

MIXTURE PROPORTIONING OPTIONS FOR IMPROVING HIGH VOLUME FLY ASH CONCRETES

By

Dale P. Bentz (corresponding author)
Chemical Engineer
Materials and Construction Research Division
National Institute of Standards and Technology
100 Bureau Drive, Stop 8615
Gaithersburg, MD 20899
(301) 975-5865 Voice
(301) 990-6891 Fax
dale.bentz@nist.gov

Chiara F. Ferraris
Physicist
Materials and Construction Research Division
National Institute of Standards and Technology
100 Bureau Drive, Stop 8615
Gaithersburg, MD 20899
(301) 975-6711 Voice
chiara.ferraris@nist.gov

Igor De la Varga (presenting author)
Ph.D. student
Purdue University
West Lafayette, IN 47907
idelavar@purdue.edu

Max A. Peltz
Engineering Technician
Materials and Construction Research Division
National Institute of Standards and Technology
100 Bureau Drive, Stop 8615
Gaithersburg, MD 20899
(301) 975-3175 Voice
max.peltz@nist.gov

John A. Winpigler
Engineering Technician
Materials and Construction Research Division
National Institute of Standards and Technology
100 Bureau Drive, Stop 8615
Gaithersburg, MD 20899
(301) 975-6710 Voice
john.winpigler@nist.gov

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ABSTRACT

High volume fly ash (HVFA) concretes are one component of creating a more sustainable infrastructure. By replacing 50 % or more of the portland cement with fly ash, a significant reduction is achieved in the carbon footprint of the in place concrete. While HVFA mixtures can be proportioned to produce equivalent long term performance as conventional (cement-only) mixtures, performance problems are often encountered at early ages, including low early-age strengths, long delays in finishing, and potentially greater susceptibility to curing conditions. In this paper, a variety of mixture proportioning options to mitigate these deficiencies are investigated within the framework of a proposed mixture proportioning methodology. Variables examined in laboratory studies include cement type, fly ash class, the provision of internal curing, and the addition of either calcium hydroxide or a rapid set cement to the binder.

Switching from a Type II/V to a Type III cement enhanced one-day compressive strengths by over 50 %. Using a Class C fly ash produced a mixture with a higher calcium-to-silicate ratio than a comparable Class F fly ash and increased the measured 7-d compressive strength. However, in this study, sulfate balance was a problem in the Class C HVFA mixtures, requiring 2 % additional gypsum to provide a proper sulfate balance. Internal curing was found to significantly reduce autogenous deformation by 50 % or more, with a concurrent 13 % decrease in compressive strength. Excessive retardations of 3 h to 4 h were observed in both mixtures with the Class C and the Class F fly ashes; powder additions of either a rapid set cement or calcium hydroxide were found to be effective in reducing this retardation (and setting time delays) in pastes and mortars.

INTRODUCTION

One of the defining characteristics of the concrete industry in the 21st century is a new emphasis on sustainability (1). High-volume fly ash (HVFA) mixtures are promoted as one potentially significant contributor to reducing the carbon footprint of in-place concrete, while concurrently increasing the utilization of a readily-available waste stream material (2). HVFA concrete mixtures, where fly ash replaces 50 % or more of the cement, would substantially reduce the CO₂ footprint of a concrete structure. While such mixtures can be proportioned to meet or even exceed the long term performance properties of conventional (cement-only) concretes (2, 3), short term performance deficiencies may limit their acceptance by the construction industry. Deficiencies include reduced early-age strength (requiring longer waiting times prior to form removal), unacceptable increases in setting time (delaying finishing operations and reducing crew efficiency), and an increased sensitivity to curing conditions (as the pozzolanic and hydraulic reactions generally occur at a much slower rate than conventional cement hydration).

This paper evaluates various strategies for mitigating these early-age deficiencies within the framework of a proposed mixture proportioning methodology, as shown in Figure 1. The influences of both cement type (II/V or III) and fly ash class (C or F) on early-age performance are characterized. Strategies are also evaluated for reducing the setting time delays commonly experienced with HVFA mixtures, based on the addition of either a rapid set cement (3) or calcium hydroxide powder. Finally, the incorporation of internal curing (4) via pre-wetted lightweight aggregates is evaluated for its influence on early-age properties including autogenous deformation and compressive strength.

MATERIALS AND EXPERIMENTAL PROCEDURES

The measured particle size distributions (PSDs) for the two cements, the two classes of fly ash, and the powder additions employed in this study are provided in Figure 2. Both a Type II/V and a Type III cement ground from the same clinker were employed; each has been optimized by the manufacturer with respect to sulfate level. Their detailed chemical compositions as provided by the manufacturer are listed in Table 1, and a variety of their early-age performance properties have been published recently (5). The Blaine finenesses of the Type II/V and Type III cements are 387 m²/kg and 613 m²/kg, respectively, as supplied by the manufacturer, and each has a density of 3250 kg/m³. A supply of a Class C fly ash (density of 2690 kg/m³ or 168 lb/ft³) was obtained from a concrete ready-mix producer and a Class F fly ash (density of 2100 kg/m³ or 131 lb/ft³) from a local fly ash producer. Detailed oxide compositions for the two fly ashes, as determined at a private testing laboratory, are included in Table 1 (6).

