Available online at www.sciencedirect.com

ScienceDirect

Procedia Engineering 171 (2017) 572 – 583

Procedia Engineering

www.elsevier.com/locate/procedia

Sustainable Civil Engineering Structures and Construction Materials, SCESCM 2016

Effect of curing temperature and fiber on metakaolin-based geopolymer

Januarti Jaya Ekaputri[a,c,] *, Subaer Junaedi[b,c], Wijaya[c]

[a]*Institut Teknologi Sepuluh Nopember, Surabaya 60111, Indonesia*
[b]*Universitas Negeri Makassar, Indonesia*
[c]*Konsorsim Riset Geopolimer Indonesia, Surabaya 60111, Indonesia*

Abstract

This paper presents mechanical properties of geopolymer mortar with metakaolin as a base material. Cylindrical specimens were prepared with a diameter of 5 cm and 10 cm height. Four compositions of geopolymer paste varied with adding polyvinyl alcohol fiber (PVA) from 0% to 1% by volume of paste. After casting, steam curing method was conducted at 40ºC, 60ºC and 80ºC for 24 hours. A control specimen was cured at room temperature. Some tests were performed for setting time, compressive strength, split-tensile, direct-tensile and porosity. It showed that the strength of fibrous specimens was 67.29 MPa at of 56 days without steam curing. When curing temperature was risen from room temperature 80ºC, the strength increased up to 14% at 28 days. Ratio of split to compressive strength was about 10% when 1% fiber was applied. However, an optimum result was shown by specimens containing 0.6% fibers according to direct-tensile test. It is recommended to apply steam curing at 60ºC-80ºC to increase the tensile strength.

Peer-review under responsibility of the organizing committee of SCESCM 2016.

Keywords: Metakaolin; geopolymer; PVA; curing temperature.

---

* Corresponding author.
E-mail address: januarti_je@yahoo.com

Peer-review under responsibility of the organizing committee of SCESCM 2016.
doi:10.1016/j.proeng.2017.01.376

1. Introduction

Global warming has become a major topic in the wide range of science and engineering research, within the last decade. The problem is related with exhaust emissions such as CO_2 from human activities. It is about 65% of greenhouse gases caused by CO_2 where Portland cement production contributes in a range of 7-8% [1]. Therefore, it is necessary to obtain green concrete materials as an innovative solution. One of the solutions is geopolymer manufacture, which is also known as zero-cement concrete. Most of geopolymer mixtures use by-products in their composition. Fly ash is the most favorable material for making geopolymer concrete [2]. Mud, clay and slag are also utilized as the raw material in geopolymer binders [3-5]. However, in Indonesia, some by products such as fly ash and bottom ash are considered as hazardous materials. It comes to being the application limitation to develop geopolymer products from laboratory to industry scale. In addition, physical characters of by-product vary in some aspects such as origin materials treatment, shape, size, etc. [6-8]. Kaolin is one of the natural pozzolanic materials, which is rich in silica and alumina. With a proper calcination, metakaolin can be produced from 75%-80% by mass of kaolin, which contains soluble silica. Since it consists of reactive oxides, metakaolin requires low concentration of alkali activator [9].

In this paper, polyvinyl alcohol fiber (PVA) is introduced to improve tensile strength of metakaolin geopolymer paste. In addition, variations of curing methods were also introduced to investigate the effect of temperature to increase compressive strength. Some influential factors to the mechanical strength of fresh and hardened paste were analyzed and discussed comprehensively. Therefore, this study will be a bench-mark formalization of environmental friendly material using kaolin as one of the alumina-silicate sources.

2. Experiment

2.1. Materials and mix proportions

Kaolin with 2.5 g/cm^3 of density adopted in this study was obtained from Bangka Belitung, Indonesia. Its chemical composition is listed in Table 1.

