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Effect of elevated temperatures on geopolymer paste, mortar and concrete

Daniel L.Y. Kong [a], Jay G. Sanjayan [b,*]

[a] Department of Civil Engineering, Monash University, Clayton, Victoria 3800, Australia
[b] Faculty of Engineering and Industrial Sciences, Swinburne University of Technology, PO Box 218, Hawthorn, Victoria 3122, Australia

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ABSTRACT

Geopolymers are generally believed to provide good fire resistance due to their ceramic-like properties. Previous experimental studies on geopolymer under elevated temperatures have mainly focused on metakaolin-based geopolymers. This paper presents the results of a study on the effect of elevated temperature on geopolymer paste, mortar and concrete made using fly ash as a precursor. The geopolymer was synthesized with sodium silicate and potassium hydroxide solutions. Various experimental parameters have been examined such as specimen sizing, aggregate sizing, aggregate type and superplasticizer type. The study identifies specimen size and aggregate size as the two main factors that govern geopolymer behavior at elevated temperatures (800 °C). Aggregate sizes larger than 10 mm resulted in good strength performances in both ambient and elevated temperatures. Strength loss in geopolymer concrete at elevated temperatures is attributed to the thermal mismatch between the geopolymer matrix and the aggregates.

1. Introduction

Ordinary Portland Cement (OPC) concretes generally provide adequate fire resistance for most normal applications. However, the strength of OPC concrete decreases at elevated temperatures due to chemical and physical changes [1]. Further, spalling of conventional concrete occurs in fire which causes a rapid layer-by-layer loss of concrete cover, potentially leading to the exposure of the main reinforcements within the concrete to fire [2]. Efforts have, therefore, been made to identify an alternative binder which possesses good fire resistance both in terms of strength loss in elevated temperature and spalling resistance.

Geopolymer has recently emerged as a novel engineering binder material with environmentally sustainable properties [3]. Geopolymer is a term used to describe inorganic polymers based on aluminosilicates [4] and can be produced by synthesizing pozzolanic compounds or aluminosilicate source materials with highly alkaline solutions [5]. Due to their ceramic-like properties, geopolymers are believed to possess good fire resistance [5,6]. Therefore, concretes produced using geopolymers may possess superior fire resistance compared to conventional concretes produced with OPC. Geopolymers in this study are produced by mixing fly ash, which contains high contents of silicon and aluminium, with an alkaline liquid. Fly ash used for this purpose is Class F type. Fly ash is an industrial by-product of coal-fired power stations [7], and hence the production of raw materials for geopolymers does not require a high level of energy consumption. The energy consumption is calculated to be approximately 60% less than that required by OPC [8].

The objective of this investigation is to experimentally examine the performances of various forms of fly ash-based geopolymer and geopolymer composites (mortar and concrete) and their performances under elevated temperatures. The investigation studied the effects of specimen sizing on the paste as well as aggregate type and aggregate grading and sizing on the concrete. Superplasticizers were also used to conduct a concrete workability study.

2. Specimen preparation, test variables and testing methods

2.1. Materials characterization

The fly ash was sourced from Pozzolanic Gladstone. The fly ash was low calcium fly ash (class F) which is generally glassy with some crystalline inclusions of mullite, hematite and quartz. The fineness of fly ash was 89% passing through a 45 µm sieve. The chemical composition and loss on ignition (LOI) of the fly ash was determined by X-ray Fluorescence (XRF) and is given in Table 1. The total does not sum up to 100% because of rounding-off of the percentages.

Alkaline activators in the investigation consisted of alkali silicate and hydroxide solutions. The alkali silicate used was Grade D sodium silicate solution (Na_2SiO_3) supplied by PQ Australia with a specific gravity of 1.53 and a modulus ratio (M_s) equal to 2 (where $M_s = SiO_2/Na_2O$, $Na_2O = 14.7\%$ and $SiO_2 = 29.4\%$). The hydroxide solution needed for activation was prepared to a concentration of 7.0 M using potassium hydroxide (KOH) flakes of 90% purity and distilled water.

