

Hydration and Strength Characteristics of Green Cementitious Mortars with Ultrahigh-Volume Limestone-Calced Clay and Class F Fly Ash

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Abstract

This study aims to investigate the hydration and strength characteristics of green cementitious mortars with ultrahigh-volume limestone-calced clay as well as two kinds of Class F fly ash. Using the ASTM C311 strength activity index test method, the effect of different pozzolan replacement levels of cement (0%, 20%, 50%, and 80%, by weight) were investigated. Compressive strength at 3, 7, 14, 28 and 90 days under standard curing was recorded, and hydration heat of the 20% and 80% replacement mixes was studied using iso-thermal calorimetry. It was observed that the effectiveness of the pozzolan in mortars depends on particle size distribution, glassy or amorphous nature, surface area and replacement level. The sum of all these effects can be captured by the strength activity test only if the standard recommended 20% pozzolan mix is substituted with the actual mix composition. The results in this study provide insights into the mix design and applications of ultrahigh-volume pozzolanic cementitious

materials specifically made with limestone-calcined clay, and promote greener cement and concrete in construction industry.

Keywords

Pozzolan; Limestone-Calcined Clay; Fly Ash; Compressive Strength; Hydration Heat

Introduction

Sustainable development is a kind of development that meets the needs of the present without compromising the ability of future generations to meet their own needs [1]. The exponential increase in greenhouse gas emissions even after Kyoto protocol having come to effect indicates serious potential consequences of climate change and its impact on global efforts towards sustainability. China and India are among major countries besides the United States, Europe and Japan who are the leading contributors. Unlike developed countries, China and India with their much higher economic growth rates are likely to continue to increase in their share of greenhouse gas emissions in coming decades. As shown in Fig. 1, China and India account for almost 37% of the total carbon emissions as of 2014 [2].

Cement industry accounts for 6-7% of the global greenhouse gas emissions [2]. Concrete being the most consumed man-made material per capita after water bears the burden in turn. Portland cement, the primary manufactured component of concrete, is particularly polluting as it contributes almost a metric ton (MT) of CO₂ per MT of cement produced. About 40-50% of the CO₂ emissions, originate from the heating of the cement kiln up to around 1450 °C and from grinding and transportation to a lesser extent. The other 50-60% of the total amount of CO₂ originates from the

decarbonation (calcination) of calcium carbonate decomposing to calcium oxide, thereby liberating CO₂. The raw materials used to produce cement clinker consist of about 75-79% of calcium carbonate, which is added in the form of limestone and rest is clay.

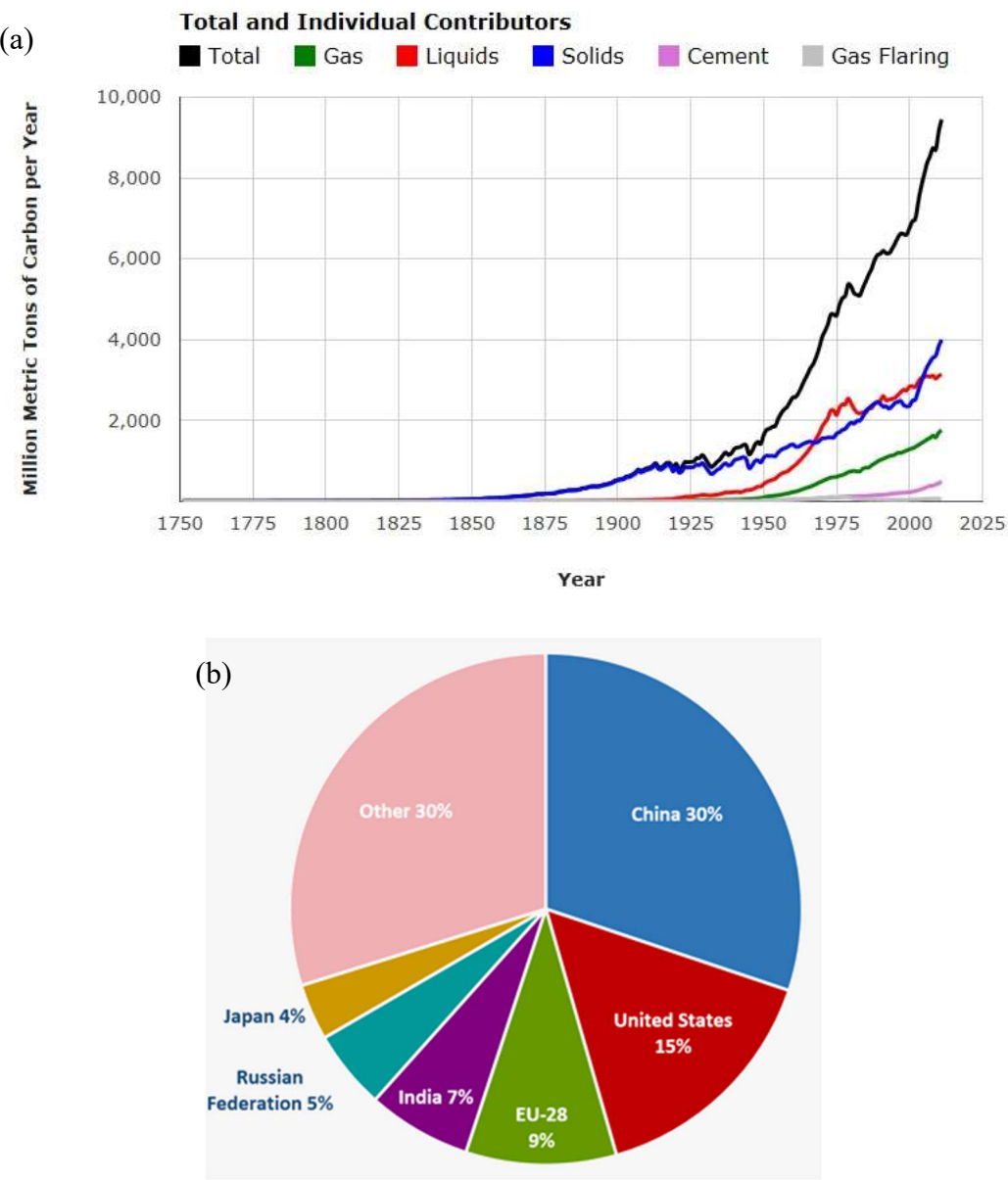


Figure 1. (a) Global carbon emissions from fossil fuels & (b) Emissions by countries in 2014 [2].

