



# Compressive behaviour of sodium and potassium activators synthesized fly ash geopolymer at elevated temperatures: A comparative study



Anwar Hosan, Sharany Haque, Faiz Shaikh\*

Department of Civil Engineering, Curtin University, Perth, Western Australia, Australia

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## ABSTRACT

This paper presents the effects of sodium and potassium based activators on compressive strengths and physical changes of class F fly ash geopolymer exposed to elevated temperatures. Samples were heated at 200 °C, 400 °C, 600 °C and 800 °C to evaluate the residual compressive strength after 28 days of curing. The fly ash geopolymer were synthesized with combined sodium silicate and sodium hydroxide solutions and potassium silicate and potassium hydroxide solutions by varying mass ratios of  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  and  $\text{K}_2\text{SiO}_3/\text{KOH}$  of 2, 2.5 and 3. Results show significant improvement in compressive strength in the case of  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  ratio of 3 than 2 and 2.5, where the residual compressive strengths are increased up to 600 °C. Better results on the geopolymer synthesized with potassium based activators are obtained where the residual compressive strength up to 600 °C are much higher than their sodium based counterparts. It is also found that the fly ash geopolymer synthesized with potassium based activators is more stable at elevated temperatures than its sodium based counterparts in terms of higher residual compressive strengths, lower mass loss, lower volumetric shrinkage and lower cracking damage. X-ray diffraction (XRD) and thermogravimetric analysis (TGA) results of sodium and potassium activator synthesized fly ash geopolymer also corresponds to the measured residual compressive strengths.

## 1. Introduction

Many reinforced concrete (RC) structures experience fire during their service life. The mechanical properties and stiffness of concrete decrease significantly due to exposure to elevated temperatures during fire. This reduction in strength is associated with the dehydroxylation of  $\text{Ca}(\text{OH})_2$  between 400 and 500 °C and continuous dehydration of Calcium Silica Hydrate (CSH) from 105 °C in cement matrix in concrete. The difference in thermal expansion/contraction between cement paste and aggregates cause significant cracking in the concrete, which also contributes to the reduction in mechanical strengths and stiffness of concrete. In addition thermal gradient in concrete also contributes to its poor performance at elevated temperatures. Geopolymer is a new construction material which has gained its popularity in recent years due to its environment friendliness and excellent mechanical, durability and fire resistant properties in severe environment. Geopolymer is first introduced by Davidovits [1] and is generally synthesized by reacting aluminate and silicate bearing source materials with alkali activator. The hardening mechanism for geopolymers essentially involves the poly-condensation reaction of polymeric precursors, usually alumina-silicate oxides, with alkali polysilicates yielding a polymeric silicon-oxygen-aluminium framework.

Due to this inorganic framework, geopolymers are intrinsically fire resistant and have been shown to have excellent thermal stability with very little gel structural degradation up to 700–800 °C, which is the most significant advantage of geopolymer over cement based binder as the latter breakdown due to dehydration of its hydrates at elevated temperatures.

Extensive researches have been conducted to study various mechanical and durability properties of geopolymer. Significant efforts have also been made by many researchers to study the effect of elevated temperatures on mechanical properties of geopolymer. However, most of the studies were on different geopolymers which were made by different types of source materials and alkali activators, e.g. fly ash activated by sodium based alkali activators [2–5], fly ash activated by combined sodium and potassium based activators [6–8], combined fly ash and slag activated by sodium based activator [9], metakaolin activated by combined sodium and potassium based activators [10], etc. Among all above source materials the class F fly ash is rich in silica and alumina and low in calcium oxide, which enables its higher stability at elevated temperature in fire than others. Moreover, it is cheaper, easily and widely available than other source materials.

This paper presents the effects of sodium and potassium based activators on the compressive strength and damage behaviour of fly ash

\* Corresponding author.

E-mail address: [s.ahmed@curtin.edu.au](mailto:s.ahmed@curtin.edu.au) (F. Shaikh).

geopolymer at various elevated temperatures of 200, 400, 600 and 800 °C. The effects of silicate to hydroxide ratios for both sodium and potassium based activators on the compressive strengths and physical changes like the mass loss, volumetric shrinkage, cracking behaviour at above elevated temperatures are also evaluated. X-ray diffraction (XRD) and thermogravimetric analysis (TGA) are also used to support the observed physical behaviour and strength properties.

## 2. Experimental program

The experimental program is consisted of two parts. The first part is geopolymer paste containing sodium based activators combinations, where three different sodium silicate to sodium hydroxide ratios of 2, 2.5 and 3 are considered and for each ratio the geopolymers are heated at 200, 400, 600 and 800 °C temperatures as well as at ambient temperature. Thus fifteen series of pastes are cast and tested at elevated temperatures in the first part. The second part is similar to the first part in every aspect except the alkali activators where potassium silicate and potassium hydroxide are used. For each series, in both parts, six 50 mm cube specimens are cast and tested and the average value is shown in the results. Detail experimental program and mix proportions are shown in Table 1. In all pastes a constant alkali activator/fly ash ratio of 0.35 is considered.

