



Effect of elevated temperature on alkali-activated geopolymeric binders compared to portland cement-based binders



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ARTICLE INFO

Article history:

Received 22 September 2015

9 September 2016

Accepted 12 September 2016

Available online xxxx

Keywords:

Alkali-activated binders

Geopolymers

Elevated temperatures

Mechanical properties

TGA

XRD

CT microtomography

ABSTRACT

This research focused on developing thermally-stable materials based on alkali-activation of slag, fly ash, and metakaolin compared to portland cement mixtures by using a hierarchical approach to material design. At lower length scales, X-ray diffraction (XRD) characterized the mineralogy that coupled to higher length scale experiments using thermogravimetric analysis (TGA) for determining the materials thermal stability. Additionally, high-energy X-ray computed microtomography (μ CT) determined the best-performing material formulation that minimized thermal damage when exposed to high temperatures (650 °C). The thermal loading was ramped up to 650 °C from ambient temperature in 60 s and then held for a total of 10 min. The μ CT identified that the alkali-activated fly ash mortar had less initial porosity than the ordinary portland cement mixtures, with more than 66% of the pores between 20 and 50 μ m in diameter. Consequently, the alkali-activated fly ash mortar was able to dissipate approximately 565 °C in just 50 mm of material, outperforming all the other mixes studied in this paper with μ CT confirming minimal damage after the temperature exposure.

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1. Introduction

Conventional structural concretes begin losing strength quickly when exposed to temperatures above 300 °C [1]. Typically, a hydrocarbon fire can generate temperatures in excess of 1000 °C with heat fluxes around 150 kW/m² within minutes of ignition [2,3]. Additionally, a hydrocarbon jet fuel can have the same temperatures, but the heat flux could be doubled [2,3]. At those temperatures, the compressive strength of portland cement concrete can be reduced by as much as 90% [2]. This loss of strength is commonly attributed to the degradation of the calcium silicate hydrate (C-S-H) as it begins to lose structural water along with dehydration of other hydrates (e.g., calcium hydroxide (Ca(OH)₂) and ettringite (Ca₆Al₂(SO₄)₃(OH)₁₂·26H₂O)) and initiation of internal thermal stress gradients. In addition, high-density, high-performance concretes (HPCs) with large amounts of portland cement and low water-to-cementitious materials ratios have issues when exposed to high temperatures. HPCs have a high density with low porosity, and as temperature increases the water vapor is unable to escape, causing pressure to build-up in the pores that results in explosive spalling [4,5]. In cases where HPC is in a confined space, flying debris represents a danger to the personnel responding to fires and sudden failures of supporting structural components create a hazard for the entire structure.

Development of a cementitious material capable of withstanding elevated temperatures due to extreme environments is required for a variety of civilian and military applications. Currently, an environmentally-friendly construction material capable of retaining mechanical performance at elevated temperatures is being developed that also reduces the amount of carbon dioxide emissions into the atmosphere. Instead of using portland cement as the binding component, industrial by-products such as fly ash, slag, and/or metakaolin are being incorporated to create a geopolymeric composite material [6–12]. A review of the geopolymer technology prepared by Duxson et al. [13] concluded that a significant amount of research has been performed, however, considerably more research is required to advance the technology to commercial applications. Geopolymer technology has the capacity for wide-scale applications in the construction industry, as well as in other applications due to similar engineering properties of portland cement and in some cases better thermal properties.

Research by Zhao and Sanjayan [14] has shown that geopolymer concretes consisting of non-hydrated aluminosilicate gel binding phases combined with thermally-stable aggregates have survived exposure to fire testing, while similar portland cement concrete structures have failed. Side-by-side experiments of geopolymer and portland cement concretes designed to have comparable compressive strengths revealed that the geopolymer concrete has a higher fire spalling resistance than the portland cement-based concretes. Zhao and Sanjayan [14] attributed this spalling resistance to a highly porous structure that

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facilitates the release of the internal steam pressure build-up after exposure to the elevated temperatures.

The research by Zhao and Sanjayan built upon the progress made by Duxson et al. [13] and Kong and Sanjayan [15] that showed that alkali-activated aluminosilicate gels present in aluminosilicate-based concretes are inherently different from portland cement-based concrete. The use of these type of binders requires a sodium and/or potassium-based alkali source that reacts with silica- and alumina-rich phases (e.g., clays and glasses), which results in a gel that does not contain the large amounts of chemi- or physi-sorbed water when compared with C-S-H gels produced by portland cement hydration. This open pore structure that distinguishes the aluminosilicate composites reflects the particle sizes of the starting materials [16]. The open pore structure results in a novel inorganic composite material that can withstand temperatures in the range of 600–800 °C with minor loss of strength. Furthermore, Zhao and Sanjayan [14] studied a fly ash based geopolymer and compared the material's response to a portland cement in a simulated gas fire, where the portland cement spalled while the fly ash did not spall.

Some examples of the research on high temperature behavior of metakaolin and fly ash based geopolymers is by Kong et al. [15,17–19], where the Si/Al ratio of the geopolymer was reported as a key parameter influencing the strength reductions after exposing the materials to elevated temperatures. Additionally, Kong reported that elevated temperature curing provided improved compressive strength, while room temperature curing resulted in lower strengths. Kong et al. [17] also reported that the fly ash based geopolymers mixed achieved average ambient compressive strengths of 59.0 MPa, while exposure to an 800 °C elevated temperature increased the strength to 62.8 MPa. This corroborates research done by Pan et al. [20] that showed a fly ash paste exposed to 550 °C had a compressive strength increase of 192% over ambient compressive strengths. Alternatively, Kong et al. [17] also examined metakaolin based geopolymers before and after elevated temperature exposure of 800 °C finding compressive strength decreased from 38.5 to 25.4 MPa.

