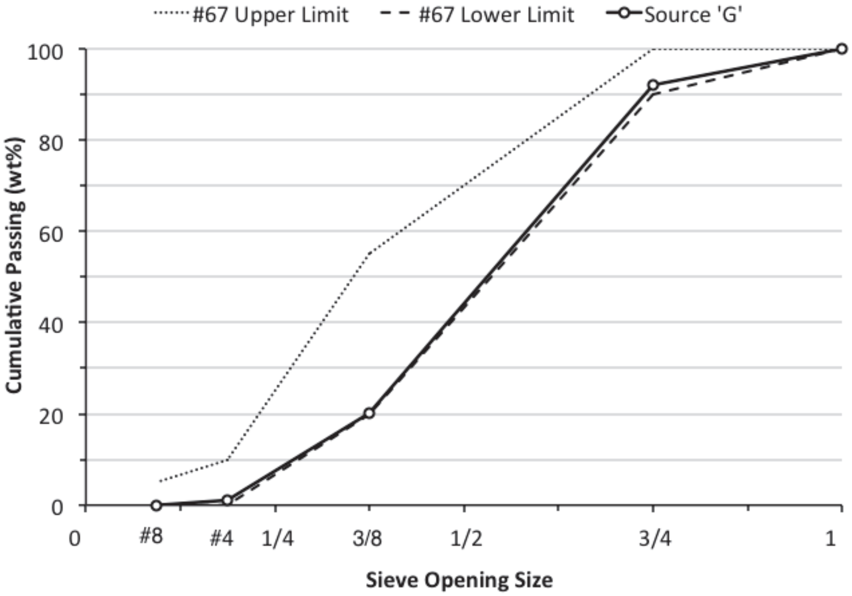


Figure 3.1. Gradation of the glacial gravel coarse aggregate source after sieve crushing and sizing.

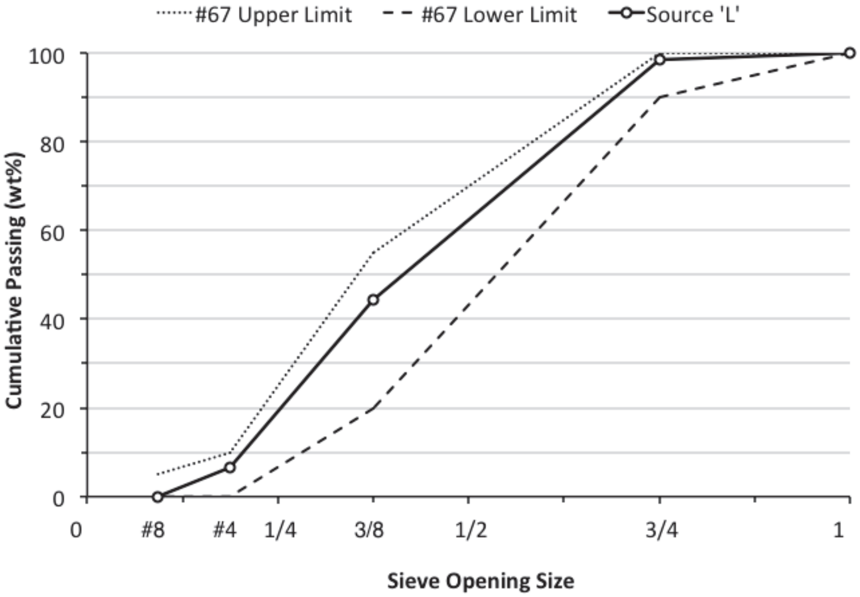


Figure 3.2. As-received gradation of the quarried carbonate aggregte source. No processing was required.

Table 3.2. Final grading used for the concrete mixture designs.

Size	Glacial Gravel Source		Quarried Carbonate Source	
	Weight Percent Retained	Weight Percent Passing	Weight Percent Retained	Weight Percent Passing
+1"	0	100	0	100
-1" +3/4	8.0	92.0	1.5	98.5
-3/4 + 1/2	49.5	42.5	33.1	65.4
-1/2 + 3/8	22.3	20.2	21.0	44.4
-3/8 + 4 mesh	19.2	1.0	37.9	
- 4 mesh + 8 mesh	1.0	0	6.5	

3.2.3 Chemical/Physical Characterization of the Fly Ash and Portland Cement Sources

All four (4) fly ash sources were characterized to establish the chemical and physical properties specified in AASHTO M 295 Tables 1 and 3. In addition, the optional properties of available alkali and air entrainment were determined Only chemical analyses and LOI tests were conducted on the three (3) cement samples. All tests were conducted in the Iowa State University Materials Analysis Research Laboratory. The list of all baseline characterization tests performed is provided below in Table 3.3.

Table 3.3. Summary of baseline tests conducted on the four (4) coal fly ash sources.

Test	Specification	# Specimens per Test	# of Repetitions	Comments
Moisture content	ASTM C311	1	2	
Loss on Ignition	ASTM C311	1	2	
Oxide Analysis	ASTM C311	1	2	XRF
Available Alkali	ASTM C311	1	2	
Density	ASTM C311	1	2	Helium pycnometer
Fineness	ASTM C311	1	2	
Soundness	ASTM C311	1	2	Autoclave method
Air Entrainment	ASTM C311	1	2	
Strength Activity Index (includes water requirement)	ASTM C311	3	1	

- **Moisture Content** – all samples were dried overnight at a temperature of 105 to 110°C. Single determinations were conducted on two different days to provide an average value for each sample of fly ash.
- **Loss on Ignition** – fly ash samples were ignited to a constant mass at 750 ± 50°C. Cement samples were ignited to a constant mass at 950 ± 50°C. Single determinations were conducted on two different days to provide an average value for each sample.
- **Oxide Analysis** – all cement and fly ash samples were analyzed using X-ray fluorescence (XRF) analysis via a PANalytical PW2404 spectrometer. This method consisted of fusing the sample with a lithium borate flux to produce a glass disk. Single determinations were conducted on two different days to provide an average value for each sample.
- **Available Alkali** (soluble Na and K, expressed as oxides and sodium oxide equivalent) – all samples were measured using the test procedure described in ASTM C311 and the calibration procedure described in ASTM C114. Single determinations were conducted on two different days to provide an average value for each sample of fly ash.

- **Density** – was determined using a helium pycnometer. Single determinations were conducted on two different days to provide an average value for each sample of fly ash.
- **Fineness** – was determined by wet-washing on a 45µm sieve (#325 mesh). The sieves were calibrated in accordance with ASTM C311, and single determinations were conducted on two different days to provide an average value for each sample of fly ash.
- **Soundnes**s – Soundness of fly ash-cement pastes was determined via the autoclave expansion test. Mixtures containing 20% (by mass of cement) of each fly ash were molded at normal consistency. Single determinations were conducted on two different days to provide an average value for each sample of fly ash.
- **Air Entrainment** – the amount of air-entraining solution required to produce a mortar air content of 18% was determined in accordance with ASTM C311. Single determinations were conducted on two different days to provide an average value for each sample of fly ash. Only a single air-entraining solution (a vinsol resin) was used for the determinations.
- **Strength Activity Index (SAI) with portland cement** – standard mortar mixtures containing 20% fly ash (by mass of cement) were used in this study. Strength Index values were calculated after 7 days, 28 day and 56 days of standard curing (23°C, lime-water cure). A control mixture (containing only cement) was mixed on each day to accompany the mixtures containing fly ash. Test values consisted of the average of three cubes broken at 7, 28 and 56 days, respectively. Water requirement for each mortar mixture was determined by maintaining the flow of the test mixture within ±5% of the flow of the control mixture

3.2.4 Adsorption Characteristics of the Fly Ash Sources

The four (4) fly ash sources used in this research were characterized for adsorption potential using the newly developed tests summarized in the Background section of this report. These include the foam index test, iodine number test, and the direct measurement of adsorption capacity. The test methods used for each are included in Appendix A.

3.3 Task 3 – Preparation of Concrete Mixtures

To assess the performance of the Class F coal fly ash sources relative to the Class C sources, concrete mixtures were prepared using the three (3) portland cement sources, two (2) aggregate sources and four (4) fly ash sources previously described. The fly ash was substituted for portland cement at three (3) replacement levels (i.e., 15, 30, and 40 by weight of total cementitious material). A partial factorial approach was employed to simplify the testing matrix. Development of a partial factorial experiment was complicated by using three portland cement sources. Typically, experimental matrices with an equal number of factor levels are preferred. However, a partial factorial experiment was developed based on a D-Optimal design that allows for reducing the testing matrix to forty-two (42) separate mixtures. A design summary of is shown in Table 3.4. In addition to the mixtures shown in Table 3.4, 6 mixtures were prepared using the three (3) portland cement sources and two (2) aggregate sources, without fly ash.

All concrete mixtures were prepared using a total cementitious material content of 470 lbs/yd^3, a target total air content of 6% +/- 1%, and a slump of 3 +/- 1 inches. A constant *w/c* was used at 0.42.

As part of preparing the concrete mixtures, fresh concrete testing was performed by American Concrete Institute (ACI) Level I Certified Concrete Technicians. The fresh concrete tests conducted were:

- Slump of Hydraulic Concrete (AASHTO T 119)
- Air Content Of Freshly Mixed Concrete By The Pressure Method (AASHTO T 152)
- Density (Unit Weight), Yield and Air Content (Gravimetric) of Concrete (AASHTO T 121)
- Semi-Adiabatic Calorimetry

Table 3.4. Summary of partial factorial concrete mixture experiment. An X indicates that combination was used for a concrete mixture.

Cement Type	Aggregate Type	Ash Type	Ash Replacement Level 15%	30%	40%
PC1	G	C1			X
		C2	X		X
		F1			X
		F2	X		X
	L	C1	X	X	
		C2	X	X	
		F1	X	X	
		F2	X	X	
PC2	G	C1	X		
		C2	X	X	
		F1	X		
		F2	X	X	
	L	C1		X	X
		C2		X	X
		F1		X	X
		F2		X	X
PC3	G	C1		X	
		C2		X	X
		F1		X	
		F2		X	X
	L	C1	X		X
		C2	X		X
		F1	X		X
		F2	X		X

3.4 Task 4 – Testing of Hardened Concrete Mixtures

For concrete mixtures prepared in Task 3, the hardened concrete properties were determined using the following test methods:

- Standard Method of Test for Resistance of Concrete to Rapid Freezing and Thawing (AASHTO T 161).
- Standard Method of Test for Compressive Strength of Cylindrical Concrete Specimens (AASHTO T 22).
- Standard Method of Test for Flexural Strength of Concrete (AASHTO T 97).
- Standard Practice for Estimating Concrete Strength by the Maturity Method (ASTM C1074a)

The Standard Method of Test for Resistance of Concrete to Rapid Freezing and Thawing (AASHTO T 161) was modified as follows:

- Three (3) replicate prisms were tested per mixture.
- Prisms were moist cured per standard test procedures for 28 days, then air cured in a room with 50% (+/-5%) relative humidity for 28 days at standard curing temperature, until just prior to freeze thaw cycling.

- Each specimen was tested for a minimum of 300 cycles unless the specimens deteriorated to a state of being unusable prior to reaching 300 cycles.
- Testing was conducted using Method A in a 4% calcium chloride solution.
- The dynamic modulus was recorded every 30 cycles.

The Standard Method of Test for Compressive Strength of Cylindrical Concrete Specimens (AASHTO T 22) and Standard Method of Test for Flexural Strength of Concrete (AASHTO T 97) were modified so that testing was performed on two (2) specimens each at test ages of 3, 7, 28, and 90 days.

3.5 Task 5 - Data Analysis

As previously described, a D-Optimal design experimental design was used to establish the mixture design matrix for the concrete mixtures. This reduced the required experimental matrix from 72 treatments to 42. This experimental design approach is useful when standard design approaches are not applicable, such as when the design matrix is not orthogonal. In addition to the D-Optimal experimental design, a small, full-factorial matrix was developed producing six (6) additional concrete mixtures without fly ash. These mixtures were prepared using each cement type in combination with each aggregate type. Having these six (6) additional mixtures allowed for comparison between mixtures with and without fly ash.

Once the experimental data was acquired, the analysis of the data was performed. All analyses were performed using the SAS software suite. Four (4) independent variables (also referred to as *factors*) and twenty-three (23) dependent variables (also referred to as *response variables*) were identified. Table 3.5 provides a list of the independent variables identified for the statistical analysis. Table 3.6 provides a list of the dependent variables identified for the statistical analysis.

Table 3.5. Summary of independent variables identified for the statistical analysis of the hardened concrete data.

Independent Variable	Number of Levels	Definition of Levels
Cement Type	3	1 – PC1 2 – PC2 3 – PC3
Aggregate Type	2	1 – G 2 – L
Fly Ash Type	4	1 – C1 2 – C2 3 – F1 4 – F2
Fly Ash Content	3	15 – 15 weight % 30 – 30 weight % 40 – 40 weight %

Table 3.6. Summary of dependent variables identified for the statistical analysis of the hardened concrete data.

Dependent Variables: *Air-void System & Freeze-Thaw*	Dependent Variables: *Maturity*	Dependent Variables: *Strength*
Pressure Meter Air Content	Maturity Factor 1-day	Compressive Strength 1-day
Gravimetric Air Content	Maturity Factor 3-day	Compressive Strength 3-day
Durability Factor	Maturity Factor 7-day	Compressive Strength 7-day
	Maturity Factor 14-day	Compressive Strength 14-day
	Maturity Factor 28-day	Compressive Strength 28-Day
	Maturity Factor @ 300 psi	Compressive Strength 90-Day
	Maturity Factor @ 700 psi	Flexural Strength 3-day
	Maturity Factor @ 1000 psi	Flexural Strength 7-day
	Maturity Factor @ 2000 psi	Flexural Strength 28-Day
	Maturity Factor @ 3000 psi	Flexural Strength 90-Day

The objectives of the data analysis were:

1. Compare the dependent (i.e., *response*) variables for mixtures with and without fly ash.
2. For mixtures with fly ash, identify which independent variables (i.e., *factors*) significantly affect each of the response variables.
3. For factors identified as affecting the response variables, determine the effect of ash replacement level on that response.
4. Determine if there were any interactions between factors, meaning a combination of factors created a response differing from the response obtained from the individual factors.

These goals were addressed as follows:

Goal 1: Compare the dependent variables for mixtures with and without fly ash.

For each response variable, the sample means of the data were calculated for the mixtures without fly ash and for the mixtures with fly ash. Two statistical tests can be used to compare the two sample means; 1) a paired *t*-test and 2) a 2-independent *t*-test. Since the data was not paired, the 2- independent *t*-test was used to compare the means of the two data sets to see if any differences noted were statistically significant.

Goal 2: For samples with fly ash included, identify which independent variables (i.e., factors) have significant impact on each of the response variables.

The variation within each level of a factor, and the variation among the levels of a factor, were calculated and compared using an F-test for each of the response variables. If the variation among the levels of a factor was significantly larger than the variation within each level, the factor had a significant impact on the response variable. As part of this analysis, the coefficient of determination (R^2) indicates how closely a response variable is related to the four factors. An $R^2=0$ indicates the response variable is not affected by the factors while an $R^2=1$ indicates 100% of the variation measured in the response variable is explained by the factors.

Goal 3: For factors identified as impacting the response variables, determine the affect of factor levels on that response.

From Goal 2 factors were identified that had a significant impact on the response variables. To determine which levels of the factors cause the impact, Tukey's Honest Significant Difference (HSD) test was used. There are many tests available that could be used including Fisher's Least Significant Difference (LSD) test and Student-Newman-Keuls (SNK) test. However the HSD test is the most conservative test, which means it is the least likely to detect a difference between results but once a difference is detected, it is more likely to be a true difference rather than one due to random error.

Goal 4: Determine if there were any interactions between factors where a combination of factors created a response differing from the response obtained from the individual factors.

Two-factor interactions occur only if both factors themselves have a significant impact on the response. Therefore, it is only necessary to examine two-factor interactions for those factors shown to be significant themselves. Similarly, it only makes sense to consider three-factor interactions when all three factors themselves have significant impact on the response, and all possible two-factor interactions between each pair of the three factors have significant impact on the response. These types of models are called hierarchical models. To consider each response, an F-test was used to examine each of the four factors and determine if any one of the factors was important to the response. Once two factors were identified as being important, a hierarchical model was developed to analyze the two-factor interaction using an F-test and ANOVA to determine if the interaction term was important. If three factors were found to be important to a response, and all 2-factor interactions of those three factors were important, a hierarchical model would be developed to test for the three-factor interaction.

4 Results and Discussion

4.1 Material Characterization – Chemical and Physical Properties

The results of the fly ash chemical and physical properties characterization study are provided in Table 4.1 through Table 4.4. The results of the chemical characterization of the three portland cement sources are provided in Table 4.5 along with pertinent physical data extracted from the producer's certifications.

Table 4.1. Results of chemical and physical characterization of fly ash source C1. Measured values were determined as described in Chapter 3. Producer certified values were provided by the ash supplier at the time of acquisition.

Chemical Composition	**Measured Values**	**Producer Certified Values**	**AASHTO M 295 Limits**	
			Class F	*Class C*
Silicon Oxide (% SiO_2)	38.8	39.23		
Aluminum Oxide (% Al_2O_3)	20.1	20.72		
Iron Oxide (% $Fe_2O_{3(T)}$)	6.4	6.65		
SUM (% SiO_2+% Al_2O_3+% $Fe_2O_{3(T)}$)	65.3	66.6	70.0 min.	50.0 min.
Sulfur Trioxide (% SO_3)	1.1	1.1	5.0 max.	5.0 max.
Calcium Oxide (% CaO)	22.7	20.60		
Magnesium Oxide (% MgO)	5.0	4.38		
Sodium Oxide (% Na_2O)	1.6	1.54		
Potassium Oxide (% K_2O)	0.7	0.91		
Total Alkali (as % Na_2O_e)	2.1			
Moisture Content	0.0	0.08	3.0 max.	3.0 max.
Loss on Ignition	0.3	1.25	5.0 max.[a]	5.0 max.[a]
Available Alkali (as % Na_2O_e) [b]	1.37		1.5 max.	1.5 max.
Physical Tests				
Fineness				
Retained on a 45-µm sieve, (% wt)	12.5	17.0	34 max.	34 max.
Strength Activity Index (% control)				
Ratio to Control @ 7 days	89	91	75 min.	75 min.
Ratio to Control @ 28 days	98		75 min.	75 min.
Ratio to Control @ 56 days	102			
Water Requirement, (% control)	95	95	105 max.	105 max.
Soundness				
Autoclave Expansion, (%)	0.02	0.02	0.8 max.	0.8 max.
Density (grams per cubic cm)	2.69	2.68		
Air Content of Mortar (fl-oz/cwt cement)	2.03			

[a] The loss on ignition (LOI) specification limit in ASTM C618 is 6.0%.

[b] Supplementary optional requirement in AASHTO M 295 – Applicable only when specifically required by the purchaser. Available alkali is not specified in ASTM C618.

Table 4.2. Results of chemical and physical characterization of fly ash source C2. Measured values were determined as described in Chapter 3. Producer certified values were provided by the ash supplier at the time of acquisition.

Chemical Composition	**Measured Values**	**Producer Certified Values**	**AASHTO M 295 Limits**	
			Class F	*Class C*
Silicon Oxide (% SiO_2)	38.8	34.44		
Aluminum Oxide (% Al_2O_3)	20.6	19.39		
Iron Oxide (% $Fe_2O_{3(T)}$)	5.6	5.82		
SUM (% SiO_2+% Al_2O_3+% $Fe_2O_{3(T)}$)	65.0	59.6	70.0 min.	50.0 min.
Sulfur Trioxide (% SO_3)	1.2	1.78	5.0 max.	5.0 max.
Calcium Oxide (% CaO)	23.1	26.62		
Magnesium Oxide (% MgO)	4.7	5.19		
Sodium Oxide (% Na_2O)	1.6	1.89		
Potassium Oxide (% K_2O)	0.7	0.49		
Total Alkali (as % Na_2O_e)	2.1			
Moisture Content	0.0	0.05	3.0 max.	3.0 max.
Loss on Ignition	0.2	0.73	5.0 max.[a]	5.0 max.[a]
Available Alkali (as % Na_2O_e) [b]	1.30		1.5 max.	1.5 max.
Physical Tests				
Fineness				
Retained on a 45-µm sieve, (% wt)	7.9	10.8	34 max.	34 max.
Strength Activity Index (% control)				
Ratio to Control @ 7 days	96	100	75 min.	75 min.
Ratio to Control @ 28 days	105		75 min.	75 min.
Ratio to Control @ 56 days	109			
Water Requirement, (% control)	95	94	105 max.	105 max.
Soundness				
Autoclave Expansion, (%)	0.02	.04	0.8 max.	0.8 max.
Density (grams per cubic cm)	2.62	2.72		
Air Content of Mortar (fl-oz/cwt cement)	2.44			

[a] The loss on ignition (LOI) specification limit in ASTM C618 is 6.0%.

[b] Supplementary optional requirement in AASHTO M 295 – Applicable only when specifically required by the purchaser. Available alkali is not specified in ASTM C618.

Table 4.3. Results of chemical and physical characterization of fly ash source F1. Measured values were determined as described in Chapter 3. Producer certified values were provided by the ash supplier at the time of acquisition.

Chemical Composition	**Measured Values**	**Producer Certified Values**	**AASHTO M 295 Limits**	
			Class F	*Class C*
Silicon Oxide (% SiO_2)	52.3	53.75		
Aluminum Oxide (% Al_2O_3)	16.1	16.18		
Iron Oxide (% $Fe_2O_{3(T)}$)	6.3	6.21		
SUM (% SiO_2+% Al_2O_3+% $Fe_2O_{3(T)}$)	74.7	76.14	70.0 min.	50.0 min.
Sulfur Trioxide (% SO_3)	0.6		5.0 max.	5.0 max.
Calcium Oxide (% CaO)	13.2	12.13		
Magnesium Oxide (% MgO)	4.6			
Sodium Oxide (% Na_2O)	2.6	0.59		
Potassium Oxide (% K_2O)	2.4			
Total Alkali (as % Na_2O_e)	4.2			
Moisture Content	0.0	0.03	3.0 max.	3.0 max.
Loss on Ignition	0.1	0.11	5.0 max.[a]	5.0 max.[a]
Available Alkali (as % Na_2O_e) [b]	1.37	1.44	1.5 max.	1.5 max.
Physical Tests				
Fineness				
Retained on a 45-µm sieve, (% wt)	22.0	27.51	34 max.	34 max.
Strength Activity Index (% control)				
Ratio to Control @ 7 days	85	79	75 min.	75 min.
Ratio to Control @ 28 days	87	86	75 min.	75 min.
Ratio to Control @ 56 days	96			
Water Requirement, (% control)	95	94	105 max.	105 max.
Soundness				
Autoclave Expansion, (%)	0.01	0.00	0.8 max.	0.8 max.
Density (grams per cubic cm)	2.49	2.42		
Air Content of Mortar (fl-oz/cwt cement)	1.82			

[a] The loss on ignition (LOI) specification limit in ASTM C618 is 6.0%.

[b] Supplementary optional requirement in AASHTO M 295 – Applicable only when specifically required by the purchaser. Available alkali is not specified in ASTM C618.

Table 4.4. Results of chemical and physical characterization of fly ash source F2. Measured values were determined as described in Chapter 3. Producer certified values were provided by the ash supplier at the time of acquisition.

Chemical Composition	**Measured Values**	**Producer Certified Values**	**AASHTO M 295 Limits**	
			Class F	*Class C*
Silicon Oxide (% SiO_2)	47.3	45.68		
Aluminum Oxide (% Al_2O_3)	23.4	23.61		
Iron Oxide (% $Fe_2O_{3(T)}$)	17.7	17.78		
SUM (% SiO_2+% Al_2O_3+% $Fe_2O_{3(T)}$)	88.4	87.1	70.0 min.	50.0 min.
Sulfur Trioxide (% SO_3)	0.7	0.54	5.0 max.	5.0 max.
Calcium Oxide (% CaO)	3.8	4.29		
Magnesium Oxide (% MgO)	0.9	1.05		
Sodium Oxide (% Na_2O)	0.7	0.73		
Potassium Oxide (% K_2O)	1.7	1.59		
Total Alkali (as % Na_2O_e)	1.8			
Moisture Content	0.2	0.19	3.0 max.	3.0 max.
Loss on Ignition	2.0	2.39	5.0 max.[a]	5.0 max.[a]
Available Alkali (as % Na_2O_e) [b]	0.43		1.5 max.	1.5 max.
Physical Tests				
Fineness				
Retained on a 45-µm sieve, (wt. %)	15.3	14.0	34 max.	34 max.
Strength Activity Index (% control)				
Ratio to Control @ 7 days	79	75	75 min.	75 min.
Ratio to Control @ 28 days	83	95	75 min.	75 min.
Ratio to Control @ 56 days	91			
Water Requirement, (% control)	100	97	105 max.	105 max.
Soundness				
Autoclave Expansion, (%)	-0.02	0.00	0.8 max.	0.8 max.
Density (grams per cubic cm)	2.50	2.51		
Air Content of Mortar (fl-oz/cwt cement)	2.48			

[a] The loss on ignition (LOI) specification limit in ASTM C618 is 6.0%.

[b] Supplementary optional requirement in AASHTO M 295 – Applicable only when specifically required by the purchaser. Available alkali is not specified in ASTM C618.

Table 4.5. The results of the chemical characterization of the three portland cement sources and pertinent physical data extracted from the producers certifications.

Chemical Composition[a] (all values weight %)	Cement Identification Code					
	PC1		PC2		PC3	
	Measured Values	Producer Certified Values	Measured Values	Producer Certified Values	Measured Values	Producer Certified Values
Na_2O	0.25	-	0.16	0.19	0.12	-
MgO	2.32	2.3	3.93	3.9	2.43	2.6
Al_2O_3	4.99	4.9	5.03	4.9	4.59	4.5
SiO_2	19.80	19.4	19.00	19.0	19.60	19.7
P_2O_5	0.13	-	0.07	-	0.07	-
SO_3	2.82	2.6	3.85	3.9	3.14	3.2
K_2O	0.47	-	1.16	1.20	0.56	
CaO	63.10	64.0	61.70	62.1	63.20	64.3
TiO_2	0.25	-	0.25	-	0.23	-
Fe_2O_3	2.92	2.8	2.82	2.8	3.22	3.2
SrO	0.08	-	0.04	-	0.05	-
Mn_2O_3	0.12	-	0.19	-	0.10	-
Loss on Ignition (LOI)	2.78	2.4	1.82	1.8	2.67	2.5
Insoluble Residue	-	0.21	-	0.28	-	0.49
Free Lime	-	1.20	-	1.1[b]	-	0.34[b]
Total Alkalis (Na_2O_e)	0.56	0.55	0.92	0.98	0.49	0.55
CO_2	1.5[c]	1.5	1.5[c]	1.5	1.3[c]	1.3
Limestone	3.4[c]	3.4	3.5[c]	3.5	3.4[c]	3.3
$CaCO_3$ in Limestone	96[a]	96	95[a]	95	89[a]	89
Inorganic Processing Additions	-	-	-	0	-	0
C_3S (Bogue, calculated)	59	62	56	54	62	65
C_2S (Bogue, calculated)	12	9	12	13	9	8
C_3A (Bogue, calculated)	8	8	8	8	6	6
C_4AF (Bogue, calculated)	9	8	8	8	9	9
$C_3S + 4.75C_3A$	97	100	95	92	93	94
Physical Test Results[a]						
Blaine Fineness (m^2/kg)	-	780	-	390	-	387
Passing #325 sieve (%)	-	97.2	-	98.2	-	-
Compressive strength (C 109)	-	-	-	-	-	-
1 day (psi)	-	2070	-	3223	-	-
7 days (psi)	-	4550	-	5100	-	5360
28 days (psi)	-	5570	-	5946	-	-
Air content (%)	-	8.1	-	8	-	7
Autoclave expansion (%)	-	0.01	-	0.18	-	0.009
Density (g/cc)	-	-	-	-	-	-

[a] Dash indicates the value was not reported

[b] Estimated from producer certificate Bogue results.

[c] Values were not determined separately. Producer certified values were used to correct Bogue calculated results.

4.2 Material Characterization – Fly Ash Adsorption Properties

4.2.1 Test Method Introduction

Three new test methods were evaluated for assessing the air entraining admixture (AEA) adsorption capacity of fly ash; the direct adsorption isotherm (Ahmed, 2012; Sutter et al., 2013), iodine number, and foam index test (Kueber, 2013; Sutter et al., 2013) were developed as part of a recent NCHRP study (see Appendix A for details of each method). The direct adsorption isotherm and iodine number tests were modified as part of this research and differ from the versions published in the NCHRP final report (Sutter et al., 2013; Sutter et al., 2013a) The foam index test included is the same as published in the NCHRP report and is reprinted here with permission. These methods provide data that can be used to either assess the adsorption capacity of one fly ash relative to another, or predict the amount of additional AEA required to produce a target air content when portland cement is partially replaced by fly ash.

4.2.2 Direct Adsorption Isotherm Test

The direct adsorption isotherm (DAI) provides a quantitative method for determining the amount of AEA adsorbed by fly ash (Ahmed, 2012; Sutter et al., 2013). This is accomplished by measuring the chemical oxygen demand (COD, in mg/l) of AEA solutions equilibrated with known quantities of cement and fly ash. The COD provides a measure of solution concentration before and after adsorption equilibrium and is used to determine the change in AEA concentration resulting from adsorption onto carbon in fly ash. Solutions with known concentrations of AEA are equilibrated with cement, fly ash, and also a combination of cement and fly ash, then filtered, and the COD of the filtrate is determined. The change in COD (i.e., change in solution concentration) upon addition of fly ash is calculated and an isotherm is plotted with a curve fit based upon the Freundlich equation shown in equation 4.1 (Ahmed, 2012; Sutter et al., 2013a).

$$\mathrm{q} = \mathrm{K} \times \mathrm{C}^{1/n} \tag{4.1}$$

Where:

K= Freundlich capacity parameter, $\frac{\mathrm{ml}}{\mathrm{g}} \times (\frac{1}{\%\mathrm{vol}})^{1/\mathrm{n}}$

$1/n$ = Freundlich intensity parameter, unit less

The adsorption capacity of the fly ash, reported as the volume of AEA adsorbed per unit mass of fly ash (e.g., ml AEA/g fly ash) is determined as a function of AEA solution concentration, expressed in volume % AEA solution. The user estimates the solution concentration as the volume of AEA added to a concrete batch divided by the total water content, by volume, in the batch.

Direct adsorption isotherms were generated for each combination of AEA1, three cements and four fly ashes, following the DAI procedure detailed in Appendix A. Tests were performed in triplicate and for comparative purposes, the fly ash adsorption capacity was calculated using a constant AEA concentration of 0.5%. (Note: 0.5% is chosen as an arbitrary reference value that falls within the range of most AEA-fly ash isotherms. This value can be used to compare two different fly ash sources with the same AEA. To estimate changes in AEA dosage, the adsorption capacity of the fly ash should be estimated based on the solution concentration of AEA in the concrete batch.). An example isotherm for fly ash C2 and AEA1 is shown in Figure 4.1 with data from three replicates in a single plot. Isotherms for all combinations of materials (i.e., AEA, cement, and fly ash) are included in Appendix B. The isotherm in Figure 4.1 was prepared using PC3 cement but this and previous research has demonstrated the adsorption isotherm is not cement-type dependent, although cement must be included in the isotherm determination (Ahmed, 2012; Sutter et al., 2013a). As read from the plot in Figure 4.1, the adsorption capacity of fly ash C2 for AEA1, at a solution concentration of 0.5%, is approximately 0.005 mL/g fly ash, based upon an average of the three data sets. The fly ash adsorption capacity for all ash sources and cement types at a constant AEA concentration of 0.5% are presented in Table 4.6 and in Figure 4.2.

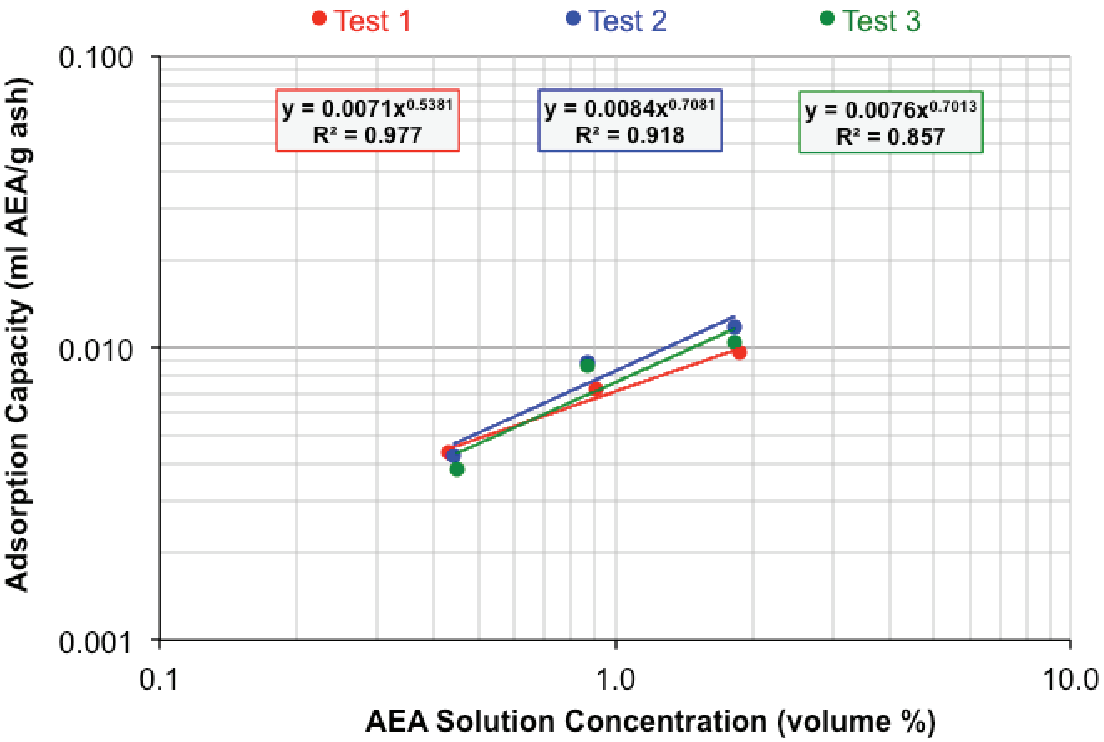


Figure 4.1. An example isotherm for one AEA type and one fly ash type.

Table 4.6. Values of adsorption capacity for each fly ash source in combination with each cement type.

	Adsorption Capacity for Fly Ash Sources (ml AEA/g fly ash)			
Cement	C1	C2	F1	F2
PC1	0.0023	0.0033	0.0008	0.0055
PC2	0.0029	0.0038	0.0001	0.0045
PC3	0.0011	0.0031	0.0012	0.0031

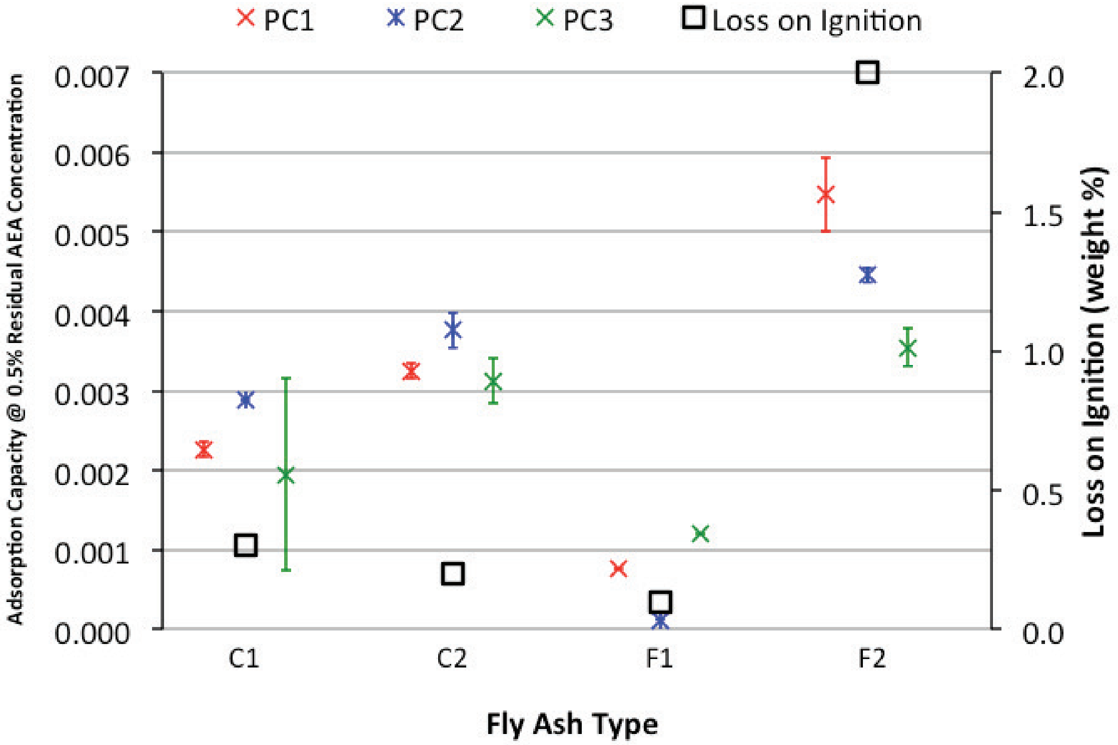


Figure 4.2. Fly ash adsorption capacity determined at a constant AEA concentration of 0.5% for each combination of AEA, cement and fly ash. The loss on ignition (LOI) value for each ash source is also presented for comparison purposes. Error bars show the range of two determinations (n=2).

As can be seen in Table 4.6 and Figure 4.2, the adsorption capacity for all ash types, with the exception of C2, vary in proportion to the LOI fraction of the ash. Additionally, for a given ash type, the adsorption capacity was not affected significantly by cement type. Fly ash F1 exhibited a very low adsorption capacity, particularly for a Class F ash. It should be noted the adsorption results in this research are for one AEA type and adsorption is dependent upon both the ash source and the AEA type. As is seen in Figure 4.2, and will be discussed with later test results, fly ash C2 demonstrates an anomalous adsorption behavior. That is, fly ash C2 has a low LOI content (i.e., lower than source C1), but the adsorption capacity is significantly higher than ash sources with a similar LOI content. This speaks to the advantage of adsorption based tests for evaluating coal fly ash, particularly as materials such as powdered activated carbon are added to fly ash for pollution control. Although two ash sources may have the same LOI content, the adsorption characteristics of the LOI fraction can differ significantly.

4.2.3 Iodine Number Test

The iodine number test is used by producers and consumers of activated carbon and carbon black. It is a measure of a material’s capacity to adsorb iodine. In an aqueous solution, iodine concentration can easily be determined by simple titration with thiosulfate solution, making it an attractive adsorbate to test. Given the active adsorbent in fly ash is carbon, the test yields an estimate of the adsorption capacity of fly ash. The iodine number test has the advantage of measuring the adsorption capacity of an adsorbent (e.g., fly ash) with a standard solution. This allows direct comparisons of the adsorption capacity of different fly ash sources. A draw back is the method does not use an AEA and therefore, the test only identifies the general adsorption characteristics of the fly ash, not how the ash performs with a specific AEA. However, the test has been shown to correlate well with other measurements of AEA adsorption (Sutter et al., 2013). The complete method is included in Appendix A.

The iodine number is determined by contacting a carefully prepared iodine solution of known concentration with a known mass of fly ash and measuring the residual solution concentration of iodine after the adsorption process has reached equilibrium. A blank iodine solution (not equilibrated with fly ash) is also titrated, establishing a reference. The iodine number is then calculated as shown in equation 4.2.

$$I = \left(\frac{V_{BK} - V_{FA}}{V_{BK}}\right) \times \left(\frac{100 \times C_I \times 126.91}{M_{FA}}\right) \tag{4.2}$$

Where:

I = Iodine number, g I / g fly ash
V_{BK} = ml of titrant required for blank
V_{FA} = ml of titrant required for filtrate from fly ash equilibration
100 = ml of standard iodine solution equilibrated
C_I = Normality of iodine solution, meq/ml
126.91 = Equivalent mass of iodine, mg/meq
M_{FA} = Mass of fly ash equilibrated, g

Iodine numbers were determined for all four of the fly ashes, with four replicates per fly ash. Iodine numbers for the four replicates are presented in Table 4.7.

Table 4.7. Fly ash iodine numbers.

Fly Ash	Iodine Number
C1	1.004
C2	1.409
F1	0.878
F2	1.348

A comparison of the direct adsorption isotherm and iodine number methods is presented graphically in Figure 4.3 with the DAI adsorption capacity shown being the average determined for the three different cement sources at a solution concentration of 0.5 volume % AEA. Included in Figure 4.3 are the LOI values for each ash source. The anomalous behavior of fly ash C2 is more clearly accentuated using the iodine number test. The general trend between LOI and adsorption is seen and the very low adsorption capacity of fly ash source F1 is confirmed.

In Figure 4.4 the standard deviation of the test is shown graphically. The coefficient of variation (CV%) for each test is shown in Table 4.8 and as can be seen the CV% is approximately 10% except for fly ash source C2. In all cases, the variation in the measurements is in part due to the low adsorption potential of the ash sources tested. When adsorption is higher, the change in solution concentration is greater and the variation in determining the solution concentration decreases, improving the precision of the test. The very low adsorption capacity of fly ash requires precise titrations and if more accurate determinations are needed (not recommended by this research) then Class A volumetric glassware may be required to assure reproducibility with ash sources. Class A glassware was not used for this study.

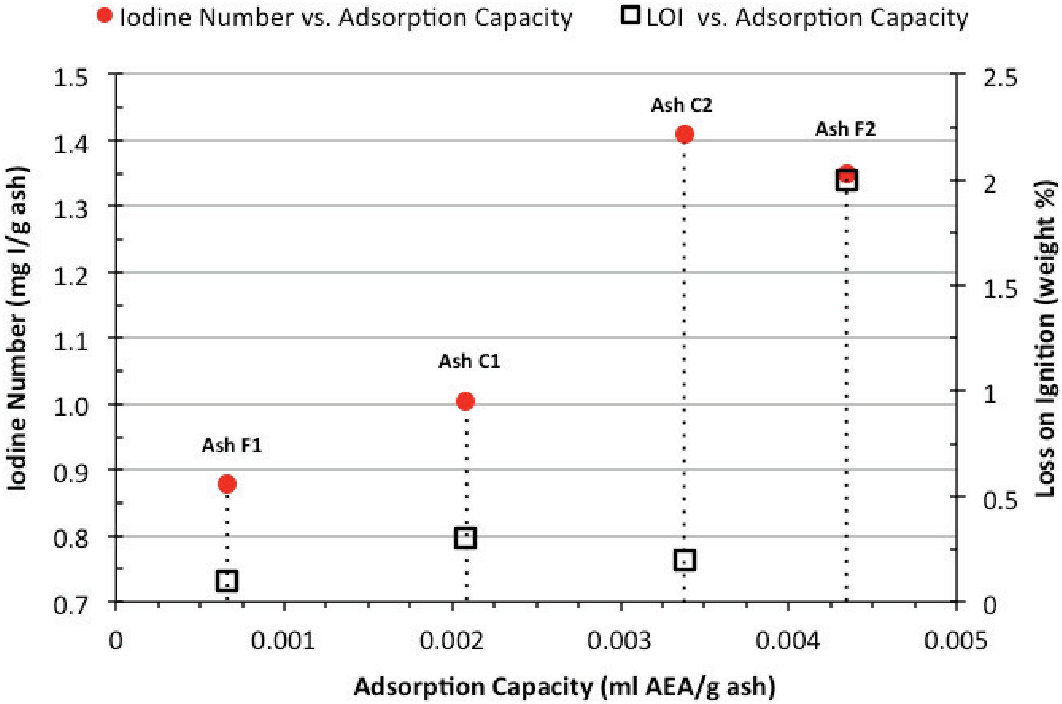


Figure 4.3. Comparison of iodine number and loss on ignition to average DAI adsorption capacity determined at a residual AEA concentration of 0.5%.

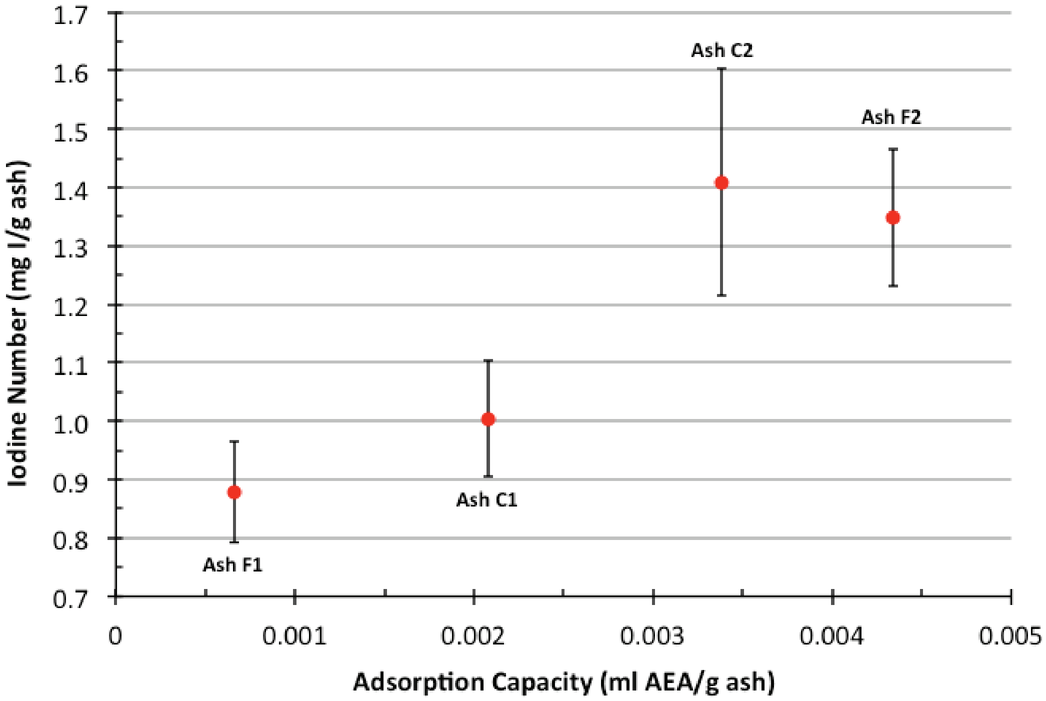


Figure 4.4. Results of the iodine number test versus average DAI adsorption capacity determined at a residual AEA concentration of 0.5%. Error bars show one (1) standard deviation for each data point which were are the average of four (4) separate determinations (n=4).