Local aggregates comprising of coarse and fine aggregates, in saturated surface dry conditions were used. The coarse aggregates

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* Corresponding author. Tel.: +61 3 9905 4985; fax: +61 3 9905 4944.
E-mail address: sanjayan50@gmail.com (J.G. Sanjayan).

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Table 1
Chemical composition of fly ash precursor.

Chemical	Component (wt. %)
Al_2O_3	27.0
SiO_2	48.8
CaO	6.2
Fe_2O_3	10.2
K_2O	0.85
MgO	1.4
Na_2O	0.37
P_2O_5	1.2
TiO_2	1.3
BaO	0.19
MnO	0.15
SrO	0.16
SO_3	0.22
ZrO_2	–
LOI[a]	1.7

[a] Loss on ignition.

primarily consisted of crushed old basalt aggregates from a local quarry and the fine aggregates consisted of river sand. The other coarse aggregates used in this investigation were slag aggregates which were procured from a local steel mill. The maximum size of the coarse aggregates was 20 mm. The grading of the aggregates used is presented in Fig. 1.

Two commercially available superplasticizers, both from BASF, were used as an additive to some of the concretes to conduct workability studies. One admixture was Glenium 27, a carboxylic ether polymer with long lateral chains. The other, Rheobuild 1000, was a sulphonated polymer-based superplasticizer.

2.2. Mixture design

All the fly ash-based geopolymer mixes in this test series had a solids-to-liquids ratio of 3.0. Studies on various trial mixes showed that this ratio provided near optimum strength and workability. When the solids-to-liquids ratio exceeded this figure, the workability of the pastes decreased, causing difficulties during compaction and loss of strength.

A large number of samples in various forms of geopolymer types were investigated, both at room temperature and after elevated temperature exposures. A summary of mixture compositions studied is given in Table 2.

2.3. Specimen preparation

2.3.1. Mixing of geopolymer paste

The geopolymeric fly ash precursor and alkaline silicate solution were mixed by hand for 10 min and a further 5 min with a mixer. The mixture was then cast into molds of various shapes and sizes and compacted in three layers of equal height. The samples were then vibrated to release any residual air bubbles. Subsequently, the molded samples were sealed with a film and transferred to an air-tight container to prevent moisture loss. Each sample batch was prepared in duplicates. Twelve samples were prepared for each study (i.e. 6 unexposed and 6 for elevated temperature exposure).

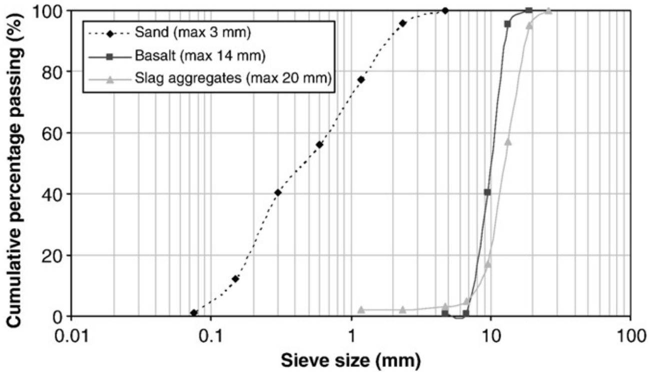


Fig. 1. Grading properties of coarse and fine aggregates.

Table 2
Mixture proportions of geopolymer paste, mortar and concrete test series.

