

PROPERTIES OF LIGHTWEIGHT AERATED GEOPOLYMER SYNTHESIS FROM HIGH-CALCIUM FLY ASH AND ALUMINIUM POWDER

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ABSTRACT: In the present work, efforts to decrease unit weights of the geopolymer mortar around 2,400 kilograms per cubic meter to be lightweight aerated geopolymer which density lower than 1,800 kilograms per cubic meter and study the properties of lightweight aerated geopolymer based on binders composed of high calcium fly ash, and the aluminum powder was studied. Compressive strengths and densities of lightweight geopolymers with aluminium powder contents of 0-0.20% wt, NaOH concentrations of 7.5-12.5 molar, liquid to fly ash ratios of 0.45-0.65, Na₂SiO₃ to NaOH ratios of 0.33-1.5, sand to fly ash ratio of 0.6-1.4, curing temperatures of 25-100 degree Celsius, and curing period of 1-24 hours. were tested. In addition, SEM and ultrasonic pulse velocity of lightweight aerated geopolymer were determined. Results showed that the lightweight aerated geopolymer high calcium fly ash and aluminum powder with the 28-day compressive strength of 1.2-12.6 MPa and densities of 770-1,560 kilogram per cubic meter, with satisfactory strength, could be made.

Keywords: Geopolymer, High Calcium fly ash, Aluminium powder, Lightweight aerated Geopolymer, Curing.

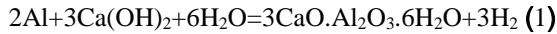
1. INTRODUCTION

The unit weight of normal concrete is 2,300 - 2,500 kg/m³ which contributes a large dead load on the building design. Lightweight concrete as shown in Table 1 has thus been invented to reduce the unit weight and in effect reduce the dead weight of the structure. Lightweight concrete with unit weight less than 1,800 kg/m³ [1] but still maintains the required compressive strength for various tasks. For example, lightweight structural concrete is defined as concrete with compressive strength more than 17.0 MPa and a 28- day air-dried unit weight not exceeding 1,850 kg/m³ [2]. Special structural lightweight concrete with a density of 1,725 kg/m³ and maximum compressive strength of 60.0 MPa has been reported [3]. Lightweight concrete can be used in roof decks and floors or lightweight concrete masonry block.

Table 1 Classification of concrete in accordance with unit weight [1]

Classification	Unit Weight (kg/m ³)
Ultra-lightweight concrete	<1200
Lightweight concrete	1200 < UW < 1800
Normal-weight concrete	~2400
Heavyweight concrete	>3200

Lightweight can be divided into three categories. The first type is no-fine concrete (NFC) consisting of cement, coarse aggregate, and water with no sand. The cement paste is coated around the coarse aggregate and provide point-to-point contact to leave interstitial voids. The second type is lightweight aggregate consisting of both coarse and fine lightweight aggregate for producing lesser unit weight concrete. Lightweight aggregates were drawn from 2 sources viz., natural materials such as pumice stone and burnt clay, and synthetic materials such as foam and plastic. The last type is aerated and foamed concrete with voids in the hardened cement paste or mortar matrix causing low-density. This type of concrete can be divided into two main types; foam concrete and aerated concrete. Foam concrete is obtained by mixing cement paste or mortar with stabilized foam. After hardening, the foam cells form concrete of a cellular structure [4]. Foam filling is divided into two processes; the first drawing foam from foam machine and injecting into concrete for mixing and second filling foaming agent into mortar admixture then mixing by the high shear mixer. Aerated concrete mostly related with this study since aerated or gas concrete can be mixed with or without aggregates which fizz within concrete by a chemical reaction between hydrate calcium oxide and aluminum following equation (1).



However, to obtain good quality aerated concrete, the products need autoclaving at a temperature of 175°C and a pressure of 8 atm [4]. The manufacturing thus requires high energy and also involves high budget in the assembly line.

