

A comparative study of the utilization of synthetic foaming agent and aluminum powder as poreforming agents in lightweight geopolymer synthesis

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Research Article

Ufafa Anggarini*, Suminar Pratapa, Victor Purnomo, Ndaru Candra Sukmana

A comparative study of the utilization of synthetic foaming agent and aluminum powder as pore-forming agents in lightweight geopolymer synthesis

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Abstract: Lightweight geopolymer concrete was synthesized using fly ash as an aluminosilicate source with the addition of a pore-forming agent. The synthesis of a geopolymer was conducted by employing various volume ratios of geopolymer paste to the foaming agent: 1:0, 1:0.67, 1:0.75, 1:1.00, 1:1.33, 1:1.50, and 1:2.00, while the ratios of aluminum powder weight percentage to the fly ash weight varied between 0.01 - 0.15 %wt. The results showed that the higher foaming agent content, the lower the compressive strength and density of the geopolymer. The ratio of the geopolymer paste to the foaming agent, 1:1.33 was found to produce the strongest light weight geopolymer whose compressive strength and density were 42 MPa and 1760 kg/m³, respectively. With the addition of 0.01%wt aluminum powder, the geopolymer specimen showed the highest compressive strength of 42 MPa and density of 1830 kg/m³, respectively. X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM) and FT-IR were utilized to study the effects of foaming agent and aluminum powder addition onto the microstructure, surface morphology, and functional groups of the geopolymer. Both types of synthesized geopolymers have the potential to be developed in terms of compressive strength and density in the future.

Keywords: geopolymer; foaming agent; aluminum powder; compressive strength; density.

1 Introduction

An earthquake occurs due to the mass transfer in the rock layer of the earth, where the strength of an earthquake is affected by the amount of energy released during the shifts and collisions [1]. Indonesia is one of the countries that is heavily struck by earthquakes every year. Therefore, it is necessary to establish an advancement that may reduce the impact, especially in terms of infrastructure. The selection of construction materials with lightweight structures is one of the reasonable inventions to reduce damage and safety risks.

The mass of building construction depends on the types of materials used. The current application of Ordinary Portland Cement (OPC) for construction has a clear advantage in terms of strength but would result in high weight structure [2]. Should any disaster cause a building to collapse, heavier construction materials would significantly slow down the required rescue actions. Therefore, the development of light weight concrete has become one of the concerns today to produce a lighter, safer structure and to reduce the cost of construction [3]. Lightweight concrete was classified as one of materials possessing a typical density ranging from 1000 – 2000 kg/m³ for structural concrete [4]. Conventional lightweight concrete was generally produced from an OPC-based cement. The use of OPC has an adverse effect in terms of the amount of CO₂ emitted from the fuel combustion, limestone decarbonization in kiln, and electricity usage at grinding plants [5]. The amount of CO₂ produced from the cement industry released into the earth's atmosphere reaches about 13,500 tons per year which contributes to the greenhouse effect with an annual increase of 7% [6]. Thus, the geopolymer is an alternative material that can

*Corresponding author: Ufafa Anggarini, Department of Chemical Engineering, Universitas Internasional Semen Indonesia, E-mail: ufafa.anggarini@uisi.ac.id

Suminar Pratapa, Department of Physics, Institut Teknologi Sepuluh Nopember Surabaya

Victor Purnomo, Department of Chemical Engineering, Universitas Internasional Semen Indonesia,

Ndaru Candra Sukmana, Department of Engineering Management, Universitas Internasional Semen Indonesia

Table 1: Chemical composition of fly ash class C.

Oxide	Al ₂ O ₃	SiO ₂	CaO	Fe ₂ O ₃	MgO	K ₂ O
Mass (%)	27.11	46.93	9.07	6.18	3.18	1.49

be used to substitute cement in a more environmental-friendly way that does not require any combustion process.

