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Author

Leong, Hsiao Yun, Ong, Dominic Ek Leong, Sanjayan, Jay G, Nazari, Ali

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The effect of different Na_2O and K_2O ratios of alkali activator on compressive strength of fly ash based-geopolymer

Hsiao Yun Leong[a], Dominic Ek Leong Ong[a], Jay G. Sanjayan[b] and Ali Nazari[b*]

a) Research Centre for Sustainable Technologies, Faculty of Engineering, Science & Computing, Swinburne University of Technology Sarawak Campus, 93350 Kuching, Sarawak, Malaysia.

b) Centre for Sustainable Infrastructure, Faculty of Science, Engineering and Technology, Swinburne University of Technology, PO Box 218, Hawthorn Victoria 3122, Australia.

* Corresponding author, Tel: +61 3 92148370, E-mail: <u>alinazari@swin.edu.au</u>

Abstract

Fly ash sourced from Sarawak, Borneo was alkali-activated by a combination of alkali hydroxides (i.e. NaOH, KOH or $Ca(OH)_2$) and sodium silicate (i.e. industrial or commercial grade Na_2SiO_3) in this research. A comprehensive overview of the compressive strength of geopolymer due to different alkali activator/fly ash and $Na_2SiO_3/NaOH$ (Na_2SiO_3/KOH or $Na_2SiO_3/Ca(OH)_2$) were studied. The results show that geopolymer made using Sarawak fly ash can be a potential construction material depending on the requirements of workability and compressive strength. The core study of this research was the investigation on the variations of compressive strength due to the oxide molar ratios. The results revealed that higher oxide molar ratios do not necessarily lead to higher compressive strengths. SiO_2/Na_2O, Al_2O_3/Na_2O, SiO_2/K_2O and Al_2O_3/K_2O weight ratios had dissimilar effects on the compressive strength of NaOH- and KOH-based geopolymer. The reduction of SiO_2/Na_2O and Al_2O_3/Na_2O in NaOH- and KOH-based geopolymer using commercial grade Na_2SiO_3 intensively

increased the strength capability. The ratios of SiO_2/Na_2O and Al_2O_3/Na_2O in $Ca(OH)_2$-based geopolymer were the highest, nevertheless, the compressive strength and workability of $Ca(OH)_2$-based geopolymer were the weakest in comparison to NaOH- and KOH-based geopolymers. Na_2O was postulated as the major alkali oxide required for the strength development rather than K_2O in both NaOH- and KOH-based geopolymer. However, K_2O in KOH-based geopolymer may govern the strength development when Na_2O is low in the mixture.

Keywords

Geopolymer, Sarawak fly ash, alkali activator/ash, mixture proportions, compressive strength

1. Introduction

In Malaysia, there are a total of 6 power stations in the country. 4 power stations situate in Peninsula Malaysia namely, Tanjung Bin Power Station in Johor (700 MW × 3 units), Jimah Power Station in Negeri Sembilan (700 MW × 2 units), Sultan Azlan Shah Power Station in Manjung (700 MW × 3 units) and Sultan Salahuddin Abdul Aziz Power Station in Kapar (752 MW × 2 units) [1, 2]. 2 power stations are located in Sarawak, Borneo namely, Sejingkat Power Station in Kuching (50 MW × 2 units and 55 MW × 2 units) and Mukah Power Station in Mukah (135 MW × 2 units).

The consumption of coal for annual combustion is approximately 1 million tons at Sejingkat Power Station. The production of fly ash is approximately 7 to 10% out of the annual coal consumption. Most of the captured fly ash is disposed into the ash ponds nearby and only some of the fly ash has been utilised for construction purposed. As the

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environmental issue becomes global concern, it is crucial to widen the application of this industrial waste in order to reduce the disposal rate.

There are several types of alkali activators that can be used to activate fly ash. For example, sodium hydroxide (NaOH) [3-8], potassium hydroxide (KOH) [3, 4], sodium silicate (Na_2SiO_3) [3-8], potassium silicate (K_2SiO_3)[3, 4], calcium hydroxide ($Ca(OH)_2$) [9-11] etc. The combined use of NaOH and Na_2SiO_3 is the most common practice in term of cost effectiveness to produce geopolymer with good compressive strength than the solely use of alkali silicate or hydroxide.

The ratio of alkali activator/ash can be a critical parameter to the strength development [12]. High alkali activator/ash increases the viscosity and difficulty in compaction [13]. The workability and compressive strength decrease by further increment of the alkali activator/ash and Na_2SiO_3/NaOH [8]. Hardjito and Rangan [14] suggested that Na_2SiO_3/NaOH=2.5 by mass is suitable for fly ash-based geopolymer synthesis in term of cost effective and consistency of the results. The change of Si content and Na+ ion due to different ratios of alkali activators have significant effect on the geopolymerisation as well as the strength development [15]. The increase of Si content increases the SiO_2/Al_2O_3 ratio resulting in more formation of Si-O-Si bonds [16], which are stronger than Si-O-Al and Al-O-Al bonds [17], thus leading to strength gain.

Xu and Deventer [18] claim that KOH-based geopolymer obtains higher compressive strength compared with NaOH-based geopolymer. Although NaOH solution exhibits higher extent of mineral dissolution, better geopolymerisation in KOH solution enhances the strength development. However, Palomo, Grutzeck [4] reported that KOH-based geopolymer does not certainly lead to the highest compressive strength. NaOH-

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based geopolymer may obtain higher compressive strength than KOH-based geopolymer when the curing temperature, curing age or alkali activator/ash is varied. Duxson, Mallicoat [19] concluded that the Si/Al is a significant parameter on the study of effect of alkali cation from the alkali activators on the compressive strength.

Na_2O and K_2O ratios of alkali activator are different in various mixture ratios. It has been reported in term of SiO_2/Na_2O and Al_2O_3/Na_2O elsewhere. However, limited study on the ratio of K_2O has been reported. The increment of silicate solution increases the ratio of SiO_2/Na_2O [9, 20]. Geopolymer tends to be less stable at low SiO_2/Na_2O than at high SiO_2/Na_2O [20]. The highest compressive strength is obtained at intermediate ratio of SiO_2/Na_2O [7]. However, it declines simultaneously with the increment of SiO_2/Na_2O and Al_2O_3/Na_2O [5].

The effect of different ratios of alkali activator/ash and $Na_2SiO_3/NaOH$ (KOH) on compressive strength of fly ash-based geopolymer has been studied by many researchers as mentioned earlier on. However, the study of mentioned ratios on geopolymer using Sarawak fly ash is still very limited. The appropriate mixing ratio to obtain the desired compressive strength and workability for industrial usage remains unclear. The effects of Si/Al, SiO_2/Na_2O and Al_2O_3/Na_2O have been reported elsewhere [3, 5, 7, 9, 20-24]. However, the study on variation of Na_2O and K_2O due to different ratios of alkali activator is very limited. The main objective of this research is to develop a comprehensive overview on compressive strength of geopolymer using Sarawak fly ash at different mixture ratios. It is aimed to provide a better understanding of Sarawak fly ash-based geopolymer for developing industrial applications in Sarawak. Essentially, this research studies the compressive strength of geopolymer in term of

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oxide molar ratios in particular the effect of different Na_2O and K_2O due to different ratios of alkali activator.

2. Experimental Procedure

2.1. Materials

Sarawak fly ash obtained from Sejingkat Power Station was used in this research. **Table 1** shows the chemical compositions of the fly ash using WD-X-ray Fluorescence Spectrometer (WD-XRF). This fly ash can be classified as Class F type in accordance to the ASTM-C618 [25] standard.

The chemical compositions of fly ash sourced from other coal fired power stations in Malaysia reported by other researchers [26, 27] are also tabulated in **Table 1**. It is worth to mention that fly ash source from same power station may behave differently in chemical compositions when the collection time is different [2, 28-31]. However, the studies of fly ash sourced from Mukah Power Station and Jimah Power Station is very limited.

The results show that the chemical compositions of fly ash sourced from different power stations in Malaysia show dissimilarity. It could be due to the sources of combustion coal, coal type, geology of coal etc. [32]. All the fly ash is classified as Class F type except the fly ash sourced from Sultan Azlan Shah Power Station which is classified as Class C type. The studied fly ash shows moderate percentage of SiO_2 and Al_2O_3 content but low Fe_2O_3 and CaO content in comparison to the other fly ash sourced from different power stations. The MnO content in the studied fly ash is found to be extraordinary. Furthermore, it is similar to the MnO content in manganese slag as shown in **Table 1**. Juda-Rezler and Kowalczyk [33] reported that Mn content can be

bounded in pyrite and illite minerals in the original coal which should be removed during pre-combustion coal cleaning. The results have shown well agreement to Sia and Abdullah [34] reported that the Mn content in fly ash produced from Balingian coal in Sarawak is much higher and commonly higher than the original coal. It implies the low volatility of this content when it is subjected to combustion.