Table 4.8. Coefficient of variation for iodine number measurements of all fly ash sources. Value shown is in percent (%). For all determinations n=4.

Coefficient of Variation (%) for Iodine Number Test			
C1 (n=4)	C2 (n=4)	F1 (n=4)	F2 (n=4)
9.9	13.8	9.8	8.7

The iodine number test is a simple, non-subjective way to evaluate fly ash for quality control purposes. Also, the results of the iodine number test can be correlated with the direct adsorption isotherm determination of adsorption capacity. Then, using the iodine number test, estimates of correction to AEA dosage can be made (Sutter et al., 2013),

4.2.4 Foam Index Test

The foam index test provides an estimate of the affect fly ash has on AEA performance. A known concentration of AEA (C_S) is added to a cement-ash-water slurry in a drop-wise fashion and the mixture is then shaken for a fixed time period. Shaking is stopped and the mixture is observed to note how long foam persists on the surface of the slurry. This procedure is repeated, adding more drops of AEA solution, until a “stable” foam persists for 15 seconds or more. The total number of AEA solution drops added (N_D) is recorded. Based on C_S and N_D, the following calculations can be made for either a cement-only mixture, or a mixture with cement and fly ash:

$$\text{Foam Index (ml AEA solution)} = N_D * 0.02 \tag{4.3}$$

$$\text{Absolute Volume (ml AEA)} = \text{Foam Index} * C_S \tag{4.4}$$

$$\text{Specific Foam Index (ml AEA / 100 kg cementitious)} = \text{Absolute Volume} * 10{,}000 \tag{4.5}$$

$$\text{Relative Foam Index (\%)} = [(\text{Absolute Volume}_{\text{ash}}) / (\text{Absolute Volume}_{\text{cement}})] * 100 \tag{4.6}$$

In equation 4.3, the value 0.02 is the ml solution per drop. This is a typical number used but if the apparatus being used produces a different drop size, the alternate value should be entered in place of 0.02. The relative foam index is determined by repeating the process with a cement-water slurry and calculating the ratio of the foam index determined with fly ash to that without fly ash. The relative foam index should indicate the additional AEA required upon partial replacement with fly ash, with a given cement source.

Unlike the other tests described, the foam index test is an empirical test. Therefore, the test should be conducted using both the job cement and the job fly ash. The results of the specific foam index for the materials tested are shown in Table 4.9 and Figure 4.5. The results of the relative foam index for the materials tested are shown in Table 4.10.

Table 4.9. Values of specific foam index for each fly ash source in combination with each cement type.

	Specific Foam Index for Fly Ash Sources (ml AEA/100kg cementitious)			
Cement	C1	C2	F1	F2
PC1	52	70	52	60
PC2	56	55	52	56
PC3	49	62	50	58

Table 4.10. Values of relative foam index for each fly ash source in combination with each cement type.

	Relative Foam Index for Fly Ash Sources [(specific foam index fly ash + cement)/(specific foam index cement)] x 100			
Cement	C1	C2	F1	F2
PC1	89	121	90	104
PC2	106	105	98	107
PC3	101	129	104	120

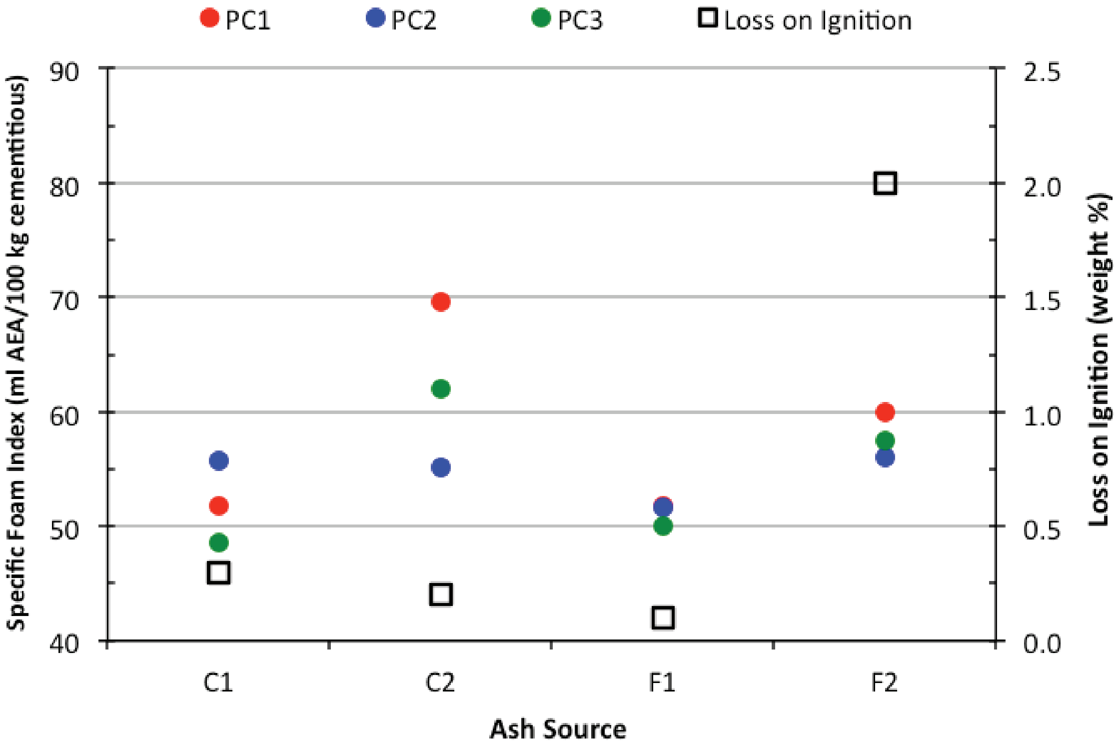


Figure 4.5. Specific foam index for each fly ash source in combination with each cement source. Also the loss on ignition value for each ash source is shown for relative comparison.

The relative foam index for each materials combination is shown in Figure 4.6. As seen in Figure 4.5, the general trend of an increase in foam index with increasing LOI holds, except for ash C2. However, the trend is more subtle. Note the foam index difference between ash sources F1 and F2 can be seen more clearly in Figure 4.6, where an approximate 5-20% difference in AEA dosage is predicted, depending upon the cement used.

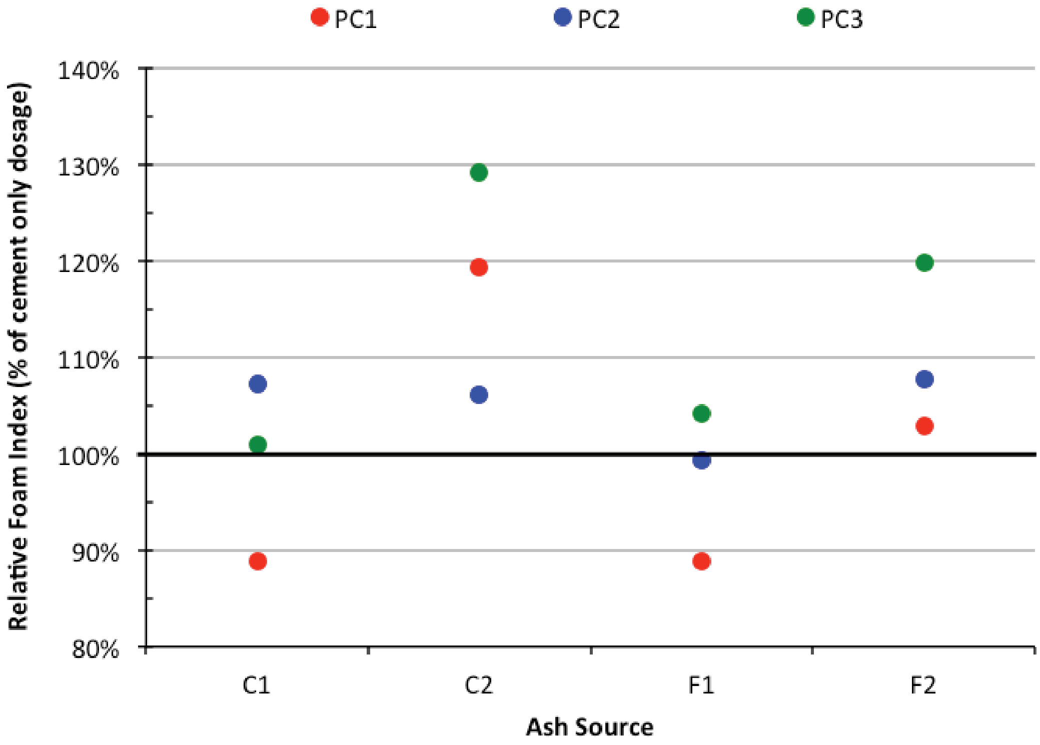


Figure 4.6. Relative foam index for each fly ash source in combination with each cement source. A value of 100 indicates the fly ash and cement combination required the AEA dosage to achieve a stable foam, as compared to cement alone.

The foam index test is a subjective test, meaning very operator dependent. There are numerous variations of the test in the literature and as with any empirical test, as test parameters are varied, results obtained vary and cannot be compared to results obtained under different testing conditions. The AEA solution concentration used in the test is one of the more critical values and needs to be reported. Regarding subjectivity, the condition of a "stable" foam is a matter of judgment by the person performing the test. Likewise the operator can affect the test by more or less shaking vigor.

The methodology used here was developed as part of other research that specifically focused on developing a standard test (Kueber, 2013; Sutter et al., 2013). The test provided in Appendix A was developed to minimize the subjectivity by using a mechanical shaker and a prescriptive approach to establishing the proper AEA solution concentration (Kueber, 2013; Sutter et al., 2013). As a result, the repeatability of the test has been improved. Table 4.11 shows the coefficient of variation for the tests performed with the different materials combinations in this research.

Figure 4.7 shows the specific foam index results obtained with the various materials. The error bars in Figure 4.7 indicate one (1) standard deviation of the measured foam index data.

Table 4.11. Coefficient of variation (%) for foam index test with each combination of cement and fly ash. Value shown is in percent (%). For each determination the value of n is included in brackets [].

	Coefficient of Variation (%) for Foam Index Test			
Cement	C1	C2	F1	F2
PC1	15.6 [7]	11.9 [5]	9.3 [7]	8.2 [5]
PC2	5.3 [7]	7.3 [5]	9.8 [6]	11.1 [6]
PC3	11.0 [5]	5.0 [6]	12.5 [3]	6.1 [6]

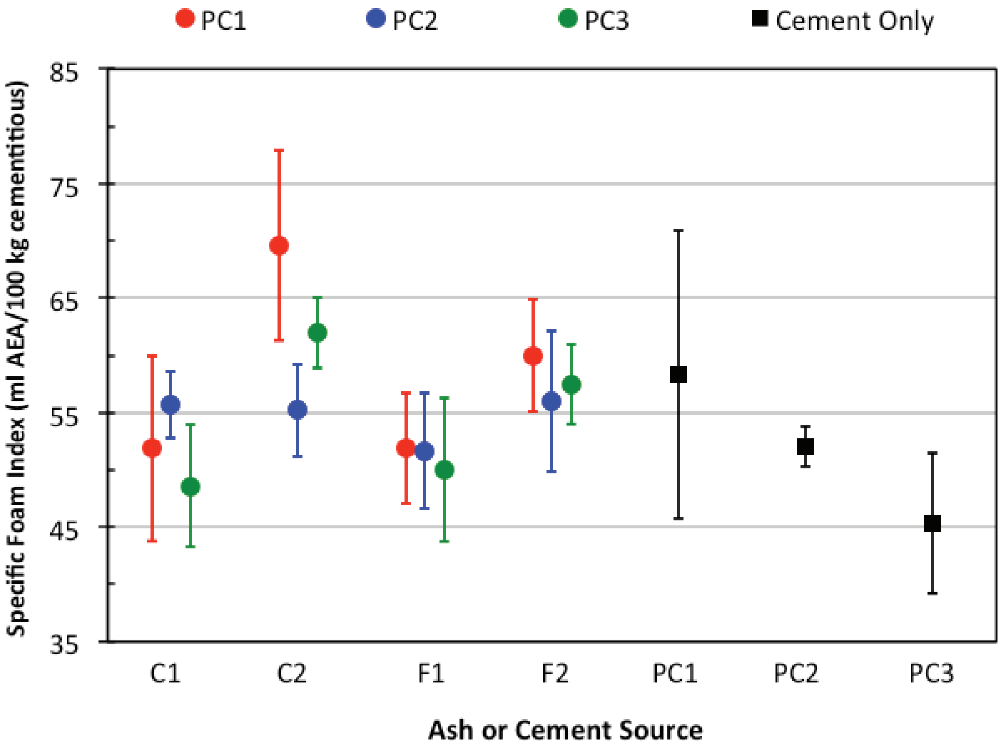


Figure 4.7. Specific foam index for each fly ash source in combination with each cement source. Also specific foam index for each cement source. In both cases the range of measured values is shown. Error bars show 1 standard deviation of n measurements for each combination. For cement & fly ash n is as indicated in Table 4.11; for cement-only tests n=3.

Although the precision of the test is adequate, the foam index test also suffers from not allowing time for the AEA and fly ash to achieve adsorption equilibrium. This fact contributes to the variability of the test and also reduces the test sensitivity relative to the previously described tests that do reach equilibrium. Figure 4.8 and Figure 4.9 illustrate this point. Both plots show the specific foam index for each fly ash source in combination with each cement source plotted against the measured adsorption capacity. Figure 4.9 includes the linear regression results to help illustrate. As can be seen, the foam index test is relatively insensitive to changes in adsorption capacity.

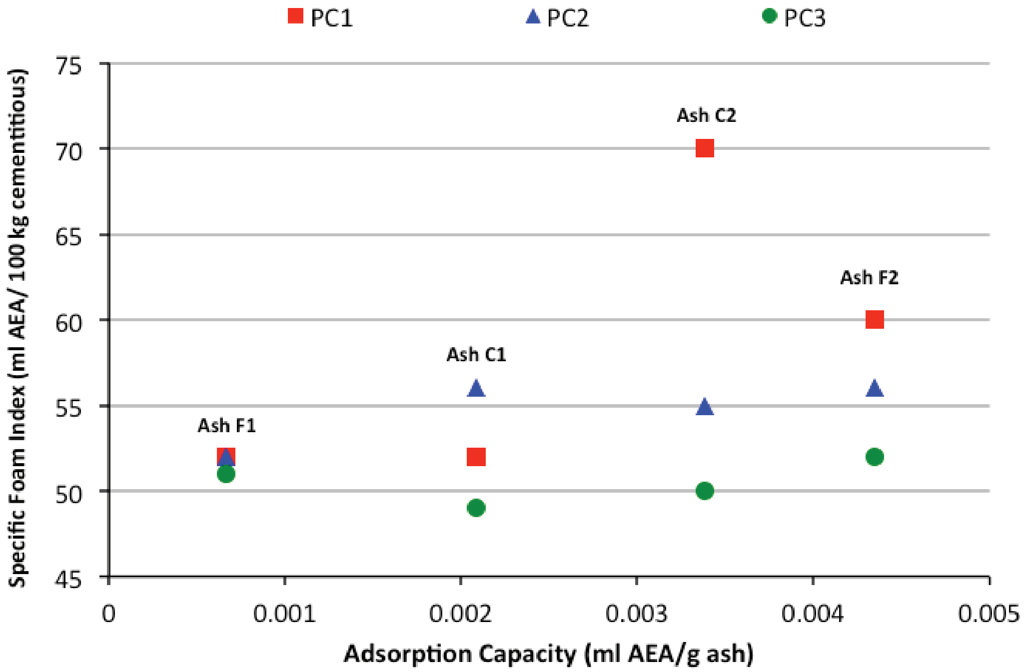


Figure 4.8. Specific foam index for each fly ash source in combination with each cement source plotted against the measured adsorption capacity.

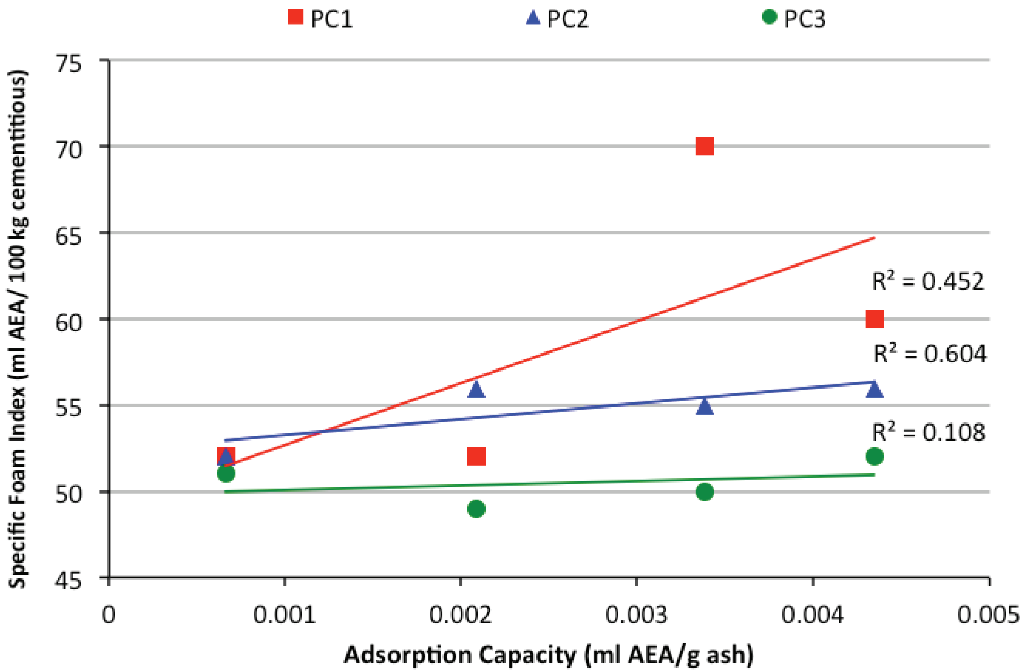


Figure 4.9. Specific foam index for each fly ash source in combination with each cement source plotted against the measured adsorption capacity showing the linear regression best fit lines.

It should be noted this correlation with adsorption capacity is worse with low adsorption capacity fly ash. Because the foam index test uses a dilute AEA solution and a short contact time, adsorption does not reach equilibrium. As the adsorption capacity of the ash increases (i.e., higher LOI), the sensitivity of the tests improves (Sutter et al., 2013). For fly ash sources typically used in highway construction, the direct adsorption isotherm and iodine number tests provide the broadest range of applicability, as they are able to characterize fly ash sources with a wide range of adsorption capacity.

4.3 Concrete Mixtures

4.3.1 Sample Designation

The mixture identification used throughout this report indicates the key properties of each mixture . As examples, the mixture ID *PC1-G-C1-40* represents a mixture with PC1 cement, the glacial gravel aggregate source (G), and fly ash source C1 replaced at 40% of the total cementitious content by weight. The mixture ID *PC2-L-X* represents a mixture with PC2 cement, the quarried carbonate source (L) and no fly ash (X).

4.3.2 Mixture Design and Batch Weights

The mixture designs for the concrete prepared in Task 3 are presented in Table 4.12 through Table 4.14. The batch weights and the results of fresh concrete testing are presented in Table 4.15 through Table 4.17.

Table 4.12. Mixture designs for concrete mixtures with PC1 cement.

Mixture ID	Cement Type	Aggregate Type	Fly Ash Source	Fly Ash Replacement	Cement Content	Fly Ash Content	Aggregate		Water Reducer	Air Entrainer	Water	Design *w/cm*	Air-Free Unit Weight
							Coarse	Fine					
				weight % cementitious	lbs/yd^3	lbs/yd^3	lbs/yd^3	lbs/yd^3	fl oz/yd^3	fl oz/yd^3	lbs/yd^3		lbs/ft^3
PC1-G-X	PC1	G	-	0	470.0	0.0	1864.3	1486.4	14.9	3.5	197.4	0.42	158.3
PC1-G-C1-40	PC1	G	C1	40	282.0	188.0	1845.3	1471.2	1.8	4.6	197.4	0.42	157.2
PC1-G-C2-15	PC1	G	C2	15	399.5	70.5	1857.2	1480.7	5.3	3.9	197.4	0.42	157.8
PC1-G-C2-40	PC1	G	C2	40	282.0	188.0	1845.3	1471.2	0.9	6.1	197.4	0.42	157.0
PC1-G-F1-40	PC1	G	F1	40	282.0	188.0	1843.2	1469.5	0.7	4.0	197.4	0.42	156.6
PC1-G-F2-15	PC1	G	F2	15	399.5	70.5	1856.4	1480.1	17.6	3.9	197.4	0.42	157.7
PC1-G-F2-40	PC1	G	F2	40	282.0	188.0	1843.2	1469.5	5.3	6.5	197.4	0.42	156.7
PC1-L-X	PC1	L	-	0	470.0	0.0	1803.1	1486.4	14.0	3.9	197.4	0.42	155.9
PC1-L-C1-15	PC1	L	C1	15	399.5	70.5	1796.2	1480.7	10.5	4.9	197.4	0.42	155.5
PC1-L-C1-30	PC1	L	C1	30	329.0	141.0	1789.3	1475.0	5.3	4.4	197.4	0.42	155.1
PC1-L-C2-15	PC1	L	C2	15	399.5	70.5	1796.2	1480.7	8.8	4.4	197.4	0.42	155.4
PC1-L-C2-30	PC1	L	C2	30	329.0	141.0	1789.3	1475.0	7.0	4.9	197.4	0.42	155.0
PC1-L-F1-15	PC1	L	F1	15	399.5	70.5	1795.4	1480.1	10.5	3.9	197.4	0.42	155.3
PC1-L-F1-30	PC1	L	F1	30	329.0	141.0	1787.7	1473.7	7.0	4.2	197.4	0.42	154.7
PC1-L-F2-15	PC1	L	F2	15	399.5	70.5	1795.4	1480.1	7.0	4.2	197.4	0.42	155.3
PC1-L-F2-30	PC1	L	F2	30	329.0	141.0	1787.7	1473.7	3.5	4.6	197.4	0.42	154.7

Table 4.13. Mixture designs for concrete mixtures with PC2 cement.

Mixture ID	Cement Type	Aggregate Type	Fly Ash Source	Fly Ash Replacement	Cement Content	Fly Ash Content	Aggregate		Water Reducer	Air Entrainer	Water	Design *w/cm*	Air-Free Unit Weight
							Coarse	Fine					
				weight % cementitious	lbs/yd^3	lbs/yd^3	lbs/yd^3	lbs/yd^3	fl oz/yd^3	fl oz/yd^3	lbs/yd^3		lbs/ft^3
PC2-G-X	PC2	G	-	0	470.0	0.0	1864.3	1486.4	14.1	3.5	197.4	0.42	158.3
PC2-G-C1-30	PC2	G	C1	30	329.0	141.0	1850.0	1475.0	9.7	4.0	197.4	0.42	157.5
PC2-G-C2-30	PC2	G	C2	30	329.0	141.0	1850.0	1475.0	8.8	4.2	197.4	0.42	157.3
PC2-G-C2-40	PC2	G	C2	40	282.0	188.0	1845.3	1471.2	6.3	4.6	197.4	0.42	157.0
PC2-G-F1-30	PC2	G	F1	30	329.0	141.0	1848.4	1473.7	5.3	4.0	197.4	0.42	157.1
PC2-G-F2-30	PC2	G	F2	30	329.0	141.0	1848.4	1473.7	5.3	4.2	197.4	0.42	157.1
PC2-G-F2-40	PC2	G	F2	40	282.0	188.0	1843.2	1469.5	3.5	4.9	197.4	0.42	156.7
PC2-L-X	PC2	L	-	0	470.0	0.0	1803.1	1486.4	14.1	3.5	197.4	0.42	155.9
PC2-L-C1-15	PC2	L	C1	15	399.5	70.5	1796.2	1480.7	8.8	3.7	197.4	0.42	155.5
PC2-L-C1-40	PC2	L	C1	40	282.0	188.0	1784.7	1471.2	4.4	4.0	197.4	0.42	154.8
PC2-L-C2-15	PC2	L	C2	15	399.5	70.5	1796.2	1480.7	7.0	3.5	197.4	0.42	155.4
PC2-L-C2-40	PC2	L	C2	40	282.0	188.0	1784.7	1471.2	2.2	4.6	197.4	0.42	154.6
PC2-L-F1-15	PC2	L	F1	15	399.5	70.5	1795.4	1480.1	9.7	4.0	197.4	0.42	155.3
PC2-L-F1-40	PC2	L	F1	40	282.0	188.0	1782.6	1469.5	0.7	6.1	197.4	0.42	154.3
PC2-L-F2-15	PC2	L	F2	15	399.5	70.5	1795.4	1480.1	3.5	4.2	197.4	0.42	155.3
PC2-L-F2-40	PC2	L	F2	40	282.0	188.0	1782.6	1469.5	2.6	5.3	197.4	0.42	154.3

Table 4.14. Mixture designs for concrete mixtures with PC3 cement.

Mixture ID	Cement Type	Aggregate Type	Fly Ash Source	Fly Ash Replacement	Cement Content	Fly Ash Content	Aggregate		Water Reducer	Air Entrainer	Water	Design *w/cm*	Air-Free Unit Weight
							Coarse	Fine					
				weight % cementitious	lbs/yd^3	lbs/yd^3	lbs/yd^3	lbs/yd^3	fl oz/yd^3	fl oz/yd^3	lbs/yd^3		lbs/ft^3
PC3-G-X	PC3	G	-	0	470.0	0.0	1864.3	1486.4	14.1	2.5	197.4	0.42	158.3
PC3-G-C1-15	PC3	G	C1	15	399.5	70.5	1857.2	1480.7	8.8	3.2	197.4	0.42	157.9
PC3-G-C2-15	PC3	G	C2	15	399.5	70.5	1857.2	1480.7	7.0	3.0	197.4	0.42	157.8
PC3-G-C2-30	PC3	G	C2	30	329.0	141.0	1850.0	1475.0	6.7	3.5	197.4	0.42	157.3
PC3-G-F1-15	PC3	G	F1	15	399.5	70.5	1856.4	1480.1	14.0	3.3	197.4	0.42	157.7
PC3-G-F2-15	PC3	G	F2	15	399.5	70.5	1856.4	1480.1	7.9	3.5	197.4	0.42	157.7
PC3-G-F2-30	PC3	G	F2	30	329.0	141.0	1848.4	1473.7	5.3	3.9	197.4	0.42	157.1
PC3-L-X	PC3	L	-	0	470.0	0.0	1803.1	1486.4	14.1	2.8	197.4	0.42	155.9
PC3-L-C1-30	PC3	L	C1	30	329.0	141.0	1789.3	1475.0	5.3	3.3	197.4	0.42	155.1
PC3-L-C1-40	PC3	L	C1	40	282.0	188.0	1784.7	1471.2	1.8	4.2	197.4	0.42	154.8
PC3-L-C2-30	PC3	L	C2	30	329.0	141.0	1789.3	1475.0	4.4	3.2	197.4	0.42	155.0
PC3-L-C2-40	PC3	L	C2	40	282.0	188.0	1784.7	1471.2	1.8	3.5	197.4	0.42	154.6
PC3-L-F1-30	PC3	L	F1	30	329.0	141.0	1787.7	1473.7	3.5	3.0	197.4	0.42	154.7
PC3-L-F1-40	PC3	L	F1	40	282.0	188.0	1782.6	1469.5	1.6	3.5	197.4	0.42	154.3
PC3-L-F2-30	PC3	L	F2	30	329.0	141.0	1787.7	1473.7	6.1	3.5	197.4	0.42	154.7
PC3-L-F2-40	PC3	L	F2	40	282.0	188.0	1782.6	1469.5	7.0	4.2	197.4	0.42	154.3

Table 4.15. Batch weights and fresh concrete properties for concrete mixtures with PC1 cement.

Mixture ID	Cement Content	Fly Ash Content	Aggregate (SSD)		Water Reducer	Air Entrainer	Total Water	*w/cm*	Measured Unit Weight	Slump	Air Content Pressure Meter	Air Content Gravimetric	Relative Yield
			Coarse	Fine									
	lbs	lbs	lbs	lbs	ml	ml	lbs		lbs/ft^3	inches	volume %	volume %	percent
PC1-G-X	90.5	0.0	359.0	286.3	85	20	38.0	0.41	148.4	2.0	5.9	6.3	100.3
PC1-G-C1-40	54.3	36.2	355.4	283.3	10	26	38.0	0.41	147.0	2.5	6.9	6.5	100.4
PC1-G-C2-15	76.9	13.6	357.6	285.2	30	22	38.0	0.41	147.6	2.0	6.9	6.5	100.5
PC1-G-C2-40	54.3	36.2	355.4	283.3	5	35	38.0	0.42	147.2	4.0	6.8	6.2	100.2
PC1-G-F1-40	54.3	36.2	355.0	283.0	4	23	38.0	0.41	147.4	2.0	5.4	5.9	100.0
PC1-G-F2-15	76.9	13.6	357.5	285.1	100	22	38.0	0.40	147.8	2.0	6.7	6.3	100.3
PC1-G-F2-40	54.3	36.2	355.0	283.0	30	37	38.0	0.41	147.4	2.0	6.0	5.9	100.0
PC1-L-X	90.5	0.0	347.0	286.3	80	22	38.0	0.41	146.4	2.0	6.5	6.0	100.1
PC1-L-C1-15	76.9	13.6	345.7	285.2	60	28	38.0	0.41	146.0	4.0	6.2	6.1	100.0
PC1-L-C1-30	63.4	27.2	344.3	284.1	30	25	38.0	0.42	145.8	4.0	6.8	5.9	99.9
PC1-L-C2-15	76.9	13.6	345.7	285.2	50	25	38.0	0.41	146.0	3.0	6.1	6.0	100.0
PC1-L-C2-30	63.4	27.2	344.3	284.1	40	28	38.0	0.40	145.6	4.0	6.0	6.0	100.0
PC1-L-F1-15	76.9	13.6	345.5	285.1	60	22	38.0	0.42	145.9	3.5	6.5	6.0	100.1
PC1-L-F1-30	63.4	27.2	344.0	283.8	40	24	38.0	0.41	145.5	4.0	6.0	5.9	100.0
PC1-L-F2-15	76.9	13.6	345.5	285.1	40	24	38.0	0.42	145.9	3.0	6.4	6.0	100.1
PC1-L-F2-30	63.4	27.2	344.0	283.8	20	26	38.0	0.42	145.6	2.0	5.8	5.8	99.9

Table 4.16. Batch weights and fresh concrete properties for concrete mixtures with PC2 cement.

Mixture ID	Cement Content	Fly Ash Content	Aggregate (SSD) Coarse	Aggregate (SSD) Fine	Water Reducer	Air Entrainer	Total Water	*w/cm*	Measured Unit Weight	Slump	Air Content Pressure Meter	Air Content Gravimetric	Relative Yield
	lbs	lbs	lbs	lbs	ml	ml	lbs		lbs/ft^3	inches	volume %	volume %	percent
PC2-G-X	90.5	0.0	359.0	286.3	80	20	38.0	0.42	148.4	3.5	7.0	6.3	100.3
PC2-G-C1-30	63.4	27.2	356.3	284.1	55	23	38.0	0.42	147.6	2.5	6.7	6.3	100.2
PC2-G-C2-30	63.4	27.2	356.3	284.1	50	24	38.0	0.42	147.9	2.0	5.8	6.0	100.0
PC2-G-C2-40	54.3	36.2	355.4	283.3	36	26	38.0	0.41	147.4	2.8	7.5	6.1	100.1
PC2-G-F1-30	63.4	27.2	356.0	283.8	30	23	38.0	0.42	147.4	3.0	7.5	6.1	100.3
PC2-G-F2-30	63.4	27.2	356.0	283.8	30	24	38.0	0.42	147.8	2.5	5.2	5.9	99.9
PC2-G-F2-40	54.3	36.2	355.0	291.9	20	28	38.0	0.42	147.6	2.5	5.2	5.8	99.9
PC2-L-X	90.5	0.0	347.0	286.3	80	20	38.0	0.41	146.3	3.5	6.8	6.1	100.2
PC2-L-C1-15	76.9	13.6	345.7	285.2	50	21	38.0	0.42	145.8	3.5	7.9	6.2	100.2
PC2-L-C1-40	54.3	36.2	343.5	283.3	25	23	38.0	0.41	145.0	3.8	6.9	6.3	100.2
PC2-L-C2-15	76.9	13.6	345.7	285.2	40	20	38.0	0.42	146.4	3.0	5.5	5.8	99.8
PC2-L-C2-40	54.3	36.2	343.5	283.3	13	26	38.0	0.40	145.0	3.0	7.0	6.2	100.2
PC2-L-F1-15	76.9	13.6	345.5	285.0	55	23	38.0	0.42	145.8	3.5	7.0	6.1	100.2
PC2-L-F1-40	54.3	36.2	343.1	283.0	4	35	38.0	0.42	145.0	4.0	6.9	6.0	100.1
PC2-L-F2-15	76.9	13.6	345.5	285.1	20	24	38.0	0.42	146.2	2.5	5.2	5.8	99.9
PC2-L-F2-40	54.3	36.2	343.1	283.0	15	30	38.0	0.42	145.4	2.5	5.2	5.7	99.8

Table 4.17. Batch weights and fresh concrete properties for concrete mixtures with PC3 cement.

Mixture ID	Cement Content	Fly Ash Content	Aggregate (SSD)		Water Reducer	Air Entrainer	Total Water	*w/cm*	Measured Unit Weight	Slump	Air Content Pressure Meter	Air Content Gravimetric	Relative Yield
			Coarse	Fine									
	lbs	lbs	lbs	lbs	ml	ml	lbs		lbs/ft^3	inches	volume %	volume %	percent
PC3-G-X	90.5	0.0	359.0	286.3	80	14	38.0	0.41	148.4	3.5	7.8	6.3	100.3
PC3-G-C1-15	76.9	13.6	357.6	285.2	50	18	38.0	0.42	148.3	3.8	5.9	6.1	100.0
PC3-G-C2-15	76.9	13.6	357.6	285.2	40	17	38.0	0.40	148.5	3.0	5.7	5.9	99.9
PC3-G-C2-30	63.4	27.2	356.3	284.1	38	20	38.0	0.42	148.3	2.8	5.4	5.7	99.7
PC3-G-F1-15	76.9	13.6	357.5	285.1	80	19	38.0	0.42	148.2	2.0	5.7	6.0	100.1
PC3-G-F2-15	76.9	13.6	357.5	285.1	45	20	38.0	0.41	148.0	2.3	6.2	6.1	100.2
PC3-G-F2-30	63.4	27.2	356.0	283.8	30	22	38.0	0.40	148.0	2.3	5.0	5.8	99.9
PC3-L-X	90.5	0.0	347.0	286.3	80	16	38.0	0.42	146.2	3.3	6.5	6.2	100.2
PC3-L-C1-30	63.4	27.2	344.3	284.1	30	19	38.0	0.41	145.2	4.0	7.6	6.3	100.3
PC3-L-C1-40	54.3	36.2	343.5	283.3	10	24	38.0	0.42	145.5	3.5	6.3	6.0	99.9
PC3-L-C2-30	63.4	27.2	344.3	284.1	25	18	38.0	0.42	145.2	4.0	8.2	6.3	100.3
PC3-L-C2-40	54.3	36.2	343.5	283.3	10	20	38.0	0.42	145.4	3.5	6.8	5.9	99.9
PC3-L-F1-30	63.4	27.2	344.0	283.8	20	17	38.0	0.42	145.2	4.0	7.7	6.1	100.2
PC3-L-F1-40	54.3	36.2	343.1	283.0	9	20	38.0	0.42	144.8	2.8	7.2	6.1	100.3
PC3-L-F2-30	63.4	27.2	344.0	283.0	35	20	38.0	0.43	145.6	3.8	5.7	5.8	99.9
PC3-L-F2-40	54.3	36.2	343.1	283.0	40	24	38.0	0.43	145.4	3.5	5.0	5.7	99.8

4.3.3 Air Entraining Admixture Dosage

The AEA dosage established for each mixture was developed through a series of trial mixtures that served to bracket the dosage needed for a given ash and cement combination. For each mixture, based on the results from the trial mixtures, initial AEA dosages were estimated and adjustments were made to that dosage while mixing, based on the measurement of the fresh air content using the pressure meter.

When using a Class F fly ash, a primary concern is the affect of the ash on air entrainment. Figure 4.10 through Figure 4.21 present a series of combined plots that show for each mixture i) the fresh air content by pressure meter and by gravimetric determination, ii) the AEA dosage used for each mixture, and iii) the AEA dosage predicted based on the DAI. The DAI test reports the AEA demand per gram of fly ash. Therefore, knowing the AEA dosage required for the cement only mixtures (i.e., baseline dosage), and knowing the result of the DAI test for each ash, a correction to the baseline dosage can be estimated. In each combined plot, the baseline (cement-only) admixture dosage is provided for reference. Note, the AEA dosage for a given cement type varied as a function of the aggregate used, so both dosages are presented. When comparing fly ash mixtures to cement-only mixtures, baseline mixtures using the same aggregate type should be compared. The air content should be 5 – 7 volume % across all plots if the AEA dosage was corrected properly for the substitution of the fly ash. The numeric values of percent change in AEA dosage are shown in Table 4.18 while the numeric values of percent change in air content by the pressure meter and gravimetric methods are shown in Table 4.19 and Table 4.20, respectively

The general trend is an increase in AEA dosage as ash content increases and higher percent increases in dosage when Class F ash is used, as compared to Class C. However, it should be noted in most cases the Class C ashes also required an increase in AEA dosage. When examining the air contents in general, even with an increase in AEA dosage, many air contents were lower in the fly ash mixtures as compared to the cement only mixtures. Also, the variation in pressure meter air content was noticeably larger than seen in the gravimetric air content determination. This was unexpected as the pressure meter and gravimetric air contents were determined on exactly the same concrete sample within a few minutes of each other.

In Figure 4.10 through Figure 4.21, the AEA dosages predicted by the DAI tests are also shown. The AEA dosage corrections were determined based on the actual mixture AEA concentration, which ranged from 0.08 to 0.21 % volume AEA. The predicted dosage increases with ash content, as expected. In most cases, the predicted dosage was more than the actual dosage arrived at by trial and error. Given the AEA dosage used resulted in a decrease in air content when comparing fly ash mixtures to cement-only mixtures, the higher dosages predicted by the DAI method may be valid. Overall, for a given fly ash, the DAI predicted AEA dosage is a reasonable first estimate of the needed AEA dosage and represents quite accurately the trend in AEA dosage as the fly ash content or fly ash adsorption increases.