Mix no	Size/type of aggregate	Unit weight (kg)		
		Coarse aggregates	Fine aggregates	Superplasticizer
1	None	0	0	0
2	None	0	15	0
3	Graded basalt[a]	22.5	15	0
4	2.37–5 mm basalt	22.5	15	0
5	5–10 mm basalt	22.5	15	0
6	10–14 mm basalt	22.5	15	0
7	20 mm basalt	22.5	15	0
8	Graded slag aggregates[b]	22.5	15	0
9	Graded basalt[a]	22.5	15	0.28[c]
10	Graded basalt[a]	22.5	15	0.1[d]

FA = 8.4 kg, Na_2SiO_3 = 2.0 kg, KOH = 0.8 kg (constant for all Mixes 1–10).
[a] Max size 14 mm (refer to Fig. 1).
[b] Max size 20 mm (refer to Fig. 1).
[c] Glenium 27.
[d] Rheobuild 1000.

2.3.2. Mixing of geopolymer mortar and concrete

The fly ash and aggregates (coarse and/or fine) were first dry mixed in a laboratory concrete pan mixer for 3 min. The alkaline solutions used for synthesis were then added into the blend and mixed for a further 7 min before being cast into standard 100 mm diameter × 200 mm height cylindrical molds. The cylinders were compacted in three layers of equal height with standard compaction using rod and vibrating table. The molds were sealed to minimize moisture loss.

2.4. Curing regime

All specimens were cured undisturbed for 24 h at room temperature before being subjected to high temperature curing (80 °C; relative humidity 93%) for a further 24 h in a laboratory grade oven. At the end of the curing regime, the specimens were removed from their molds and allowed to cool before initial physical properties were recorded.

2.5. Elevated temperature heating methods

An electrically-heated furnace designed for a maximum temperature of 1200 °C was used. Specimens were subjected to temperatures of up to 800 °C at an incremental rate of 4.4 °C per minute from room temperature. The temperature was sustained at 800 °C for 1 h before the specimens were allowed to cool naturally to room temperature inside the furnace. Meanwhile, the unexposed samples were left undisturbed at ambient temperature.

2.6. Thermogravimetric analysis and dilatometry measurements

Weight loss was measured as specimens were exposed to gradually increasing temperatures. This thermogravimetric analysis (TGA) was conducted using a Settaram TGA92 device. Powdered specimens, which consisted of sub-samples from the prepared specimens used for strength analysis, were used in TGA to ensure that thermal equilibrium during transient heating was achieved.

Dilatometry measurements were also conducted with specimens of 7.5 mm diameter × 30 mm height that were cored out of paste specimens and aggregate blocks using a core barrel attached to a drill press. Each specimen was then placed into a vertical dilatometer tube using a push rod inserted into the axis of the tube. One end of the push rod rested on the specimen while a dial gauge rested on the other end, allowing linear changes in the length of specimen to be recorded. As the push rod was made of fused silica material, its thermal expansion did not have a significant effect on the thermal expansion of the specimen overall. The specimens were heated up to 800 °C under a stepwise heating program then allowed to reach thermal equilibrium at selected temperatures before length changes were recorded.

3. Results and discussions

3.1. Strength of geopolymer: paste, mortar and concrete

Compressive strength tests of all specimens were conducted using an Amsler compressive strength device in load control regime with a loading rate of 20 MPa/min. A minimum of six specimens (100× 200 mm cylinders) for each type were tested for 3-day compressive strengths after casting, which is equivalent to a typical OPC strength development after 28-days [9]. It has been shown [10,11] that age does not have a significant effect on strength of geopolymers after the completion of the heating curing cycle. A summary of air content, density and compressive strengths results are given in Table 3. The strength of the paste, mortar and concrete before exposure were approximately the same. However, it was found that the paste specimens underwent 73.4% strength loss after elevated temperature exposure. Mortar on the other hand did not retain any residual strength after similar temperature exposure. As the temperature reached 600 °C, the mortar cylinder split into two halves. Conversely, the geopolymer concrete with graded aggregates and fine aggregates displayed a 58.4% strength reduction after similar exposure.

The strength of the existing fly ash-based geopolymer paste is not significantly increased by the inclusion of aggregates because the paste itself undergoes high early strength development at an accelerated rate. This behavior is characteristic of the quick geopolymerization process [12] which contrasts with the hydration process of OPC that gains strength over longer time periods [13]. However, the inclusion of aggregates in the paste affects performances at elevated temperatures. This is further discussed in Section 3.4.