Washed sand with saturated surface dry condition was prepared prior to sample casting. Four series of alkali activators were studied in this research. Series 1 was a combination used of 8M NaOH and industrial grade Na_2SiO_3. Series 2 was a combination used of 8M KOH and industrial grade Na_2SiO_3. Different ratios of alkali activator/ash (0.3, 0.4, 0.5 and 0.6) and Na_2SiO_3/NaOH or Na_2SiO_3/KOH (0.5, 1, 1.5, 2, 2.5 and 3) were studied. Industrial grade Na_2SiO_3 (8.8%~9.5% Na_2O and 28%~30.6% SiO_2 by weight) was then completely replaced with the commercial grade Na_2SiO_3 (12% Na_2O and 30% SiO_2 by weight) in samples of alkali activator/ash=0.4; this was denoted to Series 3. This series provides in-depth study on the effect of different grades (i.e. different compositions of SiO_2 and Na_2O) of Na_2SiO_3 on compressive strength in particular to the oxide molar ratio. Finally, a combination used of $Ca(OH)_2$ and commercial grade Na_2SiO_3 was used to produce $Ca(OH)_2$-based geopolymer (Series 4). KOH or NaOH was replaced by $Ca(OH)_2$ in order to study the performance of strength and workability in comparison to the more commonly use of NaOH- and KOH-based geopolymer.

Table 1. Chemical Composition of Fly Ash

Elements (%)	Fly Ash				Manganese Slag [35]
Power Station	Sejingkat Power Station	Sultan Azlan Shah Power Station [26]	Sultan Abdul Aziz Shah Power Station [26]	Tanjung Bin Power Station [27]	-
Location	Kuching, Sarawak	Manjung, Perak	Kapar, Selangor	Pontian, Johor	-
SiO_2	43.8	26.4	52.11	40.08	28.3
Al_2O_3	18.1	9.25	23.59	18.81	10.5
Fe_2O_3	7.7	30.13	7.39	17.7	0.3
CaO	3.9	21.6	2.61	14.32	11
MgO	0.5	-	-	1.52	14.9
MnO	22.8	0.27	0.49	-	26
K_2O	2	2.58	0.42	1.84	5.1
Na_2O	0.3	-	0.78	0.72	2.7
SO_3	0.1	1.3	1.31	0.1	-
TiO_2	0.6	3.07	0.88	4.44	-
P_2O_5	0.1	0.67	0.8	0.37	-
BaO	-	-	-	0.85	-
LOI	0.5	-	-	-	-

Note: The chemical compositions of fly ash source from Mukah Power Station and Jimah Power Station are not available elsewhere

2.2. Sample Preparation and Tests

The mixture ratios of the geopolymer samples are tabulated in **Table 2**. Fly ash was initially mixed with sand using the Mortar mixer. The alkali activators were then added into the mixture for further mixing of 5 minutes. The mixture was casted into the cubic moulds with dimensions of 50mm × 50mm × 50mm. The samples were placed on the vibrating table to remove the air bubbles within the sample. After the vibration finished, the samples were sealed with plastic sheet to prevent the loss of moisture during the

heat curing in oven at 60°C for 24 hours. The samples were then demoulded for cube test or further curing at room temperature until the date test.

The workability of the mixture and the compressive strength of the samples were obtained using the flow table in accordance to the ASTM-C1437 [36] standard and compression test machine in accordance to the ASTM-C109/C109M [37] standard, respectively.

The designation of each sample is represented as 'Series No. / Use of NaOH, KOH or Ca(OH)$_2$/ Na$_2$SiO$_3$/NaOH(KOH or Ca(OH)$_2$)/ Alkali activator/ash/ Water/binder'. For example, S1/Na/0.5/0.3/0 denotes the sample at Series 1, use of NaOH with Na$_2$SiO$_3$/NaOH=0.5, alkali activator/ash=0.3 and water/binder=0.

Table 2. The mixture ratios of the geopolymer samples

Series	Ash/sand ratio	Alkali activator type	Na$_2$SiO$_3$/NaOH (KOH or Ca(OH)$_2$) ratio	Alkali activator/ash ratio	Water /binder
1	1:2	NaOH and industrial grade of Na$_2$SiO$_3$		0.3,0.4,0.5,0.6	-
2	1:2	KOH and industrial grade of Na$_2$SiO$_3$	0.5, 1, 1.5, 2, 2.5, 3	0.3,0.4,0.5,0.6	-
3	1:2	NaOH or KOH and commercial grade of Na$_2$SiO$_3$		0.4	-
4	1:2	Ca(OH)$_2$ and commercial grade of Na$_2$SiO$_3$	0, 0.5, 1, 2, 3	0.3	0.4
			0.5	0.3,0.4,0.5,0.6	0.4
			0.5	0.3	0.4,0.5,0.6,0.7

3. Results and Discussion

3.1. Workability

From the alkali activator/ash point of view, the workability increases when the ratio is increased as shown in **Figs. 1a-1b**. The highest workability (i.e., approx. 250 mm) is

obtained at alkali activator/ash of 0.6. As the amount of the liquid content is greater than the solid content at higher ratio, the mixture tends to become more fluid thus enhancing the workability. It is also observed that the final volume of the mixture decreases when more liquid content is added to the mixture. It is most likely due to the better dissolution of fly ash when higher alkali activator/ash is used. When less liquid content is presented in the mixture at alkali activator/ash of 0.3, the workability is unmeasurable. It is because that the mixture collapses instead of flowing when it is subjected to flow table test. It is supposed that the alkali activators are insufficient to dissolve the fly ash particles. Most of the solid particles are still clearly seen throughout the mixture. In general, KOH-based geopolymer shows higher workability than NaOH-based geopolymer. It is most probably due to higher solubility of potassium than sodium.

The effect of Na_2SiO_3/NaOH(KOH) on the workability is in the decreasing manner as represented in **Fig. 1c-1d**. The highest workability is obtained on the samples with Na_2SiO_3/NaOH(KOH) ratio of 0.5 but the lowest at ratio of 3. It can be explained by the different percentage of Na_2SiO_3 and NaOH(KOH) in the mixtures. As Na_2SiO_3 is highly viscous, it increases the viscosity of the mixture when the ratio is increased thus reducing the workability. Inversely, the fluid-like nature of NaOH or KOH increases the workability.

As shown in **Figs. 1c-1d**, the workability of KOH-based geopolymer is generally higher than the NaOH-based geopolymer when commercial grade Na_2SiO_3 is used. It is found that the workability of NaOH-based geopolymer decreases but KOH-based geopolymer increases when commercial grade Na_2SiO_3 is used in comparison to industrial grade Na_2SiO_3. It is noted that, according to our visual observations, the viscosity of commercial grade Na_2SiO_3 is slightly higher than the industrial grade

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Na$_2$SiO$_3$. The increase of concentration in silicate may increase the viscosity of solution [38] thus reducing the workability. However, it is supposed that the solubility of potassium has superior effect than the viscosity of silicate on workability when Na$_2$SiO$_3$/KOH is low (i.e. higher amount of KOH than Na$_2$SiO$_3$). It explains why the workability of geopolymer using commercial grade Na$_2$SiO$_3$ is similar or lower than those using industrial grade Na$_2$SiO$_3$ at higher Na$_2$SiO$_3$/KOH.

(a) NaOH + Industrial grade of Na$_2$SiO$_3$ (b) KOH + Industrial grade of Na$_2$SiO$_3$

(c) (NaOH) Industrial grade of Na$_2$SiO$_3$ vs. Commercial grade of Na$_2$SiO$_3$

(d) (KOH) Industrial grade of Na$_2$SiO$_3$ vs. Commercial grade of Na$_2$SiO$_3$
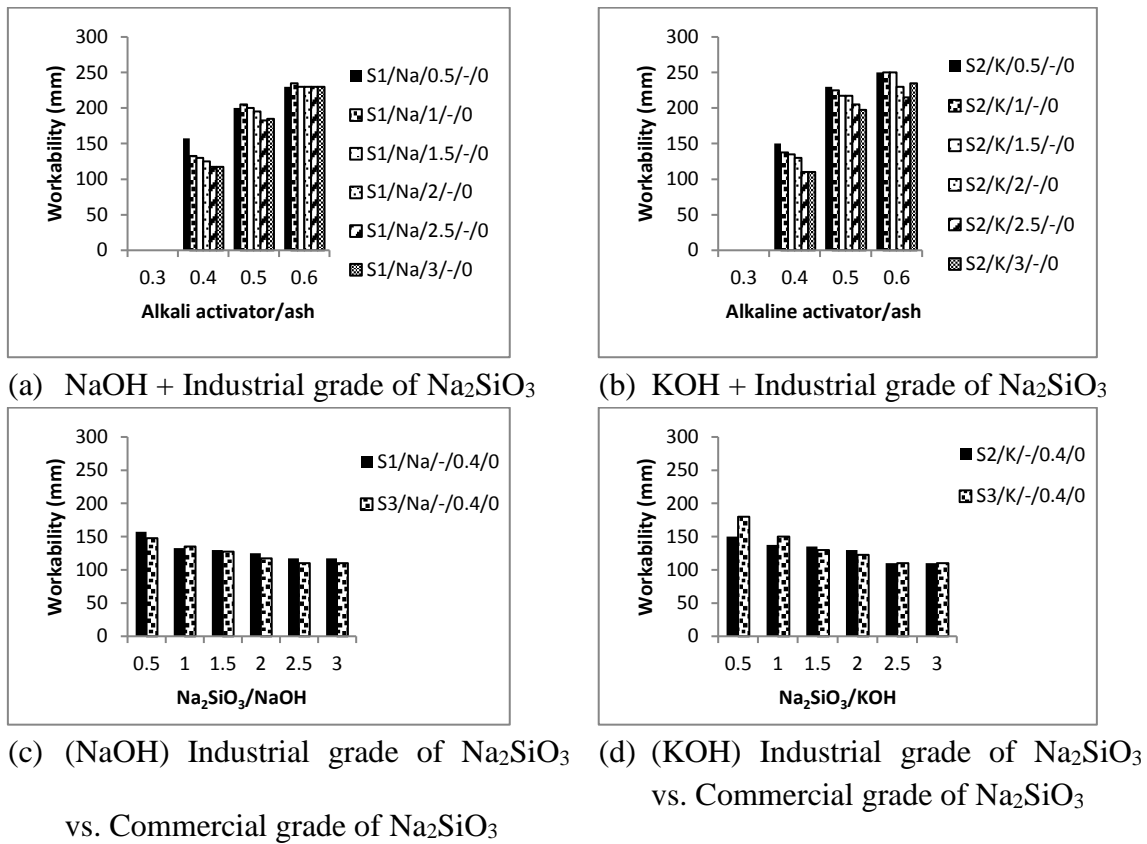
Fig. 1 Workability of the mixtures