The synthesis of a geopolymer is generally carried out by reacting an alkali activator solution with an aluminosilicate source, such as methakaoline [7], fly ash [8], and so forth. Hardjito et al., [9] stated that F class of fly ash as the starting material was suitable for a geopolymer synthesis, but in this research, the fly ash used was classified as class C with a high content of CaO (more than 8% weightage). Several studies have indicated that the use of geopolymer as lightweight non-concrete as conducted by Posi et al., [3] where the synthesized lightweight geopolymer contained an aggregate recycle lightweight block with a density and compressive strength of 1400 kg/m³ and 16 MPa, respectively. Another study by Abdullah et al. [10] explained that the use of foaming agents in their study produced lightweight geopolymers with the mechanical compressive strength of 18.19 MPa. According to the Indonesian National Standard (SNI-03-3449-2002), the minimum compressive strength of lightweight concrete was 17.24 MPa, with the highest density of about 1850 kg/m³. Since the compressive strength of lightweight geopolymer obtained in previous studies were low, this study aims to improve the characteristics of lightweight geopolymers as non-cement lightweight concrete with the addition of a foaming agent and aluminum powder as the pore-forming agent.

2 Experimental Section

2.1 Materials and preparation

The fly ash of class C from Paiton Power Plant was used in this experiment as a raw aluminosilicate source. Table 1 shows the chemical composition of this material measured by X-Ray Fluorescence (XRF). An alkali activator solution was prepared by mixing the prepared 7M sodium hydroxide (NaOH pellet, technical grade) solution with sodium silicate solution (Na₂SiO₃, technical grade). The dissolution of sodium hydroxide is an exothermic reaction; thus, the solution was cooled for 24 hours before it was mixed with sodium silicate.

The synthetic foaming agent and aluminum powder as pore-forming agents were used in this study. Synthetic foaming agent is a chemical reagent containing sodium laureth sulphate that facilitates the formation of foam which would be suitable for the production of lightweight concrete with a density of 1000 kg/m³ and above [11]. The foaming agent was a colorless liquid with a specific gravity of 1.05, whereas the aluminum powder was a silver-colored powder with a specific gravity of 2.7. The aluminum powder is commonly used to obtain hydrogen gas produced from chemical reaction that tends to generate cavity in mortar [12].

2.2 The synthesis of geopolymer

The geopolymer paste was prepared by mixing the alkali activator solution with fly ash, followed by a 5-minute stirring process. The Si/Al ratio of 4 [13] and solid/liquid ratio of 2.3 [14] were used to synthesize the geopolymer. The addition of pore-forming agent was performed separately to produce the expanded paste. The geopolymer paste was then casted in cubical moulds with dimensions of 5 x 5 x 5 cm³ and cured for 28 days at room temperature.

2.2.1 Foaming agent (Cellular Lightweight Concrete)

Preformed foam from the synthetic foaming agent was added into the geopolymer paste in various ratios of geopolymer paste to the foaming agent volume of 1:0.50, 1:0.67, 1:0.75, 1:1.00, 1:1.33, 1:1.50, and 1:2.00. Formerly, the foaming agent was pre-formed by mixing the synthetic foam and water by using foam generators. The foam was, then, added and mixed for 5 minutes. Subsequently, the mixing rotation speed was lowered to avoid formation of defects. The lightweight foaming geopolymer was then poured into cubical moulds and cured at an ambient temperature for 28 days.

2.2.2 Aluminum powder (Non-Autoclaved Aerated Concrete)

¹ The ratios of aluminum powder weight percentage to the fly ash weight used in this study were 0.01, 0.02, 0.03, 0.04, 0.05, 0.10 and 0.15 %wt. The addition of aluminum powder was carried out directly without the foam preforming process. A certain amount of aluminum powder was added and mixed with the geopolymer paste for 30 seconds. Finally, the mixture was poured into steel cubical moulds and cured at the same condition with the foaming agent.

2.3 The measurement of density and compressive strength

The dry density measurement for both the lightweight geopolymer types were performed on the cured specimens according to ASTM C138. The density of all samples was determined by dividing the mass of dry lightweight geopolymer by its volume. The compressive strength of the lightweight geopolymer was measured at 7, 21, and 28 days by using the compressive strength machine test instrument at the Physical Laboratory of PT Semen Indonesia (Persero), Tbk. The evaluation of the mechanical strength was performed according to ASTM C109. Table 3 compares the lightweight geopolymers produced from the synthetic foaming agent with that of aluminum powder.

2.4 Characterization of geopolymer specimens

The microstructure of the lightweight geopolymer was characterized by using the X-Ray Diffraction (XRD) PHILIPS Xpert MPD with Cu-K α radiation at a scanning range from 10° to 70°. Functional groups of geopolymer products were identified by using the Fourier Transform Infrared Spectroscopy (FTIR) Thermo Scientific Nicolet IS10 with the wavenumber range of 500 – 4000 cm⁻¹. The surface morphology for both the lightweight geopolymer types were characterized by using the Scanning Electron Microscope (SEM) FEI Inspect 850. All types of lightweight geopolymers were coated with gold palladium metal prior to their analysis in order to enhance their material conductivity.