However, the essential material for autoclaved aerated concrete is Portland cement which constitutes the cost and environmental considerations. The production of Portland cement in 2014 was around 4.3 billion tons [5]. The production of 1 ton of cement produces nearly 1 ton of CO² emission [6]. Geopolymer is an alternative binder with lower carbon dioxide emission than Portland cement with many similar or better qualities such as heat-resistant, high performance and chemical-resistant [6]. Besides good quality, a number of recycled waste materials can be used for producing geopolymer [7-9] with the implication that it is liable to be significant cementing material in the future. It is synthesized using starting material rich in silica and alumina and activated with a high alkali solution at normal room temperature or higher temperatures of 40-80 °f [10,11].

In the alkaline environment of geopolymer, the aluminum powder can react and form hydrogen [12,13]. The reaction between NaOH and aluminum is used to produce lightweight aerated geopolymer [14-16]. The present paper aims to produce lightweight aerated high calcium fly ash-based geopolymer mortars using aluminum powders. The density, compressive strength, microstructure and spatial distribution of void were studied. With the different amount of aluminum powder, NaOH concentration, liquid/fly ash ratio, Na₂SiO₃/NaOH ratio, sand/fly ash ratio, various curing temperatures and curing periods.

2. EXPERIMENTAL PROGRAM

2.1 Materials

The fly ash from Mae Moh power station in the north of Thailand was used as a starting material with basic qualities as shown in Table 2 and the chemical composition analyzed by X-ray fluorescence (XRF) technique as shown in Table 3. The chemical composition of 64.3% of SiO₂+Al₂O₃+Fe₂O₃ and 23% CaO indicate that it is a high calcium fly ash according to ASTM C 618 [18]. From the XRD analysis, it consists mainly of amorphous phase with some peaks of crystalline phases of magnetite, magnesioferrite, dachiardite, and calcium aluminum oxide as shown in Fig. 1. This lot of fly ash was rather fine with the Blaine fineness of 5,100 cm²/g. The fly ash particles are spherical with a smooth surface as shown in Fig. 2.

The aluminum powder with the minimum assay of 93.0 % is irregular in shape as shown in Fig.3. River sand passed sieve #50 (300 um) and retained on sieve #100 (150um), with basic properties as shown in Table 2 was used. The 7.5, 10, and 12.5 molars (M) NaOH solutions and sodium silicate (Na₂SiO₃) solution with 15.32% Na₂O, 32.87% SiO₂, and 51.8% water were used as alkali activators.

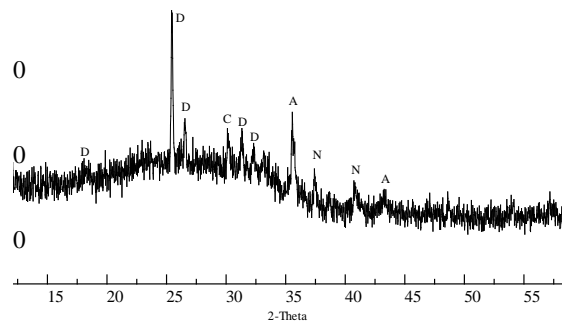


Fig. 1 XRD patterns of fly ash. A-Magnetite: Fe₃O₄;C-Magnesioferrite: Fe₂MgO₄;D-Dachiardite:Na1.1K.7Ca1.7Al₅.2Si1₈.8O₄₈ (H₂O)12.7; N-Calcium Aluminum Oxide: CaAl₂O₄.