A further series of tests was conducted on paste made from the same mixture composition, but cast into molds of several different dimensions. The results of these tests are reported in Fig. 2. Paste specimens cast into 25×25×25 mm cube molds recorded a 6.4% strength gain, whereas similar paste cast into 35×70 mm and 100×200 mm cylindrically-shaped molds recorded a strength loss of 52.3% and 73.4% respectively. Results show that exposure to elevated temperatures affect larger specimens more adversely. The strength gain phenomenon after temperature exposure, as observed in the small specimens, was no longer evident in the larger cast specimens. Heat conduction decreases significantly as specimen size increases. Therefore, there is a greater difference in thermal gradient between the hotter exterior and cooler interior of these larger specimens. Thermal gradients increase with the increasing specimen size, which consequently induces thermal cracking.

Fig. 3 shows the temperature–time profile at different distances from the heated surface of a typical 100×200 mm fly ash geopolymer paste cylinder during heating. Maximum thermal gradient between the surface exposed to heating and centre thermocouple reading was observed to occur during the 70 to 120 min. This clearly shows that the distribution of temperature within the cross section is not uniform and caused a thermal gradient to exist. This thermal gradient is likely to have adverse effects on the integrity of the geopolymer paste at elevated temperatures.

Table 3
Mean values for strength of geopolymer paste, mortar and concrete.

Mix	Type	Air (%)	Density (kg/m^3)	3-day strength (MPa)	
				Unexposed	Temp exposed
1	Paste	2.35	1940.4	71.2	19.0
2	Mortar	2.30	2291.8	72.3	0.0[a]
3	Concrete	2.40	2575.8	70.5	29.3

[a] No strength were recorded as all mortar specimens cracked and split.

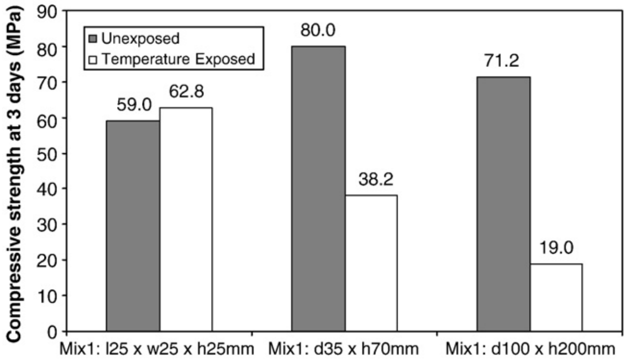
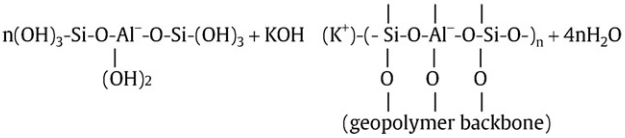


Fig. 2. Effect of specimen size on the geopolymer paste behavior.

The schematic formulation of geopolymer material can be shown in Eq. (1) [12,14]. The last term in this equation shows that water is released during the formation of geopolymers. This water, expelled from the geopolymer matrix during heat curing, causes discontinuous nano-pores within the matrix, which improves the strength of geopolymers. However, not all moisture will be expelled from the geopolymer matrix especially in larger specimens. Higher surface tensions in large specimens tend to cause trapped moisture to dissipate at slower rates compared to smaller specimens. A combination of these two behaviors is the most likely cause of variability in temperature resistance due to size.



