Properties And Microstructural Characteristics Of Lightweight Geopolymer Concrete With Fly Ash And Kaolin

Adji Sutama, Anis Saggaff, Saloma, Hanafiah

Abstract: Lightweight geopolymer is a new innovation in concrete industry. It has an advantage because it could act as geopolymer as well as lightweight concrete. It is environmentally friendly since it is not produced with Portland cement which normally emit CO_2 gas, a major contributor of greenhouse gases and global warming in the atmosphere. The material used in this experimental study are precursors, fine aggregates, alkaline activator solutions, superplasticizers, and foam. The precursors used were fly ash and kaolin, in which the percentage of kaolin were 0%, 5%, 10%, 15%, and 20% of the weight of fly ash. The concentration of NaOH at 12M and 14M. The results from the flow table test revealed that the optimum diameter of the mixture was 25.28 cm, which was found when the percentage of kaolin was 0% and the concentration of NaOH was 12M. The results of setting time showed that the fastest initial and final setting time of the mixture were 245 and 360 minutes respectively, also discovered when the percentage of kaolin was 0% and 12M NaOH. In addition, the optimum compressive strength of the mixture was 19.20 MPa, discovered when the density was 1481.12 kg/m^3, percentage of kaolin was 0% and at 14M NaOH. Then, the microstructure analysis of the mixture showed that increasing the concentration of NaOH without adding kaolin produced a dense geopolymer matrix with very small pores, however, when kaolin is increasingly added to the mixture, it produced a less dense matrix with large pores. In conclusion, mixtures without kaolin resulted in lightweight geopolymer with better properties and characteristics.

Index Terms: Lightweight geopolymer concrete, fly ash, kaolin, concentration of NaOH.

1 Introduction

Lightweight concrete usually have lower density compared with the conventional ones. Its density ranges from 300 to 1850 kg/m^3, while that of the conventional concrete is between 2200 and 2600 kg/m^3 [1]. Lightweight concrete has become more popular in the recent years due to its low density, which simplifies the construction process thereby reducing the weight of the building, its lower production cost as well as its low thermal conductivity [1]. Considering the fact that Indonesia, as a country, is prone to earthquakes, it is necessary to make buildings lighter. Therefore, lightweight concrete is perfect for this purpose. This helps to reduce the impact of the force caused by earthquakes on buildings. A commonly used lightweight concrete is the foam, usually produced by mixing foaming agents and water. Foam can be produced using two methods, the pre-formed and the mixed-foaming method [2]. However, the pre-formed is more commonly used, because it does not require much foaming agents and there is a measurable foaming agent/water ratio needed in producing it [3]. Raupit et al. [4] reported that stable foam can be produced through generator at an air pressure of 0.5 MPa. The foam stability factor is around 0.5 to 2 hours depending on the ambient temperature and the target density of the concrete based on the amount of foam needed [5].

