

Effect of Sodium Aluminate on Compressive Strength of Geopolymer at Elevated Temperatures

Ji Hoi Heo, Jun Seong Park, Hyo Kim

Abstract—Geopolymer is an inorganic material synthesized by alkali activation of source materials rich in soluble SiO_2 and Al_2O_3 . Many researches have studied the effect of aluminum species on the synthesis of geopolymer. However, it is still unclear about the influence of Al additives on the properties of geopolymer. The current study identified the role of the Al additive on the thermal performance of fly ash based geopolymer and observing the microstructure development of the composite. NaOH pellets were dissolved in water for 14 M (14 moles/L) sodium hydroxide solution which was used as an alkali activator. The weight ratio of alkali activator to fly ash was 0.40. Sodium aluminate powder was employed as an Al additive and added in amounts of 0.5 wt.% to 2 wt.% by the weight of fly ash. The mixture of alkali activator and fly ash was cured in a 75°C dry oven for 24 hours. Then, the hardened geopolymer samples were exposed to 300°C, 600°C and 900°C for 2 hours, respectively. The initial compressive strength after oven curing increased with increasing sodium aluminate content. It was also observed in SEM results that more amounts of geopolymer composite were synthesized as sodium aluminate was added. The compressive strength increased with increasing heating temperature from 300°C to 600°C regardless of sodium aluminate addition. It was consistent with the ATR-FTIR results that the peak position related to asymmetric stretching vibrations of Si-O-T (T: Si or Al) shifted to higher wavenumber as the heating temperature increased, indicating the further geopolymer reaction. In addition, geopolymer sample with higher content of sodium aluminate showed better compressive strength. It was also reflected on the IR results by more shift of the peak position assigned to Si-O-T toward the higher wavenumber. However, the compressive strength decreased after being exposed to 900°C in all samples. The degree of reduction in compressive strength was decreased with increasing sodium aluminate content. The deterioration in compressive strength was most severe in the geopolymer sample without sodium aluminate additive, while the samples with sodium aluminate addition showed better thermal durability at 900°C. This is related to the phase transformation with the occurrence of nepheline phase at 900°C, which was most predominant in the sample without sodium aluminate. In this work, it was concluded that sodium aluminate could be a good additive in the geopolymer synthesis by showing the improved compressive strength at elevated temperatures.

Keywords—Compressive strength, fly ash based geopolymer, microstructure development, sodium aluminate.

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I. INTRODUCTION

CEMENT is a general term for inorganic binders that binds substances together, or reacts with other substances such as water solidifying itself or to aggregate other substances together. However, as limestone, mud, and gypsum are used to bind or harden building materials in the field of construction, the term “cement” began to be treated as a kind of construction material. Concrete that is made by mixing cement with aggregates has a lot of advantages such as high compressive strength, durability, low cost, and possible in-situ construction, making it widely used for constructing infrastructure such as buildings, bridges, roads and dams all around the world [1].

The term “cement” generally refers to the Ordinary Portland Cement (OPC) as it is the most widely used, accounting for more than 95% of the total concrete use [2]. OPC is rich in raw materials and its production process is not complicated, making it suitable for mass production. However, the production process of cement is very energy intensive and at the same time emits large amounts of carbon dioxide (CO_2). According to [3], 1 ton of cement are produced emitting approximately 1 ton of CO_2 . In the cement industry, various efforts have been made to reduce carbon dioxide emission by using biofuels or introducing new clinker types that consume less energy and by developing ecofriendly concrete [4]. However, proportion of CO_2 emission released from the cement industry in total global CO_2 emission has increased from 5% in 2000 to 8% in 2014 due to the growing demand of cement in developing countries like China and India [5], [6]. In this respect, geopolymer is getting attention as a ecofriendly substitute of OPC.

