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Strength Development and Durability of Alkali-Activated Fly Ash

Mortar with Calcium Carbide Residue as Additive

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Abstract

    The strength development and durability of alkali-activated fly ash (FA) mortar with
calcium carbide residue (CCR) as additive cured at ambient temperature were investigated in
this paper. CCR was used to partially replace FA as additional calcium in the alkali-activated
binder system by a weight percentage of 0%, 10%, 20% and 30%. Sodium hydroxide and
sodium silicate solutions were used as liquid alkaline activation in all mixtures. Test results
show that the incorporation of CCR has an effect on the strength development of alkali-
activated FA mortar with CCR. The setting time of alkali-activated FA mortar with CCR has

decreased whereas its strength development has increased. This is further confirmed by XRD, SEM, and FTIR analyses, which show that the reaction products were increased when the alkali-activated FA incorporated with CCR. The highest 28-day compressive strength of alkali-activated FA mortar was found in the mix of 70% FA and 30% CCR, which is about 40.0 MPa. In addition, the resistances of alkali-activated FA mortar incorporated with CCR to tap water, 5% H_2SO_4 solution, and 5% $MgSO_4$ solution are found to be superior to those of alkali-activated FA mortar without CCR as indicated by the relatively low strength loss. For the samples immersed in 5% H_2SO_4 solution and 5% $MgSO_4$ solution for 120 days, the alkali-activated FA incorporated with 30% CCR showed a low strength reduction of around 71% and 53%, respectively.

Keywords: Alkali-activated Fly ash, Calcium carbide residue, Strength development, Microstructure, Durability.

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1. Introduction

Nowadays, Portland Cement (PC) is still widely used for construction work although its manufacturing produces a significant amount of greenhouse gas. It was reported that, about one ton of carbon dioxide is created for every one ton PC produced. To solve this problem, attempts have been made in recent years [1, 2] to find alternative cementitious materials to replace PC in concrete. One of these alternative cementitious materials is the alkali-activated binder which uses sodium hydroxide and sodium silicate solutions [3]. The alkali-activated binder has received great attention in recent years due to its low carbon dioxide emission although it has some disadvantages. According to the reports of Turner and Collins [4] and Teh et al. [5], the low carbon footprint of the alkali-activated bind is only when it is used with sodium hydroxide solution [6].

Alkali-activated binder is made from aluminosilicate materials such as fly ash (FA), calcined kaolin and blast furnace slag, activated with high alkali solutions [7]. Alkali-activated binder has emerged as one of the possible alternative cements to PC binder because it has an excellent properties, e.g., high compressive strength, low shrinkage, and good durability against chemical attacks [1-3]. In Thailand, FA from Mae Moh power station is suitable to be a good source material for making alkali-activated binder [8-10]. However, this FA needs a temperature curing at around 40 to 75°C for improving the degree of geopolymerization, though its strength when cured at ambient temperature is sufficiently used in construction work as reported by researchers [9, 11]. Although alkali-activated FA cured at ambient temperature could be used in real construction, the mechanical properties and durability of alkali-activated FA are generally not good in terms of strength, shrinkage and durability against chemical attacks. To improve these properties, some additives have been used to help the geopolymerization of alkali-activated FA. One of such additives is the material that consists of calcium oxide [12-14]. For example, Pangdaeng et al. [15] investigated the use of PC to replace alkali-activated high calcium FA for making alkali-activated binder under different curing conditions. They reported that the CaO from PC could improve the properties of alkali-activated high calcium FA due to additional reaction products within the matrix. Phoo-ngernkham et al. [16] investigated the properties of alkali-activated high calcium FA paste with PC as additive. They found the reactive CaO from PC is essential for the strength development of alkali-activated binder, which is consistent with what was reported by Pangdaeng et al. [15]. Many researchers [15-21] claimed that an exothermal process at ambient temperature from PC and water is important for accelerating the geopolymerization within the matrix. Also, coexistences of calcium silicate hydrate (C-S-H) and sodium aluminosilicate hydrate (N-A-S-H) gels resulted in a high strength developed in the alkali-activated binder [14]. Recently, FA-based alkali-acivated binder has been

considered as a sustainable future construction material. For example, recycled asphalt pavement (RAP) and FA were used to produce a road construction material [22]. Alkali-activated FA incorporated with slag was used to stabilize the pavement base/subbase applications [23]. More recently, novel low-carbon masonry units by using alkali-activated FA with recycled glass were developed by Arulrajah et al. [24].

Over the past few years, calcium carbide residue (CCR) has been very attractive for using as a promoter similar to the use of PC because it has rich calcium hydroxide ($Ca(OH)_2$) [25]. CCR is a by-product of acetylene production process through the hydrolysis of calcium carbide (CaC_2) regarded as a sustainable cementing agent [26-29]. In Thailand, approximately 21,500 tons CCR is produced annually and is mainly disposed in landfills, which causes a huge local environmental problem due to its high alkalinity [30]. Currently, some researchers have used the CCR with rice husk ash [31], bagasse ash [32], and FA [33-36] as new cementitious materials used in construction work. This is because its main reaction product is calcium silicate hydrate (C-S-H), which is similar to the hydration products of PC. For example, a combination of CCR and FA without PC has been used by Makaratat et al. [36] for producing concrete, and for improving the strength characteristics of silty clay and soil [34, 35]. Somna et al. [37] reported that CCR-FA blends at the ratio of 30:70 as a binder without PC gave sufficient strength in construction work and also had the reaction products similar to the pozzolanic reaction. Other by-products have been also used as the alkali-acivated binder. For instance, Arulrajah et al. [38] investigated recycled demolition aggregates such as the recycled concrete aggregates and crushed brick, stabilized by alkali activation of CCR with supplementary components of FA and slag for pavement base/subbase applications. Phummiphan et al. [39] studied the sustainable pavement based material made from marginal lateritic soil stabilized with alkali-acivated FA with CCR, in which CCR replacement was recommended for low sodium hydroxide system because

calcium hydroxide from CCR could enhance the strength development of alkali-activated binder similar to FA-PC blends activated with alkali solutions. Note that the use of CCR in construction materials is not only helping the strength development of the materials; but more importantly, it has the economic and environmental perspectives. Phetchuay et al. [40] [41] have used a combination of CCR and FA as a precursor for making alkali-activated binder to stabilize strength development in soft marine clay. Hanjitsuwan et al. [25] presented a comparative study by using PC and CCR as a promoter in alkali-acivated bottom ash (BA) mortar. It was found that the use of CCR to replace BA provided high compressive strength of alkali-activated BA mortar similar to the use of PC. However, the FA with PC provided more reaction products and the geopolymerization degree was better than that of CCR.