A rapid set cement (mainly a mixture of calcium sulfoaluminate, dicalcium silicate, and gypsum) was obtained from a commercial supplier. Calcium hydroxide and calcium sulfate dihydrate (gypsum, 98 % purity) were purchased from an international chemical company. The high range water reducing agent (HRWRA) was of the polycarboxylate type (43 % solids and a specific gravity of 1.08) and was obtained directly from a chemical admixture supplier. For the HVFA mortars with internal curing, a portion of the sand was replaced with pre-wetted lightweight aggregate (LWA) of a similar particle size distribution as the sand being replaced. The LWA sand had a saturated surface dry (SSD) density of 1700 kg/m³ and a 24 h absorption of 22 % per unit mass of dry aggregate.

Example mixture proportions for the HVFA mortars with 50 % replacement of fly ash for cement are provided in Table 2. The total sand (normal weight and lightweight) volume fraction was maintained constant at 54 % in all mixtures, with the normal weight sand consisting of a

blend of four commercial silica sands (density of 2610 kg/m^3). Mortars were prepared according to ASTM C 305 procedures (7). For each mixture, the HRWRA dosage (as indicated in Table 2) was adjusted to provide adequate workability to cast mortar cubes and corrugated tubes for measurement of autogenous deformation. In general, 60 % of the HRWRA mass was counted as water in proportioning to a constant water-to-cementitious materials by mass ratio (w/cm) of 0.3. Additionally, for comparison purposes, results for a $w/c=0.4$ control mortar without HRWRA were available from a previous study (5). For a subset of the mortars, equivalent paste mixtures were prepared in a high shear blender for the evaluation of rheology using a stress growth technique (8) and setting using needle penetration (ASTM C 191 (7)). Low temperature calorimetry measurements were conducted on these pastes stored under saturated conditions for various times, to assess the percolation state of their capillary porosity as a function of curing age (9).

Mortar characterization typically included measurements of isothermal calorimetry for the first 7 d (ASTM C 1702 (7)), semi-adiabatic calorimetry for 3 d (10), compressive strength (ASTM C 109 mortar cubes (7)), and autogenous deformation (ASTM C 1698 corrugated tubes (7)). Compressive strengths were assessed at the ages of 1 d, 7 d, 28 d, 56 d, 182 d, and 365 d on cubes that were demolded after 1 d and subsequently stored in water saturated with calcium hydroxide. Autogenous deformation was monitored for 56 d, with the sealed specimens being maintained at constant temperature conditions of $25 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$ in a walk-in environmental chamber.

RESULTS

Results will be presented in the context of the proposed mixture proportioning methodology of Figure 1.

Assuring Compatibility (Sulfate Balance)

Once potential fly ash and cement sources have been identified for a particular project, one of the first steps should be to determine whether they form a compatible blend. This can be readily examined using an isothermal calorimetry technique (ASTM C 1702 (7)), as further detailed in the ASTM C 1679 standard practice (7). For the materials selected for this study, considerable incompatibility was observed in the 50:50 mixture prepared with the Type II/V cement and the Class C fly ash. This problem with materials was first noted when the 1 d average compressive strength of mortar cubes with 50 % of the Class C fly ash was measured as only 870 psi (5.9 MPa) ± 10 psi (standard deviation for three cubes is reported) instead of the expected 2000 psi to 2500 psi. As demonstrated by the isothermal calorimetry curves in Figure 3, this incompatibility was identified as a sulfate balance issue (11, 12) that substantially reduced the magnitude of the primary hydration peak and produced a second peak only after 24 h of hydration. Subsequently, a 2 % addition of gypsum was found to provide a restoration to some semblance of “normal” hydration behavior as observed in Figure 3, with a larger primary hydration peak and a 2nd peak occurring as a shoulder off of this primary peak. This addition level was subsequently employed in all Class C fly ash mixtures investigated in this study.

Mitigating Excessive Retardation

As shown in Figure 4, based on further isothermal calorimetry results, HFVA mortars with either class of fly ash at the 50 % level exhibited considerable retardation in their hydration reactions. For the Class F fly ash, this was mainly due to the higher dosage of HRWRA (Table 2) required

to maintain adequate workability when using this less dense, larger particle size (Figure 1) fly ash, as verified previously by calorimetry measurements made on pastes with and without the HRWRA (6). While the Class C fly ash actually allowed for a reduction in the HRWRA dosage (as is often observed for fly ash), it caused considerable retardation by itself (Figure 3). It can be observed in Figure 4 that switching to a Type III cement produces a slight reduction in this retardation of perhaps 1 h for either fly ash, but clearly doesn't restore the heat release curve to that observed for the Type II/V cement-only mortar.

An extensive search was conducted to find powder additions with the potential to mitigate this excessive retardation in both fly ash mixtures (6, 13). While a variety of unsuccessful candidates were identified (cement kiln dust, limestone powder, aluminum trihydroxide powder, and silica fume), both calcium hydroxide (CH) and a rapid set cement indicated potential for restoring the setting times to those experienced by the Type II/V cement-only mortar. Initial calorimetric indications of this mitigation (6) were subsequently verified by rheological and setting time measurements (13). For pastes, the measured initial and final setting times are summarized in Table 3, from which it can be observed that either the CH or the rapid set cement additions at levels of 5 % to 10 % per unit mass of binder were able to substantially reduce the setting times for the mixtures based on both Class C and Class F fly ashes from the 8 h to 10 h range back to a range of 3 h to 6 h, in line with those of the control mixture.