Ethical approval: The conducted research is not related to either human or animal use.

3 Results and Discussion

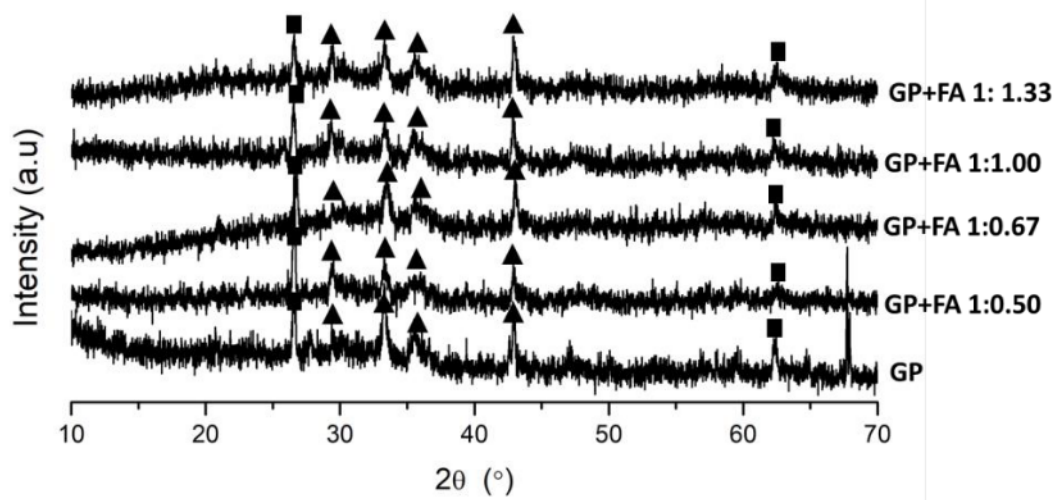
3.1 The effect of different pore-forming agents on geopolymer structure

The geopolymerization was initiated with the dissolution of aluminosilicate source into an alkaline activator solution, resulting in a dissolved alumina-silica species, followed by the initial polymerization which was aided by the alkaline media to produce oligomers. This process was continued to a supersaturated condition. Afterwards, the gel production promotes the structure transformation into an amorphous, solid bound structure [15]. Diffractograms seen in Figure 1(A) and (B) belong to the lightweight geopolymers that were synthesized by utilizing the foaming agent lightweight geopolymer and aluminum powder, respectively.

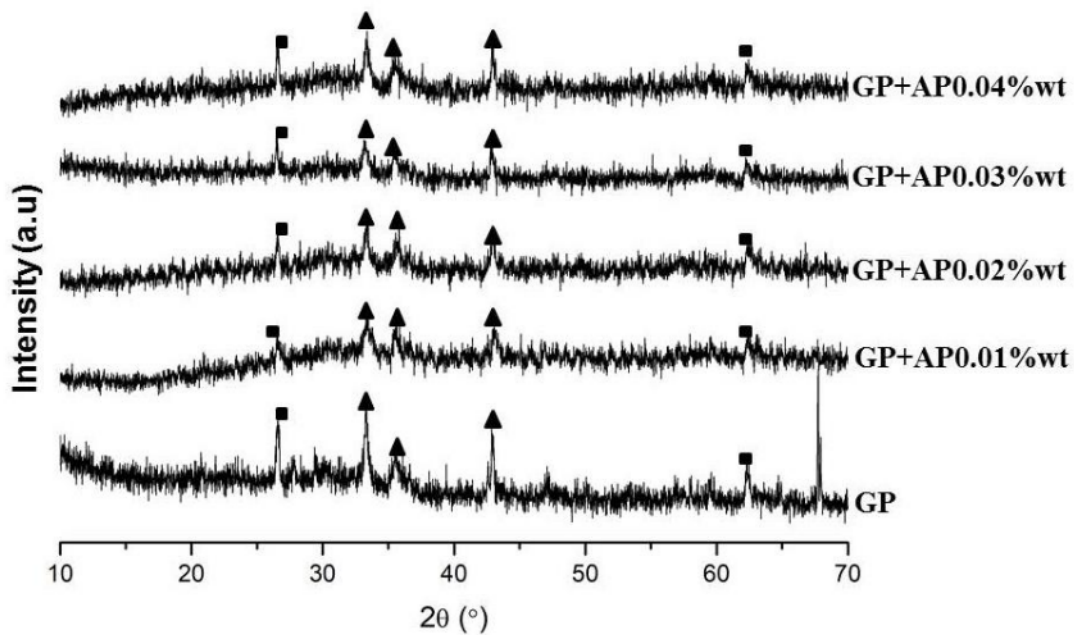
The fact that most peaks in the diffractograms are broad, indicated the existence of an amorphous phase rather than sharp configurations. The broad peak at 2 θ between 20° until 40° confirmed the formation of a polymer structure of the geopolymer [16]. All the diffractogram patterns show two types of phase: quartz and mullite, but with different intensities for both lightweight geopolymer types. The intensity of quartz phase (I_q) and mullite phase (I_m) are shown in Table 2 and the quartz phase weight fraction (W_q) was calculated according to Eq. (1).

