CHARACTERIZATION ALKALI-ACTIVATED MORTAR MADE FROM FLY ASH AND SANDBLASTING

*Yuyun Tajunnisa1, Ridho Bayuaji1, Nur Ahmad Husin1, Yosi Noviari Wibowo1, Mitsuhiro Shigeishi2
1Faculty of Vocations, Institut Teknologi Sepuluh Nopember, Indonesia; 2Faculty of Advanced Science and Technology, Kumamoto University, Japan

*Corresponding Author, Received: 23 Dec. 2018, Revised: 07 Feb. 2019, Accepted: 21 Feb. 2019

ABSTRACT: This research is to find out the contribution of waste energy utilization in Indonesia as a binding agent of alkali-activated mortar. In a previous study, researchers investigated mortar made from class F fly ash/GGBFS/micro-silica in Japan. The inclusion of GGBFS is to shorten/normalize the setting time and micro-silica is to improve mortar performance. This research is then continued by using abundant waste material in Indonesia, namely class C fly ash, by making cubic mortar specimens. Setting time of class C fly ash paste from Indonesia is very fast, in contrast to that of class F fly ash paste from Japan. Sandblasting as abundant waste material in Indonesia is substituted to class C fly ash to lengthen the setting time of paste and to improve standard deviation of a compressive test of mortar specimens. On the other hand, the addition of sandblasting waste has a negative effect, because it reduces a compressive strength of mortar specimens.

Keywords: Alkali-Activated Material, Geopolymer, Fly Ash, Sandblasting, Waste Material

1. INTRODUCTION

Alkali-activated concrete, commonly called geopolymer concrete is one of alternative materials to replace concrete made from Portland cement, with the advantages of being more environmentally friendly - in the manufacturing process without releasing CO emissions into the atmosphere, resistant to corrosive environments, more resistant to chemical attacks [1,2], and more resistant to temperature [3,4]. Glukhovsky found that aluminosilicate transformation as the basis of the structure formation process in a paste [5]. The reaction of aluminosilicate minerals with strong alkali metal silicate solutions is called the geopolimerization or reaction of alkali-activated materials [6]. Solid materials for geopolymerization can be either natural aluminosilicate minerals [7,8].

One of the most widely available aluminosilicate materials is fly ash with a total of 2260 million tons per year or 12 times the availability of Portland cement [9] in its use. Class C fly ash is rarely used in geopolymer concrete mixtures, because it has a CaO level greater than 10%. Most researchers have learned about class F fly ash which has lower CaO. Geopolymeric paste from class C fly ash has a very quick setting time because it contains high calcium. The hydraulic reaction was increased by higher calcium levels. It becomes difficult to lengthen the setting time [10]. However, this paper discusses the utilization of class C fly ash, due to the availability of abundant class C fly ash in Indonesia. Therefore, its application requires a certain combination of admixture to extend the setting time of geopolymer.

Sandblasting is an admixture used in shipyards to clean dirt, corrosion, paint or other coatings. Sandblasting has the potential to become a concrete binder. Sandblasting waste has a binding chemical characteristic which is stronger than cement because of the high content of silica and alumina compounds. The high content of SiO2 which reaches 85% to 98% is useful for cement mix purposes [11]. Further, it was also visually observed that the addition of sandblasting increases the workability of geopolymer paste [12].

A prior study [13], the alkali-activated mortar was successfully developed by utilizing fly ash type F, Ground-granulated Blast Furnace Slag (GGBFS), and micro-silica. A preliminary experiment was also studied the setting time of class F fly ash paste from a power plant in Kyushu-Japan was relatively long, about 7 days [13]. The addition of GGBFS as fly ash replacement on their mixtures have shortened and normalized time set. The use of micro-silica on their mixtures have improved the performance of mortar.

This present work aims to apply the use of abundant waste material from Indonesia, high calcium fly ash from Paiton power plant, in order to contribute as additional data to the alternative environmentally friendly concrete topics. Fly ash class used for this research is different from that of previous research at Kumamoto University in Japan. However, this present work synchronizes research of Institut Teknologi Sepuluh Nopember in Indonesia.
2. METHODOLOGY

Fig. 1. Mixture Proportion of Alkali-activated Mortar

The methodology used in this work is laboratory experiments, cube-mortars of 50 x 50 x 50 mm were used as specimens for compressive strength tests. Mix design of alkali-activated mortar refers to previous work [13-16]. The proportion of mixtures can be seen in Fig 1.