There is a global effort to produce greener cement and reduce the consumption of the materials by using alternative binders and supplementary cementitious materials (SCM) to produce blended

cement as well as concrete with lower cement content [3-6]. Coal Combustion Ash or Pulverized Fuel Ash, simply known as fly ash (FA), is a by-product of thermal power production in coal fired plants. This industrial waste has proven to be an effective pozzolanic material with properties suitable to be used in cement production and as a mineral admixture in concrete [7-9]. The annual generation of fly ash is around 600 million MT in China and around 220 million MT in India, out of which only approximately 70% and 60% of the fly ash is reused in these countries [10, 11]. Fig. 2 shows the trend of exponential growth in production of cement as opposed to competing construction materials such as steel and timber. A lot of research has been carried out over the years on the use of SCM in making of blended cements and also their use as mineral admixtures in concrete.

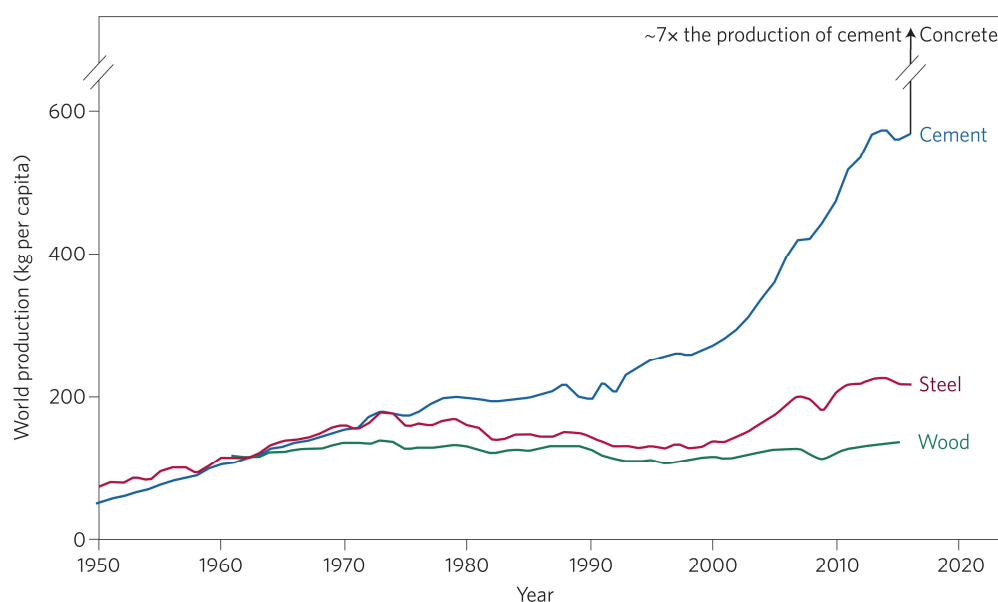


Figure 2. Historical growth in infrastructure material demand as exemplified through per capita cement, steel and wood production [12].

It is likely the total amount of SCM such as fly ash and blast furnace slag (by-product of steel making) available globally is inadequate to satisfy the likely demand from the construction

industry [13, 14]. In this context, limestone-calcined clay (LC2) has been proposed as a low cost, readily available and green substitute that can be used both for making cement as well as in concrete as a pozzolanic admixture [13, 14]. The green cement with LC2 is being actively promoted by a consortium of researchers from Switzerland, Cuba and India. Given that limestone and clay are the same raw materials used to produce LC2 and also Portland cement, it is particularly suitable for the purpose. It is estimated that if all Portland cement can be replaced by a green LC2 cement with 50% clinker, 30% calcined clay, 15% limestone and 5% gypsum, the carbon emission from cement manufacturing can be reduced by up to 30% [13, 15, 16].

Research Significance

The current research is aimed at studying the fundamental behavior of High-Volume Pozzolan Cementitious Materials with fly ash and LC2. This would supplement the earlier research which showed that it is possible to substitute up to 80% of cement by fly ash in concrete and achieve 28-day compressive strength of 45 MPa as well as required early strength [17, 18]. Such low carbon mortar compositions can also be used in practical green concrete and Strain-Hardening Cement Composite (SHCC) mixes which can address the major weaknesses of conventional concrete, viz its environmental sustainability and brittle fracture under tension [19-21]. The research reported here is also designed to support future development of green concrete mixes with a combination of fly ash and LC2 material in order to optimize the packing density and pozzolanic activity. The research results also provide important information about the fundamental characteristics of LC2 material which is a relatively new SCM being used to develop sustainable cementitious materials.

Experimental Investigation

Materials

Type I 52.5N Portland cement which met all the requirements of BS EN 197-1 [22] was used. Two kinds of fly ash - one supplied by the China Power and Light Co., Ltd in Hong Kong, China; another obtained from NTPC Thermal power plant in Kaniha in the eastern state of Odisha in India [23] were used. The limestone-calcined clay (LC2) material was also sourced from India from a batch of pilot production described in Bishnoi et al. [24]. Standard silica sand per ASTM C778 [25] was used as the fine aggregates.

