



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



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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

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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 Na_2SiO_3 and NaOH solutions as well as K_2SiO_3 and KOH solutions. The Na-based activator was composed of 8.0 M sodium hydroxide (NaOH) and D Grade sodium silicate (Na_2SiO_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 Na_2SiO_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 Na_2SiO_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 (K_2SiO_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 K_2SiO_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

K_2SiO_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 θ) 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

Table 2
Chemical Composition of Fly Ash (mass %).

Compounds	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	Na ₂ O	K ₂ O	MgO	P ₂ O ₅	SO ₃	TiO ₂	MnO	LOI
Fly ash	51.11	25.56	12.48	4.3	0.77	0.7	1.45	0.885	0.24	1.32	0.15	0.57



Fig. 1. Geopolymer specimens inside the kiln.

itored. Once the specimen's surface reached the target temperature, the temperature inside the kiln was hold for one hour. The rate of temperature increase in the kiln and in the specimen is shown in Fig. 2. As can be seen in the figure, there is a significant lag between the surface temperature of cubes and temperature inside the kiln, particularly for the 200 and 400 °C temperature profiles. This is due to the heat capacity of the specimens and the rate at which they are able to absorb heat. However, the difference in temperature between the kiln

and the cubes at 600 and 800 °C is less. Even though the difference in temperature between the kiln and the cubes existed, the target test temperatures in all cubes were maintained for one hour, which can be seen in the cylinders thermocouples readings in Fig. 2.

5. Results and discussions

5.1. Residual compressive strengths

The measured compressive strengths of geopolymer pastes containing different Na₂SiO₃/NaOH ratios of 2, 2.5 and 3 after exposed to 200, 400, 600 and 800 °C temperatures are shown in Fig. 3. It can be seen that the residual compressive strengths of geopolymer paste containing Na₂SiO₃/NaOH ratio of 2 and 2.5 are increased by about 9–11% at 200 °C compared to ambient temperature strength. However, with further heating at 400, 600 and 800 °C temperatures the residual compressive strengths of above geopolymer pastes are decreased below the ambient condition (see Fig. 4). On the other hand, the same geopolymer paste containing Na₂SiO₃/NaOH ratio of 3 showed significant increase in residual compressive strength by about 25–35% up to 600 °C with exception at 800 °C, where the residual compressive strength is decreased by more than 30% (See Fig. 4).

The effect of K-based activators on the compressive strength of geopolymer paste at elevated temperatures is also evaluated in this study and is shown in Figs. 5 and 6. Fig. 5 shows the measured compressive strengths of geopolymer pastes containing different K₂SiO₃/KOH ratios of 2, 2.5 and 3 after exposing to 200, 400, 600 and 800 °C temperatures. It can be seen in the figure that for all three K₂SiO₃/KOH ratios the residual compressive strengths of geopolymer

