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Properties of Cementless Mortars Activated by Sodium Silicate

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Abstract

The present paper reports the testing of twelve alkali-activated mortars and a control ordinary portland cement (OPC) mortar. The main aim is to develop cementless binder activated by sodium silicate powder. An alkali quality coefficient combining the amounts of main compositions of source materials and sodium oxide (Na_2O) in sodium silicate is proposed to assess the properties of alkali activated mortars, based on the hydration mechanism of alkali-activated pastes. Fly ash (FA) and ground granulated blast-furnace slag (GGBS) were employed as source materials. The ratio of Na_2O -to-source material by weight for different mortars ranged between 0.038 and 0.164; as a result, alkali quality coefficient was varied from 0.0025 to 0.0365. Flow loss of fresh mortar, and shrinkage strain, compressive strength and modulus of rupture of hardened mortars were measured. The compressive strength development of alkali activated mortar was also compared with the design equations for OPC concrete specified in ACI 209 and EC 2. Test results clearly showed that the flow loss and compressive strength development of alkali-activated mortar were significantly dependent on the proposed alkali quality coefficient. In particular, a higher rate of compressive strength development achieved at early age for GGBS-based alkali-activated mortar and at long-term age for FA-based alkali-activated mortar. In addition, shrinkage strain and modulus of rupture of alkali-activated mortar were comparable to those of OPC mortar.

Keywords: alkali-activated mortar, sodium silicate, fly ash, ground granulated blast furnace slag, compressive strength, modulus of rupture, shrinkage strain.

1. Introduction

Extensive investigations on alternative inorganic materials which could replace conventional cement have been recently conducted to reduce greenhouse gas emissions from cement kilns. As a result, alkali-activated ground granulated blast-furnace slag (GGBS) cement or fly ash (FA)-based polymer cement have been investigated in many fields [1-8] to act as binder to produce cementless mortar or concrete. Based on extensive literature review, Wang et al. [7] concluded that alkali-activated cement using GGBS should be considered as an effective binder to produce better quality concrete than ordinary portland cement (OPC) concrete. In addition, the alkali-activated binder can offer a possible solution to deal with by-product materials such as GGBS and FA; around 49% of which is simply disposed in landfill [9].

The knowledge of the mechanism of alkali activation process is considerably advanced since the alkali-activated GGBS cement and concrete were invented in the USSR in 1957 by Glukhovskiy [10]. However, there are still many challenges to be tackled. For the alkali-activated cement, it is commonly known that calcined materials having abundant silicon oxide (SiO_2) and aluminum oxide (Al_2O_3) are more suitable as source materials [2, 3], and alkali hydroxide (ROH), non-silicic salts of weak acids (R_2CO_3 , R_2S , RF), or silicic salts of the $\text{R}_2\text{O}\cdot(n)\text{SiO}_2$ type can be the most effective activator [4, 7], where R indicates an alkali metal ion such as Na, K or Li. Wang et al. [6] pointed out that sodium silicate solutions (water glass solutions) of modulus in the range of 1-1.5 is the best formulation for good strength development of alkali-activated GGBS concrete. On the other hand, based on tests of alkali-activated low-calcium FA concrete, Hardjito et al. [2] concluded that a

combination of sodium hydroxide and sodium silicate solutions can be a good application for activator and higher concentration of sodium hydroxide solution and curing temperature enable the concrete compressive strength to be higher. However, alkali solutions based pastes would set within 15 mins as experimentally proved by several researchers [1, 3, 5, 7]. This would cause a quick workability loss of mortar or concrete against elapsed time, which would constitute an obstacle for practical applications. In addition, handling of sodium hydroxide solutions requires a great caution. These quick setting and risk handling problems should be solved to improve the use of alkali activated pastes as construction materials.

In the present study, twelve alkali-activated cementless mortars and a control OPC mortar were mixed and tested. To reduce the risk of handling of alkali-activated binder, powder typed sodium silicate was employed as an activator. FA and GGBS were used as source materials and the mixing ratio of sodium silicate powder was selected as the main variable. The variation of workability flow loss against elapse of time was recorded for fresh mortars. Compressive strength, modulus of rupture, and shrinkage strain were also measured in the hardened mortar. The properties of mortars tested were evaluated according to the alkali quality coefficient combining the amounts of main compositions in source materials and molar ratio of sodium silicate. In addition, compressive strength development of alkali-activated mortar tested was compared with the design equations specified in ACI 209 [12] and EC 2 [13], which are based on extensive test results of OPC concrete.

