

Enhancing High Volume Fly Ash Concretes Using Fine Limestone Powder

by Jussara Tanesi, Dale Bentz, and Ahmad Ardani

Synopsis: One of the primary approaches to producing more sustainable concretes consists of replacing 50 % or more of the portland cement in a conventional concrete with fly ash, producing a so-called high volume fly ash (HVFA) concrete. While these mixtures typically perform admirably in the long term, they sometimes suffer from early-age performance issues including binder/admixture incompatibilities, delayed setting times, low early-age strengths, and a heightened sensitivity to curing conditions. Recent investigations have indicated that the replacement of a portion of the fly ash in these concrete mixtures by a suitably fine limestone powder can mitigate these early-age problems. The current study investigates the production of concrete mixtures where either 40 % or 60 % of the portland cement is replaced by fly ash (Class C or Class F) and limestone powder, on a volumetric basis. The mixtures are characterized based on measurement of their fresh properties, heat release, setting times, strength development, rapid chloride penetrability metrics and surface resistivity. The limestone powder not only accelerates the early age reactions of the cement and fly ash, but also provides significant benefits at ages of 28 d and beyond for both mechanical and transport properties.

Keywords: Constructability; fine limestone powder; high volume fly ash concrete; setting; sustainability.

Biography:

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INTRODUCTION

One challenge facing the transportation industry in the 21st century is the development of a more sustainable infrastructure, without sacrificing constructability (Cost, 2011). For example, concrete mixtures with a lower portland cement factor, such as high volume fly ash (HVFA) mixtures, often suffer from problems at early ages, including incompatibilities, unacceptable delays in setting times, insufficient early-age strength, and an increased sensitivity to curing conditions (Bentz *et al.* 2010). Due to these issues, the replacement level of fly ash for cement is often directly limited in construction specifications and in some cases, state Departments of Transportation (DOTs) prohibit the utilization of fly ash mixtures during a portion of their construction season, typically the winter months, or limit its use to lower levels of replacements. Clearly, these issues must be addressed to successfully foster an increase in the utilization of HVFA concrete mixtures throughout the US.

Early age cement hydration and pozzolanic reactions can be accelerated by fine filler particles, with limestone being superior to both silica fume and alumina particles (Kadri *et al.* 2009). Limestone additions may be particularly beneficial in systems with supplementary cementitious materials such as those containing fly ash (De Weerd *et al.* 2011a, Bentz *et al.* 2012, Cost and Bohme 2012) or slag (Menendez *et al.* 2003, Mounanga *et al.* 2011, Cost and Bohme 2012). In the current study, the addition of a fine limestone with a median particle diameter of 0.7 μm to replace a portion of the fly ash in HVFA concretes with either a 40 % or a 60 % cement replacement on a volumetric basis is investigated. While previous studies (De Weerd *et al.* 2011a, Bentz *et al.* 2012, Gurney *et al.* 2012) have focused mainly on heat release, setting times, and compressive strength development, here, the concrete mixtures will be characterized with respect to a wide variety of fresh and hardened properties, including transport characteristics such as surface resistivity and rapid chloride penetrability (RCPT).

RESEARCH SIGNIFICANCE

A variety of mitigation strategies have been developed for offsetting the early-age deficiencies of HVFA concrete mixtures, including the use of chemical admixtures (Cost 2011), switching to a (finer) Type III cement (Bentz *et al.* 2010), and lowering the water-to-cementitious materials ratio (w/cm) by increasing the cement content of the mixture (De la Varga *et al.* 2012). From an economic and environmental viewpoint, however, each of these options generally lacks making a positive contribution to the sustainability of an HVFA mixture. Readily available fine limestone powders generally cost substantially less and have lower CO₂ and energy footprints than the cement being replaced in a mixture, and may therefore potentially offer both performance and sustainability benefits. Quantification of these benefits requires a side-by-side comparison of 100 % ordinary portland cement mixtures to HVFA mixtures with and without the fine limestone additions. Based on such a data set, the perceived advantages of these ternary blends can be verified, paving the way for their wide spread introduction into practice by the construction industry.

MATERIALS AND EXPERIMENTAL PROCEDURES

A Type I/II ordinary portland cement was obtained from a local producer for this study. The cement has a reported Blaine fineness of 373 m²/kg and a calculated Bogue phase composition of 52.6% C₃S, 16.9 % C₂S, 6.9 % C₃A, and 10.4 % C₄AF, with a reported limestone content of 2.9 %, all mass fractions. Its density was measured to be 3270 kg/m³. Both a Class C and a Class F fly ash were used individually in the various concrete mixtures. Their chemical compositions, measured densities, and particle size characteristics are provided in Table 1. The Class C fly ash had a median diameter similar to that of the cement, while the Class F fly ash was coarser with a larger median diameter than the cement. Their CaO percentages, 24.63 % and 0.73 % by mass, respectively, provide a reasonable representation of the expected extremes in these values for the fly ashes commonly available in the US. The Class C fly ash is hydraulic, and a paste that is composed of only water and this fly ash will flash set just a few minutes after mixing, accompanied by the liberation of a significant amount of heat. This fly ash has been observed to produce considerable delays in setting times when used with a variety of portland cements in the past (Bentz *et al.* 2010, Gurney *et al.* 2012, De la Varga *et al.* 2012, Tanesi *et al.* 2012). Based on previous research (Gurney *et al.* 2012), a fine limestone powder with a median particle diameter of 0.7 μm was used in the ternary blends. It has a reported density of 2710 kg/m³ and a reported MgCO₃ content of 1 % by mass.