The utilization of these powder additions as mitigation strategies in actual concrete mixtures would need to be further evaluated in terms of their influence on slump, unit weight (air content), and other fresh and hardened concrete properties. In this study, mortar mixtures with the rapid set cement were evaluated for compressive strength. In comparison to a mixture without the rapid set cement addition, the mortar based on a Class C fly ash with a 2 % addition of gypsum and 10 % of the rapid set cement provided higher strength values at ages of 7 d and beyond. However, for the Class F fly ash, a 5 % addition of the rapid set cement did not achieve strength equivalence to the comparable mortar without rapid set cement until an age of 56 d.

Increasing Early-Age Strength

While it is recognized that properly designed HVFA mixtures can obtain long term strengths that meet or exceed performance specifications, early-age strengths are generally significantly reduced relative to those of conventional concretes. For example, the mortar cube compressive strength results provided in Figure 5 indicate that HVFA mixtures with either a Class C or a Class F fly ash have 1 d strengths that are only approximately 30 % of that of a $w/c=0.3$ control mortar or 60 % of that of a $w/c=0.4$ control mortar. The $w/c=0.4$ mortar results have been included because of the generally accepted practice of reducing the w/cm for a HVFA mixture relative to a conventional concrete (2, 3). It can be seen that in the long term, the strengths of the HVFA mixtures do in fact approach those of the $w/c=0.3$ control, while actually meeting or exceeding those of the $w/c=0.4$ control at ages of 28 d and beyond. Specifically, after 365 d, the various mixtures with 50 % fly ash have achieved compressive strengths that are greater than 85 % of the value achieved by the $w/c=0.3$ control 100 % cement mixture, with all of the mortars in Figure 5 exceeding 14,500 psi (100 MPa) at 1 year of age.

Figure 5 indicates that switching from a Type II/V cement to a Type III cement increased the 1 d compressive strengths by about 60 %, so that the strengths basically matched those of the $w/c=0.4$ control mortar based on only cement. Thus, while a Type III cement may not be a viable option for mitigating excessive retardation and finishing delays in HVFA mixtures (Figure 4), it can provide a significant boost to 1 d strengths. In Figure 5, one can also observe

that while the Class F and Class C fly ash mortars have nominally equivalent strengths at 1 d, between 1 d and 7 d, the strength development occurring in the Class C fly ash mortars is superior to that in the Class F fly ash mixtures. The generally higher calcium oxide content (reflecting a higher calcium-to-silicate ratio) of the Class C fly ash implies that it can be both hydraulic (14) and pozzolanic, and potentially offer a greater contribution to early age reactions and strength development, as observed in this study. Beyond 1 d, the HVFA mortar mixture consistently exhibiting the highest strength values was that utilizing the Class C fly ash, the Type III cement, and a 2 % gypsum addition, which achieved a strength that was actually 97 % of that of the control (Type II/V cement, $w/c=0.3$) at 365 d.

Maintaining Saturation and Reducing Autogenous Shrinkage

Maintaining saturation of the capillary porosity of a hydrating cementitious binder is critical for maximizing hydration and for reducing autogenous shrinkage that accompanies self-desiccation. Self-desiccation occurs due to the ongoing chemical shrinkage resulting from the simple fact that the volume of the cement (and pozzolanic) hydration products is less than that of the reactants. After setting, this reduction in volume manifests itself in the creation of vapor-filled pores that generate capillary stresses within the microstructure, producing a measurable autogenous shrinkage and potentially contributing to early-age cracking. One method of maintaining saturation is via the incorporation of internal curing (IC), using pre-wetted lightweight aggregates as water-on-demand reservoirs (4). Water can also be provided by external curing (e.g., misting, ponding), but the effectiveness (travel distance) of this water is severely limited once the capillary porosity depercolates. For the blended cement pastes examined in this study, the following depercolation times have been determined using low temperature calorimetry (9): II/V cement, C ash, gypsum – 10 d; III cement, C ash, gypsum – 3 d; II/V cement, F ash – 21 d; and III cement, F ash – 17 d. Both the Class C fly ash and the utilization of a Type III cement are seen to reduce the curing time required to achieve depercolation of the capillary porosity, in line with their tendencies to increase early-age compressive strengths due to increased cement hydration and pozzolanic reactions. IC may prove beneficial in mixtures where this depercolation occurs at an early age, or for providing a long term water resource to support the more slowly developing pozzolanic reactions between fly ash and calcium hydroxide.

Internal curing efficiency can be assessed by measuring the autogenous deformation of mortars using sealed corrugated tubes, as described in the newly issued ASTM C 1698 standard test method (7). Figure 6 provides the measured autogenous deformations of sealed HVFA mortars with and without IC out to an age of 56 d. For both the Class C and Class F fly ashes, the incorporation of IC to provide an additional 0.08 mass units of water per unit mass of binder (cement, fly ash, and gypsum) significantly reduced the autogenous shrinkage, actually producing a measured expansion of about 200 microstrains at 56 d. Cusson has pointed out that even more important than the expansion/shrinkage produced at a given age is the difference between the maximum (expansion) deformation and the longer term values (15). With respect to this criterion, the HVFA mixtures with IC produced approximately 100 microstrains of net shrinkage, while those without IC exhibited net shrinkages of 280 microstrains to 400 microstrains. For the HVFA mortars examined in this study, this improvement in autogenous shrinkage provided by IC must be balanced against the observed reduction in mortar cube compressive strength, as the mixtures with IC produced 182 d compressive strengths that were at least 85 % of those of the corresponding mixtures without IC, but which still exceeded 12,800 psi (88 MPa). While the HVFA mortar cubes without IC were cured in saturated

limewater immediately after demolding at an age of 1 d, those with IC were cured under sealed conditions (to allow for the lightweight aggregates to release their IC water).