Table 1 Chemical composition of source materials

Oxides (%)	SiO2	Al2O3	Fe2O3	CaO	MgO	Na2O	K2O	TiO2	MnO2	SO3	LOI
	50.26	43.00	0.73	0.04	0.12	0.04	0.57	0.28	0.01	4.72	4.72

Calcination method to activate raw material from kaolin to metakaolin is based on an experimental work developed by Triani [10]. Kaolin with particle size of 75 μm was immersed and stirred in distilled water for 10-15 minutes. It was kept in room temperature for 24 hours until sedimentation occurred. This precipitated materials were not used for making the geopolymer mixture since it may contain unreactive quartz. The top part of silt was collected to be dried in oven. Calcination process is illustrated in Fig. 1 as temperature history of furnace setting. Temperature target of 700^{o}C was set constant for six hours. Metakaolin was then pulverized until its maximum particle size of 75μm as shown in Fig 2.

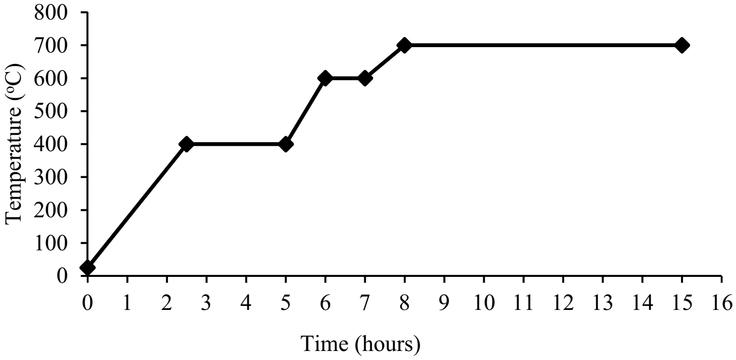

Fig. 1. Temperature History of Kaolin Calcination

Fig. 2. Metakaolin

Alkali activators consisted of sodium hydroxide and sodium silicate. Flake form of sodium hydroxide with 98% purity was prepared for concentration of eight molars. Sodium silicate (SiO_2/Na_2O = 2.0 and solid content 55%) was mixed with sodium hydroxide with a mass ratio of 56:44. Both liquids were mixed together, prepared one day before mixing with metakaolin and kept in environmental control room. Ratio of metakaolin to alkali activators of 1:1 was used for all variations.

PVA-RECS15 as shown in Fig.3 varied with 0%, 0.3%, 0.6% and 1% by paste volume. Fresh paste became more hardened and difficult to cast if it contained more than 1% of PVA. Properties of fibers are listed in Table 2.

All variations of paste were cast in cylindrical molds with 50 mm in diameter and 100 mm in length. Immediately after casting, the specimens were covered with plastic and then cured at a steam chamber with temperature varied at 40°C, 60°C and 80°C for 24 hours. After steam curing, specimens were moist-cured at room temperature until specific age for mechanical and physical testing.

Table 2 Fibers Properties

Physical Properties	Specification
Diameter	38 µm
Length	8mm
Density	1.3 gr/cm3
Tensile strength	1600 MPa
Bending strength	40 GPa
Melting Point	225°C
Absorption	Less than 1 %

Fig. 3. PVA-RECS 15

Variations of mix proportion and curing temperature are provided in Table 3.

Table 3. Specimen variations

No.	Code	Curing Temperature (ºC)	PVA (% Volume)
1	G-0-25	25	0
2	G-0.3-25		0.3
3	G-0.6-25		0.6
4	G-1-25		1
5	G-0-40	40	0
6	G-0.3-40		0.3
7	G-0.6-40		0.6
8	G-1-40		1
9	G-0-60	60	0
10	G-0.3-60		0.3
11	G-0.6-60		0.6
12	G-1-60		1
13	G-0-80	80	0
14	G-0.3-80		0.3
15	G-0.6-80		0.6
16	G-1-80		1

2.2. Testings

Setting time for fresh paste was conducted according to ASTM C191 [11]. Compression test to obtain the strength of some specimens at 3, 7, 14, 21, 28 and 56 days was conducted according to ASTM C39 [12]. To obtain the effect of fiber on density of specimens, which the specimens are categorized lightweight concrete, weight and volume of specimens were examined according to ASTM C1693-11 [13]. Splitting test according to ASTM C 496 [14] was conducted for cylindrical specimens at 28 days. Direct tensile test according to CRD-C 260-01 [15] was conducted for dog bone-like shape specimens at 28 days. Porosity test was also performed at 28 days to obtain portions of closed and open porosity as a function of total porosity.