2. Review of binder activation mechanisms

The general understanding of the hydration mechanism of GGBS-based alkali-activated (AA) paste was advanced over the past five decades, although several important questions remain unanswered. The micro-structural development of GGBS-based AA pastes is greatly influenced by synthesis of the anions of GGBS and the cations in the alkaline activators. The chemical reaction by ion exchange between the silicate anions of GGBS and cations of alkaline activators induces the formation of silica gel. This silica gel may evolve into silica-rich calcium silicate hydrates gel (CSH gel) by further reacting with calcium ion of the GGBS, which is also the principal binding phase in the OPC pastes. Richardson and Groves [14] proposed a general model for the structure of substituted CSH gels in the OPC pastes blended with GGBS, where alumina element (Al) can substitute for silica element (Si) in the bridging tetrahedral of silica chain structures. In addition, some researchers [15] postulated the formation of a solid solution of $\text{Na}_2\text{O}-\text{CaO}-\text{SiO}_2-\text{H}_2\text{O}$ (NCSH), since Na^+ ions in alkali-activated cement have a very low solubility in water [16], although no convincing evidence for the presence of NCSH gels has been presented. Based on Taylor's theory [17] on the CSH gels in OPC pastes, Wang et al. [7] also proposed that a limited solid solution would be formed by partially replacing the cations such as Na and K of the activators into calcium ions of the GGBS.

On the other hand, hardening mechanism of FA-based AA paste is generally characterized as geopolymerisation of three-dimensional alumino-silicate materials, although evidence for the geopolymerisation mechanism is still insufficient [18, 19]. Xu and Deventer [18] explained the

geopolymerisation mechanism using ion-pair theory that the geopolymerisation involves a chemical reaction between various alumino-silicate oxides with silicates under highly alkaline conditions, indicating Si-Al elements would become sources of geopolymerisation. According to Davidovits [19] the polymerisation process requires highly alkaline solutions to dissolve the silica and alumina ions in the FA. Therefore, the amount of cations in the alkaline solutions, the extent of dissolution of Si and the molar Si-to-Al ratio in FA would be a significant factor to form geopolymerisations.

The above review plainly shows that the activation mechanisms for the formation of CSH gels in GGBS-based AA pastes and polymerisation in FA-based AA pastes are commonly influenced by the molar ratio of Si to Al in the total binder, organized by source materials and alkaline activators, and the concentration of the alkaline activators. In addition, the amount of Ca^{2+} ion in source materials would have some effect on the extent of the formation of CSH gels. Therefore, the mechanical strength of alkali-activated binders depends on a silicon oxide-to-sodium oxide ratio ($\text{SiO}_2/\text{Na}_2\text{O}$) in the alkaline activator, and a silicon oxide-to-aluminum oxide ($\text{SiO}_2/\text{Al}_2\text{O}_3$) ratio and calcium contents in the source material, which was also empirically proved by Davidovits [20].

GGBS and FA are commonly rich in silicon and aluminium (See Table 1 for their compositions used in the current investigations). In the alkali-activated binder using sodium silicate powder, therefore, an alkali quality coefficient Q_A combining the concentration of activator and main compositions in source materials influencing the activation mechanism can be simply proposed as follows:

$$Q_A = \left(\frac{Na_2O}{(SiO_2)^2} \cdot Al_2O_3 \cdot CaO \right) / B \quad (\text{by weight}) \quad (1)$$

where B = the total weight of binder organized by source materials, such as GGBS and FA, and alkaline activator, sodium silicate. Although Na₂O-to-source material ratio in alkali activated binder is an important parameter as proposed by Wang et al. [6], it cannot reflect the effect of the molar Si-to-Al ratio and the amount of Ca²⁺ ion in a source material on the mechanical strength of alkali-activated binder. Therefore, the proposed alkali quality coefficient would be a more effective index to evaluate the properties of alkali-activated mortar or concrete.

