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Effect of Silica Fume on the Volume Expansion of Metakaolin-Based Geopolymer Considering the Silicon-to-Aluminum Molar Ratio

Sungwoo Park, Juan Yu, Jae Eun Oh* and Sukhoon Pyo*

Abstract

This paper investigates the effect of silica fume on the mechanical properties of metakaolin-based geopolymers with different silicon-to-aluminum molar (S/A) ratios. Geopolymer has been extensively studied as an alternative to traditional cementitious material because of its low CO₂ emissions. Previous studies revealed that the application of silica fume can improve the compressive strength of geopolymer, however, the optimum dosages are different. To examine the reason for the different optimum dosages of silica fume, this study prepares geopolymer specimens of which variables are the S/A ratio and silica fume dosage, and conducts compressive strength and initial setting time tests. To examine whether the strength degradation is caused by the expansion due to the added silica fume in geopolymer, the volume and dynamic modulus are also measured. The results show that a part of silica fume dissolves and changes the S/A ratio of geopolymer, and that a part of silica fume remains in the geopolymer matrix. These combined effects of silica fume result in an irregular compressive strength trend, and, thus, an optimum dosage of silica fume can vary depending on the S/A ratio. Furthermore, the volume expansion of geopolymer with silica fume is observed, however, no sign of damage on the compressive strength is found.

Keywords: volume expansion, metakaolin, geopolymer, silica fume, compressive strength

1 Introduction

As an alternative building material to ordinary Portland cement, geopolymer has been extensively studied because of the reduced amount of CO₂ emission during its production process (Oyebisi et al., 2020; Luukkonen et al., 2018; Tempest et al., 2015; Ashish and Verma, 2019), excellent chemical resistance (Oyebisi et al., 2020; Chindapasirt et al., 2014; Tempest et al., 2015), and thermal resistance (Sabbatini et al., 2017; Natali Murri et al., 2017; Provis et al., 2009). These substantial studies have

developed mix designs of geopolymer resulting in higher mechanical properties.

The compressive strength of geopolymer can be improved using a sodium silicate solution. The silicon-to-alumina molar (S/A) ratio of 1.5–1.9 in geopolymer mix design exhibits the highest compressive strength (Duxson et al., 2005; Silva et al., 2007; Latella et al., 2008; Nmiri et al., 2019; Provis et al., 2009). Given that the inherent S/A ratios of precursors are typically less than 1.5 (e.g., metakaolin), adding extra silicon supply into geopolymer is essential. Sodium silicate solution as a source of silicon enhances polymerization by promoting the dissolution of silicon and aluminum of the raw aluminosilicate (Damilola, 2013), and by making the geopolymer structure denser, resulting in higher compressive strength (Provis et al., 2009; Rowles and O'Connor,

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2003). Consequently, the use of sodium silicate solution has beneficial effects on the compressive strength of geopolymer.

The addition of silica can also improve the mechanical properties of geopolymer. Silica fume has been used as a silica source; it enhances the compressive strength and abrasion resistance of geopolymer by reducing voids and, thus, increasing the compactness (Khater, 2013; Chindaprasirt et al., 2014; Nuruddin et al., 2010; Dutta et al., 2010; Okoye et al., 2016; Rostami and Behfarnia, 2017; Jithendra and Elavenil, 2020; Duan et al., 2017; Uysal et al., 2018). However, the excessive amount of silica fume decreases the strength, and the optimum replacement of precursor with silica fume is debatable among studies. The highest compressive strengths were achieved with the different replacement ratios of 3.75% (Chindaprasirt et al., 2014), 7% (Nuruddin et al., 2010; Khater, 2013), or 10% (Sukontasukkul et al., 2020) while other studies reported that silica fume degrades the compressive strength of geopolymer paste specimens but improves that of geopolymer mortar specimens (Dutta et al., 2010; Thokchom et al., 2011). However, what factors decide the optimum level of silica fume remain unclear.

The hypothesis in this study is that the optimum dosage of silica fume can depend on the S/A ratio of geopolymer. Previous studies have reported that the compressive strength degrades with an excessive amount of silica fume because of its volume expansion (Nuruddin et al., 2010; Sukontasukkul et al., 2020). If this conclusion holds true, the optimum dosage of silica fume can vary with different S/A ratios, because the S/A ratio affects the density of geopolymer matrix. To demonstrate this hypothesis, the geopolymers of multiple S/A ratios from 1.0 to 2.0 were prepared with various dosages of silica fume up to 15%. Metakaolin was adopted as a precursor because its reactivity with an alkaline activator is adequate for a fundamental study (Silva et al., 2007). The compressive strength and initial setting time were measured, and the volume change was observed using Archimedes' principle to investigate the expansion of silica fume. The dynamic modulus test was also conducted to examine damages in geopolymer caused by silica fume. The results might give important insights on how to incorporate silica fume in metakaolin-based geopolymer.

2 Experimental Details

2.1 Raw Materials and Specimen Preparation

A commercial product of metakaolin was used as an aluminosilicate precursor, and sodium hydroxide and silica fume were used to synthesize sodium silicate.

The chemical compositions of metakaolin and silica fume are shown in Table 1. The S/A ratios of geopolymer

Table 1 Chemical compositions of raw materials (wt%).

Constituent	Metakaolin	Silica fume
SiO ₂	50.37	97.30
Al ₂ O ₃	43.33	0.06
CaO	0.45	0.09
Fe ₂ O ₃	2.82	0.19
TiO ₂	2.29	–
MgO	0.15	0.42
SO ₃	0.05	0.9

used in this study were 1.0, 1.4, 1.8, and 2.0, and the molar ratio of sodium to aluminum (N/A) ratio was 1/0.