In regard to the durability of alkali-activated binders, several researchers [42-46] reported that the alkali-activated binder has excellent resistance to sulfate and acid, which is superior to that of normal PC mortar and concrete. For example, Bakharev [43] did the 5-month resistance test of alkali-activated low calcium FA to 5% sulfuric acid. Ariffin et al. [42] did the one-year resistance test of alkali-activated binder concrete made from pulverized flue FA and palm oil fuel ash to 2% sulfate acid. Sata et al. [47] did the resistance test of alkali-activated BA mortar to sodium sulfate and sulfuric acid solutions. It was reported that alkali-activated BA under different particle sizes showed less susceptible to the attack by sodium sulfate and sulfuric acid solutions than that of PC mortar. Chindaprasirt et al. [48] reported that the alkali-activated binders made from ground fluidized bed combustion FA and silica fume have both good strength and resistance to sulfate and sulfuric acid attack. In addition, Chindaprasirt et al. [49] also investigated the resistance of microwave-assisted alkali-activated high calcium FA to acid and sulfate attack. However, in the literature the durability of alkali-activated FA with CCR has not been discussed. In this paper the mechanical and durability properties of alkali-activated FA mortar with CCR as an additive

were investigated using various different experimental methods. The work includes the setting time test, compressive strength test and immersion test of alkali-activated FA mortar incorporated with CCR, while the material characterization of alkali-activated FA paste incorporated with CCR was analysed using XRD, SEM, and FTIR techniques. The experimental details are described in Section 2 and corresponding results are presented in Section 3. The results show that the binder made from alkali-activated FA incorporated with CCR has the advantages of high compressive strength, short setting time and good resistance to H_2SO_4 and $MgSO_4$ attack. It can potentially be used, for example, as the binder for the precast structural members, pavement of roadworks, and repair materials for replacing the damaged concrete in existed RC structures.

2. Experimental details

2.1 Materials

The precursors used in this study are the lignite coal FA and CCR. The FA is the by-product from the Mae Moh power plant in northern Thailand with a specific gravity of 2.65 and the mean particle size of 15.6 micron, respectively. The CCR is the by-product from acetylene gas process in Sai 5 Gas Product Co., Ltd. It was oven-dried at 100°C for 24 hours and then ground by a Los Angeles abrasion machine. After that, it was passed through a sieve No. 100 (150 µm) before used as the precursor. The specific gravity and mean particle size of CCR are 2.25 and 21.2 micron, respectively. Table 1 summarizes the chemical compositions of the FA and CCR used in the present experimental work, which were determined using X-ray Diffractometry (XRD) analysis. Note that the FA mainly contains silica (SiO_2), alumina (Al_2O_3), iron oxide (Fe_2O_3), and lime (CaO). ASTM C618 [50] classifies FA into two categories: Class F and Class C. Class F FA is produced from anthracite and bituminous coal and contains less than 5-10% lime. It is essentially pozzolanic material, meaning it does

not react with water on its own, but reacts with $Ca(OH)_2$, a byproduct of cement hydration reaction, in the presence of water to give calcium silicate hydrates. In contrast, Class C FA is typically derived from lignite and subbituminous coal and contains a high amount of lime (15-30%). Apart from its pozzolanic property, Class C FA has a hydraulic (cementitious) property as well. It can react with water on its own to form calcium silicate hydrates. The major components (SiO_2, Al_2O_3 and Fe_2O_3) of the CCR are 9.88% with high $Ca(OH)_2$ and/or CaO contents at 89.24%. Figure 1 shows the mineral compositions of the FA and CCR as determined by XRD analysis. The FA consists of hump peak around 20-40° 2theta and crystal of quartz and ferric oxide, whereas the CCR consists of crystal phase of calcium hydroxide, quartz, calcium carbonate and aluminite. The fine aggregate used in this study is the local river sand (RS) with a specific gravity of 2.63 and fineness modulus of 2.40.

2.2 Sample preparation of alkali-activated FA mortar with CCR

The mix proportions of alkali-activated FA mortar with CCR as additive used in this study are summarized in Table 2. The alkali-activated FA with CCR sample is a combination of FA, CCR and liquid alkaline activator, i.e., commercial grade sodium silicate (Na_2SiO_3) with 13.45% Na_2O, 32.39% SiO_2, 54.16% H_2O and 10 molar sodium hydroxide (NaOH). According to Table 2, the FA:CCR ratios were fixed at 100:0 (control), 90:10, 80:20 and 70:30; while the Na_2SiO_3/NaOH ratio, liquid alkaline activator/binders ratio, and fine aggregate/binders ratio were kept unchanged in all mixtures. For the mixing of alkali-activated FA mortar incorporated with CCR, precursors with and without fine aggregate were dry mixed first until the mixture was almost uniformly mixed, which took approximately one minute. Liquid alkaline activators were then added into the mixture and the mixing was continued again for another three minutes.

2.3 Testing procedure of alkali-activated FA mortar with CCR

The setting time of the alkali-activated FA mortar with CCR was tested using Vicat apparatus as per ASTM C191 [51]. The fresh alkali-activated FA mortar with CCR was placed into a 50x50x50 mm^3 cube molds and compacted as per ASTM C109 [52]. After that, all samples were wrapped with vinyl sheet to protect moisture loss. They then were cured for a day and then demolded with immediately wrapped by vinyl sheet and kept in ambient temperature until testing age. The compressive strengths of the samples were tested at the ages of 1, 3, 7, 28 and 90 days. Five identical samples were tested for each case and their average result was used as the reported experimental result.

After 28 days of curing time, the alkali-activated FA pastes with CCR were broken into small fragments approximately 3-6 mm for observation in growth of the reaction products via Scanning Electron Microscopic (SEM) analysis. Furthermore, the pastes were also ground as fine powder for XRD and Fourier Transform Infrared Spectroscopy (FTIR) analyses. The XRDs were performed for 2 theta from 10 and 60° [25], whereas the FTIRs were conducted in the wave number region of between 400 and 4000 cm^{-1}. For the FTIR analysis, the powder specimens were mixed with KBr at the concentration of 0.2–1.0 wt% to make the KBr disks. Then the disks were tested using a FTIR spectrometer.

For the durability test, the alkali-activated FA mortars incorporated with CCR after 28 days of curing time were then immersed into tap water (H_2O), 5% sulfuric acid (H_2SO_4) solution, and 5% magnesium sulfate ($MgSO_4$) solution. The durability test of alkali-activated FA mortars incorporated with CCR was modified from ASTM C267-01 [53]. After the 30, 60, 90 and 120 days immersion, the retained compressive strengths of the alkali-activated FA mortars with CCR were tested. Again, five identical samples were tested for each case and their average result was used as the reported experimental result.

