

EFFECT OF GGBFS AND MICRO-SILICA ON MECHANICAL PROPERTIES, SHRINKAGE AND MICROSTRUCTURE OF ALKALI-ACTIVATED FLY ASH MORTAR

*Yuyun Tajunnisa¹, Masaaki Sugimoto¹, Takahiro Uchinuno¹, Takahiro Sato², Yoshinori Toda², Arisa Hamasaki², Toru Yoshinaga², Kenji Shida² and *Mitsuhiro Shigeishi³

¹ Graduate School of Science and Technology, ²Technical Division, Faculty of Engineering,

³Faculty of Advanced Science and Technology, Kumamoto University, Japan

*Corresponding Author, Received: 4 June 2017, Revised: 25 June 2017, Accepted: 13 July 2017

ABSTRACT: Performance of drying shrinkage, flow rate, mechanical properties, and microstructure of three materials - alkali-activated fly ash (FA); ground granulated blast-furnaced slag (GGBFS); and un-densified micro-silica (M) are investigated. Mixtures used herein are referred to as AAM – alkali activated materials – of four types according to composition: AAM-IV, AAM-V, AAM-VI and AAM-VII (note: types 1 through 3 were investigated in previous research) with corresponding mixture FA, GGBFS and M ratios of 47.5/47.5/5, 45/45/10, 42.5/42.5/15, 40/40/20 by weight percentage. The AAM samples were air-cured under a sealed condition for 6 days followed by unsealed curing up to the test at 18-20° C and a low relative humidity of 30-50%. The samples AAM-IV through VII were composed of a progressive decrease of fly ash and GGBFS and an increase in M content. Results show that flow rate and compressive strength increased from AAM-IV to AAM-VI; contrarily, it decreased in AAM-VII. Both flexural strength – a positive aspect - and drying shrinkage – negative as it leads to cracking - increased in all samples. Incorporation of both GGBFS and M in alkali-activated fly ash mortar was found to improve performance compared to that incorporated solely of GGBFS in alkali-activated fly ash. The incorporation of M of certain values improves strength and flowability of AAM; conversely, it results in a higher drying shrinkage value, which leads to increased cracking. SEM and XRD results confirm these results. Unreacted particles of AAM-VI and AAM-VII appear to act as a ‘micro-aggregate,’ resulting in increased compressive and flexural strength.

Keywords: Slag, Micro silica, Alkali-activated fly ash, Geopolymer, Shrinkage, Mechanical properties

1. INTRODUCTION

Glukhovsky hypothesized a relationship between alkaline and cementitious materials in 1957; now called alkaline cements, these have become a widely-used basic new type binder [1-2]. Alkali-activated fly ash represent promising sustainable alternatives to ordinary Portland cement, with benefits including 70% lower CO₂ emission than Portland cement [3], higher mechanical properties [4-6], fire resistance [7], thermal resistance [8], lower heat of hydration [9] and heat release [10], strength against chemical attack [11] [12] [11-12], durability in an aggressive environment [13-14], freeze-thaw resistance [15], lower shrinkage in fly ash-based geopolymer [6;16-18], and comparable shrinkage value in blended slag-fly ash geopolymers [19-20]. Conversely, drawbacks include setting periods [21-22], high shrinkage in alkali-activated materials [23-25], high salt efflorescence [5], higher carbonation [26], and cracking while curing [27-28]. A compilation of material regarding largescale application of alkali-activated cement for materials, cements, concrete, structures, tests procedures, and system benefits is presented in [29].

Researchers have been attempting to solve these drawbacks using different additives and admixtures in an alkali-activated material (AAM) system. The system involves a chemical reaction of aluminosilicate minerals under alkaline condition on Si-Al minerals [30]. The final products of reaction are influenced mainly by the chemical composition of the alkaline and source materials [31-35].

Recent studies have shown that blending of ground granulated blast furnace slag with low-calcium fly ash have significant effects on the setting and early strength development of systems cured at room temperature [19;36]. The material source, fly ash, is produced at around a billion tons annually worldwide by coal-fired power plants [9;31;37-38]. The large usage of land for fly ash disposal severely impacts the environment of surrounding areas. Fly ash activation has been extensively studied over the past year [36;39-41]. The other source material, ground granulated blast furnace slag (GGBFS), is a waste obtained from iron manufacturing [42]. While slag is used in the cement industry, the majority of GGBFS is still disposed in landfills [43]. This AAM system alleviates disposal problems of both fly ash and

GGBFS. Silica fume is a by-product during the production of silicon and ferrosilicon alloys [44]. Silica fume is also known as micro silica enhancing mechanical properties [45]. Other researchers utilized other waste materials as in [46].