The S/A and N/A ratios were calculated based on the chemical components of metakaolin, silica fume, and sodium hydroxide. The water to solid (w/s) weight ratios were 1.0 and 0.48 for all specimens. The solid part includes all of the geopolymer ingredients except for a mixing water in this study. The mix designs of geopolymer specimens are summarized in Table 2. The silica fume in Table 2 was used to synthesize sodium silicate solution to adjust the S/A ratio of geopolymer. Silica fume was also added to each geopolymer specimen in powder form and the amount was 0%, 5%, 10%, and 15% to metakaolin by weight. The maximum dosage of 15% was determined based on previous studies that have indicated the optimum dosage of silica fume is achieved in the range of 3.75 to 10%. It is noted that water was added along with silica fume to keep the w/s ratio of geopolymer as 0.48.

The sodium silicate solution was prepared by dissolving silica fume in sodium hydroxide (Rowles and O'Connor, 2003; Billong et al., 2021; Alnahhal et al., 2021). The solution was cured in the oven at 75°C for more than 12 h to extend the dissolution of silica fume, and cooled down to ambient temperature before geopolymer mixing. After mixing the dry materials of metakaolin and silica fume, the sodium silicate solution was added. The specimens were mixed for 5 min using a laboratory planetary mixer, and cast in molds. All of the specimens were cured in a sealed condition at a room temperature until tested.

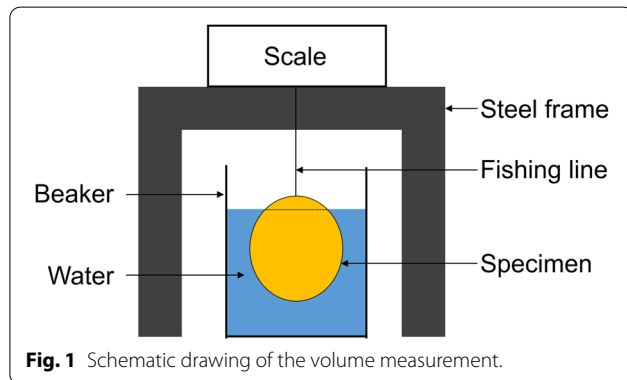
2.2 Initial Setting Time and Compressive Strength

The initial setting time was measured according to the standard of ISO 9597 (ISO 9597:2008 2008). The elapsed time was calculated after adding the alkaline solution to dry mixes of metakaolin and silica fume. The compressive strength test was conducted on the specimens cured for 28 and 90 days following the ASTM C109 (ASTM Standard C109, 2020). For the compressive strength test, three specimens were cast in 50 × 50 × 50mm cubic molds.

Table 2 Mixing proportion of geopolimer specimens.

Specimen	Silica fume addition (%)	Precursor		Sodium silicate solution			
		Metakaolin (wt%)	SF ^a (wt%)	NaOH (wt%)	SF ^b (wt%)	Water (wt%)	Mol ^c
S/A 1.0	0	51	0	17	0	32	13.2
	5	49	2	17	0	32	12.7
	10	47	5	16	0	32	12.3
	15	45	8	15	0	32	11.9
S/A 1.4	0	43	0	15	10	32	11.3
	5	42	2	14	10	32	11.0
	10	41	4	14	9	32	10.7
	15	40	6	13	9	32	10.3
S/A 1.8	0	38	0	13	17	32	10.0
	5	37	2	13	16	32	9.7
	10	36	4	12	16	32	9.4
	15	35	6	12	15	32	9.2
S/A 2.0	0	36	0	13	19	32	9.4
	5	35	2	12	19	32	9.2
	10	34	3	12	19	32	8.9
	15	33	5	12	18	32	8.7

^a Silica fume was added into geopolimer in powder form, ^b Silica fume was used to synthesize sodium silicate solution, ^c The molarity of sodium hydroxide solution.



After the specimens were hardened, they were taken out of the molds, sealed in plastic bags, and cured at an ambient temperature until tested. The average results from the triplicate specimens were used for the compressive strength data.

2.3 Volume Change Measurement

The volume of a specimen was measured using the Archimedes' principle as shown in Fig. 1. Note that the initial casting volume is measured when the material was still fresh, and, thus, the length change measurement method of ASTM C490 was not applicable. Instead, this study exploited the gravity method or Archimedes' method (Li et al., 2019; Bouasker et al., 2008). The specimens were cast in a latex membrane, its neck was tied, and the tip

of the membrane was sealed to minimize water evaporation. The volume measurement was conducted following this procedure: (i) pouring the water of 700g into the beaker, (ii) measuring the weight of the surface dried specimen (w_1), and (iii) measuring the weight in water of the specimen (w_2). The beaker was filled with water up to 700 g after each measurement and around 1 cm below the top of the specimen was submerged under water. The temperature of the room was around 22 – 25°C. The water was kept in the room for 24 h before usage to ensure that it had the same temperature of the room. The volume of the specimens were obtained by subtracting w_1 from w_2 . The initial weights and the initial volume of specimens including latex membrane were 145–150 g and 85 – 90 cm³, respectively. The volume change was calculated by subtracting the initial casting volume from the volume measured regularly. The average value of three replicates was used. It was noted that the specimen lost weight with time, which was assumed to be water evaporation through the sealed membrane. The total weight losses were around 6–7% of initial weights after 90 days.

2.4 Dynamic Test

To examine the specimen damage caused by the expansion of silica fume, the dynamic test was conducted following the standard of ASTM C215 (ASTM Standard C215, 2020).

The dynamic modulus of elasticity has been used to detect damage by macro-cracks inside concrete specimen caused by freeze-thaw or alkali-silica expansion (Al Rikabi et al., 2018; Cho, 2007; Sargolzhai et al., 2010). A longitudinal resonance frequency was measured using a specimen in the shape of a rectangular prism, sized 40×40×160mm. Since the specimens were moistened by the water produced while being cured in air due to the condensation process of the geopolymer reaction (Park and Pour-ghaz, 2018), their surface was dried with a towel before measurement. The accelerometer was installed at the center of one end surface and the steel impactor hit the center of the other end surface. The data sampling rate used was 50 kHz.