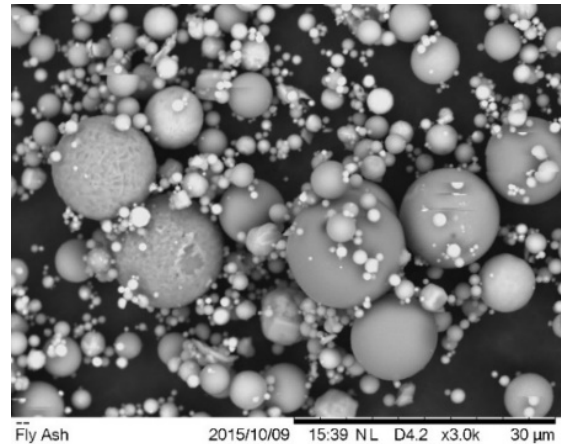


Fig. 2 SEM micrograph of fly ash powder

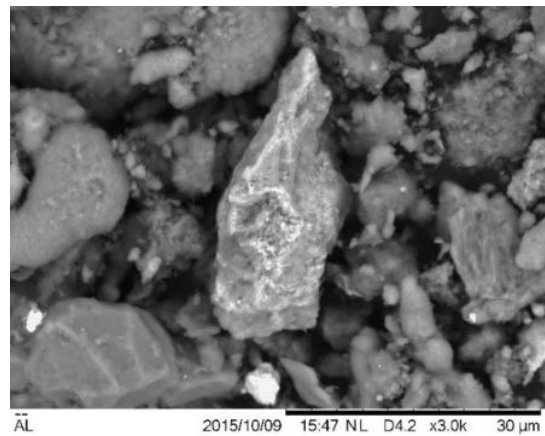


Fig. 3 SEM micrograph of aluminum metal fine powder

Table 1 Physical properties of materials

Materials	Fly ash	Sand
Specific gravity	2.49	-
Fineness by Blaine air permeability (cm ² /g)	5,166.00	-
Fineness modulus	-	1.00
Density (kg/m ³)	-	2.60
Water absorption (%)	-	3.63

Table 2 Chemical composition of Fly Ash (by weight)

Chemical composition	Content (wt. %)
SiO ₂	30.58
CaO	22.81
Al ₂ O ₃	17.10
Fe ₂ O ₃	16.60
SO ₃	5.64
MgO	2.24
K ₂ O	2.21
Na ₂ O	1.47
TiO ₂	0.53
P ₂ O ₅	0.21
BaO	0.15
SrO	0.14
MnO	0.12
L.O.I.	0.20

2.2 Mix Proportion

2.2.1 Mix compositions

From the preliminary test, a mix with NaOH concentration 10 molar, liquid to ash ratio (L/A) of 0.55, sodium silicate/ sodium hydroxy ratio (NS/NH) of 1.5, and/ash ratio (S/A) of 1, curing temperature of 40 C, curing period of 24 hr. and test age of 28days was used as the standard mix. Eight series of mixes were summarized in Table 4.

Table 3 Weight ratios of geopolymer lightweight mixes

Seri es	Variant	Unit
AL	0.0(A),0.05(B),0.1(F),0.15(J),0.2(K)	%
NH	7.5(E),10(F),12.5(G)	M
LA	0.45, 0.55(F),0.65	-
SH	3/2,3/3,2/3(F),1/3	-
SA	0.6(H),0.8,1.0(F),1.2,1.4(D)	-
CT	25(I),40(F),60,80,100(°C)	°C
CP	1,3,6,12,24(F)	hr.
DA	3,7,14,28	Day

Note: AL: Percent of Aluminium powder by weight of fly ash, NH: NaOH concentration, LA: Liquid alkaline/ash ratio, SH: Sodium silicate/NaOH ratio, SA: Sand/ash ratio, CT: Curing temperature, CP: curing period, DA: Age of sample

2.2.2 Details of mixing

The mixing process began with mixing fly ash and sodium hydroxide solution for three minutes. After that, sand was added, and mixing was continued for minutes. Sodium silicate and aluminum powder were then added and mixed for another 3 minutes. The fresh mortar was placed into 50 mm cube molds. The samples were left to expand for 30 minutes and leveled off the excess. The samples were left in a controlled 25 °C room for 24 hr. Finally, the specimens were demolded and wrapped with plastic sheet and stored in a 25°C and 50% RH controlled room.

2.2.3 Details of the test

The unit weight of aerated geopolymer was determined as described in ASTM C 138/C138M-14 [17]. The compressive strength was tested after the unit weight determination in accordance with ASTM C109/C109M-13 [18]. The report results are the average of three samples. The test of ultrasonic pulse velocity (UPV) was done in accordance with ASTM C597 [19]. The microstructure of the sample was studied with SEM.