Alkali activation of slag involves dissolution of calcium and participation of Al to sodium aluminosilicate (N-A-S-H) gel, partially replacing sodium with calcium to form (Ca-Na)-A-S-H gel; this enables rapid hardening of fly ash-based concrete at ambient temperature [47-48]. In addition, the use of silica fume to improve concrete performance offers several benefits such as compressive strength [49], tensile and flexural strength [50], durability [51], porosity [52], and modulus of elasticity [53]. In contrast, silica fume incorporation reduces workability of fresh mortar, and thus requires the addition of an agent to improve workability [54]. Poly-carboxylate based agents are a type of water-reducing admixtures able to improve workability and mechanical properties of geopolymer [55-56]. In addition, drying shrinkage is an important part of alkali-activated slag in early stages and is a critical parameter for durability design of structures [4;57].

A previous study [22] reported initial and final setting time of type II fly ash paste at 45 and 108 hours. However, initial setting time of OPC (Ordinary Portland Cement) with Vicat Needle by [58] at 30 min and final setting time should not be more than 10 hours. This relatively long setting disqualifies fly ash paste from most applications unless it can be shortened by adding admixture. This study thus applied incorporation of slag and micro-silica to enhance system performance. Though drying shrinkage is a major weakness of alkali-activated fly ash/slag, there has not yet been published research. Therefore, this work aims to reveal effects of ground granulated blast furnace slag and micro silica incorporated to fly ash mixed alkaline solution (sodium silicate and 8 M sodium hydroxide) under unsealed curing at 18-20° C and low relative humidity of 30-50% on drying shrinkage performance. Scanning electron analysis is also used to assess and compare mechanical properties of mortars.

2. EXPERIMENTAL DETAILS

2.1 Materials

Material origin is as follows: Portland cement as [59] for OPC (Ordinary Portland Cement) Mortar; type II fly ash as [60], GGBFS (particle fineness grade 4000-4120 cm²/g of specific surface area), and un-densified micro-silica Grade 940 as [61] for AAM. A high-performance AE water reducing agent, a polycarboxylic acid ether-based agent was added at a quantity of 2% of raw precursor mass.

Fine aggregate used in saturated surface dry (SSD) condition was standardized sand [58;62], 1350g per pack as described in Table 1 and Table 2. The chemical composition of raw precursors is listed in Table 1. Specific gravity (density) of materials is described in Table 3. A commercial liquid sodium silicate in bulk consisted of 18.5% Na₂O, 36.5% SiO₂, and 45.1% H₂O by mass. The 8M sodium hydroxide solution was prepared by dissolving pellets in distilled water.

Images of material precursors and sand are shown in Fig. 1. Fly ash powder characteristically consists of spherical particles with diameters ranging from 10 to 200 mm and a relatively rough surface. GGBFS powder consists of irregular and angular morphology of individual particles. Nano-sized micro silica particles are mainly of two types, spheres or semi-cylinders. The morphology of sand particles shows large size and angular appearance of these grains.

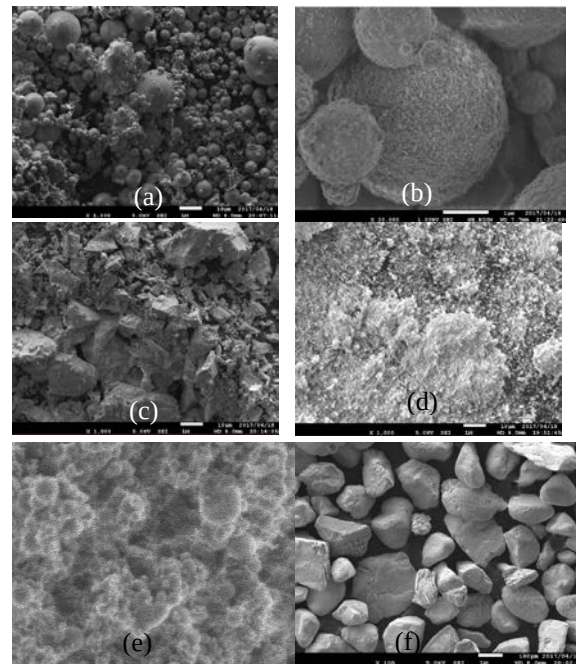


Fig. 1 SEM micrographs of typical shape: (a) fly ash magn.1k x; (b) 20k x; (c) slag 1k; (d) micro silica 1k x; (e) 20k x; (f) sand 0.1k.

