



Pozzolanic properties of reject fly ash in blended cement pastes

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Abstract

Low-grade fly ash (reject fly ash, r-FA), a significant portion of the pulverized fuel ash (PFA) produced from coal-fired power plants and rejected from the ash classifying process, has remained unused due to its high carbon content and large particle size. But it may be used in certain areas, such as in solidification and stabilization processes of hazardous waste and materials for road base or subbase construction, which require relatively lower strength and reactivity. It is therefore necessary to extend research on the properties of r-FA and explore its possible applications. This paper presents experimental results of a study on the mechanical and hydration properties of cementitious materials prepared by blending r-FA with ordinary Portland cement (OPC). Parallel mixes were also prepared with the good ash [i.e., classified fine fly ash (f-FA)] for comparison. Selective chemical activators were added to the mix to study the effects of the activators on the properties of the blend system. The results show that r-FA generally has a lower rate of hydration than f-FA particularly at the early stage of hydration. Adding $\text{Ca}(\text{OH})_2$ alone almost had no effect on accelerating the hydration of r-FA. But adding a small quantity of Na_2SO_4 or K_2SO_4 together with $\text{Ca}(\text{OH})_2$ significantly accelerated the hydration reaction. The results of the compressive strength measurement correlated nicely with the degree of hydration results. It was also found that water-to-binder ratio (w/b) was an important factor in affecting the strength development and the hydration degree of r-FA pastes.

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Keywords: Reject fly ash; r-FA; PFA; Strength development; Hydration degree; Chemical activator

1. Introduction

Chemical stabilization/solidification (S/S) process based on cement and other cementitious materials are being increasingly used in many countries as a final treatment step prior to land disposal of hazardous industrial wastes. The aim is to produce a durable, monolithic material that will not leach hazardous components under the disposal conditions. Cost is always a key factor in waste treatment, and although there are commercially available processes that use cement or cement/pulverized fuel ash (PFA) as binders, these materials tend to be expensive. It is becoming increasingly desirable to develop solidification systems that can utilize binders that are derived from other industrial wastes.

PFA is a by-product generated from burning coal during the generation of electricity. Its finer fraction ($<45\ \mu\text{m}$) produced by passing the raw ash through a classifying process is being increasingly used in building construction [1,2]. But the reject of the classifying process (i.e., coarse

fraction) is not suitable for use as a construction material due to its high carbon content and large particle size ($>45\ \mu\text{m}$). In Hong Kong, this material is currently being dumped in large ash lagoons. But it is thought that the reject ash may have potential uses in waste solidification process particularly if the carbon fraction can adsorb contaminants.

Studies on the use of fine fly ash (f-FA) as a construction material have been widely reported. Previous research mainly focused on studying (a) the chemical and physical properties of f-FA blended cement pastes and concrete [3–7], (b) how to accelerate the reaction of f-FA by chemical activation [8–12], elevated temperatures [13,14], and mechanical treatment [15–17], and (c) how to effectively utilize the f-FA in hazardous waste S/S processes [18,19]. But few studies have been conducted on the potential application of the reject ash.

The objective of this paper is to study the pozzolanic properties of reject fly ash (r-FA) blended cement pastes with or without the use of calcium hydroxide and other chemicals as activators. The strength and microstructure developments and the degree of hydration of the r-FA in the blended cement are investigated.

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2. Experimental details

2.1. Materials

Two fly ashes were used in this study. One was a classified fly ash passed through the 45- μm classifying process. The other was r-FA whose particle size was larger than 45 μm . Both of the fly ashes corresponded to the low-calcium Class F and were generated as by-products from a local coal-fired power plant. Fig. 1a shows the X-ray diffraction (XRD) patterns of the two ashes. The quartz peaks of the f-FA were stronger than the r-FA, but the latter has obvious mullite peaks, which may be the result of the ash classifying process. The particle distributions of the ashes are shown in Fig. 1b. A commercially available ordinary Portland cement (OPC) equivalent to ASTM type I was used as a blending agent. A commercially available white lime [$\text{Ca}(\text{OH})_2$] and reagent grade Na_2SO_4 , K_2SO_4 , and CaCl_2 were used as chemical

activators. The chemical and physical properties of the fly ashes and the cement are given in Tables 1 and 2.