3. Experimental Details

3.1. Materials

Powder typed sodium silicate and either fly ash (FA) or ground granulated blast-furnace slag (GGBS), which can be commercially available worldwide, are employed as alkaline activator and source material, respectively, to produce cementless mortar. The chemical compositions of these materials evaluated by x-ray fluorescence (XRF) analysis are given in Table 1. The specific gravity and specific surface area of the three materials were also measured as explained below. Fly ash used as a source material had a low calcium oxide (CaO) and a silicon oxide (SiO₂)-to-aluminum oxide (Al₂O₃) ratio by mass of 1.91. On the other hand, the GGBS used had a high CaO and SiO₂-to-Al₂O₃ ratio by mass of 2.29, which is very similar to OPC.

The specific gravity and specific surface area were 2.2 and 4200 cm²/g, respectively, for FA and 2.9 and 4400 cm²/g, respectively, for GGBS. On the other hand, sodium silicate powder used for alkaline activator was a compound of sodium oxide (Na₂O) of 50.2 % and silicon oxide (SiO₂) of 45%, producing a molar ratio of 0.9. The sodium silicate powder had also a specific gravity of 1.2 and a maximum particle size of 425 μm.

Locally available sand was used as fine aggregate in saturated surface dry condition. The specific gravity, grading and maximum size of sand used were 2.54, 2.97 and 5mm, respectively.

3.2 Mix Proportions

Twelve alkali-activated mortar mixes and a control OPC mortar mix were prepared. Table 2 refers to the details of mixing proportions of different designed mortar. The designed 28-day compressive strength and initial flow of the control OPC mortar were 40 MPa and 150 mm, respectively. Water-binder ratio (W/B) of 50% and fine aggregate-binder ratio (S/B) of 3.0 were used for all mortar mixes tested. No water-reducing admixture was added to mortar mixes. According to Wang et al.'s proposal [6], the compressive strength of GGBS concrete activated by sodium silicate solution was significantly influenced by Na₂O-to-source material ratio by weight. Therefore, Na₂O-to-source material ratio was selected as a main variable to determine the addition amount of sodium silicate powder and design the mortar mixes. Na₂O-to-source material ratio in the present study ranged between 0.038 and 0.164, regardless of the kind of source material. The alkali

quality coefficient proposed by Eq. (1) was varied from 0.0025 to 0.0365 as given in Table 2 owing to different composition of each source material and the amount of added sodium silicate.

The mortar mix notation given in Table 2 includes two parts except for the control OPC mortar. The former part stands for the type of source material: FA for low-calcium fly ash and GGBS for high-calcium ground granulated blast-furnace slag, whereas the latter is used to identify the Na₂O-to-source material ratio. For example, FA-0.038 indicates an alkali activated mortar using low-calcium fly ash and a ratio of sodium oxide (Na₂O) in the added sodium silicate powder to FA of 0.038.

3.3. Casting, curing, and testing

The source material, sodium silicate and fine aggregate were dry-mixed in a mixer pan for 3 minutes, and then water was added and mixed in for another 3 minutes. After testing for the initial flow, each mix was poured in various steel moulds to measure compressive strengths, modulus of rupture and shrinkage strain. However, the mortar mix GGBS-0.164 was quickly set in less than 1 minute; therefore it was not possible to cast any specimen from this mix. Immediately after casting, all specimens were cured at a constant temperature and relative humidity of 23 ± 2 °C and $70 \pm 5\%$, respectively, until testing at the specified age. All steel moulds for OPC and GGBS-based AA mortars were removed at an age of one day. On the other hand, steel moulds for FA-based AA mortars were removed after three days of casting as they require more setting time.