2.2 Mixture Proportions

Four types of alkali-activated fly ash/slag/micro silica mortars (AAM) and an OPC mortar are compared here. AAM specimens, namely AAM-IV, AAM-V, AAM-VI, and AAM-VII were prepared by alkali activation of fly ash, which was partially replaced by GGBFS of 47.5, 45, 42.5, 40% and micro-silica of 5, 10, 15, 20%, respectively, by total mass raw precursors.

Table 1 Chemical composition of raw precursors in weight percentage (wt.%) by XRF and chemical analysis standardized sand

Compound	Fly ash	GGBFS	Micro silica	Sand*
SiO ₂	55.19	35	93.67	98.4
Al ₂ O ₃	25.35	16	0.83	0.41
Fe ₂ O ₃	7.57	0.7	1.3	0.36
CaO	4.06	46	0.31	0.16
Na ₂ O	-	-	0.4	0.01
H ₂ O	-	-	-	<0.2
K ₂ O				0.01

Note: This table illustrate chemical composition of the raw precursors by XRF applied in this research. Standardized sand* as [58-62].

Table 2 Particle size distribution of standardized sand as [58]

Square mesh size (mm)	Cumulative sieve residue (%)
2	0
1.6	7 ± 5
1	33 ± 5
0.5	67 ± 5
0.16	87 ± 5
0.08	99 ± 1

Table 3 Specific gravity of materials

Materials	Specific Gravity (g/cc)
OP cement	3.16
Fly ash, Type II	2.33
GGBFS	2.89
Micro silica	2.22
Standardized sand	2.64
Na ₂ SiO ₃ gel	1.6
8 M NaOH solution	1.22
AE Agent	1.0

Table 4 Raw precursors of AAM proportions

Mix	Symbols	FA (%)	S (%)	M (%)
A	FA ₅₀ S ₅₀	50	50	0
B	FA ₉₀ M ₁₀	90	0	10
C	FA ₄₀ S ₅₀ M ₁₀	40	50	10
D	FA _{47.5} S _{47.5} M ₅	47.5	47.5	5
E	FA ₄₅ S ₄₅ M ₁₀	45	45	10
F	FA _{42.5} S _{42.5} M ₁₅	42.5	42.5	15
G	FA ₄₀ S ₄₀ M ₂₀	40	40	20

Note: This table illustrates raw precursor proportions by weight applied in AAM mortars.

Fixed alkali liquid concentration dosage with ratio sodium silicate to sodium hydroxide was 1.5 for all AAM mixtures, by weight. Alkaline liquid/raw precursors and liquid/raw precursor ratios were fixed at 0.538 and 0.558 by weight for all AAM. Mass of AE agent was 2% of raw precursor mass. Liquid consisted of AE agent and alkaline solution. The volume of each AAM mixture for three prisms of 40x40x160mm was constant at 878.8 cc and consisted of a fixed volume of paste, liquid, raw precursors, and fine aggregate at respective amounts of 367.4, 183.8, 462.4, and 511 cc. Three other AAM, namely AAM-I, AAM-II and AAM-III, were subjected to a 14-day compression strength test to support the investigation of effects of GGBFS and micro-silica on alkali-activated fly ash mortar. Raw precursors consist of alkali-activated FA/ GGBFS/M, encoded, AAM-I, AAM-II and AAM-III mixtures at weight percent ratios of 50/50/0, 90/0/10 and 40/50/10. Raw precursors of AAM mixture proportions are given in Table 4.

These AAM mixtures were compared to the original mixture proportion OPC mortar as [58] having mass proportion ratio of OPC mortar of cement: sand: water ratio of 1:3:0.5; water/cement ratio of 0.5; 450 g of ordinary cement estimated 3.15g/cm³ or 143 cc; and 225 g of water at 20° Celsius estimated 1.00g/cm³. Estimated volume OPC mortar same with those of AAM, each mixture mortar was 878.8 cc. Volume ratio of paste to fine aggregate is 368:768 estimated of 0.48:1 resulting 2.304 g/cc OPC mortar density.