3. Results and discussions

3.1. Setting Time

Both initial setting time and final setting time were carried out to observe the effect of fiber to setting time of paste. The result is provided in Fig 4.

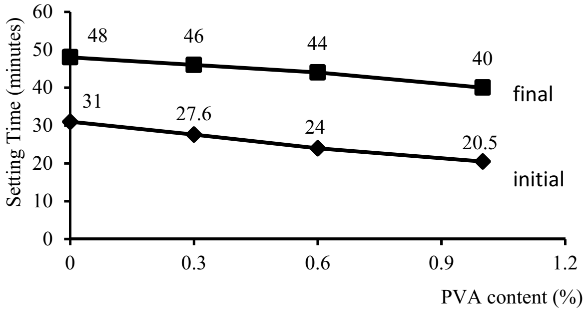

Fig 4. Setting Time

More addition of PVA fibers caused faster initial and final setting time. It is because of more fiber in mixtures turns the mixtures to be more viscous resulting in the decreasing of workability, and the setting becomes quicker. It has a good agreement as a result conducted by Noushini [16]. In this case, for larger scale of application, superplasticizer and retarder are necessary to ensure the mixture workability.

3.2. Density

Fig 5. describes the addition of fiber and curing temperature show less effect to density of geopolymer paste. The average density is about 1800 kg/m^3, which indicates that geopolymer paste is a sort of lightweight concrete. In addition, it was due to the amount of fiber was added at most only 1% by paste volume, and water absorption of fiber is less than 1% of the fiber mass. As a result, thee of PVA does not affect the weight to the volume of mortar geopolymer [17] .

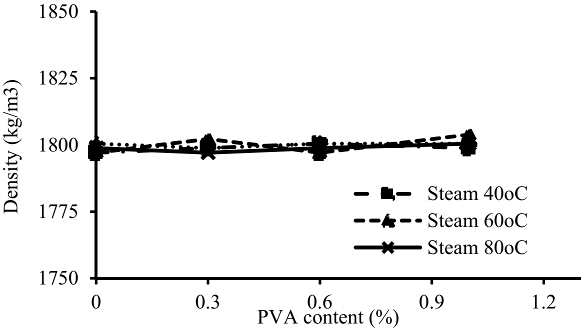

Fig 5. Relation of density and fiber content at different curing

3.3. Compressive strength

Compressive strength test performed at the age of 3,7,14,21,28 and 56 days. An average from three identical specimens was determined for each variation. The results for each variation can be seen in Figure 6 to Figure 9.

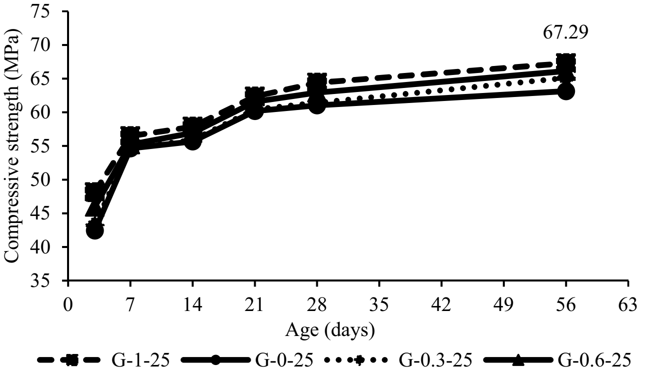

Fig 6. Compressive strength of specimens cured at room temperature

In Fig. 8, it is discovered that adding fiber increases the compressive strength. However, the fiber has less effect to contribute the strength at early age. The strength significantly increases up to seven days. After one week, it increases gradually and still has a tendency to improve until 56 days. Specimens containing 1% of fiber shows the highest compressive strength from the early age until the later age.