2.5 Scanning Electron Microscope (SEM)

A commercial SEM instrument was used to examine the morphology of each geopolymer system. After the compressive test, fractured samples after 90-day curing were collected and stored in a low vacuum desiccator for 7 days to remove free water and then crushed into small pieces until tested. The surface of the pieces was coated with Pt to improve image quality and resolution. The accelerating voltage in this study was 15 kV with a working distance of 5 mm.

3 Results

3.1 Initial Setting Time

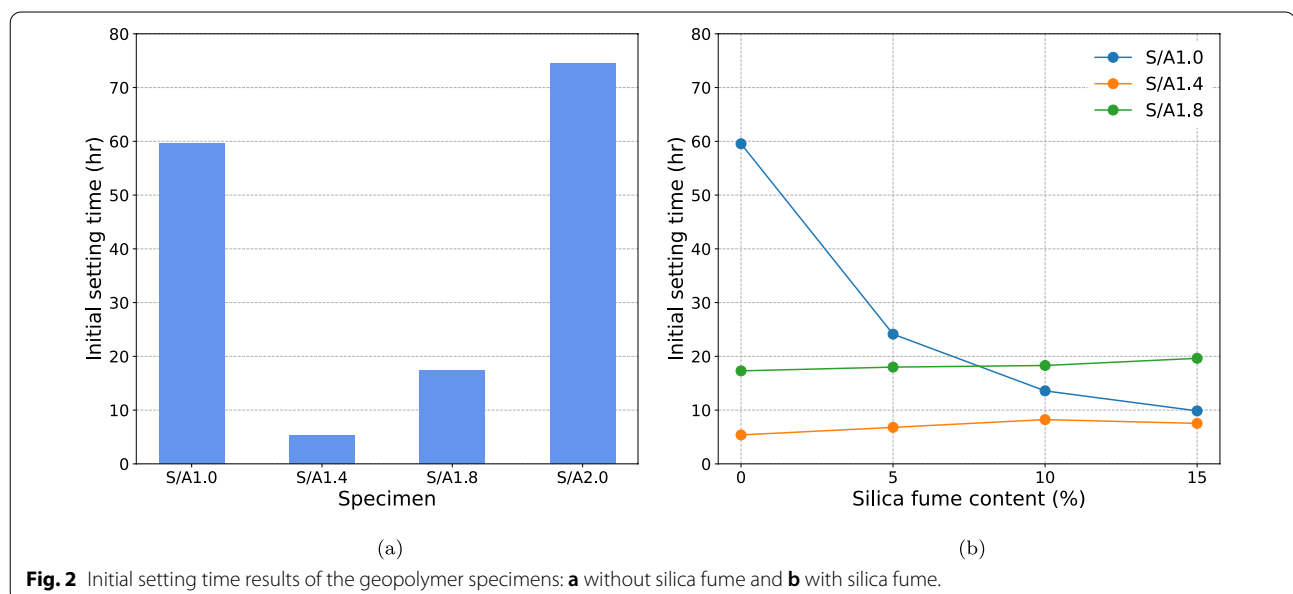
The initial setting time test results are shown in Fig. 2. In the absence of silica fume (Fig. 2), the effect of sodium silicate solution on the initial setting time is significant. The S/A 1.0 specimen synthesized with sodium hydroxide

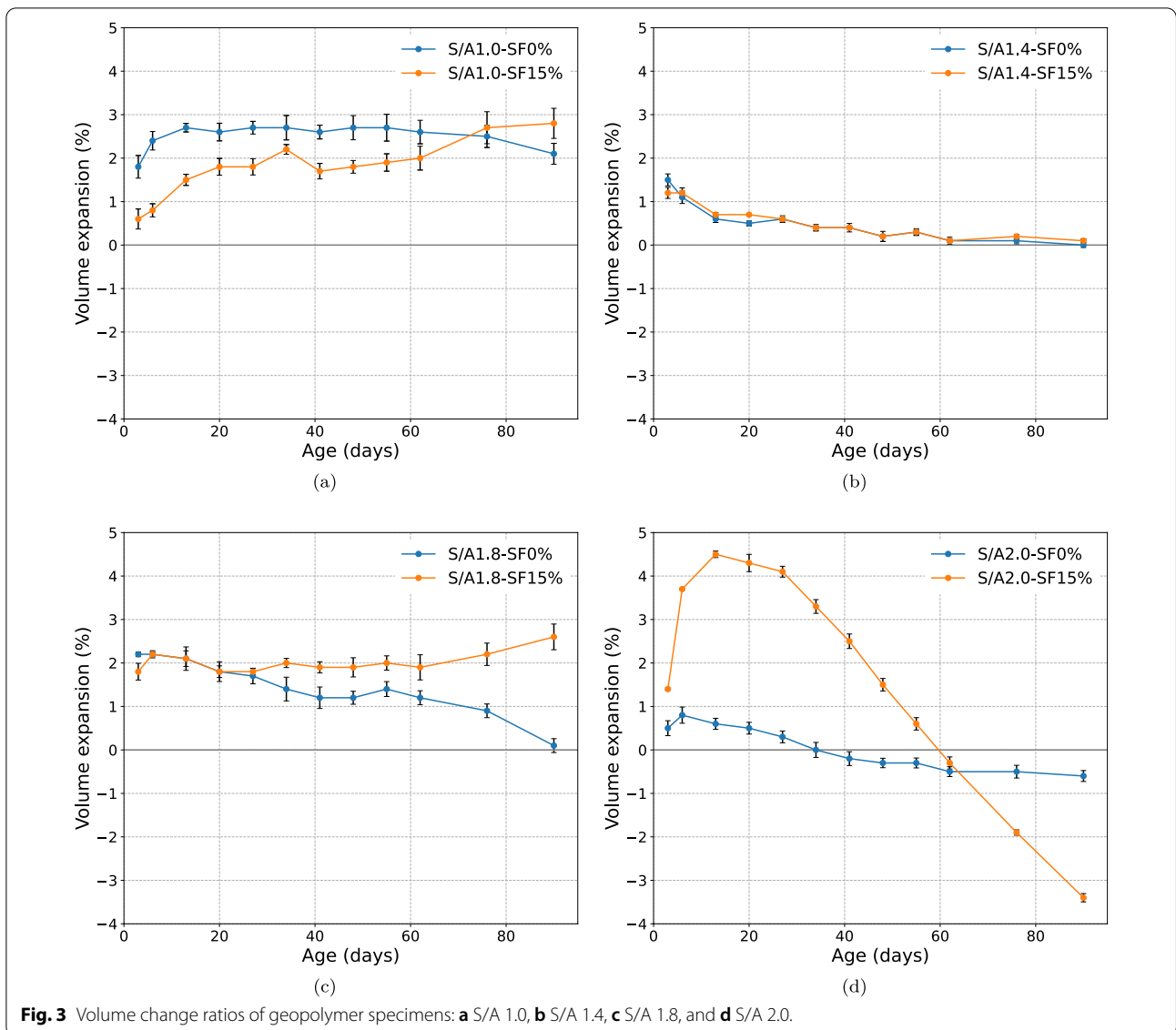
solution exhibits an initial setting time of almost 60 h. When the sodium silicate solution is incorporated, the setting time decreases to less than 30 h. This is because sodium silicate solution likely accelerates the geopolymer reaction by increasing the speed of dissolution of metakaolin as reported (Wan et al., 2017). However, when the silica concentration in sodium silicate solution increases, the S/A ratio of 1.4 to 1.8, the initial setting time of geopolymer increases, which is in accordance with previous studies (Suraneni et al., 2014; Silva et al., 2007).