3.2. Compressive Strength

3.2.1. Effect of alkali activator/ ash on compressive strength

Fig. 2 shows the effect of alkali activator/ash on compressive strength of NaOH- or KOH-based geopolymer using industrial grade Na$_2$SiO$_3$. When the alkali activator/ash

is very low (i.e., 0.3), the compressive strength is insignificant. It is supposed that the low amount of alkali activator at lower alkali activator/ash is insufficient to react with the fly ash as mentioned earlier. Therefore, the formation of rigid and coherent structure is unlikely consequently lowering the compressive strength. The compressive strength initially increases but decreases with the further increment of alkali activator/ash. The optimum compressive strength was observed at alkali activator/ash of 0.4 regardless of the type of alkali activators and curing conditions. It could be due to the higher extent of dissolution of fly ash when the alkali activator/ash is increased.

It was observed that fewer air bubbles are released from the sample surface at alkali activator/ash of 0.4 during the vibration. In comparison, greater amount of continuous air bubbles move towards the surface when the alkali activator/ash is increased. It is supposed that air bubbles which are not able to release from the sample may create pores thus producing more porous structure. The pores leave within the sample may weaken the geopolymer structure and compressive strength.
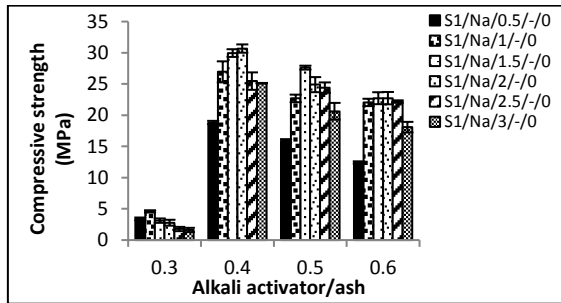
(a) Series 1: 60 degree – 1 day

(d) Series 2: 60 degree – 1 day

(b) Series 1: 60 degree – 7 days
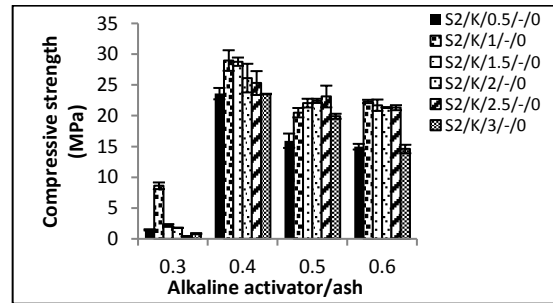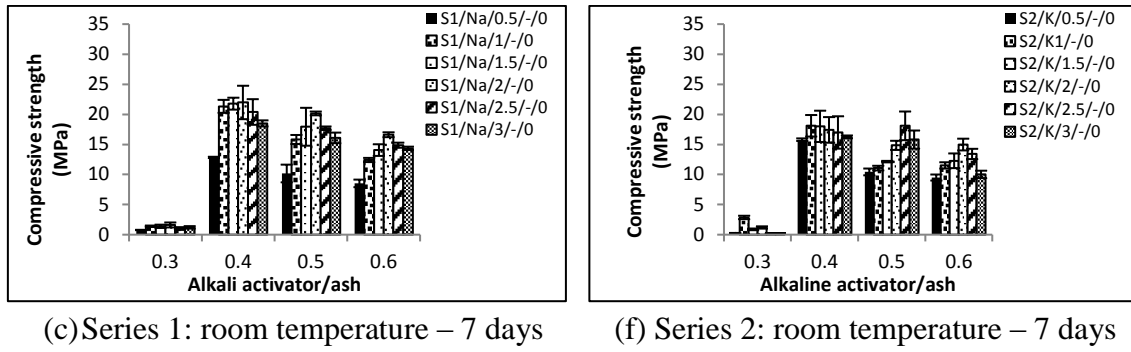
(e) Series 2: 60 degree – 7 days
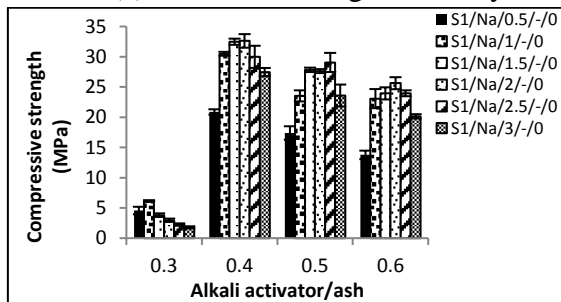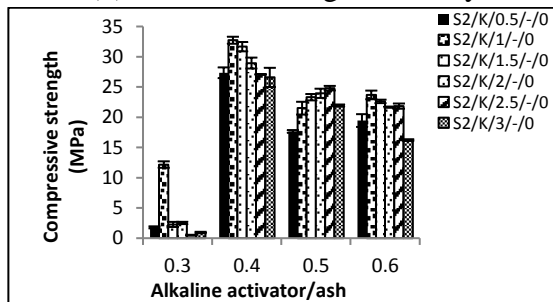
(c) Series 1: room temperature – 7 days   (f) Series 2: room temperature – 7 days

Fig 2. The effect of alkali activator/ash on compressive strength using industrial grade of Na_2SiO_3 with (a-c)Series 1: NaOH (d-f) Series 2: KOH

3.2.2. Effect of $Na_2SiO_3/NaOH$ or Na_2SiO_3/KOH ratio on compressive strength

Fig. 3 shows that the compressive strength increases when $Na_2SiO_3/NaOH(KOH)$ is increased. Duxson, Provis [20] claim that the amount of aluminosilicate gel form is correlated with the compressive strength of geopolymer, in which higher formation of gel will lead to higher geopolymer strength. The highest compressive strength was obtained at $Na_2SiO_3/NaOH=2$ and $Na_2SiO_3/KOH=1$, both have alkali activator/ash of 0.4. Further increment results in strength loss. As the amount of Na_2SiO_3 within the sample rises, it provides more Si content to the system for geopolymerisation thus improving the strength gains [18]. However, excessive Na_2SiO_3 inhibits the geopolymerisation due to the formation of aluminosilicate gel precipitation [39-42]. It furthers apart the aluminosilicate source (i.e. fly ash) and the alkali activators. Therefore, it is supposed to obstruct the alkali hydroxide to leach out the Al and Si content from the reacting material and hinder the alkali hydroxide as catalyst in the system. Additionally, this excessive silicate from higher ratio of $Na_2SiO_3/NaOH(KOH)$ affects the water evaporation during the condensation reaction [43]. The excessive Na+ may

form sodium carbonate when it contacts with CO_2 from the atmosphere subsequently reducing the strength development [44].
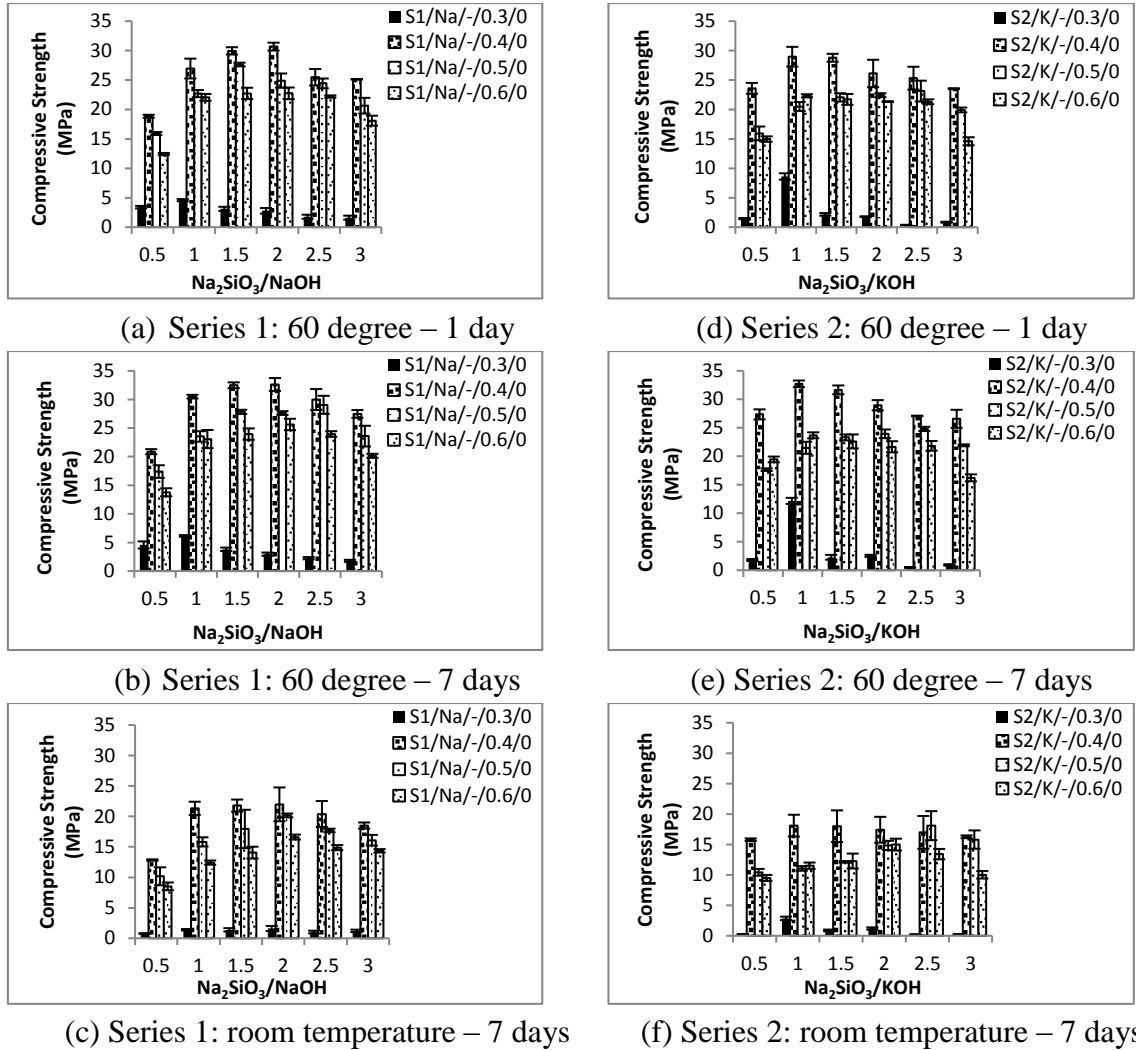