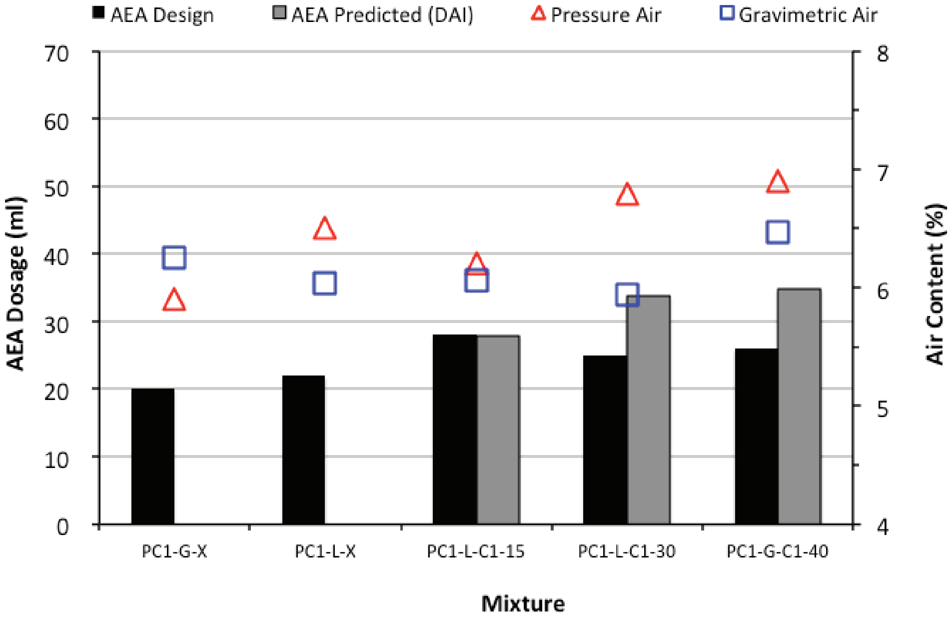


Figure 4.10. Summary graph for the PC1-C1 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

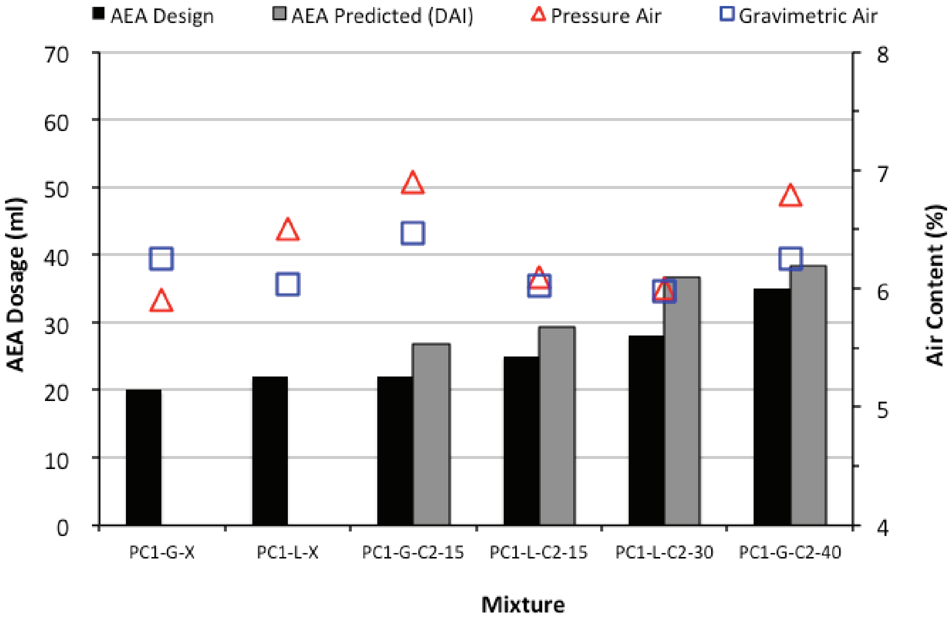


Figure 4.11. Summary graph for the PC1-C2 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

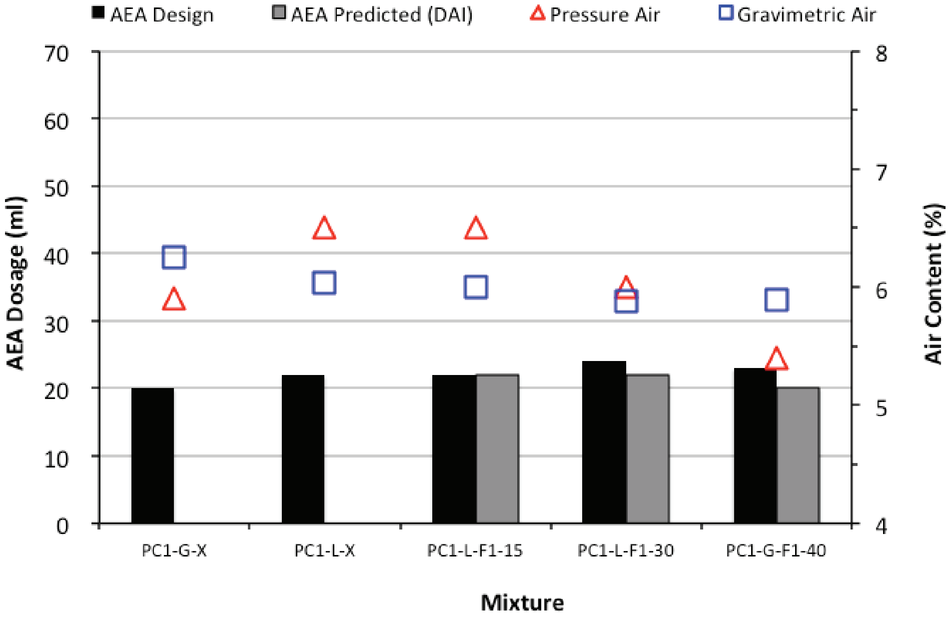


Figure 4.12. Summary graph for the PC1-F1 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

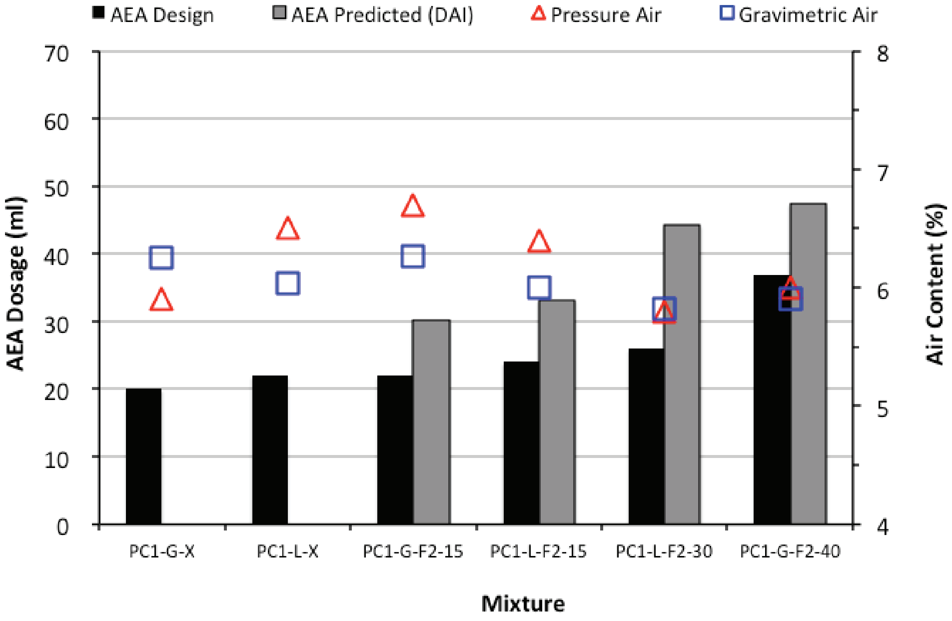


Figure 4.13. Summary graph for the PC1-F2 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

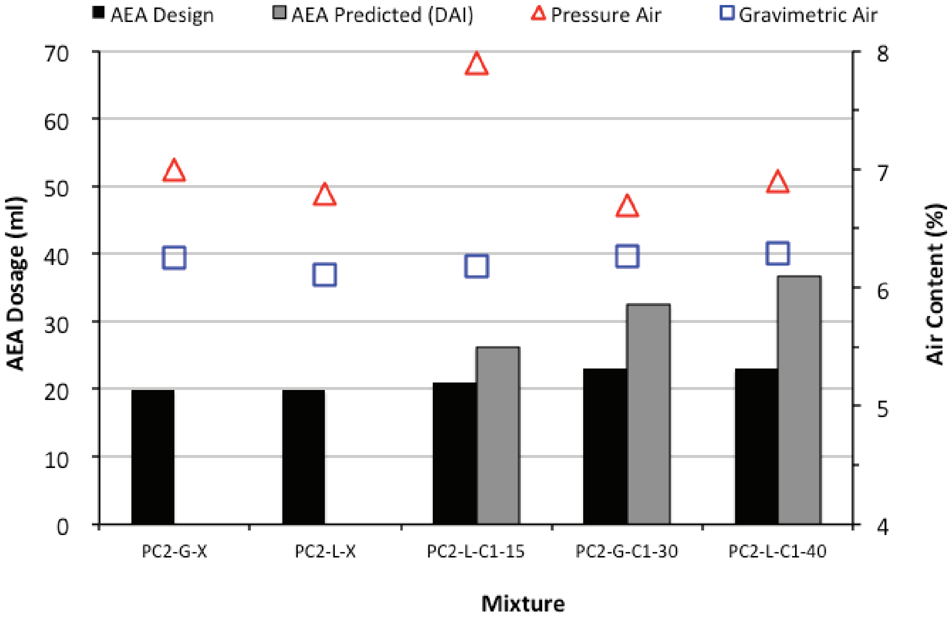


Figure 4.14. Summary graph for the PC2-C1 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

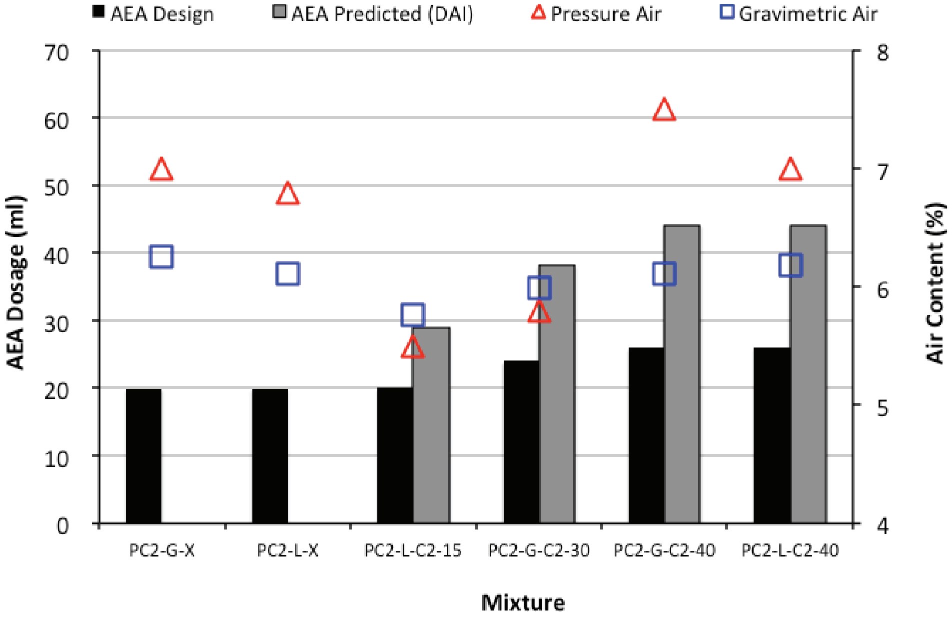


Figure 4.15. Summary graph for the PC2-C2 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

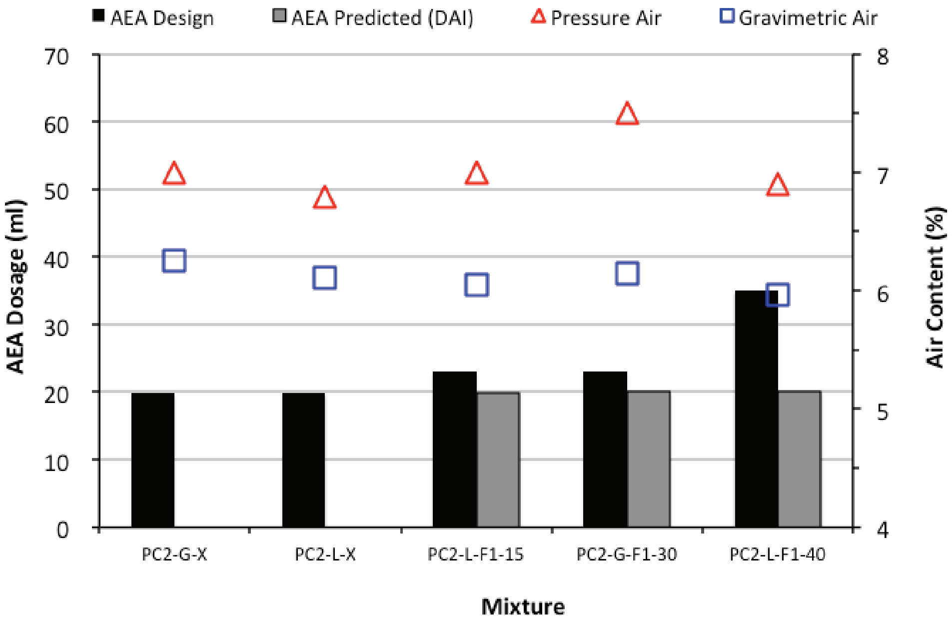


Figure 4.16. Summary graph for the PC2-F1 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

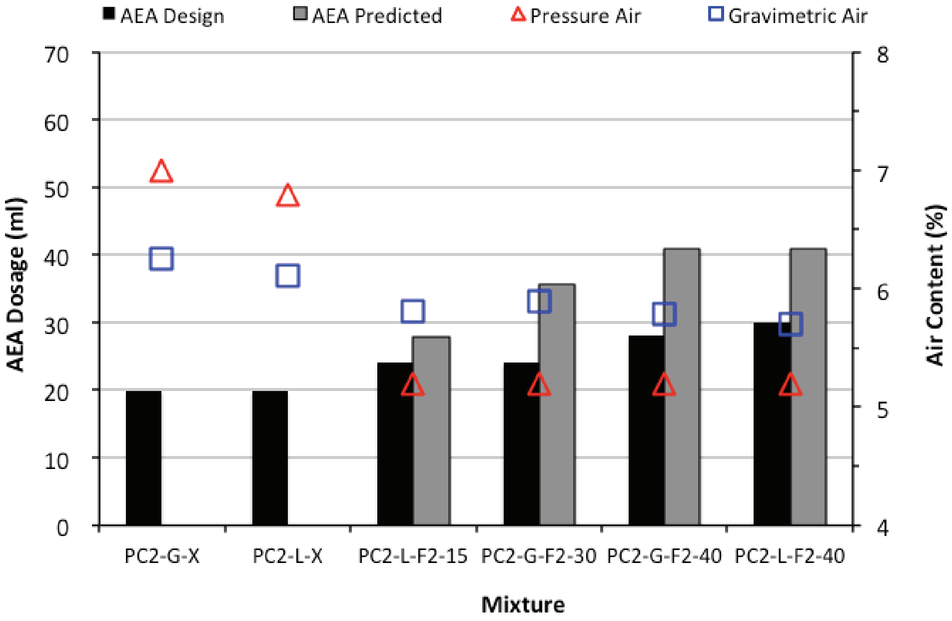


Figure 4.17. Summary graph for the PC2-F2 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

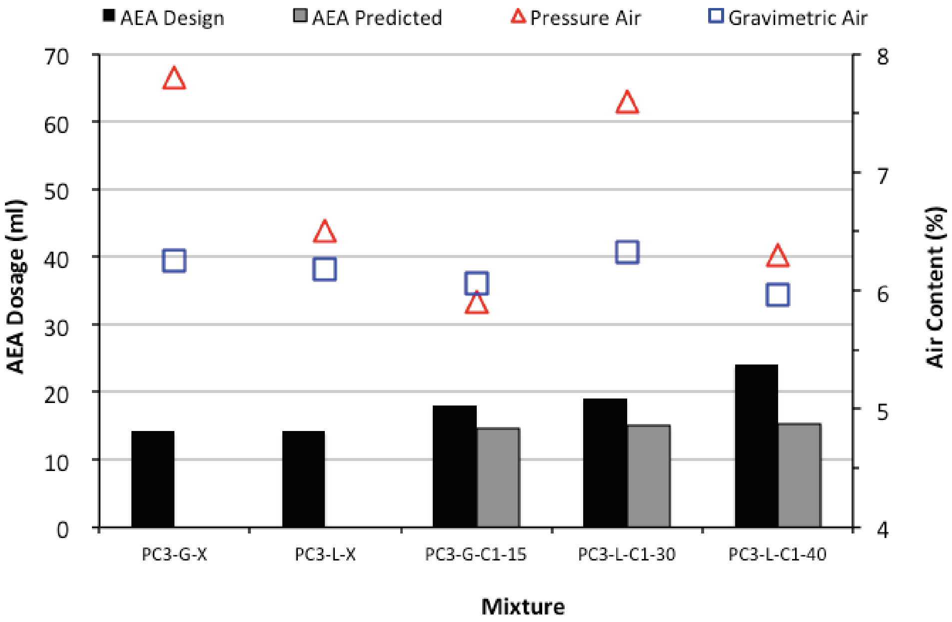


Figure 4.18. Summary graph for the PC3-C1 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

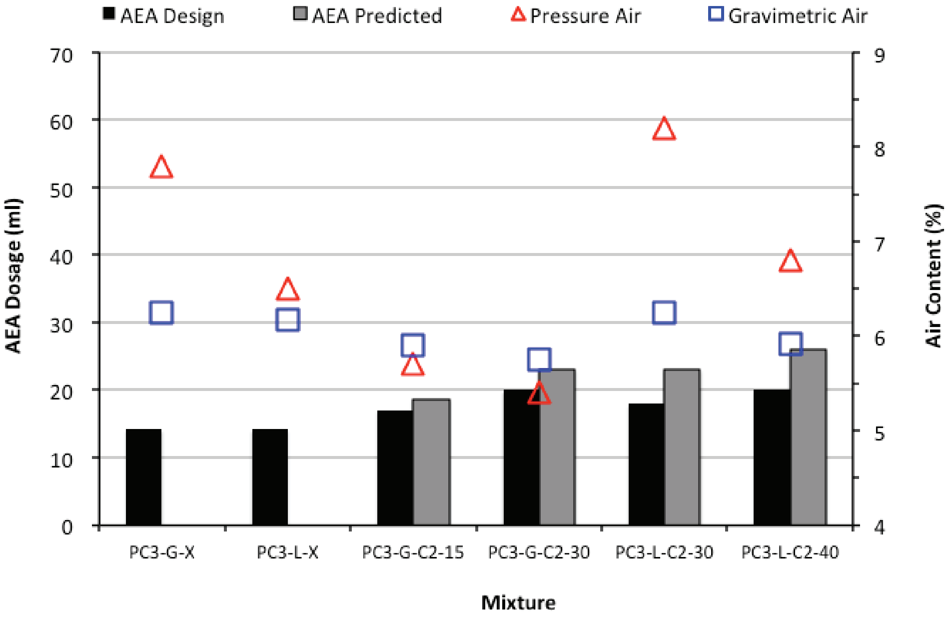


Figure 4.19. Summary graph for the PC3-C2 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

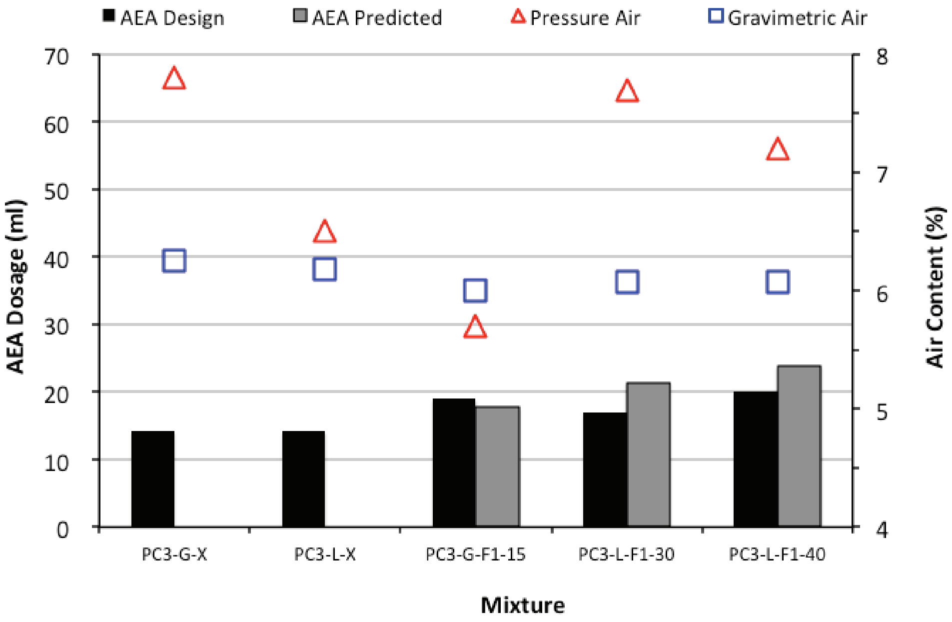


Figure 4.20. Summary graph for the PC3-F1 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

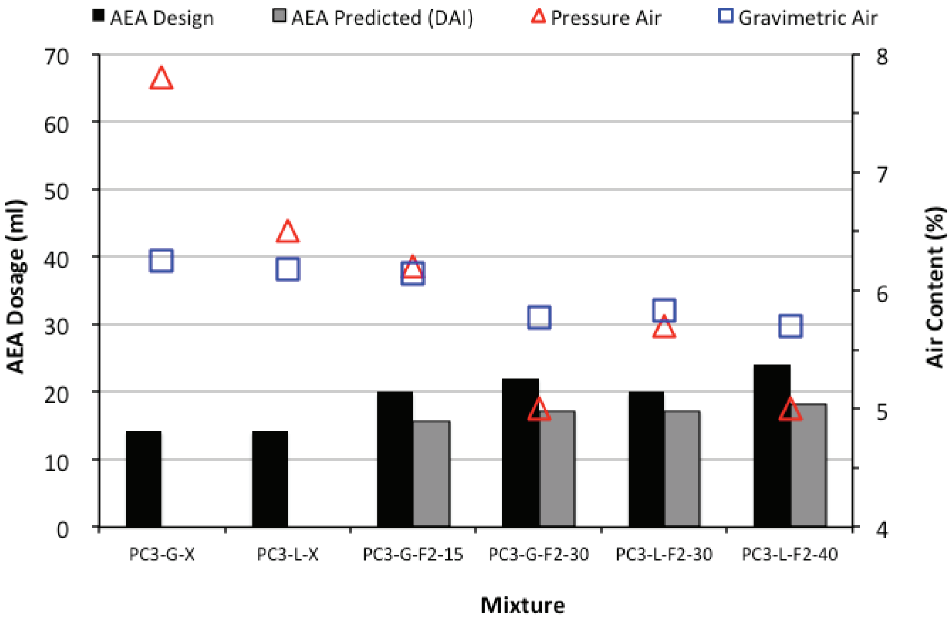


Figure 4.21. Summary graph for the PC3-F2 mixtures showing the AEA dosage used in the mixtures (AEA Design), AEA dosage predicted based on the results of the direct adsorption isotherm test (AEA Predicted DAI), measured air content as determined by the AASHTO T 152 pressure meter test (Pressure Air), and the measured air content as determined by the AASHTO T 121 gravimetric air test (Gravimetric Air).

Table 4.18. Percent change in AEA dosage comparing the fly ash containing mixtures with the cement-only mixtures. For all mixtures, AEA dosage was determined by trial and error.

	Aggregate Source G						Aggregate Source L					
	Class C Replacement (weight %)			Class F Replacement (weight %)			Class C Replacement (weight %)			Class F Replacement (weight %)		
Mixture	*15*	*30*	*40*	*15*	*30*	*40*	*15*	*30*	*40*	*15*	*30*	*40*
PC1-C1			30.0				27.3	13.6				
PC1-C2	10.0		75.0				13.6	27.3				
PC1-F1						15.0				0.0	9.1	
PC1-F2				10.0		85.0				9.1	18.2	
PC2-C1		15.6					5.5		15.6			
PC2-C2	0.5		30.7				0.5		30.7			
PC2-F1					15.6					15.6		75.9
PC2-F2					20.6	40.7				20.6		50.8
PC3-C1	26.8							33.8	69.0			
PC3-C2	19.7	40.8						26.8	40.8			
PC3-F1				33.8							19.7	40.8
PC3-F2				40.8	54.9						40.8	69.0

Table 4.19. Percent change in air content measured by the pressure meter method AASHTO T 152, comparing the fly ash containing mixtures with the cement-only mixtures. For all mixtures, AEA dosage was determined by trial and error.

	Aggregate Source G						Aggregate Source L					
	Class C Replacement (weight %)			Class F Replacement (weight %)			Class C Replacement (weight %)			Class F Replacement (weight %)		
Mixture	*15*	*30*	*40*	*15*	*30*	*40*	*15*	*30*	*40*	*15*	*30*	*40*
PC1-C1			16.9				-4.6	4.6				
PC1-C2	16.9		15.3				-6.2	-7.7				
PC1-F1						-8.5				0.0	-7.7	
PC1-F2				13.6		1.7				-1.5	-10.8	
PC2-C1		-4.3					16.2		1.5			
PC2-C2	-21.4		7.1				-19.1		2.9			
PC2-F1					7.1					2.9		1.5
PC2-F2					-25.7	-25.7				-23.5		-23.5
PC3-C1	-24.4							16.9	-3.1			
PC3-C2	-26.9	-30.8						26.2	4.6			
PC3-F1				-26.9							18.5	10.8
PC3-F2				-20.5	-35.9						-12.3	-23.1

Table 4.20. Percent change in air content measured by the gravimetric method AASHTO T 121, comparing the fly ash containing mixtures with the cement-only mixtures. For all mixtures, AEA dosage was determined by trial and error.

	Aggregate Source G						Aggregate Source L					
	Class C Replacement (weight %)			Class F Replacement (weight %)			Class C Replacement (weight %)			Class F Replacement (weight %)		
Mixture	*15*	*30*	*40*	*15*	*30*	*40*	*15*	*30*	*40*	*15*	*30*	*40*
PC1-C1			3.5				0.4	-1.7				
PC1-C2	3.3		-0.2				-0.3	-1.0				
PC1-F1						-5.9				-0.7	-2.6	
PC1-F2				0.1		-5.6				-0.6	-3.6	
PC2-C1		0.2					1.1		2.9			
PC2-C2	-8.0		-2.2				-5.8		1.2			
PC2-F1					-1.7					-1.0		-2.5
PC2-F2					-5.7	-7.4				-4.9		-6.7
PC3-C1	-3.1							2.4	-3.4			
PC3-C2	-5.7	-8.1						1.2	-4.2			
PC3-F1				-4.0							-1.6	-1.7
PC3-F2				-1.8	-7.7						-5.6	-7.6

4.4 Calorimetry

Calorimetry is used to monitor the progress of cement hydration in a concrete mixture. Generally speaking, heat evolution as a result of cement hydration begins with an initial rapid increase and then subsequent rapid decrease in concrete temperature at the time of mixing (duration ~ 15 minutes), a dormant period where the concrete is plastic and remains slightly above ambient temperature (duration ~ 2-4 hours), and then a hardening period where a steady increase in concrete temperature is observed as the concrete stiffens, commencing with the initial set and progressing to final set (duration ~ 2-4 hours). These periods are followed by cooling and further densification through hydration. For the calorimetry plots shown in this report, the initial temperature increase at mixing is not recorded. Calorimetry was performed using a Grace Adiacal™ semi-adiabatic calorimeter. The plots provided show the measured temperature increase of each mixture, which is representative of the heat evolution.

Calorimetry can be used as a quantitative tool to determine the actual heat evolution of a concrete mixture. Or, it can be used as a qualitative tool to compare the heat evolution curves from different mixtures and thereby determine the result of blending various materials or admixtures. For this study, a partial factorial approach was used for the experimental design and therefore, not every combination of materials were prepared. However, by qualitatively comparing those prepared, some general effects can be identified. The calorimeter temperature curves from all mixtures are included in Appendix C. Selected temperature curves are shown below to illustrate the main points observed.

Figure 4.22 and Figure 4.23 show the temperature increase of mixtures prepared using no fly ash. Figure 4.22 shows the mixtures with the glacial gravel aggregate while Figure 4.23 shows the mixtures prepared using the quarried carbonate aggregate. The PC1 and PC3 cements show similar temperature curves while the PC1 cement shows a lower maximum temperature and a heat evolution curve shifted to a later time, indicating a slower setting time.

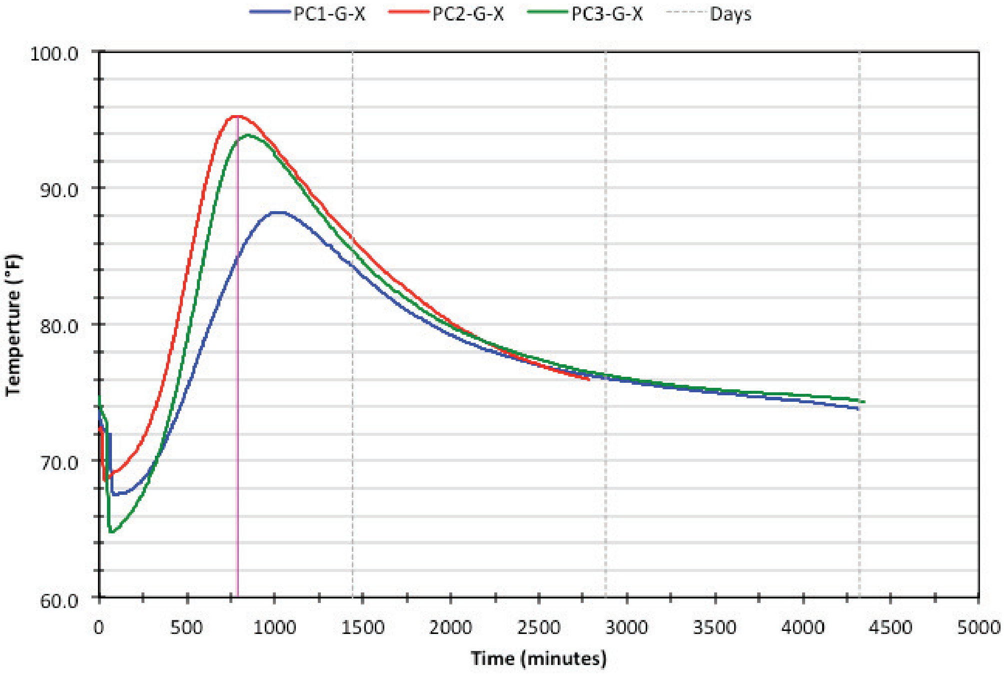


Figure 4.22. Temperature curves determined by semi-adiabatic calorimetry for mixtures with each cement, no fly ash, and the glacial gravel aggregate. Magenta line indicates earliest temperature maxima.

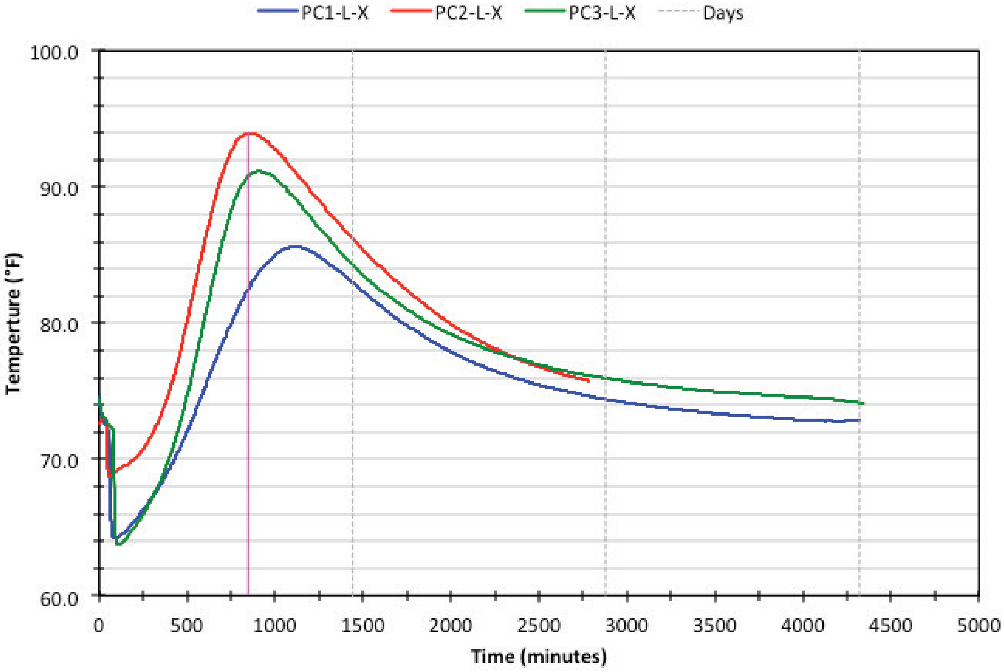


Figure 4.23. Temperature curves determined by semi-adiabatic calorimetry for mixtures with each cement, no fly ash, and the quarried carbonate aggregate. Magenta line indicates earliest temperature maxima.

Figure 4.24 through Figure 4.29 show the temperature curves for selected mixtures containing fly ash and these illustrate the key observations from all of the calorimetry data. In Figure 4.24 the PC1-G-C2-15 mixture shows a temperature curve slightly lower than the cement-only curve. This is indicative of a reduced heat of hydration due to a lower portland cement content, but also because fly ash is slower to react. As will be discussed with the maturity data, this results in a slower rate of strength gain. Class C ash is often found to retard the final set as shown by a shift in the temperature maxima to a later time. Examining the calorimetry data in Appendix C, this effect can be seen in a number of cases, though not pronounced. In Figure 4.24 the PC1-G-C2-40 curve shows the effect of a higher fly ash replacement level. Namely, the maximum temperature is further reduced but because the fly ash hydrates more slowly, and starts later, final set is delayed and the temperature stays near its maximum for a longer time period, extending the duration of the hydration process. This slower rate of temperature reduction can be seen in PC1-G-C2-15 but to a lesser extent. These same trends can be seen in Figure 4.26 and Figure 4.28, which show different combinations and replacement levels of cement and Class C ash. In all cases the heat of hydration is reduced and the hydration period is extended; these effects increase as the amount of Class C ash in the mixture is increased.

In Figure 4.25, Figure 4.27, and Figure 4.29, the affect of Class F ash is seen. Namely, a clear reduction in the maximum temperature and a reduced rate of heat evolution in the first 12-16 hours is observed. When comparing hydration of a Class F ash to a Class C ash, the biggest difference is after the maximum in the temperature curve. With Class F ash, the temperature reduction occurs at a faster rate, similar to the cement-only mixtures. The prolonged maxima and extended hydration process common with Class C ash is not observed. This is because the Class F ash is primarily pozzolanic while the Class C ash has cementitious properties. The pozzolanic reaction does not begin in earnest until a number of days later and therefore no evidence is seen in the early calorimetry curves. As will be seen when examining the maturity data, the pozzolanic reaction does not contribute significantly until week 2 or week 3. Regardless of the type of ash, it can be seen that both Class C and Class F ash reduce the initial temperature, which can result in reduced shrinkage cracking and in the case of mass concrete placements, reduced thermal cracking. The primary difference is a Class F ash will result in a reduced early strength due to the ash's hydration characteristics.

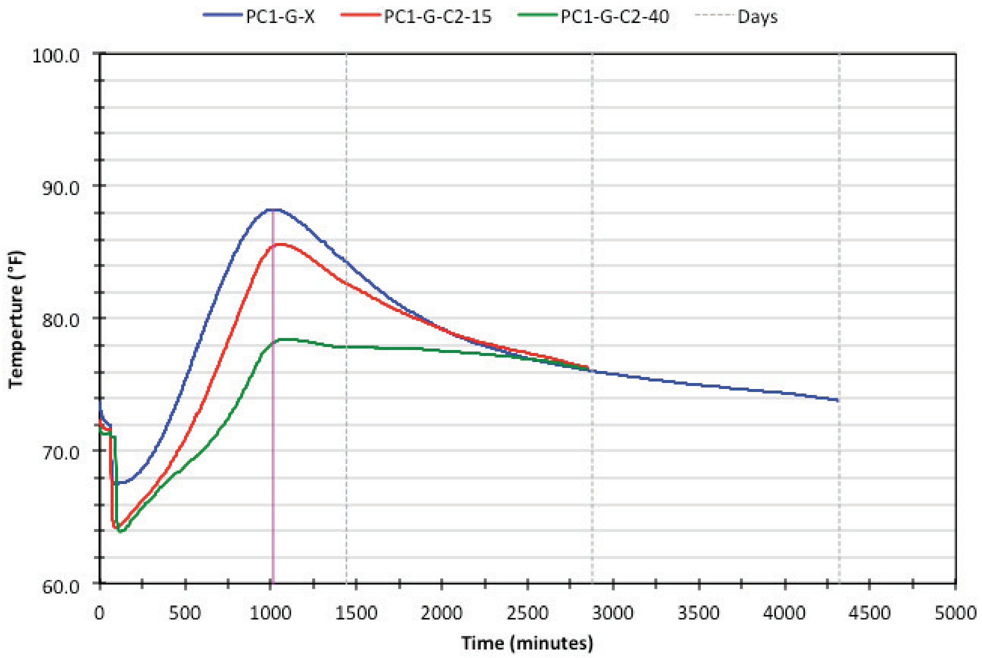


Figure 4.24. Mixture temperature determined by semi-adiabatic calorimetry for mixtures with PC1 cement, fly ash C2 at 15 and 30%, and the glacial gravel aggregate. The temperature curve for PC1 cement, glacial gravel aggregate, and no ash is included for comparison. Magenta line indicates earliest temperature maxima.

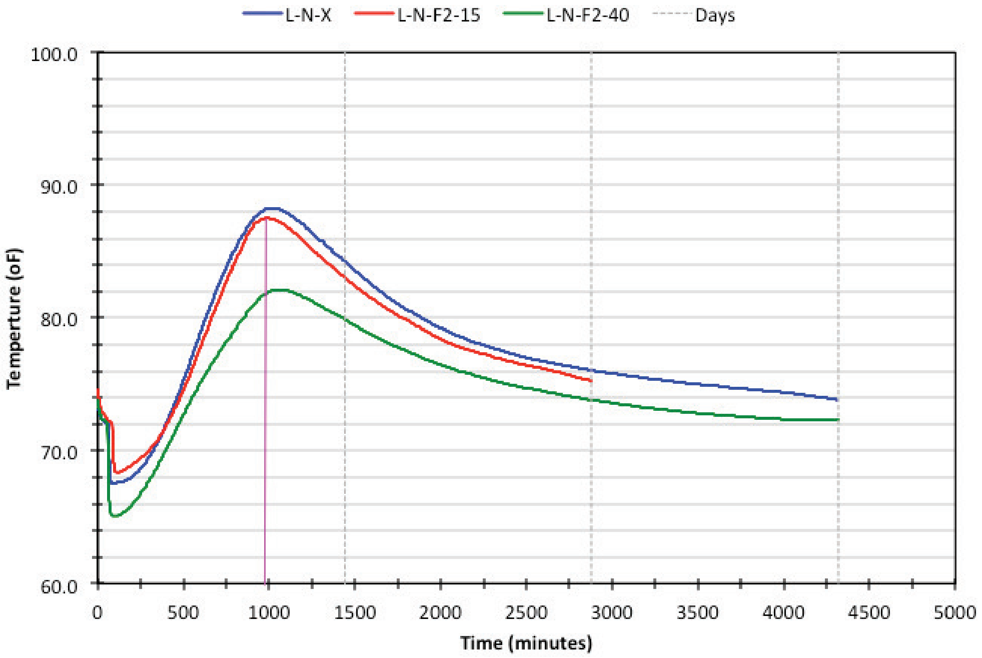


Figure 4.25. Mixture temperature determined by semi-adiabatic calorimetry for mixtures with PC1 cement, fly ash F2 at 15 and 40%, and the glacial gravel aggregate. The temperature curve for PC1 cement, glacial gravel aggregate, and no ash is included for comparison. Magenta line indicates earliest temperature maxima.

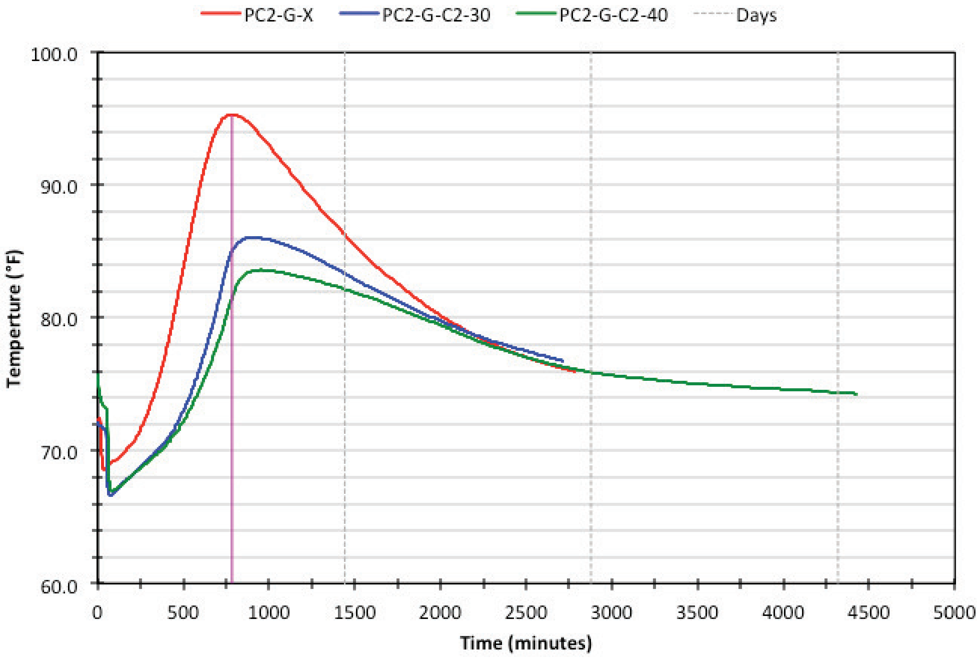


Figure 4.26. Mixture temperature determined by semi-adiabatic calorimetry for mixtures with PC2 cement, fly ash C2 at 30 and 40%, and the glacial gravel aggregate. The temperature curve for PC2 cement, glacial gravel aggregate, and no ash is included for comparison. Magenta line indicates earliest temperature maxima.

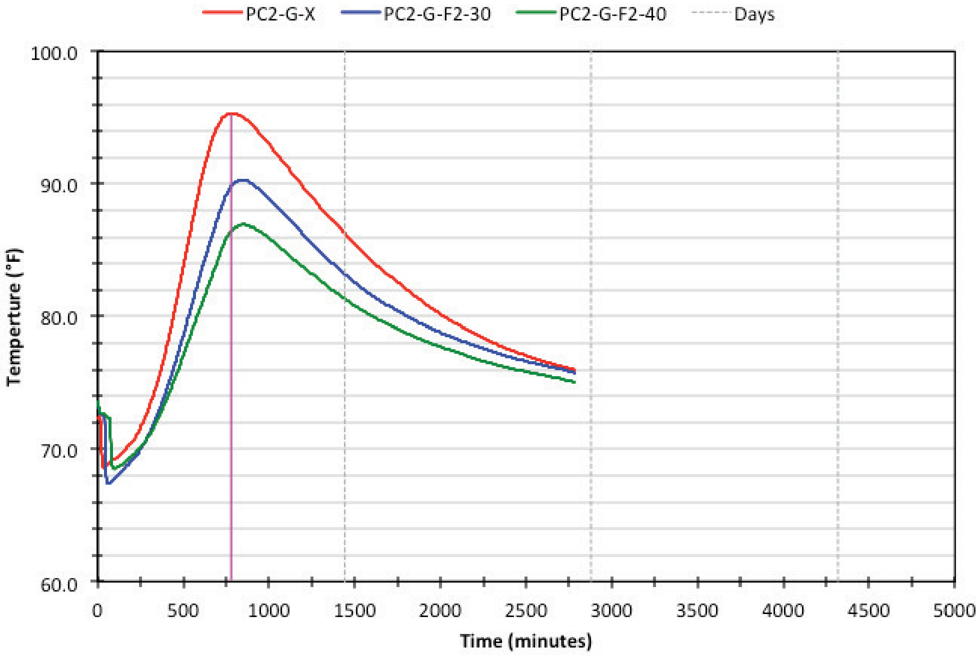


Figure 4.27. Mixture temperature determined by semi-adiabatic calorimetry for mixtures with PC2 cement, fly ash F2 at 30 and 40%, and the glacial gravel aggregate. The temperature curve for PC2 cement, glacial gravel aggregate, and no ash is included for comparison. Magenta line indicates earliest temperature maxima.

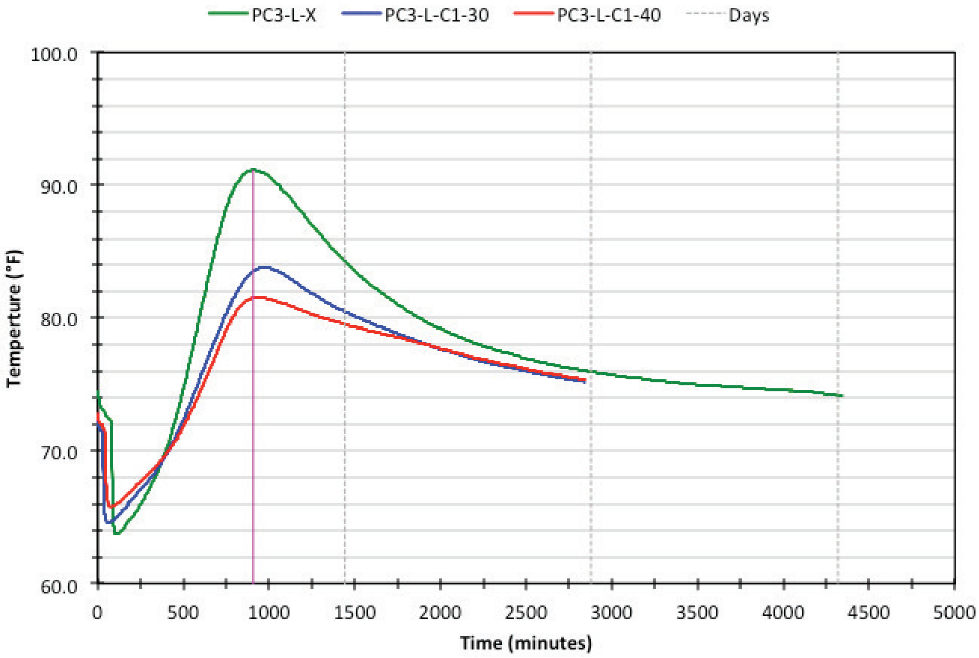


Figure 4.28. Mixture temperature determined by semi-adiabatic calorimetry for mixtures with PC3 cement, fly ash C1 at 30 and 40%, and the quarried carbonate aggregate. The temperature curve for PC3 cement, quarried carbonate aggregate, and no ash is included for comparison. Magenta line indicates earliest temperature maxima.

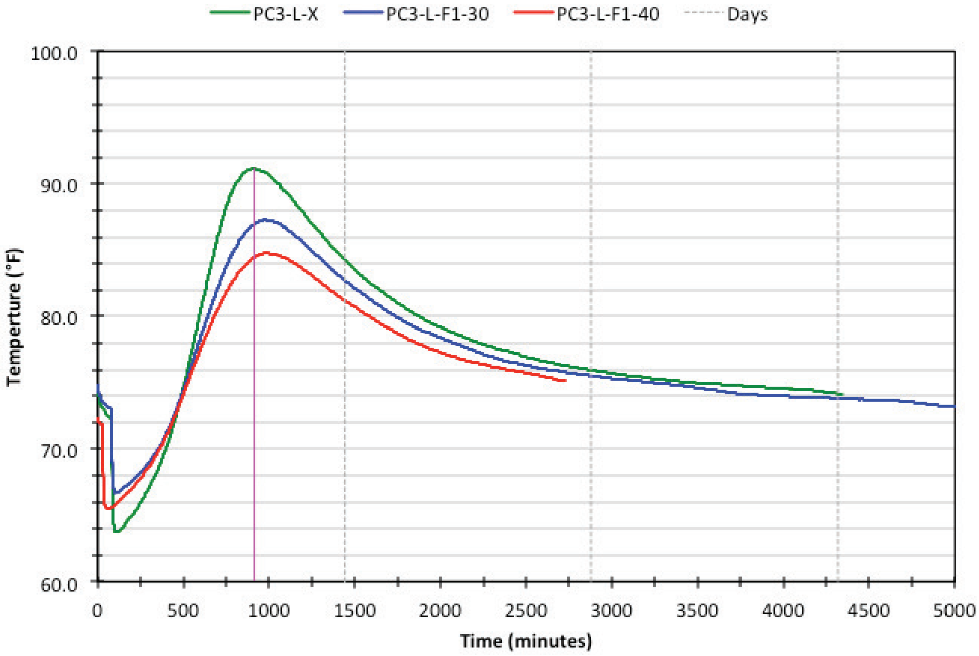


Figure 4.29. Mixture temperature determined by semi-adiabatic calorimetry for mixtures with PC3 cement, fly ash F1 at 30 and 40%, and the quarried carbonate aggregate. The temperature curve for PC3 cement, quarried carbonate aggregate, and no ash is included for comparison. Magenta line indicates earliest temperature maxima.

4.5 Hardened Concrete Testing

4.5.1 Freeze-Thaw Test Results

The results of freeze-thaw testing are presented in Table 4.21 and Table 4.22, and Figure 4.30. Five mixtures terminated with a durability factor less than 80, but greater than 60. Five mixtures terminated with a durability factor of less than 60. Only two mixtures did not reach the full 300 cycles: PC2-L-F2-15 (n=210) and PC2-L-F2-40 (n=115). Given the harsh exposure of Method A performed with 4% $CaCl_2$ solution, overall, the majority of the mixtures performed very well. An example of the harshness of the modified test is sample PC1-L-X. The PC1-L-X mixture is straight portland cement, no fly ash substitution. It is the experience of the research team that properly prepared straight portland cement mixtures, with a cement factor of 470 lbs., do not fail the AASHTO T 161 test Method A when performed with water. The fact that a straight portland cement mixture dropped below the 80% threshold for the durability factor, indicates the extraordinary exposure scenario used in this test. All five mixtures that fell below 60% durability factor contained the quarried carbonate aggregate. Two of the five mixtures that fell between 60% and 80% durability factor contained the quarried carbonate aggregate. Four of the five mixtures that fell below 60% durability factor contained ash source F2. Three of the five mixtures that fell between 60% and 80% durability factor contained the either ash source F1 or F2. There was no clear trend with cement type although as previously mentioned, one mixture with cement and no ash fell below the 80% durability factor threshold.

Beyond the physical data shown in Table 4.21 and Table 4.22, and Figure 4.30, the other observation from the freeze-thaw testing was the pronounced scaling that occurred with some mixtures. Figure 4.31 shows example images. Additional images are provided in Appendix D. The scaling was more severe then typically observed in the AASHTO T 161 test, but not unusual for exposure to dilute $CaCl_2$ solution. Interestingly, most of the prisms showing scaling did not show an associated drop in relative dynamic modulus of elasticity. Based solely on visual observation, the scaling appeared to be more prominent when quarried carbonate aggregate, Class F ash, or PC1 cement was part of the mixture. It should be reiterated this is a limited sample, only a visual observation and also, the dynamic modulus did not always diminish with the presence of scaling.

Table 4.21. Durability factor at 300 cycles for mixtures prepared with the glacial gravel aggregate source. All tests reached 300 cycles (n=300). Mixtures failing AASHTO T 161 (DF < 80%) highlighted in gray.