2.3 Mixing, Molding, Curing and Testing Methods

2.3.1 Alkali-activated Mortar (AAM)

The raw precursors for AAM were formulated with FA/S/M ratios of 47.5/47.5/5, 45/45/10, 42.4/42.5/15 and 40/40/20, respectively. A mixture mortar of 887.8 cc was mixed in the 2 litre batch mixer in control room at 18-20° C and 30-50% RH. Raw precursors were mixed for 2 minutes in low speed. The 8 M sodium hydroxide solution previously dissolved at least one day before mortar mixing, was then mixed manually with sodium silicate gel for 5 min followed by 10 min resting; then was added with AE agent. This alkaline liquid was then added and mixed with raw precursors in 2 min-low speed mixing; stopped the mixer; added with sand; continued for another 3 min-low speed mixing. After mixing, the mortars were immediately poured into 3 prism molds of 40x40x160 mm³ or cylindrical molds of 50x100 mm³. The molds were compacted on vibrator table for 2 min to remove air bubbles. The specimens were cured at 18-20°C and 30-50% RH, sealed with

plastic until demolding after 6 days in air condition, continued in unsealed condition up to the testing date.

Flow rates were tested twice for each mixture as JIS [58]. Flexural test was determined for prisms in accordance with JIS [58]. Flexure load speed was 50 N/s at a range of 20 kN; compressive test speed was $2.4 \text{ kN/s} \pm 0.2 \text{ kN/s}$ at a range of 200 kN. Cylindrical compressive tests were conducted at 14 and 28 days as [63]. Drying shrinkage tests of 3 prisms of $40 \times 40 \times 160 \text{ mm}$ were conducted as [64]. Determination of drying shrinkage of hardened mortars was started at day 7 and recorded daily until day 28; continuous twice-weekly recording continued until 84 days.

Scanning Electron Microscopy (SEM) testing was conducted to observe microstructure on pieces of cylinder specimens of AAM. Unfractured cylindrical samples of AAM-IV, AAM-VI and AAM-VII aged 80 days were cut into slices of 3 mm thickness in a vertical direction of the cylindrical sample from three different points: one at the center, one near the bottom, and one near the top. SEM test was applied to these non-coated AAM samples by JEOL JSM-7600F machine using voltage of 5 KV (low voltage LV, low current LC) for the shape of mortar and raw precursors (fly ash and GGBFS); and voltage of 1 KV (LV), LC for the of micro-silica morphology.

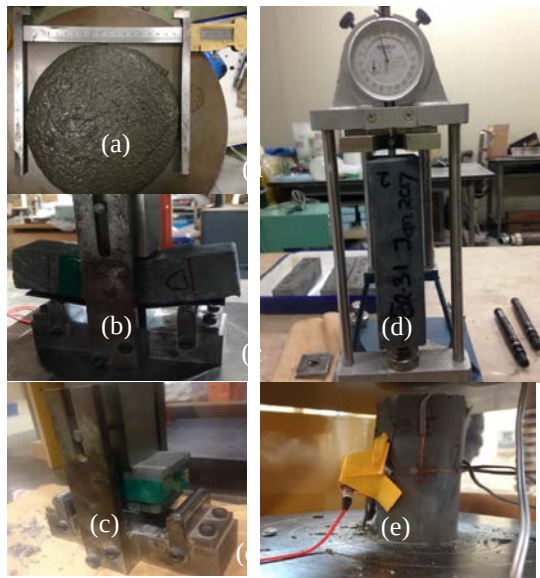


Fig. 2 Tests: (a) Flowability, (b) Flexure, (c) Compressive Test on Prisms, (d) Drying Shrinkage, (e) Compression on Cylinders

2.3.2 Ordinary Portland Cement (OPC) Mortar

OPC Mortar was mixed under JIS [58]. Cement and water were mixed for 30sec at low speed. Sand was then added over 30sec while mixing at high

speed. Mixing was stopped at 90sec, and then restarted to continue for another 60sec at high speed. The OPC mortar molds were compacted using a vibrator table as [58]. The mortars were poured a half in the molds for 15sec, continued for 15sec, then added for another half in 15 min, continued for further 75 sec. Several tests for all specimens were illustrated as shown in Fig. 2