$$n(OH)_3\text{-Si-O-}\underset{\displaystyle (OH)_2}{\underset{|}{Al^-}}\text{-O-Si-}(OH)_3 + KOH \quad (K^+)\text{-(-}\underset{\displaystyle |}{\underset{\displaystyle O}{\underset{|}{\overset{|}{Si}}}}\text{-O-}\underset{\displaystyle |}{\underset{\displaystyle O}{\underset{|}{\overset{|}{Al^-}}}}\text{-O-}\underset{\displaystyle |}{\underset{\displaystyle O}{\underset{|}{\overset{|}{Si}}}}\text{-O-)}_n + 4nH_2O \qquad (1)$$

(geopolymer backbone)

An additional study was conducted to compare the behaviors of geopolymer and OPC under elevated temperatures. The strength of geopolymer paste specimens observed in this study was compared to the equivalent OPC paste findings of Mendes et al. [15] as shown in Fig. 4. The strength of geopolymer paste specimens increased as temperature increased, attaining a peak strength of 77 MPa at 300 °C. Subsequently, this strength was observed to deteriorate gradually for the remainder of the heating regime. However, a final strength of 58 MPa was recorded after the 800 °C temperature exposure, which

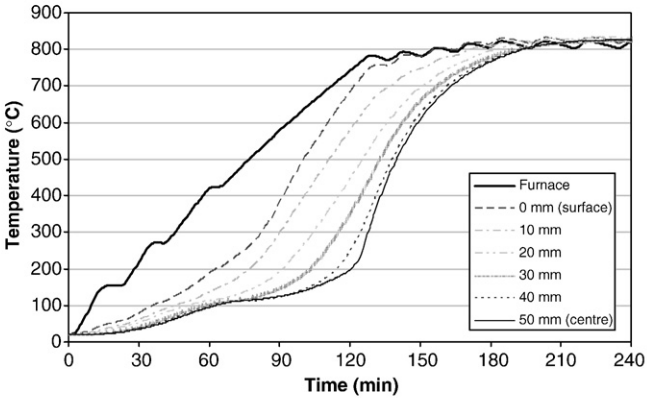


Fig. 3. Measured temperature–time profile of 100×200 mm fly ash geopolymer cylinders at various distances from heated surface.

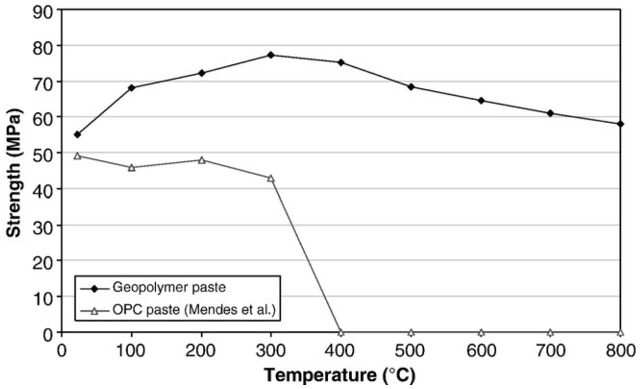


Fig. 4. Compressive strength of geopolymer and OPC [15] pastes at various temperatures.

was still a 5.4% improvement over the strength of the reference specimen. On the other hand, the strength of OPC specimens dropped considerably between temperatures of 400 °C and 800 °C. This strength deterioration is attributed to the $Ca(OH)_2$ decomposition that occurs at about 400 °C [16]. In contrast, geopolymer concrete (Table 3) and OPC concrete [17] behave very similarly when exposed to elevated temperatures, with both concretes showing strength deterioration after exposures. This strength loss is influenced by the difference in thermal strain of paste and aggregate of the concrete which takes over the strength gain behavior of geopolymer paste or stress loss behavior of Portland cement paste. Further investigation of this issue is presented in Section 3.4.

3.2. Effect of coarse aggregate size variation in geopolymer concrete

The results of the compressive strength test on the geopolymeric composites are summarized in Fig. 5, which shows the geopolymer composites made with aggregate sizes of below 10 mm spalled when exposed to elevated temperatures. Concretes made with aggregate sizes between 2.36–5 mm and 5–10 mm spalled at 420 °C and 505 °C respectively. A violent outward explosion was recorded at these temperatures. Specimens split into several smaller pieces but the bulk of it remained intact. Meanwhile, a consistent 61.8% strength loss was recorded for concretes made with 10–14 mm and 20 mm sized-aggregates.

