

# Strain Hardening Behavior of Engineered Geopolymer Composites: Effects of the Activator Combination

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## Abstract

Fly ash-based engineered geopolymer composites (EGCs) exhibiting strain hardening behavior under uni-axial tension were developed employing two different sodium-based (Na-based) and potassium-based (K-based) activator combinations. The relatively brittle low calcium (Class F) fly ash-based geopolymer matrix was reinforced with randomly oriented short poly vinyl alcohol (PVA) fibers (2% v/v). Na-based activator combination was composed of 8.0 M NaOH solution (28.6% w/w) and Na<sub>2</sub>SiO<sub>3</sub> solution (71.4% w/w) with a SiO<sub>2</sub>/Na<sub>2</sub>O ratio of 2.0; whereas, K-based activator combination was composed of 8.0 M KOH solution (28.6% w/w) and K<sub>2</sub>SiO<sub>3</sub> solution (71.4% w/w) with a SiO<sub>2</sub>/K<sub>2</sub>O ratio of 2.23. The matrix and composite properties of the developed fly ash-based EGCs including workability of the fresh matrix, density, compressive strength and uni-axial tensile behavior were evaluated. The experimental results revealed that the sodium-based EGC (EGC-Na) exhibited superior tensile strain capacity, compressive and uni-axial tensile strengths with significantly enhanced ductility.

**Keywords:** Engineered geopolymer composite (EGC), ECC, strain hardening, geopolymer, fly ash, activator combination, PVA fiber.

## INTRODUCTION

Ordinary Portland cement (OPC) production is responsible for almost 5% of total CO<sub>2</sub> emission which is the main cause of global warming in the world [1-2]. Development of geopolymer as an alternative cement-less binder to OPC was one of the significant breakthroughs in the field of concrete technology in the 20th century. The term geopolymer was initially introduced by Davidovits [3]. Geopolymers are synthesized from materials of geological origin (e.g. metakaolin) or industrial by-products such as fly ash and slag that are rich in silica and alumina with high alkaline activators. In this study, a Class F (low calcium) fly ash was used as the source material. Production of fly ash based geopolymer requires approximately 60% less energy and has at least 80% less CO<sub>2</sub> emission compared to manufacture of OPC [4-10]. Although the greenness potential of geopolymer promotes its application as a promising alternative binder to OPC; however, the inherent brittleness of geopolymer is one of the main hindrances towards the large-scale structural applications of geopolymer in the construction industry which should be suppressed [11]. Fiber reinforcing is one of the highly effective methods in suppressing the

brittleness of cementitious materials. Over the years significant interests have been demonstrated by several researchers around the world in the area of fiber reinforced cementitious composites (FRCCs) leading to the development of high performance fiber reinforced cementitious composites (HPFRCCs). HPFRCC is a class of FRCC characterized by strain hardening under uni-axial tension accompanied by multiple fine cracks [12]. Micromechanically-based designed engineered cementitious composite (ECC) [13] is a special class of HPFRCC with advanced ductility which is about 600 times of the ductility of normal concrete in uni-axial tension [14]. Currently, 100% cement-based matrix is mainly used for the manufacture of ECCs; however, several efforts have also been made to incorporate slag and high volume fly ash as partial replacement of OPC in ECCs mixture design to reduce the use of OPC; thereby reducing the global warming issues associated with the CO<sub>2</sub> emission of the cement industry [15-16].

A more sustainable approach to reduce the use of OPC is the use of alternative cement-less binder such as geopolymer. Recently, an initial study was conducted to demonstrate the feasibility of