Flow of different mortars was measured at 0 (initial), 30, 60, 90 and 120 minutes of mixing to ascertain flow loss against the elapse of time. Cubes of 50 mm in size were used for the evaluation of the mortar compressive strength in a Universal Testing Machine (UTM) with a capacity of 200 kN. Compressive strengths of mortars were measured at 1, 3, 7, 28, 56 and 91 days so that the strength gain with time would be investigated. The modulus of rupture of different specimens at age of 91 days was also measured using 150×150×450 mm prismatic beams under a symmetrical one-point top loading system. The test procedures of the flow and compressive strength were conducted according to the specifications of the Korean Industrial Standard [21] for testing OPC mortar. Shrinkage strains of each mortar were monitored using 100 mm waterproof electrical resistance strain (ERS) gages attached longitudinally at the center of the steel mould of 100 × 75 × 420 mm in size before casting mortar. After de-molding, shrinkage strains were measured using ERS gages attached to the hardened mortar. All shrinkage strain readings was recorded and saved automatically using a data logger.

4. Test results and Discussions

Table 3 gives the summary of test results of the variation of flow F of fresh mortars, and compressive strength f_c' at different ages and modulus of rupture f_r at age of 91 days of hardened mortars. Test results obtained from the present study are analyzed according to the alkali quality coefficient Q_A . Compressive strength development of AA mortar tested is compared with design equations for OPC concrete specified in ACI 209 [12] and EC 2 [13]. The modulus of

rupture of AA mortar is also normalized by the corresponding compressive strength to compare with the design equation of ACI 318-05 [22].

4.1 Initial slump

The initial flow F_i of AA mortar commonly decreased with the increase of Na₂O-to-source material ratio as given in Table 3. As a result, the initial flow F_i is inversely proportional to Q_A , indicating that the initial flow F_i of alkali-activated mortars having Q_A larger than 0.0325 would be lower than that of the control OPC mortar. In addition, lower F_i appeared in GGBS-based AA mortar than FA-based AA mortar.

4.2 Flow loss

The variation of flow F and relative flow F/F_i of AA mortar against the elapse of time are given in Table 3 and Fig. 1, respectively. The flow of GGBS-based AA mortars having Na₂O-to-source material ratio above 0.114 (GGBS-0.114, GGBS-0.139 and GGBS-0.164 specimens) after 30 minutes could not be measured owing to rapid setting. The flow of AA mortar is almost linearly decreased with the elapse of time. The decreasing rate is more notable in GGBS-based AA mortar than FA-based AA mortar, indicating that the flow loss of FA-based AA mortars was very similar to that of OPC mortar, regardless of Na₂O-to-source material ratio.

From Fig. 1, the relative flow of fresh mortars against elapsed time, which is valuable index to evaluate workability loss of mortar, can be approximately expressed as follows:

$$\frac{F}{F_i} = kT + 1 \quad (2)$$

where T = elapsed time in minute, and k = rate of flow loss.

Figure 2 presents the effect of the alkali quality coefficient Q_A on the rate of flow loss, k , of different mortar mixes tested which is determined by a linear regression analysis. The rate of flow loss increases with the increase of Q_A , indicating that higher Na₂O-to-source material ratio and the amount of CaO significantly reduce workability of fresh mortars with the elapse of time. From Table 3 and Fig. 2, therefore, Q_A in the AA mortar would need to be lower than 0.0325 for practical application.

4.3 Compressive strength at 28 days

Compressive strength at the age of 28 days (f'_c)₂₈ of GGBS-based AA mortars was generally much higher than that of FA-based AA mortars as given in Table 3. No significant compressive strength at 28 days was achieved in the FA-based AA mortar having Na₂O-to-source material ratio below 0.063. Hydration mechanism of alkali-activated GGBS paste induces the formation of CHS gel, whose mechanism is very similar to that of OPC cement, whereas that of alkali-activated FA paste can be distinguished by geopolymerisation with an amorphous microstructure. Microstructure of both OPC and GGBS-based AA mortars has very analogous characteristics as presented in Fig. 3 (a) and (c) illustrated by scanning electron microscope (SEM) image. Wang et al. [7] concluded that

GGBS-based AA mortar contains more gel pores and fewer capillary pores than OPC mortar, although both mortars are similar in the formation of CSH gels. On the other hand, microstructure of FA-based AA mortar reveals a porous microcracks and a amorphous microspheres surrounded by a crust of reaction products as shown in Fig. 3 (b). These imperfect microstructures of FA-based AA mortar would be one of the main causes of poor compressive strength.