The effect of silica fume on the initial setting time is shown in Fig. 2. Silica fume decreases the initial setting time of the S/A 1.0 specimen, whereas it increases those of the S/A 1.4 and 1.8 specimens. Note that the initial setting time of the S/A 2.0 specimens with silica fume is longer than 7 days, and, thus, the data are excluded. The effect of silica fume on the initial setting time of geopolymer can be attributed to a partial dissolution of silica fume increasing the S/A ratio (Wan et al., 2017).

3.2 Volume Change Measurement

The volume changes of geopolymer specimens with and without silica fume at different ages are shown in Fig. 3. The volume change ratio is calculated based on the initial casting volume. All of the specimens exhibit an initial expansion, resulting in positive data points. A previous study also observed the expansion of geopolymer, which can be related to the formation of Al phases (Li et al., 2019). Geopolymer begins to shrink after a certain time of expansion; however, the volume change ratio remains positive up to 90 days, with the exception of the S/A 2.0 specimen.





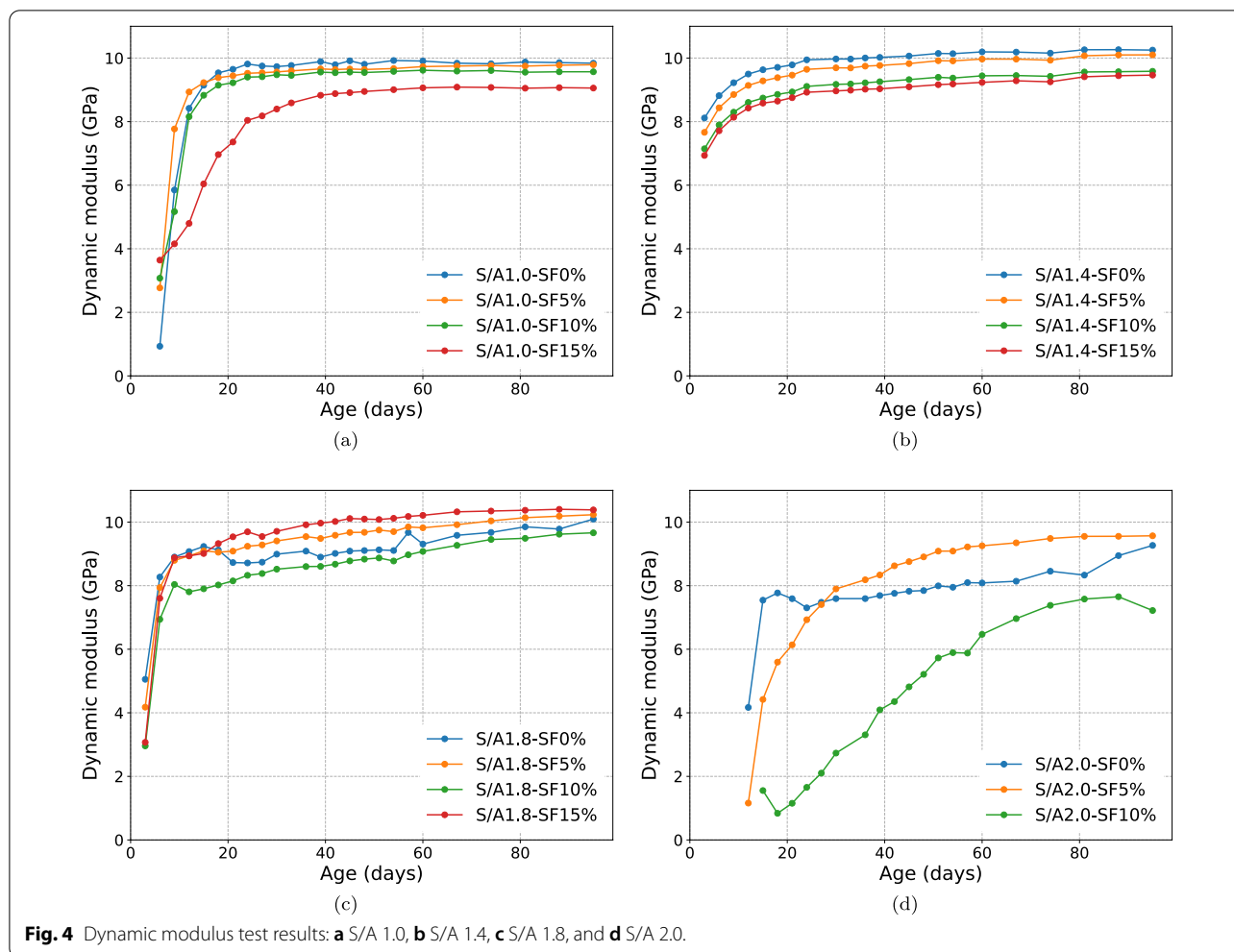
The degree of the initial volume expansion within 20 days might be related to the strength development of geopolymer. When geopolymer sets faster, it exhibits a lower degree of volume expansion. For example, the maximum volume expansion ratio of the S/A 1.4 specimen is the lowest, and its initial setting time is the fastest. The maximum volume expansion ratio of the S/A 1.0 specimens is decreased when silica fume is used, and the added silica fume decreases the initial setting time of the S/A 1.0 specimen. Therefore, it can be concluded that an initial volume expansion is reduced after the strength of the material is developed.