3. Results and Discussion

3.1 Setting time and compressive strength

Setting time is one of the important characteristics of binder materials. Figure 2 summarizes the results of the setting time and the compressive strength of the alkali-activated FA mortars incorporated with CCR. The final setting time and the compressive strength were found to vary with the amount of CCR replacement. It can be observed from Figure 2 that, the final setting time tends to decrease whereas the compressive strength tends to increase with the increase of CCR replacement. For example, the final setting times of FA:CCR ratios at 100:0, 90:10, 80:20 and 70:30 are 125, 85, 35 and 25 min, respectively; whereas the 28-day compressive strengths of them are 35.6, 41.8, 44.4, 45.8 MPa, respectively. As reported, the FA:CCR ratios at 80:20 and 70:30 meet the initial setting time and 7-day strength requirements for repair binder, which were 30 minutes and 35.0 MPa, respectively as per ASTM C881/C881M-14 [54]. Also, they meet the 7-day strength requirement of rapid-hardening mortar that was 28.0 MPa as per ASTM C928-13 [55]. In addition, only 1-day compressive strength of alkali-activated FA mortar with CCR meet the 28-day strength requirement for non-load-bearing and load-bearing brick masonry units as per ASTM C129-01 [56] and ASTM C90-02a [57]. These results appear consistent with what was reported by Pangdaeng et al. [15] and Hanjitsuwan et al. [25], which demonstrates that the CaO from PC and CCR could accelerate the geopolymerization within the matrix. The fast setting time of alkali-activated FA when incorporated with CCR could be an advantage in the conditions where a speeding setting is required, whereas in some applications too short of setting time could have a negative effect. As a comparison with the previous research, the final setting time of alkali-activated FA with CCR at the ratio of 70:30 obtained from this study (25 min) is faster than that of the alkali-activated BA with CCR at the ratio of 70:30 (35 min) [25]. Note that both the FA and BA consist of SiO_2 and Al_2O_3. However, as reported by

Chindaprasirt et al. [58], the dissolution of BA in NaOH solution would be lower than that of FA. Hence, in general, BA requires to have a high concentration of NaOH for dissolving SiO_2 and Al_2O_3 to make geopolymerization [58]. Similarly, the strength development of alkali-activated FA mortar with CCR at the ratio of 70:30 is higher than that of alkali-activated BA with CCR, but it is lower than alkali-activated FA mortar with PC at the same ratio of 70:30 [25]. Chindaprasirt et al. [58] explained that the lower strength of alkali-activated BA was mainly due to the BA particles which were much porous. Also, it is noticed from Figure 2 that the strength development of mortars tends to increase with the curing time. This implies that the present of $Ca(OH)_2$ from CCR in the system could react with silica and alumina from FA, resulting in additional formation of C-S-H co-existed with N-A-S-H gels as reported by Hanjitsuwan et al. [25] and Gue et al. [12]. The continuous strength development of alkali-activated FA mortars with CCR at later stage is a very attractive property when compared with some alkali-activated binders that exhibit strength reduction at the later stage [59].

3.2 XRD, SEM and FTIR analyses

Figure 3 shows the XRD plot of alkali-activated FA pastes with CCR as additive. The control mix (Figure 3a) consists of amorphous phase at the hump around 25-35° 2theta and crystalline phases of quartz (SiO_2), ferric oxide (Fe_2O_3), and calcium silicate hydrate (C-S-H). The presence of amorphous phases are generally well corresponding to the coexistence of C-S-H and N-A-S-H gels as reported by Hanjitsuwan et al. [10] and Garcia-Lodeiro et. al. [16]. As mentioned earlier, additional formation of C-S-H is essential for increasing strength development of alkali-activated binders similar to the reaction products from PC hydration. When both the FA and CCR are present in the mixture, the patterns exhibit broad hump peak, quartz, ferric oxide (Fe_2O_3), calcium silicate hydrate (C-S-H) and calcium carbonate ($CaCO_3$).

The SEM images of alkali-activated FA paste incorporated with CCR cured at ambient temperature for 28 days are displayed in Figure 4. The image of the control sample (Figure 4a) shows a number of non-reacted and/or partially reacted FA particles embedded in the matrix, resulting in a relatively low reaction degree of geopolymerization. Chindaprasirt et al. [11] claimed that the degree of geopolymerization at ambient temperature of alkali-activated high calcium FA is low; therefore, higher temperature curing could accelerate the strength development of alkali-activated high calcium FA as reported by Pangdaeng et al. [15]. When alkali-activated FA was mixed with 10% CCR (Figure 4b), the paste shows denser than the control one and the former is with less number of non-reacted and/or partially reacted FA particles in the matrix. Noticeable difference is observed in the 80FA20CCR (Figure 4c) and the 70FA30CCR (Figure 4d) mixes. Their SEM images appear denser and more uniform than those of the control and 90FA10CCR mixes. The increase of CCR replacement in the mix could enhance the reaction products within the matrix. The reaction of CCR with alkali solutions is an exothermal process and can liberate heat and hence the additional formation of C-S-H (see Figure 3), resulting in a better overall strength development (see Figure 2) and a shorter setting time.

FTIR spectroscopic was used to study the geopolymerization degree of alkali-activated FA pastes with CCR cured at ambient temperature for 28 days as illustrated in Figure 5. All samples of FTIR analysis are found in the range between 400 and 4000 cm^{-1}. Both of spectra band and wave number in Figures 5a to 5d had little change because the FA could react with CCR in an alkali system. According to Figure 5, the wave number was divided into four groups. First one is located at approximately 3450 and at between 1650-1600 cm^{-1} that is the O-H stretching and O-H bending of water molecule [58, 60]. Second one is located at approximately 1400-1450 cm^{-1} that is the carbonate (CO_3^{2-}) band [43-44]. According to Figures 5b-5d, the alkali-activated FA pastes incorporated with CCR are easily identified to

have the carbonate (CO_3^{2-}) band which is related to the calcium carbonate phase in XRD results (see Figure 3). Third one is located at between 1200 and 950 cm^{-1} that is the Si-O-Si and Si-O-Al stretching vibration [61, 62]. Alvarez-Ayuso et al. [61] and Barbosa et al. [62] claimed that the geopolymerization generally occurred in the matrix with this wave number. Also, Chindaprasirt et al. [58] explained that this wave number is essential for strength development of alkali-activated FA with CCR. The final one is located at approximately 460 cm^{-1} that is the O-Si-O bending mode. This wave number indicates the remain part of the unreacted quartz in the matrix [63], which is related to the quartz in XRD results. The results of FTIR spectra analysis shown herein correspond very well to the previously described results for the compressive strength, XRD and SEM analyses.

3.3 Durability

The results of alkali-activated FA mortars incorporated with CCR after immersion in H_2O, 5% H_2SO_4 solution and 5% $MgSO_4$ solution for 30, 60, 90 and 120 days, in terms of the retained compressive strength, the percentage of strength loss, and weight change of specimens are shown in Figures 6 and 7, and Table 3, respectively. The strengths of alkali-activated FA mortars incorporated with CCR after immersion in the H_2O for 30 days were found to be higher than their strengths before immersion, especially in the 80FA20CCR and 70FA30CCR mixes. This is probably due to the $Ca(OH)_2$ that could react with SiO_2 and/or Al_2O_3 in the suitable humidity to form C-S-H gels in the similar way as the pozzolanic reaction [64]. After exposure to H_2O for 60 days, the strengths of alkali-activated FA mortars incorporated with CCR are still higher than their strengths before immersion. When the samples immersed in H_2O for 90-120 days, the control, 90FA10CCR, and 80FA20CCR mixes have lower strengths than them before immersion. However, there is one exception, which is the 70FA30CCR mix that still has higher strength than it before immersion although

its strength after the 30 days immersion decreases with the immersion time (see Figure 7). This result is consistent with the physical appearance of alkali-activated FA mortar with CCR after exposure to H_2O for 30 and 120 days (see Figure 8), in which the intact surface of samples is in a good agreement with the result of their strength.