3. RESULTS AND DISCUSSIONS

Fig.4 showed that Physical description of lightweight geopolymer when sample gets a hard status that clearly when adding more aluminum powder there was more pore on the sample surface.

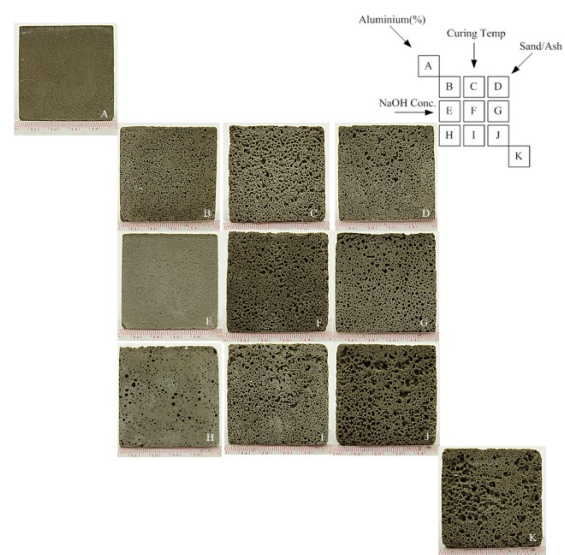


Fig.4 Physical description of lightweight geopolymer

3.1 Aluminium Powder Contents (Series AL)

The results of density and compressive strength of lightweight aerated geopolymers with various aluminum powder contents are shown in Fig. 5. The density and compressive strength decreased with the increasing aluminum powder contents as expected. The density of geopolymer was in the range of 750-1,950 kg/m³ with the corresponding strength range of 1.20-3.70 MPa. The 28-day compressive strength of samples with aluminium powder contents of 0, 0.05, 0.10, 0.15, and 2.0 % were 3.71, 2.84, 2.35, 1.73, and 1.22 MPa, respectively. The increase in aluminum powder contents increases the air bubbles [15] and thus reduce the strength of the sample which accord with SEM analysis in Fig.6. Shows structure deteriorated when more bubbles were added.

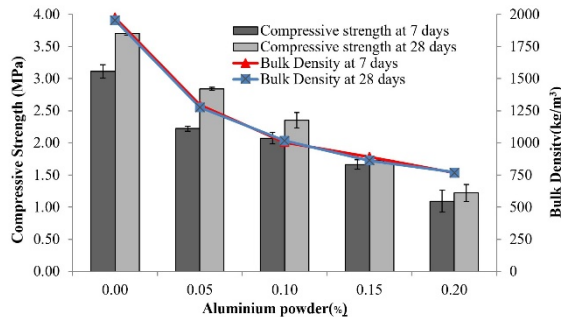


Fig. 5 Compressive strength and density at 7 and 28 days of lightweight geopolymer at various aluminum powder contents (Series AL)

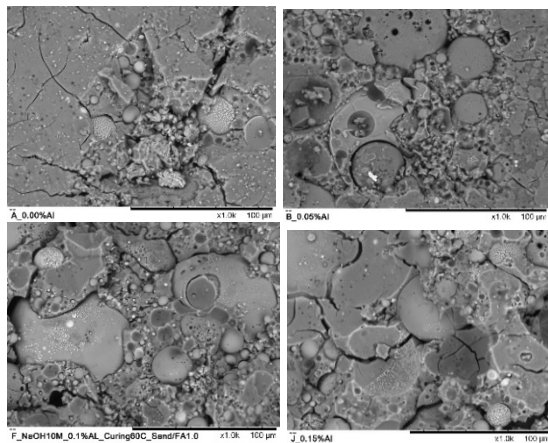


Fig.6 SEM of sample A, B, F and J Order from left to right and top to bottom, respectively.