2.2. Preparation of specimens

The cement/fly ash mixes were prepared with the following formulations. For both the f-FA and r-FA, three mixes were prepared with fly ash–OPC ratios (w/w) of 100:10, 100:25, and 100:40 and at a water-to-binder (w/b) (FA + cement) ratio of 0.28. One additional mix was prepared at a w/b of 0.35 to test the effects of different w/b on the hydration reaction. A plain cement paste without the addition of fly ash was prepared at the same w/b as a control. Additionally, lime and other activators were added to test the effects of different activators on the hydration of r-FA. The proportions of the mixes prepared are shown in Table 3.

The mixture of fly ash and cement were mixed for 10 min in a mechanical mixer at room temperature. Water was

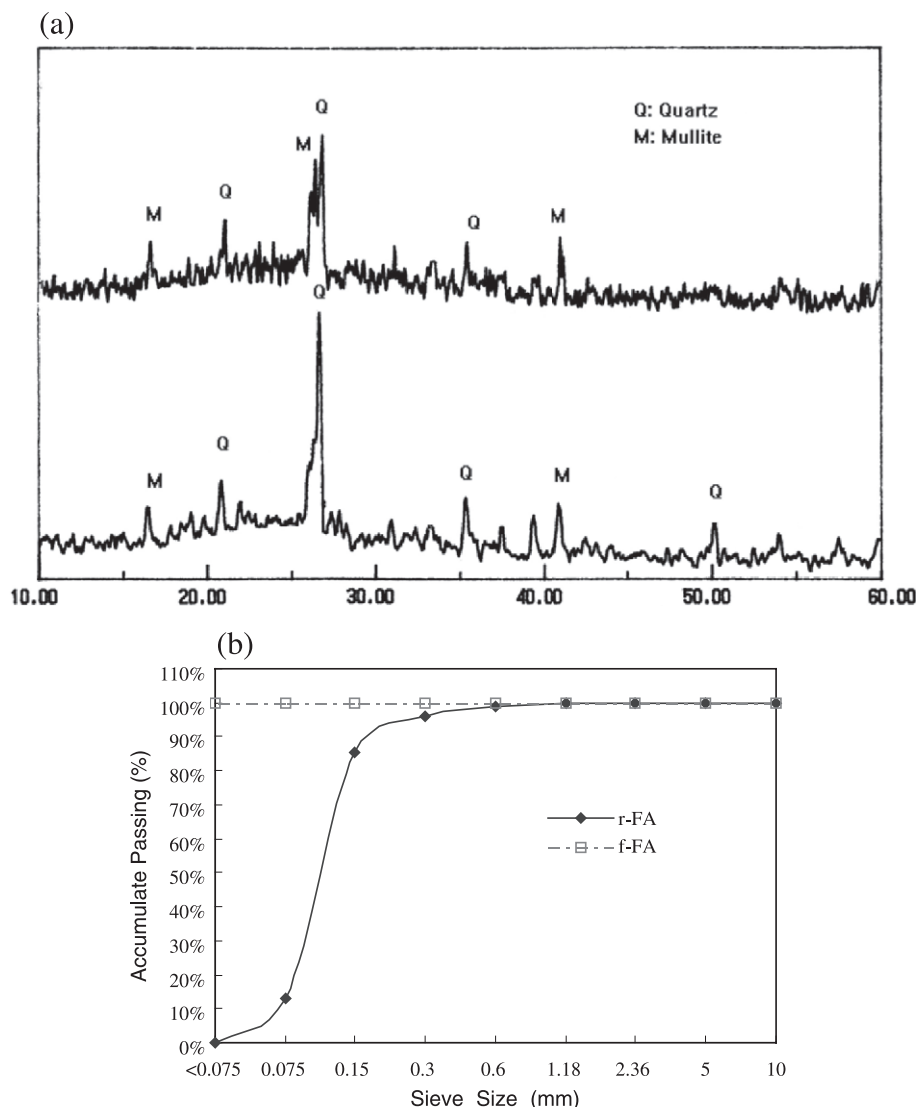


Fig. 1. (a) XRD patterns of r-FA and f-FA. (b) Particle size distributions of the r-FA and f-FA.