Geopolymer is inorganic material having a three dimensional aluminosilicate structure that is synthesized by mixing raw materials rich in silica (SiO_2) and alumina (Al_2O_3) with alkali activator. The properties of geopolymer depend on many chemical and physical factors, including a type of alkali activator, curing method, and characteristics of the raw materials, etc. In particular, the relative ratio of Al and Si in geopolymer structure was investigated to have a significant effect on the properties [7]. In addition, previous study had shown that Al-O-Al bonds are structurally unstable (Loewenstein's Rule, 1954), and Si-O-Si bonds are relatively stronger than Al-O-Si or Al-O-Al bonds [8]. Besides, many previous studies have investigated the properties of geopolymers by controlling the Si/Al ratio using additives of Si components such as silica fume, waste glass, sodium silicate, and blast furnace slag [9].

The process of dissolution of silica and alumina from raw material by alkali activation and reorientation with alkali cation is as follows [10]. First, as the raw material meets the alkaline

component and is activated, the Al and Si components are dissolved. At this time, the Al components are dissolved first since Al-O-Si bond of the raw material is relatively weaker than that of Si-O-Si [11]. Therefore, a siliceous layer is formed on the surface of the raw material from which Al components were removed in the early stage of geopolymerization and, at the same time, a primary geopolymer gel rich in Al, named gel 1, is formed around the raw material [12]. After gel 1 is formed, the Si components begin to dissolve slowly from the siliceous layer along with further geopolymerization. At this time, Si components are combined into gel 1 to form a secondary geopolymer gel, called gel 2 [12]. In the gel 2, three dimensional aluminosilicate structures are synthesized by repeating the bonding of Al and Si components forming tetrahedral structures. The types of aluminosilicate synthesized include poly (sialate) [-Si-O-Al-O-], poly (sialate-siloxo) [-Si-O-Al-O-Si-O-], and poly (sialate-disiloxo) [-Si-O-Al-O-Si-O-] and these compounds are charge balanced with alkali cations [12].

Considering this reaction process, the presence of Al before the gel 1 and gel 2 can have various effects on geopolymer synthesis. Weng et al. [13] reported that the aluminate component attracts other negative charged species better than the silicate component and then promotes condensation reaction of the geopolymer. In addition, Hajumohammadi et al. [14], who performed ATR-FTIR and SR-FTIR analysis, reported that the addition of aluminate to process of geopolymer synthesis accelerates the transition to the gel 1 state, thereby promoting the geopolymerization and forming more homogeneous geopolymer structure. Fernandez-Jimenez et al. [15] found that the more Al components in the geopolymer synthesis, the more Si components that could be incorporated in the gel 2 state, increasing properties of geopolymer, and C. A. Ress et al. [16] reported that the addition of alumina nano-particles to geopolymer reactions promotes nucleation and increases the degree of reaction.

Despite these studies, very few studies have investigated the chemical and physical properties of geopolymers by using Al additives. In particular, research on the physical and chemical properties of Al added geopolymer at high temperatures has not been reported. Therefore, this study investigates the chemical and physical properties of geopolymer synthesized by adding Al component at high temperature.

II. EXPERIMENTAL

Coal fly ash was used as a raw material and supplied from Seocheon power plant, South Korea. Fly ash is a by-product produced at coal-fired power plants and has abundant amorphous silica and alumina components. Particle size distribution, chemical composition and crystalline phase of fly ash were analyzed to investigate characteristics of the raw materials.

The particle size distribution of fly ash was analyzed using a particle size analyzer (PARTICA, LA-960, HORIBA, Japan). The mean particle size, median size, and mode size were 29.6 μm , 16.54 μm , and 12.46 μm , respectively, as shown in Fig. 1. The chemical composition was analyzed by X-ray fluorescence

and the specific result is given in Table I. The fly ash was classified as Class F fly ash according to ASTM C618 because the sum of the components of silica (SiO_2), alumina (Al_2O_3), and iron oxide (Fe_2O_3) exceeds 70 wt.% and the content of lime (CaO) is less than 7 wt.%. Also, the crystalline phase of fly ash was determined by X-ray diffraction (X-ray diffractometer, X'pert APD, PHILIPS, Netherland) analysis using $\text{Cu K}\alpha$ radiation and the result was provided in Fig. 2. As shown in Fig. 2, the fly ash contains crystalline phases of quartz and mullite with broad hump around $2\theta=22.5^\circ$ indicating the existence of amorphous phase. Sodium hydroxide solution of 14 M (mole/L) was used as alkali activator, made from NaOH pellets ($\geq 93\%$ purity, Duksan, South Korea). Sodium aluminate powder (NaAlO_2 , 97.7% pure, DAEJUNG CHEMICALS & METALS Co. Ltd, South Korea) which is utilized as an Al additive was dissolved in the sodium hydroxide solution.