Note that the H_2SO_4 solution is a strong acid and readily attacks calcium compound and other calcareous materials in concrete as reported by Chindaprasirt et al. [49] and Sata et al. [47]. For the samples immersed in the 5% H_2SO_4 solution, the strengths of alkali-activated FA mortars with CCR slightly reduced when compared to their strength before immersion. This behavior corresponds to the physical appearance of the samples as illustrated in Figure 9, which shows the alkali-activated FA mortars with CCR was slightly damaged at its surfaces. As can be seen in the figure, the retained strengths of 70FA30CCR mortar after exposure to 5% H_2SO_4 solution for 30, 60, 90 and 120 days are 45.73, 43.20, 40.00 and 37.17 MPa, respectively; indicating the strength loss at approximately -0.15%, -5.69%, -12.66% and -18.33%, respectively. According to Figure 7, the strength of alkali-activated FA mortar with CCR after immersion in the 5% H_2SO_4 solution for 30 days exhibits a slight decrease compared to its strength before immersion. After exposure to 5% H_2SO_4 solution for 60, 90 and 120 days, however, its strength loss becomes more obvious and higher. Similar results were reported by Sata et al. [47] for alkali-activated BA mortar (cured at temperature of $75^{o}C$ for 48 hours) which, after exposure to 3% H_2SO_4 solution for 120 days, exhibited a low strength loss. Mehta and Siddique [65] also reported that the strength loss of alkali-activated FA concrete with PC as additive after immersion in 2% H_2SO_4 solution for 28 and 90 days was between -15% and -28% and between -30% and -45%, respectively. In addition, Chindaprasirt et al. [49] studied the compressive strength of alkali-activated high calcium FA under 3% H_2SO_4 solution attack. They reported that after immersion in the H_2SO_4 for 3 months there was a slight strength decrease. Note that the strength loss of alkali-activated FA

with CCR after exposure to H_2SO_4 could have two reasons. One is related to the reaction of $Ca(OH)_2$ from CCR and high calcium from FA with H_2SO_4. The other is related to the reaction between C-S-H and H_2SO_4, as reported by Chindaprasirt et al. [49] and Neville [66]. The reaction of $Ca(OH)_2$ and H_2SO_4 generates the gypsum ($CaSO_4.2H_2O$) within the matrix; whereas the reaction of C-S-H and H_2SO_4 produces both the gypsum ($CaSO_4.2H_2O$) and amorphous silica gels, which become as ettringite in later stage [49, 66], causing the expansion and/or cracking of the matrix and thus a loss of compressive strength.

The compressive strength of alkali-activated FA mortar with CCR after immersion in 5% $MgSO_4$ solution for 30, 60, 90 and 120 days was found to reduce with the immersed time, which is similar to the result of the specimen immersed in the 5% H_2SO_4 solution. However, by comparing the results shown in Figures 6 and 7, one can see that the resistance of alkali-activated FA mortars with CCR is lower to the 5% $MgSO_4$ solution than to the 5% H_2SO_4 solution since the strength in the former is lower than the strength in the latter. For instance, the strength loss of the 70FA30CCR mortar after exposure to 5% $MgSO_4$ solution for 30, 60, 90 and 120 days is -21.93%, -28.66%, -32.95% and -46.77%, respectively; whereas the strength loss of the 70FA30CCR mortar after exposure to 5% H_2SO_4 solution for 30, 60, 90 and 120 days is 0.15%, -5.69%, -12.66% and -18.33%, respectively. This seems to be in line with the results reported by Chindaprasirt et al. [49], who investigated the resistance of microwave-assisted alkali-activated high calcium FA to acid and sulfate solutions. Note that many researchers [47, 49, 67] claimed that the magnesium hydroxide ($Mg(OH)_2$) and gypsum ($CaSO_4.2H_2O$) were obtained by the reaction between $Ca(OH)_2$ and $MgSO_4$ or between C-S-H and $MgSO_4$ within the matrix, leading to the deposition on the material surface. This is also demonstrated by the result shown in Figure 10, in which a white substance, known as 'efflorescence', appears on the surface and then the mortars were destroyed. Similar results were reported by Chindaprasirt et al. [49] in which the degradation of C-S-H phase after

exposure to 3% $MgSO_4$ solution gave faster than that of other sulfate, which leads to low strength.

4. Conclusion

This paper presents an experimental study on the setting time, compressive strength and durability of alkali-activated FA mortars with CCR, and the microstructure of alkali-activated FA pastes with CCR. Based on the obtained experimental results, the following conclusions could be drawn:

1) The use of CCR to partially replace FA for making alkali-activated mortar can reduce the setting time. The final setting time of the mortar with CCR is between 25 and 85 min, whereas the mortar without CCR is about 125 min. The faster setting time of alkali-activated FA mortars incorporated with CCR could be an advantage for their use in the conditions where fast setting is required.

2) The main reaction products of alkali-activated FA pastes incorporated with CCR are the amorphous phase, C-S-H and calcite. This was confirmed by the SEM images in which the pastes with CCR show denser and more uniform than the pastes without CCR. The relatively high compressive strength of alkali-activated FA mortar with 30% CCR at curing times of 7 and 28 days are 40.0 and 45.8 MPa, respectively.

3) After exposure to H_2O, the use of CCR replacement could improve the strength development of alkali-activated FA mortar at early-stage. However, a strength reduction was found at later stage except in mortar with CCR of 30%.

4) The alkali-activated FA mortar incorporated with CCR shows an excellent performance to the attack of the 5% H_2SO_4 solution when compared to the mortar without CCR. The deterioration of the alkali-activated mortar after exposure to 5% H_2SO_4 solution is

due to the reactions of Ca(OH)$_2$ or C-S-H with H$_2$SO$_4$ solution to form gypsum (CaSO$_4$.2H$_2$O) within the matrix, which causes a loss in compressive strength.

5)   The deterioration of alkali-activated FA mortar incorporated with CCR is severer to the attack of 5% MgSO$_4$ solution than to the attack of H$_2$SO$_4$ solution; the former leads to a high loss in the strength of mortars. This is because the magnesium hydroxide (Mg(OH)$_2$) and gypsum (CaSO$_4$.2H$_2$O) were generated by the reactions of Ca(OH)$_2$ or C-S-H with MgSO$_4$ within the matrix.