The effect of silica fume on the volume expansion of geopolymer differs based on the S/A ratio. The S/A 1.0 and 1.8 specimens with silica fume start to expand after

approximately 60 days, whereas the S/A 1.4 specimen does not show any expansion due to the presence of silica fume. This supports the hypothesis that the effect of silica fume on the physical properties of metakaolin-based geopolymer can differ according to the S/A ratio.

3.3 Dynamic Modulus

The dynamic modulus results sorted by the S/A ratio are shown in Fig. 4. The dynamic moduli increase with time, which indicates that the chemical reaction is still in progress. The strength development of geopolymer progresses more slowly with a higher S/A ratio. The dynamic moduli of the S/A 1.0 specimens are constant after 40 days; however, those of the S/A 1.4 and 1.8 specimens continue to increase, and the slopes of



the S/A 1.8 specimens are steeper than those of the S/A 1.4 specimens. The S/A 2.0 specimens show unstable development of dynamic moduli, which means that their strength development is slow. Note that the S/A 2.0 specimen with 15% silica fume was not set until 20 days; therefore, it was not tested. The dynamic modulus of geopolymer with silica fume increases over time similar to the specimen without silica fume. This implies that silica fume does not induce cracks in geopolymer.

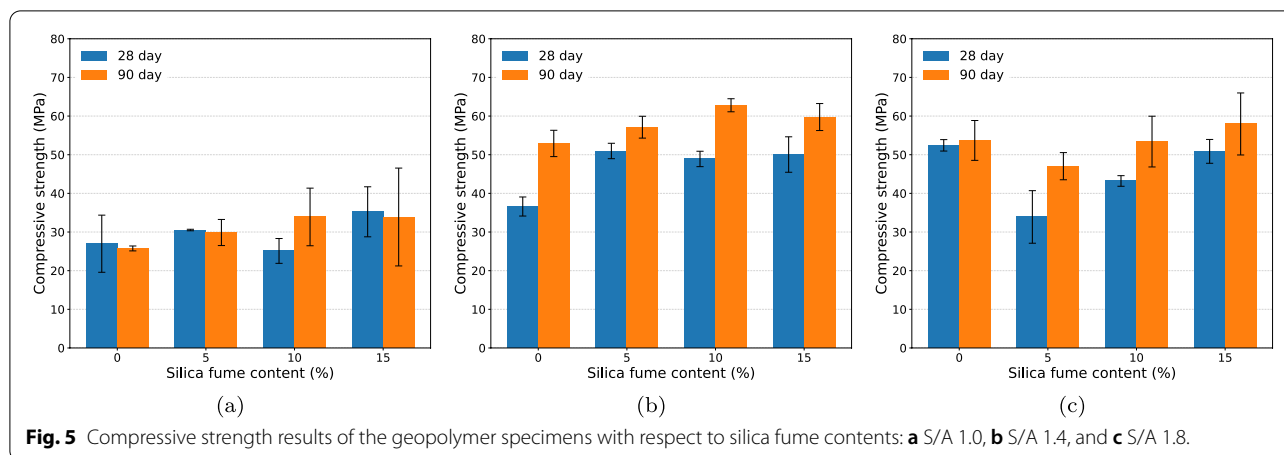
3.4 Compressive Strength

The 28-day and 90-day compressive strength results are shown in Fig. 5. It is noted that the S/A 2.0 specimens were not made in adequate dimensions for strength testing while curing and, therefore, their data were not obtained. Without silica fume, the compressive strength of geopolymer increases with an increase in the S/A ratio. The external silicon addition in alkaline solution makes the geopolymer structure denser and more homogeneous; thus, improving its compressive strength (Duxson

et al., 2005; Chen et al., 2019). The S/A 1.4 specimen shows the beneficial effect of the addition of silica fume on compressive strength; however, the compressive strength decreases with 15% silica fume. This compressive strength degradation caused by a dosage of silica fume over an optimum level has been reported in previous studies (Wu and Sun, 2010; Nmiri et al., 2019; Khater, 2013; Chindapasirt et al., 2014). The S/A 1.0 and 1.8 specimens show complex correlations between the compressive strength and the amount of silica fume, which has also been reported in other studies (Dutta et al., 2010; Nuruddin et al., 2010). Different trends in the compressive strength data between the specimens with different S/A ratios demonstrate that the effect of silica fume on the compressive strength of geopolymer is influenced by the S/A ratio.

3.5 SEM Images

Fig. 6 shows the SEM images of the geopolymer specimens of the S/A 1.0, 1.4, and 1.8 cured for 90 days.



Figs. 6a, c, and e in the left column are images of the specimens without silica fume; and Figs. 6b, d, and f in the right column are images of the specimens with 15% silica fume. The specimen with a higher S/A ratio up to 1.8 has a denser geopolymer structure of sodium aluminosilicate (N-A-S-H) gels (as noted from the top to the bottom of the figures), as reported by previous studies (Duxson et al., 2005; Chen et al., 2019).