(a) Series 1: 60 degree – 1 day

(d) Series 2: 60 degree – 1 day

(b) Series 1: 60 degree – 7 days

(e) Series 2: 60 degree – 7 days

(c) Series 1: room temperature – 7 days

(f) Series 2: room temperature – 7 days

Fig 3. The effect of Na_2SiO_3/NaOH or Na_2SiO_3/KOH on compressive strength using industrial grade of Na_2SiO_3 with (a-c) Series 1: NaOH and (d-f) Series 2: KOH

3.2.3. Comparison of strength performance between NaOH- and KOH-based geopolymer

In this section, the effect of NaOH- and KOH-based geopolymers using industrial grade Na_2SiO_3 on the compressive strength is only considered. Those using commercial grade Na_2SiO_3 will be discussed here-in-after.

As shown in **Fig. 4**, the compressive strength of both NaOH- and KOH-based geopolymers cured at 60°C after 1 day and 7 days are similar. At $Na_2SiO_3/NaOH(KOH)=0.5$ and 1, KOH-based geopolymer shows higher compressive strength than NaOH-based geopolymer. It is because KOH-based geopolymer has lower porosity in its structure [3]. Additionally, KOH solution is capable to leach out more Al content than Si content as compared with NaOH solution [45]. The negative charge of Al^{3+} in IV fold coordination can be compensated by the positive charge of alkali ion such as K^+ or Na^+ [46]. Xu and Deventer [18] state the K^+ from KOH solution favours the formation of ionic pair (i.e. cation-anion) reaction with larger silicate oligomers. It demonstrates more existence of the geopolymer precursors (i.e. long chain silicate oligomers and Al-O-Si complexes) thus revealing stronger compressive strength.

When $Na_2SiO_3/NaOH(KOH)$ is greater than 1.5, NaOH-based geopolymer obtains higher compressive strength than KOH-based. It seems that the dissolution of KOH-based geopolymer tends to become lower and the geopolymerisation is affected by the increase of silicate content. Xu and Deventer [18] claim that higher extent of dissolution is expected in NaOH-based geopolymer than in KOH-based. The smaller ionic size of Na^+ than K^+ favours the ionic pair reaction with the smaller silicate oligomers thus enhancing the bond strength. Whereas, stronger ionic pair formation is anticipated when higher amount of sodium silicate is used.

At room temperature after 7 days, NaOH-based geopolymer obtains higher compressive strength of approx. 12% to 23% than KOH-based geopolymer. It shows that NaOH-based geopolymer exhibits better performance on compressive strength than KOH-based at room temperature.
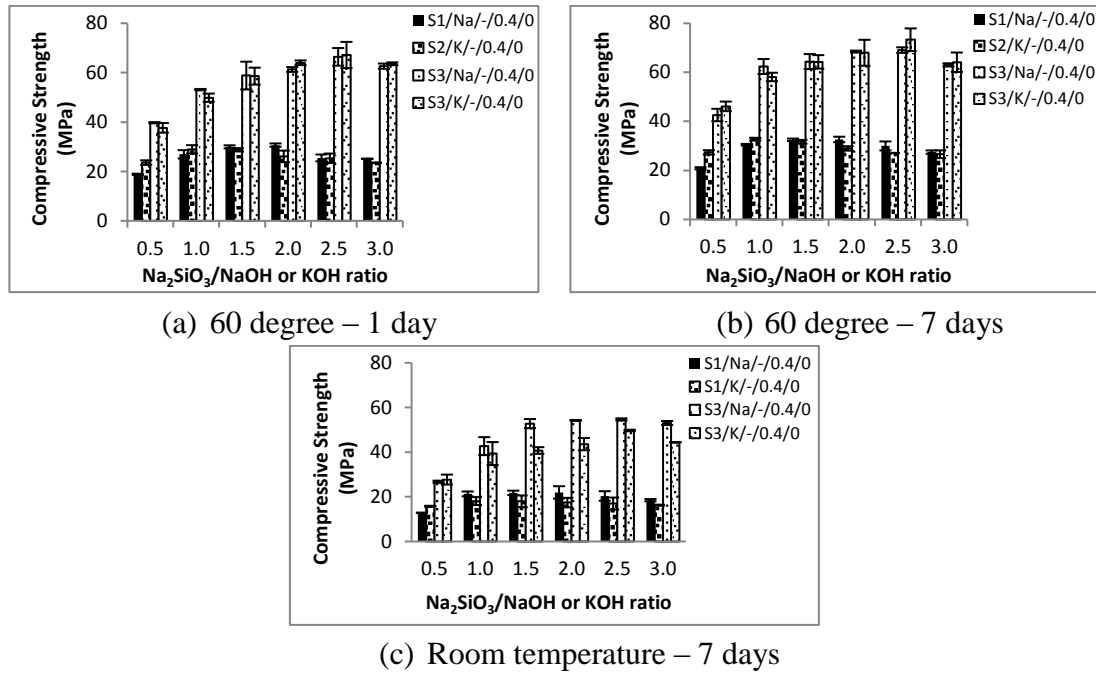
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(a) 60 degree – 1 day

(b) 60 degree – 7 days

(c) Room temperature – 7 days

Fig 4. The effect of NaOH and KOH on the compressive strength

3.2.4. Compressive strength of geopolymer in term of oxide molar ratios

As shown in **Fig. 5**, the oxide molar ratios of geopolymer is governed by the ratios of Na_2SiO_3/NaOH(KOH) and alkali activator/ash. Both amorphous and crystalline phase of SiO_2 and Al_2O_3 are considered. The optimum SiO_2/Al_2O_3, CaO/SiO_2, SiO_2/Na_2O and Al_2O_3/Na_2O ratios for the highest compressive strength on NaOH-based geopolymer in this research are 4.846, 0.081, 10.167 and 2.098, respectively. For same oxide molar ratio studies, it is 4.662, 0.084, 10.822 and 2.321 on KOH-based geopolymer, respectively. These confirm that the highest compressive strength is achievable at intermediate ratios. Higher ratios of the oxide molar do not necessarily lead to higher compressive strength. The optimum oxide molar ratios with high pH value allow the breakage of the glassy chain on fly ash surface through the alkaline activation. Thus, the internal Si and Al components can react with the alkali ion (e.g. Na^+) subsequently producing the amorphous reaction products through the geopolymerisation [5].

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When alkali activator/ash is increased, SiO_2/Al_2O_3 increases but CaO/SiO_2, $SiO_2/Na_2O(K_2O)$ and $Al_2O_3/Na_2O(K_2O)$ decrease regardless of types of geopolymer. The exception is observed when the alkali activator/ash is increased, SiO_2/K_2O of NaOH-based geopolymer increases.

For both NaOH- and KOH-based geopolymer, CaO/Al_2O_3 is constant at 0.392 as shown in **Figs. 5a-5b**. It is because the alkali activators do not contribute to CaO and Al_2O_3. These two oxides are provided by fly ash.

Figs. 5c-**5d** shows higher compressive strength is achievable when SiO_2/Al_2O_3 is increased. It is most likely due to the denser formation of Si-O-Si bond, which is shorter and stronger than Si-O-Al or Al-O-Al bonds [17, 20]. Strength loss by further increment of SiO_2/Al_2O_3 could be due to high alkali activator/ash which produces more porous structure. Large pores with some microcracks and inhomogeneity of microstructure [3], associate with the reduction of lability of the geopolymeric gel synthesis due to high Si/Al further reduces the dissolution rate of the aluminosilicate material and the structural reorganization and gel densification. When SiO_2/Al_2O_3 is low, sample appears to be loosely packed and large number of interconnected pores and voids between particles due to the unreacted material reduces the compressive strength [20].