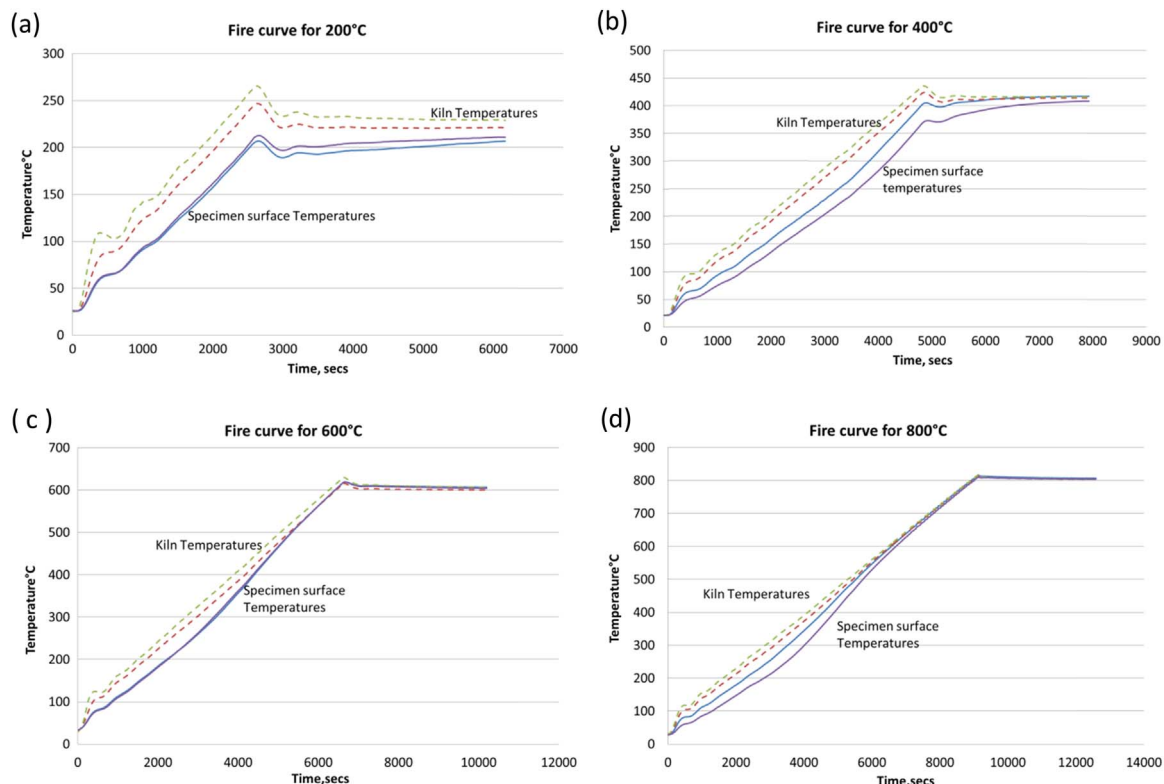


Fig. 2. Fire curves for different elevated temperatures in the kiln during heating.

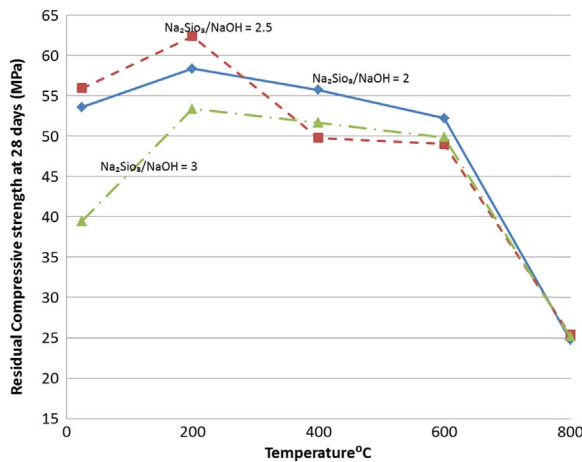


Fig. 3. Residual compressive strength of geopolymer pastes containing Na-based activators at various elevated temperatures.

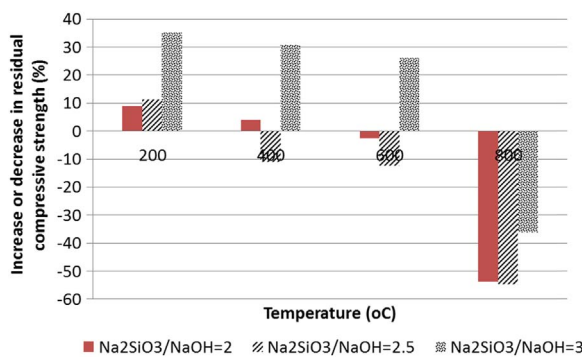


Fig. 4. Relative increase or decrease in residual compressive strength of geopolymer pastes containing Na-based activators at various elevated temperatures compared to ambient temperature.