developing a geopolymer based ECC, known as engineered geopolymer composite (EGC) where the OPC binder was completely replaced by fly ash-based geopolymer binder which exhibited strain hardening and multiple cracking behaviors [10]. The developed fly ash-based EGC possessed low to moderate compressive and tensile strengths ranging from 17.4 MPa to 27.6 MPa and from 2.9 MPa to 3.4 MPa, respectively. Further research is necessary to improve the properties namely compressive and tensile strengths of the recently developed fly ash-based EGC with relatively low concentration activator combinations. The properties of fly ash-based EGCs are influenced by many parameters such as type of fly ash, type of alkaline activator, activator to fly ash ratio, sand to fly ash ratio, temperature and duration of curing and so on. Previous studies revealed that the type of alkaline activator plays an important role in the geopolymerisation process and has significant effect on the mechanical strength of geopolymer [17-19]. Therefore, for the purpose of the current study, type of alkaline activator was selected as one of the most significant factors in governing the characteristics of the geopolymer matrix and composites. This study is aimed to evaluate the effects of two different sodium-based (Na-based) and potassium-based (K-based) activator combinations on the matrix and composite properties of the recently developed fly ash-based EGC. In terms of matrix properties, workability of the fresh matrix, density, compressive and uni-axial tensile strengths were investigated. With regards to composite properties, density, compressive strength, uni-axial tensile strength and tensile strain capacity were evaluated.

## MATERIALS

The low calcium fly ash (class F) used in this study was supplied from Gladstone power station in Queensland, Australia. Table 1 presents the chemical composition and loss on ignition (LOI) of the fly ash determined by X-ray Fluorescence (XRF). The total does not sum up to 100% because of rounding-off of the percentages. Two different Na-based and K-based activator combinations were used in this study. The Na-based activator combination was composed of 8.0 M sodium hydroxide (NaOH) and D Grade sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) solutions, while the K-based activator combination was composed of 8.0 M potassium hydroxide (KOH) and potassium silicate ( $\text{K}_2\text{SiO}_3$ ) solutions. NaOH solution was prepared with a concentration of 8.0 M using NaOH beads of 97% purity supplied by Sigma-Aldrich 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  $\text{Ms}=\text{SiO}_2/\text{Na}_2\text{O}$ ,  $\text{Na}_2\text{O}=14.7\%$  and  $\text{SiO}_2=29.4\%$ ). NaOH and  $\text{Na}_2\text{SiO}_3$  solutions were mixed together with  $\text{Na}_2\text{SiO}_3/\text{NaOH}$  mass ratio of 2.5 to prepare the Na-

based activator combination. KOH solution was prepared with a concentration of 8.0 M using KOH flakes of 90% purity supplied by Redox 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  $\text{Ms}=\text{SiO}_2/\text{K}_2\text{O}$ ,  $\text{K}_2\text{O}=11.2\%$  and  $\text{SiO}_2=24.8\%$ ). KOH and  $\text{K}_2\text{SiO}_3$  solutions were mixed together with  $\text{K}_2\text{SiO}_3/\text{KOH}$  mass ratio of 2.5 to prepare the K-based activator combination. Properties of the PVA fiber used in this study supplied by Kuraray Co. Ltd. of Japan are presented in Table 2.

**Table 1:** Chemical composition of fly ash and OPC determined by XRF

Chemical	Component (wt. %)
$\text{Al}_2\text{O}_3$	25.56
$\text{SiO}_2$	51.11
CaO	4.3
$\text{Fe}_2\text{O}_3$	12.48
$\text{K}_2\text{O}$	0.7
MgO	1.45
$\text{Na}_2\text{O}$	0.77
$\text{P}_2\text{O}_5$	0.885
$\text{TiO}_2$	1.32
MnO	0.15
$\text{SO}_3$	0.24
LOI <sup>1</sup>	0.57

<sup>1</sup>Loss on ignition

**Table 2:** Properties of PVA fiber

Fiber label	RECS 15
Diameter ( $\mu\text{m}$ )	40
Length (mm)	8
Young's modulus (GPa)	41
Elongation (%)	6
Density ( $\text{gr}/\text{cm}^3$ )	1.3
Strength (MPa)	1600

## EXPERIMENTAL PROCEDURES

### Mixture Designs and Mixing

Two appropriate fly ash-based EGC mix proportions denoted as EGC-Na and EGC-K employing the Na-based and K-based activator combinations, respectively were designed through experiments using the principles of ECC development to establish strain hardening behavior in low calcium (Class F) fly ash-based EGC. Previous studies revealed that water content plays an important role on the properties of geopolymer binders [20]. Hence, in order to compare the effects of different activator combinations, water to geopolymer solids (W/GP solids) ratio as defined by Hardjito et al. [20] was kept constant equals to 0.20 and the amounts of water in the EGC mixtures were adjusted accordingly to account for the

constant W/GP solids ratio. Table 3 presents the mix proportions of the EGCs used in this study. Both EGC mixtures were prepared in a Hobart mixer. To prepare the fly ash-based geopolymer matrix, alkaline activators in the form of solution as well as extra water in the mixture EGC-Na were added to the fly ash and mixed for about 4 min. Once a consistent matrix was reached, the PVA fibers (2% v/v) were gradually added, taking care to ensure uniform fiber dispersion. The whole mixing procedure for each composite generally took 20 min.