Cement →	PC1							PC2							PC3						
Ash Type →		C1	C2	C2	F1	F2	F2		C1	C2	C2	F1	F2	F2		C1	C2	C2	F1	F2	F2
Level →	0	40	15	40	40	15	40	0	30	30	40	30	30	40	0	15	15	30	15	15	30
Durability Factor (%)	90	100	96	100	95	94	89	98	100	100	100	**77**	**77**	**62**	98	100	100	100	100	93	90
Air Content Pressure Meter (volume %)	5.9	6.9	6.9	6.8	5.4	6.7	6.0	7.0	6.7	5.8	7.5	**7.5**	**5.2**	**5.2**	7.8	5.9	5.7	5.4	5.7	6.2	5.0
Air Content Gravimetric (volume %)	6.3	6.5	6.5	6.2	5.9	6.3	5.9	6.3	6.3	6.0	6.1	**6.1**	**5.9**	**5.8**	6.3	6.1	5.9	5.7	6.0	6.1	5.8

Table 4.22. Durability factor at 300 cycles for mixtures prepared with the quarried carbonate aggregate source. All tests reached 300 cycles (n=300) except PC2-L-F2-15 (n=210) and PC2-L-F2-40 (n=115). Mixtures failing AASHTO T 161 (DF < 80%) highlighted in gray.

Cement →	PC1									PC2									PC3								
Ash Type →		C1		C2		F1		F2			C1		C2		F1		F2			C1		C2		F1		F2	
Level →	0	15	30	15	30	15	30	15	30	0	15	40	15	40	15	40	15	40	0	30	40	30	40	30	40	30	40
Durability Factor (%)	**69**	**58**	**68**	98	89	97	100	92	100	92	99	100	100	96	97	100	**58**	**58**	90	100	100	100	100	94	97	**50**	**58**
Air Content Pressure Meter (volume %)	**6.5**	**6.2**	**6.8**	6.1	6.0	6.5	6.0	6.4	5.8	6.8	7.9	6.9	5.5	7.0	7.0	6.9	**5.2**	**5.2**	6.5	7.6	6.3	8.2	6.8	7.7	7.2	**5.7**	**5.0**
Air Content Gravimetric (volume %)	**6.0**	**6.1**	**5.9**	6.0	6.0	6.0	5.9	6.0	5.8	6.1	6.2	6.3	5.8	6.2	6.1	6.0	**5.8**	**5.7**	6.2	6.3	6.0	6.3	5.9	6.1	6.1	**5.8**	**5.7**

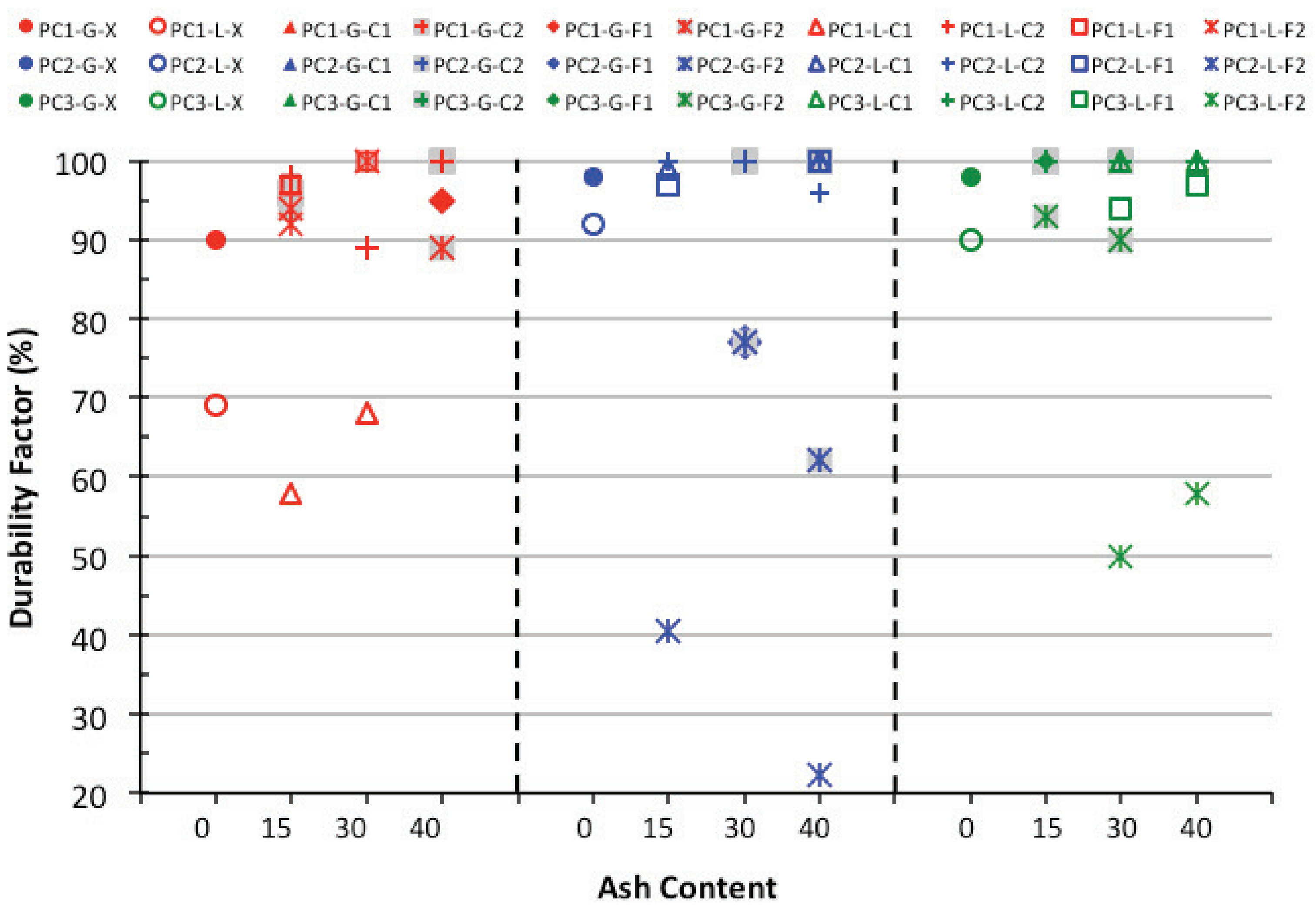


Figure 4.30. Durability factor for mixtures organized by cement type. Legend indicates the mixture design.

(Mixture PC1-L-X) (Mixture PC2-G-F2-30) (Mixture PC1-L-C1-15)

Figure 4.31. Example images of AASHTO T 161 prisms showing scaling. Additional images are presented in Appendix D.

4.5.2 Compressive and Flexural Strength Test Results

The results of compressive and flexural strength testing are presented in Table 4.23 through Table 4.25 and in Figure 4.32 through Figure 4.39. In general the following observations are made.

4.5.2.1 Compressive Strength

Class C Ash Sources

For ash source C1, the 28-day strength appears similar to the cement-only mixtures for a 15 and 30% replacement level. The strength gain at 28 days varies with cement type for a 15% replacement level. At 90 days a noticeable increase in compressive strength is observed for mixtures with a 15 and 30% replacement level. At a 40% replacement levels, early age strength gain is slower and the 28-day strengths are noticeably reduced. The 90-day strength level is approximately the same as the cement-only mixtures but the strength at 90 days also appears to be dependent on the cement type. For ash source C2, the same trends as seen with ash source C1 were observed except the strength gain at 90 days for the 15 and 30% replacement levels was noticeably higher.

Class F Ash Sources

For ash source F1 and F2 the early age strengths were noticeably reduced for all ash replacement levels. The reduction in early age strength was more affected by ash replacement level than observed with the Class C ash sources. However at 90 days, the strengths observed equaled or exceeded the strengths of the cement-only mixtures for all replacement levels with the partial exception of mixtures with a 40% replacement level of ash source F2. In the latter case, the strengths noticeably varied with cement type.

4.5.2.2 Flexural Strength

Class C Ash Sources

As compared to cement-only mixtures, the flexural strength of mixtures with Class C ash showed a very pronounced reduction in early strength for both ash sources. The rate of strength gain was noticeably reduced. The C1 source at a 15% replacement level showed a significant variation based on cement or aggregate type; the exact cause is not clear. In the case of C2 at a 40% replacement level an effect by aggregate type is clearly seen. At 90 days the strengths were comparable to the cement-only mixtures. The effect of increased fly ash replacement is more evident with flexural strength than was seen with compressive strength.

Class F Ash Sources

The Class F ash sources performed in a manner similar to the Class C sources. When compared to cement-only mixtures there is a noticeable reduction in the rate of strength gain, lower early strengths, but equal or increased 90 day strengths.

Table 4.23. Compressive and flexural strength of mixtures prepared with PC1 cement.

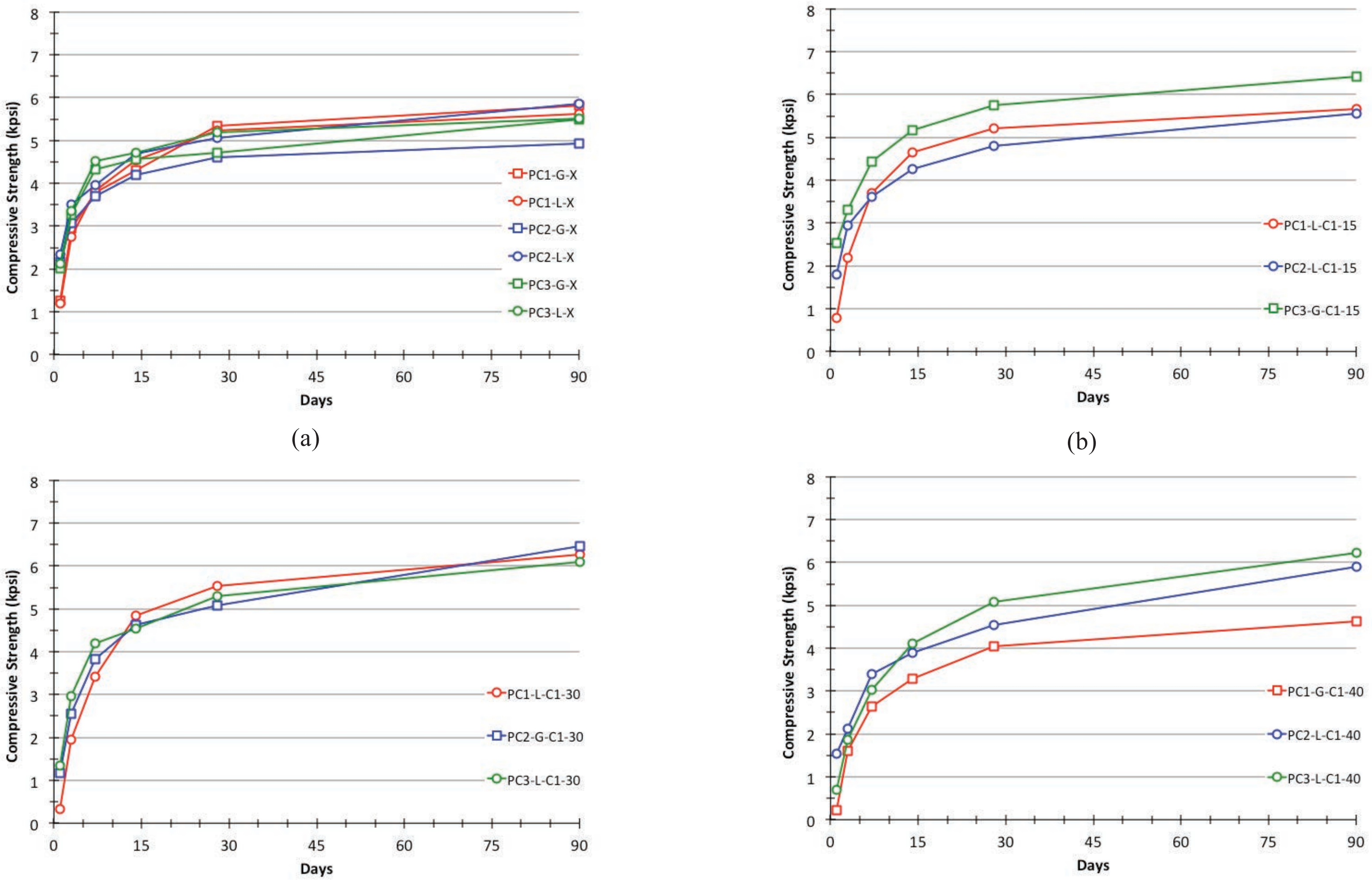
Mixture ID	Compressive Strength (psi)						Flexural Strength (psi)			
	1-day	3-day	7-day	14-day	28-day	90-day	3-day	7-day	28-day	90-day
PC3-G-X	1270	2945	3795	4310	5335	5815	440.0	580.0	642.5	695.0
PC3-G-C1-15	225	1600	2650	3295	4035	4635	337.5	445.0	647.5	692.5
PC3-G-C2-15	695	2585	4400	4920	6140	6715	437.5	567.5	622.5	667.5
PC3-G-C2-30	200	1420	3030	3830	4585	5430	347.5	437.5	587.5	612.5
PC3-G-F1-15	228	1575	2345	2675	4025	5195	312.5	467.5	545.0	652.5
PC3-G-F2-15	755	2695	3320	4340	5315	6140	385.0	522.5	617.5	702.5
PC3-G-F2-30	370	1235	1905	2530	3105	4090	300.0	357.5	502.5	607.5
PC3-L-X	1195	2750	3820	4550	5235	5625	427.5	542.5	637.5	677.5
PC3-L-C1-30	775	2195	3690	4645	5215	5660	362.5	515.0	617.5	720.0
PC3-L-C1-40	328	1955	3420	4835	5540	6265	437.5	517.5	650.0	705.0
PC3-L-C2-30	905	2765	4230	4750	5285	6340	480.0	542.5	670.0	747.5
PC3-L-C2-40	380	1930	3500	4410	4890	5780	377.5	490.0	595.0	750.0
PC3-L-F1-30	970	2775	3605	4210	4735	5795	400.0	480.0	582.5	665.0
PC3-L-F1-40	525	1900	2790	3850	4530	5275	347.5	433.5	565.0	655.0
PC3-L-F2-30	760	2765	3770	4550	4950	6485	432.5	542.5	660.0	720.0
PC3-L-F2-40	655	2275	3285	4100	4835	6050	417.5	517.5	645.0	715.0

Table 4.24. Compressive and flexural strength of mixtures prepared with PC2 cement.

Mixture ID	Compressive Strength (psi)						Flexural Strength (psi)			
	1-day	3-day	7-day	14-day	28-day	90-day	3-day	7-day	28-day	90-day
PC3-G-X	2175	3080	3700	4190	4600	4925	582.5	622.5	665.0	692.5
PC3-G-C1-15	1170	2550	3820	4635	5085	6470	420.0	502.5	590.0	667.5
PC3-G-C2-15	1145	2550	4215	5240	5840	7090	462.5	490.0	617.5	677.5
PC3-G-C2-30	615	1705	2710	3490	3865	4885	320.0	360.0	420.0	497.5
PC3-G-F1-15	1900	2825	3270	3850	4480	5110	470.0	565.0	637.5	687.5
PC3-G-F2-15	1985	2365	2945	3550	4235	5205	457.5	580.0	642.5	687.5
PC3-G-F2-30	1710	2245	2555	3265	4015	5195	362.5	460.0	572.5	640.0
PC3-L-X	2350	3500	3955	4690	5050	5865	537.5	585.0	640.0	675.0
PC3-L-C1-30	1790	2940	3615	4270	4805	5555	505.0	547.5	627.5	670.0
PC3-L-C1-40	1530	2130	3390	3895	4540	5900	430.0	482.5	592.5	727.5
PC3-L-C2-30	2395	3355	3890	4425	5225	6105	485.0	562.5	645.0	772.5
PC3-L-C2-40	1465	2115	3220	3740	4305	5775	452.5	557.5	635.0	742.5
PC3-L-F1-30	2215	3440	3895	4400	4955	6305	525.0	560.0	617.5	690.0
PC3-L-F1-40	1070	2175	2885	3640	4410	5560	390.0	460.0	625.0	720.0
PC3-L-F2-30	2330	3455	4515	5060	5755	6735	490.0	545.0	627.5	722.5
PC3-L-F2-40	1280	2455	3065	4060	4845	6165	425.0	450.0	565.0	705.0

Table 4.25. Compressive and flexural strength of mixtures prepared with PC3 cement.

Mixture ID	Compressive Strength (psi)						Flexural Strength (psi)			
	1-day	3-day	7-day	14-day	28-day	90-day	3-day	7-day	28-day	90-day
PC3-G-X	2015	3275	4325	4565	4705	5495	562.5	620.0	647.5	682.5
PC3-G-C1-15	2535	3305	4425	5175	5755	6410	545.0	620.0	735.0	812.5
PC3-G-C2-15	2195	3320	5055	5685	6170	6360	592.5	657.5	742.5	800.0
PC3-G-C2-30	1345	2960	3755	4565	5210	5695	427.5	507.5	630.0	677.5
PC3-G-F1-15	2005	3310	4300	4355	4720	5875	512.5	565.0	622.5	642.5
PC3-G-F2-15	1805	3135	4065	4480	4885	5810	457.5	525.0	597.5	645.0
PC3-G-F2-30	1250	2390	3315	3915	4715	5695	440.0	510.0	577.5	645.0
PC3-L-X	2115	3350	4520	4715	5190	5505	597.5	627.5	637.5	675.0
PC3-L-C1-30	1345	2960	4185	4530	5290	6095	377.5	465.0	540.0	685.0
PC3-L-C1-40	698	1870	3030	4100	5075	6220	367.5	445.0	577.5	710.0
PC3-L-C2-30	1325	2115	3035	3665	4965	5890	362.5	445.0	537.5	665.0
PC3-L-C2-40	1205	1840	2885	3680	4860	5895	407.5	450.0	562.5	697.5
PC3-L-F1-30	1755	3135	3365	3935	4510	5745	432.5	520.0	572.5	632.5
PC3-L-F1-40	1435	2000	3020	3745	4425	6035	415.0	510.0	567.5	720.0
PC3-L-F2-30	1330	2420	3165	3680	4455	5400	390.0	465.0	542.5	687.5
PC3-L-F2-40	875	1790	2405	2755	3590	4740	355.0	447.5	537.5	662.5



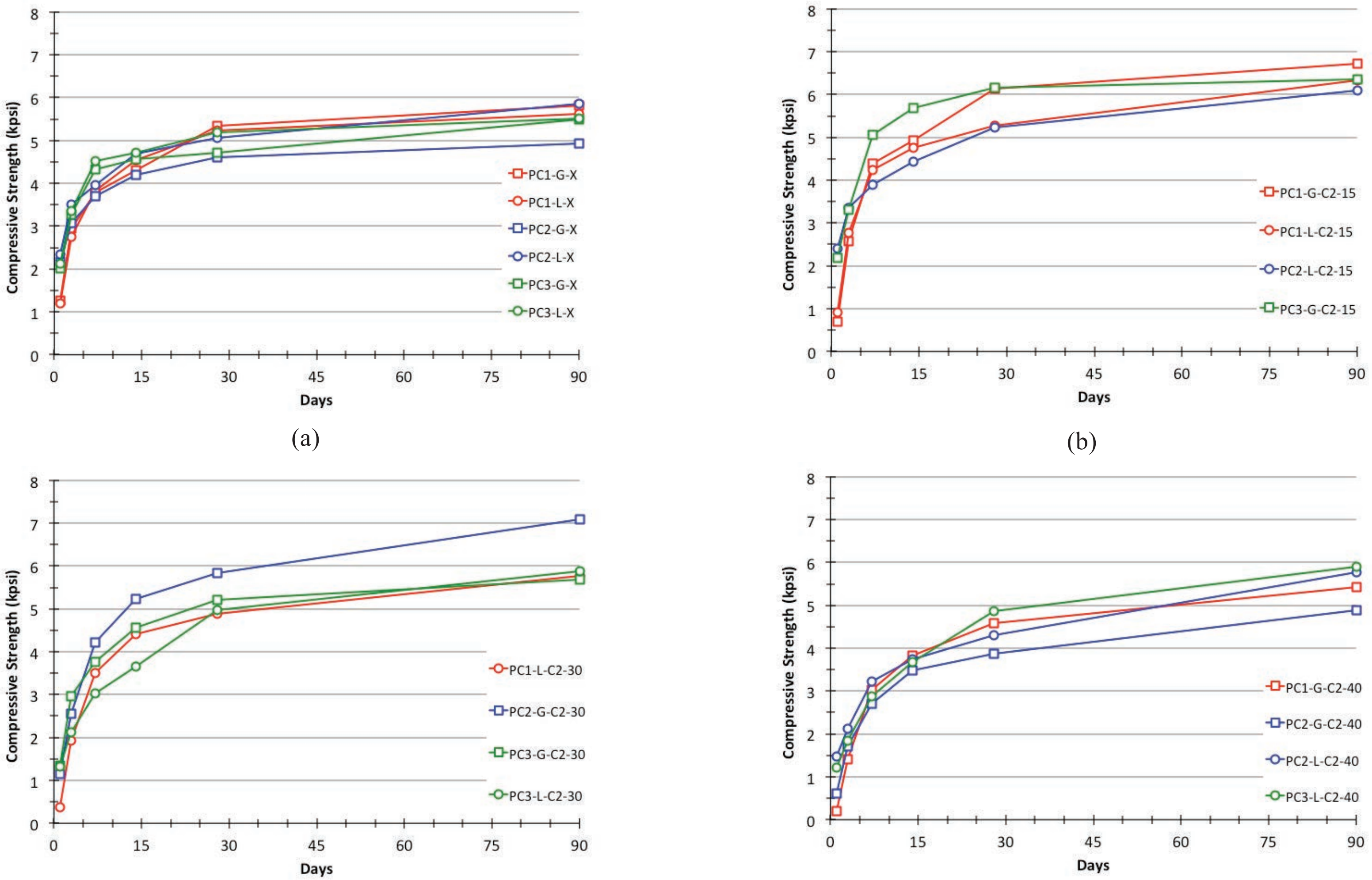
(a)

(b)

(c)

(d)

Figure 4.32. Plots of compressive strength development over time for mixtures: a) all cements without fly ash, b) all C1 ash mixtures replaced at 15%, c) all C1 ash mixtures replaced at 30%, and d) all C1 ash mixtures replaced at 40%



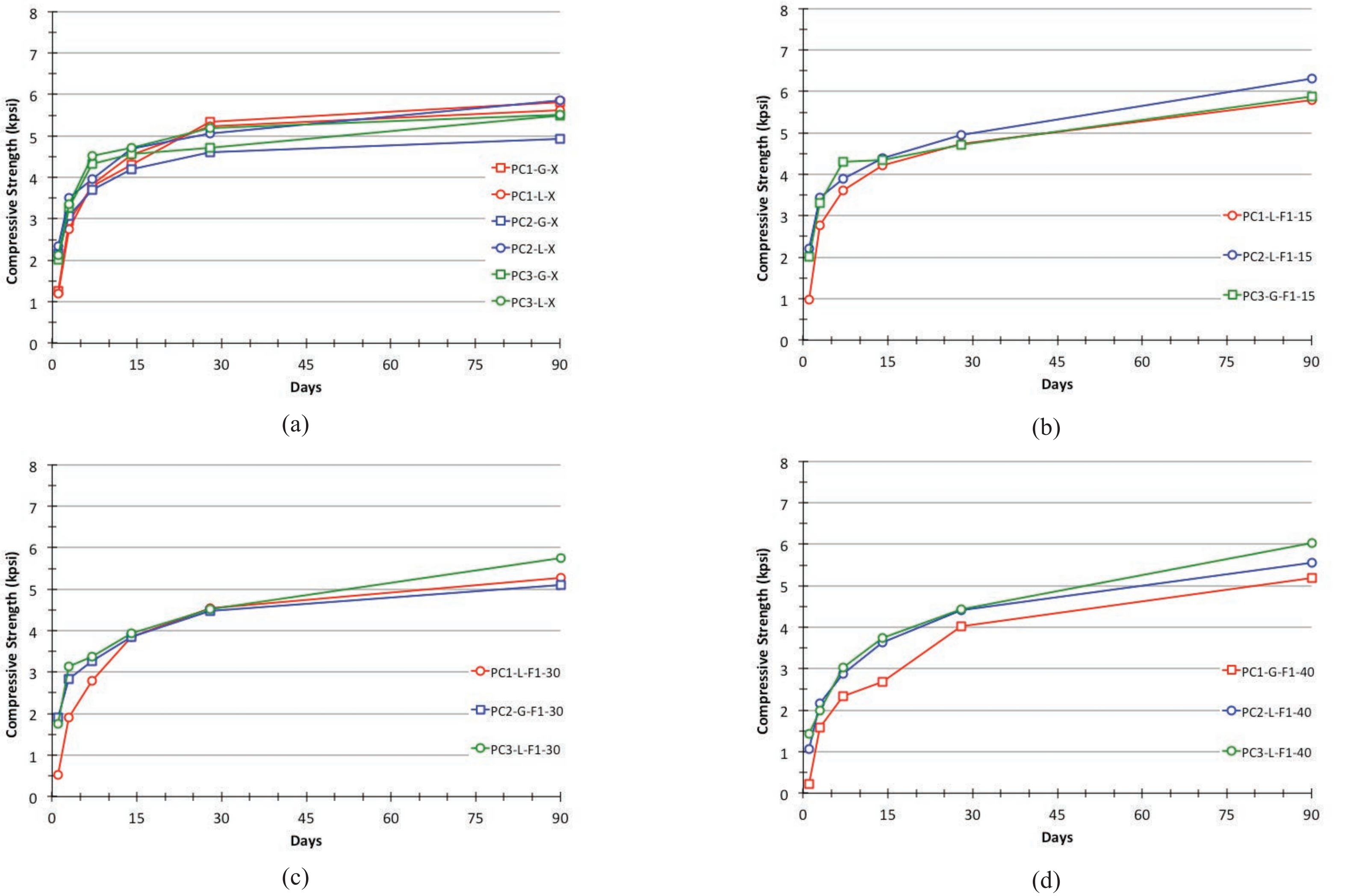
(a)

(b)

(c)

(d)

Figure 4.33. Plots of compressive strength development over time for mixtures: a) all cements without fly ash, b) all C2 ash mixtures replaced at 15%, c) all C2 ash mixtures replaced at 30%, and d) all C2 ash mixtures replaced at 40%



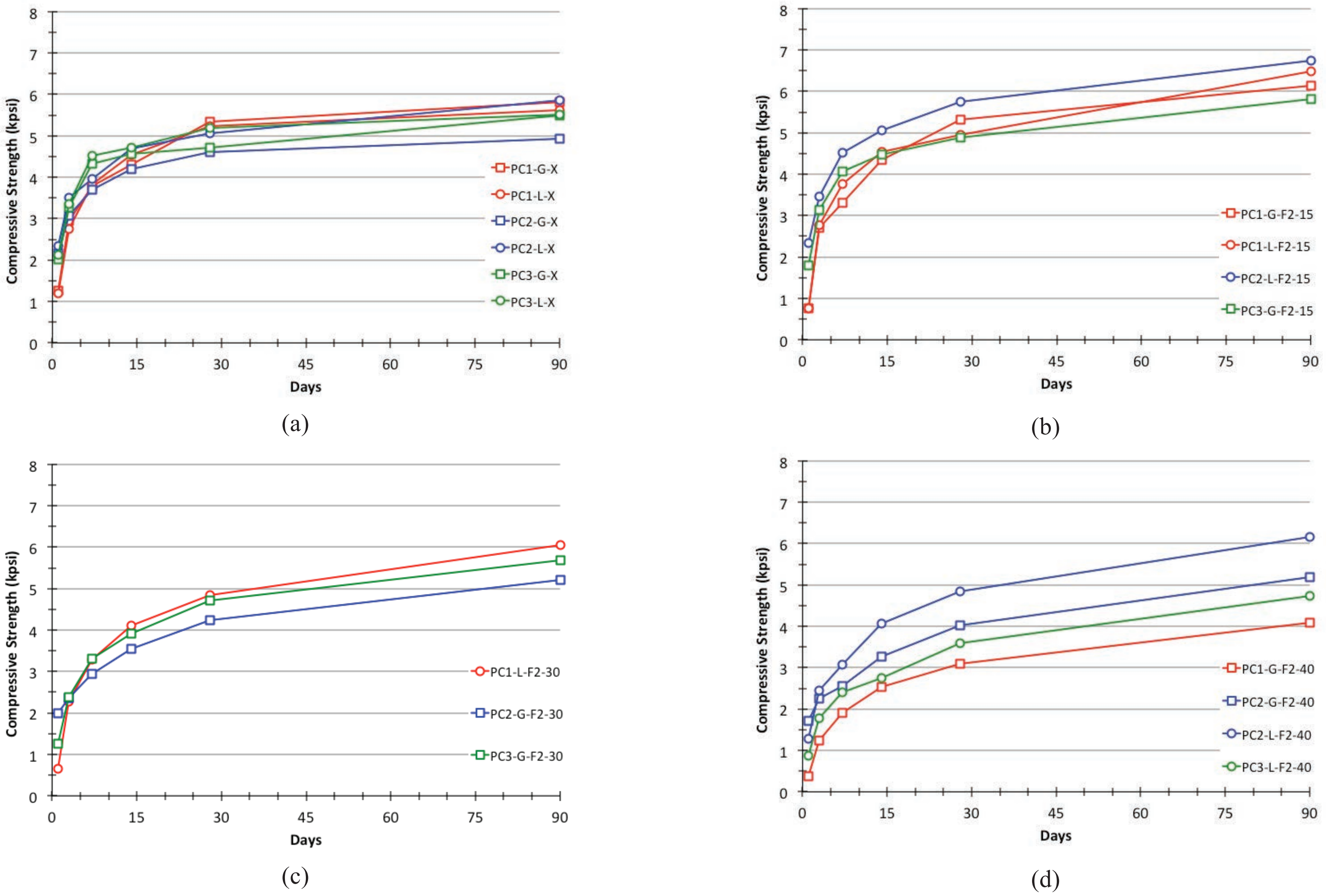
(a)

(b)

(c)

(d)

Figure 4.34. Plots of compressive strength development over time for mixtures: a) all cements without fly ash, b) all F1 ash mixtures replaced at 15%, c) all F1 ash mixtures replaced at 30%, and d) all F1 ash mixtures replaced at 40%



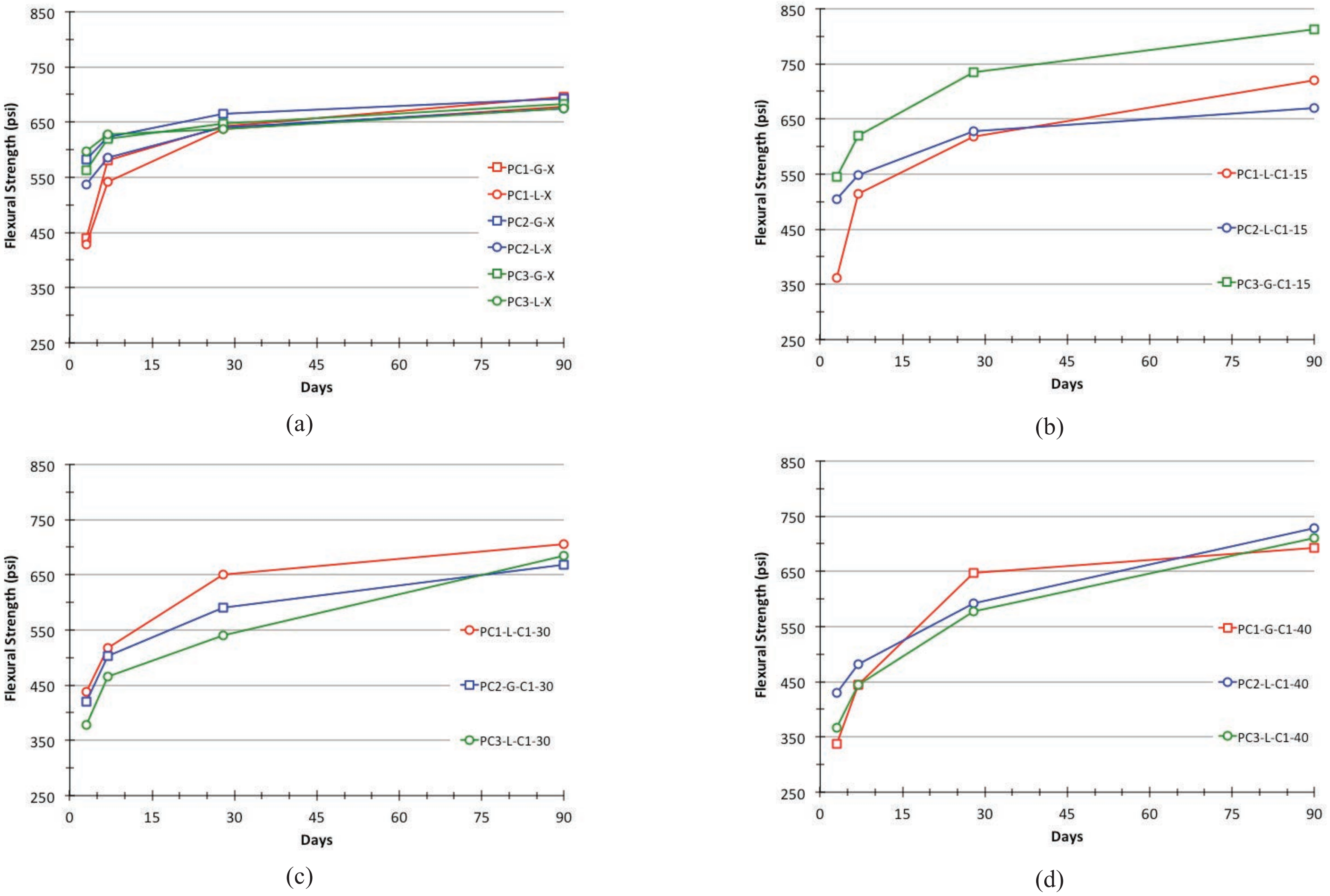
(a)

(b)

(c)

(d)

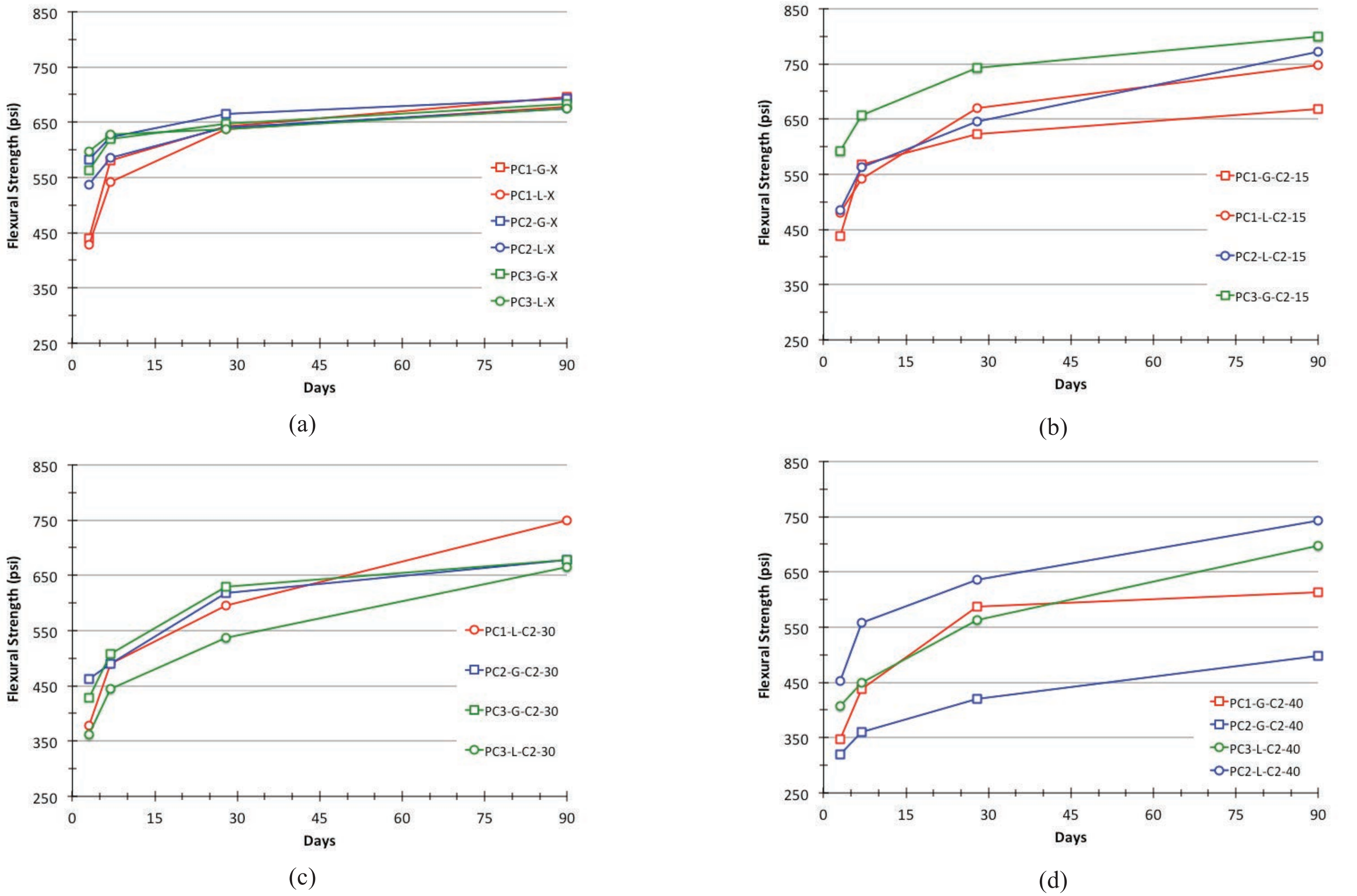
Figure 4.35. Plots of compressive strength development over time for mixtures: a) all cements without fly ash, b) all F2 ash mixtures replaced at 15%, c) all F2 ash mixtures replaced at 30%, and d) all F2 ash mixtures replaced at 40%



(a) (b)

(c) (d)

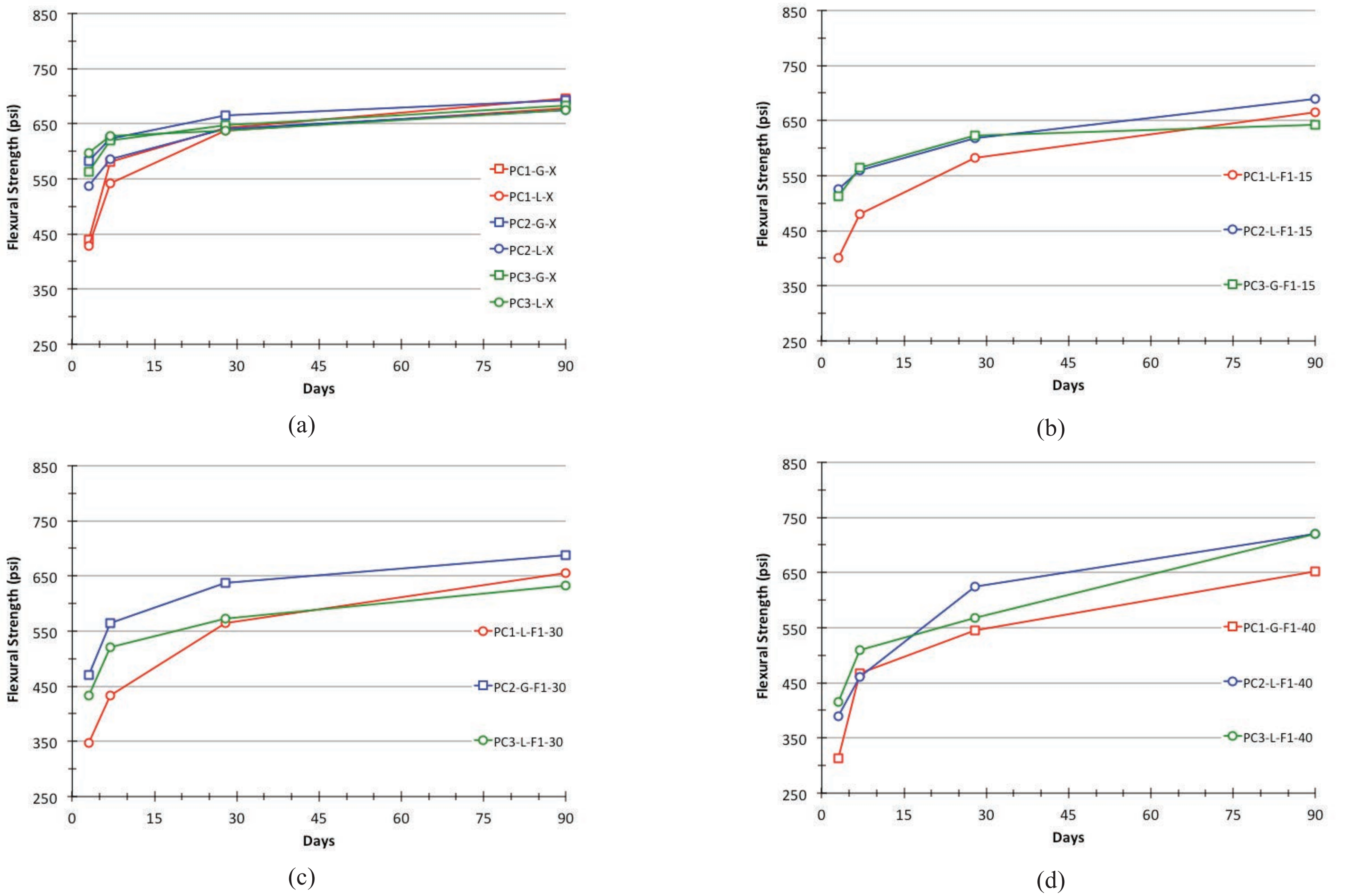
Figure 4.36. Plots of flexural strength development over time for mixtures: a) all cements without fly ash, b) all C1 ash mixtures replaced at 15%, c) all C1 ash mixtures replaced at 30%, and d) all C1 ash mixtures replaced at 40%



(a) (b)

(c) (d)

Figure 4.37. Plots of flexural strength development over time for mixtures: a) all cements without fly ash, b) all C2 ash mixtures replaced at 15%, c) all C2 ash mixtures replaced at 30%, and d) all C2 ash mixtures replaced at 40%



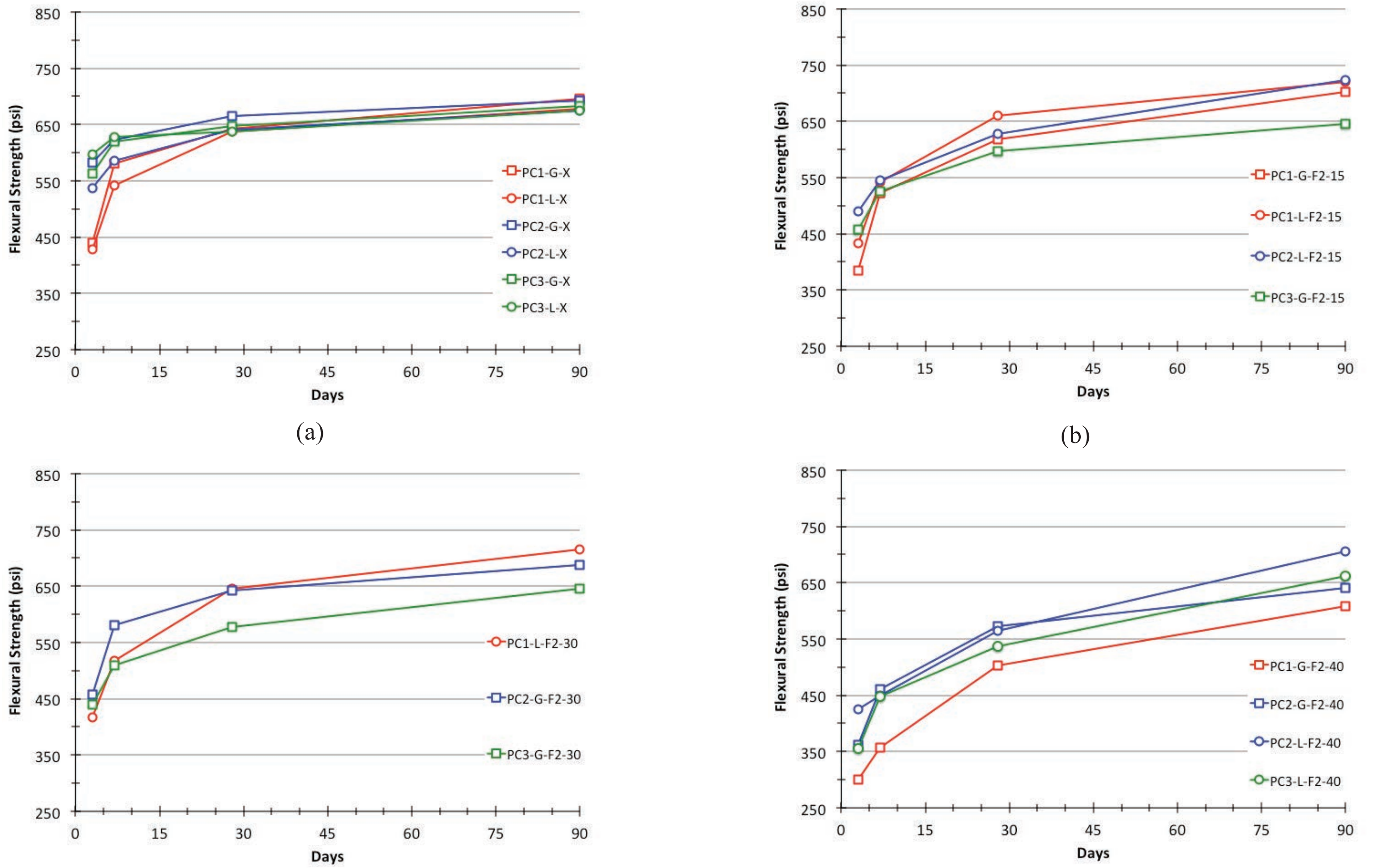
(a)

(b)

(c)

(d)

Figure 4.38. Plots of flexural strength development over time for mixtures: a) all cements without fly ash, b) all F1 ash mixtures replaced at 15%, c) all F1 ash mixtures replaced at 30%, and d) all F1 ash mixtures replaced at 40%



(a)

(b)

(c)

(d)

Figure 4.39. Plots of flexural strength development over time for mixtures: a) all cements without fly ash, b) all F2 ash mixtures replaced at 15%, c) all F2 ash mixtures replaced at 30%, and d) all F2 ash mixtures replaced at 40%

4.6 Maturity Test Results

The results of the maturity tests are summarized in Table 4.27 through Table 4.29. Maturity is one property that was significantly affected by the addition of fly ash to the concrete mixtures. In Table 4.27 through Table 4.29, columns 2-6 are the maturity factors determined by the relationship in equation 4.7.

$$M = \sum_{0}^{t}(T - T_0)\Delta t \tag{4.7}$$

Where:

M = maturity index, ºC-hours (or ºC-days)
T = average concrete temperature, ºC, during the time interval Δt
T_0 = datum temperature (usually taken to be 0 ºC)
t = elapsed time (hours or days)
Δt = time interval (hours or days)

One common approach for using the maturity data is to plot the log maturity vs. compressive strength and then perform a linear regression of the data yielding a best-fit line. Columns 7-9 present the slope, intercept, and coefficient of determination (R^2) for the regression analysis for each mixture. Columns 10-14 provide estimates of the maturity factor at specified strengths calculated using the slope and intercept presented in column 7 and 8. This estimate of maturity can be easily converted to time by dividing the table value by the average curing temperature. Therefore, smaller numbers in columns 10-14 equate to a higher rate of strength gain, or a shorter time to reach the strength specified.