Table 1 – Oxide Composition Percent by Mass and Physical Characteristics of the Class C and Class F Fly Ashes

Property	Class C Fly Ash	Class F Fly Ash
SiO ₂ (%)	38.38	59.73
Al ₂ O ₃ (%)	18.72	30.18
Fe ₂ O ₃ (%)	5.06	2.80
CaO (%)	24.63	0.73
MgO (%)	5.08	0.83
SO ₃ (%)	1.37	0.02
Na ₂ O (%)	1.71	0.24
K ₂ O (%)	0.56	2.42
Loss on ignition (%)	0.26	0.79
Density	2630 kg/m ³	2160 kg/m ³
d (10 %)	0.85 μm	3.23 μm
d (50 %)	10.30 μm	25.34 μm
d (90 %)	69.37 μm	99.06 μm

Because of the significant differences in the specific gravities of the four powders, replacing cement and proportioning on a mass basis would produce mixtures with different initial porosities, unit weights, and yields. Thus, to enable the fairest comparison among mixtures and to evaluate the influence of the fine limestone powder additions separately from any changes in initial mixture porosity or volumetric paste content, all of the concrete mixtures were designed to maintain **constant volume fractions** of water, powders, coarse aggregate, and fine aggregate based on a plain mixture with 335 kg/m³ (564 lb/yd³) of portland cement and a water-to-cement ratio by mass (*w/c*) of 0.4. All cement replacements were made on a volumetric basis, based on the measured specific gravities of the cement, fly ashes, and limestone powder. The high-range water-reducing (HRWR) admixture dosage was adjusted in order to produce mixtures with at least a 25 mm (1 in) slump and the dosages were kept as low as possible. Different dosages of HRWR were used in the plain mixture, Class F mixtures and Class C mixtures, but the dosage was kept constant for all the mixtures with the same fly ash type. No air entraining admixture was used. The coarse aggregate was a gravel having a 19 mm (¾ in) nominal maximum size, a specific gravity of 2.57 and a water absorption of 1.77 %, while the fine aggregate was a natural sand having a specific gravity of 2.61, a water absorption of 1.07 % and a fineness modulus of 2.82.

Table 2 shows the resulting concrete mixture proportions; cement, fly ash, and limestone powder are all considered as cementitious materials in computing the water-to-cementitious materials ratio (w/cm) on a mass basis. In the mixture IDs, PC represents the plain mixture with 100 % ordinary portland cement. For the other 8 mixtures, the first number indicates the percentage of fly ash (as relates to the total cementitious volume), followed by a letter that represents the class of fly ash used (either Class F or Class C). If the mixtures contained limestone powder, a second number indicates the volumetric percentage of limestone present (either 10 % or 15 % of the total cementitious volume), followed by the letter L. The 10 % and 15 % limestone levels were selected based on the results of a previous study (Gurney *et al.*, 2012).

Figure 1 shows the composite particle size distribution (PSD) of the cementitious portion of each of the nine mixtures. PSDs were determined using a laser diffraction technique, with the powders first dispersed in isopropanol.

Table 2 – Concrete mixture proportions

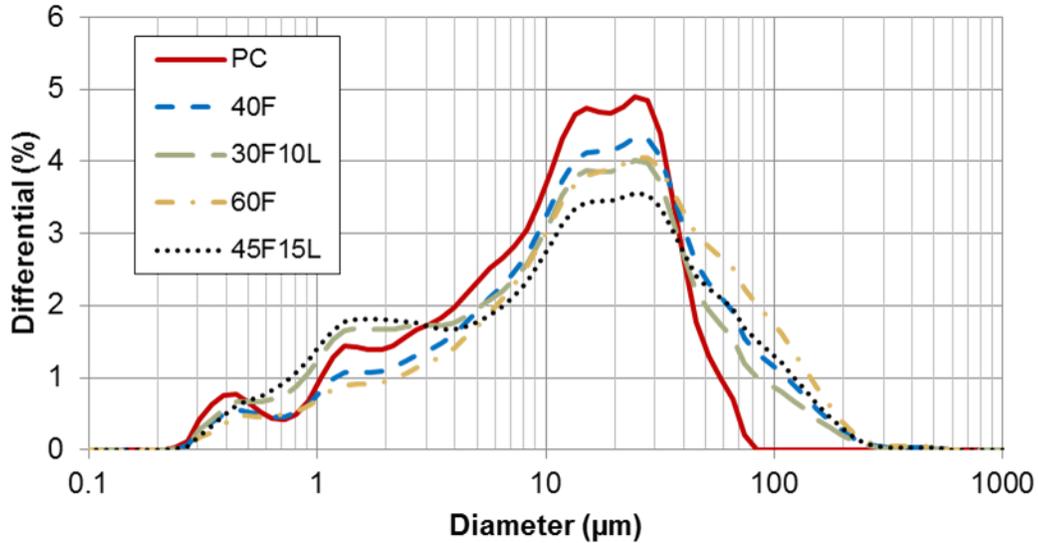
Mix ID	Cementitious (kg/m ³) [(lb/yd ³)]	Type I/II cement (kg/m ³) [(lb/yd ³)]	Class F fly ash (kg/m ³) [(lb/yd ³)]	Class C fly ash (kg/m ³) [(lb/yd ³)]	Limestone 0.7 μm (kg/m ³) [(lb/yd ³)]	Coarse aggreg. (kg/m ³) [(lb/yd ³)]	Fine aggreg. (kg/m ³) [(lb/yd ³)]	Water content (kg/m ³) [(lb/yd ³)]	w/cm	HRWR (fl oz/cwt)
PC	335 [564]	335 [564]				1038 [1750]	857 [1444]	131 [221]	0.40	7.7
40F	291 [491]	201 [338]	91 [153]			1038 [1750]	857 [1444]	131 [221]	0.46	3.8
30F10L	296 [499]	201 [338]	68 [114]		28 [47]	1038 [1750]	857 [1444]	131 [221]	0.45	3.8
40C	310 [522]	201 [338]		109 [183]		1038 [1750]	857 [1444]	131 [221]	0.43	3.0
30C10L	310 [523]	201 [338]		82 [138]	28 [47]	1038 [1750]	857 [1444]	131 [221]	0.43	3.0
60F	269 [454]	134 [226]	136 [229]			1038 [1750]	857 [1444]	131 [221]	0.50	3.8
45F15L	277 [467]	134 [226]	102 [172]		41.5 [70]	1038 [1750]	857 [1444]	131 [221]	0.49	3.8
60C	297 [501]	134 [226]		163 [275]		1038 [1750]	857 [1444]	131 [221]	0.45	3.0
45C15L	298 [502]	134 [226]		122 [206]	41.5 [70]	1038 [1750]	857 [1444]	131 [221]	0.45	3.0