3.2 NaOH Concentration (Series NH)

The results of the effects of NaOH solution concentrations on density and compressive

strengths of lightweight aerated geopolymer are shown in Fig. 7. The increase in NaOH concentration resulted in the reduction in density and compressive strength. For normal high calcium fly ash geopolymer, the increase in NaOH concentration enhanced the dissolution of fly ash [20], but the very high NaOH concentration resulted in the increased viscosity of the mix and the polycondensation was hindered [21]. The NaOH concentration for optimum strength is around 10 M. For the system with aluminum powder; the low 7.5 M NaOH resulted in high density and high strength mixture. The relatively high density was a result of the formation of a low amount of bubble. At low NaOH concentration, the setting time of geopolymer was rather fast due to the high calcium ion content. The fast setting characteristic resulted in the stiffening of the matrix and thus hindered the formation of a bubble. For the mix with high NaOH concentrations of 10 and 12.5 M, the leaching out of calcium ions was suppressed and the setting time was lengthened. The matrix was thus less stiff than that with low 7.5 M NaOH mix and the formation of the bubble could continue resulting in the lower density geopolymer and lower strength as well. After considered SEM Fig.8 found that the texture of sample E had most density but in sample G, the surface of fly ash able to react but not much in density like sample E and F

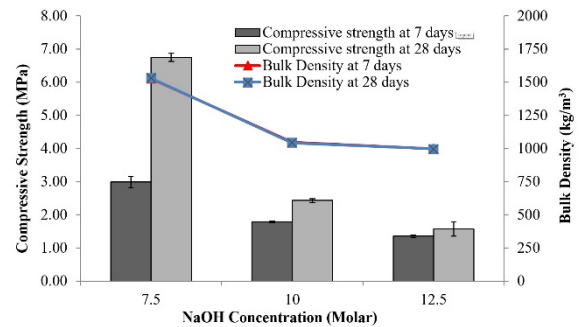


Fig. 7 Compressive strength and density at 7 and 28 days of lightweight geopolymer at various NaOH Concentration (Series NH)

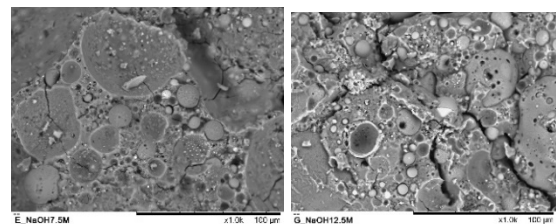


Fig.8 SEM of sample E and G Order from left to right and top to bottom, respectively.

3.3 Liquid Alkaline to Fly Ash Ratio (Series LA)

The results of density and compressive strength of lightweight aerated geopolymer with various liquid alkaline to fly ash ratios are shown in Fig. 9. The mix with L/A ratio of 0.55 had the lowest density of 1,100 kg/m³ and lowest compressive strength of 4.6 MPa. At low L/A ratio of 0.45, the workability of the mixture was low due to the low liquid in the mix. The formation of the bubble was hindered in this case as the paste was too stiff resulting in high density and slightly high strength geopolymer. Increase in the liquid portion to L/A ratio of 0.55 resulted in a mixture with adequate workability which allowed the bubble to form and resulted in the geopolymer with required low density. Further increase in the liquid portion to L/A ratio of 0.65 resulted in a high workability mix with low ability to retain the formed gas resulted in a high-density geopolymer. It is thus very important to use the proper amount of liquid to ash ratio to obtain an optimum mix with required low density with adequate strength.

