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## Thermal performance of calcium-rich alkali-activated materials: A microstructural and mechanical study





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#### HIGHLIGHTS

• Computed tomography showed thermally induced changes in pores of concrete.

• Expansion of pores reduced surface cracking via water vapour pressure release.

• Higher Na/Al decreased strength but increased post-heated strength retention.

• Lower Si/Al exhibited the lowest strength but the best thermal stability.

• Thermal performance of alkali-activated slag-fly ash is better than Portland cement.

#### ARTICLE INFO

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### 1. Introduction

Alkali activation of aluminosilicate-based materials such as blast furnace slag, fly ash, or metakaolin (collectively referred to as alkali-activated materials, AAM) can produce a binder with  $\sim$ 80-90% less associated CO<sub>2</sub> emissions and mechanical and durability properties comparable with or exceeding those of Portland cement (PC) [1]. Calcium-free and low-calcium alkali-activated aluminosilicate materials, also known as geopolymers, exhibit excellent resistance to fire and thermal stresses, evidenced by higher strength retention, a reduction in severity of cracking and an absence of spalling at an elevated temperature when compared to traditional PC binders [2]. Some AAM systems, however, exhibit poor thermal performance and fire-resistance [3,4]. The thermal

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#### ABSTRACT

The effects of Si/Al. Na/Al and water/solids ratios on thermal performance of alkali-activated materials (AAM) based on fly ash-slag blends are investigated. Higher Na/Al decreased compressive strength but increased post-heated strength retention and mass loss while reducing cracking at 1000 °C. Lower Si/ Al resulted in lowest initial strength but highest thermal stability, with an increase in strength after exposure to 1000 °C, while a high degree of cracking was observed at higher Si/Al ratio. The effect of w/s on thermal performance was subtle. Computed tomography analysis showed for the first time thermally induced expansion of pores which reduced surface cracking via water vapour pressure release. Thermal performance of alkali-activated materials (AAM) is significantly better than Portland cement (PC) of the same compressive strength because of the very low bound water content. The porosity, pore connectivity and number of pores of the AAM were considerably higher than those in the PC binder. © 2017 Elsevier Ltd. All rights reserved.

behaviour of AAM depends primarily on the chemistry and microstructure of the binder and consequently cannot be generalised across different binder systems [5]. Low-calcium AAM showed enhanced thermal stability due to lower bound water content within the gel when compared with PC [6]; bound water forms an essential part of the calcium silicate hydrate (C-S-H) gel and portlandite (CaOH) microstructure in hydrated PC [7] whereas the sodium aluminosilicate hydrate (N-A-S-(H)) gel formed in lowcalcium AAM activated with sodium silicate or sodium hydroxide contains unbound water inside the pore network (i.e. pore solution) and hydroxyl groups adsorbed to the gel surface [8-10]. Low-calcium AAM systems, however, generally show lower ambient-cured mechanical strength than high-calcium AAM systems [11]. Inclusion of ground granulated blast furnace slag (GGBFS), which enriches the calcium content of the system and promotes formation of an alkali- and aluminium-substituted calcium silicate hydrate (C-(N)-A-S-H) gel or C-(N)-A-S-H/N-A-S-(H) gel blends upon alkali activation[12], has been shown to improve the performance of metakaolin-based AAM below 800 °C [13].

Strong performance at higher temperatures is also attributed to the ability of the binder to form a network of interconnected pores which allow permeation of trapped steam to the surface of the binder. Transport of steam to the surface markedly reduces cracking and spalling by reducing the pressure build-up of water vapour in closed pores [14,15]. Lower thermal conductivity and sintering at elevated temperature has also been reported as reasons for increased fire-resistance properties of AAM compared to PC binders, which show rapid loss of strength at temperatures above 450 °C [2]. With proper AAM formulation and curing, outstanding thermal stability is achieved by retaining the amorphous gel structure at high temperatures [2]. Poorly cured AAM with remnant unreacted alkali and silicates or excess water content, however, exhibit poor thermal stability at elevated temperatures, with gel devitrification above 800 °C driving formation of a variety of crystalline phases [6,16]. Bakharev [4] found that the strength of fly ash-based AAM systems deteriorated at temperatures above 800 °C, in stark contrast to the observed increase in strength with increasing temperature (up to 800 °C). Higher curing pressure was also observed to increase strength at ambient temperature as well as post-heat shrinkage while reducing strength retention at elevated temperatures as a result of reduced porosity [4].

At elevated temperatures, thermally-induced shrinkage of alkali-activated fly ash binders coincides with the thermal expansion of aggregate and results in substantial damage and strength loss (with strength loss proportional to aggregate size) [17,18]. This shrinkage can be reduced, however, by the inclusion of alumina-based inorganic fillers in the binder [19]. Thermal shrinkage of alkali-activated metakaolin binders has been shown to increase with Si/Al ratio, and at lower Si/Al ratio is higher when the alkali cation is sodium compared to potassium [20]. At higher Si/Al, however, differences in charge-balancing alkali cations within the binder induce subtle changes in thermal shrinkage behaviour [20]. Thermal shrinkage of AAM has also been shown to increase with increasing water/solids (w/s) ratio and sodium content [4]. Potassium aluminosilicate hydrate- (K-A-S-(H)) based AAM systems exhibit increased post-heat strength up to 1000 °C but lower ambient temperature strength when compared to N-A-S-(H)-based systems due to the formation of crystalline phases, increased porosity and cracking at higher temperatures in N-A-S-(H) AAM [4].

