



Damage behavior of geopolymer composites exposed to elevated temperatures

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ABSTRACT

This paper presents a study on geopolymers and geopolymer/aggregate composites made with class F fly ash. Samples were heated up to 800 °C to evaluate strength loss due to thermal damage. The geopolymers exhibited strength increases of about 53% after temperature exposure. However, geopolymer/aggregate composites with identical geopolymer binder formulations decreased in strength by up to 65% after the same exposure. Test data from dilatometry measurements of geopolymers and aggregates provides an explanation for this behavior. The tests show that the aggregates steadily expanded with temperature, reaching about 1.5–2.5% expansion at 800 °C. Correspondingly, the geopolymer matrix undergoes contraction of about 1% between 200 °C and 300 °C and a further 0.6% between 700 °C and 800 °C. This apparent incompatibility is concluded to be the cause of the observed strength loss. This study presents the results of 15 different geopolymer combinations (i.e. mixture proportions, curing and age) and four different aggregates.

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1. Introduction

Ordinary Portland Cement (OPC) is the main ingredient used in the production of concrete – the most widely used construction material in the world. In the past, concrete was simply a composite of OPC paste with aggregates, however, modern-day concrete incorporates other cementitious materials which act as partial replacements of OPC. The manufacturing of OPC requires the burning of large quantities of fuel, and decomposition of limestone. Both, burning of fuel and decomposition of limestone, result in significant emissions of carbon dioxide. For every ton of OPC manufactured, nearly one ton of CO₂ is produced depending on the production process adopted [1]. Cement plants are reported to emit up to 1.5 billion tons of CO₂ into the atmosphere annually [2,3]. Hence, environmental preservation has become a driving force behind the search for new sustainable and environmentally friendly composites to replace conventional concrete produced from OPC.

In 1978, Davidovits [4] introduced the word ‘geopolymer’ to describe an alternative cementitious material which has ceramic-like properties. As opposed to OPC, the manufacture of fly ash-based geopolymer does not consume high levels of energy, as fly ash is already an industrial by-product. This geopolymer technology has the potential to reduce emissions by 80% [1] because high temperature calcining is not required. It also exhibits ceramic-like properties with superior resistance to fire at elevated tempera-

tures. Geopolymer can be produced by combining a pozzolanic compound or aluminosilicate source material with highly alkaline solutions [5]. Fly ash, which is available abundantly worldwide from coal burning operations, is an excellent aluminosilicate source material. In Australia, fly ash is currently underutilized; according to figures in the year 2000, 12 million tons per annum were produced but only 10% were effectively utilized in cementitious applications [6].

Lately, much research [7–10] has highlighted the potential use of fly ash-based geopolymer in cement and concrete production. Geopolymers have also been shown to have good bond strength to OPC concrete, hence it is a good repair material with superior abrasion resistance [11]. Fly ash-based geopolymer/aggregate composites have superior performance compared to OPC concrete in certain areas such as resistance to sulfate attacks [12,13], and have lower creep [14] and shrinkage [7] than conventional concrete. Geopolymers are generally believed to perform better than the conventional concretes in fire, due to their ceramic-like properties [15,16]. However, published research in fly ash-based geopolymer is limited in the area of fire resistance and responses to elevated temperatures. Given that geopolymer increases in strength after elevated temperature exposure [17], the fire resistance of geopolymer is likely to be superior to OPC concrete which loses most of its strength after elevated temperature exposure about 800 °C [18].

Though research on geopolymer as a binder has been plentiful, the study of its behavior under elevated temperature conditions is still lacking. This study investigates a number of composition and processing conditions on strength and heat resistant properties

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of geopolymer binder prepared using class F fly ashes for temperature exposures of up to 800 °C. It also investigates the effect of elevated temperature exposure on geopolymer/aggregate composites.