As can be seen by examining the tables, there is wide range of maturity values at any specified strength. The variation is summarized below in Table 4.26. To illustrate, to achieve 700 psi at an average temperature of 18 ºC [64.4 ºF], the range in time would be from 3.3 hrs (mixture PC2-L-X) to 43.0 hrs (PC1-G-F1-40).

Table 4.26. Summary of variation in estimated maturity values at specified strengths.

	Maturity Estimated at Compressive Specified Strength ([0]°C-Hrs)				
	300 psi	*700 psi*	*1000 psi*	*2000 psi*	*3000 psi*
Minimum	36	59	86	302	883.2
Maximum	524	774	1036	3757	13902.9
Average	213	309	410	1081	2988.8
Std. Dev.	146	202	261	688	2235.3
C of V	68.6%	65.3%	63.6%	63.7%	74.8%

Table 4.27. Results of maturity testing for mixtures prepared with PC1 cement.

	Maturity ([0]°C-Hrs)					Linear Regression Log maturity vs. strength			Maturity Estimated at Specified Compressive Strength ([0]°C-Hrs)				
Mixture	1-day	3-day	7-day	14-day	28-day	slope	intercept	R^2	300 psi	700 psi	1000 psi	2000 psi	3000 psi
PC1-G-X	534	1521	3700	7533	15398	2646.4	-5753.9	0.98	194	274	356	849	2027
PC1-G-C1-40	492	1461	3631	7449	15259	2539.3	-6512.5	1.00	481	691	907	2245	5557
PC1-G-C2-15	456	1565	3785	7769	15609	3535.0	-8635.3	0.99	336	436	530	1017	1953
PC1-G-C2-40	442	1549	3770	7757	15566	2940.8	-7676.2	0.99	516	705	892	1950	4265
PC1-G-F1-40	497	1607	3772	7742	15374	2366.8	-6140.0	0.97	524	774	1036	2738	7239
PC1-G-F2-15	510	1627	3797	7759	15391	2971.8	-7161.2	0.99	305	414	522	1126	2429
PC1-G-F2-40	433	1381	3598	7611	15240	1758.4	-4289.3	1.00	406	686	1016	3757	13903
PC1-L-X	447	1408	3639	7668	15329	2616.2	-5610.0	0.99	181	258	335	809	1951
PC1-L-C1-15	476	1573	3887	7821	15417	3057.8	-7425.9	0.99	335	453	568	1206	2560
PC1-L-C1-30	471	1566	3872	7813	15418	3559.3	-9268.3	0.99	487	630	766	1462	2793
PC1-L-C2-15	563	1747	4070	8038	15674	3082.8	-7327.9	0.97	297	400	501	1058	2234
PC1-L-C2-30	543	1686	4002	7943	15521	3234.2	-8397.7	0.99	489	650	805	1640	3343
PC1-L-F1-15	568	1634	3889	7828	15441	2569.6	-5806.3	0.97	238	340	445	1089	2667
PC1-L-F1-30	507	1612	3893	7790	15483	2720.5	-6848.5	1.00	423	594	765	1786	4167
PC1-L-F2-15	480	1617	3898	7792	15551	2800.4	-6478.4	0.97	263	365	467	1064	2422
PC1-L-F2-30	492	1636	3966	7913	15747	2771.5	-6725.6	1.00	341	476	610	1402	3219

Table 4.28. Results of maturity testing for mixtures prepared with PC2 cement.

	Maturity ([0]°C-Hrs)					Linear Regression Log maturity vs. strength			Maturity Estimated at Specified Compressive Strength ([0]°C-Hrs)				
Mixture	1-day	3-day	7-day	14-day	28-day	slope	intercept	R^2	300 psi	700 psi	1000 psi	2000 psi	3000 psi
PC2-G-X	542	1599	3839	7773	15604	1663.3	-2307.9	0.996	37	64	97	387	1549
PC2-G-C1-30	512	1640	3911	7688	15558	2745.8	-6210.0	0.989	192	272	354	847	2031
PC2-G-C2-30	516	1647	3921	7721	15643	3329.3	-7924.2	0.988	251	335	415	850	1741
PC2-G-C2-40	483	1563	3806	7562	15620	2244.5	-5392.6	0.991	342	516	702	1959	5470
PC2-G-F1-30	562	1669	3945	7870	15721	1730.0	-2845.7	0.993	66	112	167	631	2389
PC2-G-F2-30	517	1552	3782	7626	15604	1527.1	-2346.3	0.962	54	98	154	697	3151
PC2-G-F2-40	518	1548	3753	7599	15531	1513.0	-2552.7	0.948	77	141	223	1017	4646
PC2-L-X	538	1593	3833	7770	15604	1836.2	-2554.5	0.985	36	59	86	302	1058
PC2-L-C1-15	504	1566	3789	7509	15454	2022.9	-3612.5	0.997	96	148	204	595	1738
PC2-L-C1-40	481	1590	3806	7572	15578	2086.9	-4221.9	0.975	147	228	318	958	2885
PC2-L-C2-15	511	1608	3818	7558	15464	1843.8	-2618.9	0.991	38	63	91	318	1110
PC2-L-C2-40	443	1518	3636	7438	15496	1920.0	-3731.8	0.982	125	202	290	962	3196
PC2-L-F1-15	571	1675	3945	7840	15641	1833.3	-2696.2	0.983	43	71	103	363	1275
PC2-L-F1-40	503	1612	3794	7641	15502	2220.4	-4962.6	0.998	223	339	464	1324	3775
PC2-L-F2-15	532	1468	3687	7567	15322	2340.5	-3979.7	0.996	67	100	134	358	958
PC2-L-F2-40	524	1423	3625	7484	15242	2384.0	-5197.9	0.990	202	297	397	1043	2738

Table 4.29. Results of maturity testing for mixtures prepared with PC3 cement.

	Maturity ([0]°C-Hrs)					Linear Regression Log maturity vs. strength			Maturity Estimated at Specified Compressive Strength ([0]°C-Hrs)				
Mixture	1-day	3-day	7-day	14-day	28-day	slope	intercept	R^2	300 psi	700 psi	1000 psi	2000 psi	3000 psi
PC3-G-X	523	1620	3732	7678	15545	1891.4	-2871.8	0.923	47	77	111	374	1264
PC3-G-C1-15	484	1568	3693	7509	15607	2232.5	-3582.1	0.987	55	82	112	315	883
PC3-G-C2-15	499	1515	3609	7514	15719	2812.1	-5361.9	0.970	103	143	182	413	938
PC3-G-C2-30	467	1455	3497	7330	15324	2521.4	-5220.9	0.990	154	222	292	728	1816
PC3-G-F1-15	585	1717	3836	7537	15008	1923.2	-3046.8	0.923	55	88	126	419	1389
PC3-G-F2-15	588	1700	3998	7853	15777	2167.1	-3995.1	0.972	104	159	217	618	1757
PC3-G-F2-30	574	1685	3995	7856	15797	2385.5	-5318.5	0.999	227	334	446	1168	3062
PC3-L-X	511	1595	3695	7601	15379	2104.2	-3406.4	0.959	57	89	124	370	1105
PC3-L-C1-30	499	1474	3739	7719	15835	2578.5	-5376.5	0.974	158	226	296	723	1768
PC3-L-C1-40	500	1474	3735	7703	15789	2939.1	-7346.9	0.996	400	547	691	1513	3311
PC3-L-C2-30	413	1471	3672	7764	15786	2221.3	-4725.3	0.960	182	276	376	1060	2990
PC3-L-C2-40	452	1449	3491	7321	15317	2387.8	-5419.7	0.966	247	364	486	1277	3353
PC3-L-F1-30	567	1707	3927	7795	15578	1804.7	-3035.8	0.963	70	116	171	612	2198
PC3-L-F1-40	502	1617	3797	7632	15510	2083.1	-4391.7	0.977	178	278	387	1167	3523
PC3-L-F2-30	500	1656	3885	7701	15583	2053.6	-4213.2	0.998	158	247	346	1060	3247
PC3-L-F2-40	499	1640	3862	7670	15556	1743.0	-3844.2	0.989	237	402	598	2244	8419

4.7 Data Analysis – Statistical Analysis

4.7.1 Description of Variables

To simplify presentation of the results, a reference code was assigned to each factor and response variable. Table 4.30 provides the reference codes assigned to the factors while Table 4.31 shows the codes assigned to the responses.

4.7.2 Comparison of Mixtures With and Without Fly Ash

For each response, the sample means were calculated for the mixtures without ash content (n= 6) and mixtures with 15%, 30%, and 40% ash content, respectively (n=14 for each replacement level). These results are shown in Table 4.32.

The differences between mixtures with 0% ash and those with 15%, 30%, and 40% ash, respectively, are given in Table 4.33. For example, the sample means of PA (i.e., pressure meter air content) from using 0% and 15% ash are 6.75 and 6.279 (see Table 4.32) and the difference is given in Table 4.33 as 0.4714, obtained by subtracting 6.279 from 6.75.

When comparing mixtures with fly ash to those without fly ash, the results in Table 4.33 show the values of all 23 dependent variables are different when using fly ash. The next step is to determine whether those differences are caused by random error, or if the differences are statistically significant. Two-independent sample *t*-tests were performed using a level of significance alpha = 0.05 and the results are presented in Table 4.33. In Table 4.33, the *p*-values of the *t*-test are given. A *p*-value smaller than 0.05 indicates the difference is a true difference and it is not simply observed by chance.

Based on these results, when comparing the response variables from mixtures with fly ash to mixtures without fly ash, the response variables listed in Table 4.34 are not affected by the four factors at a statistically significant level.

Conversely, the response variables listed in Table 4.35 show a statistically significant difference when comparing mixtures with no fly ash to mixtures with fly ash. The affect on the response variable is not consistent with respect to ash content. That is, increasing ash content does not always correlate to a decrease (or increase) in any given response variable.

Table 4.30. Reference codes assigned to the independent variables (factors).

Independent Variable (factor)	Reference Code
Cement Type	CT
Aggregate Type	AT
Fly Ash Type	FT
Fly Ash Content	AC

Table 4.31. Reference codes assigned to the dependent variables (response variables).

Dependent Variable (response variable)	Reference Code
Pressure Meter Air Content	PA
Gravimetric Air Content	GA
Durability Factor	RD
Maturity Factor 1-day	MF1
Maturity Factor 3-day	MF3
Maturity Factor 7-day	MF7
Maturity Factor 14-day	MF14
Maturity Factor 28-day	MF28
Maturity Factor @ 300 psi	MS300
Maturity Factor @ 700 psi	MS700
Maturity Factor @ 1000 psi	MS1000
Maturity Factor @ 2000 psi	MS2000
Maturity Factor @ 3000 psi	MS3000
Compressive Strength 1-day	CS1
Compressive Strength 3-day	CS3
Compressive Strength 7-day	CS7
Compressive Strength 14-day	CS14
Compressive Strength 28-Day	CS28
Compressive Strength 90-Day	CS90
Flexural Strength 3-day	FS3
Flexural Strength 7-day	FS7
Flexural Strength 28-Day	FS28
Flexural Strength 90-Day	FS90

Table 4.32. Sample means for response variables from mixtures with 0, 15, 30, and 40 weight % fly ash.

Response Variable	Fly Ash Content (weight %)			
	0	15	30	40
PA	6.750	6.279	6.386	6.364
GA	6.182	6.051	5.994	6.015
RD	89.500	91.571	88.929	89.643
MF1	515.800	523.400	510.000	483.500
MF3	1556.000	1612.900	1604.000	1530.900
MF7	3739.700	3835.800	3857.600	3719.700
MF14	7670.500	7706.700	7752.100	7584.400
MF28	15476.500	15505.400	15614.100	15470.000
MS300	91.980	166.700	232.200	293.300
MS700	136.800	233.000	327.700	440.700
MS1000	184.800	300.200	425.900	600.400
MS2000	515.200	711.400	1047.700	1725.400
MS3000	1492.300	1736.700	2708.200	5162.900
CS1	1853.300	1580.700	1174.100	921.900
CS3	3150.000	3002.900	2452.100	1868.200
CS7	4019.200	4055.400	3433.200	2792.500
CS14	4503.300	4661.800	4197.100	3478.600
CS28	5019.200	5279.300	4898.600	4262.900
CS90	5538.300	6163.600	5840.400	5408.600
FS3	524.600	472.100	415.700	373.000
FS7	596.300	553.800	500.600	452.100
FS28	645.000	641.800	595.900	567.000
FS90	682.900	712.700	681.300	670.500

Table 4.33. The differences between sample means and the *p*-values for those difference, for response variables from mixtures with 0% fly ash and mixtures with 15%, 30%, and 40% fly ash. An asterisk (*) by a *p*-value indicates it is significant at the significance level of alpha = 0.05%.

Response Variable	Differences Between Sample Means			*p*-values Differences Between Sample Means		
	0%-15%	0%-30%	0%-40%	0%-15%	0%-30%	0%-40%
PA	0.471	0.364	0.386	0.1748	0.4355	0.3314
GA	0.131	0.187	0.167	0.1101	0.0344 *	0.1044
RD	-2.071	0.571	-0.143	0.7572	0.9358	0.9849
MF1	-7.524	5.833	32.333	0.7176	0.7762	0.0480 *
MF3	-56.857	-48.000	25.143	0.1562	0.2585	0.5324
MF7	-96.119	-118.000	19.952	0.1020	0.0727	0.6846
MF14	-36.214	-81.571	86.143	0.6331	0.2404	0.1488
MF28	-28.857	-137.600	6.500	0.7444	0.0648	0.9312
MS300	-74.731	-140.200	-201.300	0.1779	0.0432 *	0.0063 *
MS700	-96.193	-190.900	-303.900	0.1847	0.0331 *	0.0037 *
MS1000	-115.400	-241.100	-415.600	0.1916	0.0257 *	0.0026 *
MS2000	-196.200	-532.500	-1210.200	0.2379	0.0067 *	0.0025 *
MS3000	-244.400	-1215.900	-3670.600	0.4112	0.0014 *	0.0092 *
CS1	272.600	679.200	931.500	0.4137	0.0156 *	0.0019 *
CS3	147.100	697.900	1281.800	0.4099	0.0012 *	< 0.0001 *
CS7	-36.191	586.000	1226.700	0.8645	0.0080 *	< 0.0001 *
CS14	-158.500	306.200	1024.800	0.3941	0.1757	0.0002 *
CS28	-260.100	120.600	756.300	0.2518	0.5635	0.0047 *
CS90	-625.200	-302.000	129.800	0.0026 *	0.2228	0.6500
FS3	52.441	108.900	151.500	0.1255	0.0003 *	< 0.0001 *
FS7	42.500	95.643	144.100	0.0476 *	< 0.0001 *	< 0.0001 *
FS28	3.214	49.107	78.036	0.8713	0.0110 *	0.0044 *
FS90	-29.762	1.667	12.381	0.2079	0.8954	0.6554

Table 4.34. Response variables not showing a statistically significant difference when comparing mixtures with no fly ash to mixtures with fly ash.

Dependent Variable (response variable)	Reference Code
Pressure Meter Air Content	PA
Durability Factor	RD
Maturity Factor 3-day	MF3
Maturity Factor 7-day	MF7
Maturity Factor 14-day	MF14
Maturity Factor 28-day	MF28
Flexural Strength 90-Day	FS90

In Table 4.33, the *p*-values of the durability factor (AASHTO T 161) are the largest, indicating it is the least affected by the addition of ash, regardless of the amount being added. The smaller the *p*-value in Table 4.33, the stronger is the evidence to support the difference between mixtures with and without ash. The smallest *p*-values (< 0.0001) in Table 4.33 are obtained for the compressive and flexural strengths measured at 3 and 7 days, respectively, and mainly when comparing mixtures with no ash and mixtures with 40% ash. This indicates adding ash at a 40% level affects these response variables the most.

For many variables, no difference is observed when a low ash replacement (i.e., 15%) is compared with no ash mixtures but significant differences are observed when the level of ash content is increased to 30% or 40%. The trends observed for flexural strength should be noted. For flexural strengths at 3, 7, and 28 days, all *p*-values decrease when the level of ash content is increased, indicating the effect of increasing the ash content ash becomes more significant for those responses. However, the opposite trend is observed for the flexural strength at 90 days.

Table 4.35. Response variables showing a statistically significant difference when comparing mixtures with no fly ash to mixtures with fly ash. An x indicates the difference observed was statistically significant for the fly ash replacement level indicated.

Dependent Variable (response variable)	Fly Ash Content (weight %)		
	15	30	40
Gravimetric Air Content		X	
Maturity Factor 1-day			X
Maturity Factor @ 300 psi		X	X
Maturity Factor @ 700 psi		X	X
Maturity Factor @ 1000 psi		X	X
Maturity Factor @ 2000 psi		X	X
Maturity Factor @ 3000 psi		X	X
Compressive Strength 1-day		X	X
Compressive Strength 3-day		X	X
Compressive Strength 7-day		X	X
Compressive Strength 14-day			X
Compressive Strength 28-Day			X
Compressive Strength 90-Day	X		
Flexural Strength 3-day		X	X
Flexural Strength 7-day	X	X	X
Flexural Strength 28-Day		X	X

4.8 Factors Significantly Affecting Each Response Variables for Fly Ash Mixtures

For each of the 23 independent variables, an F-test was performed to determine if the main-effect model analyzing all four factors was useful. The *p*-values of that test are given in the column of Table 4.36 labeled *Overall F* (column 3). A *p*-value less than 0.05 indicates at least one of the four factors has a significant impact on the dependent variable. Then an F test was performed for each factor to determine the importance of each factor. The *p*-value for each factor and response combination is listed in the last four columns of Table 4.36.

Table 4.36. *p*-values of F tests of main-effect models with all four factors (Overall F). An asterisk by a value in the Overall F column indicates at least one of the four factors has a significant impact on the dependent variable. The last four columns list F test results for each factor.

Response Variable	R^2	Overall F	*p*-values			
			Cement Type	Aggregate Type	Fly Ash Type	Fly Ash Content
PA	0.331	0.0713	0.957	0.148	0.008	0.929
GA	0.374	0.0323*	0.404	0.210	0.005	0.793
RD	0.396	0.0207*	0.663	0.227	0.002	0.862
MF1	0.496	0.0019*	0.175	0.549	0.002	0.006
MF3	0.360	0.0425*	0.903	0.883	0.040	0.018
MF7	0.371	0.0342*	0.241	0.332	0.234	0.009
MF14	0.487	0.0024*	0.003	0.232	0.284	0.007
MF28	0.207	0.404	0.290	0.741	0.688	0.125
MS300	0.858	< 0.0001*	< 0.0001	0.640	0.050	< 0.0001
MS700	0.867	< 0.0001*	< 0.0001	0.454	0.204	< 0.0001
MS1000	0.866	< 0.0001*	< 0.0001	0.335	0.538	< 0.0001
MS2000	0.789	< 0.0001*	< 0.0001	0.173	0.553	< 0.0001
MS3000	0.644	< 0.0001*	0.020	0.166	0.098	< 0.0001
CS1	0.847	< 0.0001*	< 0.0001	0.937	0.397	< 0.0001
CS3	0.873	< 0.0001*	< 0.0001	0.825	0.316	< 0.0001
CS7	0.770	< 0.0001*	0.016	0.728	0.009	< 0.0001
CS14	0.691	< 0.0001*	0.387	0.865	0.004	< 0.0001
CS28	0.623	< 0.0001*	0.621	0.745	0.005	< 0.0001
CS90	0.416	0.014	0.674	0.145	0.263	0.001
FS3	0.664	< 0.0001*	0.001	0.870	0.702	< 0.0001
FS7	0.556	0.0003*	0.383	0.492	0.863	< 0.0001
FS28	0.350	0.051	0.831	0.631	0.557	0.002
FS90	0.299	0.136	0.950	0.027	0.500	0.073

The R^2 values in Table 4.36 indicate the percent of the variation of a dependent variable that can be explained by varying the levels of the factors tested. For example an $R^2 = 0.873$ for CS3 in the above table means that approximately 87.3% of the variation in CS3 can be explained by using the four factors tested. The largest R^2 values are obtained from models with maturity factors as the dependent variables (MS300, MS700, MS1000, MS2000, and MS3000). For most of those models, cement type and ash content are important factors. This means that using different cement types and ash replacement levels will have a very strong impact on maturity measures. The R^2 values for compressive strength measures are also high (i.e., CS1, CS3, CS7, CS14, CS28, and CS90) and ash content is again an important factor for those measures at all ages and cement type is a factor at early ages. Ash content is an important factor for most of the strength-related models, which means that using different levels of ash content will have a significant impact on most of the strength-related dependent variables. The response variables that cannot be explained by the variation of the four factors tested include pressure meter air content, maturity factor at 28 days, and the flexural strength at 28 and 90 days.

Based on this analysis, the factors affecting each response variable can be identified. The factors listed in Table 4.37 have a *p*-value less than 0.01 indicating a strong relationship with the corresponding dependent variable. The strength of the association can be seen from Table 4.36 with a smaller *p*-value indicating a stronger association. Notice there are no models with three important factors. This means there are no three-factor interactions for any of the response variables. Many models have two important independent variables so two factor interactions are possible for those models, which will be considered later.

Table 4.37. Factors with *p*-value less than 0.01 in Table 4.36 indicating a strong affect on the listed response variables.

Response Variable	Factors Affecting the Response Variable (alpha = 0.01)
Pressure Meter Air Content	Fly Ash Type
Gravimetric Air Content	Fly Ash Type
Durability Factor	Fly Ash Type
Maturity Factor 1-day	Fly Ash Type, Ash Content
Maturity Factor 3-day	none
Maturity Factor 7-day	Ash Content
Maturity Factor 14-day	Cement Type, Ash Content
Maturity Factor 28-day	none
Maturity Factor @ 300 psi	Cement Type, Ash Content
Maturity Factor @ 700 psi	Cement Type, Ash Content
Maturity Factor @ 1000 psi	Cement Type, Ash Content
Maturity Factor @ 2000 psi	Cement Type, Ash Content
Maturity Factor @ 3000 psi	Ash Content
Compressive Strength 1-day	Cement Type, Ash Content
Compressive Strength 3-day	Cement Type, Ash Content
Compressive Strength 7-day	Fly Ash Type, Ash Content
Compressive Strength 14-day	Fly Ash Type, Ash Content
Compressive Strength 28-Day	Fly Ash Type, Ash Content
Compressive Strength 90-Day	Ash Content
Flexural Strength 3-day	Cement Type, Ash Content
Flexural Strength 7-day	Ash Content
Flexural Strength 28-Day	Ash Content
Flexural Strength 90-Day	none

4.9 The Affect of Factor Levels on the Response Variables

In Table 4.37, ash content appeared 17 times, cement type appeared 8 times, fly ash type appeared 7 times and aggregate type did not appear. The number of occurrences indicates the importance of each factor (e.g., aggregate type is least important).

The next step is to compare the levels for each factor. For example, the *p*-value of the F-test used to examine whether MF14 and CT are related is less than 0.01 in Table 4.36 indicating a strong affect of cement type on the maturity factor at 14 days. The next step is to determine which levels of CT make a difference (e.g., is the difference in the means of MF14 obtained using CT=PC1 vs. CT=PC3 significant, and so on for each combination of cement types). The average values of MF14 are 7787.5, 7619.64, and 7636 using CT = 1, 2, and 3, respectively (these values are given in the following tables). There are two ways to test whether two treatment means are statistically significantly different using Tukey's Honest Significant Difference (HSD) test. One approach is to find the critical value and compare the difference with the critical value. If the difference is large enough to exceed the critical value, it is concluded the difference is statistically significant. The other approach is to use a simultaneous 95% confidence interval. If zero is determined to be in the confidence interval, the difference is considered to be zero with a 95% confidence level and the treatment means are considered equal. On the other hand, if zero is not in the confidence interval, the difference is considered significant. It is more convenient to use the first approach when a factor has less than four levels and the other approach is often used when the factor has four or more levels. Since CT and AC all have less than four levels, critical values using Tukey's HSD were used. The results are given in sections 4.9.1 and 4.9.3. On the other hand, fly ash type has four levels so the simultaneous confidence interval approach based on the HSD test was used. The results are given in section 4.9.2.

4.9.1 Cement Type

For each response variable in Table 4.37 where cement type (CT) was a significant factor, the HSD critical value is provided in Table 4.38 for each response variable and factor combination (columns 2-4) and summarized in Table 4.38 (columns 5-7). As an example, in the case of the MF14 values obtained using different levels of CT (i.e., cement types), using Tukey's HSD test with alpha = 0.05, the critical value is 120.48. This means if the difference between two means exceeds 120.48, the two means are considered to be statistically different. Since the difference between using CT = PC1 and CT = PC3 exceeds 120.48, the conclusion is that MF14 values are different for CT = PC1 versus CT = PC3. The same result can be obtained for CT = PC1 versus CT = PC2. However, the difference in means of MF14 using CT = PC2 and CT = PC3 is less than 120.48 indicating no statistically significant difference in MF14 with those two cements.

Table 4.38. Means of response variables where using different levels (i.e., types) of cement type was significant (columns 2-4) and the interpretation of those means using Tukey's HSD test (columns 5-7).

Response Variable	Cement Type			Comparison of Cement Types		
	PC1	PC2	PC3	PC1 vs. PC 2	PC 1 vs. PC 3	PC 2 vs. PC 3
MF14	7787.50*	7619.64	7636.00	Different	Different	Same
MS300	388.58*	137.34	166.29	Different	Different	Same
MS700	543.89*	208.69	248.83	Different	Different	Same
MS1000	702.09*	286.81	337.64	Different	Different	Same
MS2000	1681.5*	851.6	951.3	Different	Different	Same
CS1	555.1	1614.3*	1507.4	Different	Different	Same
CS3	2119.29	2593.21	2610.71*	Different	Different	Same
FS3	383.93	442.50*	434.4	Different	Different	Same

* highest value in each row.

It is interesting to note that using cement PC1 gives a statistical significantly different result for all of the affected dependent variables, while the other two types of cements give the same results for all affected dependent variables. Cement type PC2 has an alkali content different than PC1 or PC3, which indicates alkali content was not an issue with the affected dependent variables. As previously shown in the strength and maturity tests, the main difference between the cement types was the rate of strength gain, which was lower for PC1 when compared to PC2 and PC3.

4.9.2 Fly Ash Type

There are four levels for fly ash type (FT) and FT is found to be an important factor for seven dependent variables. The comparisons between each pair of the four levels are given in the following seven tables with each table representing a different dependent variable. The differences between the two response variable means are given in the second column and the simultaneous 95% confidence intervals are given in the third and forth columns. Examine the first row in the Table 4.39 as an example. The difference between the means of pressure meter air content (PA) using fly ash C1 and C2 is 0.325 and at a 95% confidence level the true difference is between -0.597 and 1.247. Since 0 is in the confidence interval, it is likely the difference is 0. Therefore, the difference is considered to be 0 and this means using fly ash type C1 and C2 give the same value of pressure meter air content. Examining the third row of Table 4.39, the difference between the means of PA using fly ash type C1 and F2 is 1.167 and at a 95% confidence level the true difference is between 0.244 and 2.089. Since 0 is not in the confidence interval, the difference is not 0. Therefore, using fly ash type C1 and F2 affects the values of pressure meter air content. The results for each response variable identified in Table 4.37 are provided in Table 4.39 through Table 4.45. These results are then summarized in Table 4.46.

Table 4.39. The difference of the means of pressure meter air content from comparing the possible combinations of fly ash type.

Fly Ash Type Comparison	Difference Between Means	95% Confidence Limits		
C1-C2	0.325	-0.597	1.247	
C1-F1	0.144	-0.842	1.131	
C1-F2	1.167	0.244	2.089	***
C2-F1	-0.181	-1.103	0.742	
C2-F2	0.842	-0.012	1.696	
F1-F2	1.022	0.100	1.945	***

*** Indicates the two means are statistically different.

Table 4.40. The difference of the means of gravimetric air content from comparing the possible combinations of fly ash type.

Fly Ash Type Comparison	Difference Between Means	95% Confidence Limits		
C1-C2	0.124	-0.083	0.331	
C1-F1	0.162	-0.059	0.383	
C1-F2	0.287	0.081	0.494	***
C2-F1	0.038	-0.168	0.245	
C2-F2	0.163	-0.028	0.355	
F1-F2	0.125	-0.082	0.332	

*** Indicates the two means are statistically different.

Table 4.41. The difference of the means of durability factor (AASHTO T 161) from comparing the possible combinations of fly ash type.

Fly Ash Type Comparison	Difference Between Means	95% Confidence Limits		
C1-C2	-6.583	-22.339	9.172	
C1-F1	-3.556	-20.399	13.288	
C1-F2	14.917	-0.839	30.672	
C2-F1	3.028	-12.728	18.783	
C2-F2	21.500	6.913	36.087	***
F1-F2	18.472	2.717	34.228	***

*** Indicates the two means are statistically different.

Table 4.42. The difference of the means of maturity factor at 1-day from comparing the possible combinations of fly ash type.

Fly Ash Type Comparison	Difference Between Means	95% Confidence Limits		
C1-C2	8.670	-31.090	48.430	
C1-F1	-49.220	-91.730	-6.720	***
C1-F2	-22.920	-62.680	16.840	
C2-F1	-57.890	-97.650	-18.130	***
C2-F2	-31.580	-68.390	5.230	
F1-F2	26.310	-13.450	66.060	

*** Indicates the two means are statistically different.

Table 4.43. The difference of the means of compressive strength at 7-days from comparing the possible combinations of fly ash type.

Fly Ash Type Comparison	Difference Between Means	95% Confidence Limits		
C1-C2	-79.90	-505.70	346.00	
C1-F1	305.60	-149.70	760.80	
C1-F2	388.10	-37.80	813.90	
C2-F1	385.40	-40.40	811.30	
C2-F2	467.90	73.70	862.20	***
F1-F2	82.50	-343.40	508.40	

*** Indicates the two means are statistically different.

Table 4.44. The difference of the means of compressive strength at 14-days from comparing the possible combinations of fly ash type.

Fly Ash Type Comparison	Difference Between Means	95% Confidence Limits		
C1-C2	8.90	-493.00	510.80	
C1-F1	524.40	-12.10	1061.00	
C1-F2	518.50	16.60	1020.40	***
C2-F1	515.60	13.60	1017.50	***
C2-F2	509.60	44.90	974.30	***
F1-F2	-6.00	-507.90	495.90	

*** Indicates the two means are statistically different.

Table 4.45. The difference of the means of compressive strength at 28-days from comparing the possible combinations of fly ash type.

Fly Ash Type Comparison	Difference Between Means	95% Confidence Limits		
C1-C2	-73.90	-599.50	451.70	
C1-F1	505.60	-56.40	1067.50	
C1-F2	479.40	-46.20	1005.10	
C2-F1	579.40	53.80	1105.10	***
C2-F2	553.30	66.70	1040.00	***
F1-F2	-26.10	-551.70	499.50	

*** Indicates the two means are statistically different.

Table 4.46. Summary of the results from Table 4.39 through Table 4.45

Response Variable	Ash Combinations With Differences in Means
Pressure Meter Air Content	C1 vs. F2, F1 vs. F2
Gravimetric Air Content	C1 vs. F2
Durability Factor	C2 vs. F2, F1 vs. F2
Maturity Factor 1-day	C1 vs. F1, C2 vs. F1
Compressive Strength 7-day	C2 vs. F2
Compressive Strength 14-day	C1 vs. F2, C2 vs. F1, C2 vs. F2
Compressive Strength 28-Day	C2 vs. F1, C2 vs. F2

This analysis indicates that ash type is a factor in early-age maturity, compressive strength, and freeze-thaw durability. The results of the durability factor and gravimetric air statistical analyses were interesting because durability factor differences were significant for C2 and F1 mixtures when compared to F2 mixtures. Intuitively, this implies a difference in air-void system parameters when these ash types are used. However, differences in gravimetric air were only significant when comparing C1 mixtures to F2 mixtures. This suggests a factor other than air content may be affecting freeze-thaw durability.

4.9.3 Ash Content

Table 4.47 summarizes the analysis of the means for response variables affected by ash content. Table 4.48 summarizes the observations from this analysis. As seen in Table 4.48, all response variables considered are different when mixtures with an ash content of 15% are compared with mixtures prepared with 40% ash content. This result is expected since the response variables were identified as related to ash content previously using the F-tests. The additional information obtained here is the other comparisons between mixtures with an ash content of 15% and 30%, and between mixtures with an ash content of 30% and 40%. For some response variables, the comparisons show no dependence on the change of ash content from 15 to 30%, or from 30 to 40%, while other response variables to indicate a difference between the two ash substitution levels. The practical implication is that simply increasing the ash replacement to 40% does not always result in degradation of properties or performance, but in some cases it does. Therefore, to use a 40% replacement level, additional scrutiny of the mixture design is warranted but 40% replacement should not categorically be restricted.

Table 4.47. Means of the 17 affected dependent variables at each level of ash content

Response Variable	Ash Content (weight %)		
	15	30	40
MF1	523.4	510.0	483.5
MF7	3835.8	3857.6	3719.7
MF14	7706.7	7752.1	7584.4
MS300	166.7	232.2	293.3
MS700	233.0	327.7	440.7
MS1000	300.2	425.9	600.4
MS2000	711.4	1047.7	1725.4
MS3000	1736.7	2708.2	5162.9
CS1	1580.7	1174.1	921.9
CS3	3002.9	2452.1	1868.2
CS7	4055.4	3433.2	2792.5
CS14	4661.8	4197.1	3478.6
CS28	5279.3	4898.6	4262.9
CS90	6163.6	5840.4	5408.6
FS3	472.1	415.7	373.0
FS7	553.8	500.6	452.1
FS28	641.8	595.9	567.0

Table 4.48. Summary of Table 4.47 using Tukey's HSD test (alpha = 0.05).

Response Variable	Comparison of Ash Contents (weight %)		
	15 vs. 30	15 vs. 40	30 vs. 40
MF1	Same	Different	Same
MF7	Same	Different	Different
MF14	Same	Different	Different
MS300	Different	Different	Different
MS700	Different	Different	Different
MS1000	Different	Different	Different
MS2000	Different	Different	Different
MS3000	Same	Different	Different
CS1	Different	Different	Same
CS3	Different	Different	Different
CS7	Different	Different	Different
CS14	Different	Different	Different
CS28	Same	Different	Different
CS90	Same	Different	Same
FS3	Different	Different	Different
FS7	Different	Different	Different
FS28	Same	Different	Same

4.10 Determine Occurrence of Two-Factor Interactions

The following models all have two significant independent variables identified from the main-effect models in Table 4.33. Models with two factor interactions were considered further. The R^2 values obtained from these models are given in Table 4.49 as well as *p*-values from testing whether the interaction terms are significant. None of the models had significant two-factor interactions.

Table 4.49. Models with two factor interactions.

Response Variable	Interaction Model	R^2	p-value
MF1	FT*AC	0.567	0.623
MF3	FT*AC	0.399	0.936
MF14	CT*AC	0.517	0.770
MS300	CT*AC	0.878	0.329
MS700	CT*AC	0.886	0.336
MS1000	CT*AC	0.886	0.304
MS2000	CT*AC	0.825	0.222
MS3000	CT*AC	0.694	0.332
CS1	CT*AC	0.872	0.248
CS3	CT*AC	0.886	0.528
CS7	FT*AC	0.816	0.387
CS14	FT*AC	0.730	0.693
CS28	FT*AC	0.695	0.411
FS3	CT*AC	0.746	0.078

5 Conclusions and Recommendations

5.1 Conclusions

5.1.1 Tests for Fly Ash Adsorption Capacity

As part of this research, three new tests were evaluated that can be used for either quality assurance (QA) or quality control (QC) of fly ash for paving concrete. The first test, the direct adsorption isotherm (DAI) test offers opportunity for use as a QA test in a number of ways. First, the test is the only test currently available that provides a direct measure of fly ash-AEA adsorption capacity. The results of the test are reported in units of ml AEA adsorbed per gram of fly ash and the test is specific to the ash and AEA tested. Therefore, the test can be used to evaluate either the fly ash, AEA, or both. What was not shown in this research but has been demonstrated in other research (Sutter et al., 2013; Sutter et al. 2013a) is that AEA adsorption is affected by both the fly ash and the AEA. Therefore, for a given fly ash source different AEAs can be screened and evaluated for how they will perform with a specific ash. Likewise, different ash sources can be evaluated with a specific AEA.

The iodine number test can be used as a QA or QC test given its simplicity and the fact it uses a standard solution to evaluate the ash; the ash is evaluated on a uniform basis (i.e., the ash and iodine are allowed to equilibrate). The drawback of the iodine number test when compared to the DAI test is the iodine number test does not capture the role of the AEA in the adsorption process.

The foam index test is a QC test that has become common in the fly ash industry. It suffers from having numerous methods in existence and the results of different tests are not easily compared to each other as the conditions of the test can vary significantly. If it is to be used, it is imperative that a standard procedure be adopted.

Overall, the recommended strategy is to use the foam index test on an hourly or daily basis to verify fly ash quality. Although it is not as sensitive to changes in adsorption as the other tests, it has acceptable precision when performed in a uniform manner (i.e., same operator), it can be easily performed in the field with a very minimum level of equipment, and it can be performed quickly. The test can readily be implemented at a batch plant or a ready-mix plant to test fly ash on a QC basis. The version provided in Appendix A uses a mechanical agitator, but the test can be performed by agitating the mixture manually. The next level of testing would be the iodine number test. It can also be implemented at batch plants or ready-mix operations, or it could be used at the ash producer's lab. The test would serve as a QA test to verify ash characteristics, particularly if new sources are being introduced. Because it is an equilibrium test, based on use of a standard solution, an ash producer, a ready-mix producer, or a DOT representative could perform the test in their respective labs and achieve the same results, within the inter-laboratory precision of the test. The last test in the suite of tools would be the DAI test. It can best be used as a QA test to verify ash-AEA combinations, it can be used to help guide mixture design decisions, or it can be used to trouble shoot problems identified by the other tests, including cases where powdered activated carbon is included in the fly ash.

5.1.2 Adsorption Properties of Ash Sources Tested

The ash sources tested in this research all showed a low to moderate level of AEA adsorption. The impact of each fly ash on required air entrainment was moderate, though noticeable especially at higher ash replacement levels. Interestingly, the fly ash that showed the lowest level of adsorption was the Class F ash F1. On the basis of both LOI and characterization with the adsorption-based tests, fly ash source F1 consistently demonstrated the best characteristics with regards to adsorption. The ash with the highest LOI content was ash source F2 at 2.0 wt.%, which is not unusually high for a Class F ash. The measured adsorption capacity by both the DAI test and the iodine number test was proportional to the measured LOI. The same can be said for fly ash C1; it had a low LOI and a low measured adsorption in all tests. However, fly ash C2 demonstrated anomalous behavior and is an excellent example of why it is important to measure adsorption properties rather than LOI. Fly ash C2 had the second lowest LOI (LOI = 0.20

wt.%), very comparable with fly ash F1 (LOI = 0.10 wt. %). However, the adsorption properties, as determined by all three tests, indicated that fly ash C2 had nearly the highest adsorption capacity of all the ash sources tested. Based on the iodine number test and the foam index test, fly ash C2 had the highest level of adsorption. The DAI test, the most definitive of the three, ranked fly ash C2 second only slightly behind fly ash F2. This case illustrates clearly the problem of relying on LOI rather than directly measuring the adsorption capacity. It should also be noted that only one AEA was tested in this research. As different combinations of ash and AEA are used, different behavior should be expected. Based on the tests conducted for this research, ash sources C2 and F2 are most likely to have air entraining issues, although no unusual issues were detected in this research.

5.1.3 Freeze-Thaw Performance

Based on the statistical analysis performed, when comparing all mixtures with fly ash to mixtures without fly ash, freeze-thaw durability was the property least affected by the addition of fly ash (see Table 4.33 through Table 4.35). This simply means from a freeze-thaw perspective, the performance of mixtures with fly ash could not be discriminated from mixtures without fly ash. This is partially the result of the experiment where a small matrix (n=6) of cement-only mixtures were prepared, making statistical comparisons less certain. For response variables affected by one of the four factors (when comparing cement-only mixtures to fly ash mixtures) it is interesting to note that any effect was not consistent across all fly ash replacement levels. For example, with gravimetric air content, an effect is seen when comparing mixtures with 30% ash to mixtures with cement-only, but no effect is seen for mixtures with 15 or 40 weight % fly ash (see Table 4.35). For the physical tests listed in Table 4.35, the trends make sense. For the air system tests, the trends are not clear.

When examining just the fly ash mixtures, Table 4.36 shows that for the freeze-thaw durability factor (DF), one of the four factors was significant and based on the *p*-value, fly ash type was most significant. When examining the mixtures that had a durability factor less than 80%, six contain ash source F2, one contained ash source F1, one contained ash source C1, and one had no fly ash. Likewise, in Table 4.36, fly ash type was the principal factor for variation in air content by gravimetric determination. The practical interpretation of these results is that when comparing cement-only mixtures to fly ash mixtures, freeze-thaw performance is not measurably impacted overall (i.e., only 10 of 48 mixtures or ~ 21% failed the test). However, for the 10 cases where FT performance was affected, fly ash type was the key variable (i.e., 6 of 10 were ash F2 and 7 of 10 were a Class F ash).

It should be mentioned, the modified freeze-thaw test required in this research is a severe test (i.e., 4% $CaCl_2$ solution, Method A) and some mixtures that failed would likely have passed under normal test conditions. If this modification of AASHTO T 161 is considered for future research, it may be prudent to also include a 56-day wet curing requirement to produce concrete specimens more suitable for the severe exposure or cure the specimens to a minimum compressive strength is achieved (e.g., 3000 psi). If a minimum compressive strength is to be adopted, allow for use of maturity to predict the required compressive strength.

5.1.4 Hardened Concrete Properties

5.1.4.1 Compressive and Flexural Strength

The compressive and flexural strength measured for all mixtures in this research followed the typical behavior of fly ash bearing concrete mixtures. At early ages (i.e., 1, 3, and 7 days), an increase in fly ash content resulted in a decrease in strength at a given age. The decrease was more pronounced for Class F ash concrete mixtures as compared to Class C ash concrete mixtures. The rate of strength gain for Class F ash concrete mixtures was slower than observed for Class C ash concrete mixtures. At later ages (i.e., 90 days) all fly ash mixtures were comparable to cement-only mixtures in strength. The statistical analysis (Table 4.36) indicates that for compressive strength at all ages, fly ash content was a significant factor in any differences measured. For flexural strength, fly ash content was significant at every age except 90 days. The fly ash type was only significant for compressive strength and only at 7, 14, and 28 days. This is indicative of the differential rate of strength gain when comparing Class C and Class F ash. It should be noted that early age compressive strength (i.e., 1 and 3 days) and 3 day flexural strength were also affected by cement type (Table 4.38). This was also seen in the calorimeter curves as well as the producer's certification where the PC1 cement was seen to develop strength more slowly.

5.1.4.2 Maturity

When statistically examining just the maturity factor alone, fly ash content is the significant factor at 1, 3, 7, and 14 days age (Table 4.36). When strength is considered and the maturity relationship is viewed in total (i.e., response variables MS300, MS700, MS1000, MS2000, and MS3000) cement type is equally as important as is fly ash content. Fly ash type is only significant for MS300 (Table 4.36). Therefore, if maturity is to be used as a quality control measure, it will be necessary to determine a maturity curve for each combination of cement and fly ash. Changes in cement or fly ash properties over a construction season will most certainly affect maturity and may require re-establishing the maturity curve on a regular basis. It should be noted that based on the analysis presented in Table 4.49, effect of cement type and ash content on maturity is not an interaction between the two factors. Each is influencing the maturity relationship independently.