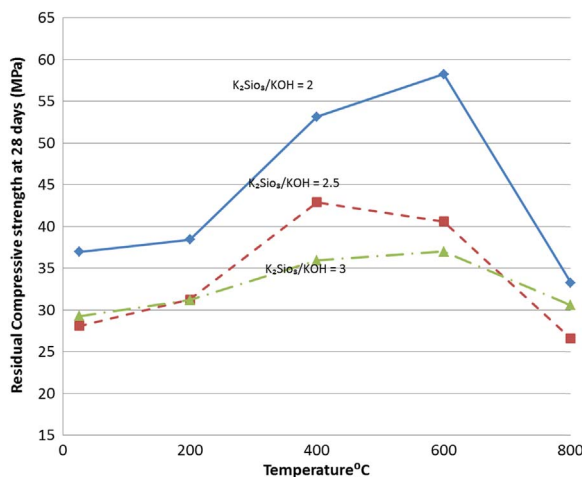


Fig. 5. Residual compressive strength of geopolymer pastes containing K-based activators at various elevated temperatures.

pastures are much higher than the ambient temperature strength and the residual compressive strengths are increased with increase in elevated temperatures until 600 °C. It is also interesting to see that at 400 and 600 °C the geopolymer paste containing K₂SiO₃/KOH ratios of 2 and 2.5 show about 40–55% increase in compressive strength compared to ambient condition. Although the increase in compressive strength of paste containing K₂SiO₃/KOH ratios of 3 is slightly lower (about 22–26%) at those temperatures, the compressive strength is increased by about 5% at 800 °C temperature, which is not observed in the case of

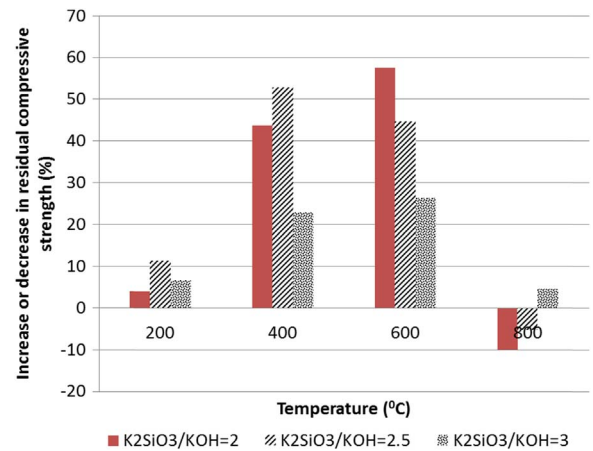


Fig. 6. Relative increase or decrease in residual compressive strength of geopolymer pastes containing K-based activators at various elevated temperatures compared to ambient temperature.

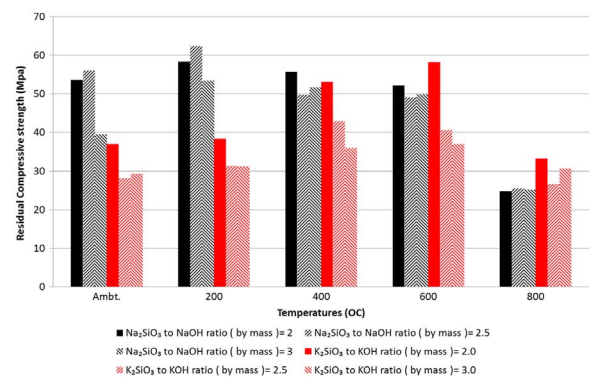


Fig. 7. Comparison of compressive strengths of Na- and K-based geopolymer pastes.

K₂SiO₃/KOH ratios of 2 and 2.5 (see Fig. 6).

By comparing both Na and K based geopolymer series in Fig. 7 it can be clearly seen that the K-based activators show higher compressive strength retention capacity for geopolymer pastes than its counterpart Na-based series compared to ambient temperature strength. It can also be seen that in both geopolymer the ambient strength decreases with increase in silicate/hydroxide ratios for both Na- and K-based activators. This is attributed to the low water evaporation and less geopolymer structure formation due to excessive sodium silicate and potassium silicate [13]. Interestingly the geopolymer with K₂SiO₃/KOH=3 exhibited higher compressive strength at all elevated temperatures than ambient temperature, which is also true for Na-based counterpart, however up to 600 °C. The increase in compressive strength in the former can be attributed to the lower diffusion coefficient of K⁺ at elevated temperatures, which results in higher melting temperature [8].