Acknowledgements

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Table 1 Chemical compositions of FA and CCR (by weight)

Materials	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	P_2O_5	SO_3	LOI
FA	31.32	13.96	15.64	25.79	2.94	2.93	2.83	-	3.29	1.30
CCR	6.16	3.54	0.18	89.24	0.37	-	-	0.50	0.87	2.79

Table 2 Mix proportions of alkali-activated FA mortar incorporated with CCR

Symbols	FA (kg)	CCR (kg)	RS (kg)	NaOH (kg)	Na_2SiO_3 (kg)
Control	1.0	-	1.5	0.233	0.467
90FA10CCR	0.9	0.1	1.5	0.233	0.467
80FA20CCR	0.8	0.2	1.5	0.233	0.467
70FA30CCR	0.7	0.3	1.5	0.233	0.467

Table 3 Weight change of alkali-activated FA mortars with CCR after immersion in H_2O, 5%$MgSO_4$, and 5%H_2SO_4 solutions

| Mixes	Weight change of alkali-activated FA mortars with CCR after immersion test (%)											
	30 days			60 days			90 days			120 days		
	H_2O	H_2SO_4	$MgSO_4$	H_2O	H_2SO_4	$MgSO_4$	H_2O	H_2SO_4	$MgSO_4$	H_2O	H_2SO_4	$MgSO_4$
Control	-1.50	-2.36	-3.33	-2.00	-2.87	-4.00	-2.60	-3.00	-4.35	-3.00	-3.30	-4.55
90FA10CCR	-1.24	-2.13	-3.00	-1.70	-2.30	-3.80	-2.20	-2.55	-4.00	-2.58	-2.96	-4.30
80FA20CCR	-1.00	-1.36	-2.79	-1.25	-1.56	-3.26	-1.90	-2.20	-3.75	-2.15	-2.45	-4.00
70FA30CCR	-0.52	-0.85	-2.38	-1.00	-1.12	-3.00	-1.50	-1.72	-3.20	-1.80	-2.22	-3.60
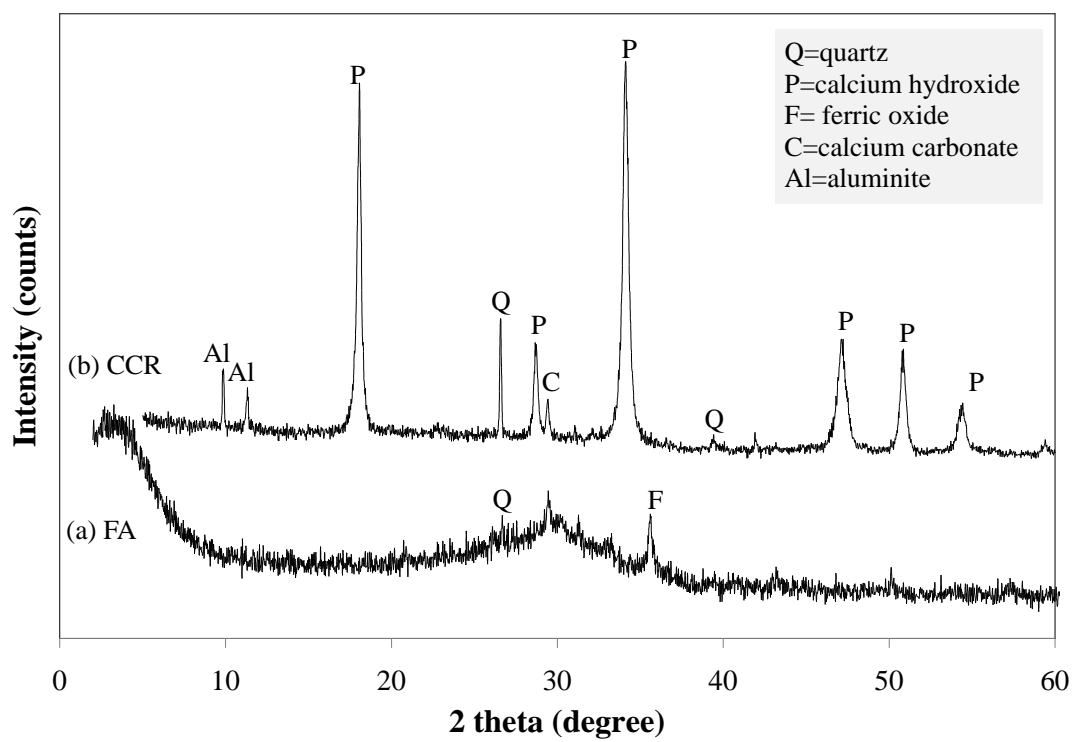
Figure 1 XRD patterns of FA and CCR
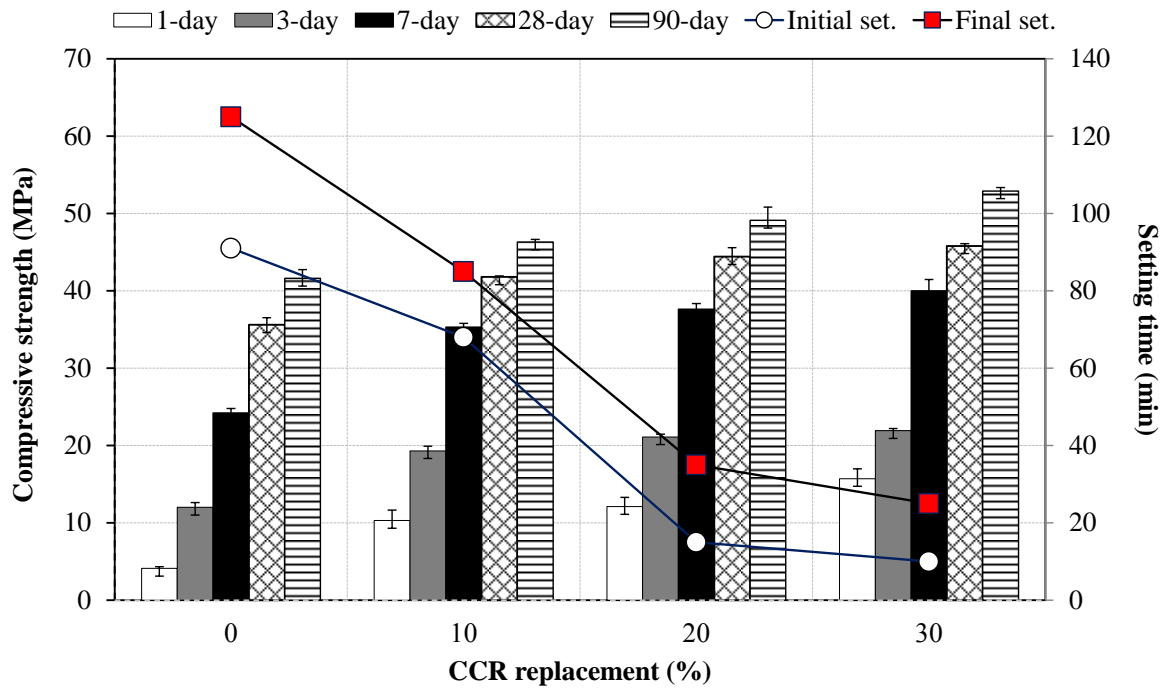
Figure 2 Compressive strength and setting time of alkali-activated FA mortars with CCR
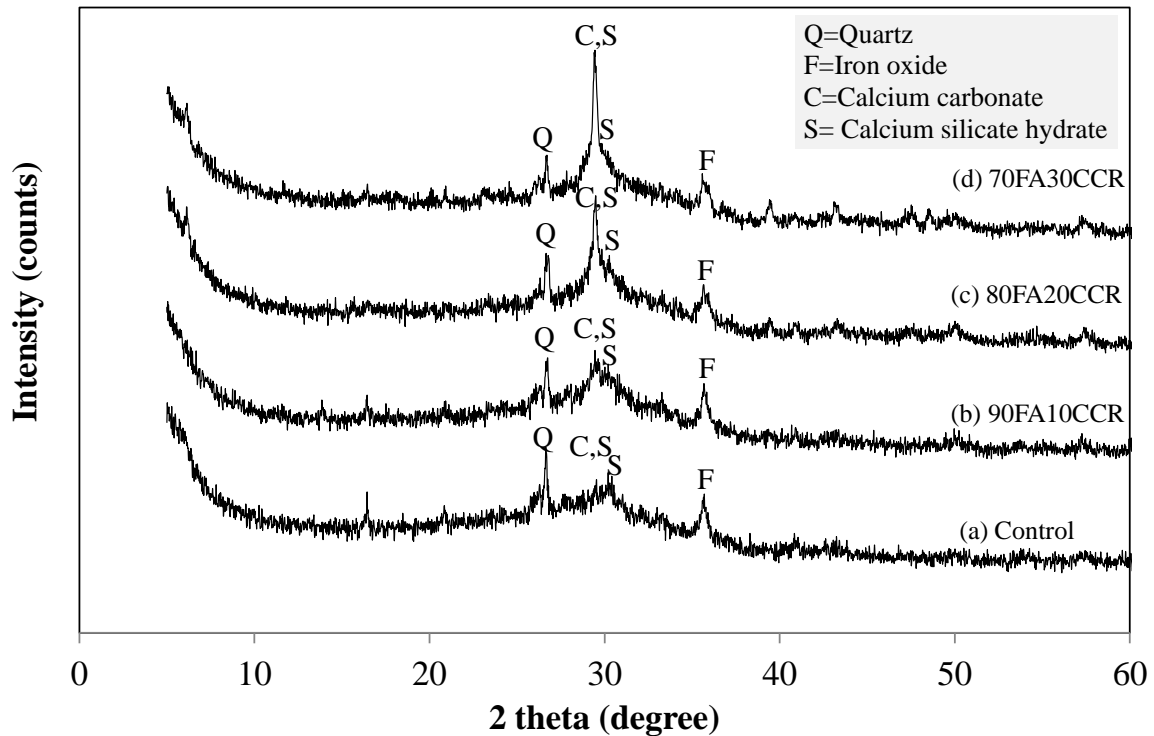