The effect of CaO/SiO_2 on the compressive strength as represented in **Figs. 5e-5f.** It shows that the compressive strength decreases when CaO/SiO_2 is decreased regardless of the ratios of alkali activator/ash or $Na_2SiO_3/NaOH(KOH)$. The formation of C-(N)-A-S-H gel due to small amount of CaO content in Class F type fly ash may contribute to some strength built up of geopolymer [47]. As shown in **Figs. 5c-5d** and **Figs. 5e-5f**, SiO_2/Al_2O_3 and CaO/SiO_2 are constant. It is because the used of NaOH or KOH in the mixture does not contribute to SiO_2/Al_2O_3 and CaO/SiO_2.

**Figs. 5g-5h** show similar pattern of results as **Figs. 5i-5j**. When $Na_2SiO_3/NaOH(KOH)$ increases and constant alkali activator/ash ratio is only considered, the compressive strength of NaOH-based geopolymer increases with the increment of SiO_2/Na_2O and Al_2O_3/Na_2O (i.e. proportional relationship). Criado, Fernández-Jiménez [7] showed that higher degree of reaction product (i.e. sodium aluminosilicate gel) is achieved when the SiO_2/Na_2O ratio is higher. More soluble silica due to higher ratio of SiO_2/Na_2O will enhance the geopolymerisation thus increasing the compressive strength. However, the compressive strength of KOH-based geopolymer increases with the reduction of SiO_2/Na_2O and Al_2O_3/Na_2O (i.e. inversely proportional relationship). The lower reactivity and slower rate of reacting material dissolution may be the reason of causing the decline of compressive strength with respect to the increment of SiO_2/Na_2O and Al_2O_3/Na_2O ratios [7].

Higher Al_2O_3/Na_2O in KOH-based geopolymer than in NaOH-based geopolymer could be attributed to the reduction of Na_2O in KOH-based geopolymer. Despite Na_2O content in fly ash, Na_2O in NaOH-based geopolymer is provided by both NaOH and Na_2SiO_3; however, Na_2O in KOH-based geopolymer is solely provided by Na_2SiO_3. Having the constant alkali activator/ash, it is supposed that the reduction of Na_2O to the increment of $Na_2SiO_3/NaOH$ increases the compressive strength. Inversely, the increment of Na_2O when Na_2SiO_3/KOH is increased lead to higher compressive strength.

SiO_2/K_2O in both types of geopolymer increases when $Na_2SiO_3/NaOH(KOH)$ is increased as shown in **Figs. 5k-5l**. However, it increases on NaOH-based geopolymer increases but it decreases on KOH-based geopolymer when the alkali activator/ash is increased.

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For Al_2O_3/K_2O, no effect has been shown in NaOH-based geopolymer as shown in **Fig. 5m**. It is because that Al_2O_3 is provided by fly ash, it is constant throughout the mixture ratios. Moreover, NaOH does not contribute to K_2O. The Al_2O_3/K_2O increases when K_2O is reduced. It seems that the compressive strength of KOH-based geopolymer increases as K_2O reduces as shown in **Fig. 5n** shows.

Both **Fig. 5l and Fig. 5n** show similar pattern of results as **Fig. 5g and Fig. 5i** respectively, although different types of alkali hydroxide are used. As mentioned earlier on, the variation of SiO_2 in these two types of geopolymer at same ratio of $Na_2SiO_3/NaOH(KOH)$ is constant. The compressive strength of geopolymer is therefore supposed to be governed by Na_2O and K_2O in the mixture. At the highest compressive strength, Na_2O and K_2O of NaOH-based geopolymer is 0.68 and 0.17 whereas it is 0.27 and 0.61 in KOH-based geopolymer. It is supposed that the strength development on NaOH-based geopolymer is mainly attributed to Na_2O (major alkali oxide) rather than K_2O (secondary alkali oxide) in the mixture. Contrariwise, the strength development on KOH-based geopolymer is mainly governed by K_2O (major alkali oxide) rather than Na_2O (secondary alkali oxide) in the mixture. Furthermore, the reduction of major alkali oxide to the increment of $Na_2SiO_3/NaOH(KOH)$ increases the compressive strength.
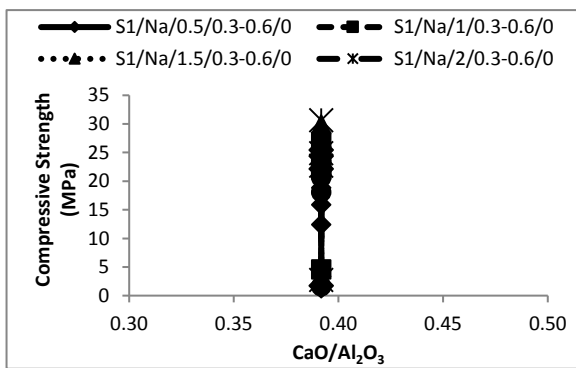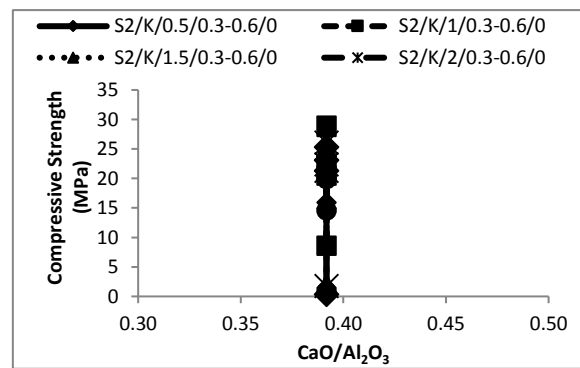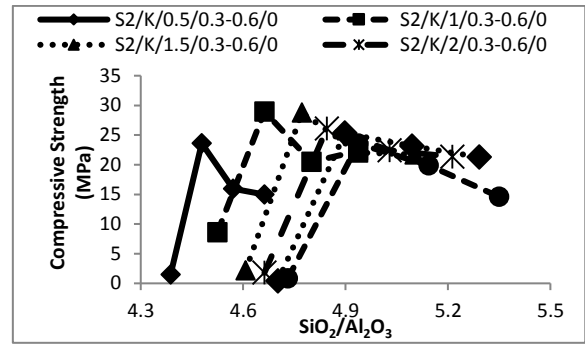

(a) Series 1: CaO/Al_2O_3 (b) Series 2: CaO/Al_2O_3
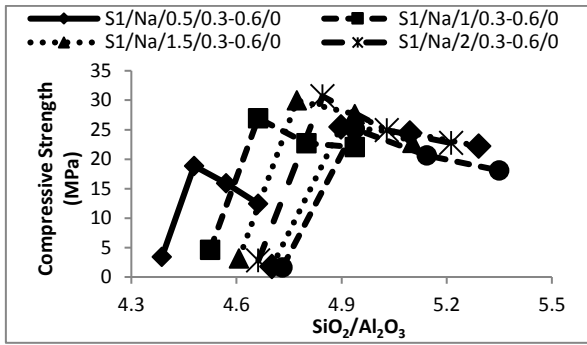
(c) Series 1: SiO₂/Al₂O₃   (d) Series 2: SiO₂/Al₂O₃
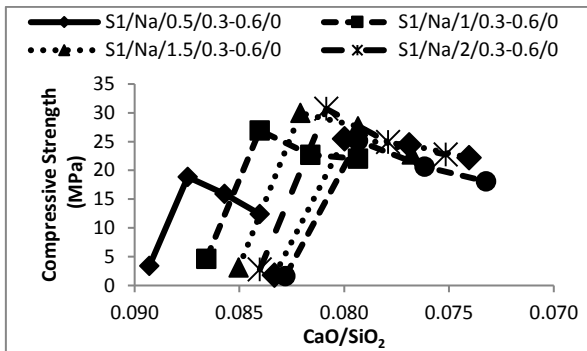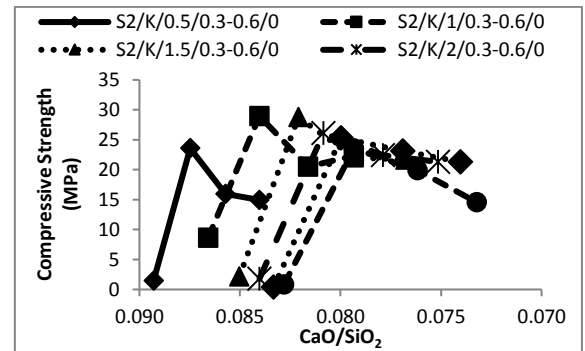
(e) Series 1: CaO/SiO₂   (f) Series 2: CaO/SiO₂
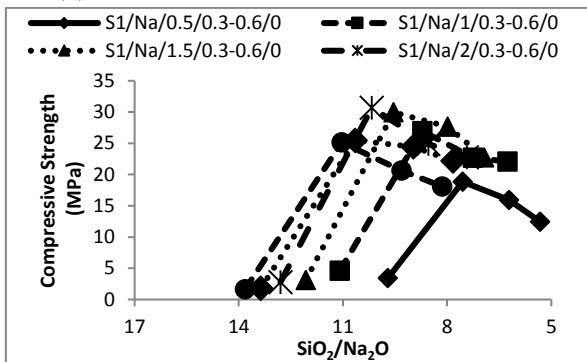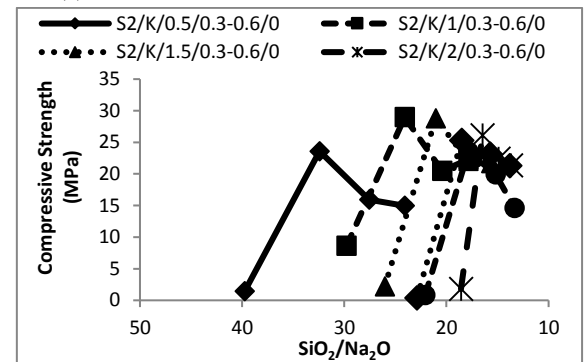
(g) Series 1: SiO₂/Na₂O   (h) Series 2: SiO₂/Na₂O
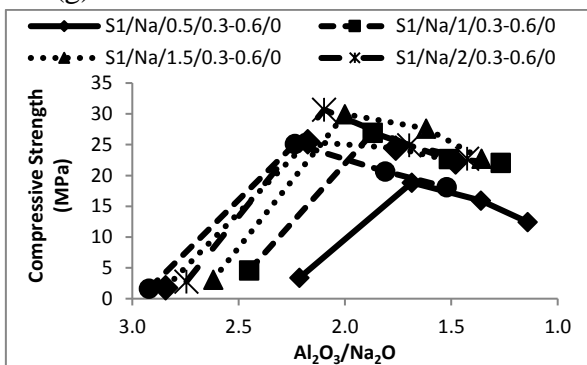
(i) Series 1: Al₂O₃/Na₂O   (j) Series 2: Al₂O₃/Na₂O