5.2. Post-heating physical behaviour

The effects of elevated temperatures on physical behaviour of both Na- and K-based activator synthesized fly ash geopolymers are shown in Figs. 8–10. Fig. 8 shows the formation of cracks on specimens' surface in both geopolymers. It can be seen that up to 400 °C no cracks are formed in both geopolymers. However, Na-based geopolymer showed signs of cracks at 600 °C and it becomes worst at 800 °C, where many wide cracks on the surface are formed. The geopolymer containing K-based activator, however, survived from surface cracking up to 600 °C, but fine cracks are formed at 800 °C. Fig. 9 shows the reduction of mass of both geopolymers at various elevated temperatures. It can be seen that the mass loss of Na-based geopolymer is

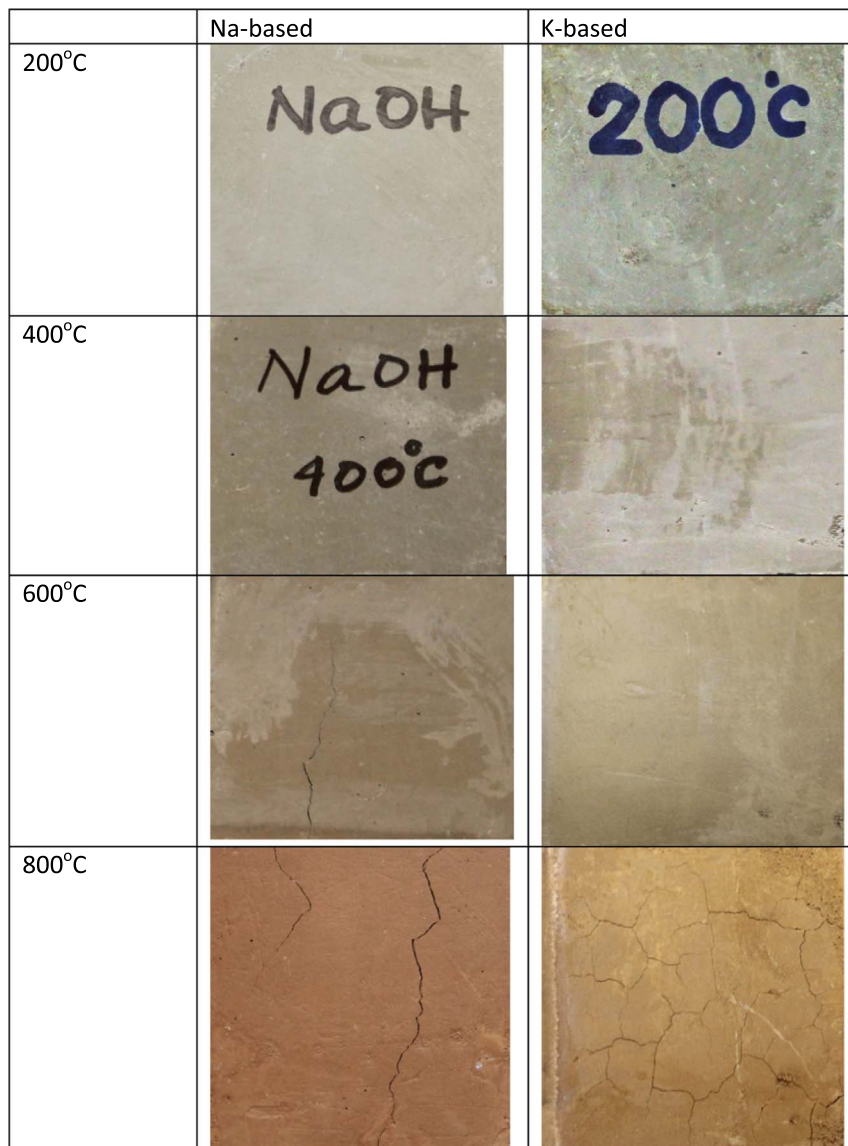


Fig. 8. : Cracking behaviour of fly ash geopolymers containing Na- and K-based activators at elevated temperatures.