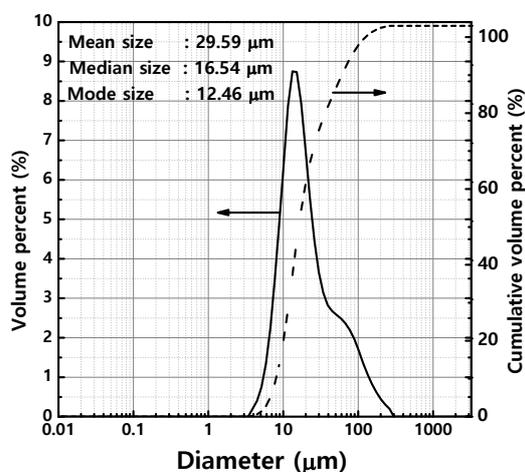


Fig. 1 Particle size distribution of fly ash

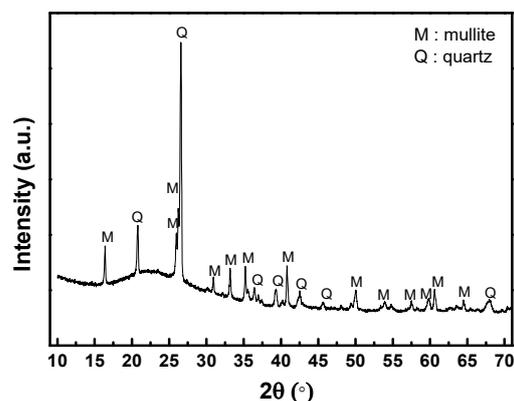


Fig. 2 XRD spectrum of fly ash

The synthesis of geopolymer samples was carried out as following processes. First, 14 M sodium hydroxide solution was prepared and sodium aluminate was dissolved in the sodium hydroxide solution, with amounts corresponding to 0.5%, 1.0%, 1.5%, and 2.0% of the weight of fly ash. The solution was rested in laboratory to cool down before use.

Then, the solution was added to the fly ash at a ratio of 0.40 to the fly ash mass and mixed for 5 minutes using the Hobart Mixer. Specific mix proportions and sample names are shown in Table II. The flowing mixture was poured into a plastic mold capable of making three 50 mm × 50 mm × 50 mm samples, then sealed with a flat plastic panel, and cured in a dry oven at 75°C for 24 hours. The cured geopolymer sample was demolded and heated in an electric furnace (electric furnace, S-1700, HANTECH, Korea). The specific heating process was presented in Fig. 3. Samples were heated from room temperature to high temperatures, 300°C, 600°C, and 900°C, respectively with an increasing rate of 8°C/min. After reaching the target temperature, heating was continued for 2 hours at that temperature, after which, the heated sample was naturally cooled to room temperature.

TABLE I
CHEMICAL COMPOSITION OF FLY ASH

Component	wt. %
SiO ₂	59.2
Al ₂ O ₃	23.3
Fe ₂ O ₃	5.95
CaO	3.50
K ₂ O	0.986
MgO	1.14
MnO	0.07
Na ₂ O	0.88
TiO ₂	1.14
LOI	3.07

Compressive strength of geopolymer samples was investigated using compression testing machine (PL-9700H, Woojin Precision Co., South Korea) and chemical properties were determined by ATR-FTIR and XRD analysis. The morphology of geopolymers were examined by SEM inspection.

