

# Effect of alkali cation type on strength endurance of fly ash geopolymers subject to high temperature exposure



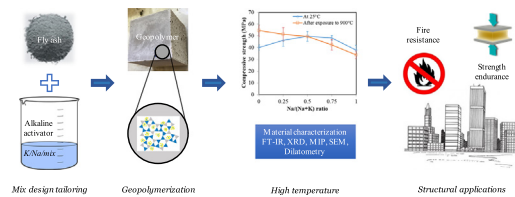
Mukund Lahoti, Keng Khang Wong, Kang Hai Tan, En-Hua Yang \*

School of Civil and Environmental Engineering, Nanyang Technological University, Singapore 639798, Singapore

## HIGHLIGHTS

- Fly ash geopolymers with stable/enhanced strengths upon thermal exposure for structural applications could be synthesized.
- Underlying competing mechanisms governing the strength endurance discovered.
- Fly ash geopolymers can be chemically stable upon thermal exposure.
- Volume stability upon thermal exposure varied with type of alkali cation used.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Choice of alkali cation in mix design of geopolymers is critical for their thermal performance. However, the influence of alkali cation type on strength endurance of geopolymers subject to high temperature exposure and the underlying governing mechanisms have not been studied. This study investigated the effects of alkali cation type on high temperature response of fly ash geopolymers aiming towards structural applications. In-depth investigation was carried out to discover the underlying mechanisms governing the strength endurance of fly ash geopolymers subject to high temperature exposure and its correlation with their volume stability and chemical stability. Results showed that potassium geopolymer exhibited significant strength enhancement (30–40%) and sodium geopolymer displayed reduced strength (10%), and the strength of mixed sodium and potassium geopolymer remained unchanged after exposure to elevated temperature. While the binders were chemically stable without deterioration and formation of new crystal phases after high temperature exposure, the volume stability varied with the type of alkali cation used. Formation of cracks and gel densification due to shrinkage, and healing of micro-cracks and change of pore sizes due to sintering were identified as responsible mechanisms at different temperature ranges. While crack development and enlargement of pores lowered the strength, densification of matrix and healing of micro-cracks favored strength gain. These competing mechanisms determine the strength endurance of geopolymers depending on the type of alkali cation used. Overall, it shows that geopolymers can be tailored, such that stable (or even enhanced) strengths upon thermal exposure are realized, for structural applications.

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## 1. Introduction

Fire damage can be critical in structures and in all cases the protection of people and property is essential [1]. Concrete is the most used construction material on Earth [2]. It is non-combustible and has a relatively low thermal conductivity. However, cracking, spalling and loss in

\* Corresponding author at: N1-01b-56, 50 Nanyang Avenue, 639798, Singapore.  
E-mail address: [ehyang@ntu.edu.sg](mailto:ehyang@ntu.edu.sg) (E.-H. Yang).

mechanical strength of concrete after exposure to fire are key concerns especially with high strength concrete [3–10]. Degradation of cement hydration products after high temperature exposure is another serious issue with ordinary Portland cement (OPC) [11,12].

Geopolymer, a novel construction material, is an alternative binder to OPC [13,14]. Geopolymer has also been referred to in literature as ‘inorganic polymer glass’, ‘alkali-activated cement’, ‘mineral polymer’, ‘alkali-bonded ceramic’ and many other names [15]. The term ‘geopolymer’ refers to a solid material formed by the reaction of an aluminosilicate source with alkaline activating solution [16]. Metakaolin, fly ash and blast furnace slag are commonly used aluminosilicate sources. It has been shown that geopolymer can be greener than OPC because many industrial by-products can be the precursor for the synthesis of geopolymer [17,18]. Geopolymers produced using coal combustion by-product, fly ash, is an effective technology to beneficially utilize fly ash; instead of stockpiling or simply dumping it to cause environmental and economic concerns [19–21]. Geopolymers with compressive strengths even higher than 100 MPa have been realized using fly ash as precursor [22]. Moreover, fire resistance potential of geopolymers, especially those synthesized using fly ash as precursor is remarkable [23–28] and their applications as heat-resistant materials [20,29–33], thermal insulators [1,34–39], thermal energy storage concretes [40], refractory materials [41] and precursors to ceramic formation [42,43] have been reported.

Geopolymer has been shown to be largely chemically stable on exposure to high temperature [44–48]. Chemical stability means that geopolymer does not undergo decomposition of the binder phase as experienced by OPC paste on high temperature exposure. However, volume stability, especially for metakaolin-based geopolymer, is a serious concern [49,50]. In absence of volume stability material would experience thermal deformation and cracking damage. Coal fly ash-based geopolymer has shown better high temperature performance than metakaolin-based geopolymer in terms of their volume stability and residual strength [51–53]. It was reported that fly ash-based geopolymers experienced less damage, as compared to their metakaolin counterparts, due to their possessing large numbers of interconnected pores which facilitated moisture escape on high temperature exposure [51]. Additionally, sintering of unreacted fly ash particles was thought to be another reason for better performance of fly ash geopolymers. Sintering enhances mechanical strength due to stronger bonding among the particles. Comparative assessment of fly ash from different sources to produce geopolymers for high temperature applications has been reported [52,54]. Recently, Colangelo et al. [40] tested several geopolymer mortars based on sodium activated fly ash and mixtures (fly ash/ground granulated blast furnace slag) for thermal storage applications. Their study led to the conclusion that binders made solely with fly ash are more thermally stable.