slightly higher than that of K-based counterpart. An interesting observation is also noted that by increasing the $\text{Na}_2\text{SiO}_3/\text{NaOH}$ ratios the mass loss is slightly increased, which is opposite in the K-based system. It can also be seen that up to 400 °C significant reduction in

mass loss of about 8–10% is observed in both geopolymers and the mass loss becomes stable afterward. In the case of volumetric shrinkage of both geopolymers at elevated temperatures similar results to those of mass loss are also observed, where the K-based geopolymer

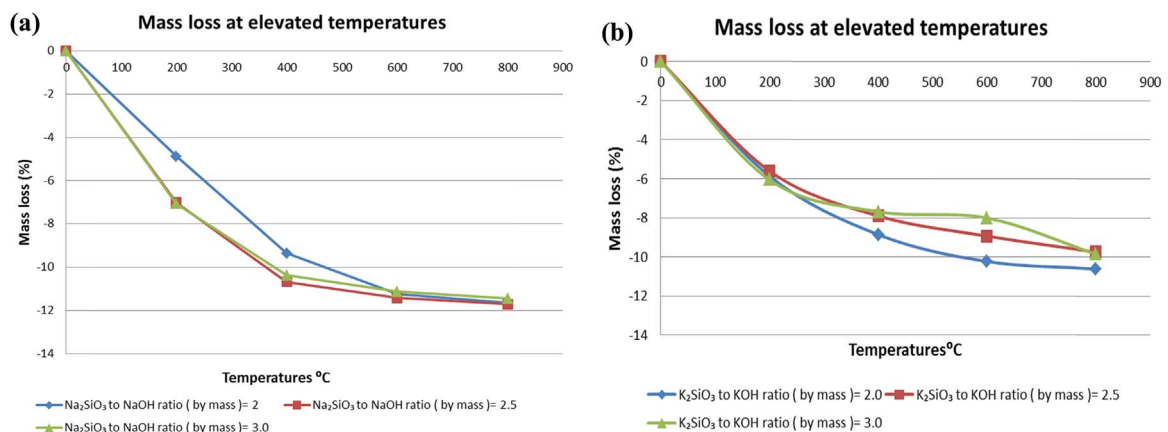


Fig. 9. Comparison of mass loss of (a) Na- and (b) K-based geopolymer pastes at elevated temperatures.

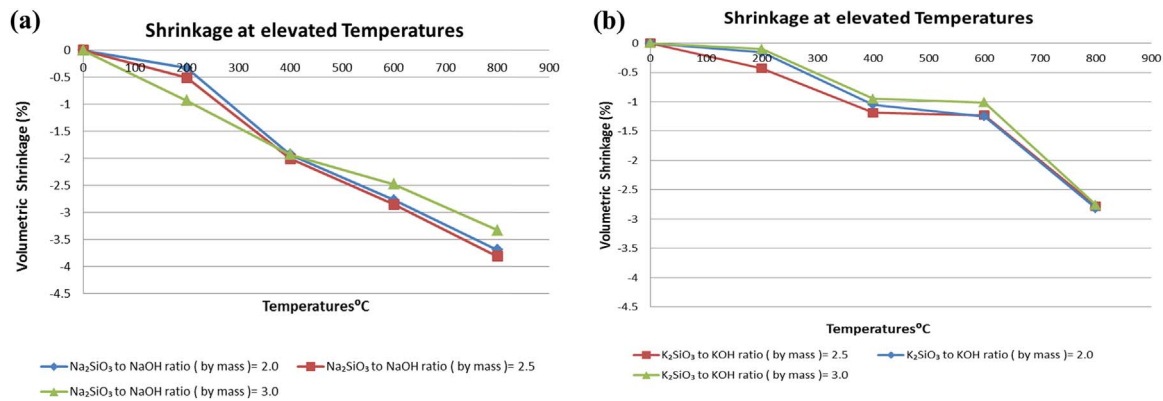


Fig. 10. Comparison of volumetric shrinkage of (a) Na- and (b) K-based geopolymer pastes at elevated temperatures.

showed lower shrinkage than its Na-based counterpart at all elevated temperatures. The observed less cracks in the K-based activator synthesized geopolymer is due to its lower mass loss and lower volumetric shrinkage than Na-based system. The higher residual compressive strength of K-based activator synthesized geopolymer than its counterpart Na-based system is also due to the less cracking in the former than the latter, as pre-existing cracks cause stress concentration in the specimen under compression and hence failure at lower loads.