The influence of Q_A on the 28-day compressive strength $(f'_c)_{28}$ of AA mortars is shown in Fig. 4. The $(f'_c)_{28}$ of AA mortar generally increases with the increase of Q_A up to a certain limit beyond which $(f'_c)_{28}$ remains constant, indicating that no meaningful increase of $(f'_c)_{28}$ developed in AA mortar having Q_A above 0.0352. In addition, the $(f'_c)_{28}$ of alkali-activated mortar having Q_A of 0.0274 is comparable to that of the control OPC mortar.

4.4 Compressive strength development

The 1-day compressive strength of FA-based AA mortar could not be measured as specimens were not hard enough to be tested. Xu and Deventer [18] proposed that the stable geopolymerisation process requires higher temperature to accelerate the hydrothermal synthesis reaction. In the present study, therefore, a slower reaction time in the geopolymerisation process at relatively low curing temperature would force the FA-based AA mortar to have lower compressive strength development at early stages. The early compressive strength of GGBS-based AA mortar was much higher than that of OPC or FA-based AA mortars as given in Table 3. However, the rate of compressive strength gain at long-term age of GGBS-based AA mortars was similar to that of OPC mortar. On

the other hand, the rate of compressive strength development at long-term age of FA-based AA mortars having Na₂O-to-source material ratio above 0.088 was very high, indicating that the higher the Na₂O-to-source material ratio, the better the compressive strength development at long-term age as presented in Table 3. In particular, the compressive strength of FA-0.164 specimen developed as much as 21 MPa at age of 91 days, in spite of using the alkaline activator in powder form under relatively low curing temperature.

ACI 209 [12] proposed the following relationship between the compressive strength development of concrete against age:

$$f'_c(t) = \frac{t}{A_1 + B_1 t} (f'_c)_{28} \quad (3)$$

where, $f'_c(t)$ = compressive strength at age t (in days). The constants A_1 and B_1 in Eq. (3) generally relate to the strength development at early age and at long-term age, respectively. The higher the value of A_1 , the lower the compressive strength at early age and the lower the value of B_1 , the higher the compressive strength at long-term age. ACI 209 specifies the two constants A_1 and B_1 of OPC concrete to be 4.0 and 0.85, respectively.

EC 2 [13] suggests the following expression for estimating the compressive strength development of concrete at age t (in days):

$$f'_c(t) = \text{Exp} \left\{ s \left[1 - \left(\frac{28}{t} \right) \right]^\alpha \right\} (f'_c)_{28} \quad (4)$$

where s and $\alpha = 0.25$ and 0.5 , respectively, for OPC concrete. The constant α determines the strength development at both early and long-term ages, indicating that the higher the value of

α , the lower the compressive strength at early age ($t < 28$ days) and the higher the compressive strength at long-term age ($t > 28$ days).

Figure 5 shows the comparison between the measured and predicted compressive strength development for different mortar specimens: Fig. 5 (a) for ACI 209 and Fig. 5 (b) for EC 2. The measured compressive strength was always higher than the predictions when Q_A was larger than 0.0196, agreeing with that the ratio between measured and predicted strengths increased with the decrease of age. On the other hand, for mortar specimens having Q_A less than 1.29, the strength measured was lower than that predicted at age below 7 days, but higher than that predicted with the increase of age. Based on Fig. 5 and Table 3, therefore, it can be concluded that the long-term strength development rate of FA-based AA mortar and the early strength development rate of GGBS-based AA mortar increases with the increase of Na_2O -to-source material ratio.

4.5 Modulus of rupture

The normalized rupture modulus $f_r / \sqrt{f'_c}$ of alkali-activated and OPC mortars against Q_A is presented in Fig. 6. The theoretical value of the modulus of rupture specified in ACI 318-05 [21] is also shown in Fig.6. FA-0.038 and FA-0.063 specimens were weak to the degree that they were fractured by their self-weight in the test set-up and, consequently, it would not be possible to test them in bending. A rupture modulus higher than $0.62\sqrt{f'_c}$ specified in ACI 318-05 exhibited by all specimens tested. The rupture modulus of AA mortars increased with the increase of Na_2O -to-source material ratio as given in Table 3. However $f_r / \sqrt{f'_c}$ of AA mortar is nearly independent

on Q_A . The $f_r / \sqrt{f'_c}$ of GGBS-based AA mortar having an average value of 1.3 is commonly much higher than that of both FA-based AA and OPC mortars. The $f_r / \sqrt{f'_c}$ value of FA-based AA mortar is similar to that of OPC mortar.