$$W_q(\%wt) = I_q / (I_q + I_m) \quad (1)$$

High intensity of quartz peak at 2 θ of 26.55° confirmed that some part of the aluminosilicate source did not react after geopolymerization. The peak intensity decreased as the ratio of the foaming agent was added into the paste was made larger. This means that more aluminosilicate source was converted into the amorphous phase. Apart from that, the addition of 0.01%wt Al powder to geopolymer paste produces the least value of W_q intensity. The more Al powder added to geopolymer matrix, the higher W_q intensity which indicates that there was more unconverted quartz phase, therefore, not forming amorphous geopolymer matrix. Generally, the amount of aluminum powder added into the geopolymer paste changed the peak intensity rather than affecting the crystal phase of the lightweight geopolymer.



(A)



(B)

■ - Quartz phase; ▲ - Mullite phase

Figure 1: The diffractogram of (A) geopolymer without and with foaming agent addition and (B) geopolymer without and with the addition of aluminum powder.

Table 2: Intensity comparison between quartz phase (Iq) and mullite phase in geopolymer and lightweight geopolymer matrix.

Sample ID	Iq	Im	Wq (%wt)
GP	90.40	85.60	51.36
Foam Agent (FA) Lightweight Geopolymer			
GP+FA 1:0.50	58.10	97.80	37.27
GP+FA 1:0.67	92.70	88.40	51.19
GP+FA 1:1.00	55.00	78.10	41.32
GP+FA 1:1.33	32.90	81.90	28.66
Aluminum Powder (AP) Lightweight Geopolymer			
GP+AP 0.01%wt	41.10	72.20	36.28
GP+AP 0.02%wt	57.90	93.60	38.22
GP+AP 0.03%wt	73.30	104.80	41.29
GP+AP 0.04%wt	107.60	92.50	53.77

3.2 The effect of pore-forming agent on geopolymer functional groups

The functional groups of the lightweight geopolymers with and without the pore-forming agent additions were characterized by using Infrared Spectroscopy, as shown in Figure 2. Infrared spectra of geopolymers without the pore-forming agent addition (GP) showed typical peaks at 1000 and 600 cm^{-1} that are related to the stretching vibration bands of Si-O-Si and O-Si-O, respectively. In addition, another peak could be observed at wavelengths of 970 and 800 cm^{-1} due to the bending vibrations of Al-O-Si and asymmetric vibration of Si-O-T, respectively. The other FTIR spectra showed the same vibrational peaks with different intensities, as it could be seen in Table 3. The intensity of FTIR peak was related to the number of functional groups that existed in the synthesized geopolymer. The addition of foaming agent and aluminum powder increased the intensity of several functional groups and, consequently, the amount of soluble silica necessary for polymeric structure formation with amorphous aluminosilicate gel phase. Abdullah et al., obtained a FTIR spectrum of lightweight geopolymer with sharper peak, indicating the formation of a new product after the dissolution of the aluminosilicate in an alkaline environment [10].