5.2 Recommendations

- Based on this research, there is strong evidence to support using Class F fly ash in paving concrete. The use of Class C ash should also continue. With the exception of freeze-thaw testing, the performance of the Class F ash sources tested was comparable to the Class C ash sources tested. Replacement levels up to 30% can be used without significant changes in fresh or hardened concrete properties.
- Class F ash source F2 demonstrated freeze-taw performance noticeably worse than the other ash sources. Additional testing should be performed to determine if the performance of ash F2 was indicative of the material performance in general, or possibly an artifact of the freeze-thaw testing regime (i.e., Method A with 4% $CaCl_2$ solution), or an interaction with the AEA used in the study.
- Use of either ash type at replacement levels greater than 30% should be considered but performance testing of those mixtures, prepared with the job-mixture materials and mixture design, must be required.
- Based on the adsorption-based tests for characterizing AEA adsorption by fly ash, the Class C ash C2 and Class F ash F2 demonstrated the most likelihood of disrupting air entrainment, although neither significantly impacted air entrainment in this study.
- The existing LOI specification of 2% should be retained but additional testing should be performed to establish the adsorption capacity of any ash used in paving concrete.
- WisDOT should adopt a standardized version of the foam index test as a QC test for concrete producers or field inspectors. It is necessary that a standardized method be adopted to minimize variability. If the intent is to make it a field test, make optional use of the mechanical shaker and allow the operator to perform the test by shaking by hand.
- WisDOT should adopt on a provisional basis use of the direct adsorption isotherm test and the coal fly ash iodine number test. By adopting, on a provisional basis, materials providers and contractors can begin developing a knowledge base of the tests and WisDOT can begin developing a historical database for establishing specification criteria in the future.
- Any construction project using maturity as a means of predicting concrete strength should require periodic and regular calorimetry testing of the cement and fly ash (e.g., each new delivery) to ensure maturity models developed are appropriate considering changes in construction materials

that can occur over a construction season. This should be required for mixtures with or without fly ash as the portland cement is a major factor in establishing the maturity relationship.

- WisDOT should discontinue freeze-thaw testing using $CaCl_2$ solutions in the test procedure. This results in a harsh testing environment, which extends what is already a harsh test (AASHTO T 161) into a more severe environment. Additionally, $CaCl_2$ solutions are corrosive and result in destruction of freeze-thaw chambers. If these solutions are to be used in future research, it is recommended that a 56-day curing regime be used to produce concrete more suitable for sever exposure, especially when the concrete mixtures being tested contain fly ash. Alternatively, a minimum compressive strength could be established for concrete tested for FT performance.

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Appendix A - Test Methods for Assessing Fly Ash Adsorption

Draft Standard Method of Test for

Determination of Air Entraining Admixture Adsorption by Coal Fly Ash

1. SCOPE

1.1. This test method covers the determination of the quantity of air entraining admixture adsorbed by coal fly ash from an aqueous solution. The result is expressed as the volume of air entraining admixture adsorbed per unit mass of coal fly ash (mL AEA/g fly ash). The quantity of air entraining admixture adsorbed is a function of the solution air entraining admixture concentration.

1.2. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations to use.*

2. REFERENCED DOCUMENTS

2.1. *ASTM Standards*:

- D2652, Terminology Relating to Activated Carbon
- C311, Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete
- ASTM D3860 Standard Practice for Determination of Adsorptive Capacity of Activated Carbon by Aqueous Phase Isotherm Technique

3. TERMINOLOGY

3.1. The terms used in this specification relative to activated carbon are defined in ASTM D2652.

4. SUMMARY OF TEST METHOD

4.1. The determination is based upon a three-point direct adsorption isotherm similar to that described in ASTM D3860. The isotherm provides a direct measurement of the amount of air entraining admixture adsorbed by a coal fly ash.

4.2. A direct adsorption isotherm is determined by equilibrating mixtures of cement, fly ash and air entraining admixture solutions to determine the reduction in the concentration of air entraining admixture due to adsorption by coal fly ash.

4.3. Solutions containing a known concentration of air entraining admixture are equilibrated with Portland cement to establish baseline ("blank") air entraining admixture concentrations. These same concentrations of AEA solutions are then equilibrated with a combination of Portland cement and fly ash and the concentration of residual AEA is determined.

4.4. The coal fly ash adsorption capacity, q (mL AEA / g fly ash), is determined by dividing the volume of air entraining admixture adsorbed by the mass of coal fly ash utilized in determining the isotherm point. Multiple isotherm points are obtained by varying the concentration of the air entraining admixture solution. The isotherm points are analyzed using the Freundlich isotherm model that describes the correlation between coal fly ash adsorption capacity and the equilibrium air entraining admixture concentration.

4.5. Chemical oxygen demand (mg COD /L) is used to determine the concentration of air entraining admixture in solution.

4.6. The concentration of air entraining admixture in the solution affects the capacity of a coal fly ash for air entraining admixture adsorption.

4.7. The apparatus required consists of various laboratory glassware used to prepare solutions and contact coal fly ash with the air entraining admixture solutions. Filtration equipment is also required. A spectrophotometric method is employed to determine COD.

5. SIGNIFICANCE AND USE

5.1. The partitioning of an air entraining admixture among the various solid phases in a concrete mixture is identified and quantified using this direct adsorption isotherm test method. Direct adsorption isotherms quantify the interaction between coal fly ash and an air entraining admixture.

5.2. An isotherm provides a quantitative measurement of the amount of air entraining admixture adsorbed by a coal fly ash, which can be used to predict and adjust the dosage of air entraining admixture, relative to a baseline dosage used for a concrete mixture with no coal fly ash, to compensate for the air entraining admixture adsorbed when a coal fly ash is added or substituted into the concrete mixture.

6. APPARATUS

6.1. *Analytical Balance*, accuracy ± 0.01 g.

6.2. *Flasks*, Erlenmeyer 250 mL capacity with a ground glass stopper or rubber stopper.

6.3. *Volumetric Flasks*, 200 mL and 1 L.

6.4. *Vacuum Flask*, 1 L.

6.5. *Aspirator or Other Source of Vacuum.*

6.6. *Buchner Funnel*, 90 mm top inside diameter.

6.7. *Filter Paper*, Grade1, 11 µm, 90mm diameter, cellulose, Whatman qualitative filter paper, or equivalent.

6.8. *Pipets*, volumetric type, 2.0 mL and 5.0 mL capacity.

6.9. *Graduated Cylinders*, 100 and 200 mL.

6.10. *Magnetic Stirrer.*

6.11. *Drying Oven.*

6.12. *COD Determination Test Kit*, HACH Method 8000, high range COD or equivalent.

7. REAGENTS

7.1. *Water*-Distilled or reagent water.

8. MATERIALS

8.1. *Coal Fly Ash* – a grab sample, regular sample, or composite sample as described in C311 Sections 6.1 – 6.3.

8.2. *Portland Cement* – select the specific cement to be used with the coal fly ash (8.1) and air entraining admixture (8.3) in any concrete mixture. If the specific Portland cement is unavailable, a portland cement of the same type (i.e. AASHTO M85 type), similar composition and Blaine fineness may be substituted but the performance of the air entraining admixture predicted by this test method may vary.

8.3. *Air Entraining Admixture* – select the specific air entraining admixture to be used with the coal fly ash (8.1) and portland cement (8.2) in any concrete mixture.

9. MEASUREMENT OF CHEMICAL OXYGEN DEMAD

9.1. The concentration of air entraining admixture in solution is determined by measuring the solution chemical oxygen demand (COD), expressed in mg COD/L.

9.2. Standard procedures for measuring COD are given in Standard Methods for the Examination of Water and Wastewater[1]. Two specific methods are described: 5220C closed reflux titrimetric method and 5220D closed reflux colorimetric method. Either method is acceptable. Method 5220D closed reflux colorimetric method is recommended.

9.3. Kits are commercially available to facilitate performing 5220D closed reflux colorimetric method, as described in Section 6.

10. PREPARATION OF SOLUTIONS

10.1. There are no specific solution strengths required to perform the test. To accurately determine COD it is recommended that three solution strengths (i.e. solutions i, ii, and iii) be selected that result in COD falling in the range of 100 – 1300 mg/L after aliquots of the solutions have equilibrated with 20g of portland cement. These will be used to determine isotherm points i, ii, and iii, respectively.

Note 1 – In most cases AEA solutions having concentrations of 5 mL/L, 10 mL/L and 20 mL/L will produce COD_{BK} measurement (step 10.3.11) between 100 – 1300 mg/L COD after equilibrating with 20g of the portland cement.

Note 2: If laboratory facilities permit, all three solutions or coal fly ash/portland cement/solution combinations can be reacted simultaneously. The procedure described herein assumes each solution or each coal fly ash/portland cement/solution combination will be reacted individually rather than simultaneously.

10.2. The relationship between air entraining admixture concentration and COD must be determined separately for each different air entraining admixture type to be evaluated.

10.3. *Determine the COD/Air Entraining Admixture Solution Concentrations:*

10.3.1. Assemble and prepare the vacuum filter apparatus.

10.3.2. Pipette 5 mL of air entraining admixture.

10.3.3. Add the air entraining admixture to a 1 L volumetric flask and dilute the air entraining admixture with distilled water to a total solution volume of 1 L.

10.3.4. Record the initial solution concentration (C_{0i}) in ml/L air entraining admixture.

10.3.5. With a volumetric cylinder, measure 200 mL of the air entraining admixture solution.

10.3.6. Weigh 20 g of Portland cement and record to nearest 0.01g (M_{PCi}).

10.3.7. Combine the 200 mL of air entraining admixture solution with the 20 g of Portland cement in a 250 mL Erlenmeyer flask. Add a magnetic stirring bar and stopper the flask.

10.3.8. Stir for 60 minutes ± 2 minutes using a stir speed sufficient to keep the solids in suspension. If necessary, use a glass rod to initiate stirring.

10.3.9. Remove the solution and Portland cement from the stirrer and filter.

10.3.10. Determine the COD of the filtrate and record as COD_{BKi}.

10.3.11. If COD_{BKi} is less than 100 mg COD/L or greater than 1300 mg COD/L, estimate by ratio the required air entraining admixture volume to achieve a COD_{BKi} of approximately 800 mg/L COD and repeat steps 10.3.3 - 10.3.11.

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[1] Eaton, A. D., L. S. Clesceri, E. W. Rice, A. E. Greenberg and M. H. Franson, eds. Standard Methods for the Examination of Water and Wastewater, 21st ed., Port City Press, Baltimore, Md., 2005, pp. 4-58 to 4-60. ISBN 0-87553-047-8.

10.3.12. Based upon the results of 10.3.11, estimate by ratio the required air entraining admixture volume to prepare solution ii and iii with final solution concentrations (i.e. solution concentration after contact with the portland cement) such that together the three prepared solutions adequately cover the range of 100 – 1300 mg/L COD. Repeat steps 10.3.3 - 10.3.10 to prepare solution ii and iii and record M_{PCii}, M_{PCiii}, C_{0ii}, C_{0iii}, V_{Fii}, V_{Fiii}, COD_{BKii}, and COD_{BKiii}.

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11. PROCEDURE

11.1. Obtain a 300 – 400 g sample of the coal fly ash to be tested (8.1). Dry this sample to constant weight at a temperature of 110° ± 2° C.

11.2. Obtain a 300 – 400 g sample of the Portland cement to be tested (8.2).

11.3. Assemble and prepare the vacuum filter apparatus.

11.4. *Determine the COD of the Isotherm Data Points:*

11.4.1. Measure 200 mL of air entraining admixture solution i.

11.4.2. Measure 40 g of the coal fly ash to be tested. Record the weight to 0.01 g (M_{FAi}).

11.4.3. Measure 20 g of the Portland cement to be tested. Record the weight to 0.01 g (M_{PCi}).

11.4.4. Combine the 200 mL of air entraining admixture solution with the 40 g of coal fly ash (11.5.2) and the 20 g of Portland cement in a 250 mL Erlenmeyer flask. Add magnetic stirring bar and stopper the flask.

11.4.5. Stir for 60 minutes ± 2 minutes using a stir speed sufficient to keep the solids in suspension.

11.4.6. Remove the slurry from the stirrer and filter.

11.4.7. Determine the COD of the final solution and record this as COD_{FCi}.

11.4.8. Repeat steps 11.5.1 – 11.5.8 for solutions ii and iii.

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12. CALCULATION

12.1. *Determine the reduction in AEA concentration (ΔA) at each solution strength resulting from adsorption by coal fly ash:*

Reduction in AEA concentration in solution i due to adsorption by coal fly ash (*mL/L*) (repeat for solutions ii and iii):

$$\Delta A_i = \frac{(COD_{BKi} - COD_{FCi}) \times C_{0i}}{COD_{BKi}} \tag{1}$$

12.2. *Determine adsorption capacity (q) for each solution strength:*

Adsorption capacity for solution i *(mL air entraining admixture / g coal fly ash)* (repeat for solutions ii and iii):

$$q_i = \frac{\Delta A_i \times 0.2}{M_{FAi}} \tag{2}$$

12.3. *Determine residual AEA concentration (C_F) for each solution strength*

Residual AEA concentration of solution i after equilibration with cement and coal fly ash *(vol% AEA)* (repeat for solutions ii and iii):

$$C_{Fi} = \frac{C_{0i} - \Delta A_i}{10} \tag{3}$$

12.4. *Plot solution concentration versus capacity:*

Plot the results using a log-log scale. Plot the residual AEA concentration (i.e. C_{Fi}, C_{Fii}, C_{Fiii}) on the x-axis and adsorption capacity (i.e. q_i, q_{ii}, q_{iii}) on the y-axis.

Fit a power line to the data, the equation for which can be used to determine the volume of air entraining admixture adsorbed per gram of coal fly ash, as a function of the air entraining admixture concentration.

An example plot is shown in Figure 1.

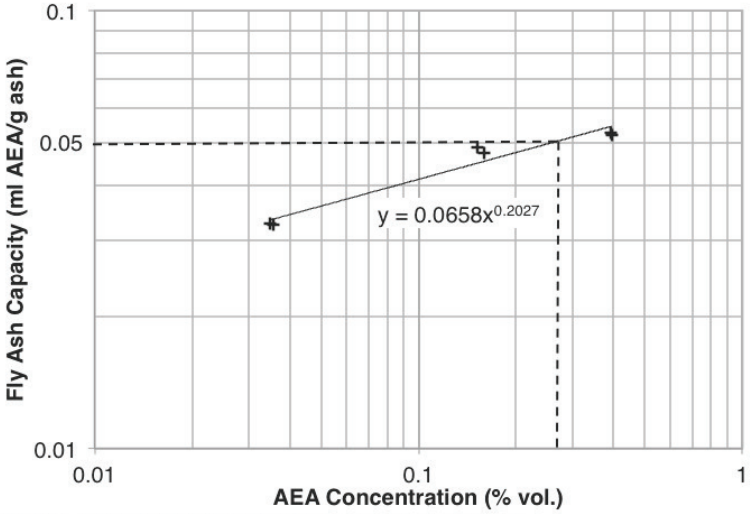

Figure 1. Example of direct adsorption isotherm for coal fly ash.

12.5. To use the graph, calculate the air entraining admixture dosage for a concrete mixture design in terms of volume % air entraining admixture. Enter the graph on the x-axis at the determined volume % air entraining admixture. Intercept the isotherm line and read off the corresponding fly ash capacity on the y-axis. The capacity determined is the volume of air entraining admixture that will be adsorbed per gram of fly ash.

12.6. An example calculation is shown below:

Concrete Mix Design Parameters:

Item	Quantity - Customary Units	Quantity - Converted Units
Water	290 lb.	34.8 gal.
Air Entraining Admixture	11.5 fl. oz.	0.09 gal.

Assume 100 pounds (45.4 kg) of fly ash is substituted for 100 pounds portland cement.

$$Volume\ \%\ Air\ Entraining\ Admixture = \frac{0.09\ gal\ Air\ Entraining\ Admixture}{34.8\ gallons\ water} \times 100 = 0.26\ volume\ \%$$

From Figure 1, fly ash capacity = 0.05 mL air entraining admixture per gram coal fly ash

$$\frac{0.05\ mL\ air\ entraining\ admixture}{g\ coal\ fly\ ash} \times \frac{45.4\ kg\ coal\ fly\ ash}{} \times \frac{1000\ g}{1\ kg} = 2270\ mL\ air\ entraining\ admixture$$

$$2270\ mL\ air\ entraining\ admixture \times \frac{1\ fl\ oz}{29.57 mL} = 76.8\ fl\ oz$$

In this example, it is estimated 100 pounds of coal fly ash has the capacity of 76.8 fl. oz. of air entraining admixture.

13. REPORT

13.1. Report the following information

- Time and date of test
- Fly ash source tested
- Cement source tested
- air entraining admixture tested
- Coal fly ash capacity plot and power fit equation for the adsorption isotherm

14. PRECISION AND BIAS

14.1. *Precision* – To be determined

14.2. *Bias* – There is no accepted standard sample that can be used to establish bias.

15. KEYWORDS

15.1. Coal fly ash; air entraining admixture; direct adsorption isotherm.

Draft Standard Method of Test for

Determination of Iodine Number of Coal Fly Ash

1. SCOPE

1.1. This test method covers the determination of the relative activation level of remnant carbon present in coal fly ash by measuring the adsorption of iodine from aqueous solution. The mass of iodine adsorbed (in milligrams) by 1 gram of carbon using test conditions listed herein is called the iodine number.

1.2. ***This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations to use.*** Specific hazard statements are given in Section 8.

2. REFERENCED DOCUMENTS

2.1. *ASTM Standards*:

- D2652, Terminology Relating to Activated Carbon
- C311, Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete
- D1510 Standard Test Method for Carbon Black—Iodine Adsorption Number

3. TERMINOLOGY

3.1. The terms used in this specification relative to activated carbon are defined in ASTM D2652.

4. SUMMARY OF TEST METHOD

4.1. A weighed sample of coal fly ash is equilibrated with a standard iodine solution. The concentration of iodine remaining in solution after equilibration is determined by titration with a standard thiosulfate solution and the adsorbed iodine is expressed as a fraction of the total mass of coal fly ash.

4.2. The apparatus required consists of various laboratory glassware for preparation of solutions, contacting coal fly ash with the standard iodine solution, filtration and titration.

5. SIGNIFICANCE AND USE

5.1. Coal fly ash is composed of inorganic and organic phases with the organic phases occurring as unburned carbon resulting from the coal combustion process. Carbon may also be present when used for flue gas treatment to meet emission standards. Carbon is assumed to be the sole adsorbent of iodine.

5.2. The iodine number is a relative indicator of porosity in an activated carbon. It does not necessarily provide a measure of the carbon's ability to adsorb other species. Iodine number may be used as an approximation of surface area for some types of activated carbons. However, the relationship between carbon surface area and iodine number cannot be generalized; carbon surface area varies with many factors relating to the source of the carbon and the conditions under which it is produced in the combustion process.

5.3. The presence of adsorbed volatiles, sulfur, and water extractable phases may affect the measured iodine number of an activated carbon in coal fly ash. This procedure includes a pre-treatment step to remove sulfur known to exist in coal fly ash.

6. APPARATUS

6.1. *Analytical Balance,* accuracy ± 0.0001 g.

6.2. *Buret,* 25 mL capacity precision buret with stand.

6.3. *Flasks,* Erlenmeyer 250 mL capacity with a ground glass or rubber stopper.

6.4. *Flask,* titration or Erlenmeyer wide-mouthed, 250 mL capacity.

6.5. *60mm filter funnels.*

6.6. *Beakers,* assorted sizes.

6.7. *Bottles,* 1 L minimum, amber, for storage of iodine and thiosulfate solutions.

6.8. *Glass-Stoppered Bottles,* 1 L minimum, for storage of potassium iodate.

6.9. *Filter Paper,* Grade1, 11 µm, 150mm diameter, cellulose, Whatman qualitative filter paper, or equivalent.

6.10. *Pipets,* volumetric type, 5.0, 10.0, and 25.0 mL capacity.

6.11. *Volumetric Flasks,* 1 L.

6.12. *Graduated Cylinders,* 100 mL.

6.13. *Eyedropper.*

6.14. *Mortar and Pestle.*

6.15. *200 Mesh Sieve.*

6.16. *Magnetic Stirring rods.*

6.17. *Hot Plate with Magnetic Stirrer.*

6.18. *Gravity Convection Drying Oven* , capable of maintaining 103° C ± 5° C.

7. REAGENTS

7.1. *Purity of Reagents*—Reagent grade chemicals shall be used in all tests

7.2. *Water*-Distilled or reagent water.

7.3. *Hydrochloric Acid,* concentrated.

7.4. *Sodium Thiosulfate,* ($Na_2S_2O_3 \cdot 5H_2O$)

7.5. *Iodine,* United States Pharmacopeia, resublimed crystals.

7.6. *Potassium Iodide*

7.7. *Potassium Iodate,* primary standard.

7.8. *Starch,* soluble potato or arrowroot.

7.9. *Sodium Carbonate*

8. HAZARDS

8.1. Several potential hazards are associated with conducting this test procedure. It is not the purpose of this standard to address all potential health and safety hazards encountered with its use. The user is responsible for establishing appropriate health and safety practices before use of this test procedure. Determine the applicability of federal and state regulations before attempting to use this test method.

8.2. Personnel conducting the iodine number procedure should be aware of potential safety and health hazards associated with the chemicals used in this procedure. The "Material Safety Data Sheet" (MSDS) for each reagent listed in Section 7 should be read and understood. Special precautions to be taken during use of each reagent are included on the MSDS. First aid procedures for contact with a chemical are also listed on its MSDS. A MSDS for each reagent may be obtained from the manufacturer. Other safety and health hazard information on reagents used in this procedure is available.[1,2,3]

8.3. Careful handling and good laboratory technique should always be used when working with chemicals. Avoid contact with hydrochloric acid or acid vapor. Care should also be taken to prevent burns during heating of various solutions during this test procedure.

8.4. The user of this test method should comply with federal, state, and local regulations for safe disposal of all samples and reagents used.

9. PREPARATION OF SOLUTIONS

9.1. *Hydrochloric Acid Solution (5 % by weight)* — Add 70 mL of concentrated hydrochloric acid to 550 mL of distilled water and mix well. A graduated cylinder may be used for measurement of volume.

9.2. *Sodium Thiosulfate* (0.025 *N)* — Dissolve 6.205 g of sodium thiosulfate in approximately 75 ± 25 mL of freshly boiled distilled water. Add 0.10 ± 0.01 g of sodium carbonate to minimize bacterial decomposition of the thiosulfate solution. Quantitatively transfer the mixture to a 1 L volumetric flask and dilute to the mark. Allow the solution to stand at least 2 days before standardizing. Store the solution in an amber bottle.

9.3. *Standard Iodine Solution* (0.025 ± 0.001 *N)* — Weigh 3.175 g of iodine and 4.775 g of potassium iodide (KI) into a beaker. Mix the dry iodine and potassium iodide. Add 2 to 5 mL of water to the beaker and stir well. Continue adding small increments of water (approximately 5 mL each) while stirring until the total volume is 50 to 60 mL. Allow the solution to stand a minimum of 4 h to ensure that all crystals are thoroughly dissolved. Occasional stirring during this 4 h period will aid in the dissolution. Quantitatively transfer to a 1 L volumetric flask and fill to the mark with distilled water. Store the solution in an amber bottle.

9.3.1. It is important that the standard iodine solution has an iodide-to- iodine weight ratio of 1.5 to 1. This achieved if by properly performing step 9.3

9.4. *Potassium Iodate Solution* (0.1000 *N)* — Dry 4 or more grams of primary standard grade potassium iodate (KIO_3) at 110 ± 5 °C for 2 h and cool to room temperature in a desiccator. Dissolve 3.5667 ± 0.1 mg of the dry potassium iodate in about 100 mL of distilled water. Quantitatively transfer to a 1 L volumetric flask and fill to the mark with distilled water. Mix thoroughly and store in a glass-stoppered bottle.

9.5. *Starch Solution*—Mix 1.0 ± 0.5 g of starch with 5 to 10 mL of cold water to make a paste. Add an additional 25 ± 5 mL of water while stirring to the starch paste. Pour the mixture, while stirring, into 1 L of boiling water and boil for 4 to 5 min. This solution should be made fresh daily.

10. STANDARDIZATION OF SOLUTIONS

10.1. *Standardization of 0.025 N Sodium Thiosulfate*—Pipet 5.0 mL of potassium iodate (KIO_3) solution from 9.4 into a 250-mL titration (or wide-mouthed Erlenmeyer) flask. Add 2.00 ± 0.01 g of potassium iodide (KI) to the flask and shake the flask to dissolve the potassium iodide crystals. Pipet 5.0 mL of concentrated hydrochloric acid into the flask. Titrate the free iodine with sodium thiosulfate solution until a light yellow color is observed in the flask. Add a few drops of starch

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[1] The "Chemical Safety Data Sheet" for the subject chemical is available from the Manufacturing Chemists Association, Washington, DC.

[2] Sax, N. I., *Dangerous Properties of Industrial Materials* , 4th edition, 1975, Van Nostrand Reinhold Company, New York, NY.

[3] *NIOSH/OSHA Pocket Guide to Chemical Hazards*, 1978, U.S. Department of Labor, Occupational Safety and Health Administration, Washington, DC. Available from U.S. Government Printing Office, Washington, DC.

indicator (9.5) and continue the titration drop-wise until one drop produces a colorless solution. Determine sodium thiosulfate normality as follows:

$$N_{\mathrm{ST}} = (\mathrm{PI} \times \mathrm{RI}) / \mathrm{ST} \tag{1}$$

where:

N_{ST} = sodium thiosulfate (N)
PI = potassium iodate (mL)
RI = potassium iodate (N)
ST = sodium thiosulfate (mL)

The titration step should be done in triplicate and the normality results averaged. Additional replications should be done if the range of values exceeds 0.003 N.

10.2. *Standardization of 0.025 ± 0.001 N Iodine Solution*— Pipet 25.0 mL of iodine solution (9.3) into a 250-mL titration (or wide-mouthed Erlenmeyer) flask. Titrate with standardized sodium thiosulfate (10.1) until the iodine solution is a light yellow color. Add a few drops of starch indicator and continue titration drop-wise until one drop produces a colorless solution. Determine the iodine solution normality as follows:

$$N_{\mathrm{I}} = (\mathrm{ST} \times N_{\mathrm{ST}}) / I \tag{2}$$

where:

N_I = iodine (N)
ST = sodium thiosulfate (mL)
N_{ST} = sodium thiosulfate (N)
I = iodine (mL)

The titration step should be done in triplicate and the normality results averaged. Additional replications should be done if the range of values exceeds 0.003 N. The iodine solution concentration must be 0.100 ± 0.001 N. If this requirement is not met, repeat 9.3 and 10.2.

11. MATERIALS

11.1. *Coal Fly Ash* – a grab sample, regular sample, or composite sample as described in C311 Sections 6.1 – 6.3.

12. PROCEDURE

12.1. Obtain a 300 – 400 g sample of the coal fly ash to be tested (11.1). Place a quantity of 5% weight HCl equivalent to or greater than four times the mass of fly ash sample in a beaker. Mix the fly ash with the 5% HCl solution, place on a hot plate and heat to boiling. Boil for five minutes then cool to ambient temperature. Filter using Grade 1, 90 mm diameter, cellulose, Whatman qualitative filter paper, or any equivalent filter paper. Dry at 103 °C to a constant weight.

12.1.1. After drying, it may be necessary to break up the treated coal fly ash using a mortar and pestle such that all of the material passes through a 200 mesh sieve.

Note 1: Due to the relatively low adsorption capacity of coal fly ash, large masses of coal fly ash are required to adsorb enough iodine to cause an accurately measurable reduction in the iodine solution concentration.

12.2. Blank Iodine Determination.

12.2.1. Pipet 25 mL of standard iodine solution (9.3) into a 250-mL titration (or wide-mouthed Erlenmeyer) flask and titrate (10.2) with the standard thiosulfate solution. Record volume of standard thiosulfate solution required for titration (V_B).

12.2.2. Perform a duplicate blank titration and average the mL of thiosulfate for use in the calculations.

12.3. Prepare filter funnel with 150 mm filter paper folded in quarters placed in 100 mL graduated cylinder.

12.4. Place 40g of the treated coal fly ash sample in a 250 ml Erlenmeyer flask, record sample weight to the nearest 0.01 g (W_F). With a graduated cylinder, measure 100.0 ml of the standard iodine solution (12.2) and add it to the flask, Add a stir bar and stopper the flask to minimize iodine volatilization. Place the flask on a stirring plate and stir the mixture for 5 minutes.

12.5. Filter the slurry pipetting 25.0 mL of filtrate into a 250-mL titration (or wide-mouthed Erlenmeyer) flask as soon as sufficient filtrate has collected in the bottom of the graduated cylinder.

12.6. Titrate with the standard thiosulfate solution. Record volume of standard thiosulfate solution required for titration (V_F).

13. CALCULATION

13.1. Calculate the iodine number (I):

$$I = \frac{(V_B - V_F)}{V_B} \times N_I \times \frac{100}{W_F} \times 126.9 \qquad (1)$$

where:

I = Fly ash iodine number (g I / kg coal fly ash)
100 = volume of standard iodine solution equilibrated (mL)
126.9 = equivalent mass of iodine (meq / mL)

14. REPORT

14.1. Report the following information

- Time and date of test
- Fly ash source tested
- Iodine number determined

15. PRECISION AND BIAS

15.1. *Precision* – To be determined

15.2. *Bias* – There is no accepted standard sample that can be used to establish bias.

16. KEYWORDS

16.1. Coal fly ash; iodine number; adsorption.

Proposed Draft Standard Method for

Determination of the Foam Index of a Coal Fly Ash and Portland Cement Slurry

1. SCOPE

1.1. This test method is for the determination of the foam index of a mixture of coal fly ash, portland cement, water, and an air entraining admixture.

1.2. The foam index can be used as a relative measure of the affect of a specific coal fly ash on the process of air entrainment in concrete batched using the same fly ash, portland cement, and air entraining agent as used in the test procedure.

1.3. The values stated in SI units are to be regarded as the standard.

1.4. *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations to use.*

Warning—Fresh hydraulic cementitious mixtures are caustic and may cause chemical burns to skin and tissue upon prolonged exposure (Note 1).

Note 1—The safety precautions given in the Manual of Aggregate and Concrete Testing, located in the related section of Volume 04.02 of the Annual Book of ASTM Standards, are recommended.

1.5. The text of these standard reference notes provides explanatory material. These notes (excluding those in tables and figures) shall not be considered as requirements of the standard.

2. REFERENCED DOCUMENTS

2.1. *ASTM Standards*:

- C125, Standard Terminology Relating to Concrete and Concrete Aggregates
- C311, Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete

3. TERMINOLOGY

3.1. *Definitions*:

3.1.1. The terms used in this specification are defined in ASTM C125.

4. SUMMARY OF TEST METHOD

4.1. The foam index test is used to estimate the affect of a specific coal fly ash on the air entrainment of concrete prepared with a specific portland cement and air entraining admixture combination. The test is performed by visually noting the stability of the foam produced when the coal fly ash, portland cement and air entraining admixture are combined with water and agitated. The test is designed to achieve a foam index value in 15 ± 3 minutes when conducted using an air entraining admixture solution strength appropriate for the coal fly ash being tested.

5. SIGNIFICANCE AND USE

5.1. The test provides an indication of possible changes in the amount of air entraining admixture required when using the same materials combination in concrete.

5.2. The foam index is not an absolute measure of air entraining admixture dosage for a concrete mixture.

5.3. The test result can be expressed in a number of different ways, depending upon the need of the users, as presented in Section 9.

6. APPARATUS

6.1. *Pipette* – capable of delivering a drop volume of 0.02 ml per drop.

6.1.1. Pipette should be calibrated prior to use in accordance standard laboratory procedures.

6.2. *250 ml wide-mouth Nalgene®-type container with a tight fitting screw top lid.*

6.3. *Wrist Action Type Laboratory Shaker* – Capable of holding a 250mL wide-mouth Nalgene®-type container.

6.3.1. Wrist Action Type Laboratory Shaker should be adjustable in sample displacement and have timer control capable of producing a 10 second and 30 second shake cycle. An example is shown in Figure 1.

Figure 1. Example wrist action type laboratory shaker.

7. MATERIALS

7.1. *Coal Fly Ash* – a grab sample, regular sample, or composite sample as described in C311 Sections 6.1 – 6.3.

7.2. *Portland Cement* – a 2 – 4 kg sample of the portland cement that is to be used, along with the coal fly ash, in the final concrete mixture.

7.3. *Distilled Water* – an adequate supply of distilled water.

7.4. *Air Entraining Admixture* - an adequate supply of the air entraining admixture to be used, along with the coal fly ash, in the final concrete mixture.

7.4.1. The air entraining admixture should be prepared as standard aqueous solutions to be used for testing. The concentration of the standard solutions can vary depending upon the coal fly ash and air entraining admixture being tested. Recommended solution strengths are 2%, 6%, 10%, and 15% air entraining admixture by volume. Solutions of any known concentration can be used.

7.4.2. To ensure accuracy in mixing standard solutions, a minimum of 1 L of solution should be prepared.

7.4.3. Consult the air entraining admixture manufacturers recommendations regarding the shelf life of the prepared solutions.

8. PROCEDURE

8.1. *Determine Blank Sample Air Entraining Admixture Requirements (Optional)*

8.1.1. This part of the procedure establishes the air entraining admixture needed to achieve a stable foam with cement only. Depending upon how the results of the foam index test are to be presented and used, this portion may be optional.

8.1.1.1 Determine the initial solution concentration to use for the test. For cement the lowest concentration solution should suffice.

8.1.1.2 In a 250 ml wide-mouth Nalgene®-type container with a tight fitting cap, combine 25 mL distilled water and 10 g of portland cement and tightly seal the container.

8.1.1.3 Secure container in the wrist action shaker and agitate the container for 30 seconds displacing it vertically approximately 20 cm.

8.1.1.4 Open the cap on the container.

8.1.1.5 With the container still in the wrist action shaker, pipette a single drop (0.02 ml) of air entraining admixture solution and tightly reseal the container.

8.1.1.6 Agitate the container with the wrist action shaker for 10 seconds displacing it vertically approximately 20 cm.

8.1.1.7 With the container still in the wrist action shaker, open the cap leaving the container undisturbed and observe the contents at the air-slurry interface for foam.

8.1.1.8 If no foam is present or the foam is stable for less than 15 seconds, repeat steps 8.1.1.4 – 8.1.1.7 until a stable foam remains for 15 seconds.

Note 2 — *A stable foam is defined as a continuous foam cover at the air/liquid interface.*

8.1.1.9 If the stable foam is achieved within a total test time of 12-18 minutes, record the total number of drops of air entraining admixture solution added to achieve a stable foam ($N_{D\,cement}$), the solution concentration of the air entraining admixture solution used ($C_{S\,cement}$), and the total test time (t_{cement}).

8.1.1.10 If the stable foam is achieved outside 12-18 minutes, adjust the solution concentration as described in Figure 2 and proceed from Step 8.1.1.2.

8.2. *Determine Combined Portland Cement & Coal Fly Ash Air Entraining Admixture Requirements*

8.2.1. This part of the procedure establishes the air entraining admixture needed to achieve a stable foam with portland cement and coal fly ash combined.

8.2.1.1 Determine the initial solution concentration to use for the test. For blends of cement and coal fly ash the choice will be based upon experience or available information (e.g. known LOI).

8.2.1.2 In a 250 ml wide-mouth Nalgene®-type container with a tight fitting cap, combine 25 ml distilled water, 8 g of portland cement and 2 g of coal fly ash and tightly seal the container.

8.2.1.3 Secure container in the wrist action shaker and agitate the container for 30 seconds displacing it vertically approximately 20 cm.

8.2.1.4 Open the cap on the container.

8.2.1.5 With the container still in the wrist action shaker, pipette a single drop (0.02 ml) of air entraining admixture solution and tightly reseal the container.

8.2.1.6 Agitate the container with the wrist action shaker for 10 seconds displacing it vertically approximately 20 cm.

8.2.1.7 With the container still in the wrist action shaker, open the cap leaving the container undisturbed and observe the contents at the air-slurry interface for foam.

8.2.1.8 If no foam is present or the foam is stable for less than 15 seconds, repeat steps 8.2.1.4 – 8.2.1.7 until a stable foam remains for 15 seconds.

8.2.1.9 If the stable foam is achieved within a total test time of 12-18 minutes, record the total number of drops of air entraining admixture solution added to achieve a stable foam ($N_{D\,ash}$), the solution concentration of the air entraining admixture solution used ($C_{S\,ash}$), and the total test time (t_{ash}).

8.2.1.10 If the stable foam is achieved outside 12-18 minutes, adjust the solution concentration as described in Figure 2 and proceed from Step 8.2.1.2.

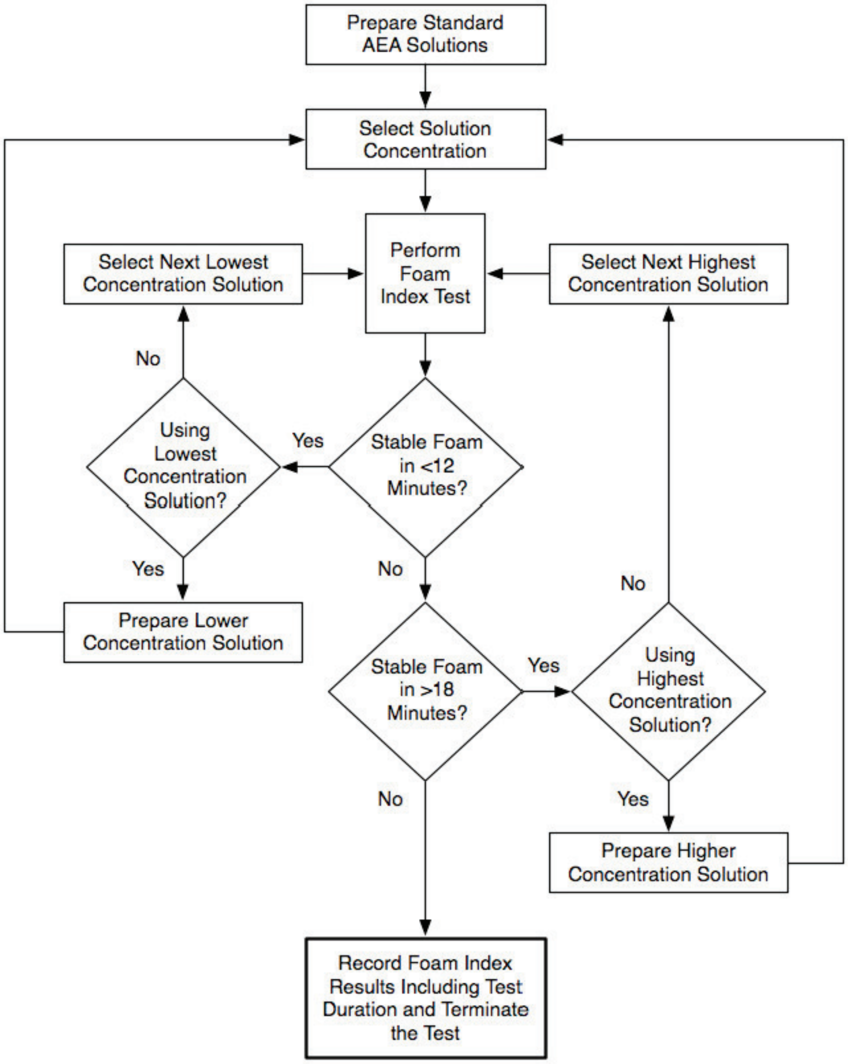


Figure 2. Protocol for conducting the foam index test and establishing the optimum AEA solution concentration to achieve an endpoint in 15 ± 3 minutes.

9. CALCULATION

9.1. The results of the foam index test can be expressed many different ways. Each of these may have application depending upon the purposes of the test.

9.1.1. Calculations are based on 10 g of cementitious material for all tests conducted

9.1.2. Data collected will consist of:

$N_{D\,cement}$ = number of drops air entraining admixture solution added to cement only sample

$C_{S\,cement}$ = concentration of air entraining admixture solution added to cement only sample

$N_{D\,ash}$ = number of drops air entraining admixture solution added to cement/coal fly ash sample

$C_{S\,ash}$ = concentration of air entraining admixture solution added to cement/coal fly ash sample

9.2. $Foam\ Index_{\,cement} = N_{D\,cement} \bullet 0.02$

9.3. $Foam\ Index_{\,ash} = N_{D\,ash} \bullet 0.02$

9.4. $Absolute\ Volume_{\,cement} = N_{D\,cement} \bullet 0.02 \bullet C_{S\,cement}$

9.5. $Absolute\ Volume_{\,ash} = N_{D\,ash} \bullet 0.02 \bullet C_{S\,ash}$

9.6. $Specific\ Foam\ Index_{\,cement} = Absolute\ Volume_{\,cement} \bullet 10{,}000$

9.7. $Specific\ Foam\ Index_{\,ash} = Absolute\ Volume_{\,ash} \bullet 10{,}000$

9.8. $Relative\ Foam\ Index = \left[(Absolute\ Volume_{\,ash}) \,/\, (Absolute\ Volume_{\,cement})\right] * 100$

Where:

Foam Index = volume of diluted air entraining admixture solution added in the test, *ml*

Absolute Volume = volume of undiluted air entraining admixture solution added in the test, *ml*

Specific Foam Index = undiluted air entraining admixture per 100kg cementitious material, *ml*

Relative Foam Index = ratio of air entraining admixture needed for cementitious mixture containing coal fly ash with that required for just cement only, expressed as a percent of that required for just cement only

10. REPORT

10.1. Report the following information

- Time and date of test
- Fly ash source tested
- Portland cement tested
- Air entraining admixture tested
- Solution strength (both $C_{S\,cement}$ and $C_{S\,ash}$ as applicable)
- Total test time (both $t_{\,cement}$ and $t_{\,ash}$ as applicable)
- Results of 9.2 – 9.8 (as applicable)

11. PRECISION AND BIAS

11.1. *Precision* – A precision statement for this test has not yet been established.

11.2. *Bias* – There is no accepted standard sample that can be used to establish bias.

12. KEYWORDS

12.1. Coal fly ash; air entraining admixture; foam index.

Appendix B - Direct Adsorption Isotherm Plots

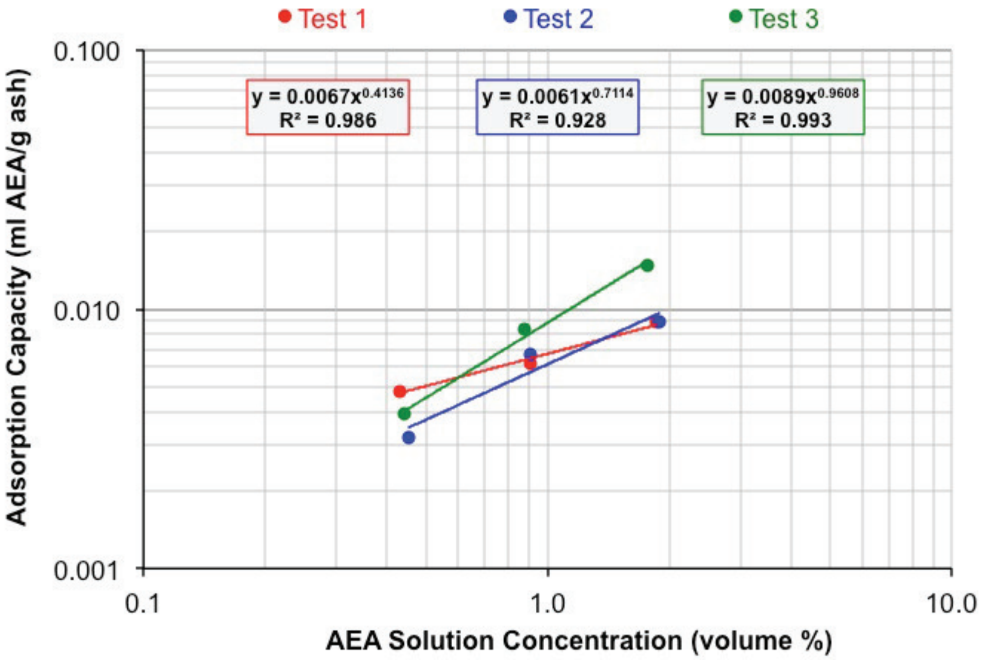


Figure B1. Direct adsorption isotherm for AEA1, PC1 Type I/II cement, and fly ash source C1.

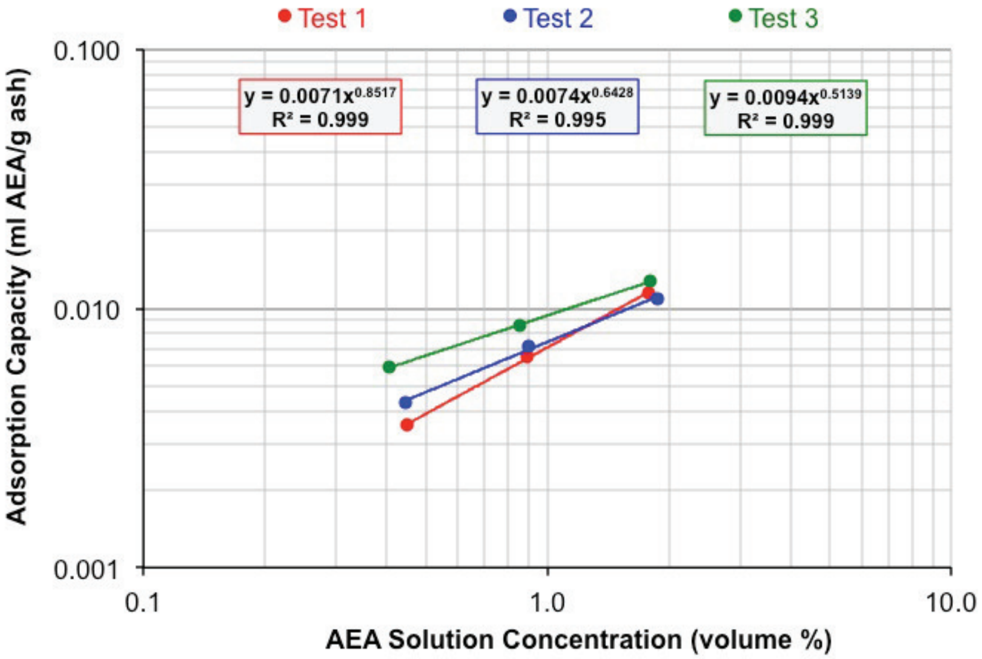


Figure B2. Direct adsorption isotherm for AEA1, PC2 Type I/II cement, and fly ash source C1.