Mixtures were prepared and cast according to ASTM C192. Slump (ASTM C143), air content (ASTM C231), unit weight (ASTM C138) and setting time (ASTM C403) tests were carried out. The ASTM C403 test method reports single-operator coefficients of variation for times of initial and final setting of 7.1 % and 4.7 %, respectively. Semi-adiabatic calorimetry was performed on 101.6 mm x 203.2 mm (4 in. x 8 in.) cylindrical concrete specimens for a period of 3 d, using a commercially available calorimeter. Two replicate specimens were evaluated for each mixture, with a maximum difference in temperature between two replicate specimens from the same mixture measured as 1.1 °C.

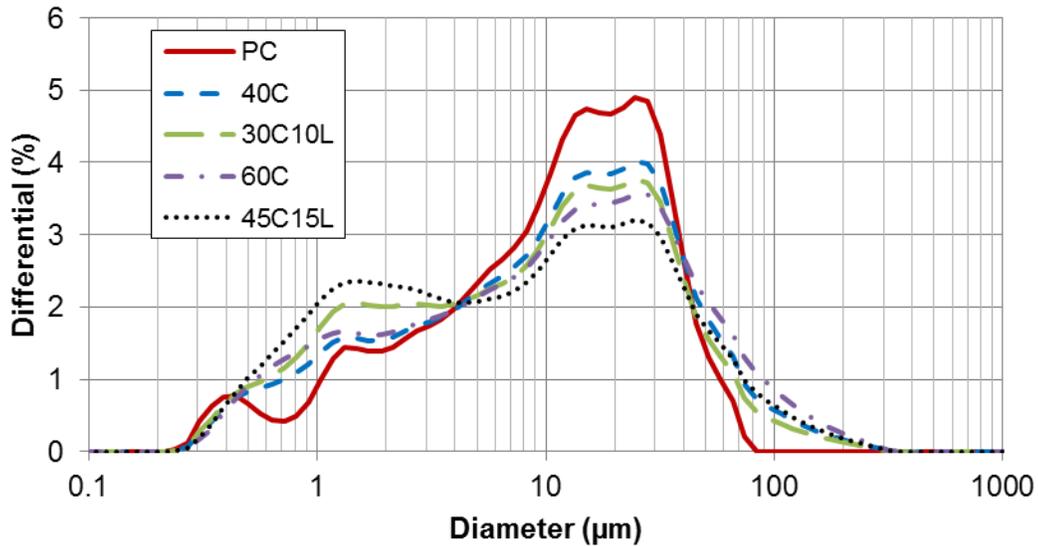
Isothermal calorimetry (ASTM C1679 and ASTM C1702) was conducted for 7 d on mortar that was wet sieved from the concrete according to ASTM C172. Two specimens were tested per mixture and the results were averaged. Since the samples were mixed, placed in the ampoules and then inserted in the calorimeter, the initial peak that occurs when water contacts cement was not examined in this study. In order to be able to normalize the isothermal calorimetry by the water volume of each specimen, the mortar water content was determined according to AASHTO T318.

Compressive strength was determined according to ASTM C39, using unbonded caps, at ages of 1 d, 3 d, 7 d, and 28 d. The transport properties of the mixtures were evaluated at 56 d through surface resistivity tests

(AASHTO TP95) and chloride penetrability tests (ASTM C1202). These ages were chosen to better represent the current specifications; most of the specifications use a maximum of 28 d strength and a 56 d RCPT. Specimens were protected from moisture loss in their molds for the first 24 h, then demolded and cured in lime water until their testing age.



(a)



(b)

Figure 1 – Composite particle size distributions of cementitious materials: a) plain mixtures and mixtures containing Class F fly ash; b) plain mixture and mixtures containing Class C fly ash.

RESULTS AND DISCUSSION

Table 3 shows a summary of the fresh properties test results. As it can be observed, the limestone powder did not impact the slump of the mixtures containing class F fly ash, but decreased the slump of the Class C fly ash mixtures considerably, especially when the mixtures with only 40 % of cement are compared (60C and 45C15L).

Because all mixtures were prepared using (constant) volumetric proportioning, there is minimal variation in their measured unit weight and of course, any variation in unit weight between mixtures should be solely due to their variable air contents.

Table 3 – Fresh properties test results

	Slump (cm) [(in)]	Air (%)	Unit Weight (kg/m³) [(lb/ft³)]	Initial Setting Time (min)	Final Setting Time (min)
PC	3.8 [1.5]	4.0	2311 [144.3]	224	312
40F	1.9 [0.75]	2.6	2323 [145.0]	285	407
30F10L	2.5 [1.0]	3.0	2305 [143.9]	202	307
40C	5.1 [2.0]	3.0	2318 [144.7]	345	473
30C10L	3.2 [1.25]	3.3	2316 [144.6]	259	393
60F	2.5 [1.0]	3.1	2297 [143.4]	291	471
45F15L	1.9 [0.75]	3.0	2297 [143.4]	221	351
60C	12.7 [5.0]	4.4	2295 [143.3]	545	719
45C15L	6.4 [2.5]	3.8	2292 [143.1]	299	474