2. Materials characterization and specimen preparation processes

2.1. Source materials

Fly ash is the aluminosilicate material utilized in the synthesis of geopolymeric binder in this investigation. The low calcium (class F) fly ash was sourced from Pozzolanica Gladstone in Queensland. It is mainly glassy with some crystalline inclusions of mullite, hematite and quartz. The breakdown of its chemical composition is shown in Table 1. Laboratory grade D sodium silicate solution (Na_2SiO_3) with $\text{Na}_2\text{O}/\text{SiO}_2 = 2$ was procured from PQ Australia while the potassium hydroxide (KOH) flakes of 90% purity supplied by Orica Chemicals was prepared to a molarity of 7.0 M. Two different coarse aggregates were used to make geopolymer/aggregate composites, namely, crushed basalt and slag aggregates, both with a maximum aggregate size of 14 mm. Fine aggregates which comprised of Lyndhurst sand had a fineness modulus of 1.82.

2.2. Specimen preparation and mixture compositions

2.2.1. Geopolymer

Geopolymer was made by hand-mixing fly ash with the alkaline solution by for 10 min and a further 5 min with an industrial mixer. All investigations involved a combination of Na_2SiO_3 and KOH (molarity of 7.0 M). A summary of the mixture compositions are tabulated in Table 2. The solutions used to activate the fly ash were prepared one day prior to usage. The paste mixture was cast into $25 \times 25 \times 25$ mm cubic-shaped moulds, vibrated for compaction and sealed with a lid to minimize any loss of evaporable water.

2.2.2. Geopolymer/aggregate composite

The same type of geopolymer used in the previous section was used to produce geopolymer/aggregate composites, however, the fly ash-to-activator and $\text{Na}_2\text{SiO}_3/\text{KOH}$ ratios were kept constant at 3.0 and 2.5, respectively. Total aggregates made up 77% of the geopolymer/aggregate composites. Of the total aggregates used, 60% comprised of coarse aggregates while the remaining 40% were fine aggregates. Details of the mixture composition are summarized in Table 3.

The fly ash and aggregates were firstly dry-mixed in a concrete laboratory pan mixer for 3 min. Subsequently, the alkaline solu-

Table 1
Chemical composition of fly ash using XRF

Chemical	Component (%)
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*	1.7

* Loss on ignition.

Table 2
Geopolymer mixture combinations

Mixture no.	$\text{Na}_2\text{SiO}_3/\text{KOH}$ ratio	FA/alkaline ratio	Curing regime	Paste age (days)
1	0.5	3.0	80 °C (24 h)	3
2	1.0	3.0	80 °C (24 h)	3
3	1.5	3.0	80 °C (24 h)	3
4	2.0	3.0	80 °C (24 h)	3
5	2.5	3.0	80 °C (24 h)	3
6	1.0	3.0	80 °C (24 h)	7
7	1.5	3.0	80 °C (24 h)	7
8	2.0	3.0	80 °C (24 h)	7
9	1.0	3.0	80 °C (72 h)	7
10	1.5	3.0	80 °C (72 h)	7
11	2.0	3.0	80 °C (72 h)	7
12	1.0	2.0	80 °C (24 h)	3
13	1.5	2.0	80 °C (24 h)	3
14	2.0	2.0	80 °C (24 h)	3

Table 3
Geopolymer/aggregate composite mixture composition

Mixture no.	Coarse aggregates (kg/m^3)	Fine aggregates (kg/m^3)	Fly ash (kg/m^3)	Na_2SiO_3 (kg/m^3)	KOH (kg/m^3)	Curing regime
1	1125 (Basalt)	750	420	100	40	80 °C (24 h)
2	1125 (Slag)	750	420	100	40	80 °C (24 h)

tions used for synthesis were added to the blend and mixed for a further 7 min before being cast into standard 100 mm diameter \times 200 mm height cylindrical moulds. The cylinders were compacted in three layers of equal height with normal rodding and vibration. Again, the moulds were sealed at the top to minimize water loss.

2.3. Curing regime

Geopolymer and geopolymer/aggregate composite specimens were left to cure undisturbed under ambient temperature for 24 h before being subjected to an elevated temperature of 80 °C for an additional 24 h. At the end of the curing regime, the specimens were removed from their moulds and allowed to cool before any physical properties were recorded; these included dimension and mass measurements.

2.4. Elevated temperature exposure regime

At the age of 2 days, the specimens were subjected to temperatures of up to 800 °C at a gradual incremental rate of approximately 5 °C/min from room temperature. As soon as the target temperature (800 °C) was attained, it was maintained for an additional 60 min before the furnace was shut down to allow the specimens in the furnace to cool down to room temperature. This cooling usually takes more than 12 h. In the meantime, a set of the concurrent counterparts were left undisturbed at room temperature for comparative study.