These results show that aggregate size affects strength performances of geopolymer concrete at elevated temperatures and are consistent with earlier observations with mortar specimens.

In addition, the concrete made with evenly graded mix in sizing of coarse aggregates (as indicated in Fig. 1) were able to perform better at elevated temperatures. A comparison of strength results from Table 3 and Fig. 5 shows that concretes produced with graded coarse aggregate (Mix 3) underwent a strength reduction of 58.4%, which compares favorably to concretes made with one constant aggregate size (Mixes 4–7).

The introduction of aggregates was expected to increase the strength of geopolymer through the reduction of pores, prevention of cracks and as well as contribution from the strength of the aggregate itself. However, the aggregate, which has higher stiffness than the geopolymer paste, restrains the matrix shrinkage. This restraining effect causes radial and tangential stresses around the aggregate particles [18]. The shrinkage resulting from moisture loss enhances these stresses, leading to the development of extensive cracks. This shrinkage behavior is clearly displayed in concretes made with smaller aggregates. The lack of irregular shapes in smaller aggregates limit the production of effective aggregate bridging.

3.3. Effect of supersplasticizer in geopolymer concrete

Superplasticizers are commonly used as an additive because of its known ability to increase workability of fresh concrete. This section studies the effects of superplasticizers on the workability and compressive strengths of fly ash-based geopolymer concrete. The strength results of geopolymer with the addition of superplasticizer (Mixes 9 and 10) are shown in Fig. 6. The strengths of the geopolymer concrete mixed purely with the silicates and hydroxides were consistently higher than those with addition of superplasticizer. The addition of superplasticizer was observed to improve the workability of the fresh concrete marginally; however, it had significant detrimental effects on the compressive strength of the original concrete. Strength degradations observed were dependent on the type of supersplasticizer used. Glenium 27 caused a 54% strength degradation from the original strength of the concrete while Rheobuild 1000 caused a 21.8% drop in strength. Hardjito et al. [10] reported that the use of superplasticizer beyond 2% of the mass of the fly ash will likely cause deterioration in compressive strengths.

Further, after elevated temperature exposure, the specimens containing Rheobuild 1000 recorded the highest drop in strength i.e. 81%, whereas strength loss for Glenium 27 specimens was significantly lower at 28%. Superplasticizers do not improve workability of fly ash-based geopolymer significantly as the fly ash particles itself are spherically-shaped [19], which allows easy dispersal within the Na_2SiO_3 and KOH environment. Superplasticizer therefore is not beneficial in reducing water content as observed in the case of OPC concrete. In this case, the solid component as well as the integral water of the superplasticizer consistently lowered the strength of the concrete.

3.4. Effect of coarse aggregates in geopolymer concrete

A summary of compressive strengths is given in Table 4. Comparatively, the compressive strength of fly ash-based geopolymer paste was higher than those recorded in the fly ash-based geopolymer

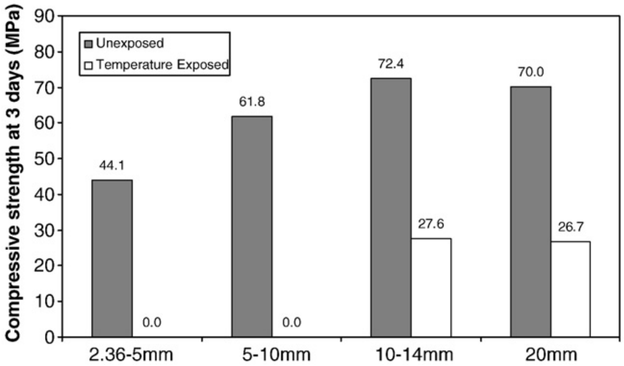


Fig. 5. Effect of aggregate size on the behavior of geopolymer concrete.