3. RESULTS AND DISCUSSION

Drying shrinkage is a time-dependent deformation due to water loss [65]. It may be shown by crack presence. The four mortar designations had 3 kinds of precursors: AAM-IV, V, VI, VII had decreasing fly ash content (47.5, 45, 42, 40%); decreasing GGBFS content (47.5, 45, 42, 40%); and increasing micro-silica (5, 10, 15, 20%). Fig. 3 shows drying shrinkage of fly ash/slag/micro silica AAM and OPC Mortar as a function of age. It is observed that drying shrinkage increases rapidly up to 14 days and then slightly with time. However, the graph shows drying shrinkage tends to flatten after 60 days. It shows that AAM has a higher drying shrinkage value than that of OPC mortar. AAM-IV has the lowest drying shrinkage value among AAM types, followed by AAM-V, AAM-VI, and AAM-VII. However, the drying shrinkage value of AAM-IV is higher than that of OPC Mortar.

Compressive strengths of AAM-IV to AAM-VII at 14 days achieved about 95-99% those of 28 days. This compares well with 14-day ordinary Portland mortar (OPC), which achieved 79% that of 28 days, as given in Table 4. GGBFS incorporation affects AAM gains early strength compared with that of OPC. The compressive strength of AAM-I, AAM-II and AAM-III at 14 days are 62.5, 16.7 and 66.2 respectively as shown in Fig. 4. The lowest strength of AAM-II (without GGBFS) indicates that the presence of Ca (GGBFS) in AAM-I causes early hardening and enhances strength, and the addition of both reactive Si (micro-silica) and Ca (GGBFS) in AAM-III makes it higher strength than that of AAM-II. Furthermore, compressive strengths of 4 kinds of AAM from 3 kinds of raw precursors (AAM-IV, AAM-V, AAM-VI and AAM-VII) are 64, 66, 68 and 64 MPa respectively, as shown in Fig. 5. This indicates slight differences among them. The compressive strength of AAM-IV to VI slightly increases; contrarily, slightly decreases at AAM-VII. This is caused by various Ca and Si additions to the mixture. However, the increase of strengths among the 4 types of AAM are slight significant.

Furthermore, precursor compounds affect flow rate values of mortars. AAM-IV, V, VI and VII have flow rate values of 172, 176, 181, and 177 mm respectively, compare with that of OPC, 179 mm. Flow rate of AAM-IV to AAM-VI slightly increases, but AAM-VII slightly decreases. Flow

rate support the results of compression strength value.

However, the trend of flexural strength slightly differs from those of compression strength results: flexure results of AAM-IV to AAM-VII increase, as can be seen in Fig. 5 and Fig. 6. The SEM image in Fig. 7 shows that results are strongly correlated with the result of mechanical properties of mortars. Cracks noticed among AAM are related to the higher drying shrinkage values as shown in Fig. 3 and Fig. 7. This may also be due to extreme conditions during curing with sealed condition in 6 days; unsealed condition up to test date and low relative humidity in the control room.

SEM images indicate that some un-reacted particles appearing in AAM act as an 'micro-aggregate', causing increased compressive and flexural strength. However, the highest compression strength was achieved at AAM-VI with 15% micro-silica, and the highest flexure strength at AAM-VII with 20% micro-silica. It seems that a certain high micro-silica content provides positive influence in flexural strength. This may due to un-reacted micro-silica filling tiny pores and resulting higher flexural strength.

XRD (X Ray Diffractogram) in Fig. 8 shows the intensity of quartz in sample AAM-VII is higher than in sample AAM-IV. The scale of the Y-axis shows the intensity wt.% of quartz in samples of type AAM-VII is greater than in sample AAM-IV.