Table 1 – Chemical composition of Portland cement (PC), fly ash (FA) and limestone-calcined clay (LC2)

Material	LOI ^a (%)	Al ₂ O ₃ (%)	SiO ₂ (%)	Fe ₂ O ₃ (%)	CaO (%)	SO ₃ (%)	MgO (%)	Na ₂ O (%)	K ₂ O (%)	TiO ₂ (%)
OPC	1.2	4.4	20.2	3.4	63.9	4.7	2.1	0.1	0.4	-
Chinese FA	2.5	18.9	49.8	11.4	9.3	2.1	3.6	1.6	1.8	-
Indian FA	1.7	27.5	64.6	3.5	1.1	-	-	-	1.3	1.9
LC2	-	31.3	45.8	3.4	14.5	1.7	0.0	0.0	0.7	2.6

^a Loss on Ignition.

Table 1 shows the chemical composition procured by X-ray Fluorescence (XRF) analysis of the two kinds of fly ash and the LC2 as compared to that of Type I Portland cement. SEM images of Indian fly ash and LC2 material are shown in Fig. 3, and the morphology of the Chinese fly ash is very similar to that of the Indian fly ash. Fig. 4 presents the particle size distributions of the cement, fly ash and LC2 material obtained from a Microtrac Bluewave LASER particle size analyzer. The X-ray Diffraction (XRD) analysis and Thermal Gravimetric Analysis (TGA) of the three powder

materials are shown in Fig. 5 and Fig. 6. The XRD analysis gives useful insights into the crystalline structure of the pozzolans, while the thermal characteristics can indicate the loss on ignition and confirm certain minerals present in the material from their thermal signature during heating under high temperature. Brunauer Emmett Teller (BET) surface area measurement was also carried out using a Beckman Coulter SA3100 surface area and pore size analyzer and the results are discussed in the analysis section later.

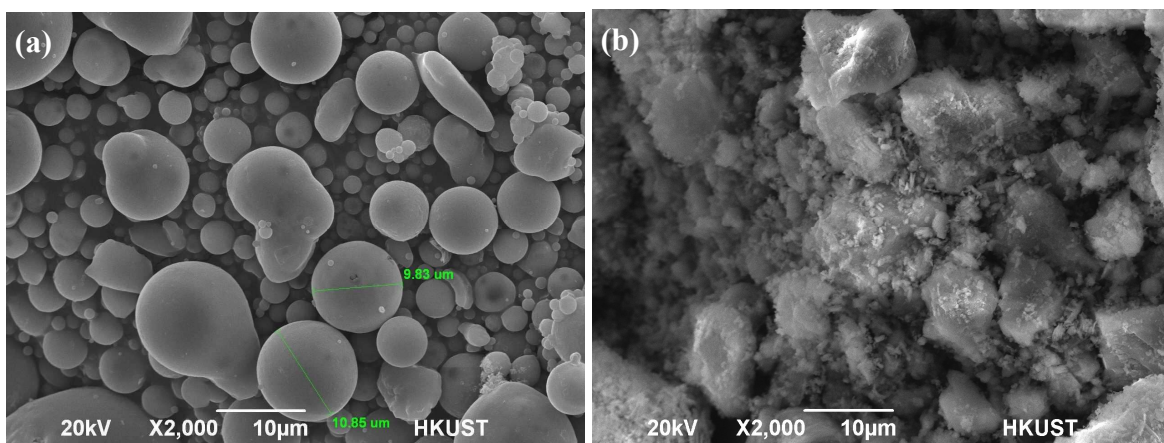


Figure 3. Scanning Electron Microscope (SEM) images of (a) Indian fly ash and (b) limestone-calcined clay.

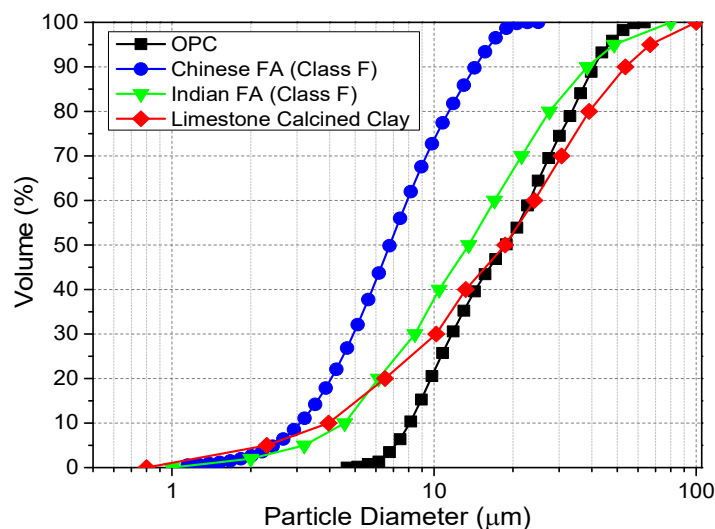


Figure 4. Particle size distribution of Portland cement, fly ash and limestone-calcined clay