Sodium silicate (Na$_2$SiO$_3$) with 18.5% of Na$_2$O, 36.4% of SiO$_2$, and 45.1% of H$_2$O were used in mixtures in Indonesia. The 8 M NaOH solution was made from NaOH flakes that 98% of purity mixed with water. Alkali solution concentration ratio of Na$_2$SiO$_3$ to 8 M NaOH was 1 for AAM1 and AAM2, by weight. Compared to previous work in Japan [13], sodium silicate consisted of a maximum 17-19% of Na$_2$O, 35-38% of SiO$_2$, and 46% of H$_2$O. Alkali solution concentration ratio of Na$_2$SiO$_3$ to 8 M NaOH was 1.5 for AAM3 to AAM9, by weight. Compressive strength was measured.

Experimental work was carried out by producing cube-mortar samples in several variations as shown in Table 1. Material testing which determines chemical content of material was done by XRF (X-Ray Fluorescence) test, as shown in Table 2. The SEM (Scanning Electron Microscopy) test was also conducted to determine the morphology of material, as can be seen in Fig 2. Based on ASTM C618-03, fly ash can be classified by calculation of the total weight percentage of SiO$_2$, Al$_2$O$_3$, and Fe$_2$O$_3$, ranged from 50% to 70% of the total content for class C and more than 70% for class F. According to the calculation, the Indonesian fly ash (IF) presented in this work is included in class C. Meanwhile, the Japanese fly ash (JF) can be classified to class F fly ash. Whereas sandblasting material has a total content of SiO$_2$, Al$_2$O$_3$, and Fe$_2$O$_3$ is about 99.31% and CaO is less than 10%.

Table 1. Variation of Alkali-activated Mixture Proportions

| Mixture | Symbols | Raw Material Proportions |
|---------|---------|--------------------------|
|         |         | Indonesian Raw Materials | Japanese Raw Materials |
|         |         | IF | Sb | JF | M | S |
| AAM1    | IF$_{100}$ | 100% | 0% | 0% | 0% | 0% |
| AAM2    | IF$_{50}$Sb$_{50}$ | 50% | 50% | 0% | 0% | 0% |
| AAM3    | JF$_{90}$M$_{10}$ | 0% | 0% | 90% | 10% | 0% |
| AAM4    | JF$_{50}$S$_{50}$ | 0% | 0% | 0% | 0% | 0% |
| AAM5    | JF$_{40}$S$_{40}$M$_{10}$ | 0% | 0% | 0% | 0% | 0% |
| AAM6    | JF$_{47.5}$S$_{42.5}$M$_{5}$ | 0% | 0% | 47.5% | 47.5% | 5% |
| AAM7    | JF$_{42.5}$S$_{42.5}$M$_{5}$ | 0% | 0% | 42.5% | 42.5% | 15% |
| AAM8    | JF$_{42.5}$S$_{42.5}$M$_{15}$ | 0% | 0% | 42.5% | 40% | 20% |
| AAM9    | JF$_{40}$S$_{40}$M$_{20}$ | 0% | 0% | 40% | 40% | 20% |

Note: IF Indonesian Fly ash, JF: Japanese fly ash, Sb: Sandblasting waste, M: Micro-silica, S: GGBFS

Table 2. The chemical composition of raw materials in weight percentage (wt.%) by XRF