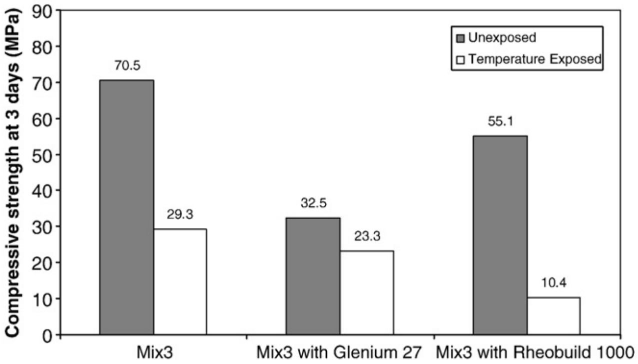


Fig. 6. Effect of the type of superplasticizer on behavior of geopolymer concrete.

Table 4
Variation in aggregate type.

Mix	Coarse aggregate type	Air (%)	Density (kg/m^3)	Slump (mm)	3-day strength (MPa)	
					Unexposed	Temp exposed
3	Basalt	2.35	2575.8	10	70.5	29.3
8	Slag	2.30	2616.1	30	61.8	21.9

concrete. The introduction of aggregates (both coarse and fine) to the geopolymer paste resulted in decreased strengths at ambient temperature from 71.2 MPa to 70.5 MPa and 61.8 MPa for basalt and slag aggregates respectively. Additionally, the strength of the geopolymer concretes further decreased after exposure to elevated temperatures. The results show that the basalt-based concrete experienced a 58.4% drop in strength while the slag aggregate-based concrete underwent a 64.6% drop after temperature exposure. Additionally the workability of fresh slag aggregate-based concrete improved over the basalt-based concrete.

In geopolymers, aluminosilicate gel is the major binding phase that provides interparticle bonding, which in turn enhances the macroscopic strength [20]. The variation in performances of the two aggregates is primarily caused by the differential thermal expansion between the aggregate and paste. Thermogravimetric analysis and dilatometry measurements were conducted to study this behavior under elevated temperature.

Fig. 7 shows the results of the TGA/DTG analysis of the geopolymer paste (Mix 1). The sharp decrease in weight before 250 °C as recorded in the TGA thermogram is attributed to the loss of evaporable water in the geopolymer. The decrease in weight peaked at 127 °C as indicated by the DTG curve. After the initial rapid decline, the geopolymer's rate of weight loss stabilized between 250 °C and 700 °C. There was little change in the percentage of weight remaining beyond 700 °C. The average total percentage of weight remaining after 800 °C exposure was 89%.

The strain behavior of geopolymer paste is presented in Fig. 8. Minor expansions were observed in the geopolymer as the temperature increased to 150 °C. The geopolymer did not experience any expansions between 150 °C and 220 °C, however substantial shrinkage occurred as the temperature increased to 320 °C. This drastic shrinkage is linked to majority of the thermal damage sustained by the geopolymer paste and is therefore considered the most critical aspect of this strain behavioral study. Rahier et al. [21] attributed dehydroxylation as the cause of shrinkages in geopolymer below 300 °C while Duxson et al. [22] hypothesize that shrinkage is related to the mass loss in the form of evaporated free pore water. Further, the slow rate of thermal shrinkage between 320 °C and 700 °C could be related to the low rate of weight loss observed in the thermogravimetric data recorded in Fig. 7. Thermal expansion data of basalt and slag aggregates for temperatures up to about 800 °C are also represented in Fig. 8. Observations show that minimal damage occurred as the aggregates and geopolymer paste both underwent thermal expansion and are therefore compatible during the initial stages of heating.