Silica fume affects the structure of geopolymers. In the S/A 1.0 specimen, the density of the structure seems to increase with silica fume. This can be observed on a larger scale, as seen in Fig. 7. The S/A 1.0 specimen without silica fume has more granulated phases, possibly zeolite, whereas the S/A 1.0 specimen with silica fume has longer networks in its structure. The trace of silica fume is not detectable; thus, the majority of silica fume seems to be dissolved by the alkaline solution. In the S/A 1.4 and 1.8 specimens, silica fume is discernible, as shown in Fig. 8. However, silica fume mainly exists in the matrix and does not fill the void around unreacted metakaolin particles (Chaipanich et al., 2019). Therefore, it can be expected that the efficiency of silica fume in improving the compressive strength of geopolymer will be lower than that in cement concrete.

4 Discussions

The addition of silica fume into metakaolin-based geopolymer is conducted in two ways. First, it increases the amount of silicon content in a geopolymer mixture. Based on the initial setting time and the SEM, partial dissolution of silica fume was observed. The dissolved silicon changed the structure of the S/A 1.0 specimen. Second, it provides a granular stack of silica fume in geopolymer. The silica fume particle that remained in the geopolymer structure was observed by the SEM. However, silica fume mainly existed in the matrix and

did not fill the voids caused by unreacted metakaolin particles when compared with the same specimen without silica fume. Previous studies have shown that silica fume is not efficient in reducing the porosity of geopolymer (Nmiri et al., 2019; Dutta et al., 2010; Thokchom et al., 2011). Consequently, although silica fume is an efficient filler for normal concrete (Ashish, 2019; Mehta and Ashish, 2020), it may not act the same for geopolymer.

The effect of silica fume on the compressive strength of metakaolin-based geopolymer can vary with different S/A ratios. Silica fume slightly enhanced the compressive strength of the S/A 1.0 specimen, which can be associated with the dissolution of silica fume. Given that no silica fume particles were observed in the SEM images of the S/A 1.0 specimen, it can be inferred that the majority of silica fume was dissolved by the alkaline solution. If all of the 15% silica fume dissolves, the S/A ratio increases from 1.0 to 1.27, which is still relatively low compared to 1.4 or 1.8. Therefore, the S/A 1.0 specimen can be inferred to accommodate a larger amount of silica fume than 15%. The S/A 1.4 specimen achieved the highest compressive strength at 10% silica fume. The lower compressive strength at 15% silica fume can be attributed to an excessive amount of silica fume in geopolymer structure. In the case of the S/A 1.8 specimens, however, silica fume was barely beneficial because the increase in the S/A ratio over 1.8 deteriorates its compressive strength. Note that the optimum S/A ratio of the 1.8 specimen was investigated through trial tests. The compressive strength of the S/A 1.8 specimen degraded with 5% silica fume. These complex trends in the compressive strength can be attributed to the combined effects of silica fume in geopolymer as additional silicon supply and fillers. As a result, the effect of silica fume should be studied considering the S/A ratio of metakaolin-based geopolymer.

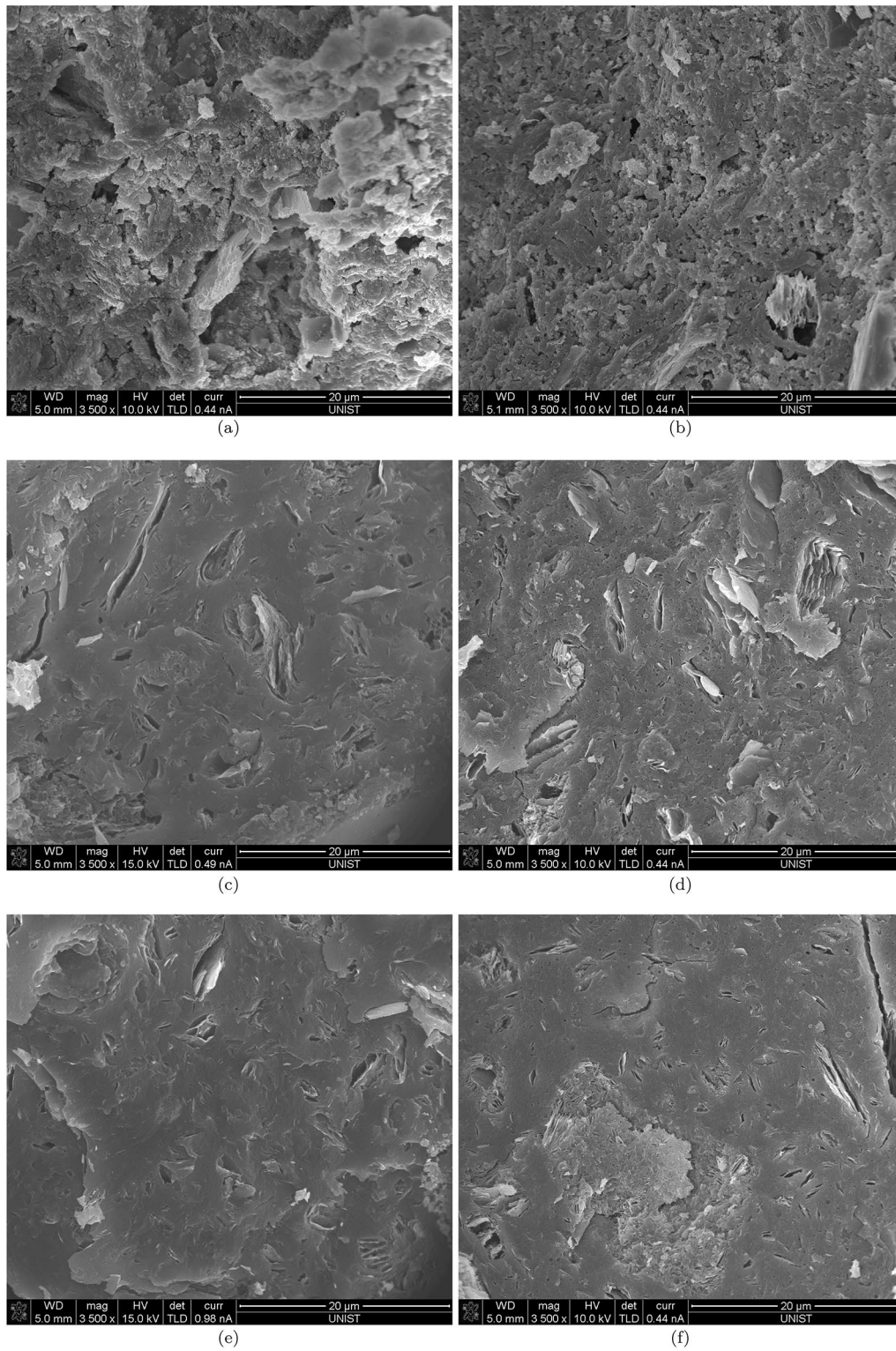


Fig. 6 SEM images of geopolymer specimens: **a** S/A 1.0, **b** S/A 1.0 - SF15, **c** S/A 1.4, **d** S/A 1.4 - SF15, **e** S/A 1.8, and **f** S/A 1.8 - SF15.