TABLE II
MIX DESIGN OF GEOPOLYMER SAMPLES WITH VARIOUS CONTENTS OF SODIUM ALUMINATE

Sample name	NaOH solution / Fly ash (wt./wt.)	Sodium aluminate / Fly ash (wt./wt.)
Con	0.40	-
SA0.5	0.40	0.005
SA1.0	0.40	0.010
SA1.5	0.40	0.015
SA2.0	0.40	0.020

III. RESULTS AND DISCUSSIONS

A. XRD Analysis

Fig. 4 shows the XRD results of fly ash and geopolymer samples of SA2.0 over the elevated temperatures of 300°C, 600°C, and 900°C. No determinative differences were observed depending on the content of sodium aluminate but differences in types of crystalline phase were observed with the various exposure temperatures. Peaks of mullite and quartz were observed in the fly ash spectrum but after geopolymerization, a peak indicating sodalite was additionally observed. The

crystalline phases of the samples exposed to 300°C and 600°C were not much different from those of the sample not heated (ambient), but dehydration of sodalite occurred after heating. The dehydration is a result of the evaporation of water that had been bonded chemically and physically to the geopolymer structure.

After exposure to 900°C, the existing crystalline phases disappeared and new crystalline phases including nepheline, albite, and magnetite were formed. The sodalite, which existed when exposed at temperatures below 600°C, had been transformed into nepheline [17] and the iron (Fe) elements that existed in small quantities in the geopolymer structure formed the magnetite along with change of the sample's color to red as being oxidized at that temperature.

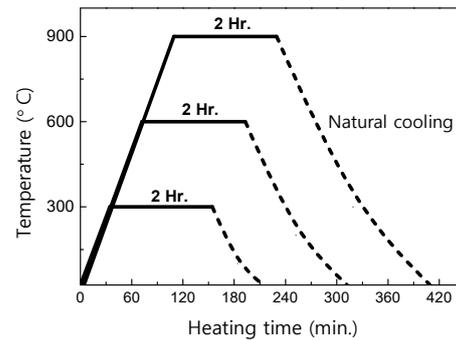


Fig. 3 Heating schedules for elevated temperatures of 300, 600, and 900°C

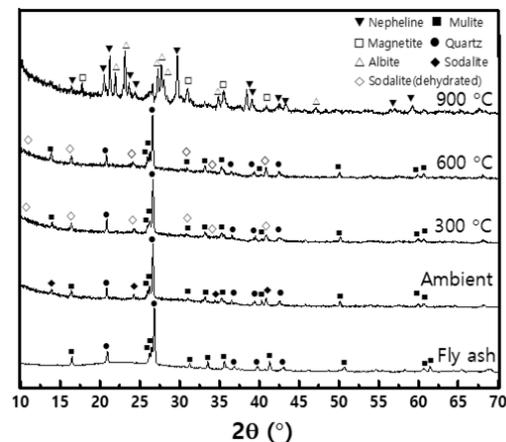


Fig. 4 X-ray diffraction patterns of fly ash and geopolymer samples of SA2.0 over the elevated temperatures of 300°C, 600°C, and 900°C

B. FTIR Analysis

Results of the FTIR analysis of fly ash and the geopolymer containing 2 wt.% of sodium aluminate (SA2.0) which exposed to the temperatures from ambient to 900°C are presented in Fig. 5. In the FTIR spectrum of fly ash, a peak that appears near 1061 cm⁻¹ indicates Si-O-T (T means Si or Al) asymmetric stretching vibration. The peak moves to a low wavelength of 970 cm⁻¹ as Al rich gel (gel 1) is formed in the early stage of geopolymerization [8], [14]. After that, the peak gradually

moves to a higher wavelength again with bonding of Si components or silanol groups present in the geopolymer structure, increasing compressive strength [8], [13]. As indicated in Fig. 5, the peak corresponding to Si-O-T asymmetric stranding vibration moves from 960 cm^{-1} to 990 cm^{-1} as the exposure temperature increases. As mentioned above, the peak moved due to the bonding of Si components or silanol groups with the Al components. It is also related to the tendency of increased compressive strengths when exposed to higher temperature.