An effect of steam curing at 40ºC and 60ºC with different fiber content is presented in Fig. 7 and Fig 8 respectively. The increase of compressive strength occurred at the early age due to the acceleration of geopolymerization reaction. At 60ºC, the strength increased up to 21 days, which was faster than the specimens cured at room temperature and at 40ºC. The geopolymeric reaction of metakaolin paste is revealed to be accelerated strongly according to the curing temperature regardless of the fiber content. Geopolymeric reaction is similar to hydration reaction in cement portland, which occurs rapidly due to heat treatment [18].

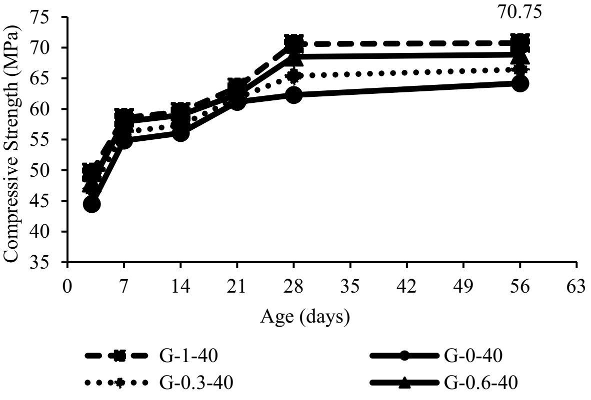

Fig 7. Compressive strength of specimens cured at 40ºC

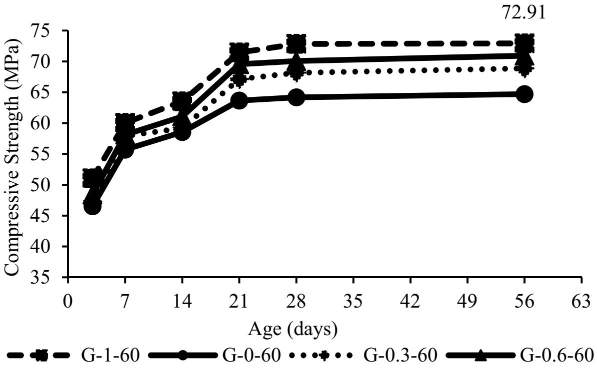

Fig 8. Compressive strength of specimens cured at 60ºC

Corresponding to spesimens cured at 40ºC and 60ºC, Fig 9 shows the influence of steam curing at 80ºC to the strength. Mechanical strength increased along with fiber content and age of specimens. At 80ºC, the strength increased rapidly until 14 days and gradually increased slowly until 21 days. It can be compared with Fig. 6, the compressive strength of specimens at 28 days, which contains 1% of fiber rises up to 14%. It is in accordance with an experimental study conducted by Hardjito [19] using fly ash as the raw material. In his research, the optimum strength of fly ash-based geopolymer concrete was achieved at 75ºC.

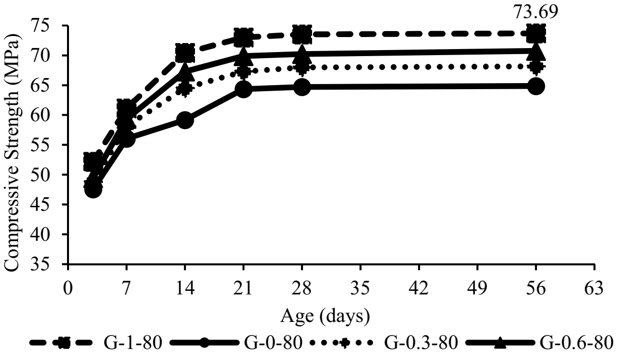

Fig 9. Compressive strength of specimens cured at 80ºC

3.4. Splitting tensile strength

Fig. 10 shows the result of splitting test at 28 days. As expected, influence of fiber content indicated more than the heat effect to the strength. It is shown by s G-1-80 containing 1% PVA 80ºC. It also implies that fiber has more contributions to tensile strength than compressive strength.