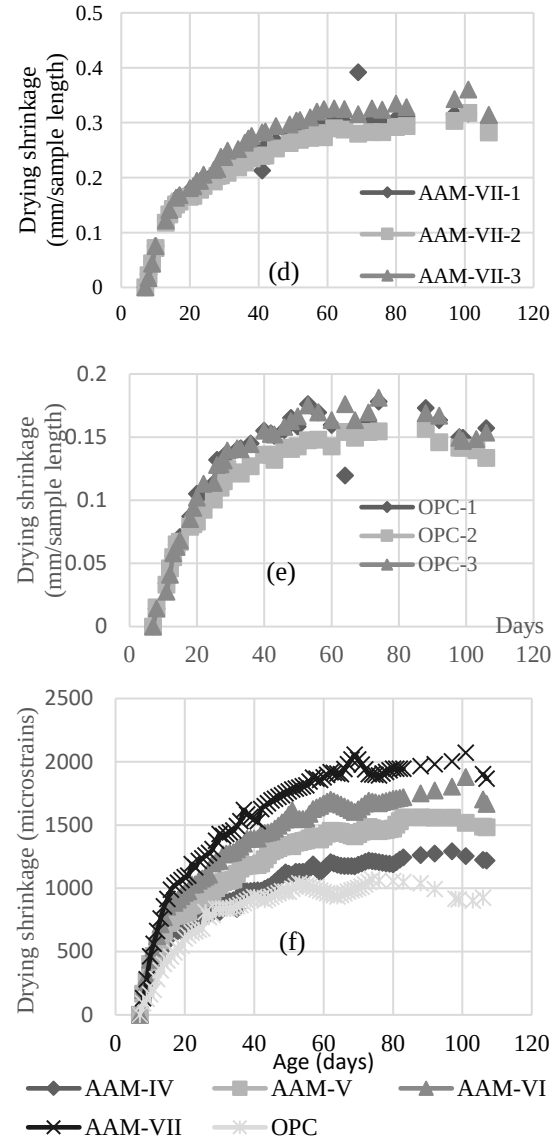
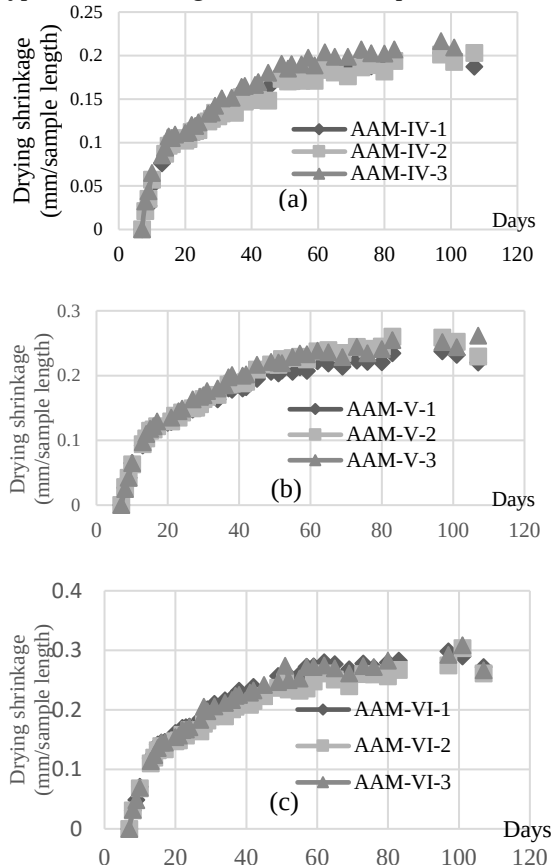


Fig. 3 Drying shrinkage of Fly Ash/GGBFS/micro silica alkali-activated mortar as function of time: (a) AAM-IV; (b) AAM-V; (c) AAM-VI; (d) AAM-VII; (e) OPC Mortar; (f) AAM and OPC Mortar

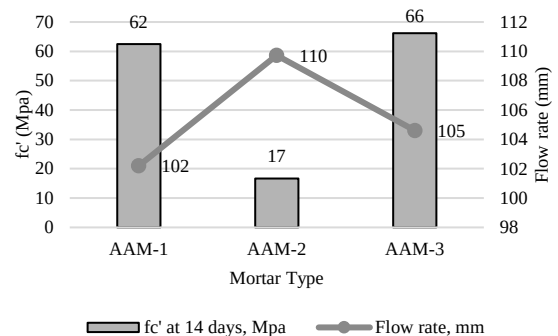


Fig. 4 Flow rate and the 14-day compressive strength values of AAM-I, AAM-II and AAM-III cylinders

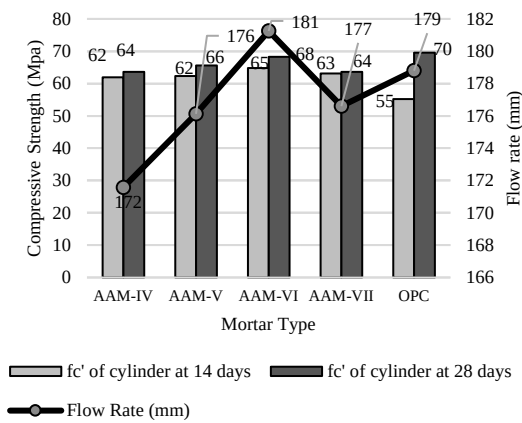


Fig. 5 Flow rate and the 14-day and 28-day compressive strength of AAM-IV, AAM-V, AAM-VI, AAM-VII cylinders