Both types of lightweight geopolymers showed broad and sharp bands at wavenumbers of 3600 – 3000 and 1650 cm^{-1} related to stretching and bending vibrations of H-O-H, respectively. The presence of those vibrational peaks were related to the water molecules that were absorbed onto the surface or trapped inside the defect cavity of the lightweight geopolymer [17]. The larger the pores formed

on the geopolymer; the more water molecules would be absorbed into the pore structure of the geopolymer. Hence, the increasing intensity of H-O-H peak indicated by the development of the pore structure that results in a lighter structure. However, this would also, inevitably, cause the reduction of the compressive strength. The intensity of the broad peak at the wavenumber of 3600 – 3300 cm^{-1} decreased as the aluminum powder weight ratio was made higher than 0.01 %wt. This was the result of aluminum powder addition quickening the reaction that produced hydrogen gas leading to the increase of the collapsing pore structure.

3.3 The effect of pore-forming agent to geopolymer surface morphology

Scanning Electron Microscope (SEM) was used to observe the surface morphology of the geopolymer with and without the pore-forming agent addition as seen in Figure 3. All the geopolymer morphology images were taken with 2500x magnification. The surface morphology of fly ash geopolymer without the addition of the pore-forming agent displayed some rough parts, indicating unreacted fly ash spherical grain. Yunsheng, et al. found that unreacted particles could produce defects, thus reducing the geopolymer compressive strength. Apart from that, some fractional lines could also be observed in the images. This is related to the quick loss of water [18].

Geopolymer is an amorphous aluminosilicate polymer that possess some crystalline structure, such as quartz and mullite shown in XRD results. The presence

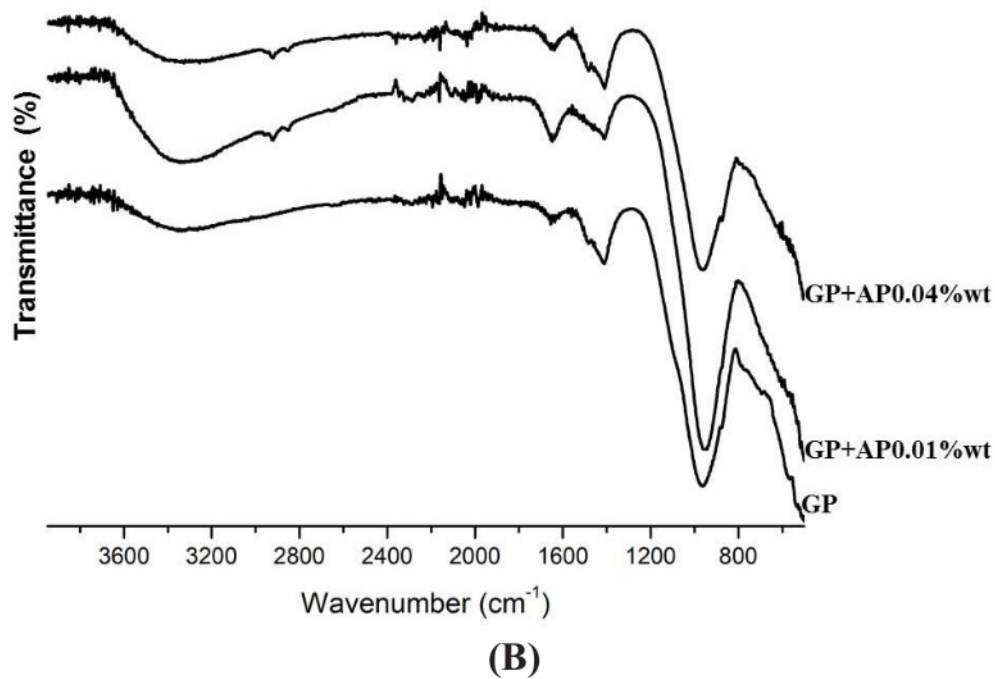
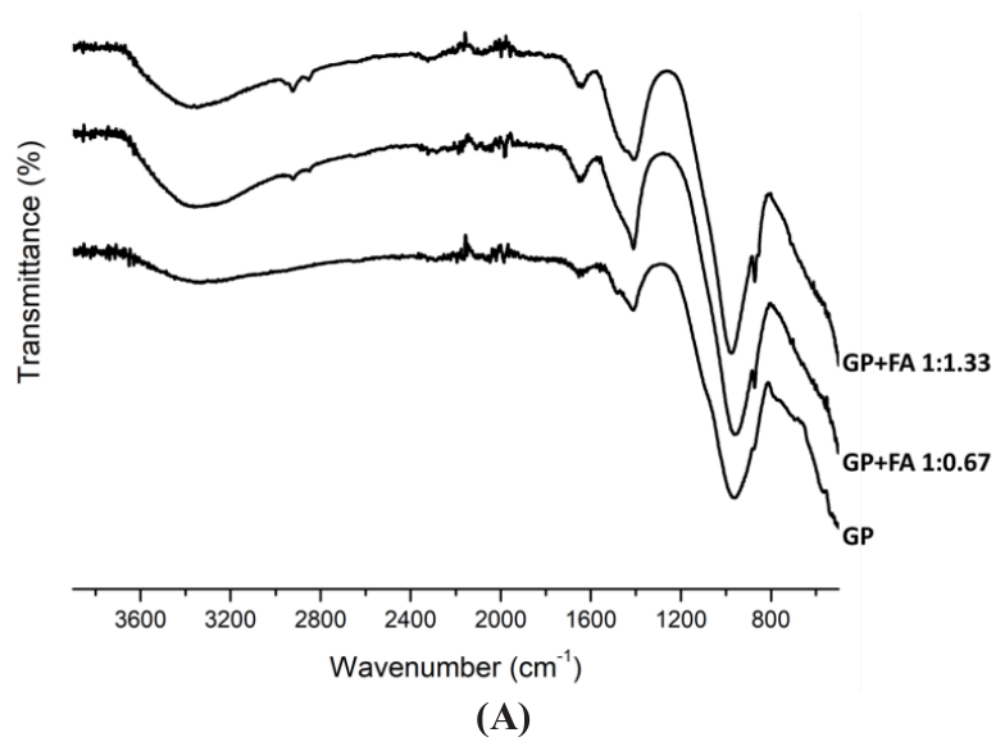