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These foam concrete are still being produced with Portland cement but other precursors, which are environmentally friendly could be used instead, in order to minimize the emission of CO_2 into the atmosphere. Geopolymer was first used as a term in 1978 when Professor Joseph Davidovits was making concrete with inorganic binder materials like polymeric alumina, also known as geopolymer. And Davidovits [6] reported that geopolymers are industrial by-products or geological materials rich in silica and alumina and are found in nature like fly ash and kaolin. Also, Sumajouw and Dapas [7] reported that geopolymer binder is made of solid components rich in SiO_2 and Al_2O_3 like fly ash, kaolin, slag, as well as alkaline activator solutions. The mixing of the components leads to a hardening process caused by the formation of aluminosilicate networks which vary between amorphous and crystalline state [7]. The alkaline activator commonly used is a combination of sodium or potassium hydroxide solution with sodium or potassium silicate. Sodium solution is widely used due to the fact that it is cheaper and when mixed with NaOH and Na_2SiO_3 solution, it gives mixtures with high compressive strength [8]. The effect of the concentration of NaOH on lightweight geopolymers was examined, and the ratios of fly ash/alkaline activator, Na_2SiO_3/NaOH solution, foaming agent/water, and foam/geopolymer paste were kept constant at 2.0, 2.5, 1:10, and 1:1 respectively while NaOH concentrations used were 6M, 8M, 10M, 12M, and 14M [9]. The results revealed that the optimum concentration of NaOH was 12M, which produced the highest compressive strength of 15.6 MPa and a low density of 1415 kg/m^3. These results were applied in making light concretes [9]. In addition, the compressive strength and density of the lightweight concrete were examined with the ratios of fly ash/alkaline activator, Na_2SiO_3/NaOH solution, foaming agent/water, and foam/geopolymer paste kept at 2:1, 2.5:1, 1:20, and 1:2 respectively [10]. A lightweight concrete with heat curing of 60°C produced maximum compressive strength of 11 MPa, 17.6 MPa, and 18.2 MPa at days 1, 7, and 28 respectively, with density of 1667 kg/m^3 [10]. The effect of temperature on lightweight geopolymer concrete based on fly ash was also examined using a foaming agent with the ratio of fly ash/alkaline activator, Na_2SiO_3/NaOH solution, foaming agent/water, and foam/geopolymer paste kept at 1:1, 1:2, and 1:3 respectively [11]. According to the results, the maximum compressive strength is in the sample (LC3) at room temperature and sample (LC4) at an oven temperature 60°C measured, revealed that LC4 showed higher compressive strength compared to LC3. This is an indication that heat curing is needed to accelerate geopolymerization process [11]. Also, Zhang and Wang [12] showed that curing with high or low temperatures can cause slight changes in the volume of the concrete produced. Also, the microstructural characteristics of the mixture was examined with the percentage of kaolin at 10%, 20%, 30%, 40%, and 50% of the fly ash weight [13]. The concentration of NaOH used was 12M and the solid to liquid ratio used was 2 while that of Na_2SiO_3/NaOH was 2.5. The results showed that samples with greater percentage of kaolin have lower compressive strength at days 7 and 28, however, that of day 28 was greater than day 7. This is due to the fact the pores on the surface affect the paste bond and the compressive strength of the sample [13].

2 EXPERIMENTAL PROCEDURES

2.1 MATERIAL

The materials used in this experimental study are precursors, fine aggregates, alkaline activator solutions, superplasticizers, and foam. The precursors used are fly ash and kaolin. The fly ash is filtered with 200 filter size before using it. Tables 1 and 2 show the chemical compositions of fly ash and kaolin as revealed by X-Ray Fluorescence (XRF), the microstructures as revealed through Scanning Electron Microscope (SEM) are shown in Figs. 1 and 2 while the X-Ray Diffraction (XRD) patterns are shown in Figs. 3 and 4. According to Tables 1 and 2, silica (SiO_2) and alumina (Al_2O_3) are more present in both fly ash and kaolin, hence, could be used in geopolymerization. Class F and N fly ash and kaolin as designated in ASTM C 618 [14] are the type used for the concrete in this study. Considering Figs. 1 and 2, the particles of fly ash are spherical with different diameters and irregular shape at 1500x magnification, while that of kaolin are hexagonal in shape and plate-like structures at 5000x magnification. The spherical shape of fly ash, makes it better during concrete mixing compared with plate-like structure of kaolin [13].

Table 1. *Chemical composition of fly ash*

No.	Chemical composition	Contents (%)	ASTM C 618 class F (%)	
1.	SiO_2	50.07	min. (1+2+3)	70
2.	Al_2O_3	30.41		
3.	Fe_2O_3	4.28		
4.	SO_3	0.348	max.	5
5.	TiO_2	0.811		-
6.	CaO	4.12	max.	10
7.	MgO	2.41		-
8.	K_2O	0.778		-
9.	Na_2O	4.88		-
10.	MnO	0.0646		-
11.	P_2O_5	0.266		-
12.	LOI	1.28	max.	6

Table 2. *Chemical composition of kaolin*

No.	Chemical Composition	Contents (%)	ASTM C 618 class N (%)	
1.	SiO_2	48.73	min. (1+2+3)	70
2.	Al_2O_3	34.49		
3.	Fe_2O_3	1.05		
4.	SO_3	0.0126	max.	4
5.	TiO_2	0.504		-
6.	CaO	0.0395		-
7.	MgO	0.207		-
8.	K_2O	1.61		-
9.	Na_2O	0.0285		-
10.	MnO	-		-
11.	P_2O_5	-		-
12.	LOI	13.17	max.	10