Table 1
Chemical compositions of the cement and fly ashes

	SiO ₂	Fe ₂ O ₃	Al ₂ O ₃	TiO ₂	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	I.L
f-PFA	47.62	7.35	27.4	1.23	8.11	3.55	0.57	0.88	0.87	0.89
r-PFA	47.23	8.42	24.54	0.99	8.28	1.62	0.39	—	—	8.06
Cement	19.61	3.32	7.33	—	63.15	2.54	2.13	—	—	2.97

added after the first minute of mixing. As for the chemical activators, they were first dissolved into the required amount of water before mixing. The mixes were then cast into plastic vials ($\phi 40 \times 80$ mm) and compacted with a plastic rod until all entrapped air was removed. The prepared samples were placed into a fog tank at 25 °C. The samples were removed from the plastic vials after 7, 28, and 90 days of curing and subject to the following tests.

2.3. Compressive strength test

At the given ages, three specimens per mix were subject to compressive strength testing using a Denison compression machine. The ends of these specimens were ground to ensure that the end surfaces were flat and parallel before testing. The strength results shown were the average of the three specimens with variations of not more than 10%.

The fractured pieces of the specimens after the compression testing were preserved for other tests. To stop the hydration reactions, the fractured pieces of the pastes were soaked in acetone for a total of 14 days, with the acetone changed after the first 7 days. Then they were dried at 60 °C for 48 h in a vacuum oven and were ground in a mortar to produce fine powders with a particle size of less than 75 μ m.

2.4. Determination of the hydration degree for fly ash

To determine the hydration degree of the fly ashes, a selective dissolution procedure using picric acid–methanol solution and water was employed [20–22]. The method is based on the phenomenon that unreacted cement and fly ash cannot be removed by the picric acid–methanol solution.

The unhydrated fly ash in the fly ash–cement system was determined as follows: 1 g of the ground sample (<75 μ m) was added to a beaker containing 9 g of picric acid and 60 ml methanol (AR Grade). The mixture was stirred using a magnetic stirrer for 15 min. Then, 40 ml of deionized water was added and stirred continuously for another 45 min. The solution was filtered through a Whatman No. 41 filter paper. The filter paper and the residue were washed with

Table 2
Physical properties of cement and fly ashes

	Fineness (m ² /kg)	Density (kg/m ³)
f-PFA	399.6	2.28
r-PFA	119.0	2.19
Cement	352.0	3.16

Table 3
Summary of mix proportions

No.	r-FA	f-FA	Cement	Ca(OH) ₂	Na ₂ SO ₄	K ₂ SO ₄	CaCl ₂	W/b
M1	100	/	10	/	/	/	/	0.28
M2	100	/	25	/	/	/	/	0.28
M3	100	/	40	/	/	/	/	0.28
M4	/	100	10	/	/	/	/	0.28
M5	/	100	25	/	/	/	/	0.28
M6	/	100	40	/	/	/	/	0.28
M7	100	/	25	/	/	/	/	0.35
M8	/	/	100	/	/	/	/	0.28
M9	100	/	25	20	/	/	/	0.35
M10	100	/	25	20	5.8	/	/	0.35
M11	100	/	25	20	/	5.8	/	0.35
M12	100	/	25	20	/	/	5.8	0.35

methanol until they appeared colorless and then were further washed with 300 ml of deionized water at 60 °C. The filter paper and the undissolved contents were transferred into a crucible. The crucible was ignited in an electric furnace for an hour at intervals of 300, 450, and 950 °C. The crucible was cooled down to room temperature in a desiccator after taken out from the electric furnace and weighed. In order to correct the weight of the sample residue, blank tests were carried out following the same procedure without adding the sample.