Table 3: Mix proportions of the fly ash-based EGCs

Materials	EGC-Na	EGC-K
Fly ash	1.0	1.0
Activator	0.35 <sup>1</sup>	0.35 <sup>2</sup>
Water	0.014 <sup>3</sup>	---
PVA fiber	0.02	0.02
W/GP solids ratio	0.20	0.20

Note: All numbers are mass ratios of fly ash weight except W/GP solids ratios and fiber contents (volume fraction)

<sup>1</sup> Composed of the Na-based activator combination.

<sup>2</sup> Composed of the K-based activator combination.

<sup>3</sup> Extra water added to the Na-based activator combination.

### Casting, Curing and Testing of Specimens

To determine workability of the fresh geopolymer matrix, mini slump tests also known as spread-flow tests were conducted. Details of the mini-slump test can be found in Nematollahi and Sanjayan [18]. Each mix was tested twice. The relative slump value was derived from the following equation:

$$\Gamma_p = (d/d_0)^2 - 1 \quad (1)$$

where  $\Gamma_p$  is relative slump,  $d$  is the average of two measured diameters of the matrix spread and  $d_0$  is bottom diameter of the conical cone, equals to 100 mm in this study [21].

For each mix, compressive strength of the matrix (before addition of the PVA fibers) and the composite (after addition of the PVA fibers) were measured. In this regard, the fresh geopolymer matrices and composites were cast into standard 50 mm plastic cube molds and compacted using a vibrating table. For heat curing, the molds were sealed to minimize moisture loss and placed in an oven at 60° C for 24 hours. At the end of the heat curing period, the specimens were removed from the oven and kept undisturbed until being cool and then removed from the molds and left in the laboratory at ambient temperature until the day of testing. All EGC specimens were tested 3 days after casting. Previous studies have shown that age does not have considerable effect on strength of

geopolymers after the completion of the heating curing period [20]. At the testing day the cube specimens were weighed to determine the density of the matrix and composite specimens. Compressive strength of the specimens was measured according to ASTM C109 [22].

Uni-axial tension tests were conducted to evaluate the behavior of the EGC mixtures under tension. For each mix, at least three composite panels with the dimensions of 400 mm×75 mm×10 mm were cast and cured similar to the cube specimens. The specimens were tested in uni-axial tension under displacement control using MTS testing machine with hydraulic wedge grips. The displacement rate was 0.25 mm/min. A schematic of the uni-axial tension test setup is shown in Fig. 1. Aluminum plates were epoxy glued onto the ends of the specimens to facilitate gripping. Care was taken to ensure proper alignment of the specimens with the machine hydraulic grips. In addition, two linear variable differential transducers (LVDTs) were also connected to the MTS machine to measure displacements between two points on the specimen with a gauge length of 200 mm as shown in Fig. 1. Resulting load versus displacement data were recorded and tensile stress versus strain curves were plotted.

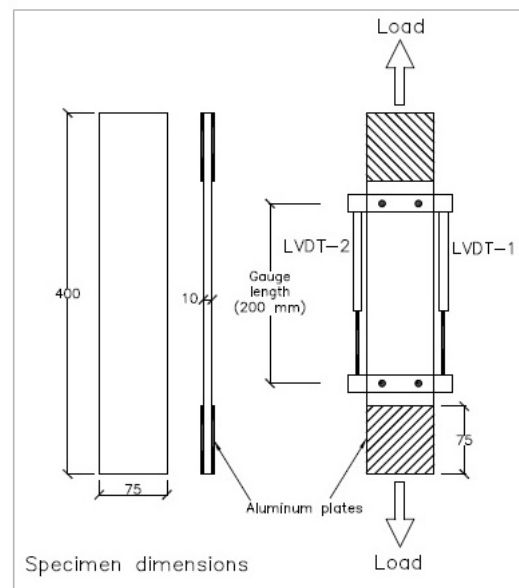


Fig. 1: Uni-axial tension test setup (all dimensions in mm)