4.6 Shrinkage strain

Shrinkage strains of AA mortars against age are plotted in Fig. 7; Fig. 7 (a) for FA-series and Fig. 7 (b) for GGBS-series. On the same figure the shrinkage strain of the control OPC mortar is also presented. Most shrinkage strains in all mortar specimens occurred at the first 14 days and then the increasing rate of shrinkage strain was slowed down. The amount of shrinkage strain of AA mortar was higher than that of control OPC mortar until the age of about 14 days, but turned to be lower than that of OPC mortar with the increase of age. In general, the trend of shrinkage strain of GGBS-based AA mortars against time was very similar to that of OPC mortar. The shrinkage strain of the tested AA mortar was nearly independent on the type of source materials and Na₂O-to-source material ratio as shown in Fig. 7. Wang et al. [6] mentioned that higher shrinkage strain may be undergone in GGBS concrete activated by water glass than in OPC concrete due to the formation of silica-rich gel during hydration, while Hardjito et al. [2] showed that drying shrinkage strains of FA-based AA concrete were smaller than those of OPC concrete. Although inelastic strain of AA mortar is still controversy [7] owing to various influencing factors, the amount of shrinkage strain of AA mortars tested in the present study was slightly lower than that of OPC mortar.

5. Conclusions

Twelve alkali-activated (AA) cementless mortars and a control OPC mortar were mixed and tested. Fly ash (FA) and ground granulated blast-furnace slag (GGBS) used as source materials were activated by sodium silicate powder. The properties of mortar tested were examined according to the alkali quality coefficient combining the amounts of main compositions in source materials and sodium oxide (Na_2O) of sodium silicate. In addition, compressive strength development of AA mortars tested was compared with design equations specified in ACI 209 and EC 2. The following conclusions may be drawn:

1. The initial flow of AA mortar decreased with the increase of alkali quality coefficient, indicating that lower initial flow exhibited by GGBS-based AA mortar than in FA-based AA mortar.
2. The rate of flow loss increased with the increase of alkali quality coefficient. GGBS-based AA mortars having Na_2O -to-source material ratio above 0.114 started to set within 30 minutes of mixing.
3. The compressive strength of AA mortars at the age of 28 days increased with the increase of alkali quality coefficient up to a certain limit beyond which the 28-day compressive strength remained constant, indicating that no meaningful increase of the compressive strength developed in AA mortar having alkali quality coefficient above 0.0352.
4. Much higher compressive strength developed in GGBS-based AA mortars than in FA-based mortars. The 28-day compressive strength of GGBS-based AA mortar having alkali quality

coefficient of 0.0274 was comparable to that of the control OPC mortar.

5. The rate of compressive strength development of GGBS-based AA mortars was more notable at early age than long-term age. On the other hand, compressive strength of FA-based AA mortar sharply increased with the increase of age, although the compressive strength development in the first 7 days was very low.

6. The long-term strength development rate of FA-based AA mortars and the early strength development rate of GGBS-based AA mortars increased with the increase of Na₂O-to-source material ratio.

7. The measured compressive strength was always higher than the predictions obtained from design equations specified in ACI 209 and EC 2 when alkali quality coefficient was larger than 0.0196. On the other hand, for the FA-based AA mortars, the measured compressive strength was higher than predictions from ACI 209 and EC 2 at age above 7 days.

8. The rupture modulus of AA mortars increased with the increase of alkali quality coefficient. However, the normalized rupture modulus of AA mortars is nearly independent on the alkali quality coefficient, indicating the normalized rupture modulus of GGBS-based AA mortars having an average value of 1.3 was commonly much higher than that of both FA-based AA and OPC mortars.

9. The amount of shrinkage strain of AA mortar was higher than that of control OPC mortar in the first 14 days, but turned to be lower than that of OPC mortar with the increase of age. The trend of shrinkage strain of the tested AA mortar was nearly independent on the type of source materials and Na₂O-to-source material ratio.

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