It has been shown for metakaolin-based geopolymers that the choice of alkali cation (whether sodium, potassium or a mixture of two), while designing a geopolymer mix, affects thermal shrinkage and phase stability subject to elevated temperatures [46,55]. However, for fly ash-based geopolymers, there has been very limited research investigating the effect of mixed alkali cation on their high temperature performance. Furthermore, the influence of alkali cation type on strength endurance of geopolymers subject to high temperature exposure and the underlying governing mechanisms have not been studied even for metakaolin-based geopolymers. Strength endurance of material subject to high temperature exposure is of particular importance for structural applications. Construction materials need to satisfy required strength criteria (e.g. 30 MPa) at ambient condition and also must endure sufficient strength to continue to serve as a structural material after exposure to elevated temperature. Geopolymers synthesized in many previous works with low strength (e.g. 10 MPa) were not aiming towards structural applications [56]. Therefore, there is a need to carry out a careful investigation on geopolymers designed using different alkaline activators to not only meet the strength endurance

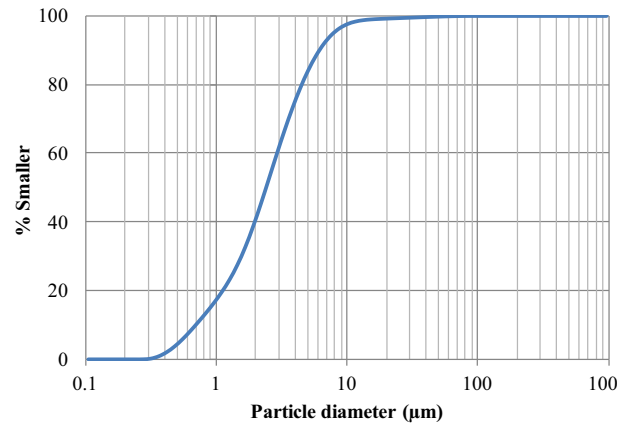


Fig. 1. Particle size distribution of fly ash.

criteria but also to reveal the underlying mechanisms that govern their thermal performance.

Hence, this study investigated the effects of alkali cation type (whether sodium, potassium or a mixture of two) on high temperature response of fly ash-based geopolymers aiming towards structural applications. In-depth investigation was carried out to discover the underlying mechanisms governing the strength endurance of the fly ash geopolymers subject to high temperature and its correlation with their volume stability and chemical stability. Various experimental techniques, namely x-ray diffractometry (XRD), Fourier transform infrared spectroscopy (FTIR), mercury intrusion porosimetry (MIP), dilatometry, and scanning electron microscopy (SEM) were utilized in this research.

## 2. Materials and methods

### 2.1. Fly ash

Class F Fly ash was used as a precursor to synthesize geopolymers in this research. This fly ash was procured from Bisley Asia Pte Ltd. The particle size distribution of fly ash is shown in Fig. 1. It illustrates a well graded or continuous particle size distribution with particle size ranging from 0.2 to 80 µm with  $d_{10} = 0.74$  µm,  $d_{50} = 2.5$  µm and  $d_{90} = 6$  µm.

Chemical composition of fly ash based on X-ray fluorescence spectroscopy (XRF) is given in Table 1. As can be seen, the fly ash has low CaO and the total content of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and Fe<sub>2</sub>O<sub>3</sub> is higher than 70%, which indicates it is a class F fly ash according to ASTM C618. The high SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> content (~90%) implies the fly ash may potentially be a suitable geopolymer precursor.

The XRD pattern for fly ash is shown in Fig. 2. Almost all crystalline peaks could be identified and were classified into either Mullite (Al<sub>4.66</sub>Si<sub>1.39</sub>O<sub>9.75</sub>) or Quartz (SiO<sub>2</sub>). The hump in the XRD pattern, in the 2theta range 17°–33°, is indicative of the amorphous phases which are considered as potential reactive components for alkaline activation [57]. Amorphous phases were quantified by means of the internal standard method. Fly ash samples were spiked with silica (5% by weight) obtained from the U.S. National Institute of Standards and Technology (NIST). The crystal structures used to calculate the patterns were taken from the Inorganic Crystal Structure Database (ICSD). The codes for the various crystal structures were: Silicon 51,688, Quartz 156,198

Table 1  
Chemical composition of fly ash.

Oxide	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	K <sub>2</sub> O	CaO	MgO
Content (wt%)	58.59	30.44	4.66	2.02	1.51	1.21	0.78
Oxide	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	ZrO <sub>2</sub>	MnO	Cr <sub>2</sub> O <sub>3</sub>	CuO	ZnO
Content (wt%)	0.53	0.09	0.04	0.04	0.03	0.03	0.02

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