## RESULTS AND DISCUSSION

### Workability, Density and Compressive strength

Based on available literature, the Na-based activator combination is the most commonly used activator combination in fly ash-based geopolymer binders [18, 20, 23]. Hence, the EGC-Na composite is selected as the benchmark in this study. The average matrix workability of each composite in terms of relative slump value is shown in Table 4.

According to this table, the matrix workability of the EGC-K composite was 59% higher compared to that of the EGC-Na composite. Moreover, through visual inspection it was noted that the matrix viscosity of the EGC-K composite was much lower than that of the EGC-Na composite. This seems to be due to much higher viscosity of D Grade  $\text{Na}_2\text{SiO}_3$  solution compared to that of the KASIL 2236 Grade  $\text{K}_2\text{SiO}_3$  solution. It should be pointed out that previous studies proved that there is no relationship between slump and viscosity [24-25]. According to Wallevik [26], the slump is influenced by the yield stress of the paste, but it is not affected by the viscosity. Therefore, it can be said that the underlying reason for different matrix workability of the EGC composites lies in their different yield stresses. It can be concluded that using the K-based activator combination resulted in a matrix with higher slump and lower viscosity compared to the matrix with using the Na-based activator combination. The average matrix and composite density of each mix is also presented in Table 4. As shown in this table, the composite densities of both EGCs were comparable. In addition, the composite densities of both EGCs were relatively lower compared with their corresponding matrix density. This may be attributed to a fiber induced damage effect, which results in a composite with higher porosity compared with the matrix material alone [27].

The average matrix and composite compressive strength of each mix is also presented in Table 4. As shown in this table, in both EGCs, the compressive strength of the composite specimens increased due to addition of the PVA fibers (2% v/v) with respect to that of the matrix specimens.

According to Table 4, the matrix and composite compressive strength of the EGC-Na composite were about 70% higher than that of the EGC-K composite. In addition, although the concentration of the activator combinations was limited to 8.0 M to account for safety consideration; however, the compressive strength of both EGCs developed in this study was higher than that of the EGCs developed by Ohno and Li [11] which was ranging from 17.4 MPa to 27.6 MPa. This could be mainly due to the longer heat curing period (i.e. 24 hours at 60°C) employed in this study. As shown in Table 4, although the matrix workability of the EGC-Na composite was 37.1% lower than that of the EGC-K composite; however, its compressive strength was 70% higher. Fernández-Jiménez and Palomo [17] found out that in fly ash-based geopolymer, regardless of the type of activator, the main reaction product formed is an alkaline aluminosilicate gel with low-ordered crystalline structure. However, the microstructure as well as the Si/Al and the Na/Al ratios of the aluminosilicate gel depend on the type of the activator used. It is thereby hypothesized that the microstructure of matrix of the EGC-Na composite is different to that of the EGC-K composite. Therefore, it can be said that the most prominent reason for different matrix compressive strength the EGC composites lies in their different microstructure of the geopolymer matrices. In addition, the price of  $\text{K}_2\text{SiO}_3$  and KOH solutions is higher than that of the NaOH and  $\text{Na}_2\text{SiO}_3$  solutions [28]. Thus, it can be concluded that in fly ash-based EGCs the use of Na-based activator combination is beneficial in terms of lower cost and higher compressive strength gain compared with the K-based activator combination.