3. Results and discussions

3.1. Behavior of geopolymers at elevated temperature

The geopolymer specimens were tested for 3-day and 7-day strengths after casting using a Mohr and Federhaff AG mechanical testing machine in load control regime with a loading rate of 20 MPa/min.

The studied geopolymer showed rapid hardening after 1-day room temperature curing and did not exhibit noticeable dimensional change. The volume of the specimen decreased by only 2% after temperature exposure. Furthermore, observations showed no visible cracks and an average mass reduction of 13%, which was significantly lower than that of the metakaolinite-based paste typically characterized by a 30% reduction [19].

Some of the parameters studied in accordance to the strength and elevated temperature performances of the geopolymers include the effect of the sodium silicate-to-potassium hydroxide ratio, the binder-to-liquids ratio, the age of binder and curing periods. These findings are summarized below.

3.1.1. Effect of sodium silicate-to-potassium hydroxide ratio

Fig. 1 shows the 3-day compressive strength measurements for fly ash-based geopolymer prepared at various $\text{Na}_2\text{SiO}_3/\text{KOH}$ ratios ranging from 0.5 to 2.5. The constant curing temperature selected was 80 °C. The strength of the geopolymer significantly improved as the ratio increased. An increase in $\text{Na}_2\text{SiO}_3/\text{KOH}$ ratio results in an increase of Na content in the mixtures which in turn produces more stable strength properties. Rapid strength development occurs within geopolymer with higher sodium concentrations especially within the Na_2SiO_3 [20]. However, the compressive strength is expected to decrease as more silicate is added into the system. This is because excess sodium silicate hinders water evaporation and structure formation [21]. Another interesting observation is that the strength of the geopolymer specimens unexpectedly increased when subjected to elevated temperature exposure. This increase of compressive strength is attributed to the low diffusion coefficient of K^+ at elevated temperatures which result in a higher melting temperature of the geopolymer [15].

3.1.2. Effect of fly ash-to-alkaline silicate activator ratio

A strength performance comparison in terms of fly ash-to-activator ratio was made to replicate water-to-cement ratio typically used to quantify compressive strength in OPC. The total mass of the liquid activators is a combined mass of both Na_2SiO_3 and KOH liquids. There was a general decrease in strength when the amount of activator introduced into the system was increased (reducing the FA/activator ratio from 3.0 to 2.0), as shown in Fig. 2.

The solids/liquids ratio contributes to the porosity level of the hardened geopolymer. Increased porosity causes a decline in strength [17,22]. Voids in concrete can be filled with air or water. Since, the solids/liquids ratio affects the volume of voids in the geopolymers, it indirectly influences the strength of the geopolymer.

However, it was found that the strength of the geopolymer with the lower fly ash-to-activator ratio (FA/activator = 2.0) decreased after elevated temperature exposure, unlike previous findings where strength increased after exposure (FA/activator = 3.0).

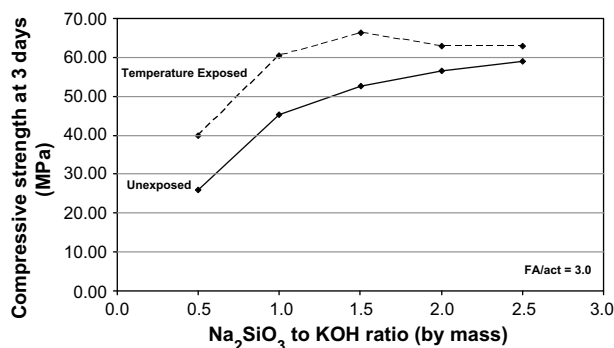


Fig. 1. Effect of sodium silicate-to-potassium hydroxide ratio on compressive strength.

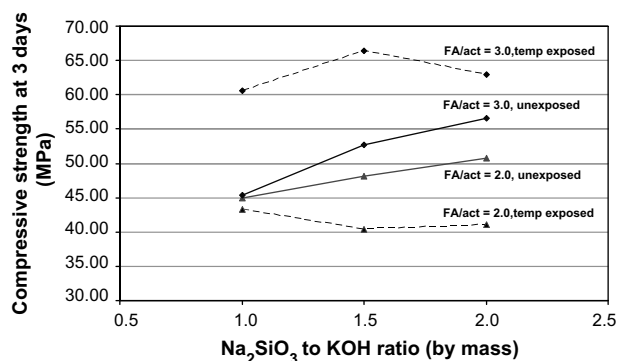


Fig. 2. Effect of fly ash-to-activator ratio on compressive strength.