Additional Benefits of HVFA Mixtures

Early-age stresses in hardening concrete include contributions from both self-desiccation (capillary) stresses and thermal stresses. The latter are due to the heating (and subsequent cooling) of the concrete caused by the exothermic hydration and pozzolanic reactions superimposed on the diurnal temperature cycle. Because fly ashes are typically much less reactive than cement at early ages, they reduce the maximum temperature rise produced in (mass) HVFA concrete structures due to a simple dilution effect. This is exemplified by the semi-adiabatic calorimetry results for the mortars examined in this study, as shown in Figure 7. The temperature rise occurring in the HVFA mortars is only about ½ of that experienced in the cement-only mortars, which should result in structures that exhibit a lower propensity for early-age thermal cracking (16). It can also be observed in Figure 7 that the two mixtures with IC exhibited a slight reduction in retardation relative to those without IC, as exemplified by their earlier occurrence (by 3 h to 4 h) of a measurable temperature rise under semi-adiabatic conditions. Part of this reduction is surely due to the fact that these mixtures with IC required a lower dosage of the HRWRA than their non-IC counterparts (see Table 2).

Economic Considerations

For HVFA concretes, the bottom line for many ready-mix producers and contractors will be the cost of the materials. With this in mind, estimates of the cost of the various HVFA mortars on a mass basis are provided in Table 4, with the costs of each of the individual materials being obtained from discussions with two separate industry sources. For reference, the cost of a $w/c=0.4$ (cement only) mortar would be \$39.50 per ton. It is clear from Table 4 that the HVFA mixtures can be cost competitive. The \$7/ton premium for a Type III cement vs. a Type II/V cement is more than offset by the savings of incorporating 50 % fly ash into the HVFA mixtures. The HRWRA is a significant portion of the total materials costs and the Class C fly ash mixtures therefore offer some cost benefits by reducing the requisite HRWRA dosage, due to their lubricating effects within the mortar. It should be kept in mind that the calculations in Table 4 were performed on a mass basis. Since the specific gravity of fly ash is generally lower than that of cement, HVFA mixtures proportioned on a mass basis will have a slightly higher yield and cost savings on a unit volume basis (perhaps more relevant to a contractor's or ready-mix producer's bottom line) should therefore be even greater than those provided in Table 4.

Table 4 only presents initial materials costs and doesn't consider a complete life cycle analysis of the mixtures. The results presented here indicate that the HVFA mixtures will likely have a reduced propensity for early-age cracking, while the long term durability-related properties of properly cured fly ash concretes are generally superior to those of conventional concretes, with the possible exception of resistance to deicing salt scaling (3, 17-19). Thus, from a life cycle perspective, it is likely that the cost savings offered by HVFA concretes will be even greater than those indicated in Table 4. Additionally, in the future, HVFA mixtures may offer additional cost savings, depending on the value (if any) assigned to their reducing the CO₂ emissions accompanying the production of a unit volume of concrete.

CONCLUSIONS

This paper has presented a preliminary mixture proportioning methodology for high volume fly ash concretes. Based on the results, the following conclusions can be drawn:

- 1) Isothermal calorimetry is a valuable experimental technique for evaluating cement-fly ash blends for potential incompatibilities and for determining the effectiveness of mitigation strategies such as the addition of gypsum.
- 2) At 50 % fly ash, most HVFA mixtures will exhibit a significant retardation in setting time and delays in finishing. In this study, these delays were linked both to retardation inherently caused by the Class C fly ash and also to retardation caused by an increase in the required HRWRA dosage. Calcium hydroxide or rapid set cement powder additions were found to reduce these setting times back to those exhibited by the control cement-only mixture, for the particular cements and fly ashes investigated in this laboratory study.
- 3) Switching from a Type II/V (or I/II) cement to a Type III cement can provide a significant boost to 1 d compressive strengths, when needed to meet construction schedules. Additionally, HVFA mixtures based on a Class C fly ash should be expected to have a greater strength development between 1 d and 7 d than a comparable mixture employing a Class F fly ash.
- 4) Internal curing can be successfully employed with HVFA mixtures, dramatically reducing autogenous shrinkage, but with a concurrent slight decrease in compressive strength for the materials employed in this study. Additionally, IC will generally significantly increase the materials costs of the HVFA mixtures.
- 5) From an economic standpoint, HVFA mixtures are cost competitive from a materials first cost basis. When a life cycle analysis is considered and/or if carbon trading becomes a reality worldwide, the cost savings of HVFA mixtures may be even greater.