Setting Time

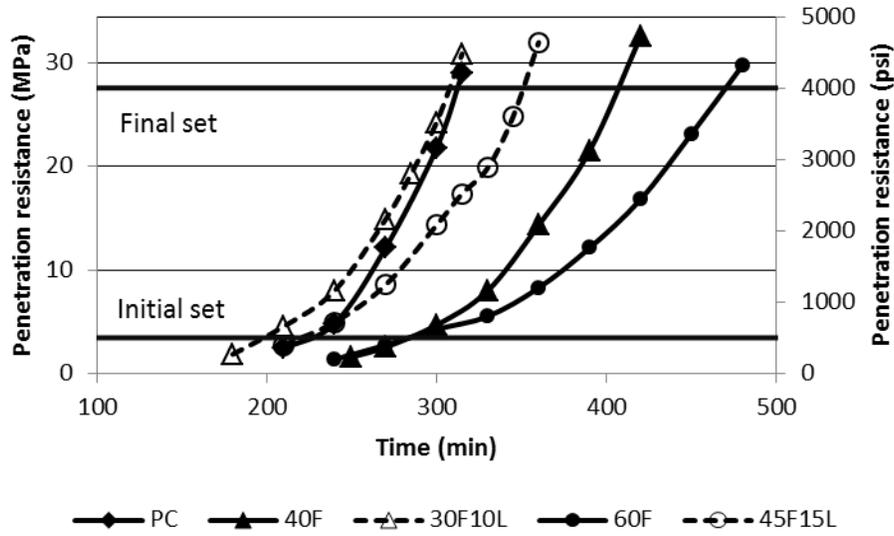
Figure 2 presents the measured setting development and Table 3 compares the initial and final setting times of all nine mixtures. Both fly ashes cause initial and final setting delays, due to both a dilution effect (Class F and Class C) and a retardation (Class C) effect, as observed in a previous study (Gurney *et al.*, 2012).

For the Class F fly ash mixtures, the presence of limestone powder accelerated the reactions to the point that both initial and final set times of mixture 30F10L were faster than the mixture containing only cement (PC). For the mixture with a higher level of cement replacement, the limestone powder eliminated the initial set delay and decreased the final set delay from 159 min (mixture 60F) to 39 min (mixture 45F15L).

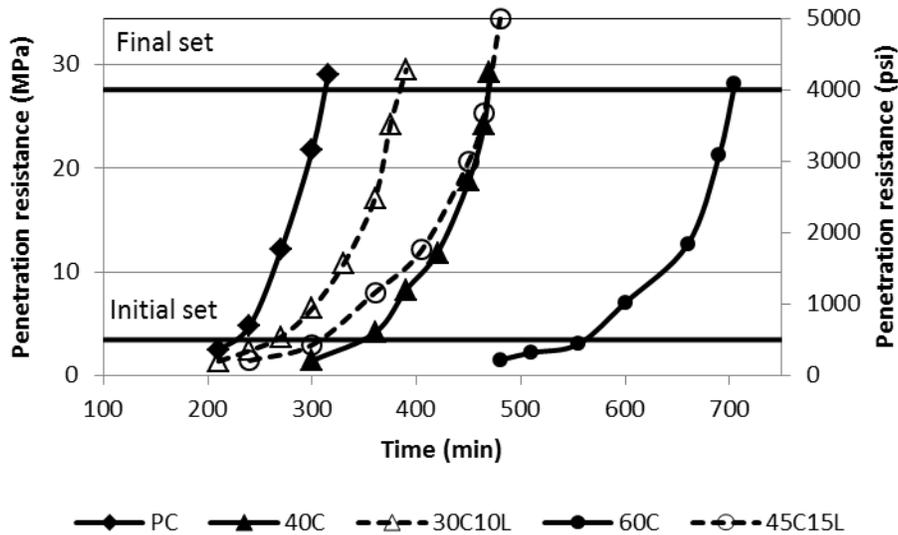
For the Class C fly ash mixtures, the limestone powder accelerated the reactions, as well, but since the presence of this fly ash retarded setting considerably, the limestone powder did not completely eliminate the delays. In mixtures with 40 % of cement replacement, the presence of limestone powder decreased the initial set delay from 121 min (mixture 40C) to 35 min (30C10L) and the final set delay from 161 min (mixture 40C) to 81 min (30C10L). In mixtures with 60 % cement replacement, the presence of limestone powder decreased the initial set delay from 263 min (mixture 60C) to 75 min (45C15L) and the final set delay from 353 min (mixture 60C) to 162 min (45C15L).

Semi-adiabatic calorimetry

The semi-adiabatic temperature measurements for the nine mixtures are provided in Figure 3. The acceleration of the reactions by the incorporation of limestone into the cement-fly ash blends is easily observed. All of the binary and ternary blends, however, exhibit a much smaller maximum temperature and a more gradual temperature decrease than those observed in the 100 % ordinary portland cement concrete, which should provide a significant advantage with respect to thermal cracking issues in some concrete constructions, such as mass concrete.



(a)



(b)

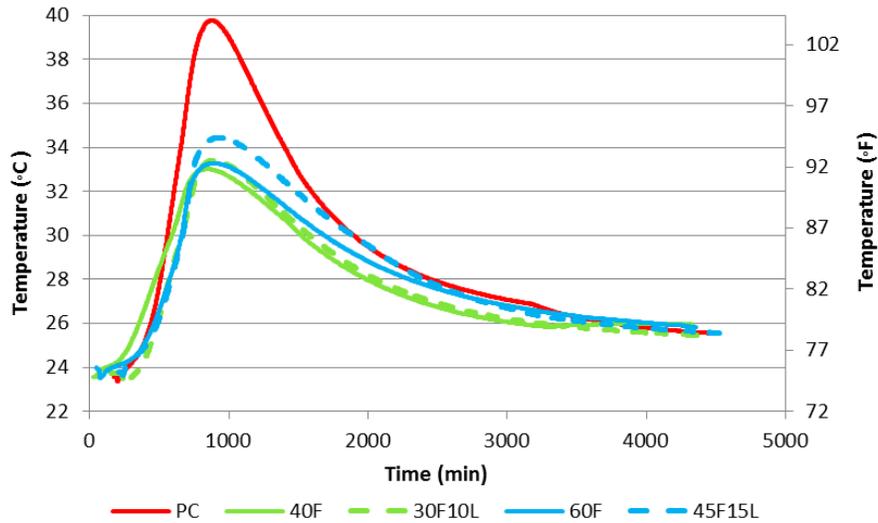
Figure 2 – Setting development: a) plain mixture and mixtures containing Class F fly ash; b) plain mixture and mixtures containing Class C fly ash.