After exposure to 900°C , a new peak appears near 578 cm^{-1} and 715 cm^{-1} . The formation of these peaks is attributed to the newly formed crystalline phase at that high temperature as revealed in the XRD results, above mentioned. In addition, in the FTIR result of geopolymer not exposed to high temperatures, a peak corresponding to OH vibration of existent moisture in the geopolymer structure appeared near 1653 cm^{-1} [18]. The peak disappears after heated at temperatures above 300°C indicating evaporation of moisture present inside the structure. These results are also consistent with the XRD results which showed dehydration of sodalite.

Figs. 6 and 7 are presented to compare the geopolymer structure not exposed to high temperature with the one exposed to 900°C with increasing content of sodium aluminate. Fig. 6 shows the FTIR results of geopolymer with various sodium aluminate content which is not exposed to high temperatures. As the content of sodium aluminate increased, the peak corresponding to Si-O-T asymmetric straining vibration appeared at the lower wavelength. The peak of geopolymer without sodium aluminate (Con) was located at 971 cm^{-1} while the peak of geopolymer with 2 wt.% sodium aluminate (SA2.0) appeared at lower wavelength 962 cm^{-1} . This arose from the increased incorporation of Al component into the geopolymer structure with increment of sodium aluminate [14].

Fig. 7 presents the FTIR spectra of geopolymer by sodium aluminate content after exposure to 900°C . The peak corresponding to Si-O-T shifted to a higher wavelength compared to the one not exposed to the elevated temperatures. Also, the wavelength number where the peak was located was larger as the sodium aluminate content increases. The peak of the geopolymer without sodium aluminate (Con) rarely moved from 971 cm^{-1} to 969 cm^{-1} , while the peak of the one containing 2.0wt.% of sodium aluminate shifted largely from 962 cm^{-1} to 991 cm^{-1} , both after exposure to 900°C .

Madavarapu and Hajimohamadi et al. [14], [19] reported that during the geopolymerization, the peak of Si-O-T asymmetric stretching vibration shifted to higher wavelengths as the Si component and silanol group were combined with the structure. Also, Fernandez-Jimenez et al. [15] reported that the more Al components in the process of geopolymer synthesis, the more Si components that can be incorporated in the gel 2 state, increasing properties of geopolymer. Consequently, the degree of reduction in compressive strength declined after exposure to 900°C with increasing addition of sodium aluminate because higher sodium aluminate content led to further bonding of Si component.

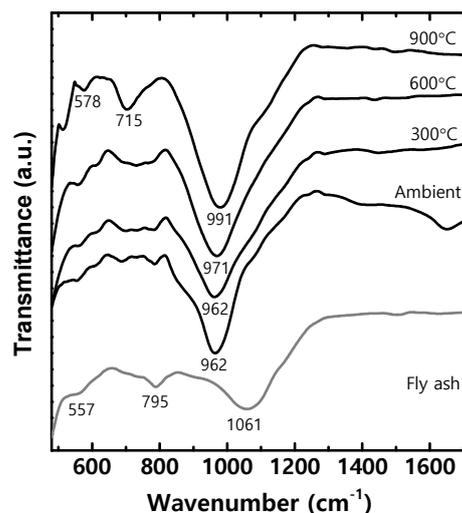


Fig. 5 FTIR spectra of fly ash and geopolymer containing sodium aluminate with the amount of 2 wt.% at the temperature from ambient to 900°C

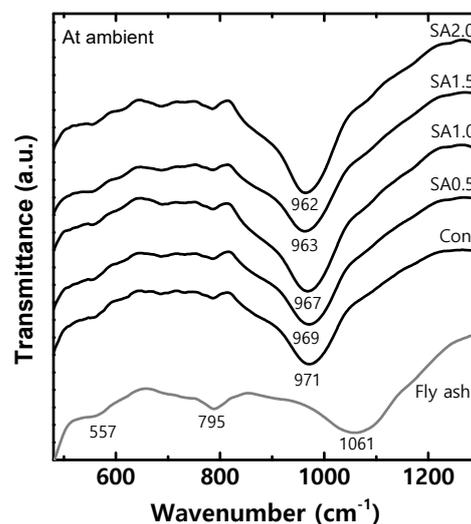


Fig. 6 FTIR spectra of fly ash and geopolymer with various contents of sodium aluminate at ambient