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TABLE 1 Oxide Compositions of Class C and Class F Fly Ashes and Portland Cements

Component	Class C fly ash (mass fraction)	Class F fly ash (mass fraction)	Type II/V cement (mass fraction)	Type III cement (mass fraction)
SiO ₂	0.3838	0.5973	0.211	0.207
Al ₂ O ₃	0.1872	0.3018	0.045	0.045
Fe ₂ O ₃	0.0506	0.0280	0.041	0.041
CaO	0.2463	0.0073	0.649	0.648
MgO	0.0508	0.0083	0.012	0.012
SO ₃	0.0137	0.0002	0.025	0.028
Na ₂ O	0.0171	0.0024		
K ₂ O	0.0056	0.0242		
TiO ₂	0.0148	0.0160		
P ₂ O ₅	0.0124	0.0008		
Mn ₂ O ₃	0.0002	0.0002		
SrO	0.0037	0.0005		
Cr ₂ O ₃	<0.0001	0.0003		
ZnO	<0.0001	<0.0001		
BaO	0.0094	0.0012		
Loss on ignition	0.0026	0.0079	0.013	0.013

TABLE 2 Example Mixture Proportions for HVFA Mortars

Component	II/V control	II/V – F	II/V - C	III - F	III - C	III-F with IC
Type II/V cement	1250 g	625 g	625 g			
Type III cement				625 g	625 g	625 g
Class C fly ash			625 g		625 g	
Class F fly ash		625 g		625 g		625 g
Gypsum			25.5 g		25.5 g	
Sand	2375 g	2695.6 g	2524.6 g	2698 g	2553.1 g	1779.6 g
Water	370 g	370 g	380.2 g	367.5 g	377.7 g	369 g
HRWRA	8.3 g	10.8 g	4.2 g	12.5 g	8.3 g	10 g
LWA (SSD)						598.2 g

TABLE 3 Setting Times for the Paste Mixtures (13)

Paste mixture	Vicat initial set (h) ^a	Vicat final set (h)
II/V cement		
0.67 % HRWRA	5.1 h	5.9 h
50 % C ash, 2 % gypsum, 0.33 % HRWRA	8.2 h	8.8 h
50 % C ash, 2 % gypsum, 5 % CH, 0.33 % HRWRA	5.3 h	6.0 h
50 % C ash, 2 % gypsum, 10 % rapid set cement, 0.33 % HRWRA	3.1 h	4.5 h
50 % F ash, 0.87 % HRWRA	8.6 h	10.2 h
50 % F ash, 5 % CH, 0.87 % HRWRA	5.2 h	5.9 h
50 % F ash, 5 % rapid set cement, 0.87 % HRWRA	3.3 h	4.5 h

^a Per the ASTM C191 standard test method (7), the single laboratory precisions are listed as 12 min and 20 min for initial and final times of setting, respectively.

TABLE 4 Estimated Materials Costs for HVFA Mixtures

Material	\$/ton	II/V cement	II/V F ash	II/V C ash	III F ash	III C ash	III F ash IC	III C ash IC
II/V cement	\$110	\$34.35	\$15.84	\$16.46				
III cement	\$117				\$16.90	\$17.35	\$18.25	\$18.82
Fly ash	\$40		\$5.76	\$5.99	\$5.78	\$5.93	\$6.24	\$6.43
Gypsum	\$80			\$0.49		\$0.48		\$0.53
HRWRA	\$2000	\$4.17	\$5.00	\$2.00	\$5.78	\$3.95	\$4.99	\$2.15
Sand	\$15	\$8.90	\$9.35	\$9.07	\$9.35	\$9.09	\$6.66	\$6.24
LWA	\$100						\$12.43	\$12.88
Water	\$0.50	\$0.05	\$0.04	\$0.04	\$0.04	\$0.04	\$0.06	\$0.06
Total (\$/ton)		\$47.46	\$35.99	\$33.92	\$37.84	\$36.85	\$48.64	\$47.10