According to Barbosa et al. [23], the excess water content from the increased activator content will dilute the reaction of the geopolymer. This reduced geopolymer synthesis is inadequate for elevated temperature resistance.

3.1.3. Effect of the age of geopolymer

In order to study the effects of geopolymer age on compressive strengths, the geopolymers were prepared at various $\text{Na}_2\text{SiO}_3/\text{KOH}$ ratios and tested for 3-day and 7-day strengths. From Fig. 3, it can be seen that there was a small increase in strength when the age of specimens were extended.

The chemical reaction of the geopolymer is a rapid polymerization process; geopolymer gains strength – about 70% of its strength is gained within the first 3–4 h of curing [5,24,25]. The compressive strength does not vary with the age since the specimens underwent accelerated heat curing for 24 h. This observation is in contrast to the well-known behavior of Portland cement, which experiences strength gain over time [26].

3.1.4. Effect of curing period

Results in Fig. 4 indicated that a longer heat curing time did not significantly affect strength performances. The authors believe most geopolymerization of fly ash binder was complete within the first 24 h of heat curing. van Jaarsveld et al. [27] claimed that curing for longer periods of time at elevated temperatures appeared to weaken the structure. However, the experimental findings in this section proved otherwise within the time frames investigated.

The authors have also considered the effect of delay time between production and heat curing of specimens which have been studied in detail by previous researches [20,28]. Chindaprasirt et al. [28] reported that a delay time between production and curing of specimens did not have a huge impact on strength on class C activated fly ash. On the contrary, Bakharev [20] suggested that a

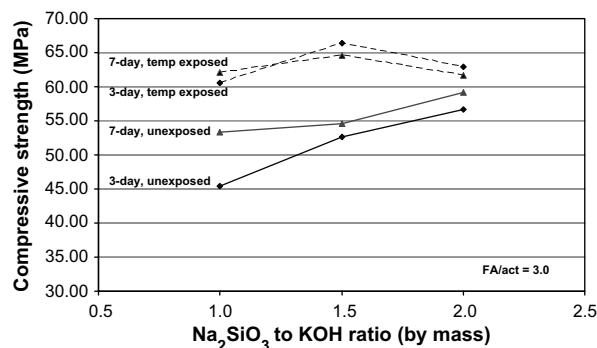


Fig. 3. Effect of geopolymer age on compressive strength.

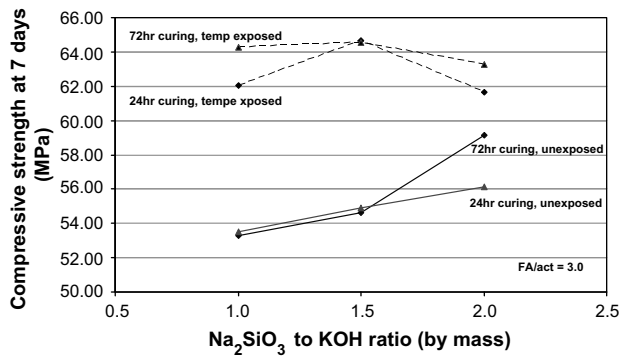


Fig. 4. Effect of curing period on compressive strength.

delay time of 24 h was an important factor for the strength development of class F activated fly ash.

3.2. Behavior of geopolymer/aggregate composites at elevated temperature

Physical observations showed that all specimens reacted well to elevated temperature exposure. Apart from decolorization, which was similar to the effect of that observed in geopolymers, there was no visible spalling observed in any of the geopolymer/aggregate composite cylinders.