It is interesting to note in Figure 3 that even though the Class C fly ash is more reactive than the Class F fly ash at intermediate ages (as confirmed by the compressive strength results to follow), the Class F concretes exhibit a 3 °C to 4 °C higher temperature rise at their peak. Apparently, the initial retardation produced by the Class C fly ash, along with its accompanying lack of any temperature increase, is substantial enough to also reduce the subsequent temperature rise produced in these concrete mixtures. Alternatively, the activation energies for these two fly ashes could be significantly different, which would also produce a different temperature sensitivity and semi-adiabatic response for the Class C and Class F fly ash mixtures.

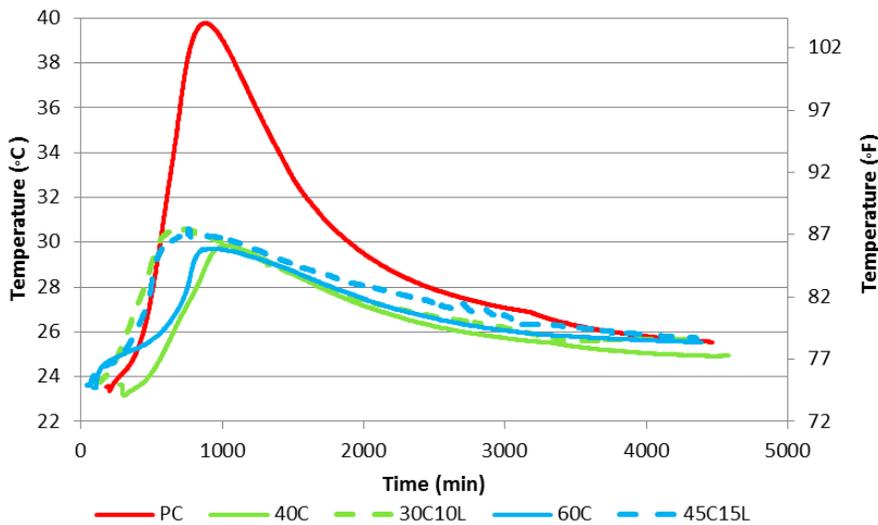
Isothermal calorimetry

Figure 4 shows the heat flow per unit volume of water for each mixture for the first 24 h of hydration. The vertical lines indicate the measured time for initial set. As expected, the substitution of cement by fly ash caused a dilution effect, due to the fact that fly ashes are normally inert during the first few hours. As a consequence,

independent of the fly ash used, the maximum heat flow decreased with the increase of fly ash content and, in some cases, there was a significant retardation in the heat flow, shown as a shift of the peaks to the right. When limestone powder was used, it both accelerated (shifted to the left) and amplified (increased in height) the heat flow by providing additional surface area for the nucleation and growth of products.



(a)



(b)

Figure 3 – Semi-adiabatic calorimetry for: a) plain mixture and mixtures containing Class F fly ash and b) plain mixture and mixtures containing Class C fly ash.

A typical cementitious system heat profile from isothermal calorimetry shows a first main peak that is related to the hydration of C_3S and a second peak that corresponds to the renewed formation of ettringite. Due to the reactive aluminate content of the Class C fly ash, mixtures containing this fly ash presented a sharp amplification of the second peak and both peaks were considerably retarded from those of the plain mixture. The presence of limestone in these mixtures amplified this peak even more and accelerated its occurrence, as the limestone may also participate in reactions of the aluminate phases in these systems, producing carboaluminates in contrast to the conventional formation of sulfoaluminate hydrates (De Weerd *et al.*, 2011b).