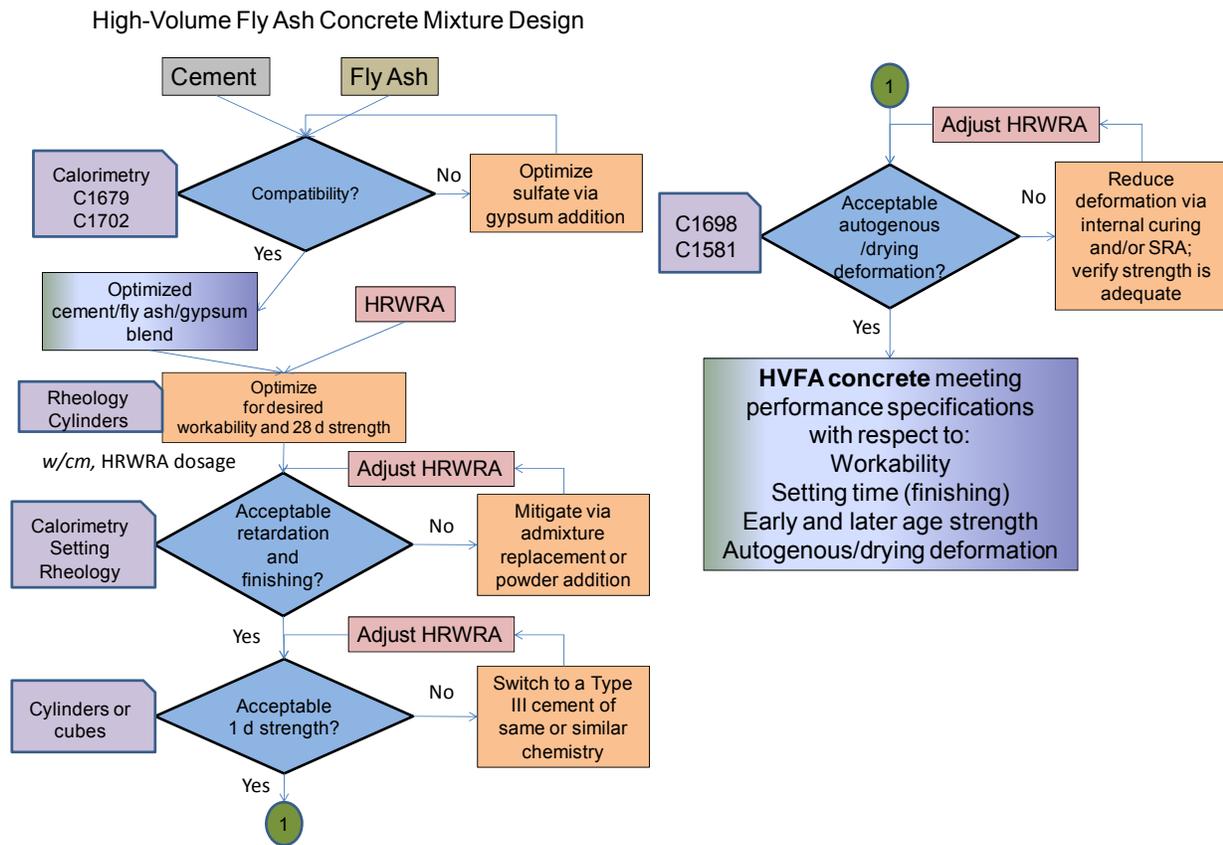


FIGURE 1 Proposed Mixture Proportioning Methodology for HVFA Concretes.

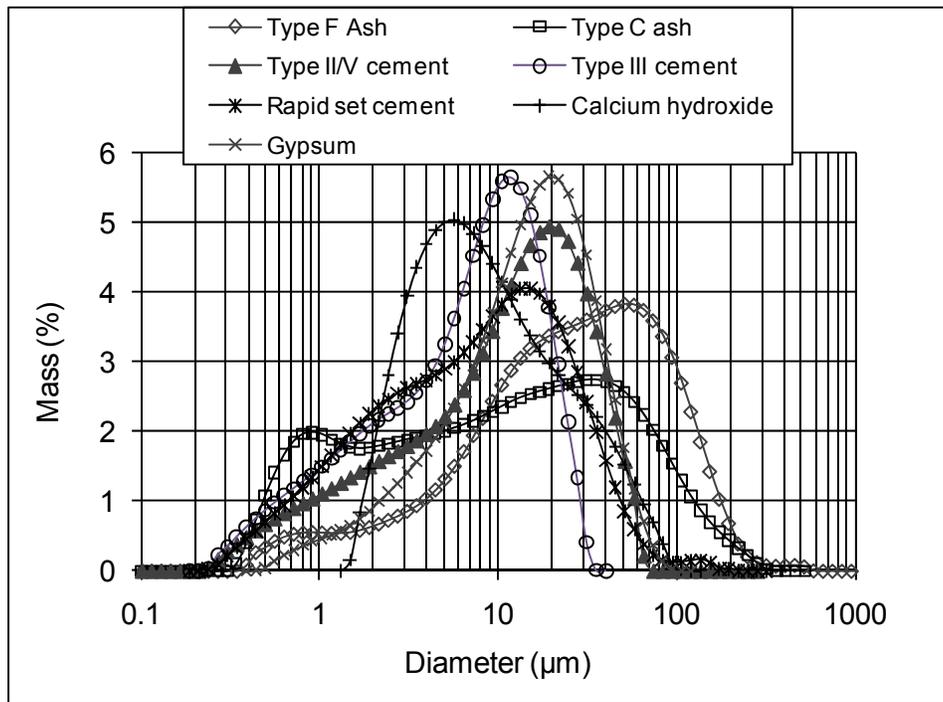


FIGURE 2 Measured particle size distributions for the powder materials employed in this study.

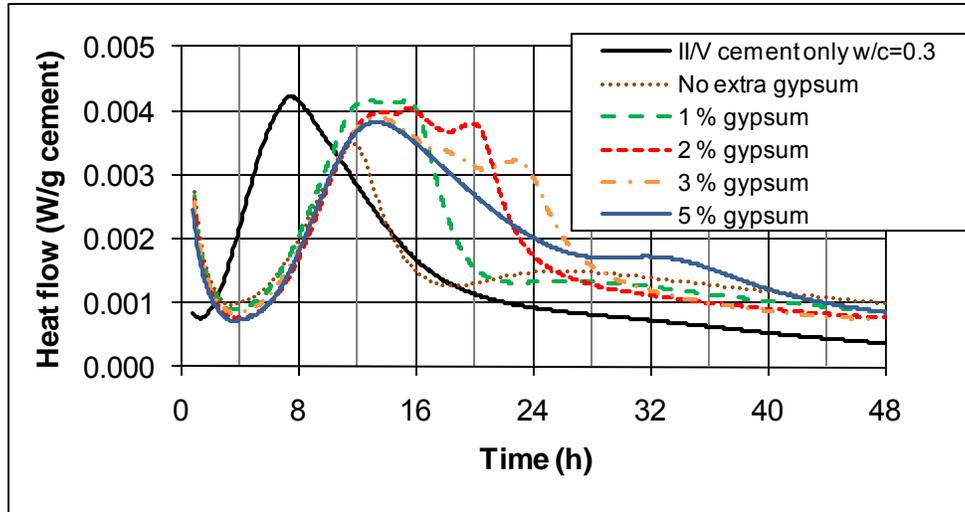


FIGURE 3 Isothermal calorimetry results normalized per gram of cement for Type II/V cement - Class C fly ash (50:50) pastes with various addition levels of gypsum.