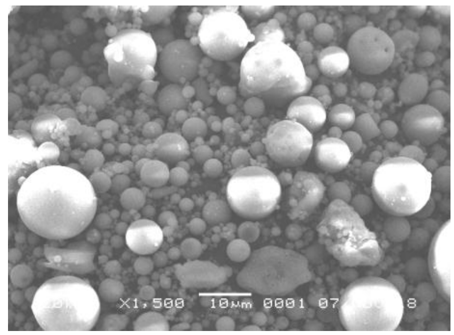

Fig. 1. *The microstructure of fly ash*

According to Figs. 3 and 4 above, the fly ash is made of quartz mineral (SiO_2), sillimanite (Al_2O_5Si), and moissanite (SiC), while kaolin consists of kaolinite ($Al_2H_4O_9Si_2$), illite ($Al_3H_2KO_{12}Si_3$), muscovite ($Al_3H_2KO_{12}Si_3$), halloysite ($Al_2H_4O_9Si_2$), and anatase (TiO_2). And all these mineral compositions are in the crystalline phase, in which fly ash are fewer at position 2θ = 21°, 27°, 36°, 37°, 41°, 43°, 50°, 60°, and 68°; but kaolin on the other hand, are dominant at position 2θ = 9°, 12°, 18°, 20°, 21°, 23°, 24°, 25°, 27°, 28°, 30°, 33°, 35°, 36°, 38°, 39°, 41°, 42°, 45°, 47°, 48°, 51°, 55°, and 57°. Therefore, to reactivate the crystalline phase of both precursors, different concentrations of NaOH were used. This plays an important role in the production of geopolymer material because the solubility of aluminosilicate in an alkaline activator solutions is highly dependent on hydroxide concentration [9].

Fig. 2. *The microstructure of kaolin*

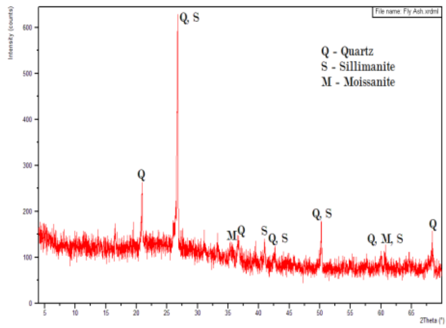

Fig. 3. *X-ray diffraction pattern of fly ash*

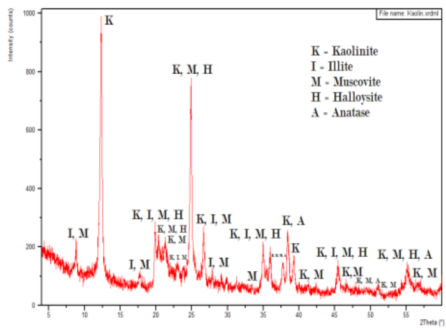

Fig. 4. *X-ray diffraction pattern of kaolin*

The fine aggregate used was river sand which has a fine modulus of 2.467 and specific gravity of 2.604. The fine aggregate need to be filtered using filter No. 16 under saturated surface dry conditions before being used [3]. The alkaline activator used was a mixture of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) solution. The NaOH was in the form of flakes with 98% purity which was prepared one day before use by dissolving 480 grams of the flakes in 1 liter of aquadest to produce 12M NaOH and 560 grams in the same volume of aquadest to produce 14M NaOH. The Na_2SiO_3 solution used was a very thick and colorless liquid, the superplasticizer improved the workability of the geopolymer concrete, and the foaming agents used are those produced with surfactant.

2.2 The Mixed Design and Mixing Process

The mixed design employed in this study is based on results from previous studies as shown in Table 3. The percentages of kaolin used were 0%, 5%, 10%, 15%, and 20% of the fly ash weight. The ratios of fine aggregate/precursor, Na_2SiO_3/NaOH solution were 2:1 and 2.5:1 respectively, with the concentration of NaOH at 12M and 14M. The ratio of the alkaline activator/precursor was 0.5 and the superplasticizer used was 3% of the precursor weight. The ratio of the foaming agent/water to produce foam was 1:40 with the percentage of foam 50% of the mixed volume, all these were subjected to an oven at 60°C for 24 hours.