5.3. Further discussion considering XRD and TGA/DTA results

Among three different ratios of silicate-to-hydroxide of both Na and K-based activators the ratio 3 exhibited the best in terms of highest residual compressive strengths after elevated temperatures exposure. Even among Na and K-based activators the K-based activator showed the best performance. In order to get more insight on the above difference in behaviour powder samples were collected from tested specimens at ambient condition, 400 °C and 800 °C for both activators for silicate-to-hydroxide ratio of 3. X-ray diffraction (XRD), thermogravimetric analysis (TGA) and differential thermal analysis (DTA) of above powder samples were conducted to identify the change in geopolymer reaction phases due to exposure to elevated temperatures for both activators. Fig. 11a–c show the XRD patterns of both Na and K-based geopolymers at ambient and after exposure to 400 °C and 800 °C temperatures. The strong peaks in the fly ash geopolymers mainly identify the presence of quartz and mullite at ambient temperature. Mullite is the stable crystalline phase of the Al₂O₃-SiO₂ system under atmospheric condition and is a refractory. Mullite retains its room temperature strength at elevated temperatures and has high temperature stability up to 1840 °C [2] with low thermal expansion and oxidation resistance. In Both Na- and K-based fly ash geopolymers at room temperature several quartz peaks at 2θ angles of 20.83°, 26.65° and 39.51° and several mullite peaks at 2θ angles of 16.46°, 26.53°, 30.26°, 33.20° and 35.66° are observed (Fig. 11a). However, in K-based geopolymer the quartz and mullite peaks are more prevalent than the Na-based system. Both geopolymers after exposure to 400 °C also show the presence of quartz and mullite peaks, with slightly stronger quartz and mullite peaks in Na-based geopolymer than the K-based geopolymer. After 800 °C an opposite trend is observed with stronger quartz and mullite peaks in K-based geopolymer than the Na-based geopolymer.

Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were used to assess the weight loss during controlled heating of both Na- and K-based fly ash geopolymers before and after exposure to elevated temperatures in order to identify the present of phases. Fig. 12a–c show the TGA and DTA curves of both geopolymers at ambient condition and after heating to 400 and 800 °C temperatures. It can be seen in Fig. 12a that the weight loss of Na-based fly ash