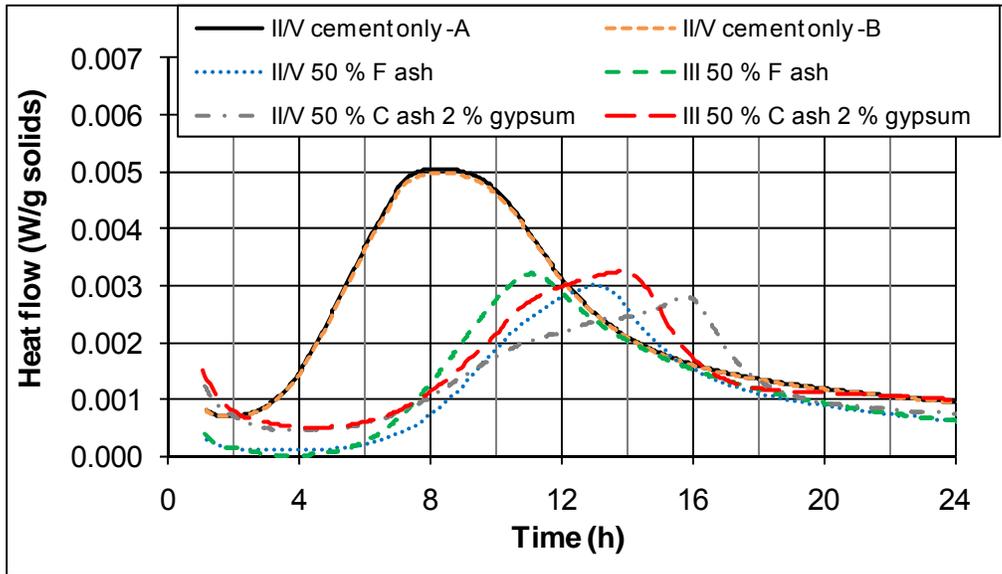


FIGURE 4 Isothermal calorimetry results normalized per gram of binder for mortars with and without 50 % fly ash. Two replicates are shown for the cement-only mortar to provide an indication of variability in the technique.

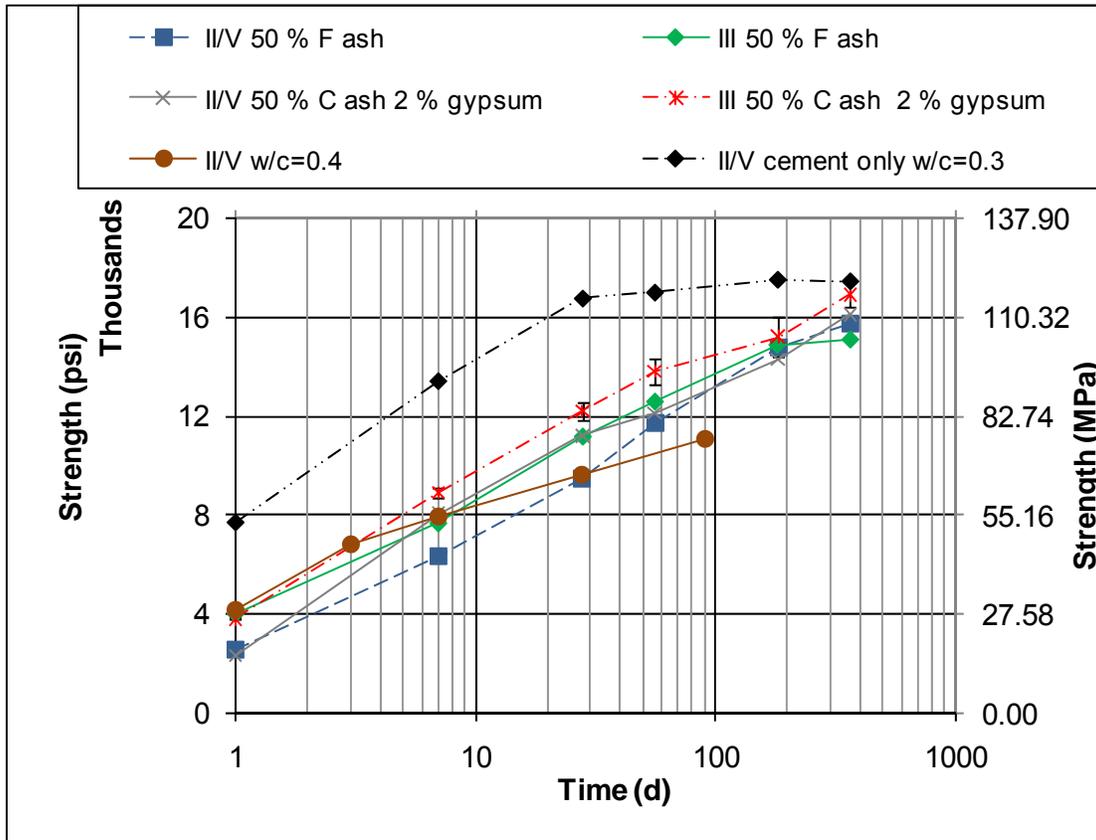


FIGURE 5 Measured mortar cube compressive strengths. Error bars (one standard deviation between three specimens) are provided for the Class C ash – III 2 % gypsum data to provide an indication of variability.