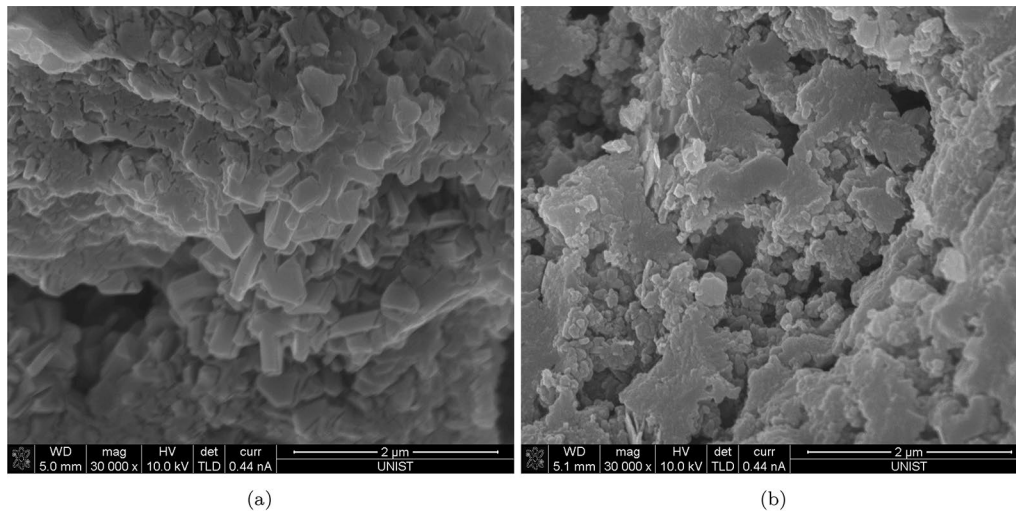


Fig. 7 SEM images of the S/A 1.0 geopolymer specimens: **a** without silica fume and **b** with silica fume.

An initial expansion of geopolymer was observed during the volume measurement tests, which occurred in all of the geopolymer specimens with or without silica fume. The degree of the initial expansion depended on the initial setting time of the specimen; if the specimen set faster, it stopped the initial expansion earlier and the increase of the volume was smaller. After the initial expansion, the geopolymer synthesized with the sodium silicate solution began to shrink as silicate oligomers transform alumina-rich structures to denser structures (Li et al., 2019). It can be deduced that the drying of the specimen during the volume measurement tests caused shrinkage. However, a previous study showed that the drying shrinkage was barely observed until approximately 50% of the initial mixing water evaporated when the geopolymer specimens were cured at an ambient temperature (Kuenzel et al., 2012). The 6–7% mass loss during the entire experiment (up to 90 days) corresponds to the evaporation of 18–22% of the initial mixing water. Therefore, the volume shrinkage measured in this study can be mainly attributed to the condensation process of polymerization, and the degree of that condensation increases with the increase of the S/A ratio.

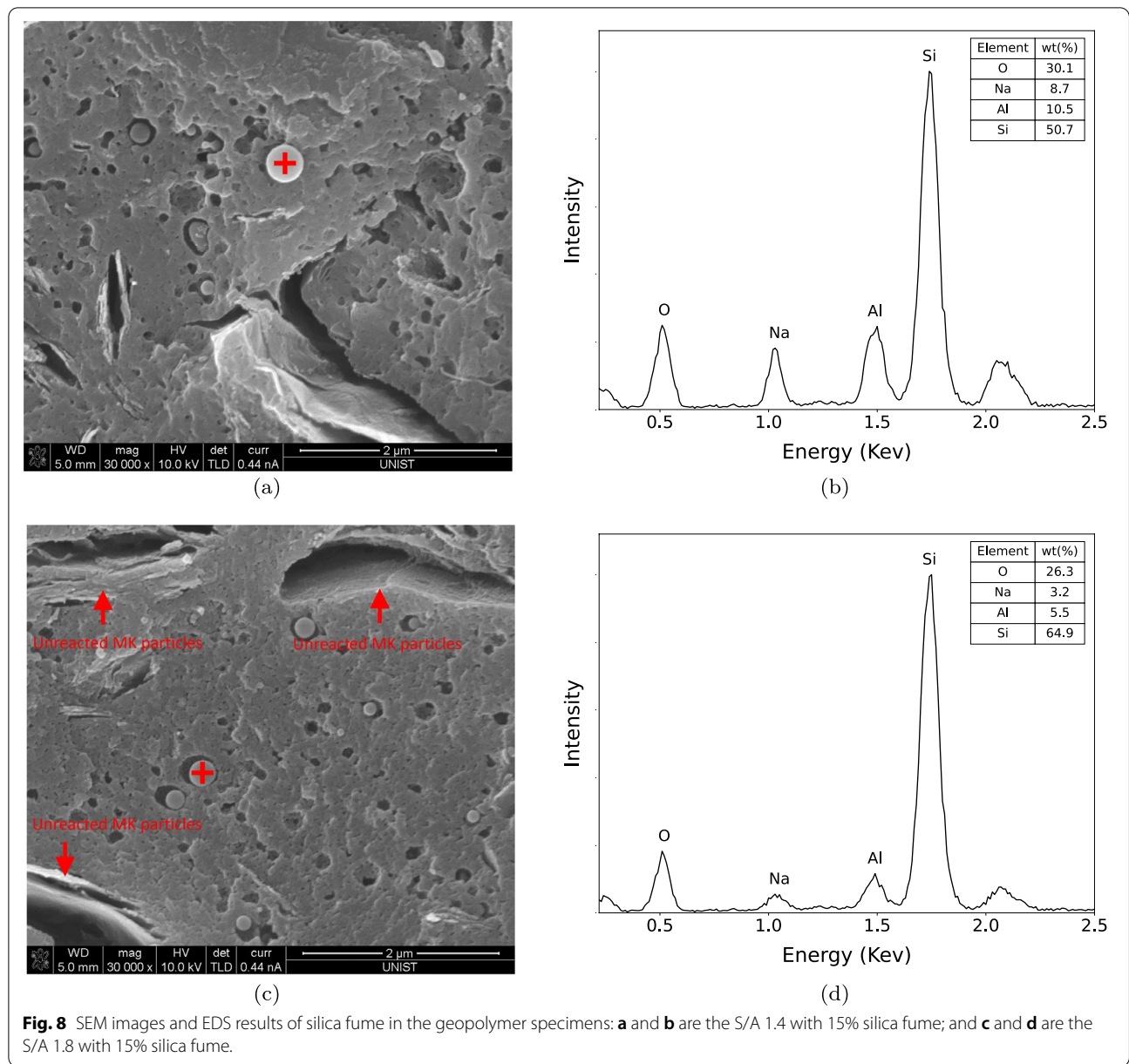
The degradation in compressive strength caused by the volume expansion of geopolymer with silica fume remains unclear. Although volume expansions of the S/A 1.0 and 1.8 specimens were observed after approximately 60 days, the S/A 1.4 specimen did not expand. Furthermore, damage due to the addition of silica fume was not observed in the dynamic modulus test. The increase of dynamic moduli of geopolymers with or without silica fume was almost the same, and did not decrease over time. In SEM images, no crack around silica fume