Figure 2: FTIR spectra of (A) lightweight geopolymer without and with foaming agent addition with ratio of 1.5:1 and 1.5:2; (B) lightweight geopolymer without and with aluminum powder addition with ratio of 0.01%wt and 0.02 %wt.

Table 3: Functional groups detected on lightweight geopolymer synthesized without and with pore-forming agent.

Absorption Peak (cm ⁻¹)	Types of Vibration	Intensity		
		Geopolymer (GP)	Foaming agent (FA) Lightweight Geopolymer	Aluminum Powder (AP) Lightweight Geopolymer
3600-3000	Stretching OH	low	high	high-medium
1650	Bending H-O-H	low	medium	medium
1400	Bending O-H	medium	high	medium
1000	Stretching Si-O-Si	high	high	high-medium
970	Bending Al-O-Si	high	high	high-medium
800	Stretching Si-O-T	medium	medium	medium
600	Bending O-Si-O	high	high	high

of this amorphous phase makes the reaction between aluminosilicate in raw materials with alkaline activator solution to form polymeric chains through polymerization possible. Furthermore, the presence of crystalline phases of quartz and mullite hinders some aluminosilicate components in fly ash from being involved in geopolymer formation. They are shown in the SEM image as some round-shaped particles.

The different ways on how bubbles developed and generated different sizes and foam distribution on geopolymer concrete were studied. In this study, the introduction of air bubbles was performed by executing the process of prefoaming which used the foam generator. After the production of air bubbles, the foaming agent was then introduced into the geopolymer paste and voids were consequently formed. The product produced through this kind of method was known as Cellular Lightweight Concrete (CLC). The surface morphology of lightweight geopolymer with low pore-forming agent, both foaming agent (FA) and aluminum powder (AP), additions showed less voids as shown in Figure 3(c) - (e). Contrary to that, bigger voids were observed on the surface when the geopolymer paste content was made lower. The higher number of pores generated on the geopolymer surface resulted in lower compressive strength. The pore distribution on the geopolymer surface also affected the density and compressive strength of the geopolymer.

In this study, aluminum powder was added into the geopolymer paste based on Non-Autoclaved Aerated Concrete (N-AAC) where the aluminum powder was mixed directly without undergoing the autoclave process. Generally, the aluminum powder lightweight geopolymer, as seen in Figure 3(f) - (h) possessed more voids. Increasing aluminum powder weight added into geopolymer paste

meant that the air voids produced were bigger. The voids produced when the aluminum powder was used resulted from the generated endogenous gas from the mixture of fine aluminum powder and geopolymer paste. Zhang et al. [15] stated that aluminum was a reactive metal that had reacted with water and hydroxide in the presence of alkaline, i.e. sodium hydroxide, to produce hydrogen gas and hydrolyzed metal complexes. The reaction stated on Eq. (2) took place when aluminum was added into the geopolymer paste.