Figure 3 XRD patterns of alkali-activated FA pastes with CCR at 28 days
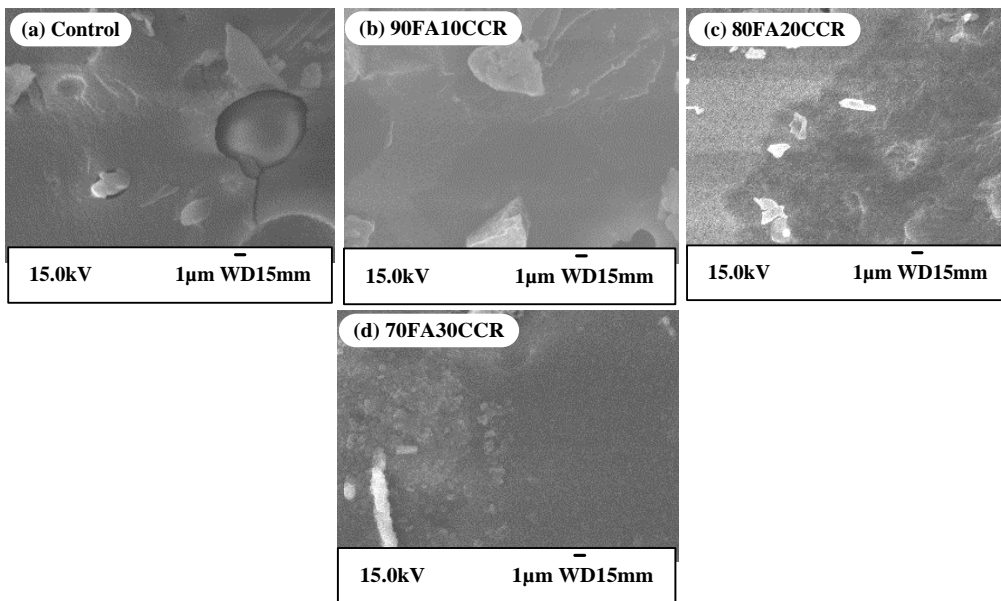
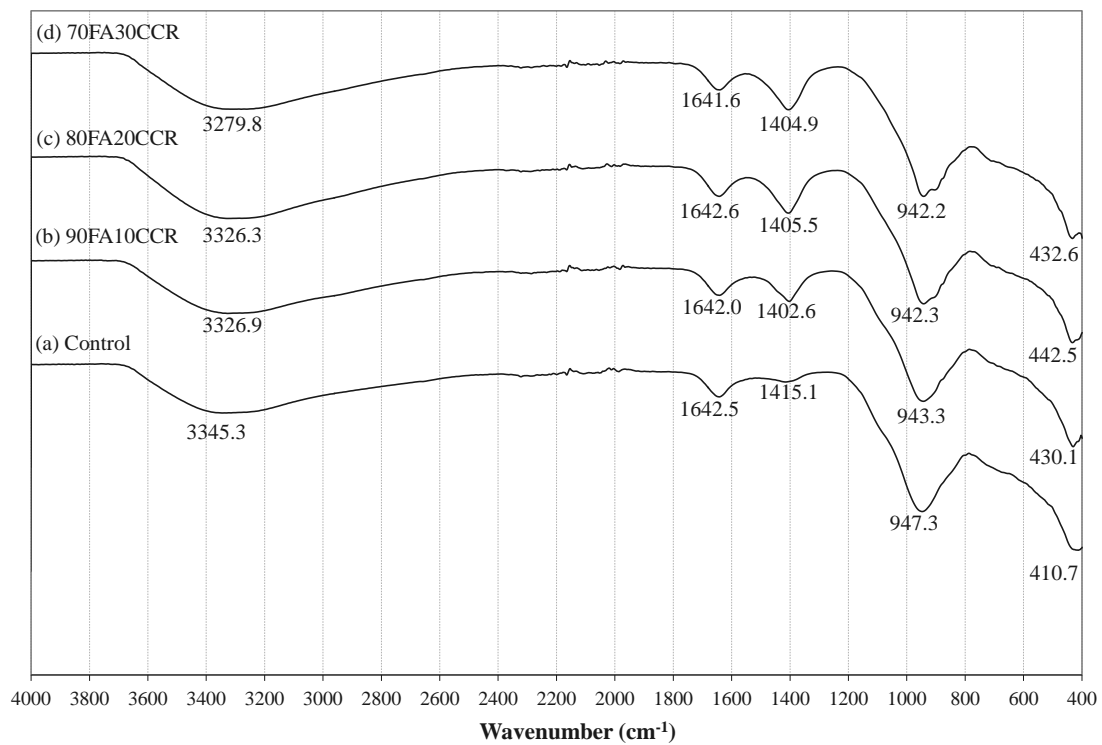**Figure 4** SEM images of alkali-activated FA pastes with CCR at 28 days