Table 3. *Mixed design of lightweight geopolymer concrete (m^3)*

LGC	FAs (kg)	K (kg)	FA (kg)	SS (kg)	SH (kg)	Sp (kg)	F (L)
0%-12M	320	0	640	114	46	10	500
0%-14M	320	0	640	114	46	10	500
5%-12M	304	16	640	114	46	10	500
5%-14M	304	16	640	114	46	10	500
10%-12M	288	32	640	114	46	10	500
10%-14M	288	32	640	114	46	10	500
15%-12M	272	48	640	114	46	10	500
15%-14M	272	48	640	114	46	10	500
20%-12M	256	64	640	114	46	10	500
20%-14M	256	64	640	114	46	10	500

Note: LGC = lightweight geopolymer concrete, FAs = fly ash, K = kaolin, FA = fine aggregate, SS = sodium silicate solution, SH = sodium hydroxide solution, Sp = superplasticizer, and F = foam.

The proper mixing process started by adding Na_2SiO_3 into NaOH solution while stirring and then allowed to cool. The fly ash, kaolin, and fine aggregate were weighed into a mixer bowl and mixed for about 5 minutes and the alkaline activator solutions was added while stirring until it was evenly mixed after which the superplasticizer was added, the mixing process of liquid material above for about 5 minutes. The foam was produced by mixing 25 mL of its agent with 1000 mL of water in a tube at an air pressure of 0.5 MPa for about 2 minutes, after which it was added into the concrete mixture while stirring for another 2 minutes. It was then allowed to set and poured into 50 mm side cube molds and cured in an oven at 60°C for 24 hours. This is wrapped in a plastic material to minimize the evaporation of water from it.

2.3 Testing

The first microstructure test conducted on fly ash and kaolin was the X-Ray Fluorescence (XRF) testing using Thermo Scientific ARL 9900, then the Scanning Electron Microscope (SEM) testing using JEOL JSM-6360LA, and lastly, the X-Ray Diffraction (XRD) testing using the PANalytical X'Pert PRO series PW3040/x0 X'Pert PRO. The test conducted with the fresh concrete was with the flow table in compliance with ASTM C 1437 [15], the setting time in accordance to ASTM C 191 [16], while that of the hard concrete involving density and compressive strength was in compliance with ASTM C 109 [17] using the automatic compression controls equipment. Each mixture was made into five cube specimens and tested at 28 days. Those samples tested for compressive strength were taken for microstructure testing. The sample used for microstructure testing is in the form of small flakes with the highest concrete compressive strength.

3 Results and Discussion

3.1 Flow Table

According to the flow table test results shown in Figs. 5 and 6, the diameter of the fresh concrete ranged from 25 to 17 cm. Also, the fresh concrete optimum diameter was 25.28 cm at 0% kaolin and 12M NaOH.

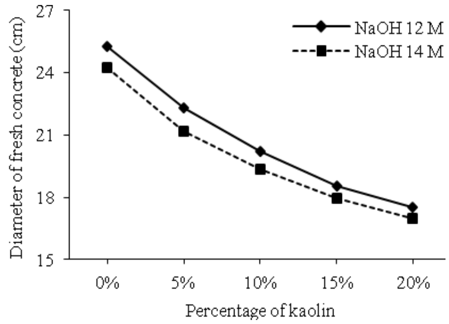

Fig. 5. *Effect of kaolin percentage on flow table test*

Considering Fig. 5, the higher the percentage of kaolin used, the smaller the diameter of the fresh concrete and the harder its workability. High kaolin content of geopolymer concrete has the tendency to make the mixture thicker [18]. Also, Ghais et al. [19] reported that concrete properties are far better with fly ash compared with the addition of kaolin.