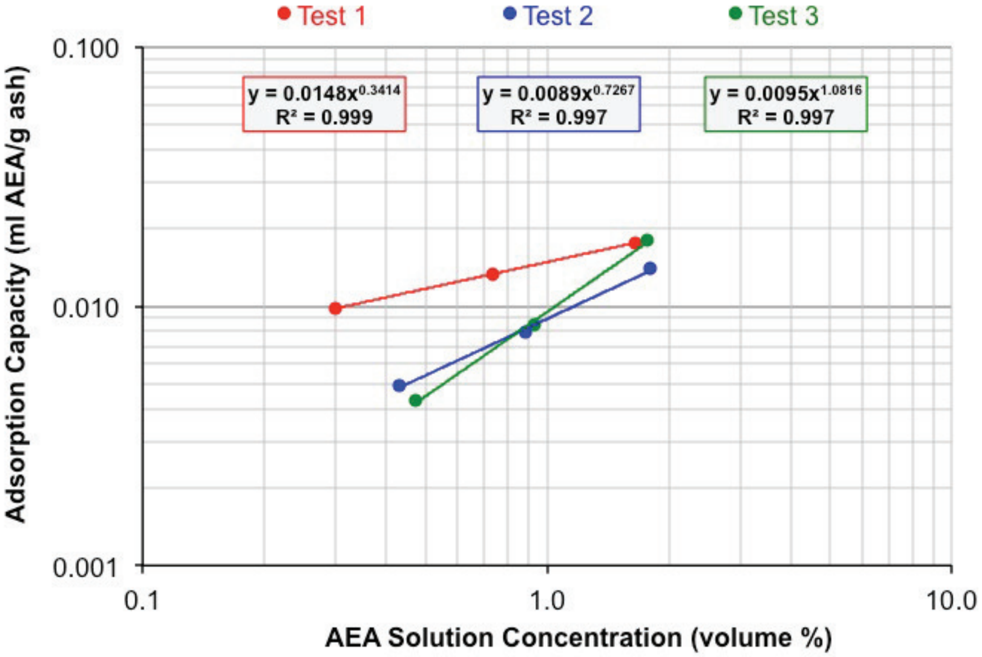


Figure B3. Direct adsorption isotherm for AEA1, PC3 Type I/II cement, and fly ash source C1.

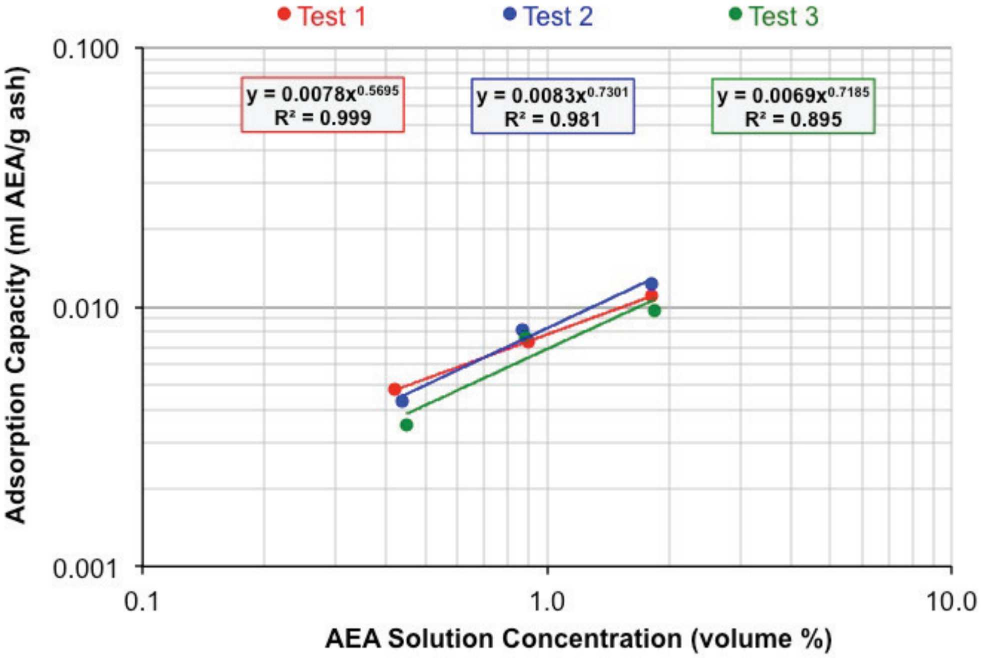


Figure B4. Direct adsorption isotherm for AEA1, PC1 Type I/II cement, and fly ash source C2.

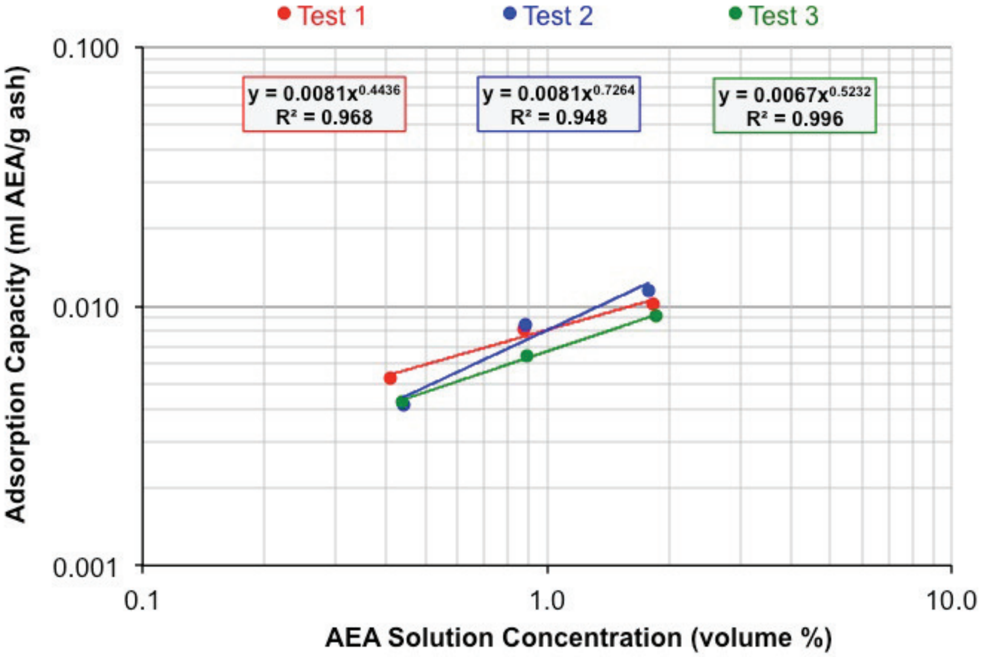


Figure B5. Direct adsorption isotherm for AEA1, PC2 Type I/II cement, and fly ash source C2.

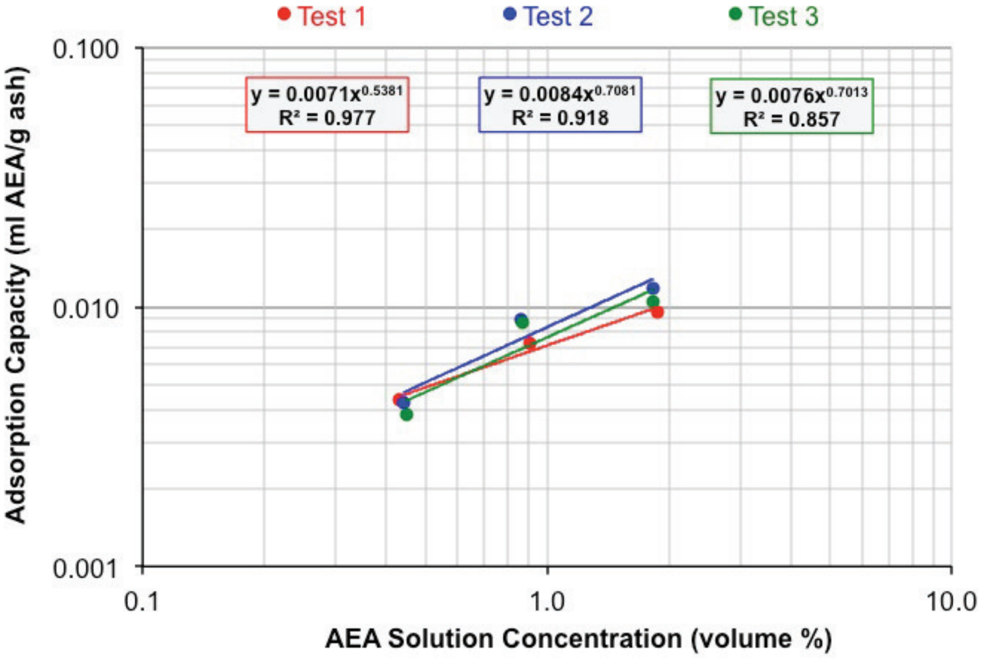


Figure B6. Direct adsorption isotherm for AEA1, PC3 Type I/II cement, and fly ash source C2.

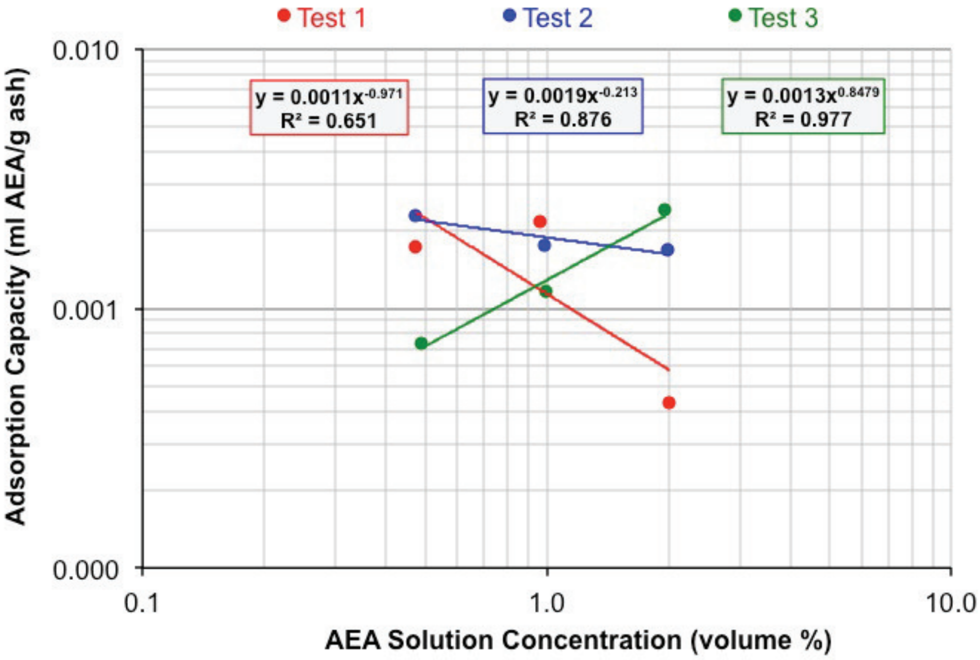


Figure B7. Direct adsorption isotherm for AEA1, PC1 Type I/II cement, and fly ash source F1.

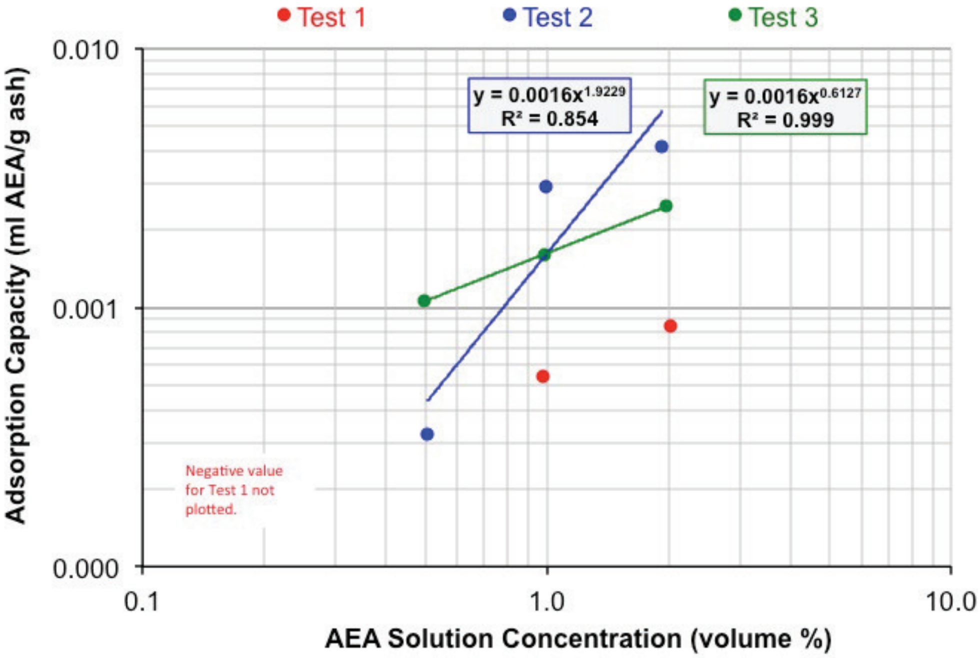


Figure B8. Direct adsorption isotherm for AEA1, PC2 Type I/II cement, and fly ash source F1.

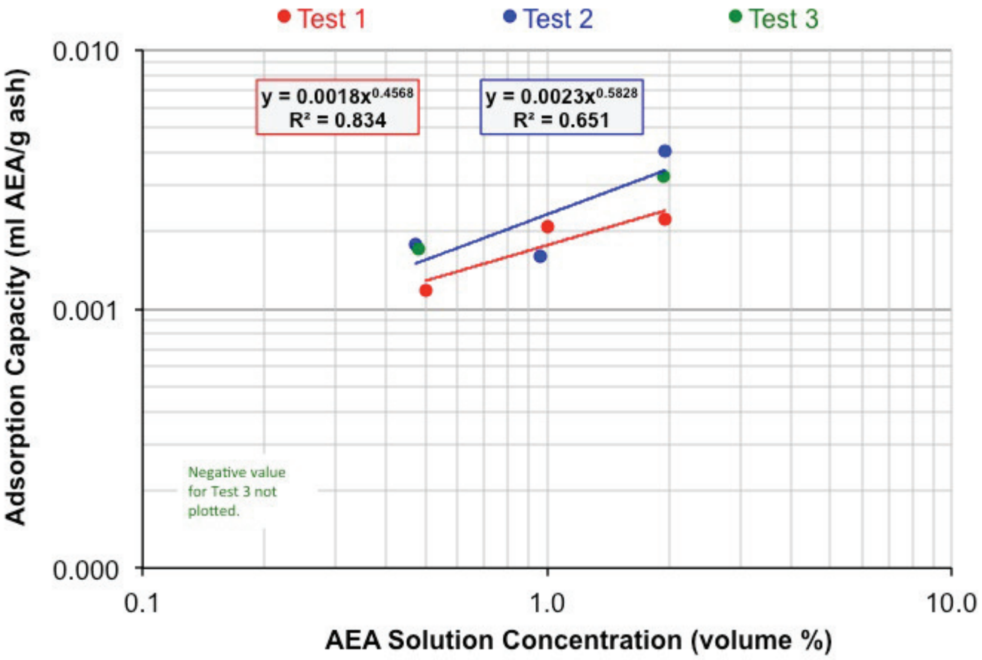


Figure B9. Direct adsorption isotherm for AEA1, PC3 Type I/II cement, and fly ash source F1.

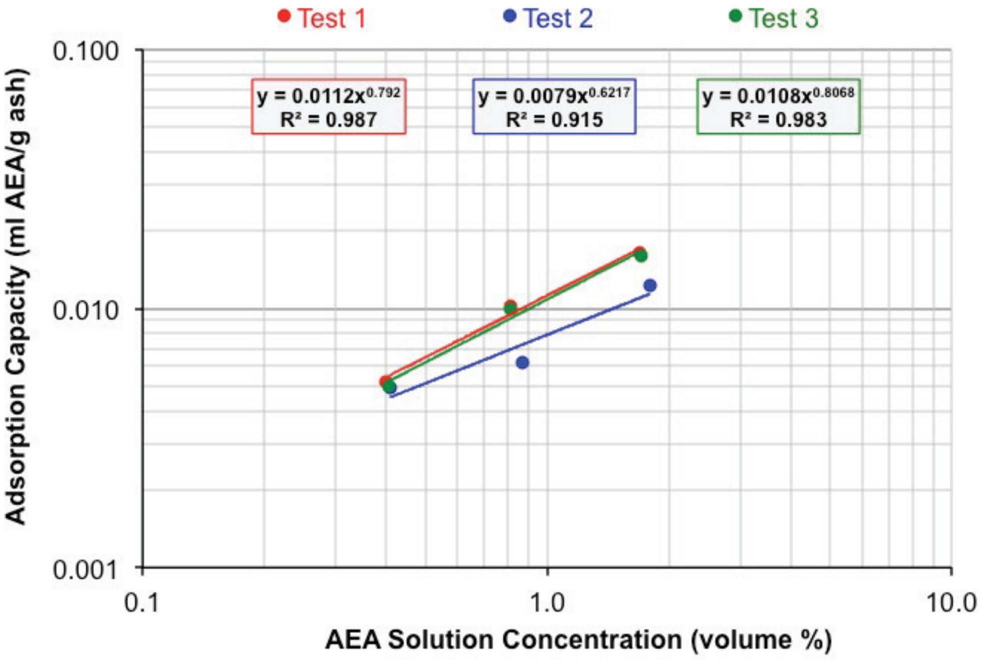


Figure B10. Direct adsorption isotherm for AEA1, PC1 Type I/II cement, and fly ash source F2.

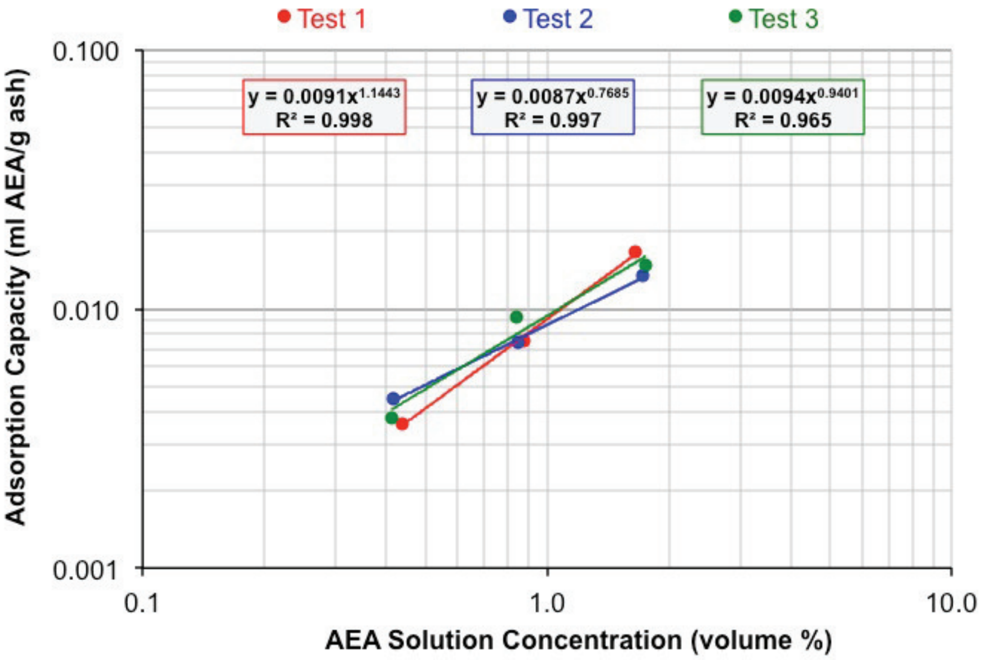


Figure B11. Direct adsorption isotherm for AEA1, PC2 Type I/II cement, and fly ash source F2.

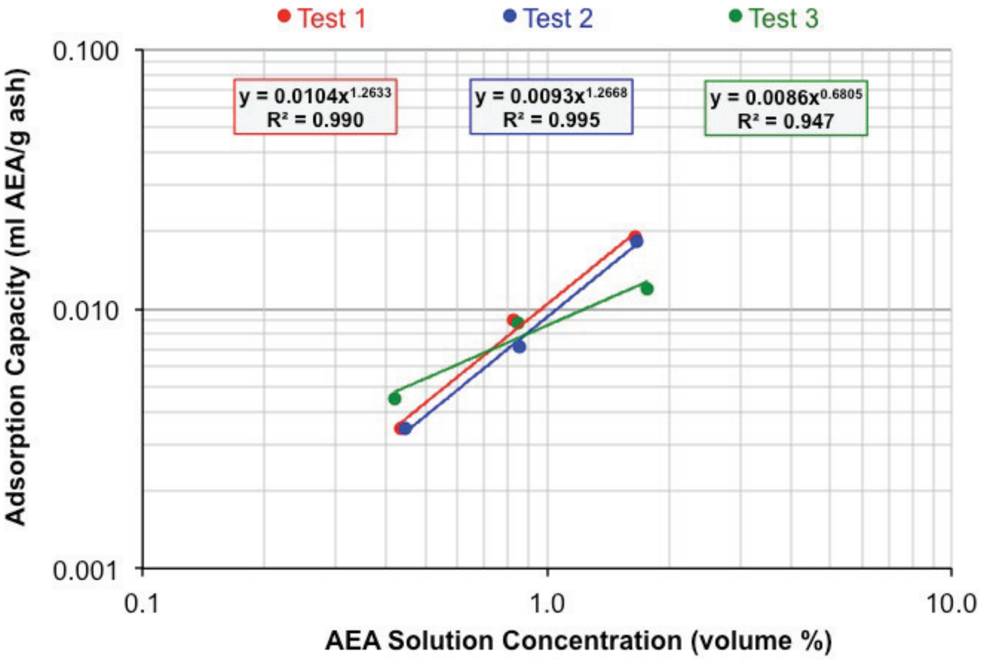


Figure B12. Direct adsorption isotherm for AEA1, PC3 Type I/II cement, and fly ash source F2.

Appendix C - Calorimetry Data

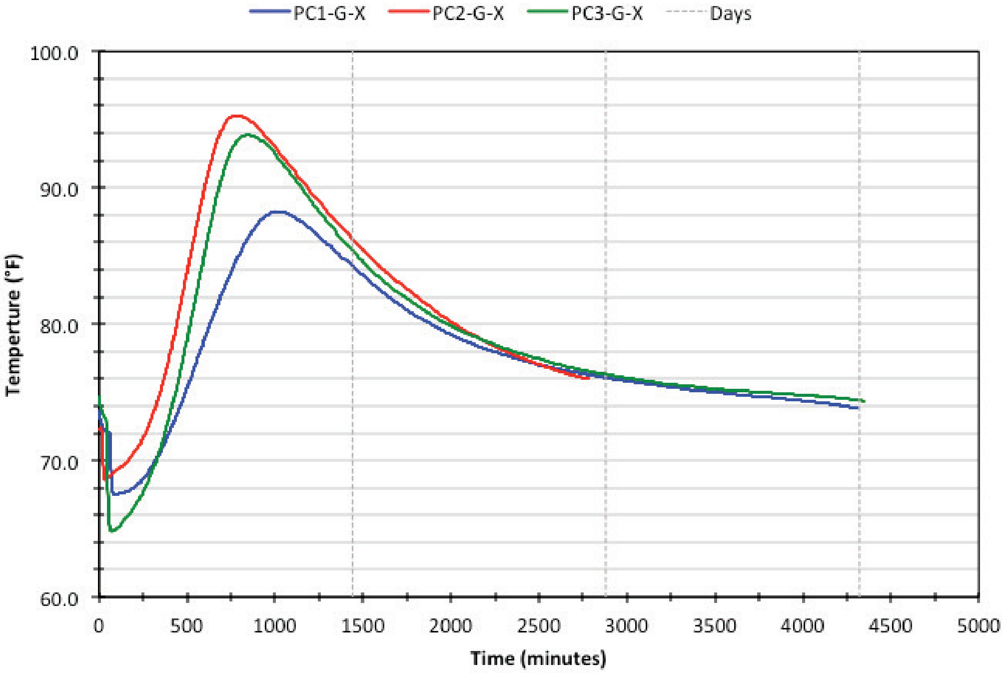


Figure C1. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with each cement, no fly ash, and the glacial gravel aggregate.

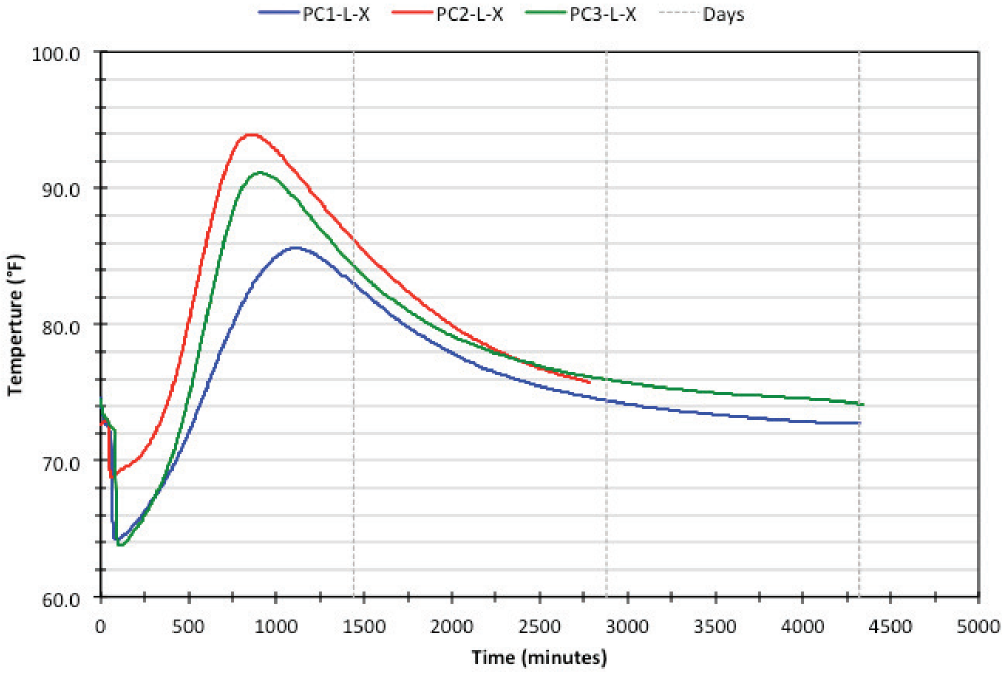


Figure C2. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with each cement, no fly ash, and the quarried carbonate aggregate.

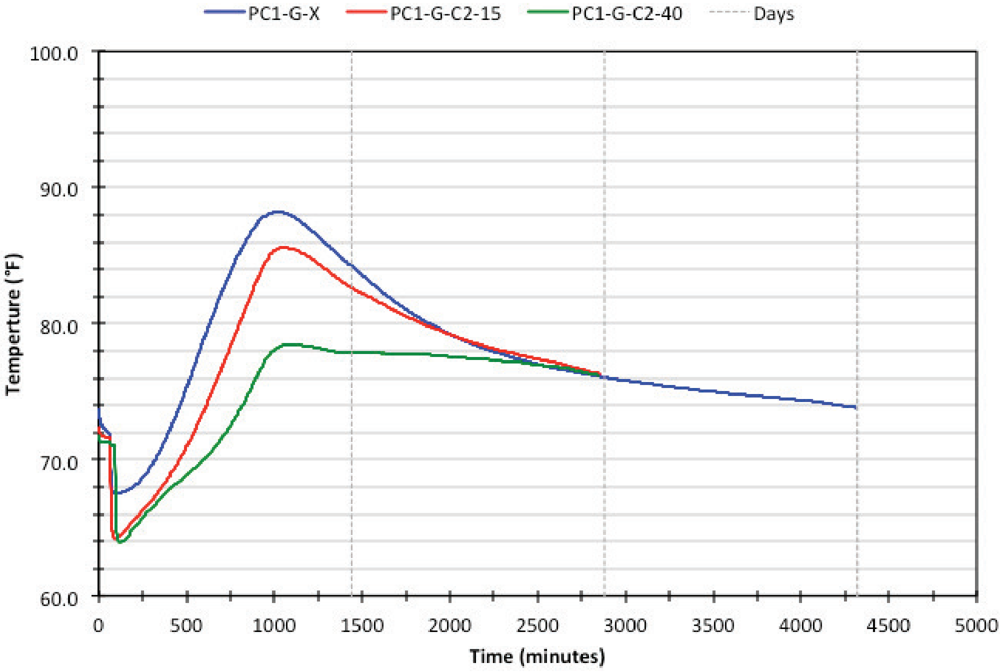


Figure C3. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC1 cement, fly ash C2 at 15 and 30%, and the glacial gravel aggregate. The heat curve for PC1 cement, glacial gravel aggregate, and no ash is included for comparison.

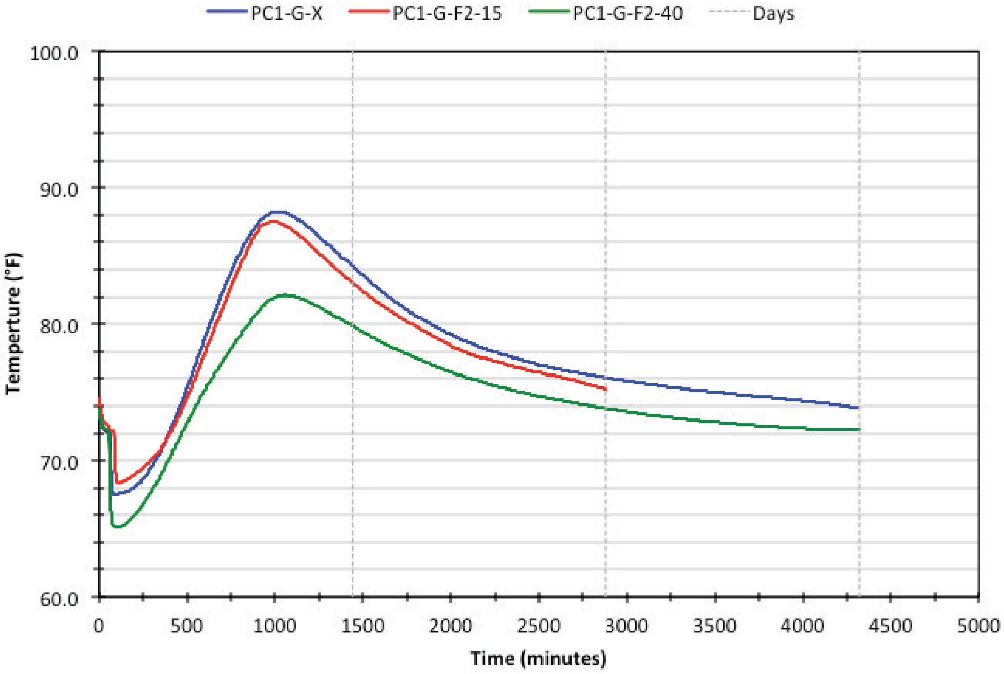


Figure C4. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC1 cement, fly ash F2 at 15 and 40%, and the glacial gravel aggregate. The heat curve for PC1 cement, glacial gravel aggregate, and no ash is included for comparison.

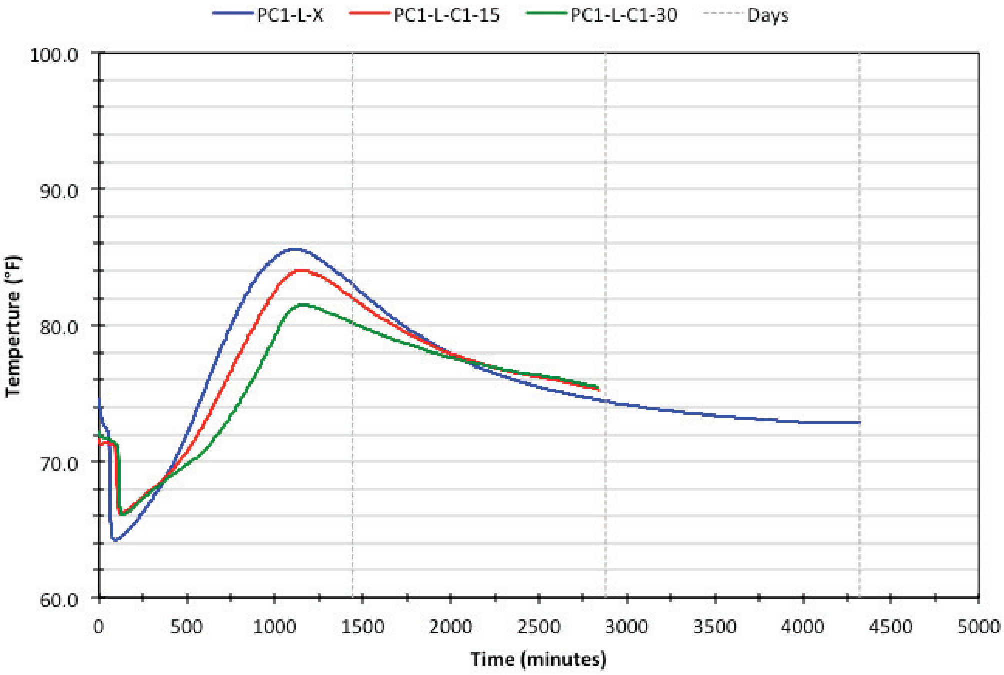


Figure C5. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC1 cement, fly ash C1 at 15 and 30%, and the quarried carbonate aggregate. The heat curve for PC1 cement, quarried carbonate aggregate, and no ash is included for comparison.

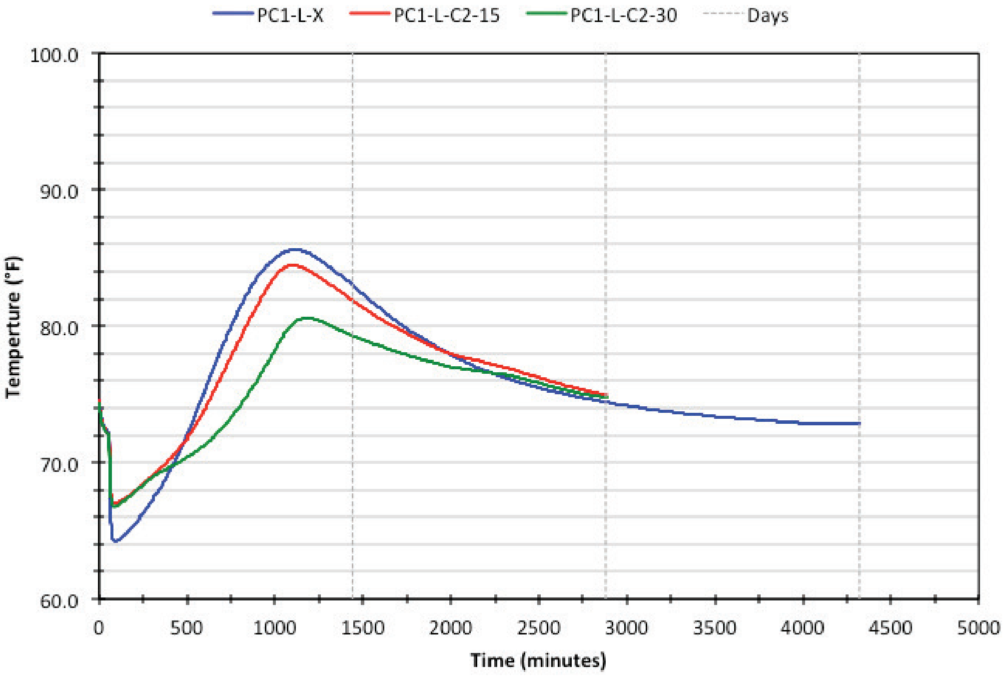


Figure C6. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC1 cement, fly ash C2 at 15 and 30%, and the quarried carbonate aggregate. The heat curve for PC1 cement, quarried carbonate aggregate, and no ash is included for comparison.

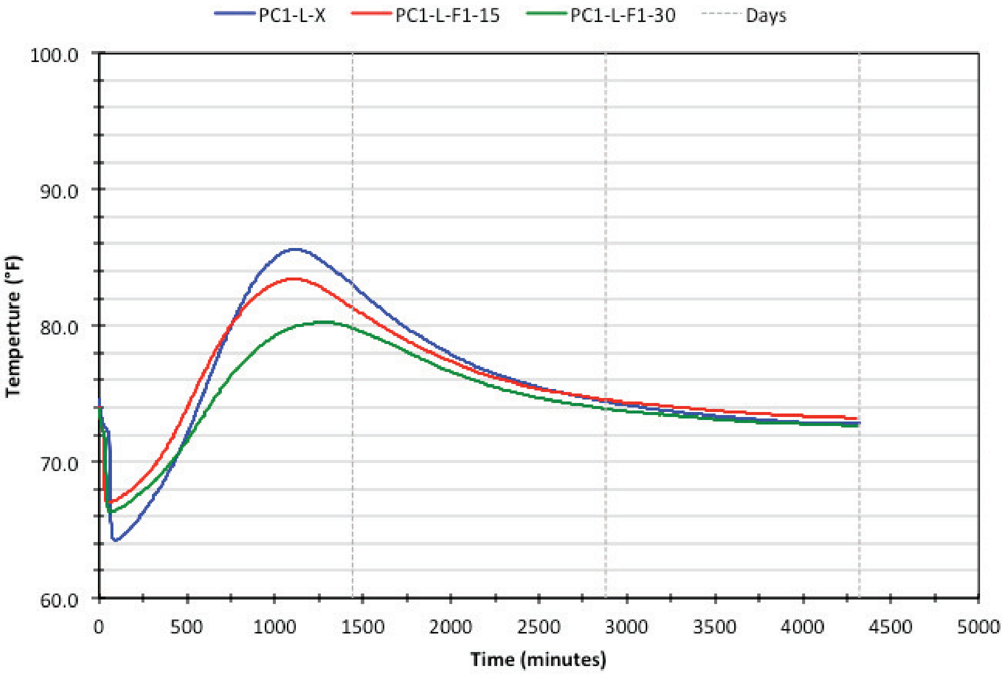


Figure C7. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC1 cement, fly ash F1 at 15 and 30%, and the quarried carbonate aggregate. The heat curve for PC1 cement, quarried carbonate aggregate, and no ash is included for comparison.

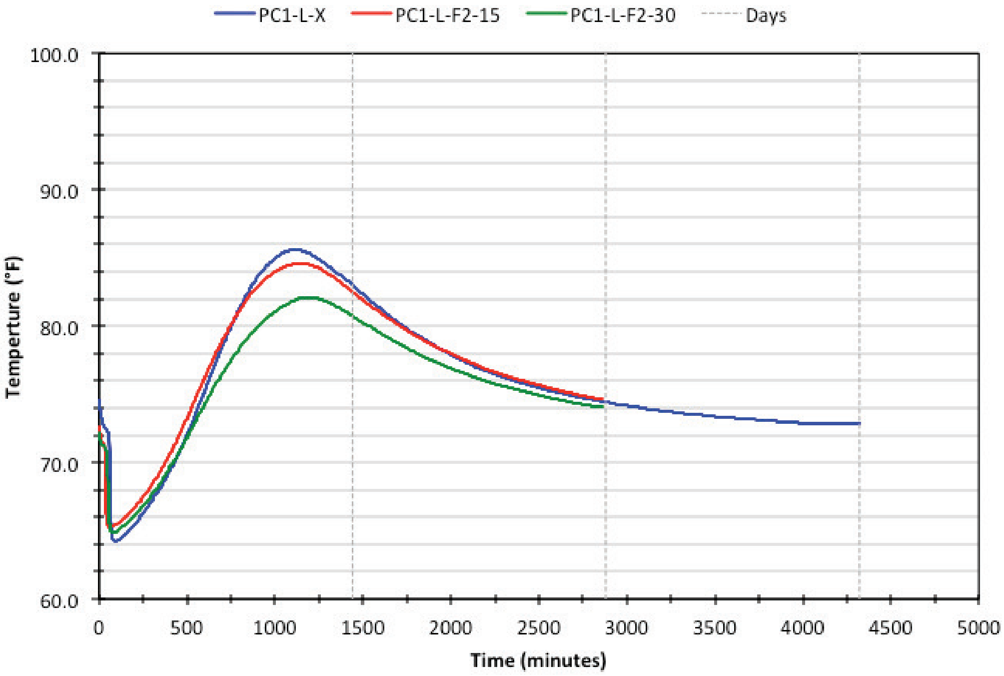


Figure C8. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC1 cement, fly ash F2 at 15 and 30%, and the quarried carbonate aggregate. The heat curve for PC1 cement, quarried carbonate aggregate, and no ash is included for comparison.

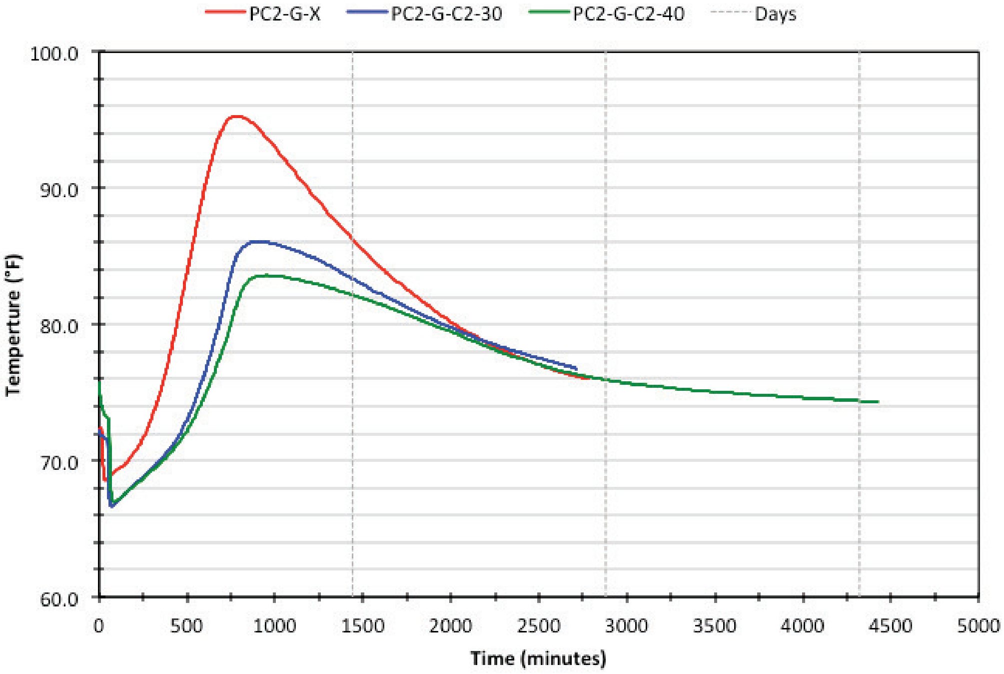


Figure C9. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC2 cement, fly ash C2 at 30 and 40%, and the glacial gravel aggregate. The heat curve for PC2 cement, glacial gravel aggregate, and no ash is included for comparison.

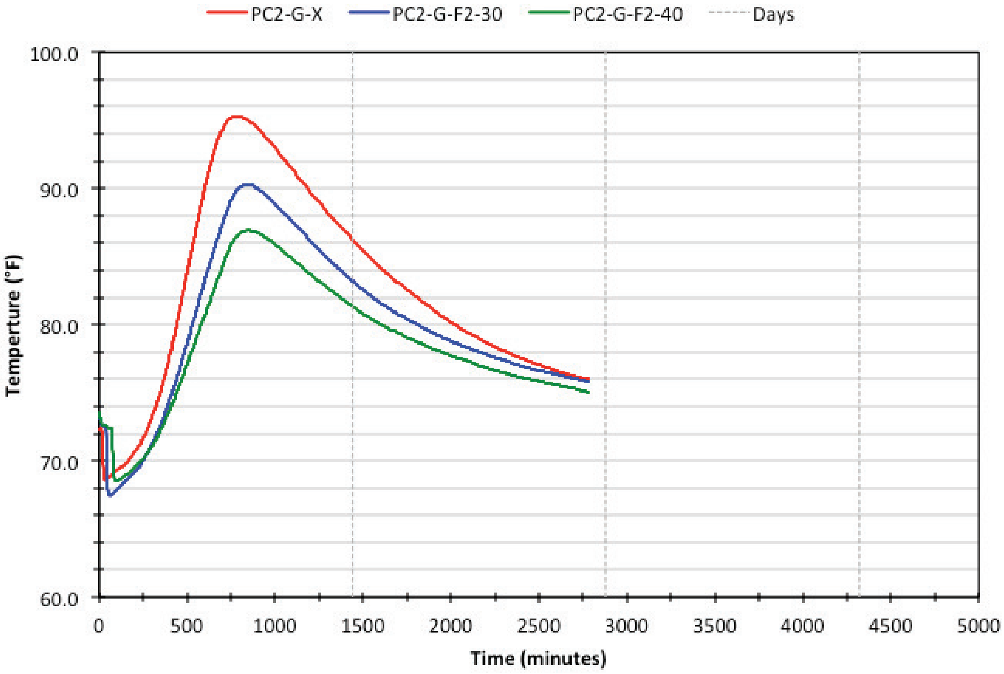


Figure C10. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC2 cement, fly ash F2 at 30 and 40%, and the glacial gravel aggregate. The heat curve for PC2 cement, glacial gravel aggregate, and no ash is included for comparison.