(k) Series 1: SiO$_2$/K$_2$O   (l) Series 2: SiO$_2$/K$_2$O
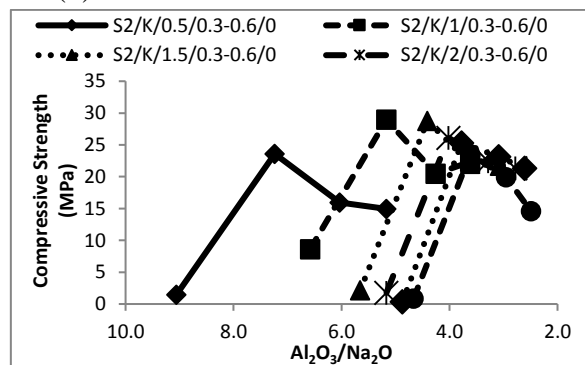
(m) Series 1: Al$_2$O$_3$/K$_2$O   (n) Series 2: Al$_2$O$_3$/K$_2$O

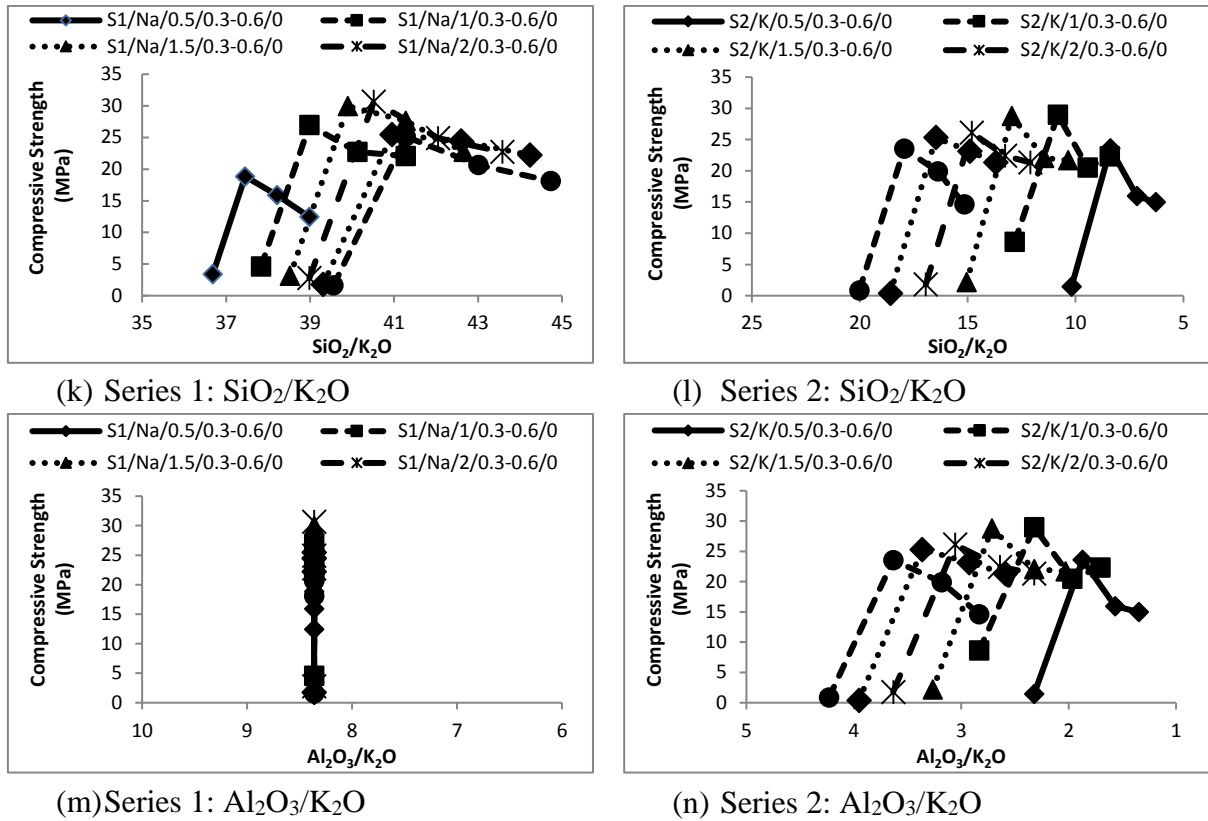**Fig 5.** Effect of oxide molar ratios on the compressive strength of geopolymer using industrial Na$_2$SiO$_3$ (left column) Series 1: NaOH and (right column) Series 2: KOH

3.2.5. Industrial grade Na$_2$SiO$_3$ vs. Commercial grade Na$_2$SiO$_3$

Geopolymer using industrial grade Na$_2$SiO$_3$ at alkali activator/ash of 0.4, which exhibited the highest compressive strength, was then replaced with the commercial grade Na$_2$SiO$_3$. The results of the geopolymer using commercial grade Na$_2$SiO$_3$ at various Na$_2$SiO$_3$/NaOH(KOH) were studied in comparison to the geopolymer using industrial grade Na$_2$SiO$_3$. **Fig. 4** shows that geopolymer using commercial grade Na$_2$SiO$_3$ obtains significantly higher compressive strength than the samples using industrial grade Na$_2$SiO$_3$. It is noted that the highest compressive strength of both NaOH- and KOH-based geopolymers using commercial grade Na$_2$SiO$_3$ were obtained at Na$_2$SiO$_3$/NaOH(KOH)=2.5. This result is consistent with the finding as reported by

Hardjito and Rangan [14]. When commercial grade Na_2SiO_3 is used, the strength increment of NaOH-based geopolymer cured at 60°C after 1 day, 7 days and room temperature after 7 days is approximately 54%, 53% and 58% respectively. In similar curing conditions, the strength increment of KOH-based geopolymer is 52%, 52% and 57% respectively. The significant strength increment may be attributed to the different percentages of SiO_2 and Na_2O in Na_2SiO_3, in which the commercial grade Na_2SiO_3 consists of higher percentage of SiO_2 and Na_2O than the industrial grade.
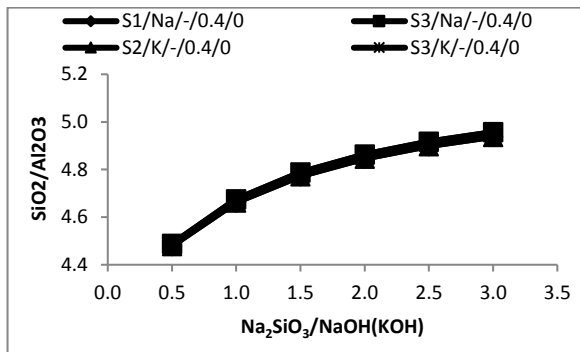
From the oxide molar ratio points of view, the effect of commercial grade Na_2SiO_3 on NaOH- and KOH-based geopolymers in comparison to industrial grade Na_2SiO_3 is illustrated in **Fig. 6**. The effect of commercial grade Na_2SiO_3 on SiO_2/Al_2O_3, CaO/SiO_2, SiO_2/K_2O and Al_2O_3/K_2O of both NaOH- and KOH-based geopolymer is insignificant as shown in **Figs. 6a-d**. For SiO_2/Na_2O and Al_2O_3/Na_2O, the results of geopolymer using commercial grade Na_2SiO_3 shows similar pattern to geopolymer of industrial grade Na_2SiO_3. However, these two oxide molar ratios decrease when the commercial grade Na_2SiO_3 is used as illustrated in **Figs. 6e-f**. This is well reflected that although the tendency of SiO_2/Na_2O and Al_2O_3/Na_2O in both NaOH- and KOH-based geopolymer are different, the increment of SiO_2/Na_2O and Al_2O_3/Na_2O at constant $Na_2SiO_3/NaOH(KOH)$ reduces the strength capability.