When lightweight geopolymer was synthesized, aluminum powder as a pore-forming agent was added to a mixture of aluminosilicate source and alkaline activator solution that contains some water and sodium hydroxide (NaOH). Water as alkaline solid solvent and hydroxide ionic alkaline solution would react with aluminum powder to generate aluminum tetrahydroxide ion and hydrogen gas. The presence of hydrogen gas as a product of this reaction led to the formation of the pore structure and expanded the geopolymer paste, promoting higher chances of yielding a lighter material.



3.4 The effect of pore-foaming agent addition on the density and compressive strength of geopolymer

The density and maximum compressive strength of the unfoamed geopolymer were 2255 kg/m³ and 51 MPa after 28 days of curing, respectively. The density and compressive strength of all geopolymer specimens as

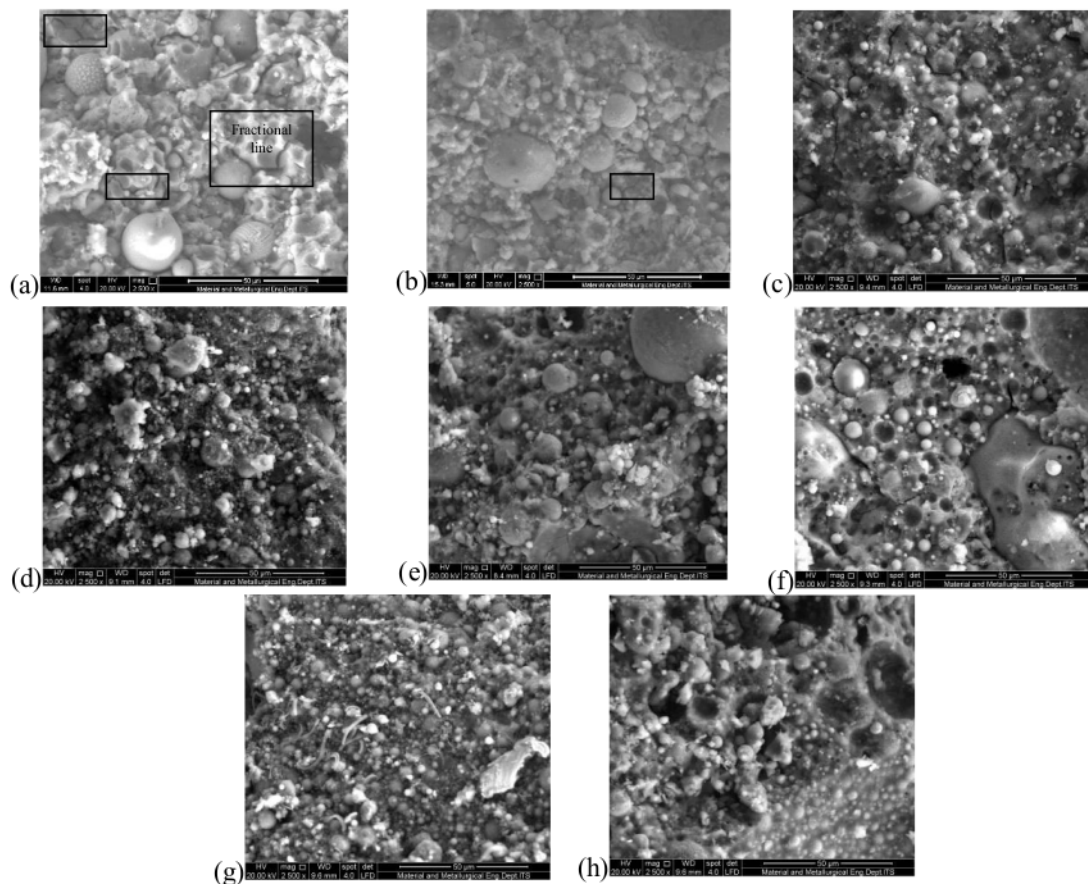


Figure 3: SEM of geopolymer sample for (a) surface geopolymer without pore-forming agent addition, (b) cross section of geopolymer - GP, (c) GP+FA 1:1.00, (d) GP+FA 1:0.67, (e) GP+FA 1:0.50, (f) GP+AP0.01 %wt, (g) GP+AP0.03 %wt and (h) GP+AP0.04 %wt.