Compressive strengths of the composites were conducted using an Amsler compressive strength device in load control regime with a loading rate of 20 MPa/min. These specimens were tested for strengths 3 days after casting. A summary of compressive strengths is given in Table 4. Comparatively, the compressive strengths of composites were higher than those recorded for geopolymers. The introduction of basalt aggregates to the geopolymer increased the strengths from 59.0 MPa to 70.5 MPa. For slag aggregates under the same conditions, the strength increase was from 59.0 MPa to 61.8 MPa. However, the strength of geopolymer/aggregate composites decreased after exposure to elevated temperatures. As can be seen in Table 4, after the temperature exposure, the specimens were weaker than their unexposed counterparts. The results show a 58% strength decrease in basalt-composite and a 65% drop in slag-composite after elevated temperature exposure. The phenomenon of temperature-exposed strengths being higher than unexposed strengths as previously displayed by the geopolymers no longer exists.

Since the performance of geopolymers at elevated temperatures declined with the inclusion of aggregates to make composites, it was hypothesized that differential thermal expansion between the aggregates and geopolymer is the cause of after exposure strength deterioration. To further investigate this hypothesis, the thermal contraction/expansion characteristics of both geopolymer and aggregate were studied.

3.3. Thermal contraction/expansion of geopolymer and aggregate

Thermogravimetric Analysis (TGA) was conducted using a Setaram TGA92 device. In this test, the mass loss was measured while

Table 4
Geopolymer/aggregate composite strength results

Mix	Coarse aggregate type	Air (%)	Density (kg/m ³)	Slump (mm)	3-Day strength (MPa)	
					Unexposed	Temp exposed
1	Basalt	2.4	2576	10	70.5	29.3
2	Slag	2.3	2616	30	61.8	21.9

the specimens were gradually exposed to increasing temperatures. Powdered specimens were used in TGA to ensure achievement of thermal equilibrium during transient heating.

Dilatometry measurements were also conducted to understand the incompatibility of geopolymer with aggregates. Specimens of 7.5 mm diameter × 30 mm height were cored out using a core barrel attached to a drill press. Each specimen was then inserted into a vertical dilatometer tube with a push rod inserted in the axis of the tube. One end of the push rod rested on the specimen while a dial gauge rested on the other end; making it easy to record linear changes in the length of specimen. The push rod was made of fused silica material; therefore, there was minimal thermal expansion due to the push rod. The specimens were heated up to 800 °C under a stepwise heating program; the sample was allowed to come to a thermal equilibrium at selected temperatures before changes in length were recorded.

One of the two curves on Fig. 5 shows the length change, Δl , of the geopolymer with respect to its original length, l_0 . Small expansions were observed in the geopolymer when the temperature increased up to about 150 °C. When the temperature was between 150 °C and 220 °C, no further expansion were observed. The geopolymer specimen then underwent substantial shrinkage between 220 °C and 300 °C. This thermal shrinkage is the most important aspect of the curve, and is responsible for the damage observed due to thermal incompatibility. Beyond 300 °C, no shrinkage or expansions were recorded until 700 °C. Between 700 °C and 800 °C, further increases in shrinkage were observed. Shrinkage occurred due to mass loss in geopolymer when subjected to elevated temperature. The TGA was able to measure mass loss as a function of temperature as displayed by the thermogravimetric curve in Fig. 5. This mass loss is believed to be entirely caused by loss of water from the geopolymer. Fig. 6 shows the differential thermogravimetric (DTG) curve of the previous TGA curve in Fig. 5. Rapid rate of mass loss (likely to be loss of water) occurred, peaking in the 120–130 °C temperature range. The average total percentage of mass remaining after being heated to 800 °C was 89%. All specimens experienced a rapid decline in percentage of mass remaining within the first 200 °C and all rates of mass loss stabilized between 250 °C and 700 °C. After 700 °C, there was little change in the percentage of mass remaining.

To investigate the thermal compatibility between the aggregates and geopolymer, the thermal expansions for various aggregates originating from typical rocks commonly used during concrete manufacture were investigated. Some of these rocks included local old basalt from Mount Shamrock, granite from Tynong, siltstone from Donnybrook, Victoria, Australia and conventional slag aggregates. The thermal expansions of different rocks for temperatures up to about 800 °C are represented in