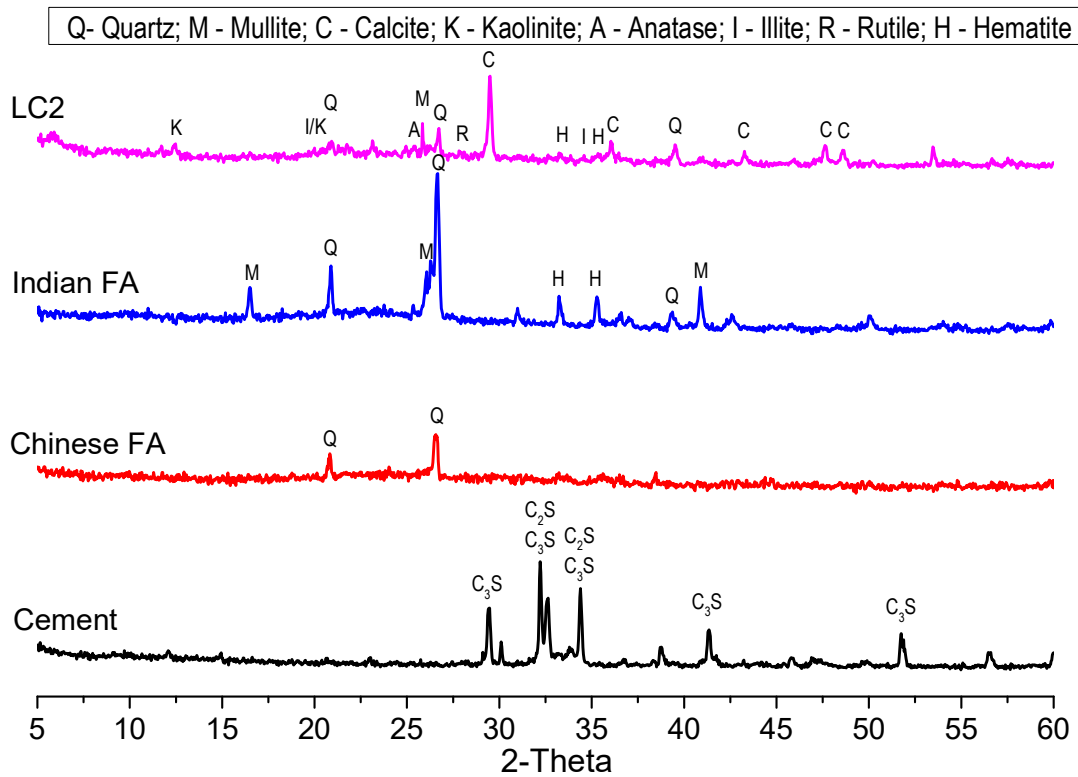
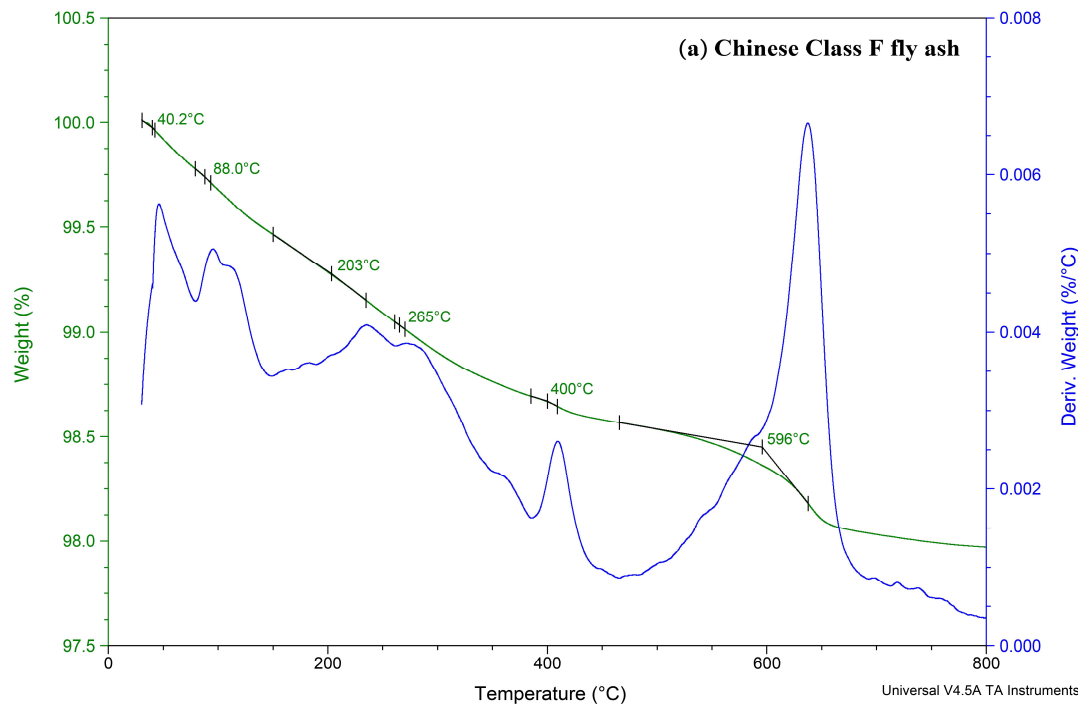


Figure 5. X-ray diffraction (XRD) results of Portland cement (PC), fly ash (FA) and limestone-calcined clay (LC2)