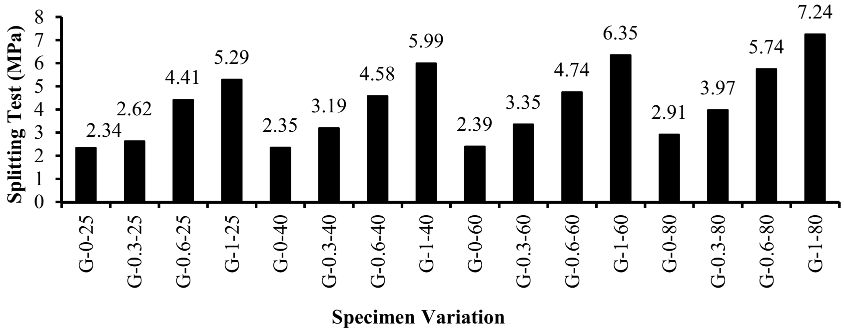

Fig 10. Splitting test of specimens cured at different temperature

3.5. Direct tensile strength

Stress-strain relation of dog bone-like specimens is provided in Fig. 11 to Fig. 14. It was predicted that at higher curing temperature, the tensile strength tended to rise. The absence of fiber showed less effect on specimen's ductility. Fiber content increased the ductility cured at the same condition. However, ductility of specimens containing fiber had a tendency to decrease at higher temperatures. Stress hardening was observed for specimens with 0.6% and 1% of fiber. It indicated that after crack occurred, tensile stresses were transferred to the fiber. Because of fiber, tensile strength is greater than the paste causing hardening phenomenon. This means that the PVA fiber starts to control the crack after paste failure to resist tensile load [20].