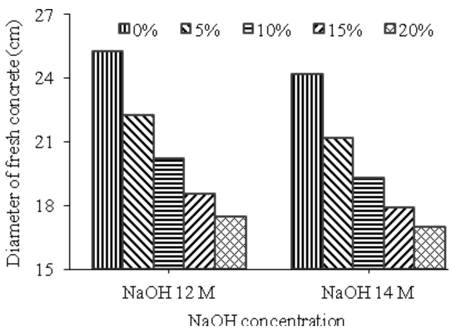

Fig. 6. *Effect of NaOH concentration on flow table test*

According to Fig. 6, the higher the concentration of NaOH, the thicker or the more cohesive the concrete mixture becomes, thereby reducing its workability [20]. A research work that provided the reasons for this was conducted by Arafa et al. [21] and according to it, an increase in the concentration of NaOH releases larger silica and alumina compounds, thereby increasing its geopolymerization and stiffness.

3.2 Setting Time

Figs. 7 and 8 show the initial and final setting time based on the percentage of kaolin while the setting time based on NaOH concentration are shown in Figs. 9 and 10. The initial setting time ranged from 245 to 395 minutes, while the final setting time ranged from 360 to 630 minutes. According to the results, the fastest initial and final setting time of the mixture are 245 and 360 minutes respectively at 0% kaolin and 12M NaOH.

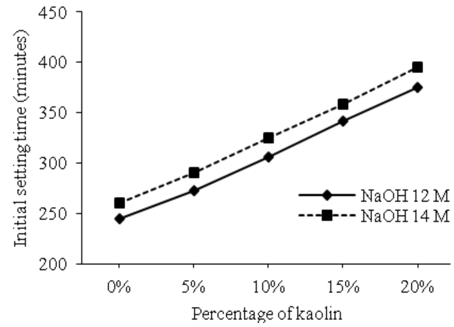

Fig. 7. *Effect of kaolin percentage on initial setting time*

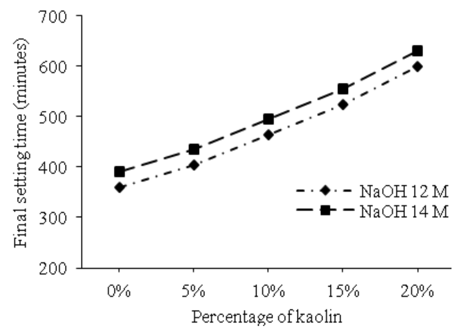

Fig. 8. *Effect of kaolin percentage on final setting time*

Figs. 7 and 8 are in accordance with the research conducted by Yahya et al. [18] which stated that the higher the percentage of kaolin in lightweight geopolymer concrete, the longer the initial and final setting time. According to Figs. 9 and 10, the initial and final setting time increase with increasing NaOH concentration. A research conducted by Phoo-ngernkham et al. [22] provided the reason for this process, and according to it, higher concentration of NaOH release more Si^{4+} and Al^{3+} ions and block more of Ca^{2+} ions. Also, Risdanareni et al. [23] reported that the higher the amount of CaO in the mixture, the shorter the setting time and the higher the compressive strength.