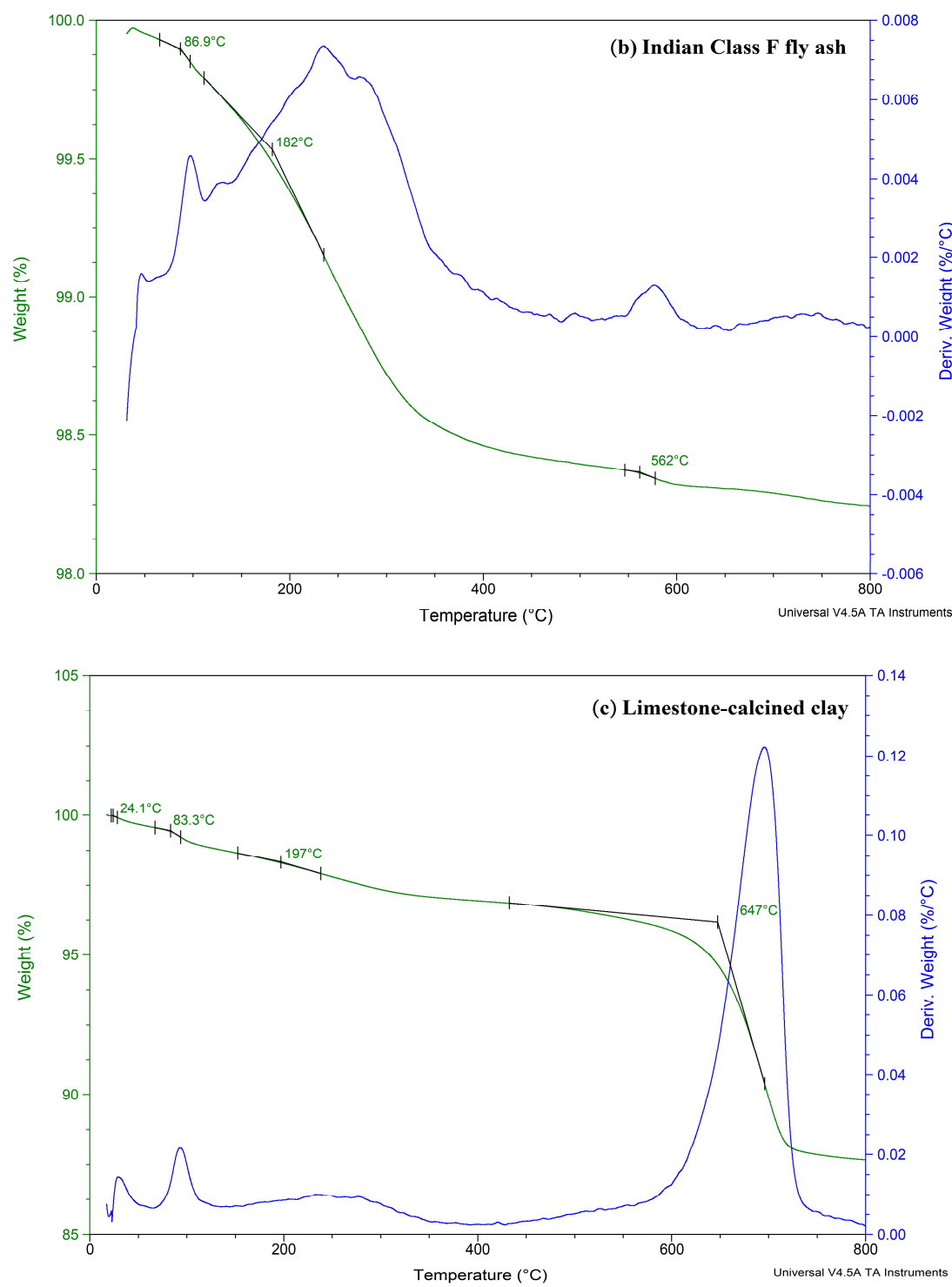


Figure 6. Thermal Gravimetric Analysis results of (a) Chinese fly ash, (b) Indian fly ash and (c) limestone-calcined clay.

From the chemical compositions of the materials listed in Table 1, the two kinds of fly ash are both categorized as Class F as per ASTM C618 [26]. The Chinese fly ash has significantly higher calcium oxide compared to the Indian fly ash, which can result in higher pozzolanic strength activity. This is particularly important in ultrahigh-volume pozzolan cementitious materials, where calcium hydroxide produced from cement hydration may not be adequate to react with all the pozzolanic material in the mix.

Mix Design and Testing Procedure

Table 2 – Mix proportions (weight ratio)

Mix	Pozzolan	Cement/Binder	Pozzolan/Binder	Sand/Binder	Water / Binder
C100 (Control)	-	1.0	0.0	2.75	0.484
C80FA20(C)	Chinese FA	0.8	0.2	2.75	0.484
C50FA50(C)	Chinese FA	0.5	0.5	2.75	0.484
C20FA80(C)	Chinese FA	0.2	0.8	2.75	0.484
C80FA20(I)	Indian FA	0.8	0.2	2.75	0.484
C50FA50(I)	Indian FA	0.5	0.5	2.75	0.484
C20FA80(I)	Indian FA	0.2	0.8	2.75	0.484
C80L20	LC2	0.8	0.2	2.75	0.484
C50L50	LC2	0.5	0.5	2.75	0.484
C20L80	LC2	0.2	0.8	2.75	0.484

Besides the characterization tests of the raw materials, three tests were conducted on the mortar samples, namely the flow table test, the isothermal calorimetry test and the compressive strength test. A series of mortar mixes were designed as shown in Table 2. The experimental plan was to test the mortar samples with 0%, 20%, 50% and 80% replacement of cement by the three pozzolans under standard curing [27], and study their effects on strength development. No superplasticizer

was used during mixing although the workability was significantly affected by the use of different pozzolans particularly LC2, given its rather irregular particle geometry (Fig. 3).

A Hobart HL800 mixer was utilized to prepare the mortars. For each mortar mix in Table 2, the fresh property was measured using the flow table test according to ASTM C1437 [28]. A truncated cone mold with a diameter of 3.94 in (100 mm) at the bottom and 2.76 in (70 mm) at the top and a height of 2.36 in (60 mm) was placed on a smooth plate, filled with mortar, and lifted upward. After that, the table was immediately dropped 25 times in 15 second [28]. A characteristic deformability factor, denoted by Γ , was calculated as:

$$\Gamma = (D_1 - D_0)/D_0 \quad (1)$$

where D_1 is the average of two orthogonal diameter measurements after dropping, and D_0 is the diameter of the bottom of the slump cone.