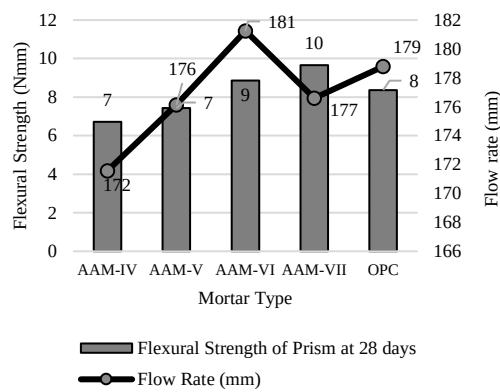


Fig. 6 Flow rate and the 28-day flexural strength of AAM-IV, AAM-V, AAM-VI, AAM-VII prisms

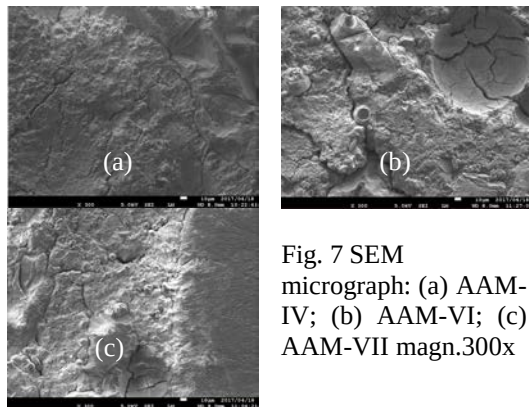


Fig. 7 SEM micrograph: (a) AAM-IV; (b) AAM-VI; (c) AAM-VII magn.300x

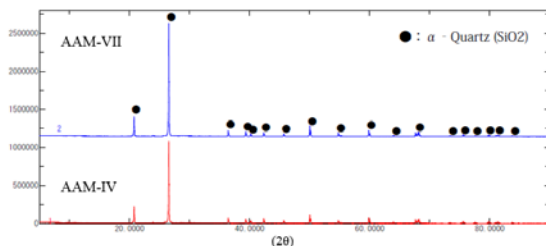


Fig. 8 AAM-IV and AAM-VII X Ray Diffractogram

4. CONCLUSION

The present work reviews and summarizes effects resulting from incorporation of Ca (GGBFS) and Si (micro-silica) of certain amounts as fly ash replacement on mechanical properties of AAM. Both GGBFS and micro-silica incorporation in AAM improves strength compared to that of only GGBFS incorporation. AAM show a higher drying shrinkage compared with OPC mortar. The trend of AAM drying shrinkage increases with increased proportion of micro-silica and decreased proportion of GGBFS and fly ash. High drying shrinkage causes some cracking in AAM; however, strength differences among AAM types were insignificant.

Authors recommend further investigation regarding curing conditions, specifically, which curing time length under sealed condition allows sufficient time for reaction; and how variations of relative humidity affect shrinkage.

5. ACKNOWLEDGEMENTS

The authors gratefully acknowledge Assoc. Prof. Januarti Jaya Ekaputri, Institut Teknologi Sepuluh Nopember, Surabaya, Indonesia for valuable comments.

This research work in Kumamoto University, Japan has been supported by BPPLN Scholarship, Ministry of Research, Technology and Higher Education of the Republic of Indonesia. I also would like to thank to Prof. Jun Otani, Assoc. Prof. Kozo Onoue, Assoc. Prof. Toshifumi Mukunoki and Mr. William Baerg, Kumamoto University, Japan, for their valuable suggestions.

6. AUTHOR'S CONTRIBUTIONS

The experiments were conducted by Yuyun Tajunnisa as part of her PhD research project, with a help of Masaaki Sugimoto and Takahiro Uchinuno; supervised by Yoshinori Toda, Arisa Hamasaki, Prof. Mitsuhiro Shigeishi; SEM test with a help of Takahiro Sato and Yoshinori Toda; XRD was conducted by Dr. Kenji Shida. The manuscript was prepared by Yuyun Tajunnisa under supervision of Prof. Mitsuhiro Shigeishi.

7. ETHICS

This article is original work. There are not conflict interest on this paper.

8. REFERENCES

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