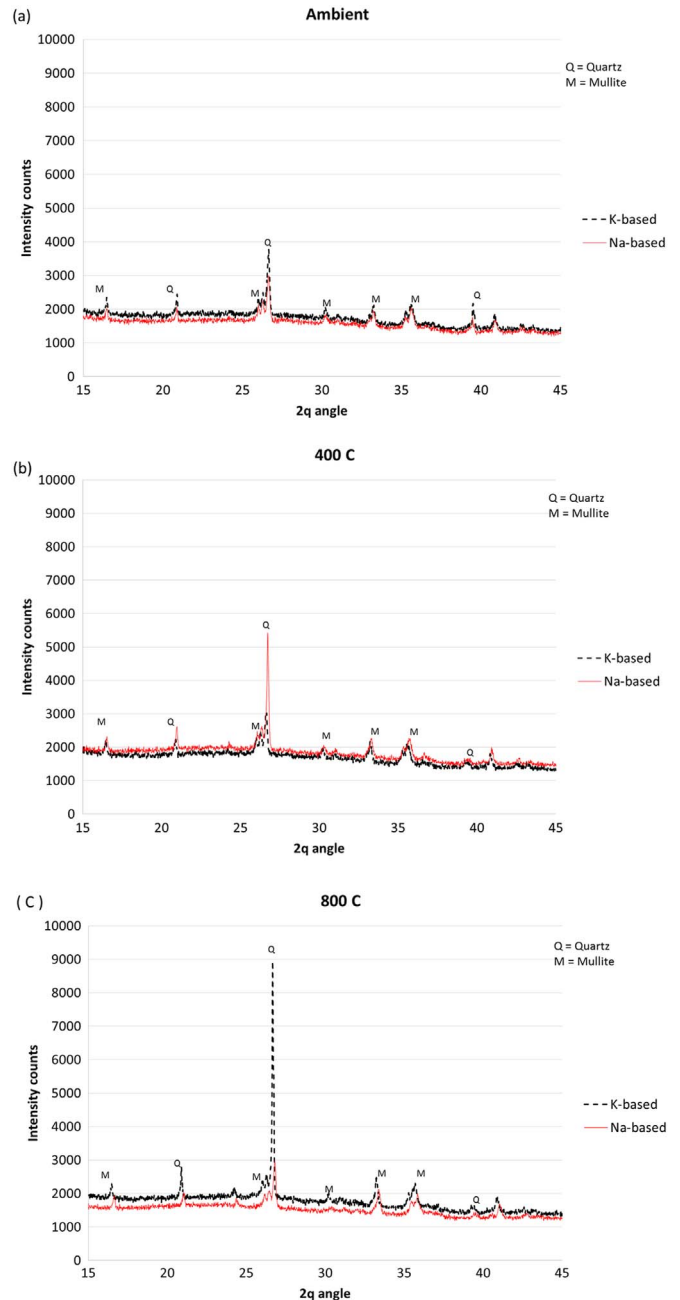


Fig. 11. XRD analysis results of Na- and K-based fly ash geopolymer at (a) ambient, (b) at 400 °C and (c) at 800 °C temperatures.

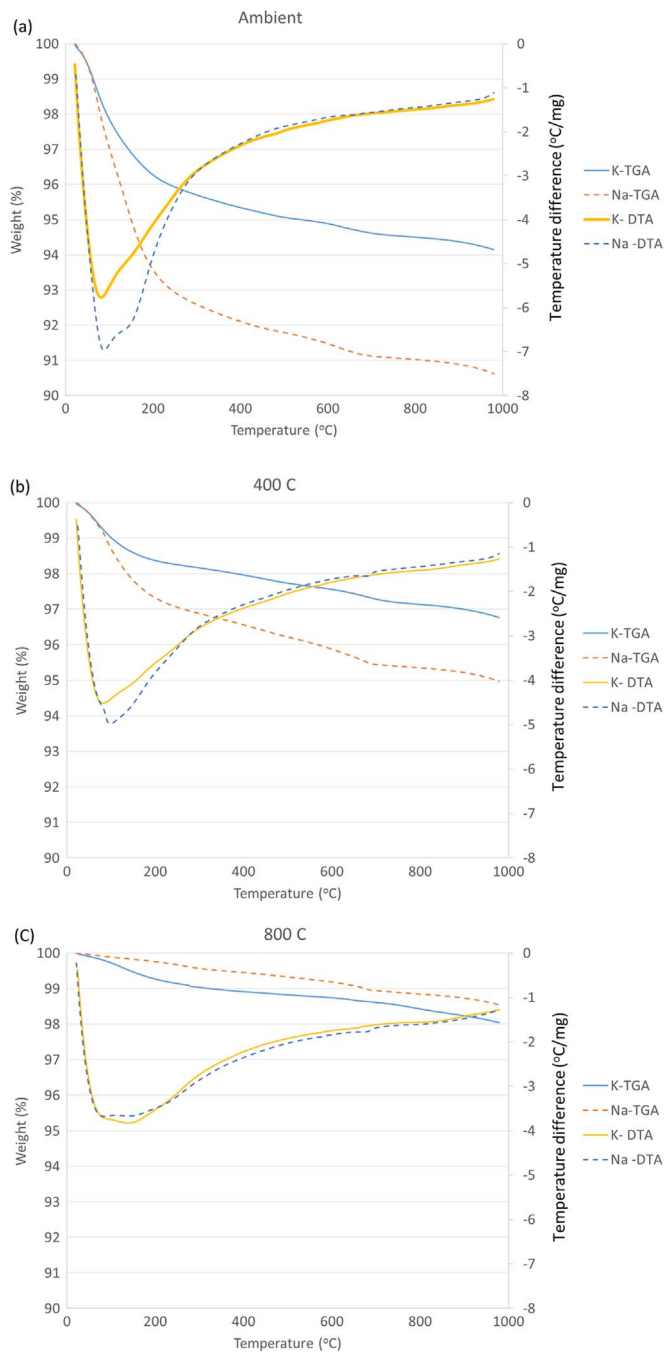


Fig. 12. TGA/DTA analysis results of Na- and K-based fly ash geopolymer at (a) ambient, (b) at 400 °C and (c) at 800 °C temperatures.