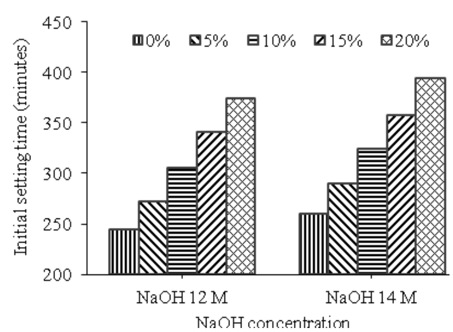

Fig. 9. *Effect of NaOH concentration on initial setting time*

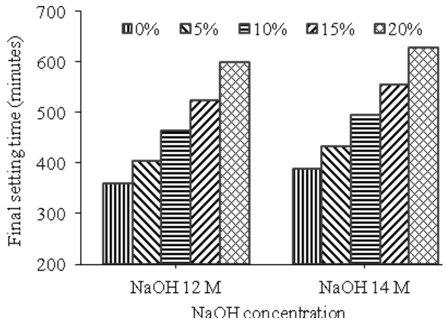

Fig. 10. *Effect of NaOH concentration on final setting time*

3.3 Density

The test involving the density of the mixture was conducted by weighing 50 mm cube sample at day 28 with the target density in the range 1200 to 1700 kg/m^3. The results are shown in Figs. 11 and 12. Fig. 11 shows that the mixed components achieved the target density between 1200 and 1700 kg/m^3. Also, the higher the percentage of kaolin used, the lower the density of the concrete formed because of the pores formed. Bakri et al. [13] reported that kaolin-based geopolymers are different from that of fly ash because they contain much larger pores. According to Fig. 12, the higher the concentration of NaOH used, the higher the density of the concrete. Ibrahim et al. [9] gave the reason for this process, higher concentration of NaOH releases larger silica and alumina compounds, thereby resulting in concrete with higher density and compressive strength.

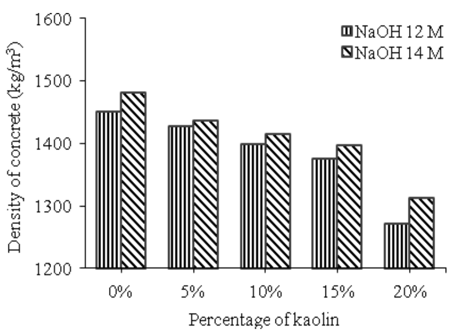

Fig. 11. *Effect of kaolin percentage on density*

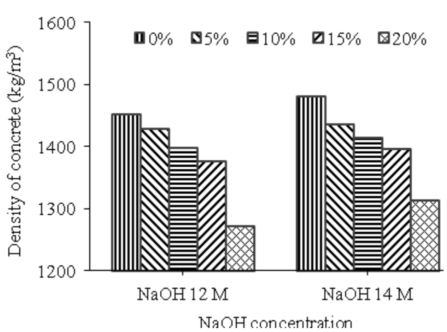

Fig. 12. *Effect of NaOH concentration on density*

3.4 Compressive Strength

The compressive strength test results at day 28 are shown in Figs. 13 and 14. The optimum of 19.20 MPa was found in the mixture with 0% kaolin, 14M NaOH, and density of 1481.12 kg/m^3. Considering Fig. 13, the higher the percentage of kaolin in the mixture, the lower its compressive strength. This is caused by the formation of pores on the sample surface [9] and the sticky or cohesive nature of kaolin. However, the addition of fly ash gives concrete with better compressive strength [19].

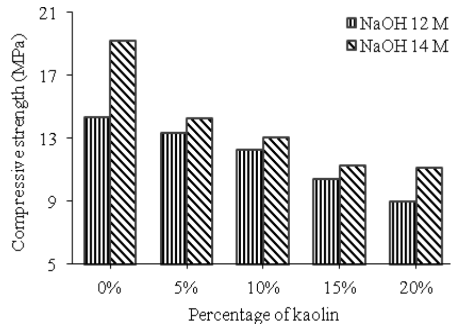

Fig. 13. *Effect of kaolin percentage on compressive strength*

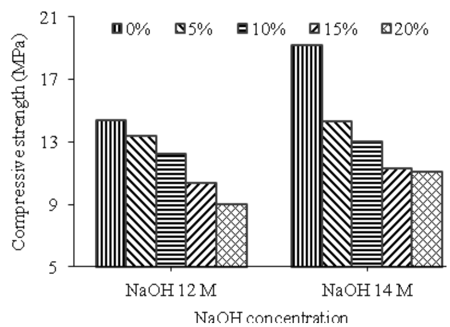

Fig. 14. *Effect of NaOH concentration on compressive strength*

The loss on ignition (LoI) test can be used to know the geopolymerization potential of the source material. The test shows if the carbon constituents of a material have not been burned, which have the capacity to absorb the alkaline activator, whenever a large amount of alkaline activator is needed, it impacts negatively on the mixture [24]. And the chemical compositions of kaolin due to LOI exceed the conditions specified by ASTM C 618 [14] which gives a lower compressive strength, as shown in Table 2. According to Fig. 14, the higher the concentration of NaOH, the greater the compressive strength of the concrete. This is caused as a result of the chemical reactions of Si and Al present in both precursor materials and by the high alkalinity state of the mixture. Also, Ibrahim et al. [9] reported that the higher the concentration of the alkali, the higher the compressive strength of the mixture.