Table 4: Workability, density and compressive strength of each mix

Mix ID	Fresh matrix workability	Density; ( $\text{kg/m}^3$ )		Compressive strength; (MPa)	
		Matrix	Matrix	Composite	Matrix
EGC-Na	6.92	1859.3	54.6	63.7	1859.3
EGC-K	11.0	1845.3	32.3	37.3	1845.3

### Uni-axial Tensile Performance

Fig. 2 presents the tensile stress-strain behavior of the fly ash-based EGCs. As shown in this figure, both fly ash-based EGCs, regardless of their type of activator combinations, exhibited strain hardening behavior accompanied by multiple cracking. The uni-axial tensile performances of the fly ash-based EGCs developed in this study are comparable to those of the fly ash-based EGCs developed by Ohno and Li [11] and the conventional PVA-ECC [29]. The first-crack strength, ultimate tensile strength and tensile strain capacity of each composite is summarized in Table 5. According to this table, in both fly ash-based EGCs, regardless of their type of activator combinations, the ultimate

tensile strength of the composite was significantly higher than the composite first-crack strength. Thus, the stress-based condition for pseudo-strain hardening (PSH) behavior is satisfied [29]. In addition, the EGC-Na composite exhibited the highest ultimate tensile strength of 4.7 MPa which was higher than that of the Na-based EGCs developed by Ohno and Li [11] which ranged from 2.9 MPa to 3.4 MPa. Moreover, the first-crack strength and ultimate tensile strength of the EGC-Na composite were 112.9% and 161.1%, respectively, higher than those of the EGC-K composite. The increase in the ultimate tensile strength, which was higher than the first-crack strength, could be due to the interfacial properties.

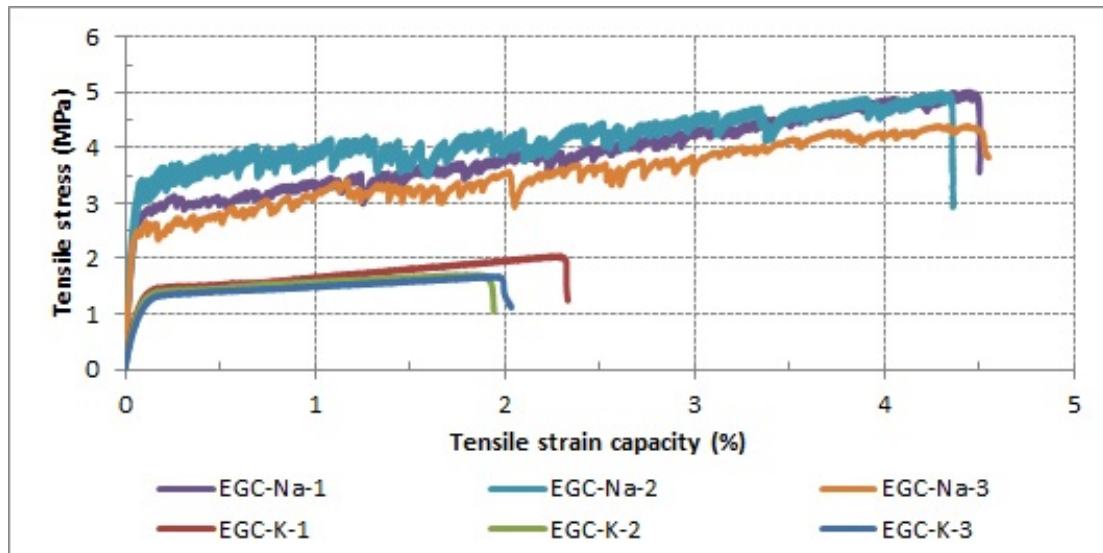


Fig. 2: Tensile stress vs. tensile strain capacity of EGC-Na and EGC-K

Table 4: Uni-axial tension test results

Properties	EGC-Na	EGC-K
1st-crack strength, $\sigma_{fc}$ (MPa)	$0.66 \pm 0.053$	$0.31 \pm 0.029$
Ultimate tensile strength, $\sigma_{cu}$ (MPa)	$4.7 \pm 0.25$	$1.8 \pm 0.21$
Tensile strain capacity, $\varepsilon_{cu}$ (%)	$4.3 \pm 0.14$	$2.0 \pm 0.26$
stress-performance index ( $\sigma_{cu}/\sigma_{fc}$ )	7.1	5.8

In other words, the chemical bonding energy and the frictional bond strength of the EGC-Na composite increased more than the cracking strength compared with the EGC-K composite; resulting in higher fiber bridging strength [30].