Cheng and Chiu [21] investigated slag based geopolymers, noticing that strength and fire resistance increased as metakaolin content increased, and reported a 79 MPa compressive strength for the material. Bakharev [22] investigated the thermal stability properties of a fly ash based geopolymer under elevated temperature (800–1200 °C) and found the material to be inappropriate for refractory insulation applications, but added the need for further investigation for fire protection applications.

The thermal deterioration mechanisms reported in these materials are likely due to elevated temperatures above 100 °C leading to the water/moisture in the concrete mixture to vaporize. Once vaporized and unable to escape, leads to cracking and potential catastrophic damage to the infrastructure. To understand this vapor-escaping phenomenon in geopolymer materials, Kong et al. [17] identified that the fly ash based geopolymers have an increased number density of small pores that help the vapor to escape at elevated temperatures. However, metakaolin based geopolymers lack this pore distribution, resulting in damage to the structure [17].

The primary goal of this research was to develop a mixture to withstand elevated temperature exposure and to characterize the damage of these novel aluminosilicate binders using a variety of different analysis techniques. The research presented herein focused on understanding the interaction between gel chemistry and high temperature behavior using thermogravimetric analysis (TGA), X-ray diffraction (XRD), and X-ray computed microtomography (μ CT) to identify material composition and infer thermal stability as well as experiments to study the influence of high temperatures on compressive strength and thermal cracking. The goal of this investigation was to identify relevant constituent materials and mixture proportioning considerations to develop castable inorganic composites that are resistant to high temperature exposures.

2. Materials and methods

2.1. Materials

Three alkali-activated mortars fabricated from slag, metakaolin, and fly ash were compared to a portland cement mortar and an ASTM C109 [23] mortar. Slag used in this study met the requirements of ASTM C989 [24], with a specific gravity (SG) of 2.89 coming from the Birmingham slag facility (Birmingham, AL, USA). The fly ash was a low calcium fly ash (Class F), which meets the ASTM C618 Class F [25] and AASHTO M295 Class F [26] requirements. The fly ash had an SG of 2.29 and came from the Bowen power plant (Euharlee, GA, USA). The metakaolin met the requirements of ASTM C618 for a Class N natural pozzolan. The metakaolin is from Advanced Cement Technologies (Blaine, WA, USA) and had an SG of 2.6. The oxide compositions measured using X-ray fluorescence and loss on ignition (LOI) of the above-mentioned materials are given in Table 1.

Alkaline activators used in this investigation consisted of potassium hydroxide and potassium silicate. Potassium hydroxide of ACS reagent grade with purity greater than 85%, was utilized to prepare the activator solutions to a concentration of 14.0 M in deionized water. The solutions cooled for a period of 24 h. following mixing. For the mortar mixtures, a graded silica sand meeting the ASTM C778 [27] requirements was used.

2.2. Sample preparation

Activators were prepared 24 h prior to mixing to allow the activator to reach ambient temperature. The alkali-activated mortars and pastes were prepared adding the liquids to the 11 l tabletop mixer, then the starting material was added slowly (for the mortars, the sand and starting material were dry blended before adding it to the table mixer). Specimens were cast in 76.2 mm \times 152.4 mm cylinders for compressive strength testing and 50.8 mm \times 101.6 mm cylinders for elevated temperature exposure tests. Samples from all of the mixtures were demolded after 24 h and cured at 23 °C in a moist room except for the fly ash samples, which were sealed in plastic bags and cured in an oven at 40 °C for 72 h. Elevated temperature curing was necessary to harden the fly ash samples. Mixtures were designed for a minimum target compressive strength of 40 MPa at 28 days. The targets for the composition of the activator solution and curing temperatures were determined from previous research [14,18,21,22,28–33]. The two control mixtures were an ASTM C109 [34] mortar with a water to cement ratio of 0.485 and a portland cement mortar (PC/M) with a water to cement ratio of 0.40. The portland cement control mixture was selected, because the cement to sand volume ratio was the same as the other three alkali-activated mortar materials. The alkali-activated mortars were prepared by first adding the liquids then the starting material and the sand. Paste mixtures for TGA and XRD were prepared using the same procedure, but without sand. Thus, there is only one portland cement control paste (PC/P). The alkali-activated slag (S/P), fly ash (FA/

Table 1
Metakaolin, fly ash and slag elemental compositions.

Chemical	Component (wt.%)			
	Metakaolin	Fly ash	Slag	Portland cement type I/II
SiO ₂	55.24	53.74	40.92	21.66
TiO ₂	1.28	1.40	0.23	0.19
Al ₂ O ₃	39.83	28.26	8.32	2.86
Fe ₂ O ₃	1.83	6.66	0.44	4.47
MgO	0.10	0.91	10.99	2.69
CaO	–	1.38	34.12	63.96
Na ₂ O	0.04	0.35	0.14	0.09
K ₂ O	0.26	2.10	0.42	0.24
P ₂ O ₅	0.15	0.26	0.04	0.11
SO ₃	–	0.08	2.72	2.74
Loss on ignition	0.32	3.58	0.35	0.81

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