## 3. Materials, mixing and methods

Class F fly ash supplied by Gladstone power station of Queensland, Australia was used as source material to prepare the geopolymer pastes. Table 2 shows the chemical compositions of the fly ash. The activating alkali liquids consisted of  $\text{Na}_2\text{SiO}_3$  and NaOH solutions as well as  $\text{K}_2\text{SiO}_3$  and KOH solutions. The Na-based activator was composed of 8.0 M sodium hydroxide (NaOH) and D Grade sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) solutions. NaOH solution was prepared with a concentration of 8.0 M using NaOH beads of 97% purity and tap water. The D Grade  $\text{Na}_2\text{SiO}_3$  solution was supplied by PQ Australia with a specific gravity of 1.51 and a modulus ratio (Ms) equal to 2.0 (where  $\text{Ms}=\text{SiO}_2/\text{Na}_2\text{O}$ ,  $\text{Na}_2\text{O}=14.7\%$ ,  $\text{SiO}_2=29.4\%$  and water-55.9%). The NaOH and  $\text{Na}_2\text{SiO}_3$  solutions were mixed together with  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 2, 2.5 and 3 to prepare the Na-based activators. The K-based activator was composed of 8.0 M potassium hydroxide (KOH) and potassium silicate ( $\text{K}_2\text{SiO}_3$ ) solutions. KOH solution was prepared with a concentration of 8.0 M using KOH flakes of 90% purity supplied by Perth Scientific, Australia and tap water. The  $\text{K}_2\text{SiO}_3$  (KASIL 2236 Grade) solution was supplied by PQ Australia with a specific gravity of 1.32 and a modulus ratio (Ms) equal to 2.23 (where  $\text{Ms}=\text{SiO}_2/\text{K}_2\text{O}$ ,  $\text{K}_2\text{O}=11.2\%$ ,  $\text{SiO}_2=24.8\%$  and water=64%). KOH and

$\text{K}_2\text{SiO}_3$  solutions were mixed together with  $\text{K}_2\text{SiO}_3/\text{KOH}$  mass ratio of 2, 2.5 and 3 to prepare the K-based activators.

All pastes were prepared in a Hobart mixer. To prepare the Na- and K-based geopolymer paste the alkaline activators in the form of solution were added to the fly ash and mixed for about 4 min. The fresh geopolymer pastes were cast into standard 50 mm plastic cube moulds and compacted using a vibrating table. The specimens were subjected to heat curing. In this regard, all moulds were sealed to minimize moisture loss and placed in an oven at 70 °C for 24 h. At the end of heat curing period, the specimens were removed from the oven and kept undisturbed until being cool and then removed from the moulds and left in the laboratory at ambient temperature until the day of testing. Compressive strength of all specimens was measured according to AS 1012.9:2014 [11]. For each mix, at least six specimens were tested in order to check the variability of performance under compression. The volumetric shrinkage of pastes was determined by measuring the length of three sides of the cubes before and after heating at respective elevated temperatures. The difference in volume changes indicates the volumetric shrinkage and six specimens were used to measure the volumetric shrinkage for each series. Similar method was also used to determine the mass loss of geopolymer pastes after exposed to respective elevated temperatures.

The thermal stability of samples was studied by thermogravimetric analysis (TGA). A Mettler Toledo TGA one star system analyser was used for all these measurements. Samples of 25 mg were placed in an alumina crucible and tests were carried out in Argon atmosphere with a heating rate of 10 °C/min from 25 to 1000 °C. In the case of XRD analysis, the samples were measured on a D8 Advance Diffractometer (Bruker-AXS) using copper radiation and a Lynx Eye position sensitive detector. The diffractometer were scanned from 7° to 70° (2 $\theta$ ) in steps of 0.015° using a scanning rate of 0.5°/min. XRD patterns were obtained by using Cu Ka lines ( $k=1.5406 \text{ \AA}$ ). A knife edge collimator was fitted to reduce air scatter.

## 4. Elevated temperature exposure

A locally manufactured kiln was used to heat the geopolymer specimens, where the specimens were heated from 200 °C to 800 °C. The specimens were positioned inside the kiln where two thermocouples were touched the specimens, and two more thermocouples were also inserted inside the kiln to monitor the kiln air temperature. The thermocouples were connected to the data logger and were used to monitor the temperature on the mortar surface and the kiln air as shown in Fig. 1. A heating rate of 5 °C per minute was selected, which is very close to the RILEM recommended heating rate [12]. During heating process the temperatures of four thermocouples were mon-

**Table 1**  
Experimental program and mix proportions.

Part 1				Part 2			
Sample ID	$\text{Na}_2\text{SiO}_3/\text{NaOH}$	Activators/fly ash	Kiln Temperature	Sample ID	$\text{K}_2\text{SiO}_3/\text{KOH}$	Activators/fly ash	Kiln Temperature
Na-2-28	2	0.35	Ambient	K-2-28	2	0.35	Ambient
Na-2-200	2	0.35	200	K-2-200	2	0.35	200
Na-2-400	2	0.35	400	K-2-400	2	0.35	400
Na-2-600	2	0.35	600	K-2-600	2	0.35	600
Na-2-800	2	0.35	800	K-2-800	2	0.35	800
Na-2.5-28	2.5	0.35	Ambient	K-2.5-28	2.5	0.35	Ambient
Na-2.5-200	2.5	0.35	200	K-2.5-200	2.5	0.35	200
Na-2.5-400	2.5	0.35	400	K-2.5-400	2.5	0.35	400
Na-2.5-600	2.5	0.35	600	K-2.5-600	2.5	0.35	600
Na-2.5-800	2.5	0.35	800	K-2.5-800	2.5	0.35	800
Na-3-28	3	0.35	Ambient	K-3-28	3	0.35	Ambient
Na-3-200	3	0.35	200	K-3-200	3	0.35	200
Na-3-400	3	0.35	400	K-3-400	3	0.35	400
Na-3-600	3	0.35	600	K-3-600	3	0.35	600
Na-3-800	3	0.35	800	K-3-800	3	0.35	800

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