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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 temperatures. 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 Pozzolanic 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 $Na_2O/SiO_2 = 2$ 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 course 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 Na₂SiO₃ 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 Na₂SiO₃/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 ₂ O ₃ | 27.0 |
| SiO ₂ | 48.8 |
| CaO | 6.2 |
| Fe ₂ O ₃ | 10.2 |
| K ₂ O | 0.85 |
| MgO | 1.4 |
| Na ₂ O | 0.37 |
| P ₂ O 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. | Na ₂ SiO ₃ /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 ₂ SiO 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.

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