geopolymer is much higher than its K-based counterpart. In DTA curves a peak is located at approximately 90 °C and is caused by loss of absorbed and combined water in the N-A-S-H gels [14]. It can also be seen that the DTA peak is greater for Na-based fly ash geopolymer than its K-based counterpart, meaning more geopolymerization has occurred with Na-based activator [14]. And this can be reflected by comparing the compressive strength values in Figs. 3 and 5 at ambient temperature for both geopolymers. On the other hand, the weight loss in both geopolymer in TGA decreased after their exposure to 400 °C and 800 °C and the difference in weight loss between the Na- and K-based systems decreased gradually at 400 °C and 800 °C exposures. This is also reflected in the reduction in the difference in peaks in DTA cures in both geopolymers at both 400 and 800 °C temperatures. This lower weight loss in both geopolymers can be attributed to the loss of

absorbed water in the geopolymer matrix due to heating at 400 °C and the combined water in geopolymer gels at 800 °C. No weight loss or DTA peak at approximately 450 °C is observed which is usually for $\text{Ca}(\text{OH})_2$. It is also interesting to note that the weight loss and the DTA peak at approximately 100 °C is higher in Na-based geopolymer than its K-based counterpart at 400 °C exposure. On the other hand, the K-based geopolymer showed slightly higher weight loss and DTA peak than Na-based geopolymer at 800 °C. These results are consistent with XRD and compressive strength results, where the XRD peaks of quartz and mullite phases are higher in Na-based fly ash geopolymer than the K-based at 400 °C compared to 800 °C. The residual compressive strength of Na-based fly ash geopolymer is also higher than the K-based geopolymer at 400 °C.

6. Conclusions

Based on limited experimental variables in terms of Na and K-based activators, the ratios of silicate to hydroxide of above activators, elevated temperatures on the residual compressive strengths, volumetric shrinkage and mass loss of geopolymer pastes the following conclusions can be drawn:

1. The geopolymer pastes containing Na-based activator exhibited higher compressive strength at ambient temperature and higher compressive strength at elevated temperatures up to 400 °C than its K-based counterpart.
2. At 600 °C the compressive strength of geopolymer containing K-based activator is slightly higher than its Na-based counterpart.
3. The geopolymer paste containing K-based activator exhibited higher residual compressive strengths at all elevated temperatures compared to ambient temperature than its Na-based counterpart.
4. The geopolymer paste containing K-based activator with $\text{K}_2\text{SiO}_3/\text{KOH}$ ratio of 3 exhibited the highest residual compressive strengths at all elevated temperatures compared to ambient temperature than its Na-based counterpart.
5. The volumetric shrinkage and mass loss of geopolymer paste containing K-based activator is lower than its Na-based counterpart.
6. The geopolymer pastes containing K-based activator exhibited fewer surface cracks than that of Na-based activator.
7. XRD peaks of quartz and mullite are observed in both geopolymers after exposure to 400 and 800 °C temperatures like those observed in ambient condition. The presence of quartz and mullite peaks are believed to be the reason for maintaining residual compressive strength by both geopolymers after exposure to elevated temperatures.
8. The Na-based fly ash geopolymer exhibited higher weight loss in TGA and higher DTA peak at about 100 °C than its K-based counterpart after exposure to 400 °C. The higher weight loss and DTA peak is associated with the loss of absorbed and combined water in geopolymer gels, which indicate that higher geopolymer gels are remained in Na based geopolymer after 400 °C exposure than K-based system, which agrees well with the observed compressive strength results. An opposite trend is observed after exposure to 800 °C.

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