Cubic samples measuring 40 mm × 40 mm × 40 mm were cast and cured for comparison purposes. All the specimens were cast in greased steel molds. After finishing the surface, the specimens were covered with a polyethylene sheet to prevent loss of moisture, and were stored for 24 hours at room temperature prior to demolding. After demolding, all of the specimens were cured in a fog room at a temperature of 23 ± 2 °C and relative humidity of $95 \pm 5\%$, until testing. Compressive testing was performed with a standard sample holder to ensure a 40 mm × 40 mm loading area. The loading rate for compressive tests was 0.35 MPa/s. For each data point, a minimum of three test results were averaged and reported along with the coefficient of variation. The Strength Activity Index (SAI) for the mixes with different contents of pozzolans can be calculated as per ASTM C311/C311M [29] as follows:

$$SAI = (f_c^{\text{pozz}}/f_c^{\text{control}}) \times 100\% \quad (2)$$

where: f_c^{pozz} = compressive strength of the mix with pozzolan at a specific age, MPa; f_c^{control} = compressive strength of the control mix at the same age, MPa.

Experimental Results and Discussions

The results of the study are discussed in two parts, including the basic characteristics of the pozzolanic materials and their likely impact on their pozzolanic strength activity.

Characterization of pozzolans

The SEM images of the fly ash contrasts sharply against the image of the LC2 (Fig. 3) in revealing their different particle structures. While the fly ash consists of spherical smooth particles, LC2 is characterized by angular particles of random shapes and sizes. This difference is expected to affect the workability of the mix.

From the particle size distribution shown in Fig. 4, the median particle size of Type I PC, Chinese FA, Indian FA and the LC2 are 18.86 μm , 6.76 μm , 13.56 μm and 18.68 μm respectively. However, it is interesting to note that the LC2 material has the largest spread while the remaining three curves have similar spread around their median value. The Chinese fly ash has the finest particle size and would be expected to have better pozzolanic strength activity as compared to the Indian fly ash. The BET surface area measurement which not only measured the external surface (such would be related to the particle size distribution) but also measures the internal surface in case of porous materials, yields values of 2.328 m^2/g for Chinese fly ash, 0.696 m^2/g for the Indian fly ash and 8.353 m^2/g for the LC2. Again this indicates an advantage for the LC2, followed by Chinese fly

ash, with their higher surface area rendering them more reactive during cement hydration and the subsequent pozzolanic reaction.

From the X-Ray Diffraction (XRD) results for the three pozzolanic materials as well as the Portland Cement shown in Fig. 5, the peaks corresponding to the Quartz (SiO_2) and CaO are visible to different degrees of intensity. While the Type I PC and Indian fly ash seem qualitatively to be more crystalline in nature, only a quantitative analysis using the Rietveld refinement can accurately reveal the degree of crystallinity and hence the presence of glassy or amorphous phase which has a strong influence on pozzolanic strength activity.

From Fig. 6, the Thermal Gravimetric Analysis (TGA) results reveal different signatures of the LC2 material with significant presence of CaCO_3 exhibiting highest loss on ignition (LOI) at around 12%. Besides, the difference in calcium content of the two kinds of fly ash is readily reflected by the slope around 700 °C.

Fresh Property of mortars

The Characteristic Deformability Factors (Γ) for all the mixes listed in Table 2 are summarized in Table 3. Compared to the control mix ($\Gamma=1.10$), the mixes with Chinese fly ash showed improved workability, from $\Gamma=1.15$ for C80FA20(C) to $\Gamma=1.35$ for C20FA80(C), and the mixes with India fly ash had a similar trend. However, the mixes incorporating LC2 had lower workability with a Γ no more than 1.05. As mentioned earlier, the water/binder ratio was constant and superplasticizer was not used to compensate for the variation in workability. Poor workability could have affected the compaction and packing density of the mixes, which can lead to variation in compressive

strength. However, LC2 mixes exhibited relatively good strength probably owing to the much higher BET surface area and calcium content.

Table 3. Summary of Characteristic Deformability Factor (Γ) and compressive strength.

Mix	Characteristic Deformability Factor	3-day Comp. Strength (MPa)	7-day Comp. Strength (MPa)	14-day Comp. Strength (MPa)	28-day Comp. Strength (MPa)	90-day Comp. Strength (MPa)
C100(Control)	1.10	27.0(1.3)	32.4(0.7)	36.6(1.8)	43.6(0.8)	45.6(1.4)
C80FA20(C)	1.15	23.6(2.1)	28.7(2.2)	30.9(3.4)	38.3(1.9)	39.2(1.4)
C50FA50(C)	1.21	14.3(0.7)	20.6(1.3)	23.6(0.9)	30.6(2.7)	38.8(1.2)
C20FA80(C)	1.35	3.4(0.4)	5.8(0.4)	6.5(0.5)	8.3(1.4)	12.0(0.4)
C80FA20(I)	1.10	19.1(1.9)	24.7(2.8)	28.6(3.5)	32.0(1.5)	41.2(2.5)
C50FA50(I)	1.19	11.3(0.7)	14.5(0.3)	15.6(1.8)	20.3(1.4)	31.9(1.6)
C20FA80(I)	1.31	4.4(0.2)	5.1(0.1)	5.8(0.4)	7.0(0.4)	12.9(0.5)
C80L20	1.05	22.2(2.2)	29.6(2.5)	33.0(4.7)	35.4(2.3)	38.3(5.6)
C50L50	1.03	10.5(1.4)	15.8(2.4)	16.4(0.4)	18.7(0.5)	18.9(0.8)
C20L80	1.00	4.7(0.1)	5.5(0.3)	5.9(0.1)	6.7(0.2)	6.9(0.2)

Note: Data in brackets is the standard deviation.

Compressive Strength and Pozzolanic Strength Activity Index

Table 3 summarizes the compressive strength of all the mixes at 3-day, 7-day, 14-day, 28-day and 90-day ages under standard curing conditions. The coefficients of variation of the results are between 3.5% to 10.3% for different mixes, which indicates reasonable uniformity among samples. The 28-day compressive strength of control sample is lower than expected due to variation in manual casting procedure and inadequate compaction. However, since identical procedure is used

for all mixes, the relative strengths are reasonable and can be used to understand the effect of pozzolan addition on strength as shown in Fig 4.