3.5 Microstructure Test Results

The microstructure test results in each of lightweight geopolymer concrete mixtures are shown in Figs. 15 to 24 at a magnification of 2000x. There are unreacted fly ash particles mixed with kaolin which produce less dense geopolymer matrix in each mixture. Also, the pores are of different sizes, though less than 10 μm, and appear more in mixtures with kaolin, which in turn affects the density and compressive strength of the concrete. Abdullah et al. [10] reported that mixtures with kaolin have more microcracks and lower compressive strength.

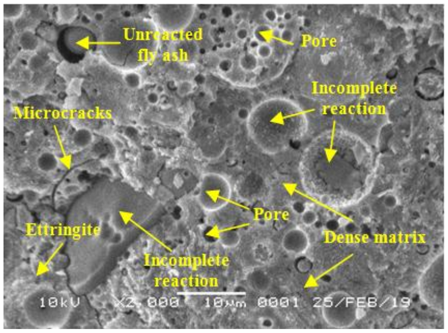

Fig. 15. *Microstructure test results on mixed LGC at 0% kaolin and 12M NaOH*

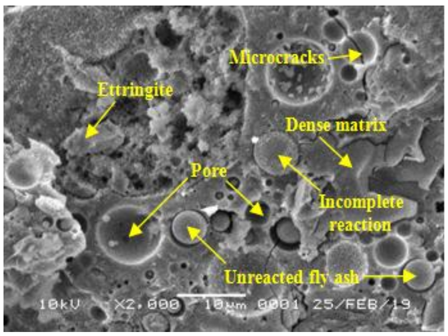

Fig. 16. *Microstructure test results on mixed LGC at 0% kaolin and 14M NaOH*

According to Figs. 15 and 16, increasing the concentration of NaOH without kaolin gives small pores or microcracks, and small amounts of ettringite, but produces geopolymer with dense matrix as a result of the complete reaction between the fly ash and alkaline activator, results in greater density and compressive strength [9]. However, Abdullah et al. [10] stated that some could also result into incomplete reactions, thereby forming a less dense matrix, if the fly ash surface is covered with aluminosilicate gel.

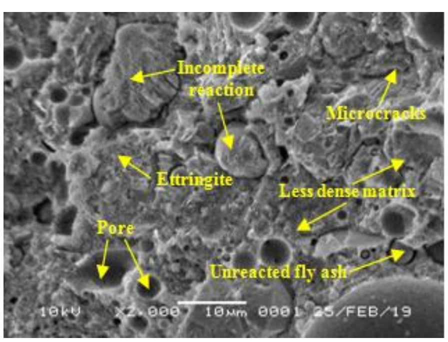

Fig. 17. *Microstructure test results on mixed LGC at 5% kaolin and 12M NaOH*

Fig. 18. *Microstructure test results on mixed LGC at 5% kaolin and 14M NaOH*

Figs. 17 to 24 show that mixtures with larger pores are produced with the addition of kaolin, which then result in less dense geopolymer matrix with low density and compressive strength. Also, the microcracks are caused by dehydration of the concrete mixture during the curing process.