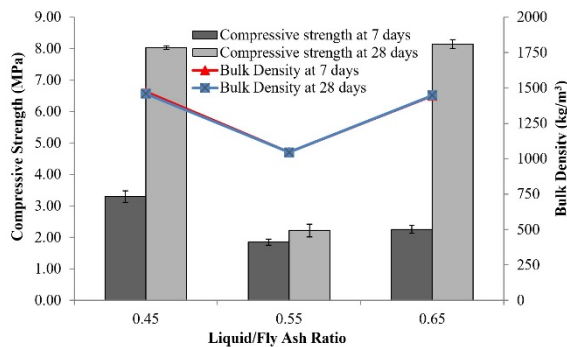


Fig. 9 Compressive strength and density at 7 and 28 days of lightweight geopolymer at various Liquid to Fly Ash Ratio (Series LA)

3.4 Na₂SiO₃ to NaOH (Series SH)

The result found that Na₂SiO₃ to NaOH plentifully affected to compressive strength and density. Even though 28-day optimum compressive strength that had 45 MPa was obtained with Na₂SiO₃ to NaOH ratios of 1.00 but density rate was over than 1,800kg/m³ and not regarded as lightweight aerated concrete. If consider in density, we might realize that Na₂SiO₃ to NaOH ratios reducing will cause lower viscosity since sodium silicate was more viscous than sodium hydroxide. Therefore, Na₂SiO₃ can keep more air bubbles [10] when Na₂SiO₃ to NaOH ratios decreased which showed in Fig.10, Na₂SiO₃ to NaOH ratios of 1/3,

found that compressive strength was reduced since the decreasing in strength in this range of Na₂SiO₃ to NaOH ratios was due to the silica content of mixture reducing [22]. Moreover, high NaOH did not cause higher SiO₂ and Al₂O₃ leaching [20] since SiO₂ and Al₂O₃ in Fly ash are limited but NaOH solution exceed will reduce compressive strength rate. A similar trend of the result was reported for the normal weight high calcium fly ash based-geopolymer mortars. The compressive strengths of mortars with high Na₂SiO₃ to NaOH ratios of 1.5 and 3.0 were reduced compared to that with Na₂SiO₃ to NaOH ratios of 1.0 [11] over OH filling will fizza a large number of air bubbles and reduce density rate [13].

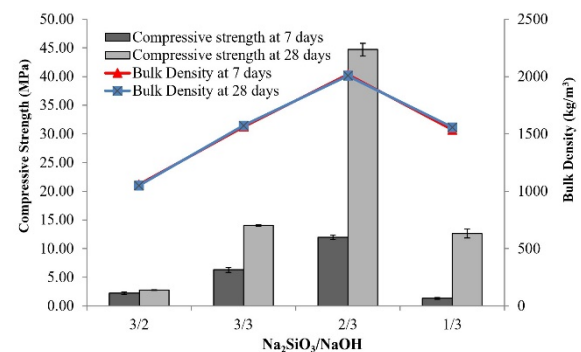


Fig. 10 Compressive strength and density at 7 and 28 days of lightweight geopolymer at various Na₂SiO₃ to NaOH (Series SH)

3.5 Sand to Fly Ash Ratio (Series SA)

The results of the effects of sand to fly ash ratio on compressive strength and density of lightweight aerated geopolymer are shown in Fig. 11. Sand to Fly ash ratio had an influence on the strength of geopolymer but the influence was rather small. The result showed that after filled sand into the compressive composition strength and density of lightweight aerated geopolymer rate will increase because of sand replacing air bubbles. After considering the results, sand to fly ash ratio more than 1 will not be beneficial to compressive strength and increase the sample density rate. From SEM Fig.12. found that texture paste of the sample H had highest homogeneous since air bubbles not kept within the sample. If we only applied the result from SEM, we might not explain the result of the low compressive strength of sample H, but after explained by SEM displays, the results that accorded with compressive strength were clearly shown. The sample that had a low rate of sand to fly ash ratio could not keep air bubbles like sample H

since most of the large bubbles will rise up because sand is the factor that increases sticky rate for fresh lightweight aerated geopolymer and compresses the bubbles in order to stop their floating as shown in Fig.6 and 12 (D, F, and H).