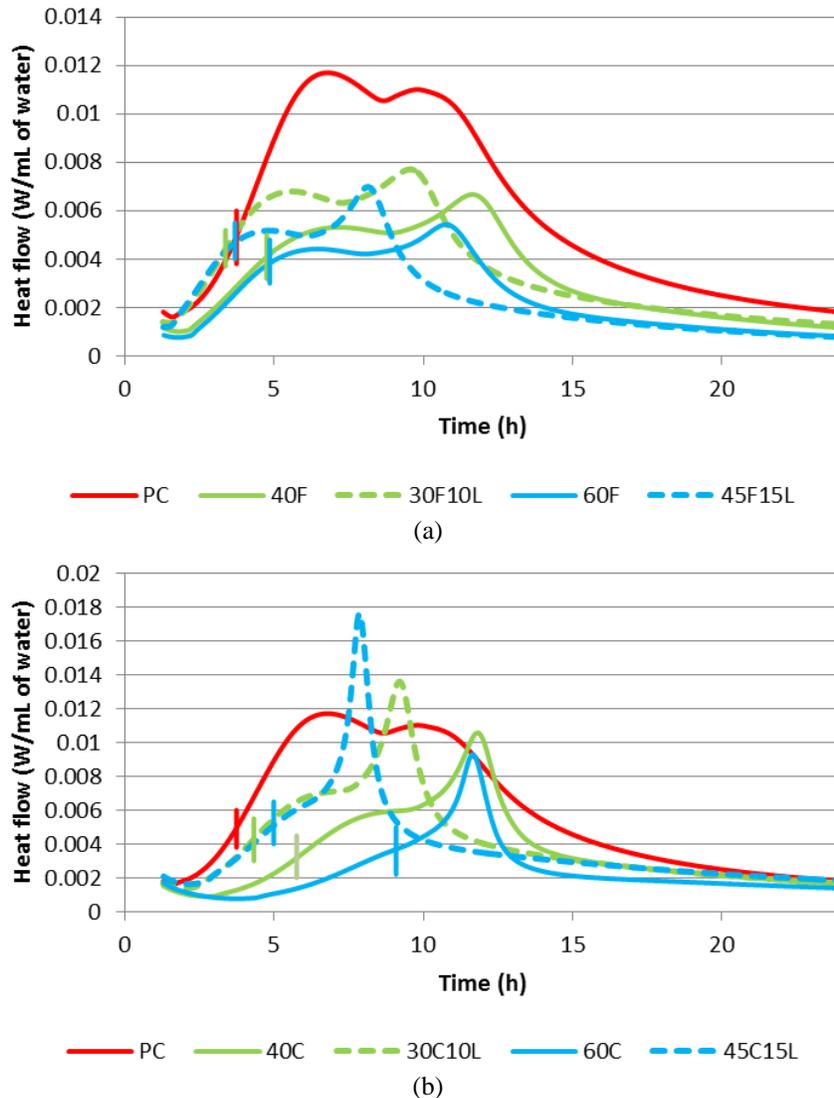


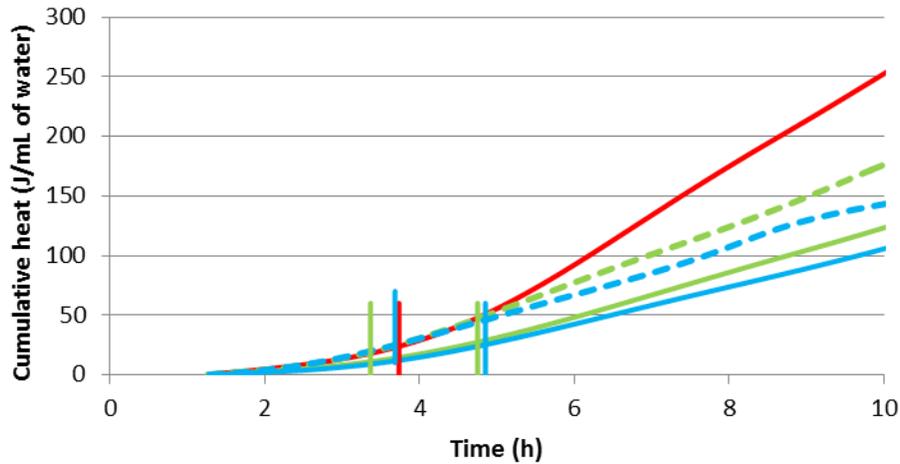
Figure 4 – Heat flow for a) plain mixture and mixtures containing Class F fly ash and b) plain mixture and mixtures containing Class C fly ash. The vertical lines indicate the measured time for initial set for each mixture. Results from two replicate specimens (not shown) overlap each other within the thickness of the plotted lines.

The cumulative heat release shown in Figure 5 is normalized per volume of water to examine the relationship between heat generation and the filling of this pore (water) volume with hydration products (Bentz *et al.*, 2012). At a given age, the cumulative heat decreases with the increase of fly ash content, but partially recovers this loss with the presence of limestone powder. Previous studies have indicated that compressive strengths should follow the same trends as these cumulative heat release results (Bentz *et al.*, 2012).

Compressive strength

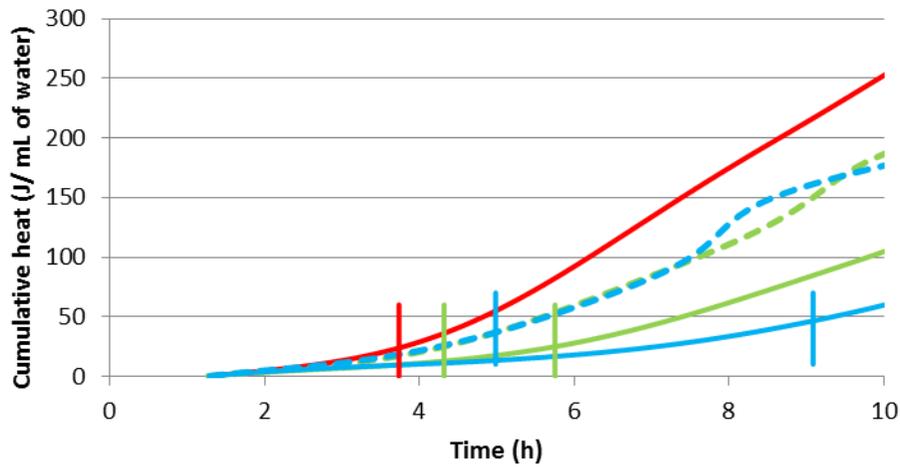
Figure 6 shows the compressive strength development for the plain mixture and mixtures containing fly ash. In general, mixtures containing Class F fly ash presented lower strength than comparable mixtures containing Class C fly ash. The early age compressive strengths (1 d and 3 d) were considered low for all of the mixtures where 60 % of the cement volume was replaced, independently of the fly ash used. The addition of limestone powder recovered a portion of the loss in compressive strength at all ages, but had a larger impact on the 28 d strength than the early age strength. The impact of limestone powder on the 28 d strength was more pronounced in the Class C fly

ash mixtures, where, for example, the strength doubled when mixture 45C15L is compared to mixture 60C. Both of the mixtures with 30 % fly ash and 10 % limestone powder would likely meet a 28 d strength target of 27.6 MPa (4000 psi). Conversely, only the 45 % Class C fly ash and 15 % limestone powder would meet a 28 d target of 20.7 MPa (3000 psi). If further strength enhancements at early ages are required, viable strategies include a non-chloride accelerator (Cost, 2011), switching to a Type III cement (Bentz *et al.*, 2010), and/or reducing the mixture's *w/cm* ratio (De la Varga *et al.*, 2012), any or all of which could be performed in combination with the fine limestone powder addition.