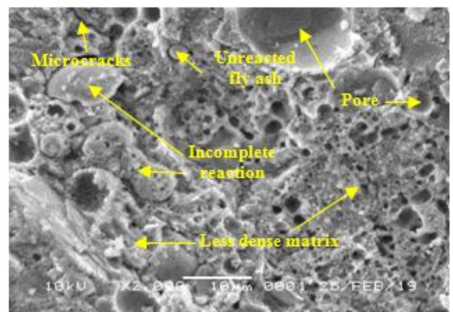

Fig. 19. *Microstructure test results on mixed LGC at 10% kaolin and 12M NaOH*

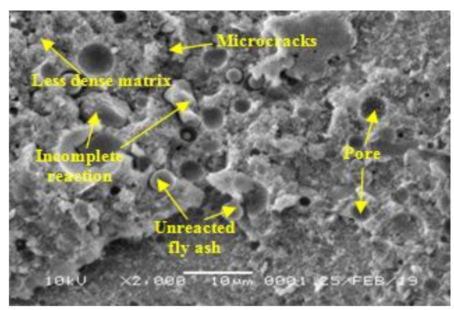

Fig. 20. *Microstructure test results on mixed LGC at 10% kaolin and 14M NaOH*

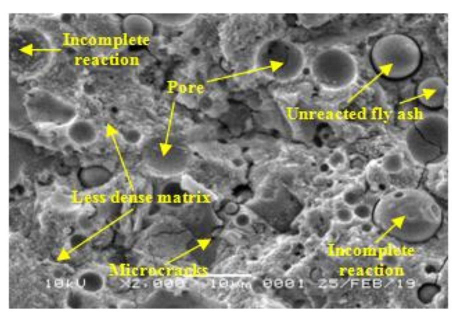

Fig. 21. *Microstructure test results on mixed LGC at 15% kaolin and 12M NaOH*

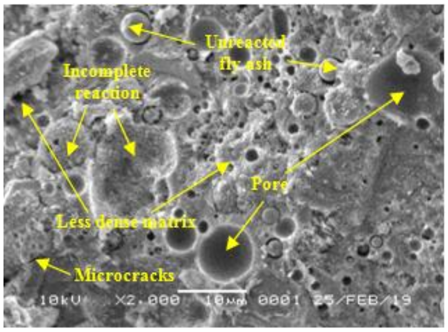

Fig. 22. *Microstructure test results on mixed LGC at 15% kaolin and 14M NaOH*

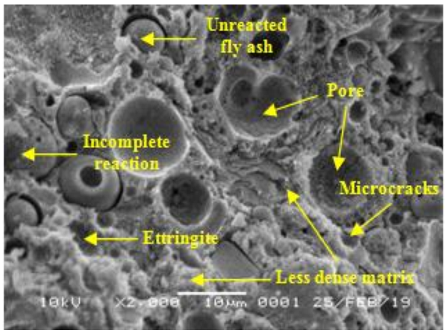

Fig. 23. *Microstructure test results on mixed LGC at 20% kaolin and 12M NaOH*

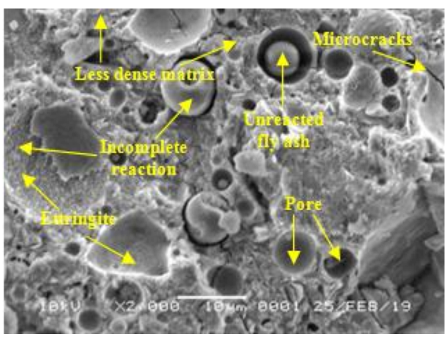

Fig. 24. *Microstructure test results on mixed LGC at 20% kaolin and 14M NaOH*

4 CONCLUSION

Considering the results of this experimental research, the authors conclude as follows:

1. The optimum lightweight geopolymer concrete was found in the mixture with 0% kaolin and 14M NaOH. This can be applied in non-structural materials like precast or brick walls.
2. According to the flow table test, the optimum diameter of the mixture was 25.28 cm, and this was found in the mixture with 0% kaolin and 12M NaOH. Also, the higher the percentage of kaolin and the concentration of NaOH added, the smaller the diameter of the fresh concrete produced and hence, the lower its workability. Then, the setting time test showed that the initial and final setting time were 245 and 360 minutes respectively, found in the mixture with 0% kaolin and 12M NaOH. Therefore, the higher the percentage of kaolin and the concentration of NaOH used, the longer the setting time.
3. The optimum compressive strength of 19.20 MPa was found in the mixture with 0% kaolin and 14M NaOH with a density of 1481.12 kg/m^3. Therefore, the higher the concentration of NaOH used, the higher the compressive strength of the mixture. However, the higher the percentage of kaolin used, the lower the compressive strength.
4. According to the microstructure test, the higher the concentration of NaOH without the adding kaolin, the smaller the pores or microcracks produced. Also, an increase in NaOH concentration resulted in a dense matrix due to the complete reaction between fly ash and alkaline activator solutions, which produced mixture with greater density and compressive strength. However, increasing the percentage of kaolin produced less dense matrix with larger pores and low compressive strength.

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