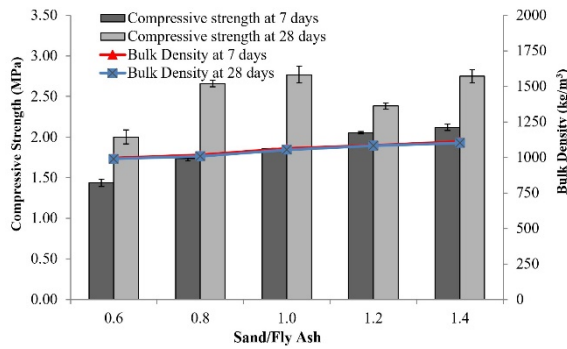


Fig. 11 Compressive strength and density at 7 and 28 days of lightweight geopolymer at various Sand to Fly ash ratio (Series SA)

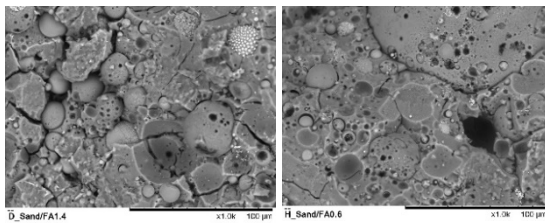


Fig.12 SEM of sample D and H Order from left to right, respectively.

3.6 Curing Temperature (Series CT)

The results of compressive strength and density of lightweight aerated geopolymer with various curing temperatures are shown in Fig.13. The optimum curing temperatures were 60 °C and the strength of 3.3 MPa. An increase in curing temperatures of 25 °C to 60 °C resulted to develop on compressive strength accorded with several research works that studied about an increase in temperature of curing to 60 °C enhanced the geopolymerization and increased the compressive strength [11], [23-25]. Even though temperature curing increasing is the factor that supports geopolymerization method but when the temperature reaches over 80 °C, the solution will rapidly evaporate, the reaction will not extremely occur, the sample will be dry, shrink and become to micro crack sample. In term of SEM analysis that showed in Fig.14, the compressive strength was plentifully reduced from temperature curing at 60 °C was 3.3 MPa to 1.8MPa at 80 °C decreasing as 45 % in order that temperature curing increasing

will reduce compressive strength as well. Moreover, the temperature that higher than 80 °C restrained the development of compressive strength and the sample's compressive strength tends to be reduced based on further ages. However, an increasing of curing temperatures reduced density rate but not much effective since the high-temperature curing caused watered solution lost their moistness more than curing at a low temperature, so the sample weight was decreased as well [11,26]. An interesting aspect of lightweight aerated geopolymer production is; autoclave treatment is not important for producing, on the other hand, OPC concrete production still needs autoclave [27,28] that will reduce the process, energy and production cost.

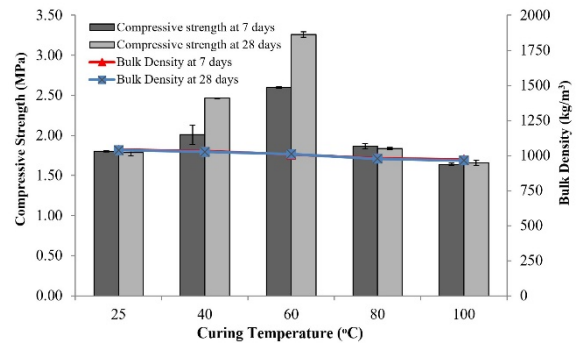


Fig. 13 Compressive strength and density at 7 and 28 days of lightweight geopolymer at the various curing temperature (Series CT)

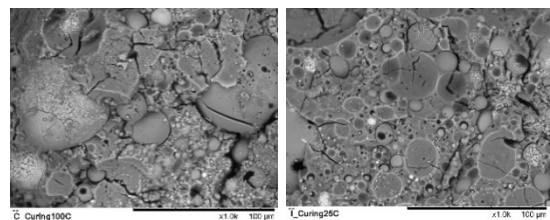


Fig.14 SEM of sample C and I Order from left to right, respectively.