It is also noted that the main alkali oxide in KOH-based geopolymer using commercial grade Na_2SiO_3 is Na_2O (0.48) instead of K_2O (0.42). It is supposed that when Na_2O content is low, the strength development of KOH-based geopolymer is mainly governed by higher K_2O content in the sample. However, the strength development in this case is mainly attributed to Na_2O when K_2O in the sample is lower than Na_2O. As mentioned earlier on, the solubility of KOH-based geopolymer decreases
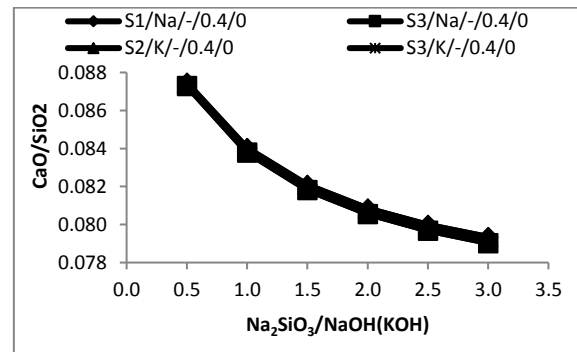
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when the silicate ratio is increased. This may explain why KOH-based geopolymer obtains the highest compressive strength at lower Na_2SiO_3/KOH when industrial grade Na_2SiO_3 is used.

The workability of geopolymer using commercial grade Na_2SiO_3 at the highest compressive strength is 110mm for both NaOH- and KOH-based geopolymer. Whereas, it is 125mm and 135mm on NaOH- and KOH-based geopolymer using industrial grade Na_2SiO_3. Although the reduction of workability is exhibited, it does not lower the compressive strength. It shows that the increment of oxide molar ratios in particular the SiO_2 and Na_2O content is most likely lead to higher compressive strength.

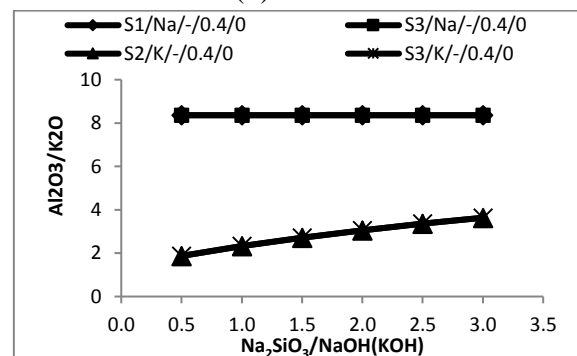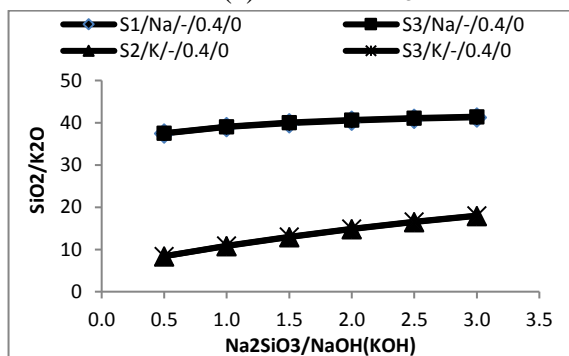From the cost effective point of view, the industrial grade of Na_2SiO_3 is much cheaper than the commercial grade. Despite having better compressive strength on geopolymer using commercial grade of Na_2SiO_3, geopolymer using industrial grade of Na_2SiO_3 is suitable for the application which requires lower compressive strength and higher workability.

(a) SiO_2/Al_2O_3

(b) CaO/SiO_2

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(c) SiO$_2$/K$_2$O   (d) Al$_2$O$_3$/K$_2$O

Chart (c):
Legend: S1/Na/-/0.4/0   S3/Na/-/0.4/0   S2/K/-/0.4/0   S3/K/-/0.4/0
Y-axis: SiO2/Na2O (0, 5, 10, 15, 20, 25, 30, 35)
X-axis: Na$_2$SiO$_3$/NaOH(KOH) (0.0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5)

Chart (d):
Legend: S1/Na/-/0.4/0   S3/Na/-/0.4/0   S2/K/-/0.4/0   S3/K/-/0.4/0
Y-axis: Al2O3/Na2O (0, 2, 4, 6, 8)
X-axis: Na$_2$SiO$_3$/NaOH(KOH) (0.0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5)

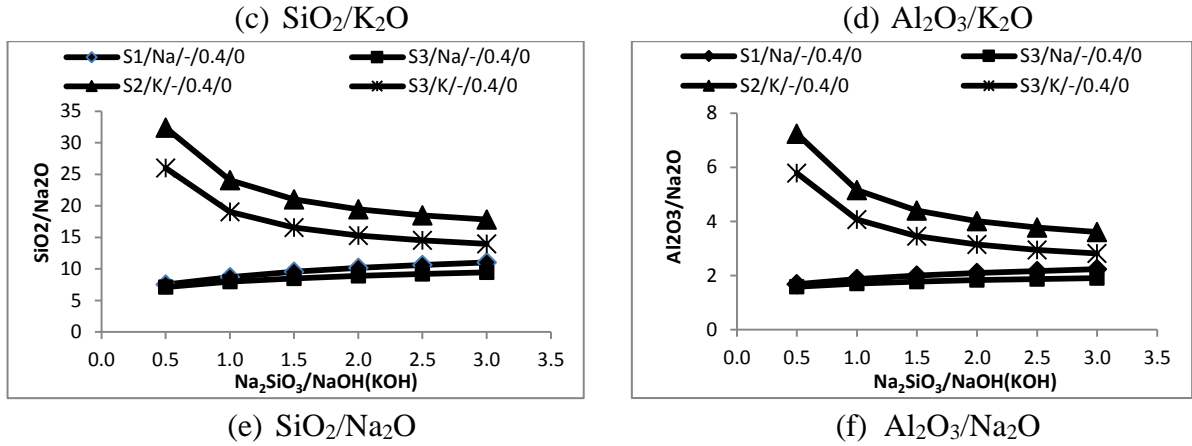(e) SiO$_2$/Na$_2$O   (f) Al$_2$O$_3$/Na$_2$O

Fig 6. Variation of oxide molar ratios on NaOH- and KOH-based geopolymer when commercial grade Na$_2$SiO$_3$ is used in comparison to industrial grade Na$_2$SiO$_3$

3.2.6. Ca(OH)$_2$-based geopolymer

The compressive strength of geopolymer using Ca(OH)$_2$ and commercial grade of Na$_2$SiO$_3$ as the alkali activator is shown in **Fig. 7**. The results show that the compressive strength of the sample after 1 day curing initially enhances with the alkali activator/ash increment but it decreases when the alkali activator/ash becomes greater than 0.4. For the samples cured for 7 days, the compressive strength gradually increases with the increment of alkali activator/ash. When the samples are exposed to the atmosphere, Ca(OH)$_2$ may react with CO$_2$. The precipitator of this atmospheric carbonation, CaCO$_3$, may precipitate in the pores of the sample thus reducing the total porosity and leading to higher compressive strength over the curing age.[48]

The effect of different ratios of Na$_2$SiO$_3$/Ca(OH)$_2$ on the compressive strength is shown in **Fig. 7b**. When Na$_2$SiO$_3$/Ca(OH)$_2$=0, the compressive strength of the sample is only 2 MPa. It can be explained by the poor self-cementing properties of Class F type fly ash due to lower CaO content as compared with Class C type fly ash [49]. As none alkali activator is added at this mixture ratio, the hardening of fly ash and strength

development are mainly attributed to the reaction between fly ash and water. Poorer self-cementing properties, therefore reduces the strength capability.

When $Na_2SiO_3/Ca(OH)_2$ is greater than 0.5, the compressive strength of the samples decreases with the increment of $Na_2SiO_3/Ca(OH)_2$. The highest compressive strength (i.e., 10 MPa) was obtained when $Na_2SiO_3/Ca(OH)_2$=0.5. Fan, Yin [10] state that little amount of Na_2SiO_3 with $Ca(OH)_2$ can accelerate the activation of fly ash. As the samples do not consist of alkali hydroxide, the activation and dissolution of aluminosilicate materials are thought to be minimal. In this case, the presence of calcium cation may compensate the negative charge of Al^{3+} in IV fold coordination or more importantly, is precipitated as C-S-H gel [50]. Therefore, the main reaction product is suggested to be C-S-H gel rather than the geopolymeric gel (i.e., alkali aluminosilicate gel). This explains the reduction of compressive strength by further increment of $Na_2SiO_3/Ca(OH)_2$. Although the soluble silicate is increased in the mixture, deficiency of calcium cation reduces the formation of C-S-H gel. The results show in this research reveal that higher ratio of $Na_2SiO_3/Ca(OH)_2$ would decrease the compressive strength.

When water/binder is increased, the compressive strength of the samples linearly decreases. It shows that the optimal water/binder for the highest compressive strength is 0.4. The reduction of compressive strength with the increment of water/binder may be attributed to the solubility of $Ca(OH)_2$ in water and the evaporation of water when curing in oven. It is known that the solubility of $Ca(OH)_2$ in water is very low.[51] When $Ca(OH)_2$ is added into the water, the solution turns into milky-like and the precipitation of $Ca(OH)_2$ is suspended in the solution. The OH^- ion of the $Ca(OH)_2$ is therefore not available for the dissolution of fly ash. Unlike $Ca(OH)_2$ being a weak base,

NaOH and KOH are categorised as strong base with high solubility. The later types can dissolve more silica and alumina from the fly ash. Additionally, alkali metal (e.g. Na and K) is more reactive than alkaline earth metal (e.g. Ca) [51]. Therefore, it explains why $Ca(OH)_2$-based geopolymer exhibited the lowest compressive strength as compared with NaOH-and KOH-based geopolymers. Moreover, the excessive water content in the sample may evaporate when it is subjected to oven dry. The pores leave behind may weaken the matrix formed, consequently reducing the compressive strength.