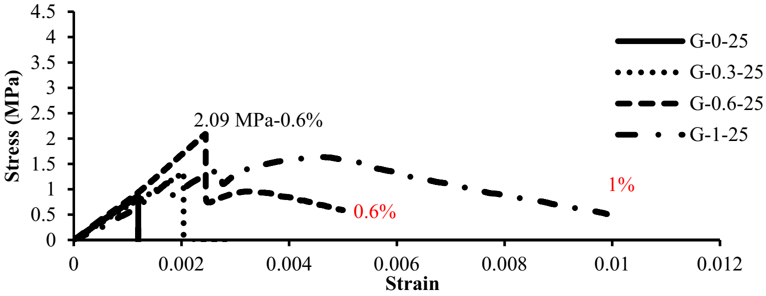

Fig 11. Tensile strength cured at room temperature

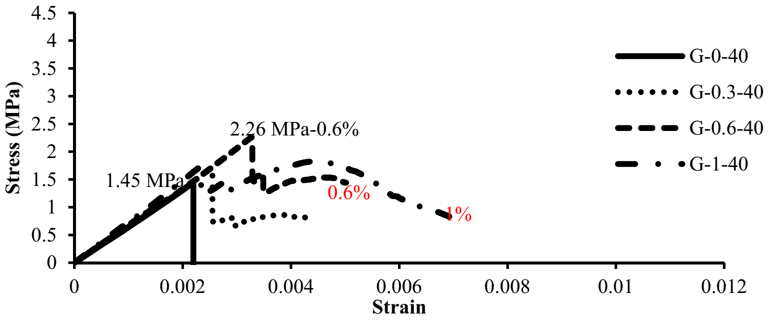

Fig 12. Tensile strength cured at 40ºC

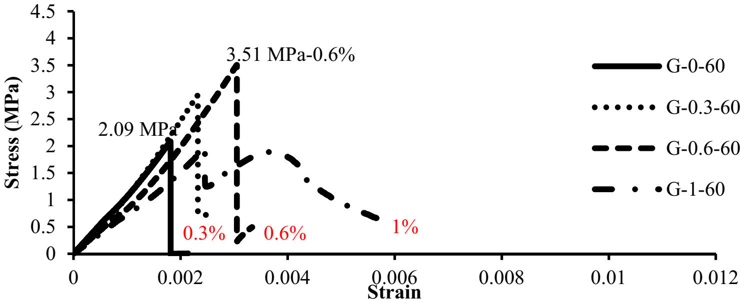

Fig 13. Tensile strength cured at 60°C

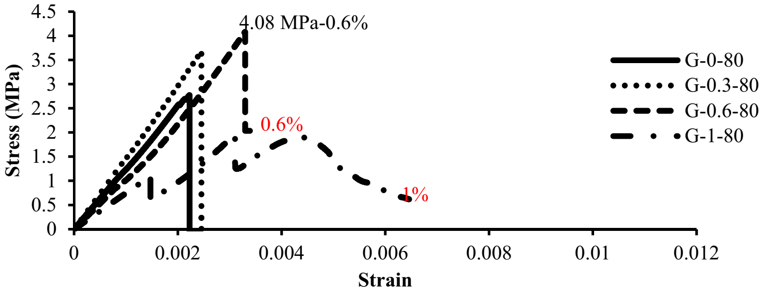

Fig 14. Tensile strength cured at 80°C

A relation of compressive strength and tensile strength at 28 days is represented by some factors. Some relationships such as f_{split}/f_c’, f_t/f_c’ and $\Delta f_t/\Delta\varepsilon_t$ are provided in Fig. 15, Fig. 16 and Fig. 17 respectively. Splitting strength, f_{split}, compressive strength, fc, and tensile strength f_t represents mechanical strength of specimens. Elastic stiffness is corresponded to a ratio of tensile stress (Δf_t) and tensile strain ($\Delta\varepsilon_t$) which is determined before cracks occur.