— PC — 40F - - - 30F10L — 60F - - - 45F15L

(a)



— PC — 40C - - - 30C10L — 60C - - - 45C15L

(b)

Figure 5 – Cumulative heat release for: a) plain mixture and mixtures containing Class F fly ash and b) plain mixture and mixtures containing Class C fly ash. The vertical lines indicate the measured time for initial set for each mixture. Results from two replicate specimens (not shown) overlap each other within the thickness of the plotted lines.

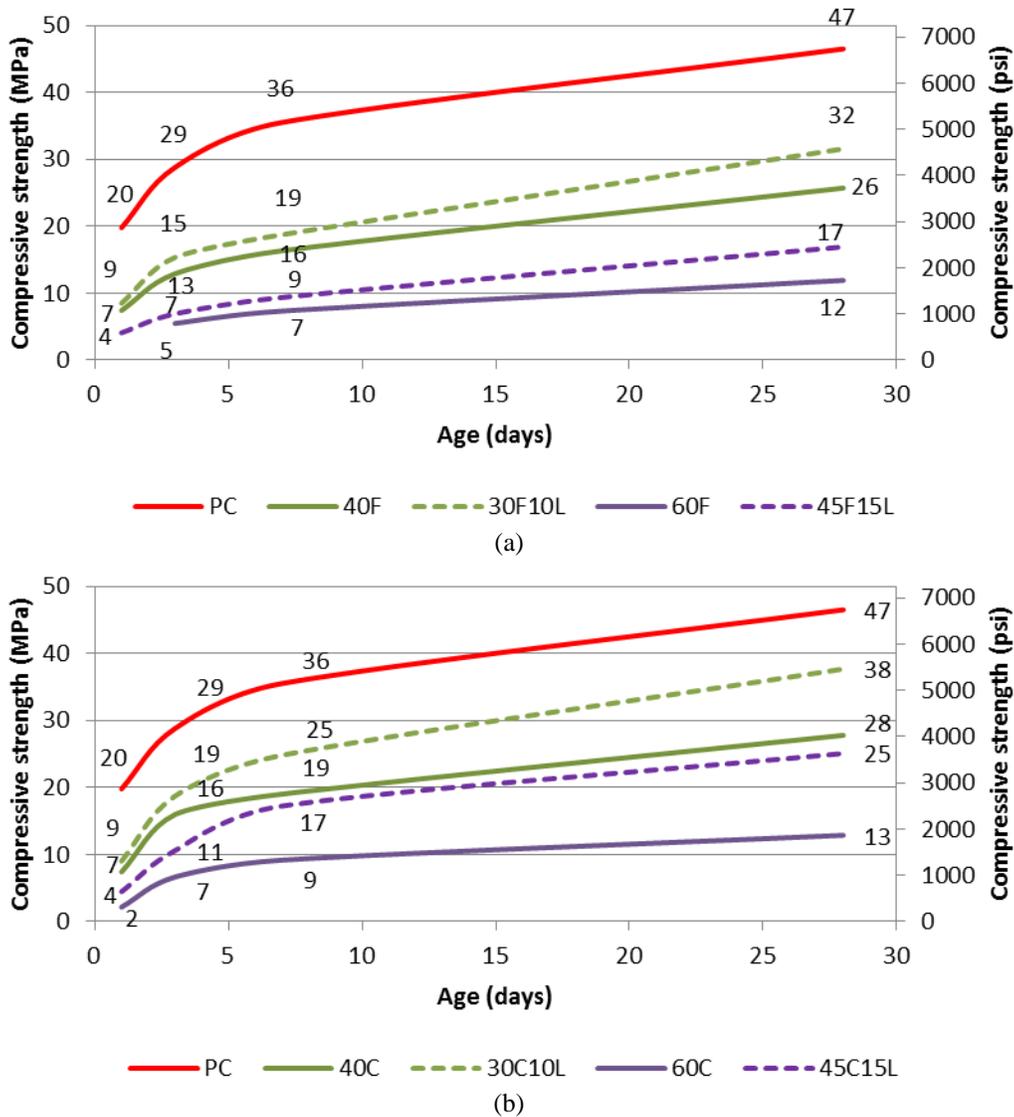


Figure 6 – Compressive strength development for mixtures containing: a) Class F fly ash and b) Class C fly ash. Coefficients of variation for three replicate specimens varied from 0.67 % to 2.5 % for the various mixtures.

Transport properties

Figure 7 shows the effects of fly ash and limestone powder on the measured chloride penetrability. At the 40 % replacement level, all mixtures, with the exception of the mixture containing only Class C fly ash (mixture 40C), presented lower or equivalent coulomb values to the plain mixture. For the 60 % replacement level, both mixtures containing limestone powder presented equivalent or lower coulomb values than the plain mixture. In mixtures with Class F fly ash, for both replacement levels, limestone decreased the charge passed to about half of that of the plain mixture. In mixtures with Class C fly ash, limestone powder decreased the charge passed to less than half of that of the corresponding mixture containing only cement and Class C fly ash. Contributions to these reductions in charge passed in the systems containing limestone powder may include the acceleration of the cement and fly ash reactions in the presence of limestone, differences in the phase assemblage due to the presence of limestone (De Weerd *et al.*, 2011a), and reductions in the conductivity of the pore solution in these systems.