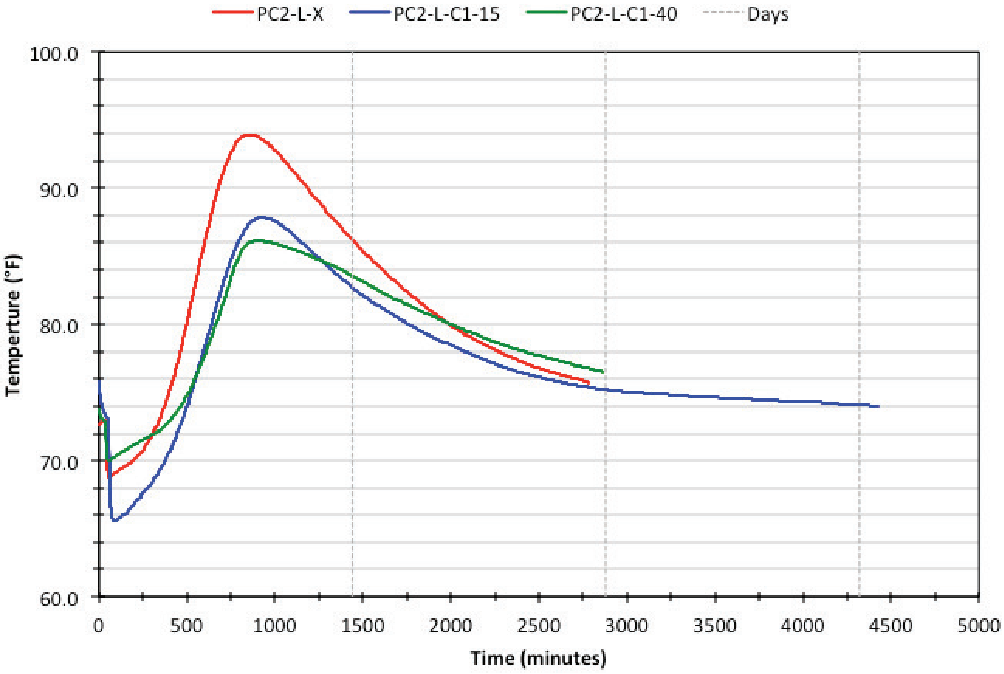


Figure C11. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC2 cement, fly ash C1 at 15 and 40%, and the quarried carbonate aggregate. The heat curve for PC2 cement, quarried carbonate aggregate, and no ash is included for comparison.

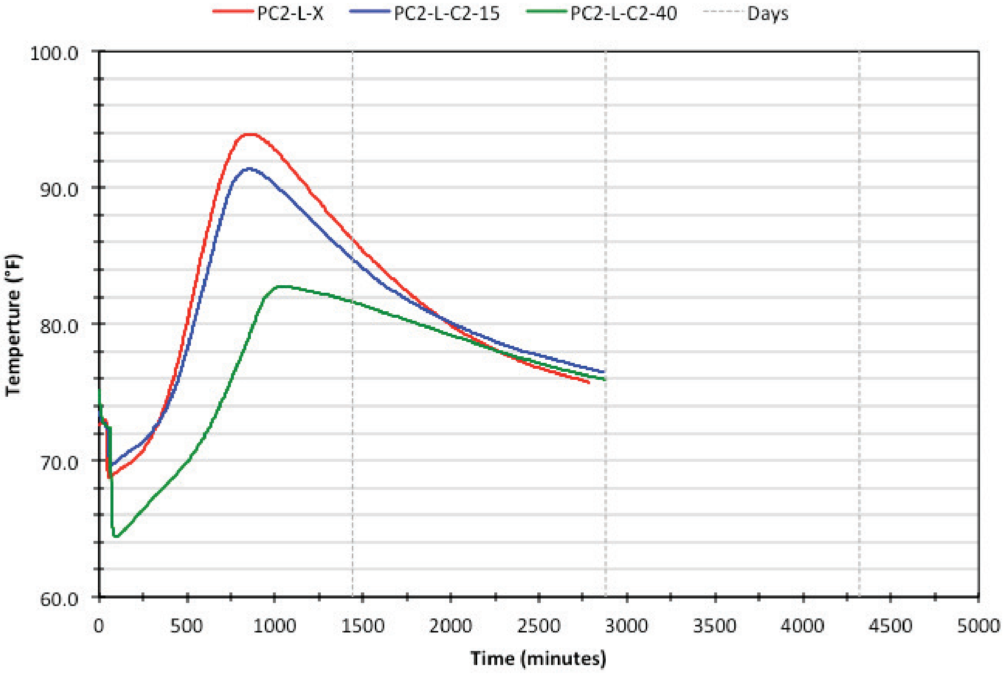


Figure C12. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC2 cement, fly ash C2 at 15 and 40%, and the quarried carbonate aggregate. The heat curve for PC2 cement, quarried carbonate aggregate, and no ash is included for comparison.

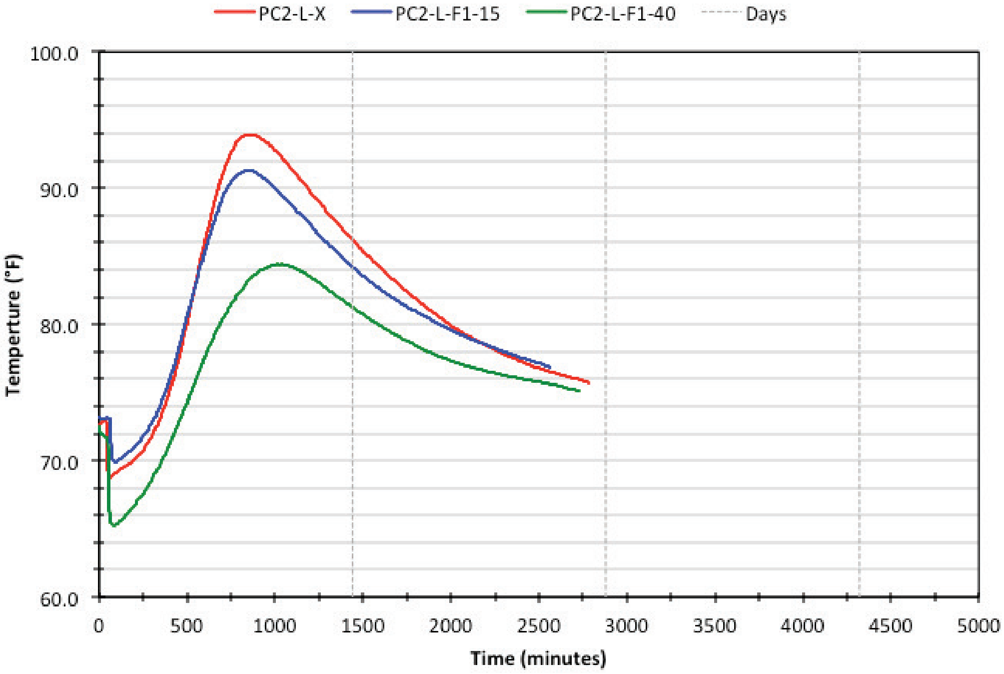


Figure C13. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC2 cement, fly ash F1 at 15 and 40%, and the quarried carbonate aggregate. The heat curve for PC2 cement, quarried carbonate aggregate, and no ash is included for comparison.

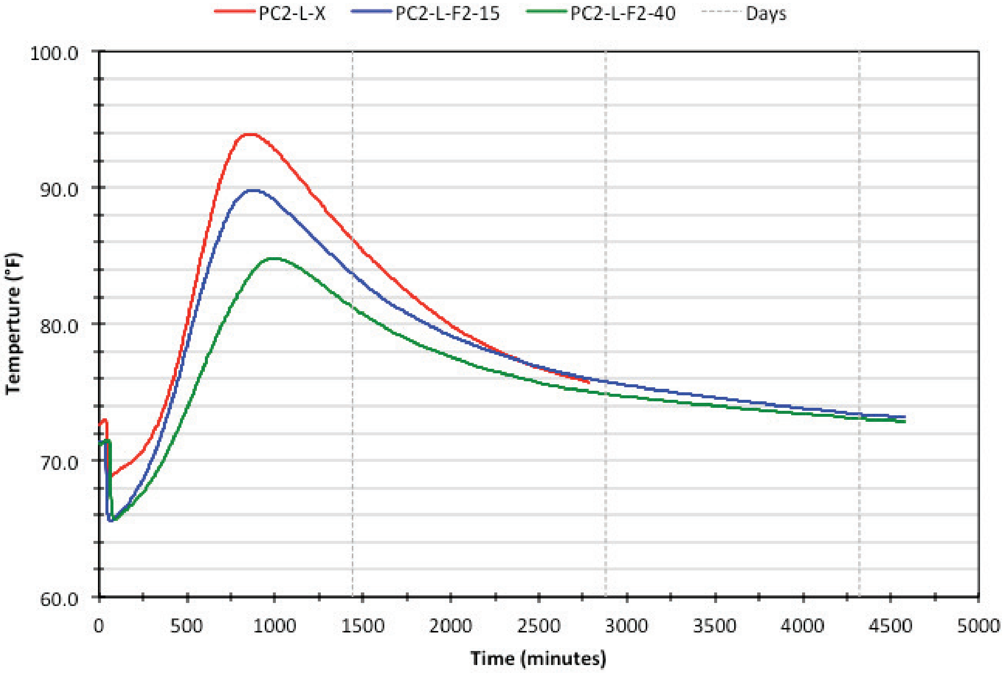


Figure C14. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC2 cement, fly ash F2 at 15 and 40%, and the quarried carbonate aggregate. The heat curve for PC2 cement, quarried carbonate aggregate, and no ash is included for comparison.

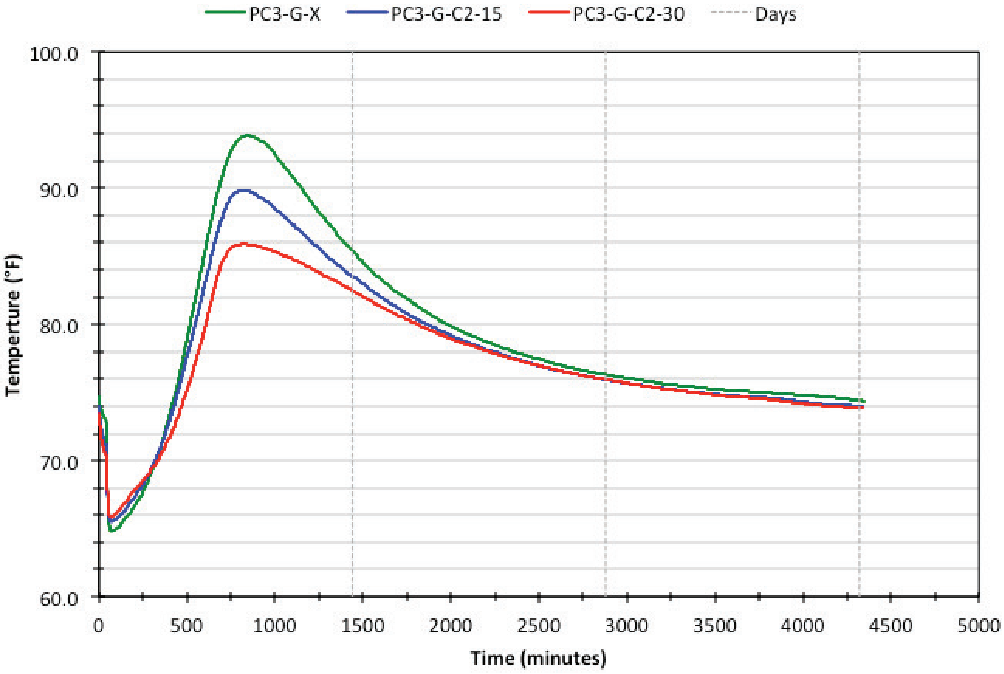


Figure C15. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC3 cement, fly ash C2 at 15 and 30%, and the glacial gravel aggregate. The heat curve for PC3 cement, glacial gravel aggregate, and no ash is included for comparison.

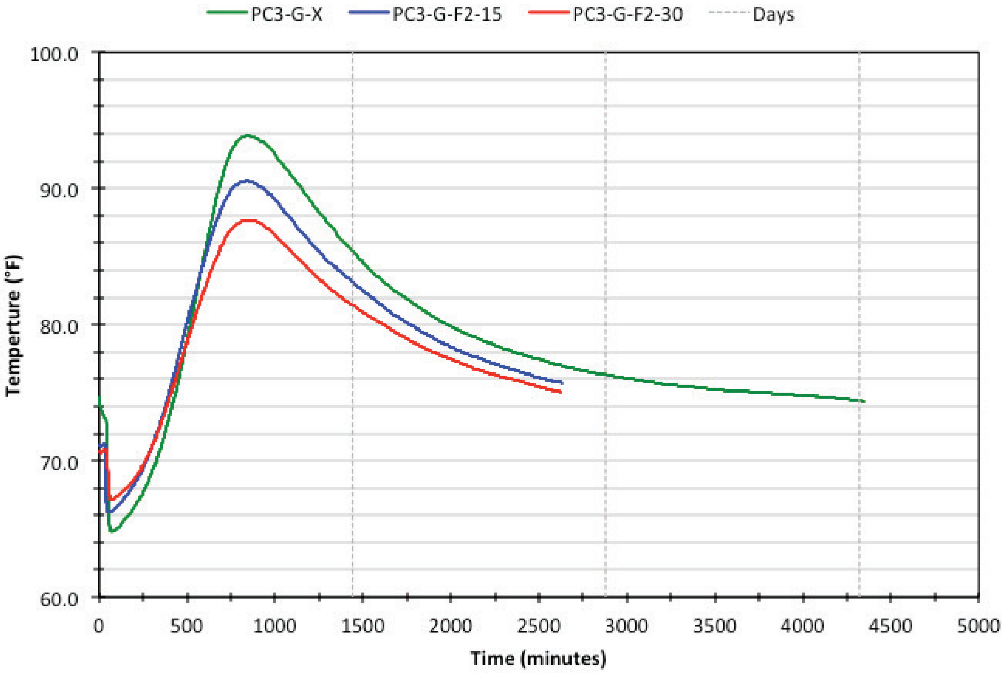


Figure C16. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC3 cement, fly ash F2 at 15 and 30%, and the glacial gravel aggregate. The heat curve for PC3 cement, glacial gravel aggregate, and no ash is included for comparison.

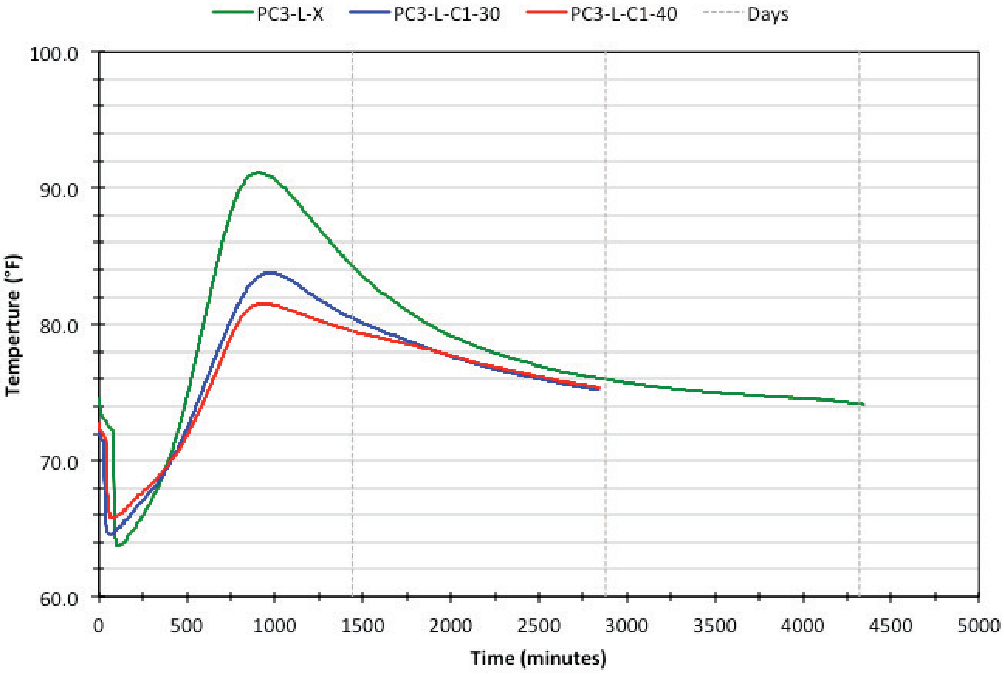


Figure C17. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC3 cement, fly ash C1 at 30 and 40%, and the quarried carbonate aggregate. The heat curve for PC3 cement, quarried carbonate aggregate, and no ash is included for comparison.

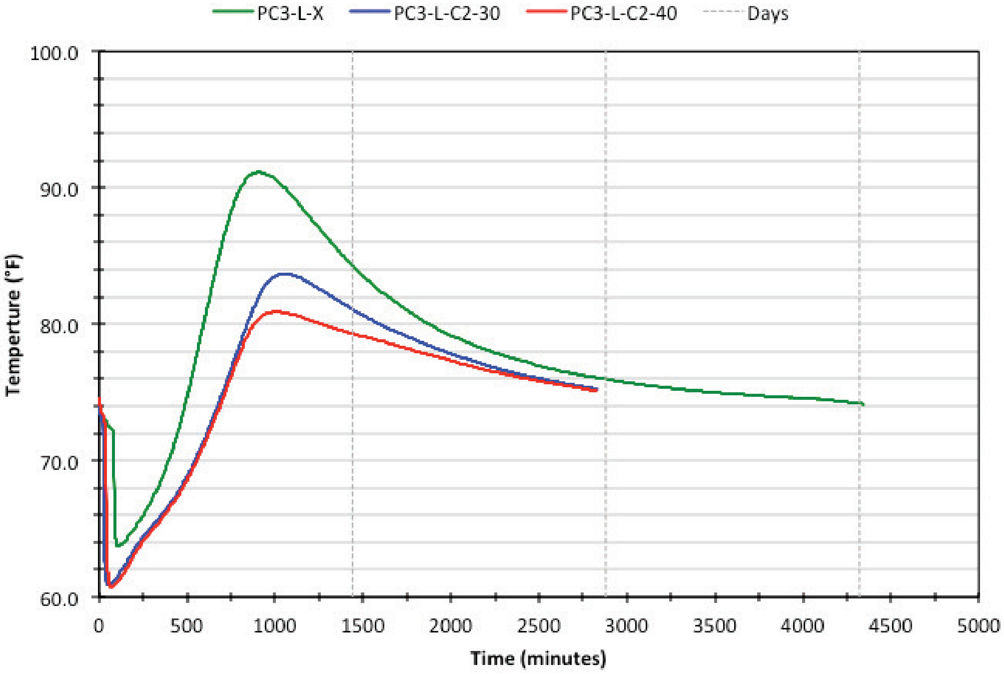


Figure C18. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC3 cement, fly ash C2 at 30 and 40%, and the quarried carbonate aggregate. The heat curve for PC3 cement, quarried carbonate aggregate, and no ash is included for comparison.

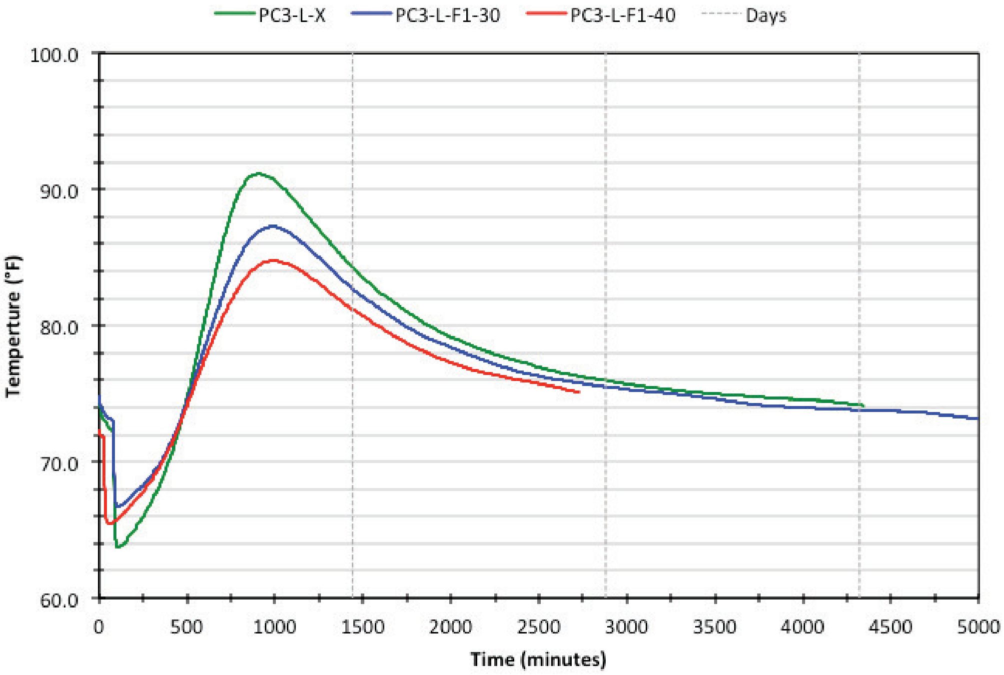


Figure C19. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC3 cement, fly ash F1 at 30 and 40%, and the quarried carbonate aggregate. The heat curve for PC3 cement, quarried carbonate aggregate, and no ash is included for comparison.

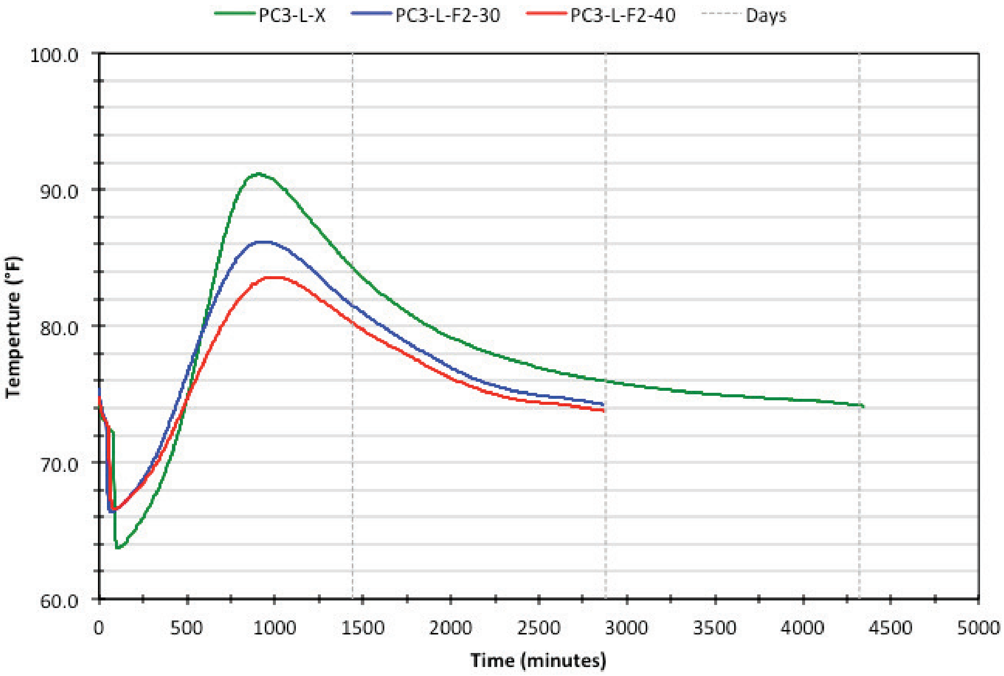


Figure C20. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC3 cement, fly ash F2 at 30 and 40%, and the quarried carbonate aggregate. The heat curve for PC3 cement, quarried carbonate aggregate, and no ash is included for comparison.

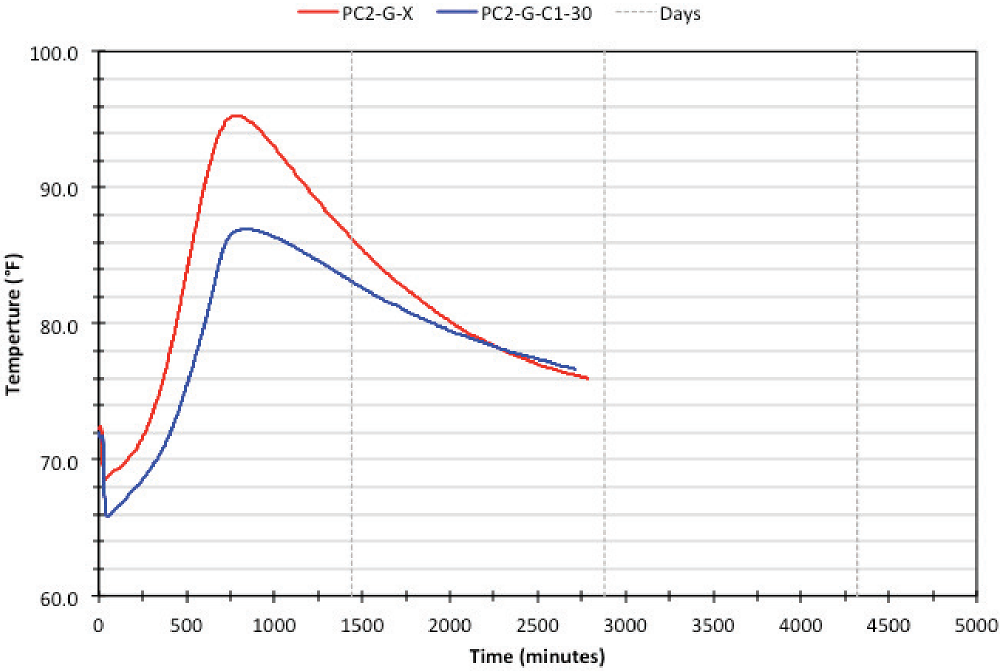


Figure C21. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC2 cement, fly ash C1 at 30%, and the glacial gravel aggregate. The heat curve for PC2 cement, glacial gravel aggregate, and no ash is included for comparison.

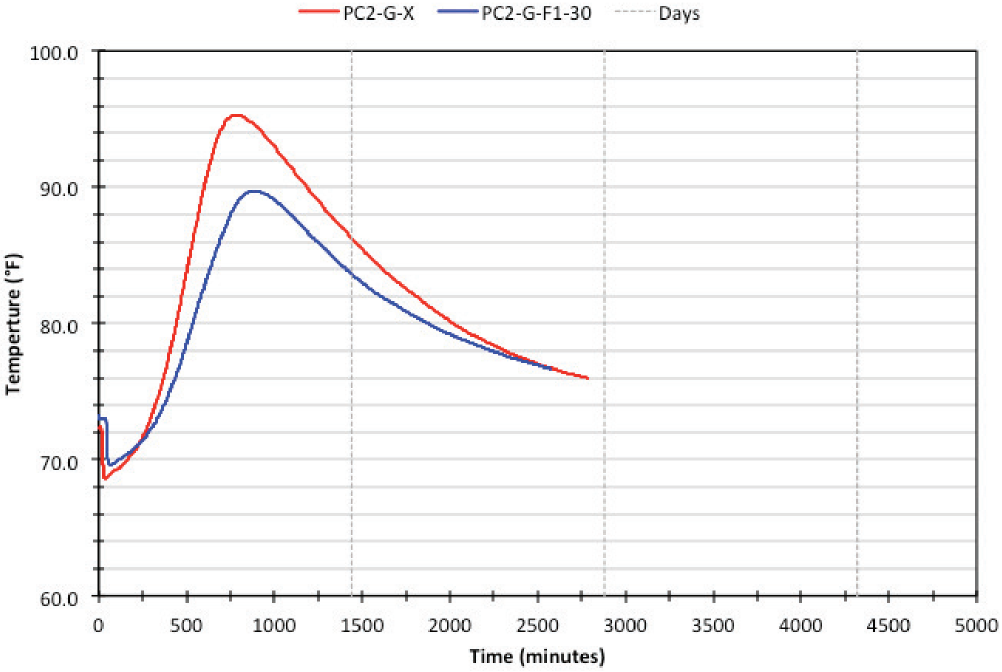


Figure C22. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC2 cement, fly ash F1 at 30%, and the glacial gravel aggregate. The heat curve for PC2 cement, glacial gravel aggregate, and no ash is included for comparison.

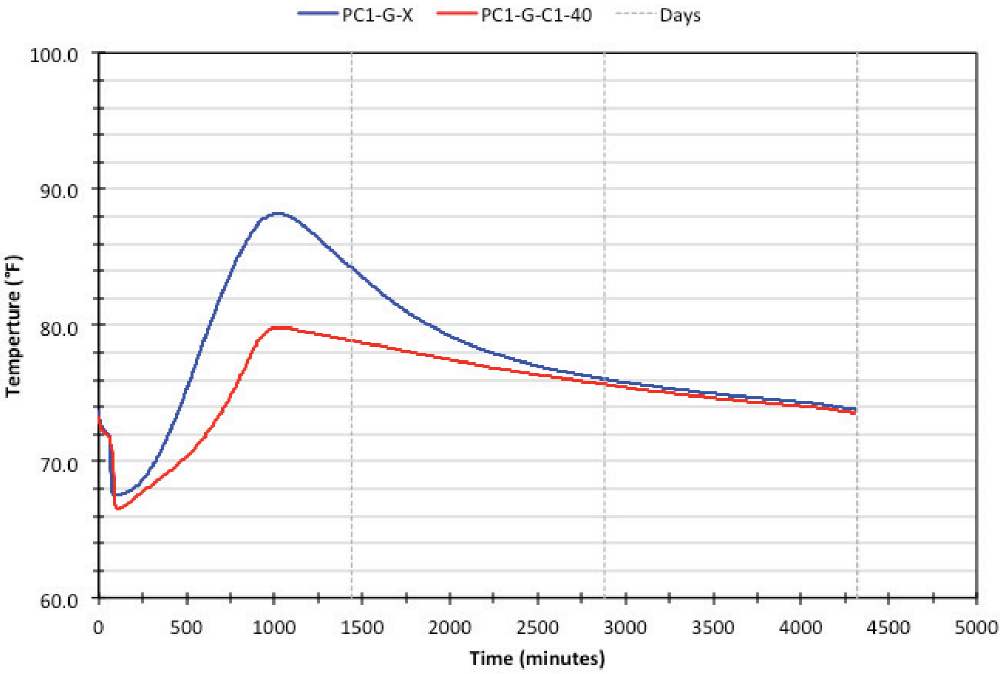


Figure C23. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC1 cement, fly ash C1 at 40%, and the glacial gravel aggregate. The heat curve for PC1 cement, glacial gravel aggregate, and no ash is included for comparison.

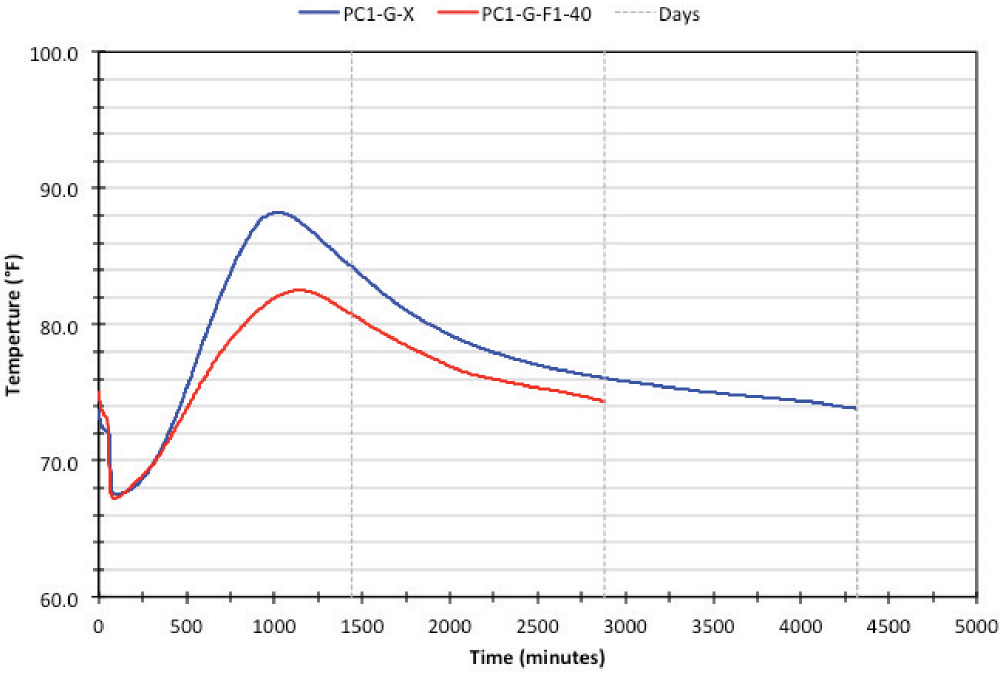


Figure C24. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC1 cement, fly ash F1 at 40%, and the glacial gravel aggregate. The heat curve for PC1 cement, glacial gravel aggregate, and no ash is included for comparison.

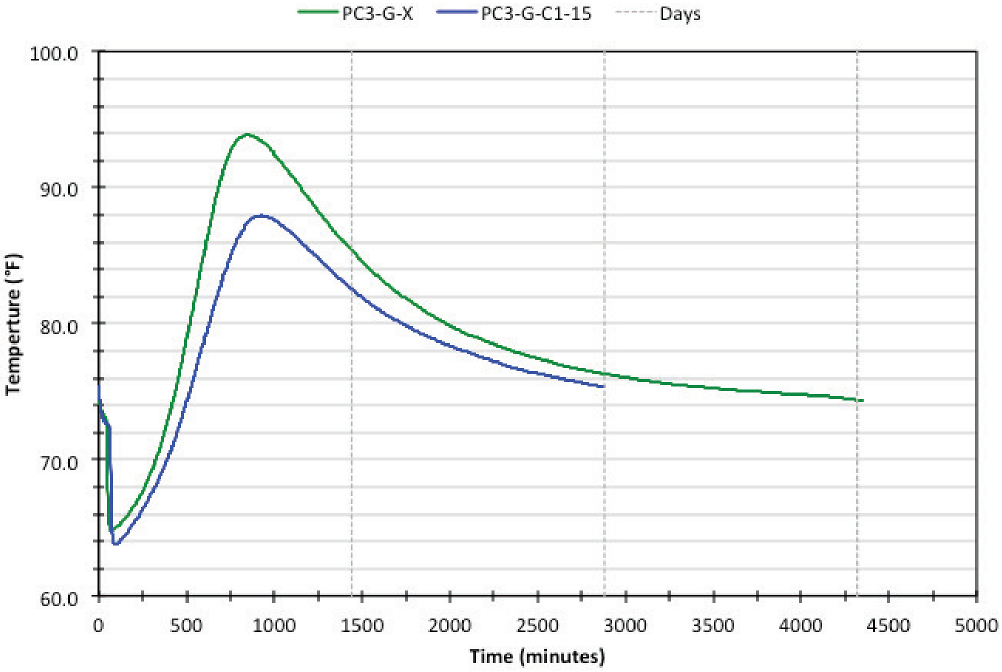


Figure C25. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC3 cement, fly ash C1 at 15%, and the glacial gravel aggregate. The heat curve for PC3 cement, glacial gravel aggregate, and no ash is included for comparison.

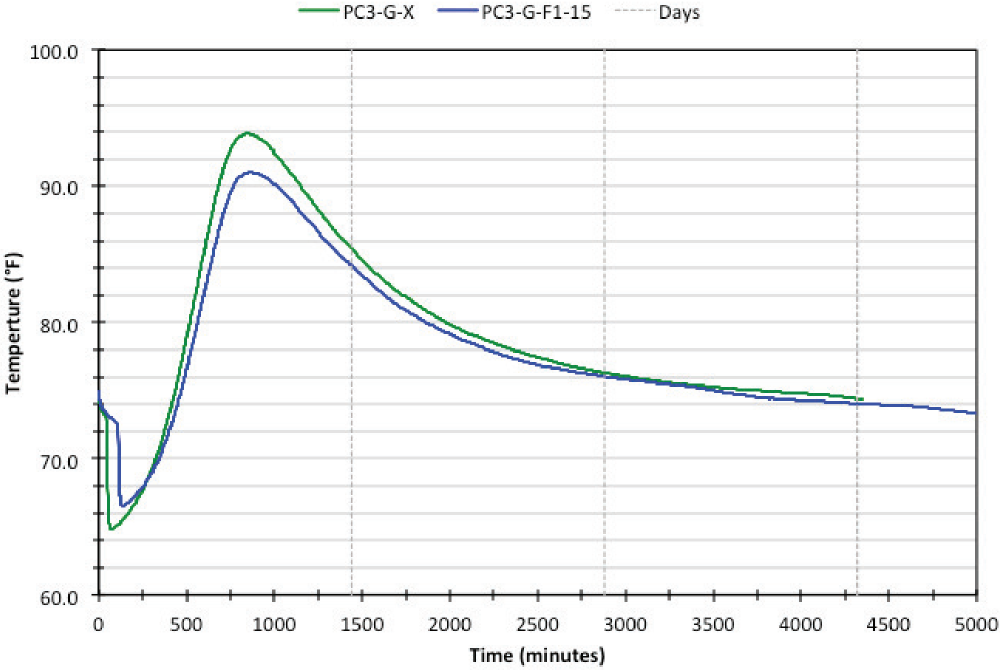


Figure C26. Heat of hydration curves determined by semi-adiabatic calorimetry for mixtures with PC3 cement, fly ash F1 at 15%, and the glacial gravel aggregate. The heat curve for PC3 cement, glacial gravel aggregate, and no ash is included for comparison.

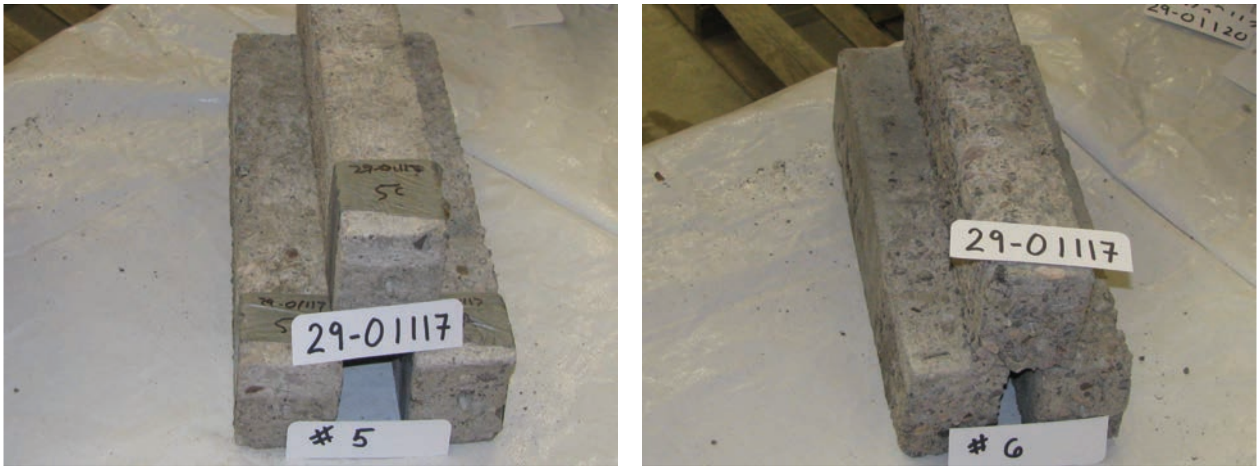
Appendix D - Images of Freeze-Thaw Prisms

(Mixture PC1-G-X)

(Mixture PC1-G-C1-40)

(Mixture PC1-G-C2-15)

(Mixture PC1-G-C2-40)



(Mixture PC1-G-F1-40)

(Mixture PC1-G-F2-15)

Figure D1. Images of freeze-thaw prisms after completion of AASHTO T 161 testing.

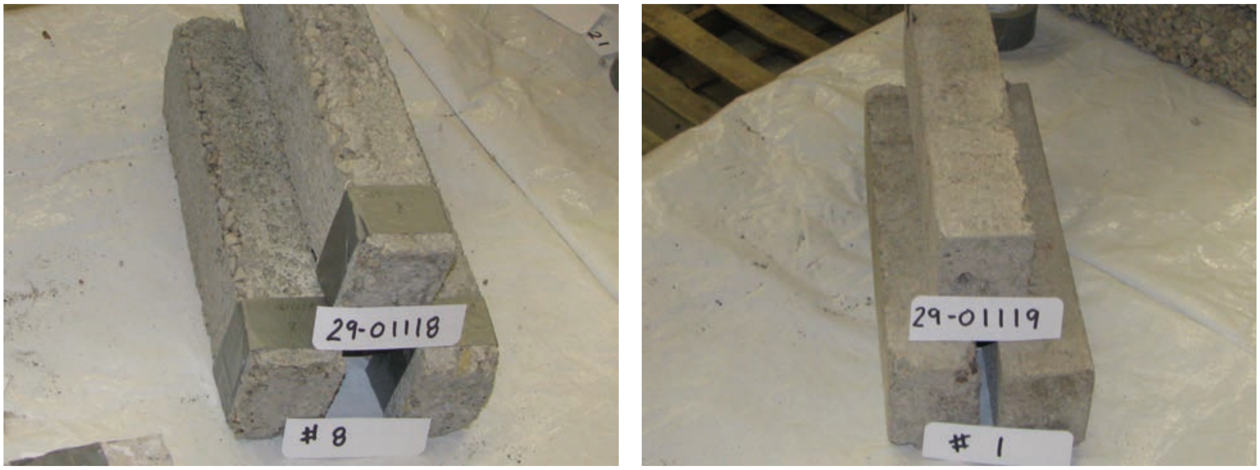
(Mixture PC1-G-F2-40) (Mixture PC1-L-X)

(Mixture PC1-L-C1-15) (Mixture PC1-L-C1-30)

(Mixture PC1-L-C2-15) (Mixture PC1-L-C2-30)

Figure D2. Images of freeze-thaw prisms after completion of AASHTO T 161 testing.

(Mixture PC1-L-F1-15) (Mixture PC1-L-F1-30)



(Mixture PC1-L-F2-15) (Mixture PC2-G-X)

(Mixture PC2-G-C1-30) (Mixture PC2-G-C2-30)

Figure D3. Images of freeze-thaw prisms after completion of AASHTO T 161 testing.

(Mixture PC2-G-C2-40) (Mixture PC2-G-F1-30)

(Mixture PC2-G-F2-30) (Mixture PC2-G-F2-40)

(Mixture PC2-L-X) (Mixture PC2-L-C1-15)

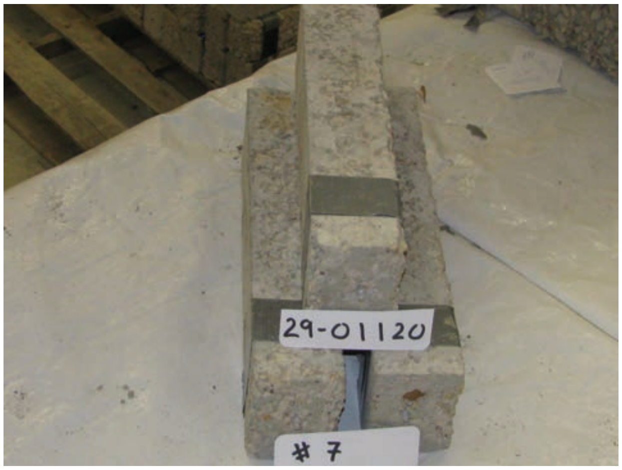
Figure D4. mages of freeze-thaw prisms after completion of AASHTO T 161 testing.

(Mixture PC2-L-C1-40)

(Mixture PC2-L-C2-15)

(Mixture PC2-L-C2-40)

(Mixture PC2-L-F1-15)



(Mixture PC2-L-F1-40)

(Mixture PC2-L-F2-15)

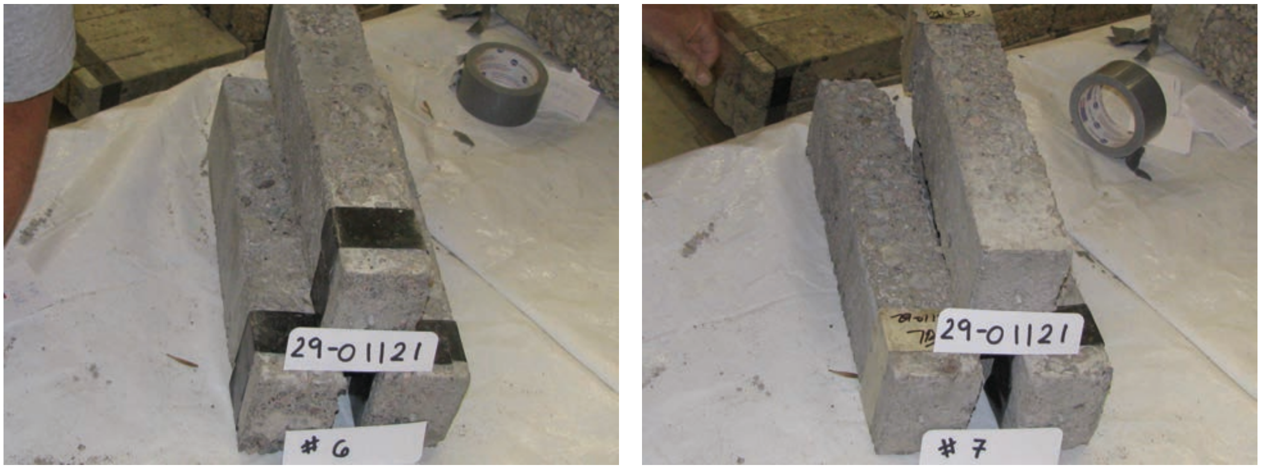
Figure D5. Images of freeze-thaw prisms after completion of AASHTO T 161 testing.

(Mixture PC2-L-F2-40) (Mixture PC3-G-X)

(Mixture PC3-G-C1-15) (Mixture PC3-G-C2-15)

(Mixture PC3-G-C2-30) (Mixture PC3-G-F1-15)

Figure D6. Images of freeze-thaw prisms after completion of AASHTO T 161 testing.



(Mixture PC3-G-F2-15) (Mixture PC3-G-F2-30)

Figure D7. Images of freeze-thaw prisms after completion of AASHTO T 161 testing.

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