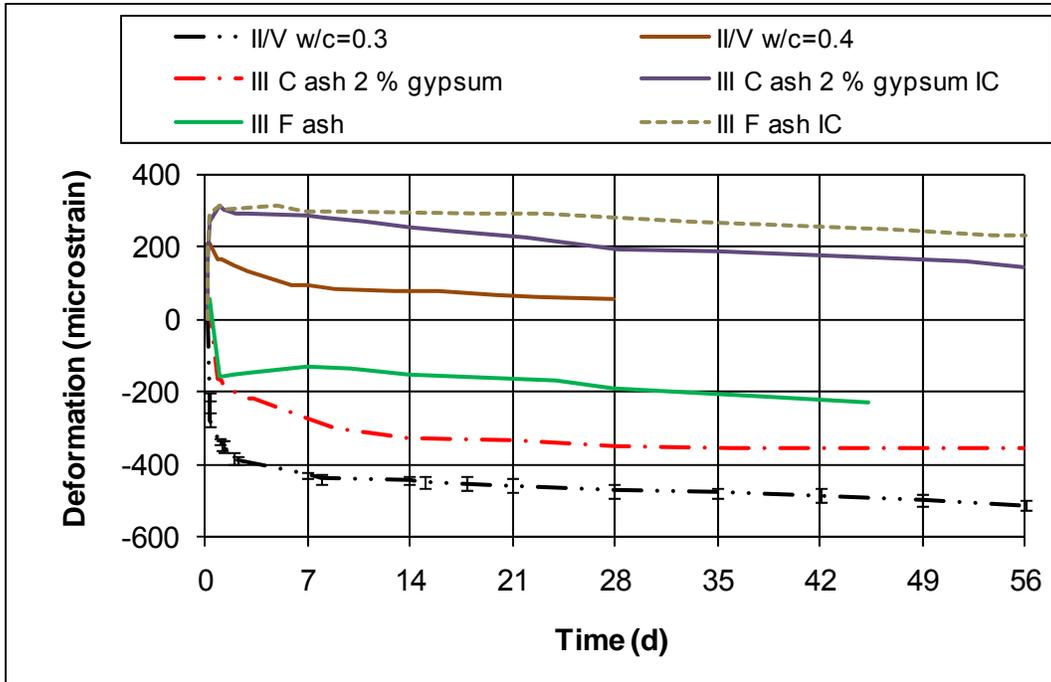


FIGURE 6 Measured autogenous deformation for sealed mortar specimens. Error bars (one standard deviation between three specimens) are provided for the II/V $w/c=0.3$ data to provide an indication of variability.

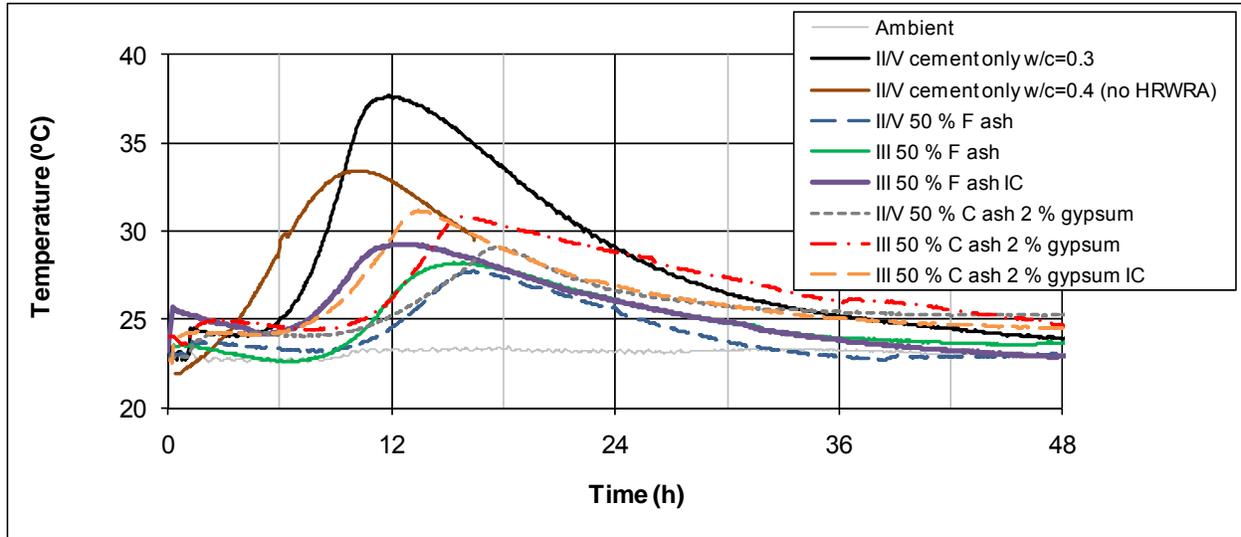


FIGURE 7 Measured semi-adiabatic temperature vs. time for HVFA mortar mixtures.