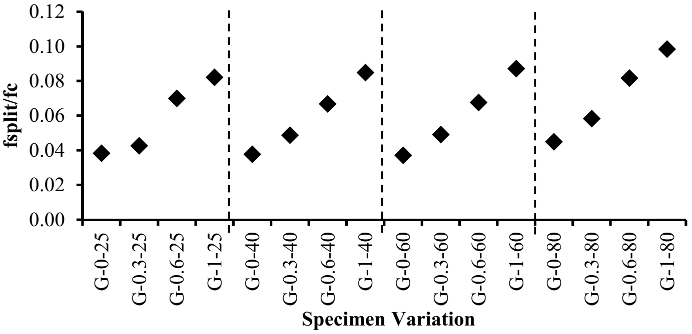

Fig 15. Ratio of splitting strength to compressive strength

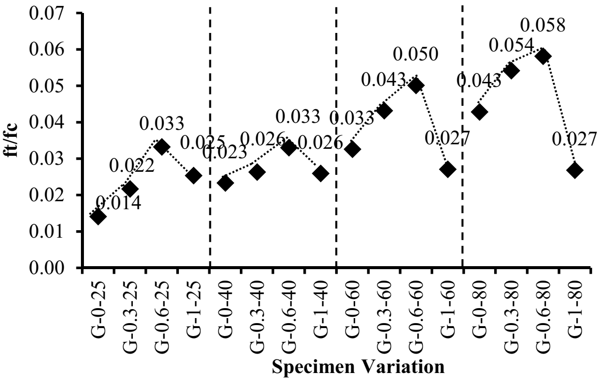

Fig 16. Ratio of tensile strength to compressive strength

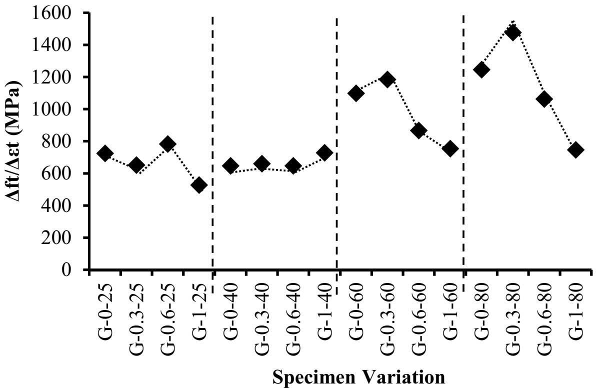

Fig 17. Ratio of tensile strength to tensile strain in elastic condition

In Fig. 15, it can be seen that fiber content has more contributions to increasing the ratio of tensile strength to compressive strength. Less influence to this ratio was exhibited by heat effect. This indicated that the increase of split tensile strength had a linear correlation to the increase of compressive strength [21]. At room temperature, this ratio rose from 3% to 9%. At 60°C, it increased up to 9.9%. However, in Fig. 16, there is an optimum point where addition of fiber at 0.6% shows the highest ratio of tensile to compressive strength. Since 1% of fiber resulted in difficulty of casting, direction of fiber in the paste caused the specimens became less uniform. Fig. 17 shows that the ratio of Δft to Δεt tends to descend along the increasing of fiber content and curing temperature. It reveals that greater stiffness generates brittleness of geopolymer paste.

3.6. Porosity

Fig.18 shows some relations of porosity of specimens containing 0% and 1% fiber. Curing temperature tended to decrease the total pores, which generated more strength. Surprisingly, fiber content showed less effect to the open porosity. In general, fiber increases the open porosity, resulted in denser of mixture and decreasing strength in certain proportion. Results in Fig. 18 reveal that PVA fiber contributes to decreasing of total porosity without affecting density of geopolymer paste.

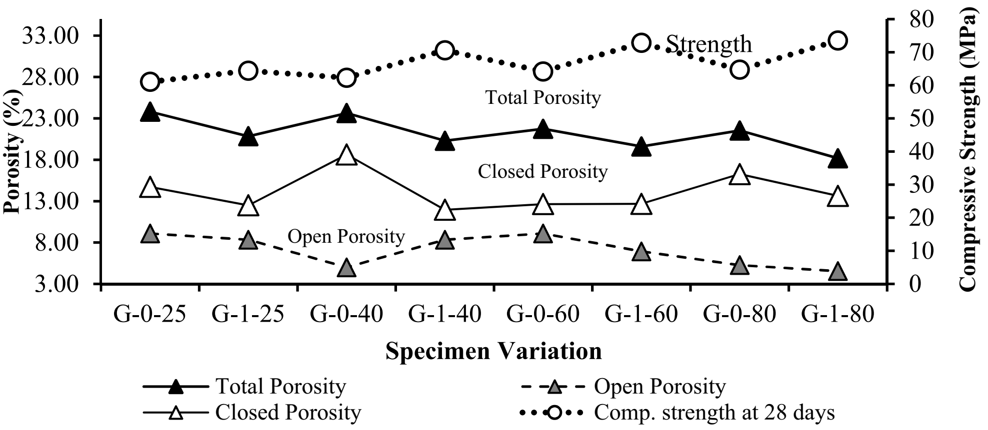

Fig 18. Porosity

4. Conclusions

- PVA fiber causes setting time becomes faster due to workability of fresh paste. However, adding PVA fiber to 1% by paste volume shows less effect on density of paste. This is because water absorption of fiber is very small.
- Curing temperature at 80ºC generates the highest compressive strength of paste containing 1% of fiber.
- Tensile stress is contributed mostly by the presence of fibers, which expanded the ductility of paste. Nevertheless, at higher curing temperature, even though the tensile stress increases, heat effect decreases the ductility.
- Stiffness of paste increases along with heat effect to the paste. However, the effect the fiber content contributes to decreasing of stiffness.
- Porosity has a strong relation with compressive strength. Regardless causing poor workability, PVA fibers contribute to decrease the total pores which support the strength of paste.
- It is recommended to apply PVA fiber up to 0.6% by volume to increase the ductility of geopolymer paste. More fiber content causes compacting problem to the fresh mixture.

References

[1] McCaffrey, Climate Change and the Cement Industry, Enviromental Special Issue 2002 (eds.), GCL Magazine, London.