C. Compressive Strength

Considering the frequent occurrence of fire in buildings, the high temperature durability as building materials is an important factor for safety. Tendencies of compressive strength over the high temperatures with increment of sodium aluminate are presented in Fig. 8. For samples not exposed to high temperatures after curing, the compressive strength tends to increase as the sodium aluminate increases. After heating the cured samples at 300°C , the compressive strength of all the samples increased above the values of ambient temperature and the higher the compressive strength with increment of sodium aluminate. The same trend was observed even after heating at 600°C , achieving the highest compressive strength for all samples. This tendency to increase compressive strength when heated from room temperature to 600°C is related to increased

concentration of the components due to water evaporation inside the sample [20] and bonding of silanol group [19], [21]. The free water present inside the geopolymer sample evaporates in large quantities near a temperature of 150°C [12], which increases the concentration of Si and Al components inside the sample and then improves the compressive strength with an additional geopolymer reaction. In addition, the compressive strength increases as the silanol group present in the structure forms Si-O-Si bonds (dehydroxylation) when geopolymer is heated to a temperature above 300°C. However, after exposed to 900°C, the compressive strength of all kinds of samples tends to decrease, which is related to deformation of the geopolymer structure and crystalline phases and the excessive evaporation of moisture inside the samples at high temperature (>600°C).

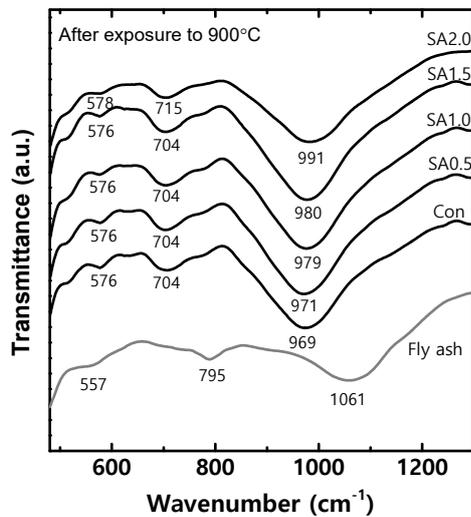


Fig. 7 FTIR spectra of fly ash and geopolymer with various contents of sodium aluminate after heating to 900°C

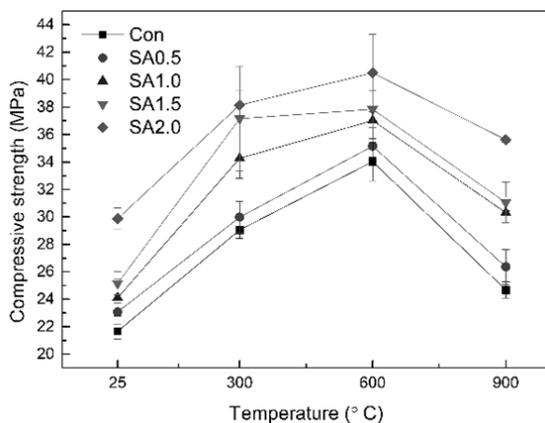


Fig. 8 The variation in compressive strength over the elevated temperatures with geopolymer sample containing sodium aluminate with the amount of 0 to 2 wt.%

Ratios of the reduced compressive strength at 900°C against the compressive strength at 600°C, which is the highest value,

are shown in Fig. 9. The degree of reduction of compressive strength at 900°C compared to the maximum compressive strength was decreased as the amount of sodium aluminate increased. The geopolymer specimen without sodium aluminate (Con) achieved the highest compressive strength of 34.0 MPa at 600°C and the value dropped to 24.7 MPa at 900°C, showing a relatively large reduction of 27.5%. In contrast, the geopolymer sample with 2 wt.% sodium aluminate (SA2.0) had the highest compressive strength of 40.5 MPa at 600°C and 35.6 MPa at 900°C, showing a small decrease of 12.0%. The smaller reduction of compressive strength at 900°C with increment of sodium aluminate can be attributed as follows. The FTIR analysis, as mentioned above, showed that Si components that combined at high temperature increased as more Al components existed in the geopolymer structure, and the combination of these Si components increased durability at high temperature. In addition, the presence of the Al components in the geopolymer gel in the early stage of geopolymerization is known to make the geopolymer structure more homogenous [14], which was also analyzed to increase durability by lessening the thermal expansion differences between the geopolymer structures at high temperatures.