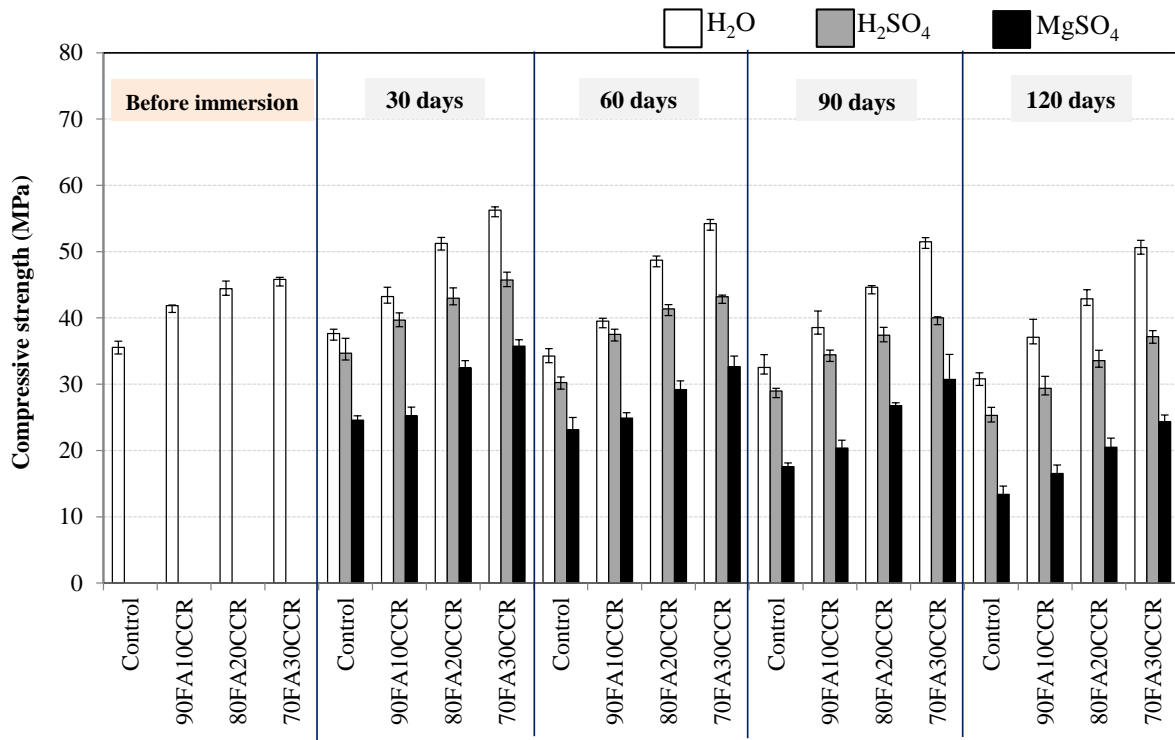**Figure 5** FTIR of alkali-activated FA pastes with CCR at 28 days

Figure 6 Compressive strength of alkali-activated FA mortars with CCR after immersion in
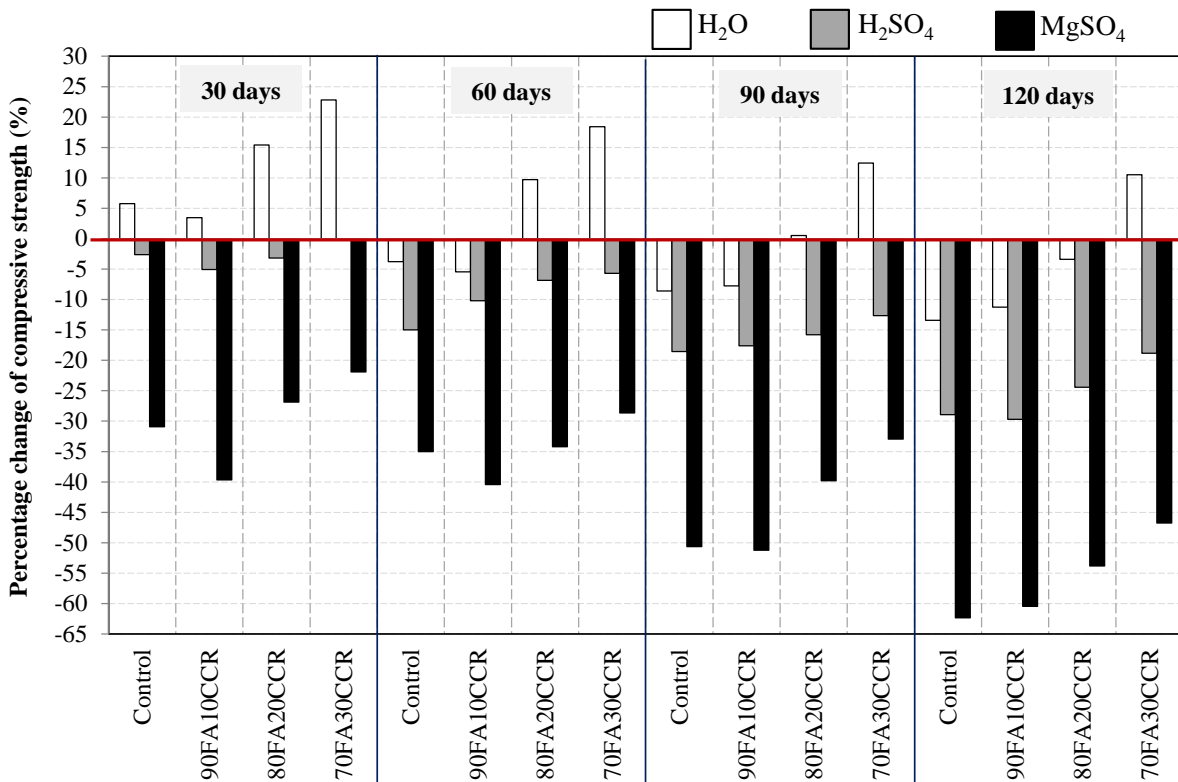
H_2O, 5% $MgSO_4$, and 5% H_2SO_4 solutions