shown in Table 4. The addition of foaming agents into the geopolymer paste promoted air bubble production that led to pore structure formation; hence, the density of the geopolymer matrix decreased. This, however, resulted in lower geopolymer compressive strength, weakening the formation of polymer cluster linkage and producing cracks of destruction. The foaming agent lightweight geopolymer exhibited the lowest density at the variation of GP+FA (1:2.00), with the lowest compressive strength of 0.2 MPa. Abdullah et al., used foaming agent to produce lightweight geopolymers with a compressive strength of 18.1 MPa after 28 days of curing [10]. In this study, when the ratio of the geopolymer paste to the foaming agent was made 1:1.33, the highest geopolymer compressive strength was observed to be 33 MPa with a density of 1766 kg/m³, while the addition of 0.01 %wt aluminum powder

resulted in the highest compressive strength of 42 MPa with a density of 1830 kg/m³. The foaming agent improves the pores formation within the geopolymer; thus, the more amount foaming agent used, the more pores will be formed. Hence, increasing the amount of foaming agent would decrease the geopolymer density since there will be more pores formed within the specimen itself. On the other hand, more pore formation would also result in less compressive strength of the geopolymer since the strength distribution will be non-uniform along the geopolymer surface.

This correlation could be applied to the aluminum powder lightweight geopolymer. While the weight percentage of aluminum powder was increased, both the density and compressive strength of the geopolymer decreased. The lowest density and compressive strength

Table 4: Dry density and compressive strength of unfoamed geopolymer (GP) and both types of lightweight geopolymer.

Specimen Number	Density (kg/m ³)	Compressive Strength (Mpa)		
		7 days	21 days	28 days
Geopolymer (GP)	2255	22.40	32.80	51.00
Foaming agent (FA) Lightweight Geopolymer				
GP + FA (1:0.50)	1990	6.80	12.40	15.40
GP + FA (1:0.67)	1969	8.20	9.60	12.60
GP + FA (1:0.75)	1842	12.80	13.00	8.00
GP + FA (1:1.00)	1341	2.20	6.54	9.60
GP + FA (1:1.33)	1766	10.00	16.00	33.00
GP + FA (1:1.50)	1040	1.00	1.20	4.00
GP + FA (1:2.00)	573	0.40	0.20	0.20
Aluminum Powder (AP) Lightweight Geopolymer				
GP + AP 0.01%wt	1830	5.00	16.00	42.00
GP + AP 0.02%wt	1815	11.00	19.00	20.00
GP + AP 0.03%wt	1373	11.80	17.00	19.20
GP + AP 0.04%wt	1424	8.20	10.00	10.00
GP + AP 0.05%wt	1965	7.20	8.80	16.00
GP + AP 0.10%wt	1093	3.72	3.80	3.80
GP + AP 0.15%wt	1031	3.04	3.20	4.60

were observed when 0.15 %wt aluminum powder was used. When the composition of the aluminum powder was higher than 0.01 %wt, the formation of hydrogen gas became faster, more pores are produced as the casting and gel hardened [19]. According to SNI 03-3449-2002, the minimum required compressive strength of the structural lightweight construction is 17.24 MPa with a maximum density of 1850 kg/m³. Based on this regulation, the density of the foaming agent lightweight geopolymer (1766 kg/m³) and aluminum powder lightweight geopolymer (1830 kg/m³) is near the requirements, hence they have the potential to be utilized as an alternative to structural binder construction.

4 Conclusion

Lightweight geopolymers were synthesized by using different pore-foaming agents, namely synthetic foaming agent and aluminum powder. XRD results showed that the addition of pore-forming agents would change the intensity of geopolymer peak, whereas the FTIR spectra

displayed that the increase of water molecules peak at lightweight geopolymer structures were related to the higher pore production that absorbs water. The morphology of lightweight geopolymer had some hollow parts, confirming the development of pore structures above the surface. The utilization of the foaming agent produced a lightweight geopolymer that has an optimum compressive strength of 33 MPa and a dry density of 1766 kg/m³, while the aluminum powder produced a binder structure with the maximum compressive strength of 42 MPa and dry density of 1830 kg/m³. It is recommended to study the enhancement of compressive strength and density for both types of lightweight geopolymer further.

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Conflict of interest: Authors declare no conflict of interest.

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