The performance of alkali-activated fly ash [21] and fibrereinforced alkali-activated metakaolin [22] samples (exhibiting a range of microstructures from porous to solid) were studied under simulated fire conditions. Despite the lower thermal conductivity of porous samples, the fire rating of solid AAM samples was higher. Phases that do not participate in alkali aluminosilicate gel formation have also been shown to affect performance at higher temperatures. Iron-rich phases which have higher thermal expansion compared to the N-A-S-(H) gel have been shown to cause cracking at elevated temperature, however crystalline silica phases were found to have a negligible effect on the thermal performance of alkali-activated fly ash [23].

In addition to thermally-induced chemical and atomic- or nanostructural changes in the gel, thermally-induced microstructural changes dictating porosity and permeability also play a significant role in thermal performance. Neutron pair distribution function (PDF) analysis showed that the alkali aluminosilicate gel ((N,K)-A-S-(H)) structure exhibited only subtle changes after heat exposure up to 1000 °C, and the majority of evaporated water in alkali-activated metakaolin binders results from water present in large pores and associated with hydration of alkali cations [23]. Sintering and densification of the AAM binder at elevated temperatures reduce pore accessibility and limits the ability of traditional gas-adsorption porosimetry (i.e. Brunauer–Emmett–Teller theory) to characterise the pore structure after exposure to elevated temperatures [24]. Micro-computed tomography ( $\mu$ -CT), however, is able to resolve the majority of the pore structure above 30  $\mu$ m in equivalent diameter in both AAM and PC binders exposed to elevated temperature [5].

Despite an abundance of studies examining the thermal performance of low-calcium AAM (using either metakaolin or lowcalcium fly ash precursors), there has been limited previous work examining thermal performance in high-calcium AAM systems based on GGBFS [25-27], and no previous work examining the thermal performance of alkali-activated fly ash-slag blends. Here, we examine the thermal performance of high-calcium AAM systems with fixed CaO content, varying Si/Al, Na/Al and w/s in the reaction mixture. These compositional parameters have been shown to be the main factors dictating the microstructure and mechanical performance of AAM [1], and are varied to investigate the resultant compositional and microstructural (including crystallinity, porosity and permeability) changes and their effects on compressive strength, elastic modulus and other physical properties in these systems at elevated temperature. In particular, a detailed discussion of the influence of porosity, pore connectivity, crack development and pore size on thermal performance within these systems is provided by application of micro computed tomography (µ-CT), thermogravimetric analysis and physical property characterisation. A comparison of the thermal performance of an AAM and a Portland cement binder with the same ambientcured strength and water to solids ratio was also performed. This study provides for the first time a comprehensive investigation of the effect of precursor chemistry, gel composition and microstructure on thermal performance to provide a novel and enhanced understanding of the fundamental interactions which dictate the structure, strength and performance of AAM systems during exposure to elevated temperatures.

#### 2. Materials and methods

GGBFS (hereinafter slag) and a commercially available blend of high-calcium fly ash (based on fly ashes from Gladstone and Callide power stations, Australia) were purchased from Cement Australia (chemical composition displayed in Table 1); Xray fluorescence (XRF) analysis showed a loss on ignition at 1000 °C of both precursors was negligible. A general purpose PC was purchased from Boral Australia.

Activating solutions were produced by dissolving solid sodium hydroxide (analytical grade with 99.9% purity, Chem-Supply, Australia) in water and subsequent addition of sodium silicate solution (Grade D, 9.4 wt% SiO2, 14.7 wt% Na2O and 55.9 wt% H<sub>2</sub>O, molar ratio SiO<sub>2</sub>/Na<sub>2</sub>O = 2, density = 1530 kg/m<sup>3</sup>, viscosity = 400 cps, PQ Australia). Eight AAM samples of differing composition were produced by dry mixing fly ash and slag in a 4:1 ratio before addition of pre-prepared activating solutions (Table 2). Each sample code starts with G then the next number after it is the Si/Al molar ratio and the number in parenthesis is the Na/Al molar ratio. For instance, G2.1(0.65) has a nominal Si/Al ratio of 2.1 and nominal Na/Al of 0.65. A water to solids (w/s) ratio of 0.35 was used unless it was noted after the sample code (e.g. 37 for w/s of 0.37). A PC sample with w/s of 0.35 was cast for comparison. After 10 min mixing using a Hobart mixer, samples were cast into moulds. A vibrating table was used for 5 min in order to compact the paste and release any entrained air. Samples were sealed in plastic bags and cured at ambient temperature (25 ± 2 °C) until the time of testing i.e. 14 days. 50 mm and 10 mm cubic samples were cast for compression tests and µ-CT scans, respectively.

Crystalline phases in the slag and fly ash precursors were neglected when calculating the mix proportions. Quartz, mullite and various iron rich crystalline phases (e.g. hematite) have been shown to be insoluble during alkali-activation over the time scale examined in this study, and consequently, only the amorphous component is considered reactive [28]. Quantitative X-ray diffraction (Q-XRD) analysis was used to calculate the amorphous portion of each phase (Table 3). The amorphous portion of the precursors as determined by Q-XRD does not necessarily indicate the total amount of reactive material (e.g. Ca present in crystalline phases such as gypsum can be reactive, and entrapment of amorphous material within insoluble crystalline phases can prevent reaction of it [29]), rather it provides an indication of the reactive precursor content. Diffraction data were collected using a Bruker D8 Advance instrument with Ni-filtered Co k $\alpha$  radiation (1.79 Å). Data were collected between 5 and 85° 20, with a step size of 0.02°, the scan rate of 1.0 s/step and spinning at 15 rpm. An anti-scatter blade was used to reduce the Download English Version:

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