According to Table 5, the tensile strain capacity of the EGC-Na composite was significantly higher (i.e. 115% higher) than that of the EGC-K composite. Kanda and Li [31] proposed two performance indices namely stress-performance index ( $\sigma_0/\sigma_{fc}$ ) and energy-performance index ( $J'_b/J_{tip}$ ) where  $\sigma_0$  is the maximum fiber bridging stress (i.e. ultimate tensile strength of the composite ( $\sigma_{cu}$ )),  $\sigma_{fc}$  is the tensile first-crack strength of the composite,  $J'_b$  is the complementary energy and  $J_{tip}$  is the composite crack tip toughness. Theoretically, both performance indices must exceed unity for achieving PSH behavior in a fiber reinforced composite. The higher the performance indices values, the greater the possibility of saturated multiple cracking or saturated PSH behavior which results in higher tensile strain capacity of the composite. One of the underlying reasons for the significant difference in the tensile strain capacity of the EGC-Na composite compared to that of the EGC-K composite lies in their different stress-performance indices. The stress-performance index ( $\sigma_0/\sigma_{fc}$ ) of each composite is also presented in

Table 5. As shown in table, the stress-performance index of the EGC-Na composite is 22% higher than that of the EGC-K composite.

In addition, the considerably different tensile strain capacity of the EGC-Na and EGC-K composites is also attributed to their considerably different energy-performance indices ( $J'_b/J_{tip}$ ). With regards to the  $J_{tip}$ , the lower tensile first-crack strength of the EGC-K composite indicates its lower  $J_{tip}$  compared to that of the EGC-Na composite [29]. On the other hand, the significantly higher ultimate tensile strength and strain of the EGC-Na composite indicates its considerably higher  $J'_b$  compared to that of the EGC-K composite. The higher the  $J'_b$  and the lower the  $J_{tip}$  values, the higher the energy-performance index. Thus, it can be concluded that the energy-performance index of the EGC-Na composite is significantly higher than that of the EGC-K composite. In other words, the margin between  $J_{tip}$  and  $J'_b$  in the EGC-Na composite is considerably higher than that of the EGC-K composite which is desirable for developing intense multiple cracking process and the saturated PSH behavior [32]. This is another reason for the significantly higher tensile strain capacity of the EGC-Na composite compared to that of the EGC-K composite.



## CONCLUSIONS

Randomly oriented short PVA fiber reinforced fly ash-based EGCs were developed in this study. The effects of two different Na-based and K-based activator combinations on the matrix and composite properties of the developed fly ash-based EGCs including workability of the fresh matrix, density, compressive strength and uni-axial tensile behaviors were evaluated. Both fly ash-based EGCs, regardless of their type of activator combinations, exhibited strain hardening behavior accompanied by multiple cracking in uni-axial tension similar to the conventional PVA-ECC. The workability of the fresh matrix of the EGC-K composite was 59% higher than that of the EGC-Na composite. However, the EGC-Na composite exhibited the highest compressive, uni-axial tensile strength of 63.7 MPa and 4.7 MPa, respectively with very high average tensile strain capacity of 4.3%. The following conclusions can be made from the experimental study conducted:

1) The tensile strain capacity of the EGC-Na composite was 115% higher than that of the EGC-K composite. The higher stress-performance index ( $\sigma_0/\sigma_{fc}$ ) of the EGC-Na composite compared to that of the EGC-K composite is one of the underlying reasons for its significantly high tensile strain capacity. In addition, it is also attributed to the considerably higher complementary energy and energy-performance index ( $J_b/J_{tip}$ ) of the EGC-Na composite compared to that of the EGC-K composite.

2) The first-crack strength of the EGC-Na composite was considerably higher (112.9%) than that of the EGC-K composite. The ultimate tensile strength of the EGC-Na composite was also significantly higher (161.1%) compared to that of the EGC-K composite. It could be attributed to the higher fiber-matrix interfacial properties of the EGC-Na composite compared to the EGC-K composite which resulted in higher fiber bridging strength of the EGC-Na composite.

Therefore, it can be concluded that in fly ash-based EGCs the use of the Na-based activator combinations is highly beneficial in terms of lower cost, higher compressive strength and superior uni-axial tensile behavior compared to the K-based activator combinations.

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