| Material       | SiO$_2$ | Fe$_2$O$_3$ | Al$_2$O$_3$ | CaO  | Source            |
|----------------|---------|-------------|-------------|------|-------------------|
| Fly Ash (IF)   | 34.52   | 18.16       | 12.36       | 19.56| Indonesian Materials |
| Sandblasting (Sb) | 92.77   | 5.19        | 1.35        | 1.58 |                   |
| Sand           | 54.38   | 9.64        | 20.17       | 8.03 |                   |
| Fly Ash (JF)   | 55.19   | 7.57        | 25.35       | 4.06 | Japanese Materials |
| Micro silica   | 93.67   | 1.3         | 0.83        | 0.31 |                   |
| GGBFS          | 35      | 0.7         | 16          | 46   |                   |
| Standardized Sand | 98.4    | 0.36        | 0.41        | 0.16 |                   |
Class C fly ash particles are irregular round shapes with different particle sizes, as shown in SEM Fig 2 (a). Class F Japanese fly ash shapes are regular round, as shown in Fig 2 (c). Sandblasting particles, as shown in Fig 2 (b), have a similar shape with GGBFS, rough and sharp with square edges, as shown in Fig 2 (d). While, micro-silica particles are very small particles with a round shape, as displayed in Fig 2 (e).

Furthermore, in order to analyze the crystalline phases of fly ash X-Ray Diffraction technique was used, as shown in Table 3. It indicates that 44.088% of fly ash solid is amorphous. The amorphous fly ash is more reactive to dissolve Si and Al by alkaline solution and produces a stronger polymer [19-20]. The higher amount of amorphous phase of fly ash is an important factor to improve its mechanical and physical properties [21].

### Table 3. XRD of Class C Fly Ash

| Solids Particles | %     |
|------------------|-------|
| Quartz           | 8.493 |
| Arcanite         | 3.149 |
| Periclase        | 8.894 |
| Hematite         | 2.873 |
| Anyhydrite       | 3.545 |
| Magnetite        | 3.307 |
| Lime             | 1.061 |
| Magnesite        | 6.541 |
| Brownmillerite   | 14.656|
| Amorphous        | 44.088|
| R_wp             | 2.737 |

Fig 3 represents the XRD (X-Ray Diffractometer) sandblasting pattern. The XRD test showed the presence of crystalline solids, namely: Quartz, Hematite, and Mullite. These crystalline solids act as filler in a mortar, because they are non-reactive materials in geopolymer reactions [18].

The EDX (Energy Dispersive Spectroscopy) test of sandblasting can be seen in Fig 4. It indicates that solid has a peak of each element clearly, with Silica (Si) being the highest level of the element. Sandblasting solids in crystalline are constituent particles of material with regular atoms. The length and angle of the bond between the atom, the melting point and chemical properties are very clear [22].
Mortar specimens of 50 x 50 x 50 mm were used in UPV, compressive strength, and porosity tests, as shown in Fig. 5. The UPV test is a test to measure mortar density through conductivity of ultrasonic pulse velocity in a mortar. Tests were carried out at the 3rd, 14th, 28th and 56th day-old specimens, by air curing at room temperature.

3. RESULTS AND DISCUSSION

3.1 Setting Time

The setting time test of alkali-activated paste using apparatus tool as ASTM C191-07 results in a significant difference between the paste of AAM1 and AAM2. The 100% fly ash in the paste of AAM1 has a short setting time of around 15 minutes. While for the variation of 50% fly Ash + 50% sandblasting waste in the paste of AAM2 has a longer setting time, which is 75 minutes. It shows that the sandblasting corporation lengthen the setting time of alkali-activated fly ash. Furthermore, visual observation shows that the substitution of sandblasting also improves the workability of alkali-activated paste. This fact explains that the presence of different organic compounds in sandblasting waste has the same properties as cement water mixtures [12]. The shape of the sandblasting waste particles is irregular and larger than that of fly ash, as shown in Fig 2. This particle shape is predicted to be able to hold back and slow down the geo-polymerization process.

3.2 Compressive Strength

The development of the compressive strength of hardened alkali-activated paste is one of the basic indicators of geopolymers performance. The compressive strength of mortar specimens increases constantly. The highest compressive strength of mortar was achieved at 56 days: AAM1 (60.7 MPa) and AAM2 (29.2 MPa), as presented in Table 4. However, the compressive strength of AAM1 specimens is higher than those of the AAM2 specimens at four-age sets. The values of AAM1 compressive strength is about three times compared to those of AAM2 values in each of the four-age tests.