The oxide molar ratio of $Ca(OH)_2$-based geopolymer is tabulated in comparison to NaOH- and KOH-based geopolymer as **Table 3** shows. The ratios of CaO/Al_2O_3, SiO_2/Al_2O_3 and CaO/SiO_2 are similar on these three types of geopolymer. The variation of SiO_2/Na_2O, SiO_2/K_2O, Al_2O_3/Na_2O and Al_2O_3/K_2O is significant. $Ca(OH)_2$-based geopolymer exhibits the highest ratios in these oxide molar ratios than the other two types of geopolymer. It is most likely due to the lowest Na_2O and K_2O present in $Ca(OH)_2$-based geopolymer in comparison to the latter types. $Ca(OH)_2$-based geopolymer shows the lowest compressive strength while the SiO_2/Na_2O and Al_2O_3/Na_2O are the highest. This has shown well agreement to the statement as postulated earlier on.

The workability of the $Ca(OH)_2$-based geopolymer is shown in **Table 4**. It decreases with the increment of alkali activator/ash and $Na_2SiO_3/Ca(OH)_2$. It is noted that $Ca(OH)_2$-based geopolymer sets and gets harden very quickly as compared with the NaOH- and KOH-based geopolymers. It is believed that $Ca(OH)_2$ accelerates the setting and hardening properties of geopolymer [10]. This hardening mechanism is most likely due to the formation of $CaCO_3$[46]. When the setting time is shortened, the dissolution of reacting material reduces. More unreacted particles present in the sample thus
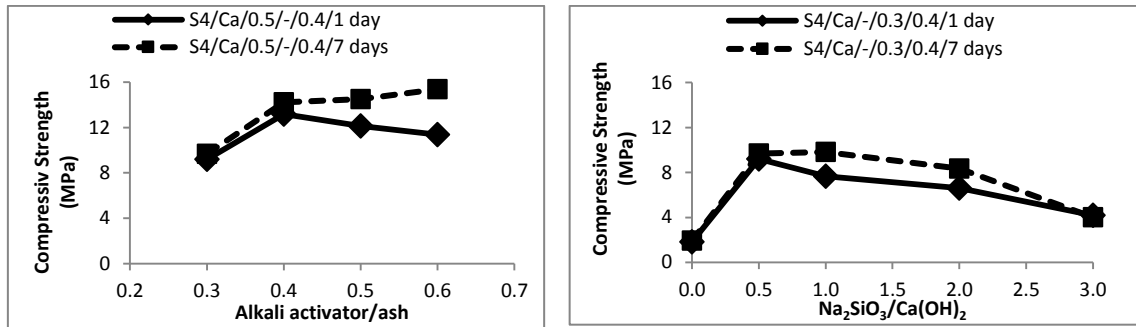
lowering the strength capability [52]. Having these properties, Ca(OH)$_2$-based geopolymer properties may beneficial for the applications that require fast drying duration. Additionally, alkali hydroxide such as NaOH or KOH can be added into the mixture to achieve higher compressive strength. Therefore, geopolymer of high strength and fast drying performance can be achievable.

Table 3. The oxide molar ratios of Ca(OH)$_2$-based geopolymer in comparison to NaOH- and KOH-based geopolymer

Type	Sample designation	CaO/ Al2O3	SiO2/ Al2O3	CaO/ SiO2	SiO2/ Na2O	SiO2/ K2O	Al2O3/ Na2O	Al2O3/ K2O
CaOH-based	S4/Ca/0.5/0.4/0	0.392	4.347	0.090	36.803	36.346	8.466	8.361
NaOH-based	S3/Na/0.5/0.4/0	0.392	4.488	0.087	7.148	37.524	1.593	8.361
KOH-based	S3/K/0.5/0.4/0	0.392	4.488	0.087	25.990	8.396	5.791	1.871

Table 4. The workability of the samples at different ratios

No.	Na$_2$SiO$_3$/ Ca(OH)$_2$ ratio	Alkali activator/ ash ratio	Water/ binder ratio	Workability (mm)
a	0.5	0.3	0.4	150
	0.5	0.4	0.4	129
	0.5	0.5	0.4	125
	0.5	0.6	0.4	100
b	0	0	0.4	200
	0.5	0.3	0.4	150
	1	0.3	0.4	100
	2	0.3	0.4	100
	3	0.3	0.4	100
c	0.5	0.3	0.4	150
	0.5	0.3	0.5	180
	0.5	0.3	0.6	200
	0.5	0.3	0.7	245

(a) Alkali activator/ash

[Na_2SiO_3/$Ca(OH)_2$ =0.5,

water/binder=0.4]

(b) Na_2SiO_3/$Ca(OH)_2$

[Alkali activator/ash=0.3,

water/binder=0.4]
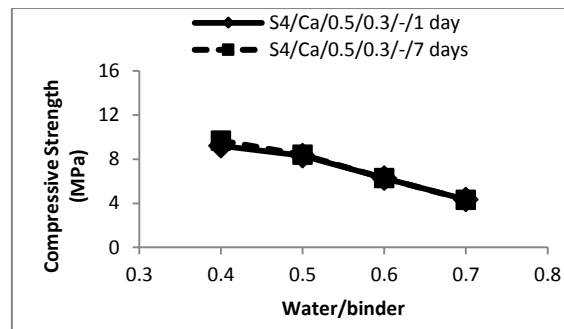
(c) Water/binder [Na_2SiO_3/$Ca(OH)_2$ =0.5, Alkali activator/ash=0.3]

Fig 7. Compressive strength of geopolymer using $Ca(OH)_2$ at different ratios of (a) alkali activator/ash (b) Na_2SiO_3/ $Ca(OH)_2$ and (c) water/binder

4. Conclusion

A comprehensive overview on compressive strength of geopolymer using Sarawak fly ash with different alkali activator/ash and Na_2SiO_3/NaOH (or KOH or $Ca(OH)_2$) is studied in this research. Oxide molar ratios due to different ratios of alkali activator and mixture have been investigated. The results show that geopolymer made using Sarawak fly ash can possibly be used as construction materials. The conclusions are summarised as follows:

1. The workability is in the increasing manner when the alkali activator/ash is increased.

   Inversely, it is in the decreasing manner when Na_2SiO_3/NaOH and Na_2SiO_3/KOH are

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increased. The commercial grade Na_2SiO_3 shows significant effect on the workability of both NaOH- and KOH-based geopolymers.

2. High ratio of alkali activator/ash and Na_2SiO_3/NaOH(KOH) does not necessarily lead to high compressive strength. The optimal compressive strength was obtained when alkali activator/ash ratio was 0.4 regardless of the type of alkali activators (i.e., NaOH or KOH) and curing condition (i.e., 60°C or room temperature and 1 day or 7 days). With alkali activator/ash of 0.4, Na_2SiO_3/NaOH=2 and Na_2SiO_3/KOH=1 exhibited the highest compressive strength for geopolymer using Sarawak fly ash.

3. When Na_2SiO_3/NaOH=0.5 and 1, the compressive strength of KOH-based geopolymer was higher than NaOH-based but lower than NaOH-based when Na_2SiO_3/NaOH>1.5. NaOH-based geopolymer exhibited better compressive strength than KOH-based geopolymer when curing at room temperature.

4. The effect of oxide molar ratios on the compressive strength was studied. Higher ratios do not lead to higher compressive strength. The effect of CaO/Al_2O_3, SiO_2/Al_2O_3, CaO/SiO_2 on NaOH- and KOH-based geopolymer is similar. However, the tendency of SiO_2/Na_2O, Al_2O_3/Na_2O, SiO_2/K_2O and Al_2O_3/K_2O to NaOH- and KOH-based geopolymer shows dissimilarity.

5. The compressive strength of geopolymer using commercial grade Na_2SiO_3 was significantly higher than those using industrial grade Na_2SiO_3 regardless of the type of alkali hydroxide. The reduction of SiO_2/Na_2O and Al_2O_3/Na_2O in geopolymer using commercial grade Na_2SiO_3 in comparison to industrial grade Na_2SiO_3 increases the compressive strength. Higher ratio of SiO_2 and Na_2O in commercial grade Na_2SiO_3 is most likely attributed to the enhancement of geopolymerisation.

6. For geopolymer using industrial grade Na_2SiO_3, the major alkali oxide in the sample is Na_2O rather than K_2O in NaOH-based geopolymer. Inversely, it is K_2O (major alkali oxide) rather than Na_2O in KOH-based geopolymer. Both NaOH- and KOH-based geopolymer show Na_2O as major alkali oxide when commercial grade Na_2SiO_3 is used.

7. $Ca(OH)_2$-based geopolymer demonstrated the lowest compressive strength than NaOH- and KOH-based geopolymers. The lowest solubility of $Ca(OH)_2$ in comparison to the latter types reduces the dissolubility of silica and alumina from the fly ash. $Ca(OH)_2$-based geopolymer appears to set and get harden faster than NaOH- and KOH-based geopolymer.

8. Sarawak fly ash-based geopolymer can be a potential construction material for industrial application depending on the requirements of the compressive strength and workability. Geopolymer using industrial grade Na_2SiO_3 is beneficial for the application which requires low cost, low compressive strength but high workability. For geopolymer using commercial grade Na_2SiO_3, it is suitable for the application that requires high compressive strength. $Ca(OH)_2$ can be added into the geopolymer mixture to reduce the drying duration.

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