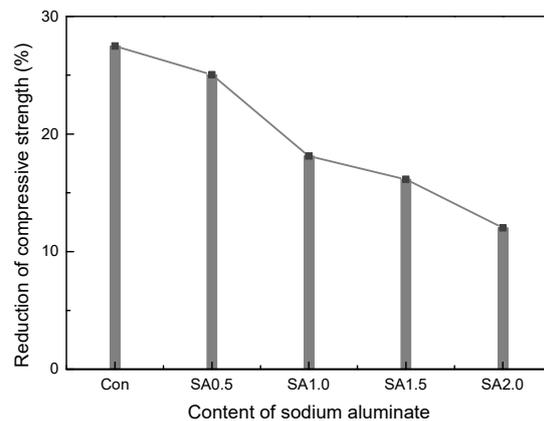


Fig. 9 The reduction rate of compressive strength at 900°C (compared to the value at 600°C) according to content of sodium aluminate

D.SEM Analysis

SEM images of geopolymer by sodium aluminate content at room temperature, 300°C, 600°C, and 900°C are provided in Figs. 10-13, respectively. As the content of added sodium aluminate increased, the larger amount of geopolymer structure was synthesized. This can be attributed to that the addition of Al component stimulated the generation of crystalline nuclei in the early stages of geopolymerization [10] and at the same time accelerated the transition to gel I state, which resulted in a lot of synthesis of geopolymer [19]. The tendency of microstructure by sodium aluminate content was the same even after being exposed to 300°C as indicated in Fig. 11. The SEM images after heating at 600°C showed that as the content of sodium aluminate increases, porous structures that appeared at room temperature and 300°C tend to become denser and more homogenous. This result was also consistent with increased compressive strength as the content of sodium aluminate

increases at that temperature. After exposure to 900°C, the geopolymer structure changed to a smooth form as sintering occurred, that is consistent with changes of crystalline phase in XRD and formation of new peaks in FTIR results at the corresponding temperature.

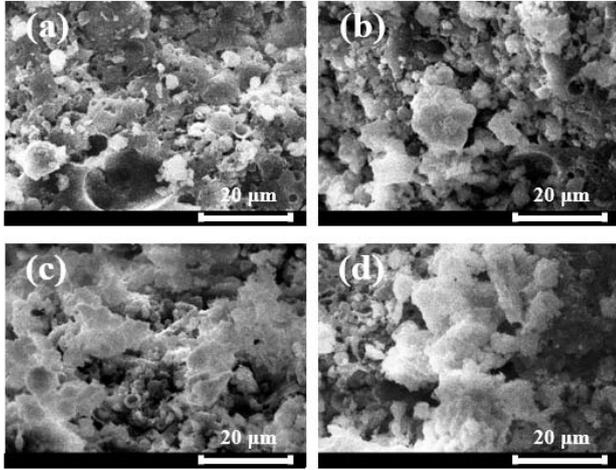


Fig. 10 SEM images of the geopolymer samples of (a) Con, (b) SA0.5, (c) SA1.0, and (d) SA2.0 at ambient

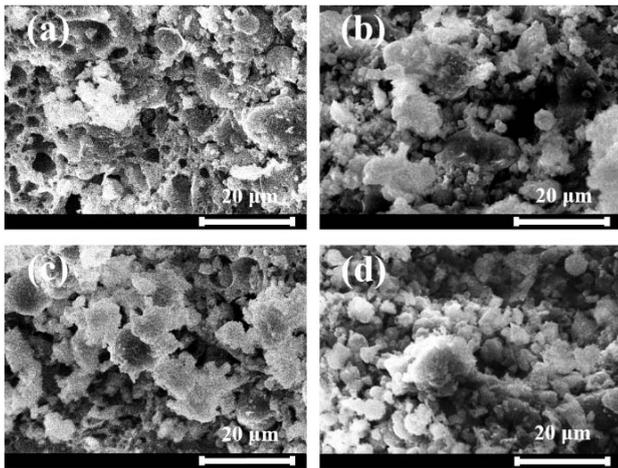


Fig. 11 SEM images of geopolymer samples of (a) Con, (b) SA0.5, (c) SA1.0, and (d) SA2.0 after heating to 300 °C