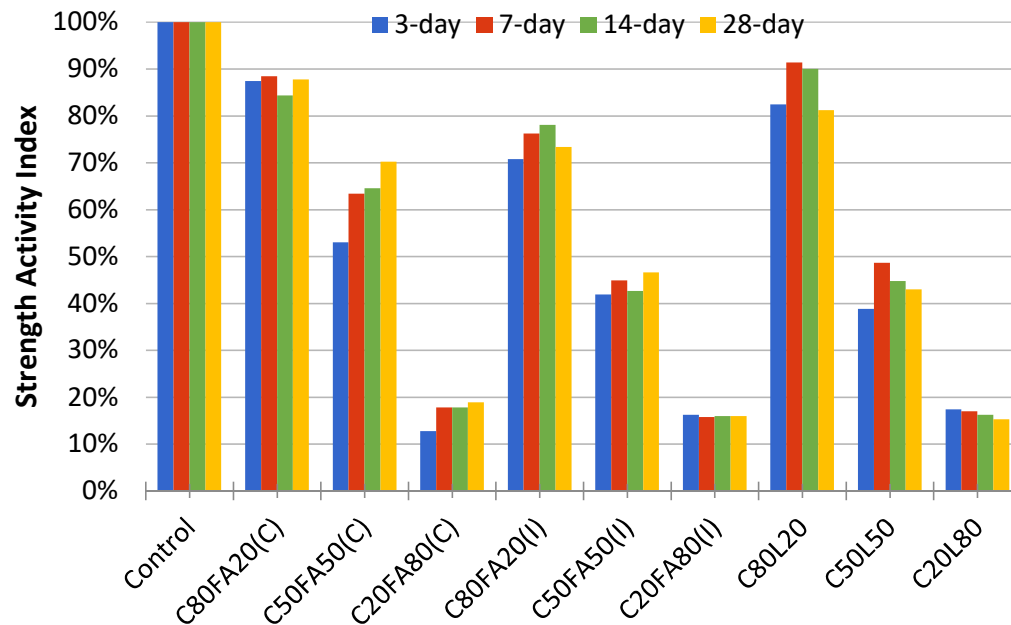


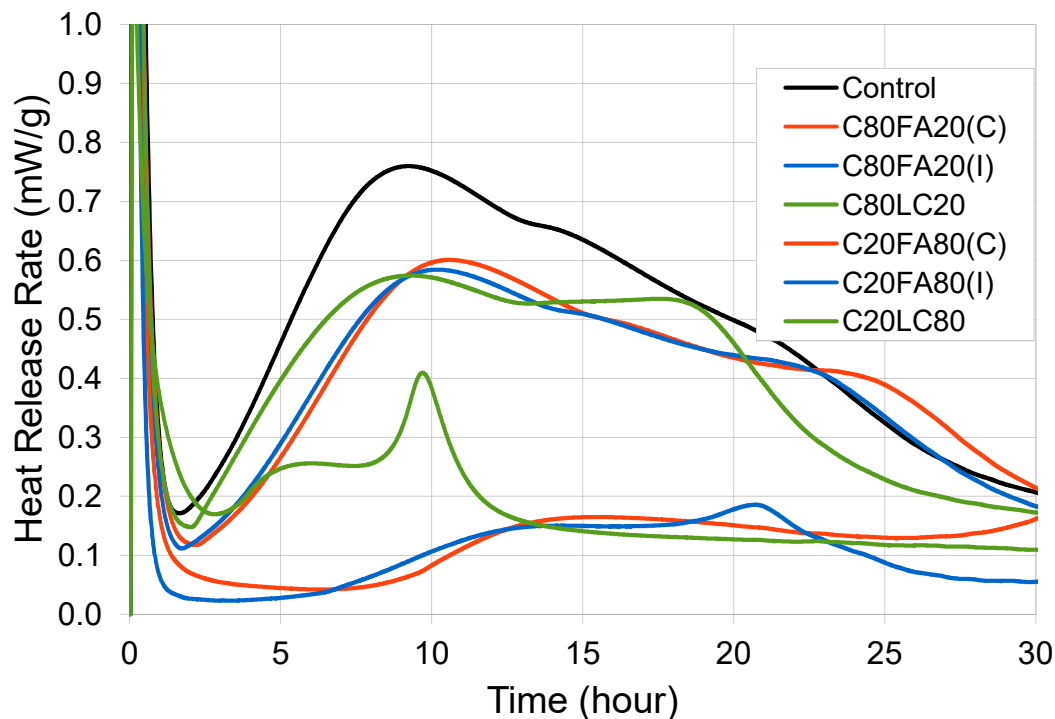
Figure 7. Strength activity indices of mortars with different pozzolan substitutes.

The overall trend of strength development with increasing age is reasonable. According to Eq. 7, the pozzolanic strength activity indices for the mixes with different replacement levels of pozzolan at different ages can be calculated, and the results are shown in Fig. 7. Generally speaking, the strength activity indices for the same mix after different curing are stable. For the mixes with Chinese fly ash, the index is about 88% for C80FA20(C), about 70% for C50FA50(C) and 19% for C20FA80(C). In other words, the index is decreased with increasing replacement level of pozzolan. The same trend can be observed for the mixes with Indian fly ash and LC2, and the indices are 73% and 81% for Indian fly ash and LC2, respectively. It is interesting to note that while there is not much increase in strength after 28 days for the control and LC2 mixes, the mixes

with both kinds of fly ash show substantial increase in strength at 90-day age compared to 28 days at almost all the replacement levels.