[2] K. Zerfu and J.J. Ekaputri, Review on Alkali-Activated Fly Ash-Based Geopolymer Concrete, MSF 841 (2016) 162-169

[3] J. J. Ekaputri, Triwulan, S. Junaedi, Fansuri and R. Bayuaji, Light Weight Geopolymer Paste made with Sidoarjo Mud (Lusi), MSF 803 (2015) pp 63-74

[4] M. M. A. B. Abdullah, M. Md Mohtar, L. Y. Ming, M. F. M Tahir, K. Hussin, and J. J Ekaputri, Flood Mud as Geopolymer Precursor Materials: Effect of Curing Regime on Compressive Strength, AMM 815 (2015) pp 177-181

[5] A. N. Hashim, K. Hussin, N. Begum, M. M. A. B. Abdullah, K. A. Razak and J. J. Ekaputri, Effect of Sodium Hydroxide (NaOH) Concentration on Compressive Strength of Alkali-Activated Slag (AAS) Mortars, AMM 754-755 (2015) 300-304.

[6] J. J. Ekaputri, M. B. Ulum, Triwulan, R. Bayuaji, T. E. Susanto and M. M. A. B. Abdullah, A Comprehensive Characterization and Determination of Fly Ashes in Indonesia using Different Methods, AMM. 754-755 (2015) pp 320-325

[7] N. Ratni, J.J. Ekaputri, M. Bahrul Ulum, Triwulan and P. Suprobo, Characterization of Fly Ash on Geopolymer Paste, MSF 841 (2016) pp 118-125

[8] J. J. Ekaputri, Triwulan and T. E. Susanto, Lightweight Geopolymer Paste made with Sidoarjo Mud, the 6th ACF 2014, 21-24 September, Gangnam, Seoul, South Korea, pp. 1053-1057

[9] S. Junaedi, A. Haris, N. Nurhayati, A. Irhamsah and J.J. Ekaputri, The Influence of Si:Al and Na:Al on the Physical and Microstructure Characters of Geopolymers Based on Metakaolin, MSF 841 (2016) 170-177

[10]D.N.D. Triani, J.J. Ekaputri, Triwulan, S. Hardono and T. E. Susanto, Application of Pozzolan as Materials of Geopolymer Paste, MSF 841 (2016) pp 111-117

[11] ASTM C 191, Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle, ASTM International, 2013

[12] ASTM C 39, Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens, ASTM International, 2013

[13] ASTM C1693, Standard Specification for Autoclaved Aerated Concrete (AAC), ASTM International, 2009

[14] ASTM C 496, Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens, ASTM 2011
[15] CRD-C 260-01, Standard Test Method for Tensile Strength of Hydraulic Cement Mortars, 2001
[16] A. Noushini, B. Samali and K. Vessalas, Influence of Polyvinyl Alcohol Fibre Addition on Fresh and Hardened Properties of Concrete, Proceeding of EASEC 2013 pp: B-5.
[17] J. J. Ekaputri, H. Limantono, Triwulan, T. E. Susanto, and M. M. A. B. Abdullah, Effect of PVA Fiber in Increasing Mechanical Strength Paste Containing Glass Powder, KEM 673 (2016) pp 83-93
[18] J. J. Ekaputri, K. Maekawa, T. Ishida, Experimental Study on Internal RH of BFS Mortars at Early Age, MSF 857, pp 305-310
[19] D. Hardjito and B. V. Rangan, Factors Influence the Compressive Strength of Fly Ash- Based Geopolymer Concrete, Civil Eng. Dimension 6, 2, pp 88-93.
[20] M. Ohno and V. Li, A Feasibility Study of Strain Hardening Fiber Reinforced Fly Ash – Based Geopolymer Composites, Cons. and Build. Ma. 57, pp 163-168.
[21] W. Hu, Experimental Research on the Mechanical Properties of PVA Fiber Reinforced Concrete, Research Journal of Applied Sciences, Eng. and Tech. 5, 18, pp 4563-4567.