Figure 7 Percentage change of compressive strength of alkali-activated FA mortars with

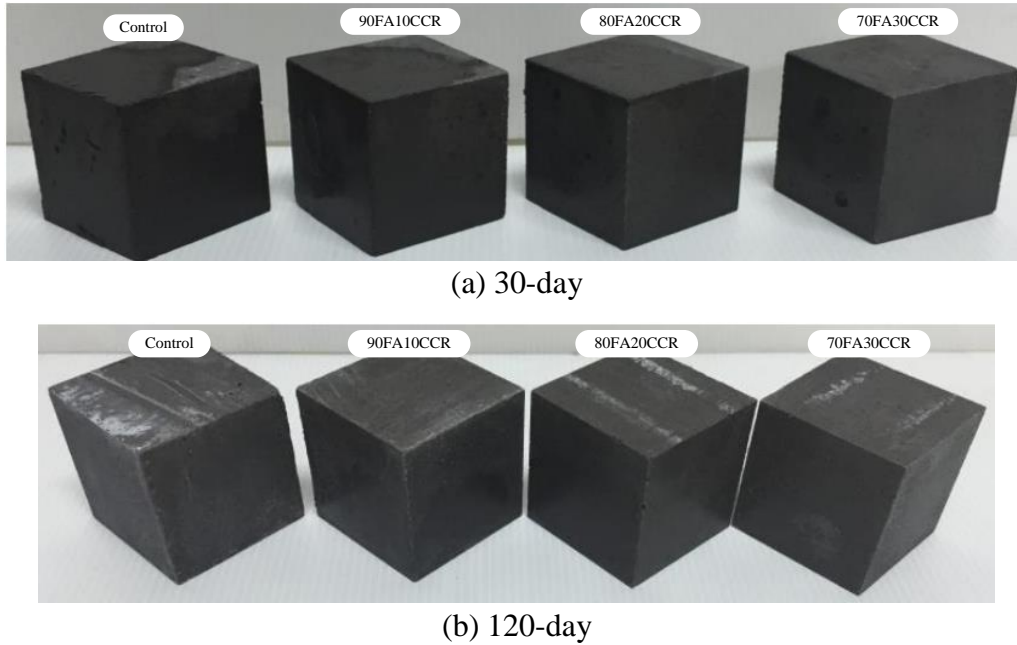CCR after immersion in H_2O, 5%$MgSO_4$, and 5%H_2SO_4 solutions
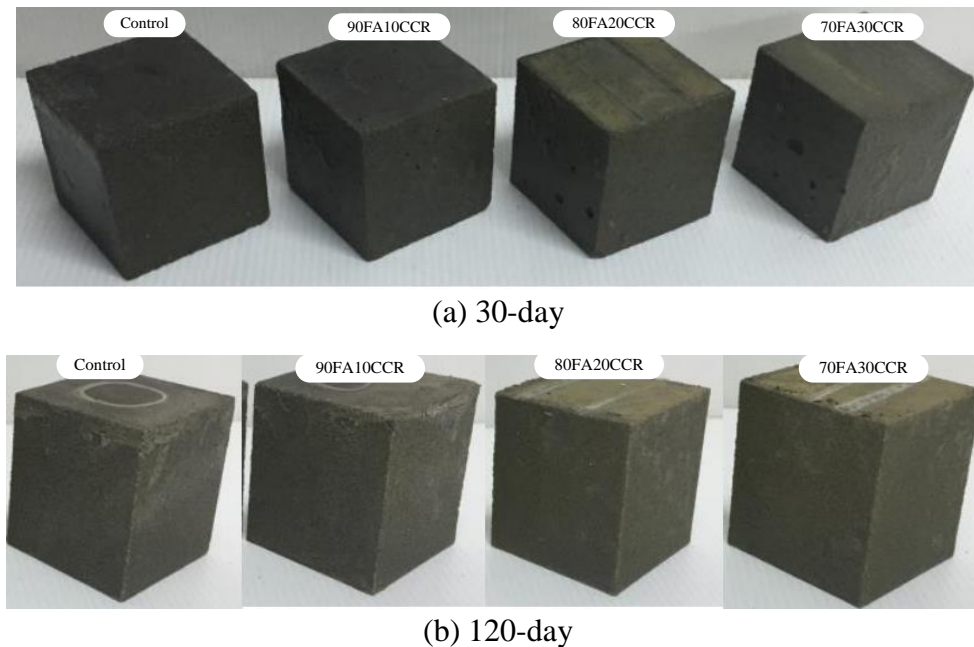

(a) 30-day

(b) 120-day

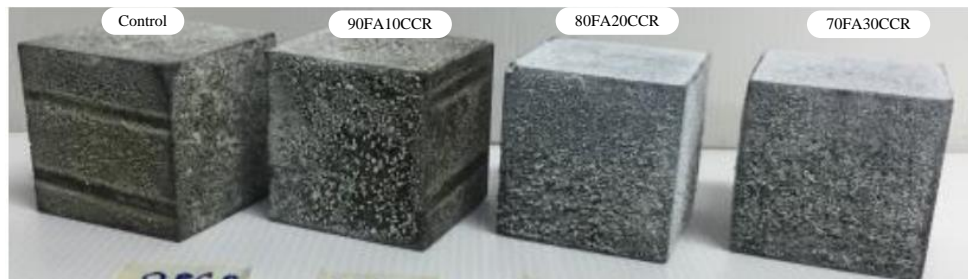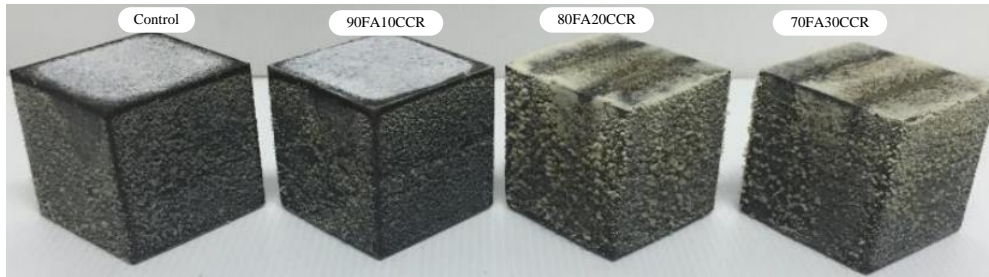**Figure 8** Physical appearance of alkali-activated FA mortars with CCR after 30-day and 120-

day immersion in H_2O

(a) 30-day

(b) 120-day

Figure 9 Physical appearance of alkali-activated FA mortars with CCR after 30-day and 120-

day immersion in 5% H_2SO_4 solution

Control   90FA10CCR   80FA20CCR   70FA30CCR

(a) 30-day

Control   90FA10CCR   80FA20CCR   70FA30CCR

(b) 120-day

Figure 10 Physical appearance of alkali-activated FA mortars with CCR after 30-day and

120-day immersion in 5% $MgSO_4$ solution