3.7 Curing period (Series CP) and Age of the sample (Series DA)

The results of compressive strength and density of lightweight aerated geopolymer with various curing period are shown in Fig.15. Density had changed without significance with 1,055 Kg/m³ as its rate signified that the curing period could not affect to air bubbles fizzing or the sample evaporation when cured with 40 °C. However, an increasing curing period will increase the compressive strength rate. The results accorded with Görhan 2014 research which described, when

curing temperature of 65°C and increasing curing period from 2, 5 and 24 hours geopolymer that compounded with NaOH 9 M's compressive strength will increase to 13, 16 and 21 MPa, respectively. [29].

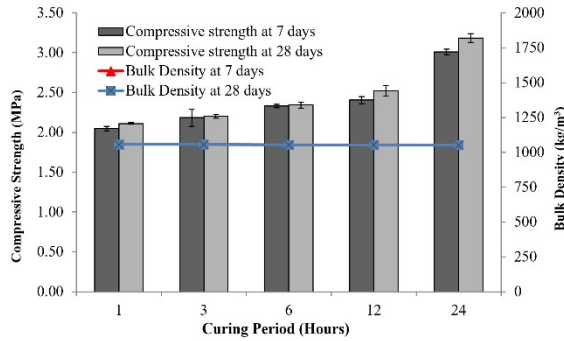


Fig. 15 Compressive strength and density at 7 and 28 days of lightweight geopolymer at various curing period (Series CP)

Fig.16 showed that when the sample aged compressive strength will continuously develop and density rate of the sample had a little change with average density rate as 1,058 kg/m³. However, in this test the sample was cured with 40 °C and the result accorded with Fernández-Jiménez 1999 research that studied about curing temperature of 45 °C [33] but cannot find out that lightweight aerated geopolymer will develop their compressive strength further aged in any cases. Since the relation of lightweight aerated geopolymer compressive strength development has to consider the main factor like curing temperature. According to the research, curing in high temperature will be beneficial to early compressive strength but it will reduce along with its ages [30] Moreover, if curing with relative humidity > 90%, the sample's compressive strength will continuously develop [31].

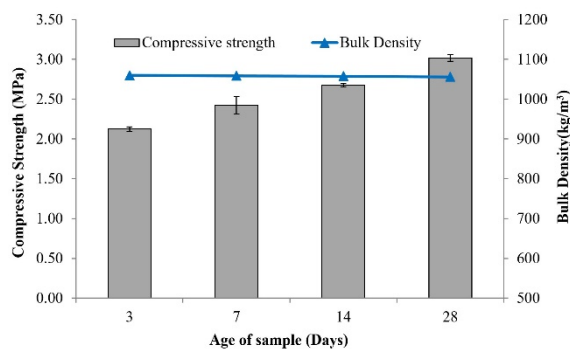


Fig. 16 Compressive strength and density of lightweight geopolymer age (DA)

4. CONCLUSIONS

Based on the obtained data, the following conclusions can be drawn.

1. Suitable Liquid to Fly Ash Ratio for lightweight aerated geopolymer production, lowest density should be 0.55.

2. An aluminum powder increasing increased H₂ bubbles and reduced sample's density and when the numbers of void increased compressive strength and its development will be reduced.

3. An increasing of NaOH Concentration make rapid and aggressive H₂ bubbles on the other hand high increasing NaOH Concentration generate large air bubbles but density not much reduced.

4. Na₂SiO₃ to NaOH have a major effect on the viscous of fresh specimens If there are high quantities the specimens will give a highly viscous, and also affect the setting time, therefore, air bubbles maintaining has to relate with setting time if setting time is late, the bubbles will rise up to the surface and density rate will increase.

5. Sand to Fly ash ratio not much affect to density rate and found that the suitable Sand to Fly ash ratio is 1.0

6. Curing temperature and curing period gave a little effect to density rate but in compressive strength, an increasing of curing temperature during 25- 60 °C will increase compressive strength on the other hand if curing with over 60 °C will harm to compressive strength and its development tends to be reduced.

7. The results showed that the lightweight aerated geopolymer High-Ca fly ash and aluminum powder with a 28-day compressive strength of 1.2-12.6 MPa and densities of 770-1,560 kg/m³ could be made.

5. ACKNOWLEDGMENTS

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