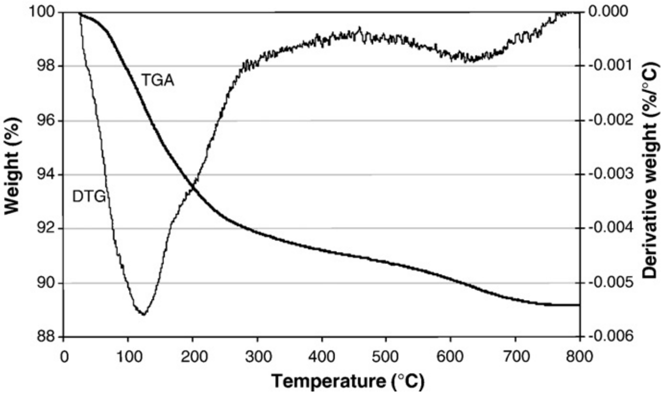


Fig. 7. TGA and DTG of fly ash geopolymer paste.

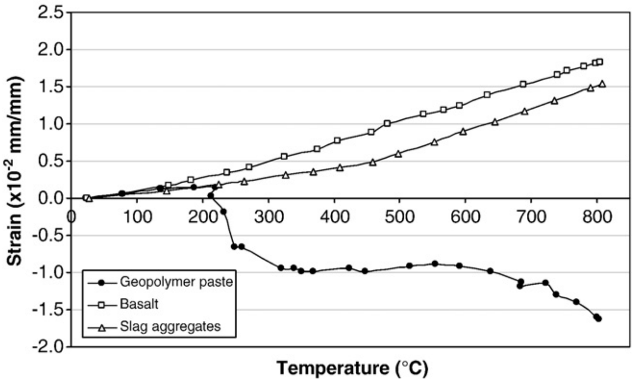


Fig. 8. Thermal expansion of coarse aggregate and paste.

The strain of geopolymer/aggregate composites is strongly linked to the expansion of aggregates, as aggregates generally occupy 75–80% of the volume of concrete. Therefore, when subjected to temperatures between 220 °C and 320 °C, the expansion of aggregates dominates over the shrinkage of the geopolymer paste leading to a net result of expansion in the concrete.

These collective results and discussions support the hypothesis that strength loss in geopolymer and geopolymer/aggregate concrete specimens at elevated temperatures is primarily caused by thermal incompatibility between the geopolymer matrix and its aggregate components. Slag aggregates should theoretically perform better than basalt aggregates when exposed to elevated temperatures, as slag aggregates are able to match the thermal expansion of the geopolymer paste more closely. However, results in Table 4 show otherwise. The authors hypothesize that this is attributed to the low strength characteristics of the slag aggregates.

4. Conclusions

Based on the obtained data, it can be concluded that:

1. Elevated temperature strength is dependent on the size of the geopolymer paste specimens. The significant temperature difference between the surface and core of the specimen cross-section induces thermal cracking. Therefore, thermal incompatibility arising from thermal gradient is likely to be the reason for the size effects.
2. The size of the aggregates is an important factor in determining the geopolymer concrete behavior under elevated temperatures. Smaller sized aggregates (<10 mm) promote spalling and extensive cracking in the geopolymer concrete while geopolymer concretes containing larger aggregates (>10 mm) are more stable in elevated temperatures.
3. Conventional superplasticizer commonly used with Ordinary Portland Cement, when being applied as an additive to the binder, does not significantly improve the overall workability of the mix. On the contrary, it deteriorates the strength of the geopolymer matrix. Elevated temperature performance in concrete with superplasticizer is also poor. The use of superplasticizers is not beneficial in geopolymer concrete for elevated temperature performance.
4. The thermal incompatibility between the geopolymer matrix and its aggregate components is the most likely cause of strength loss in geopolymer concrete specimens at elevated temperatures. This is proven by comparison of geopolymer concretes made two different aggregates with distinctly different thermal expansion characteristics, where the sample with greater incompatibility led to higher strength

loss. The rate of expansion of the aggregate with temperature is an influential factor in the performance of geopolymer concrete under elevated temperatures.

Acknowledgements

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