Hydration Heat

Figure 8 shows the heat release rate and heat of hydration for the seven out of ten mortar mixes listed in Table 2. The test was conducted for 72 hours and the results clearly indicate the effect of replacement of the cement with the three different pozzolan used in this study. At relatively low level of 20% replacement the three different pozzolans have almost indistinguishable effect on the heat release rate and total heat of hydration of the mortar mixes. However, at higher replacement level (80%), the behavior is entirely different with the LC2 having a greater early age pozzolanic strength activity which is expected to result in higher compressive strength and total heat of hydration of the mix.



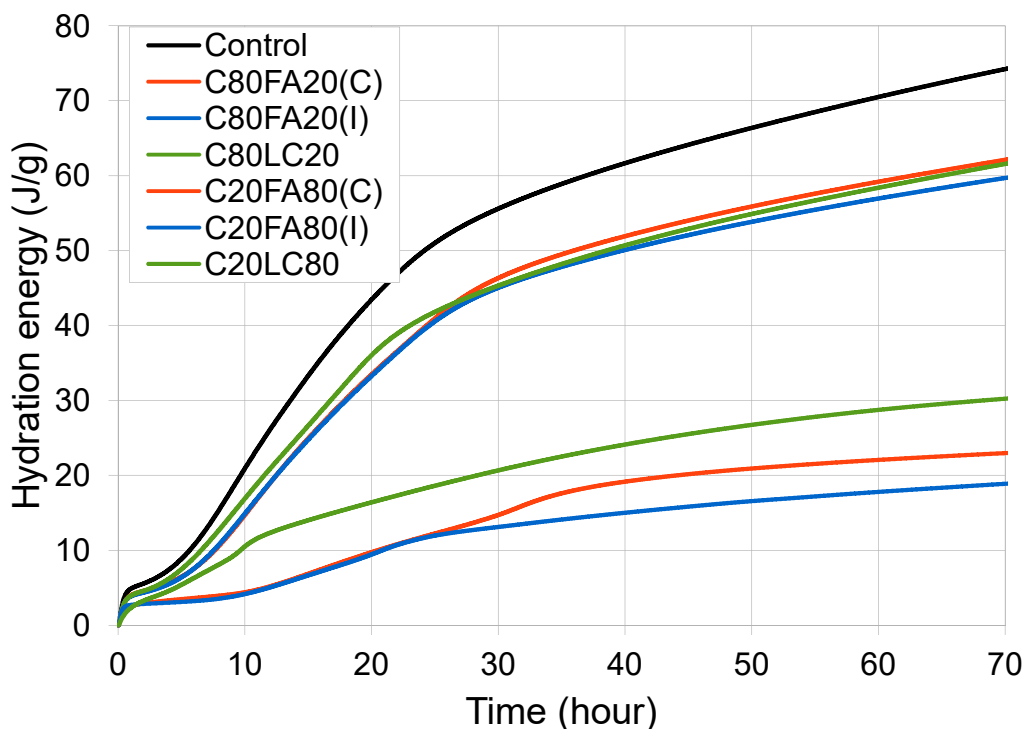


Figure 8. Isothermal calorimetry results for the mortars: (a) Hydration heat release rate at the early stage; (b) Total hydration heat up to 72 hours.

Further Research

These tests may give significantly different results if repeated maintaining constant workability by use of superplasticizer. It is desirable to test high-volume pozzolan cementitious materials at the age of one year or more to account for the long term strength gain characteristics of these materials. Further research needs also to look in to the durability aspects in particular the lower carbonation resistance of high-volume pozzolan mixes. Strength Index Factor is a good measure of efficiency of a pozzolanic material when replacing cement and accounts for the combined effect of multitude of parameters such as particle size distribution, oxide composition, glassy content etc. In the future, a quantitative correlation will greatly help mix design of high-volume pozzolan

cementitious materials. Such research will contribute greatly to a future built with sustainable concrete [12].

Conclusions

The paper has reported experimental results on hydration and strength characteristics of green cementitious mortars with ultrahigh-volume limestone-calcined clay (LC2) as well as two kinds of Class F fly ash. All three materials have been carefully characterized in terms of their chemical composition, physical characteristics such as particle size distribution, morphology of the particles, BET surface area, calorimetric measurement of heat of hydration when used in a mortar mix. Strength development up to 90 days of mortar mixes with 0%, 20%, 50% and 80% replacement following ASTM C311/C311M has been measured and reported. Longer age strength development, strength development using lower water/binder ratio and constant workability using superplasticizer will be studied in future. The following conclusions can be made according to the materials used and the test results from current phase of this study:

1. The workability of the standard mortar when using fly ash showed increased flow unlike in case of LC2, which resulted in significantly reduced flow properties. In spite of the reduced workability, LC2 seems to have a comparable strength activity index given its much higher BET surface area compared to the two kinds of fly ash studied.
2. When tested according to pozzolanic strength activity tests recommended by ASTM C311/C311M, the Strength Activity Index was found to be 88%, 73% and 81% for the Chinese fly ash, Indian fly ash and LC2, respectively. These figures were quite similar for 7-day strength as well.

3. The heat of hydration results qualitatively provided supportive evidence of the relative pozzolanic strength activity performance of the different pozzolans at 20% replacement level. It also indicates that at higher levels of cement replacement such as 80%, it is the pozzolanic reaction that is likely to dominate the mortar strength development.
4. It is postulated that the higher amorphous nature, finer particle size distribution, higher calcium content and surface area of the Chinese fly ash likely to give comparatively better performance. On the other hand, the LC2 material is likely to increase the water demand and reduce setting time of the mix given its non-spherical and angular particle structure with very high BET surface area compared to the two fly ash studied.

The results in this study provide insights into the mix design and applications of High-Volume Pozzolan Cementitious Materials with low energy content and carbon footprint. Ongoing research would reveal the validity of these conclusions under different water/binder ratios, curing conditions (thermal curing) and levels of cement substitution.

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