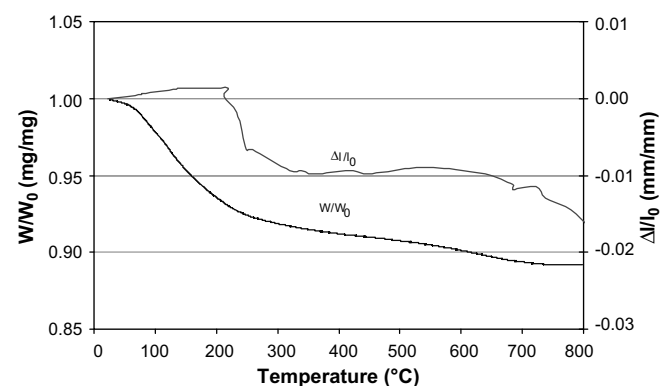


Fig. 5. TGA and dilatometric curves for geopolymer.

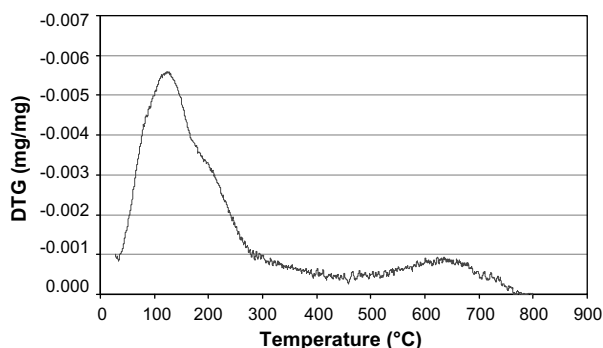


Fig. 6. DTG curve for geopolymer.

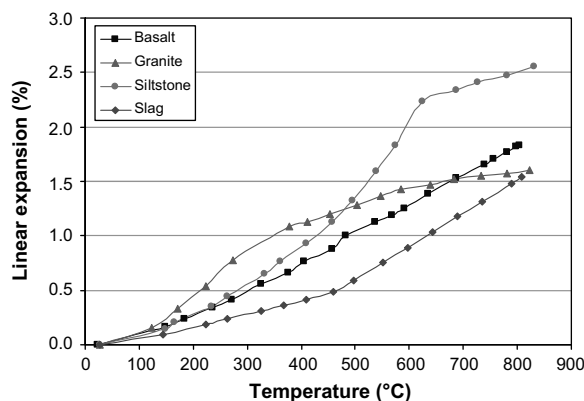


Fig. 7. Thermal expansion of various coarse aggregates.

Fig. 7. As can be seen, all the aggregates expanded with temperature and are not compatible with the curve in Fig. 5. The thermal expansion of geopolymer/aggregate composite at elevated temperatures is strongly affected by the expansion of aggregates because aggregates generally occupy 75–80% of the volume of composite. The expansion of aggregates usually predominates over the contraction of the geopolymer subjected to temperatures between 220 °C and 300 °C, which produces a net result of expansion in the composite. Meanwhile, a differential thermal expansion exists between the aggregates and the geopolymer. The results support the hypothesis that the primary reason for observed strength loss at elevated temperatures of geopolymer and geopolymer/aggregate composite specimens is the thermal incompatibility between geopolymer matrix and aggregate components of the mix.

4. Conclusions

This paper presents results on the behavior of geopolymer and geopolymer/aggregate composites when exposed to elevated temperatures. The experimental and analytical studies led to the following conclusions:

1. The fly ash-to-activator ratio appeared to be the most critical parameter with regards to general strength and fire resistance of the geopolymer paste. Other parameters such as $\text{Na}_2\text{SiO}_3/\text{KOH}$, binder age and curing period had little effect on initial strengths. Within the compositions studied, the optimal composition combination for elevated temperature performance was achieved with $\text{Na}_2\text{SiO}_3/\text{KOH} = 2.5$ and $\text{FA}/\text{activator} = 3.0$. Clearly from the parametric study conducted, fly ash-based geopolymer displayed increase in strength after temperature expo-

sure. The authors believe that this is attributed to the increase in a combination of polymerization reaction and sintering at elevated temperatures.

2. The strength of the fly ash-based geopolymer declined with the inclusion of aggregates i.e. geopolymer/aggregate composites. Evidence presented in this paper suggests the decline in strength was caused by the differential thermal expansion between the geopolymer and aggregates. Separate dilatometry tests show that while aggregates undergo expansion at elevated temperatures, the geopolymer matrix experienced contraction.

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