IV. CONCLUSIONS

Many studies have investigated geopolymer properties using additives from Si but using Al additives has been studied rarely, since the bonding of Al has been known to be chemically unstable. However, in the few studies in which geopolymers were synthesized by adding Al component, the presence of Al in the initial stage of geopolymerization could change the geopolymer reaction mechanism and thus expectable improved physical properties. Therefore, in this experiment, sodium aluminate was added by dissolving in an aqueous sodium hydroxide solution to investigate the effect of Al components on the properties of geopolymer. The cured geopolymers

were heated from room temperature to high temperatures (300, 600, and 900°C) and then their physical and chemical properties were investigated.

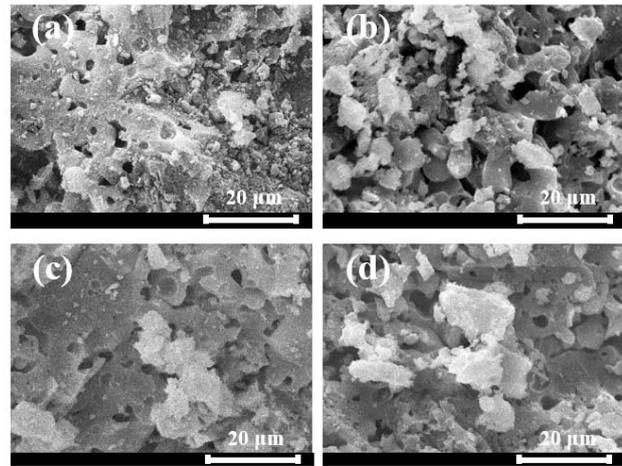


Fig. 12 SEM images of geopolymer samples of (a) Con, (b) SA0.5, (c) SA1.0, and (d) SA2.0 after heating to 600°C

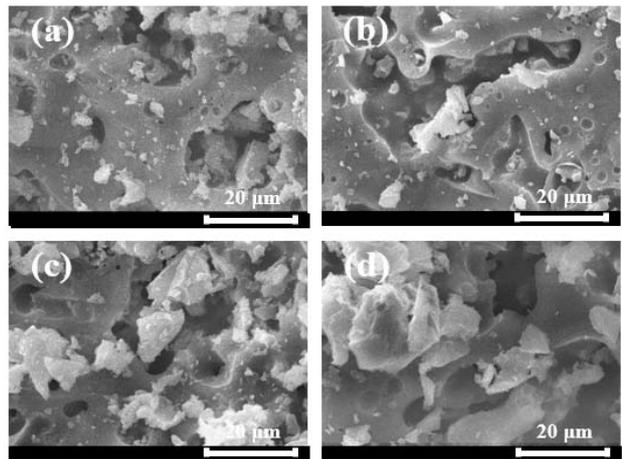


Fig. 13 SEM images of geopolymer samples of (a) Con, (b) SA0.5, (c) SA1.0, and (d) SA2.0 after heating to 900°C

By the changing content of sodium aluminate, the compressive strength and wavenumber of the peak in FTIR spectra changed at various high temperatures, as follows:

- 1) The compressive strength of all geopolymer samples with each sodium aluminate content ratio was increased up to 600°C but decreased at 900°C. In addition, with increment of sodium aluminate, the compressive strength was increased and the ratio of compressive strength reduction at 900°C compared to its maximum value was also decreased regardless of the exposed temperature.
- 2) From the results of FTIR analysis, as the content of sodium aluminate increases, more Al is incorporated in the geopolymer structure at the early stage of geopolymerization, which increased the compressive strength at high temperature by increasing the Si

component bonded and reduced the proportion of compressive strength reduction at high temperatures.

APPENDIX

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