particles was observed. Lastly, the compressive strength did not decrease between 28 and 90 days. Therefore, this study did not determine that silica fume expands and degrades the compressive strength of geopolymer. Although the 90-day period of observation used in this study was based on previous literature (Sukontasukkul et al., 2020; Songpiriyakij et al., 2010), the measurement needs to be conducted for a longer period to see whether further expansion of the S/A 1.8 specimen degrades its compressive strength. Also, further studies are required to investigate the necessary conditions for the volume expansion of geopolymer with silica fume.

5 Concluding Remarks

This paper investigated the effect of silica fume on the mechanical properties of metakaolin-based geopolymer with different S/A ratios. It was hypothesized that the S/A ratio of geopolymer influences the effect of silica fume. The compressive strength and initial setting time tests were conducted on the geopolymer with a S/A ratio from 1.0 to 2.0. To examine the finding by previous studies that silica fume expands in geopolymer and degrades its compressive strength, the volume and dynamic modulus of geopolymer were also measured. The key findings and observations are summarized below:

- 1 Silica fume influences the properties of metakaolin-based geopolymer in two ways. First, silica fume increases the S/A ratio, as a part of the silica fume is dissolved in an alkaline solution providing additional silicon. The increase in the S/A ratio affects both the initial setting time and the structure of geopolymer. Second, silica fume can exist inside geopolymer



matrix. The SEM images confirm the existence of silica fume particles although they are placed in a geopolymer structure leaving the majority of the pores in unreacted metakaolin particles empty. This implies that silica fume cannot efficiently decrease the porosity of geopolymer.

- The effect of silica fume on compressive strength differs between S/A ratios. This can be attributed to the combined roles of silica fume in geopolymer. When the S/A ratio of geopolymer is low, silica fume can enhance the compressive strength of geopolymer as silica fume increases the S/A ratio. When the S/A

ratio is high, silica fume can increase the S/A ratio over the optimum; therefore, degrading the compressive strength. Consequently, the S/A ratio of geopolymer should be considered when silica fume is incorporated.

- Geopolymer initially expands regardless of whether it contains silica fume. The degree of the initial expansion is related to the initial setting time; when material sets faster, it expands less. After the initial expansion, geopolymer begins to shrink and the degree of shrinkage is dependent on the S/A ratio.

- 4 The volume expansion of geopolymer with the presence of silica fume at a late age is also dependent on the S/A ratio. The S/A 1.4 specimen shrinks after the initial expansion, whereas the S/A 1.0 and 1.9 specimens exhibit continuous volume expansion. However, the volume expansion with silica fume does not degrade the compressive strength of geopolymer. The dynamic modulus test also confirms that no damage occurs due to the addition of silica fume.

This study explains why the optimum dosage of silica fume was found to vary in previous studies. The S/A ratio of geopolymer should be considered during the incorporation of silica fume. Specifically, as a part of silica fume dissolves in alkaline solution resulting in extra silicon content, the S/A ratio of geopolymer should be lower than the optimum value that yields the highest compressive strength without silica fume. Previous studies have reported that silica fume in geopolymer can enhance its resistance against chemical attack (Chindaprasirt et al., 2014; Rostami and Behfarinia, 2017; Thokchom et al., 2011). Therefore, the results of this study could inform the development of a more durable mix design of geopolymer using silica fume.

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Authors' contributions

Sungwoo Park: conceptualization, methodology, investigation, validation, formal analysis, investigation, data curation, writing-original draft preparation, writing-review and editing, visualization; Juan Yu: investigation, formal analysis, data curation, writing-original draft preparation, writing-review and editing, visualization; Jae Eun Oh: writing-review and editing, resources, supervision, project administration, funding acquisition; Sukhoon Pyo: writing-review and editing, resources, supervision, project administration, funding acquisition; All authors read and approved the final manuscript.

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Availability of data and materials

The data presented in this study are available on request from the corresponding authors.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare that they have no competing interests.

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