Figure 8 shows the effect of fly ash and limestone powder on the measured surface resistivity. At the 40 % replacement level, all mixtures, with the exception of the mixture containing only Class C fly ash (mixture 40C),

presented a higher resistivity than that of the plain mixture. For the 60 % replacement level, all mixtures, with the exception of the mixture containing only Class C fly ash (mixture 60C), presented equivalent or higher resistivity than the plain mixture. In mixtures with Class F fly ash, for both replacement levels, limestone increased the measured resistivity by at least 50 % when compared with mixtures with the same cement replacement level but no limestone powder. In mixtures with Class C fly ash, limestone powder increased the resistivity by at least 85 % when compared with mixtures with the same cement replacement level but no limestone powder. Both resistivity and RCPT are influenced by both the concrete microstructure and the composition (conductivity) of its pore solution. To separate out these influences, future direct measurement of chloride ingress profiles under ponding conditions for the various concretes would be required.

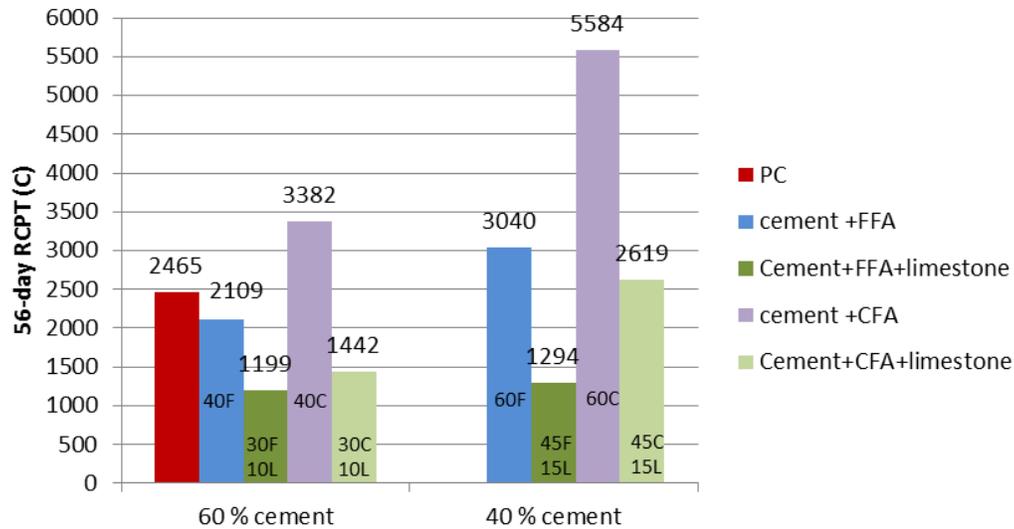


Figure 7 – Rapid chloride penetrability test (RCPT) values at 56 d as a function of cement volumetric replacement level. Coefficients of variation for three replicate specimens varied from 2.9 % to 19.1 % for the various mixtures.

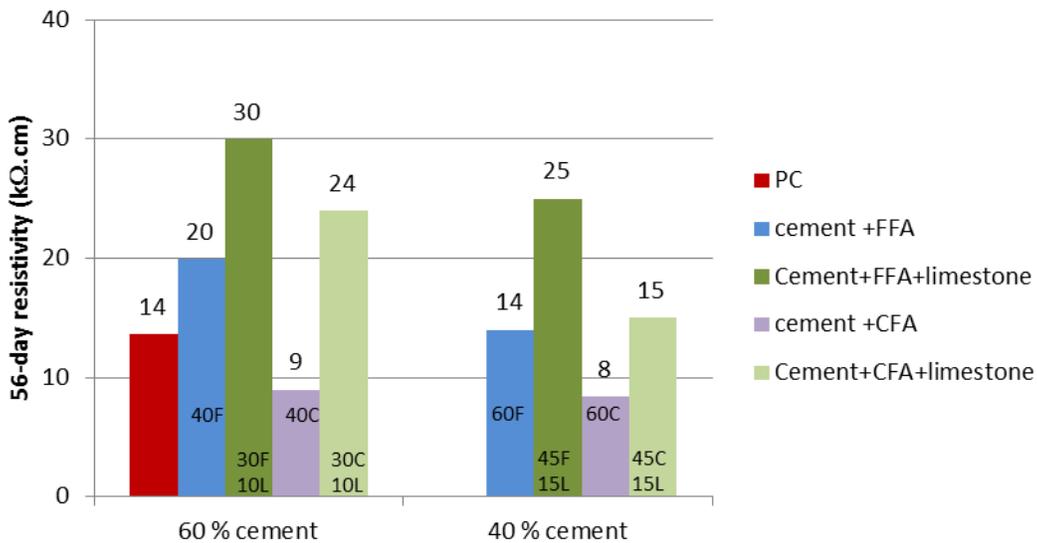


Figure 8 – Measured surface resistivity at 56 d as a function of cement replacement level. Coefficients of variation for three replicate specimens varied from 0.5 % to 9.9 % for the various mixtures.

CONCLUSIONS

In this study, the benefits of adding fine limestone powder to high volume fly ash concrete mixtures have been clearly demonstrated. In agreement with past studies on pastes (Gurney *et al.*, 2012), the 0.7 μm limestone powder was able to mitigate the excessive setting time delays conventionally produced when high volume fractions of cement are replaced by fly ash, which are a result of the effects of both dilution (Class C and Class F fly ash) and retardation (Class C fly ash). Additionally, significant improvements in 28 d strength and 56 d transport properties, as exemplified by RCPT and resistivity test results, were also observed. Isothermal calorimetry has indicated that the limestone both accelerates and amplifies the early age hydration reactions in the ternary blends, likely by providing additional surfaces for the nucleation and growth of reaction products. At later ages, the limestone may participate in the reactions of the aluminate phases, leading to the production of carboaluminate hydrates (as opposed to the conventional sulfoaluminates).

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