The higher percentage of sandblasting waste substitution results in an increase of the Si to Al ratio which causes a decrease of compressive strength. This fact fits what presented in [12]. The addition of sandblasting has a positive effect on the variation of compressive test results. It reduces the value of standard deviation, as shown in Table 4. The standard deviation of AAM1 specimens is classified as low. Classification of AAM2 specimens is medium, good and very good. It means AAM1 specimens have a larger variation of compressive strength results than those of AAM2, as shown in Table 4. By inclusion of sandblasting, the standard deviation of specimens becomes better. This is due to the large variation in heterogeneous elements contained in 100% fly ash (AAM1)
Table 4. Compressive Strength Test of AAM1 and AAM2-Mortars

| Mix   | Age Day | f'_c (MPa) Average | Standard Deviation | Classification |
|-------|---------|--------------------|--------------------|----------------|
|       | 3       | 17.9               | 4.47               | Low            |
| AAM1  | 14      | 46.3               | 4.46               | Low            |
|       | 28      | 54.1               | 5.59               | Low            |
|       | 56      | 60.7               | 4.77               | Low            |
|       | 3       | 7.5                | 1.83               | Good           |
| AAM2  | 14      | 17.8               | 0.86               | Very Good      |
|       | 28      | 19.3               | 2.51               | Medium         |
|       | 56      | 29.2               | 0.33               | Very Good      |

Compressive strength development of alkali-activated fly ash mortar is presented in Fig 7 and Fig 8. The 14-day compressive strength of AAM1 to AAM5 is shown in Fig 7. The lowest compressive strength of AAM is made from Class F Japanese fly ash (AAM3) which consisted of 90% fly ash, 10% micro silica and no GGBFS in mixtures. The GGBFS substitution on AAM4 can increase compressive strength. Substitution of reactive Si from micro-silica and Ca from GGBFS on AAM5 causes higher compressive strength than that of AAM3 at the 14-day age [17]. Class C fly ash mortar (AAM1) has a higher compressive strength than that of class F fly ash mortar (AAM3). Compared to AAM1 (100% Class C fly ash), substitution of 50% sandblasting waste on AAM2 reduces compressive strength of class C fly ash mortar, as shown in Table 4.

Furthermore, compressive strength results of 4 variations of AAM from 3 kinds of raw materials (class F fly ash, GGBFS and micro silica) at 28-day (AAM6, AAM7, AAM8 and AAM9) are 64, 66, 68 and 64 MPa respectively, as shown in Fig 8. The compressive strength of AAM6 to AAM9 at 14-days reached about 95-99% of those at 28-days. The compressive strength of AAM6 to AAM8 slightly increases, but slightly decreases at AAM9. This was caused by the mixture which contains various Ca and Si additions from admixtures. Performance of class C fly ash mortars has a lower compressive strength than those of class F fly ash mortars. It can be said, the need for different admixture for a different class of fly ash utilized for alkali-activated mortar or geopolymer mortar.

4. CONCLUSION

Alkali-activated class F fly ash, the paste time setting is very long. It requires an admixture. The combination of GGBFS, micro-silica on class F fly ash mortar improve mortar performance. However, alkali-activated class C fly ash has a fast setting time. Therefore, it needs different admixture.
Alkali-activated class C fly ash/sandblasting on AAM2 paste (50% class C fly ash + 50% sandblasting) shows a positive effect because it produces a longer setting time than that of AAM1 paste (100% class C fly ash). Sandblasting substitution also improves the workability of geopolymer. It is caused by high Si content on sandblasting. Particle shape and particle size are predicted to hold up the process of reaction. The replacement of sandblasting to class C fly ash also reduces the standard deviation of mortar specimens under the compressive test. However, sandblasting substitution has a negative impact, because it reduces compressive strength. The combination of waste sandblasting and class C fly ash shows better performance on geopolymer mortars. Thus, the addition of sandblasting is one of the solutions to solve the problem of setting time of AAM made from class C fly ash which has a fast set time.

The future research plan is to optimize the proportion of sandblasting replacement on class C fly ash to have better mechanical properties of alkali-activated concrete.

5. ACKNOWLEDGMENTS

This research conducted in Institut Teknologi Sepuluh Nopember in Indonesia and Kumamoto University in Japan. This work has been supported by Kumamoto University and by LPPM, Institut Teknologi Sepuluh Nopember and Ministry of Research, Technology and Higher Education of the Republic of Indonesia.

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