All the calculations described in this section were carried out on ignition basis (i.e., 1 g of sample was ignited at 950 °C in an electric furnace for 1 h).

The residue (S) per gram of ignited sample was calculated by Eq. (1).

$$S = (\text{residue of sample after selective dissolution} - \text{residue of blank sample}) / \text{weight of sample} \times 100\% \quad (1)$$

The residue per gram of ignited pure component i (F_i) was given by Eq. (2).

$$F_i = (\text{residue of pure component } i \text{ after selective dissolution} - \text{residue of blank sample}) / \text{weight of pure component } i \times 100\% \quad (2)$$

The percentage of component i (P_i) in the sample on ignition basis is given by Eq. (3).

$$P_i = [P_{ii}(100 - L_i) / \Sigma(P_j(100 - L_j))] 100\% \quad (3)$$

Where P_{ii} =percentage of component i in original sample (before ignition), P_j =percentage of component j (including all components of the sample) in original sample, L_i =loss of ignition for component i , and L_j =loss of ignition for component j .

The reaction degree of fly ash (Y) was thus given by:

$$Y = [1 - (S_{\text{sample}} - \Sigma(F_i \times P_i)) / (F_{\text{fly ash}} \times P_{\text{fly ash}})] 100\% \quad (4)$$

2.5. Scanning electron microscopy

A Leica Stereoscan 150 scanning electron microscope (SEM) was used to study the morphology of the fractured surfaces of the cement/fly ash pastes.

3. Results and discussion

3.1. Compressive strength development

The compressive strength data of the pastes containing r-FA or f-FA are presented in Figs. 2 and 3. They show the compressive strengths of pastes decreased with increasing fly ash content, and all of the results of the cement/fly ash pastes were lower than the control (pure cement) paste. These were similar to the results of our previous studies [12]. Figs. 2 and 3 also show that the strength development of f-FA pastes was significantly faster than that of the r-FA before 28 days. For the mixes containing 25% and 40% cement, the strength increased by 85.6% and 171.8% from 7 to 28 days, respectively. These values were much higher than those of the r-FA pastes, which showed an increase of 46% and 95.4% during the same period. For the strength development from 28 to 90 days, the increases of the f-FA pastes were lower (90.2% and 32.6%) than that of the r-FA pastes (92.6% and 46.7%). It can be concluded that the hydration rate of r-FA was slower than f-FA at the early age but increased with curing time.

From Fig. 2, it is also found that the strength of the mix with a w/b of 0.35 was higher than that with a w/b of 0.28 at all the given curing ages. The strength development of the former was also much higher than the latter. Similar results were also reported by Katz [13]. It seems that a higher but not excessive w/b benefited the hydration and strength development of the r-FA cement pastes.

Fig. 4 shows that $\text{Ca}(\text{OH})_2$ alone almost had no effect on the strength development of r-FA pastes. However, after Na_2SO_4 , K_2SO_4 , and CaCl_2 was added into the r-FA–cement– $\text{Ca}(\text{OH})_2$ system, the compressive strengths were higher than the pastes with or without $\text{Ca}(\text{OH})_2$ at all the curing ages. The pastes containing Na_2SO_4 and K_2SO_4

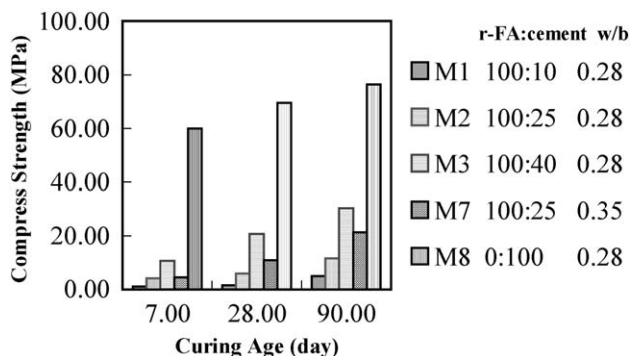


Fig. 2. Compressive strength development of r-FA paste specimens.

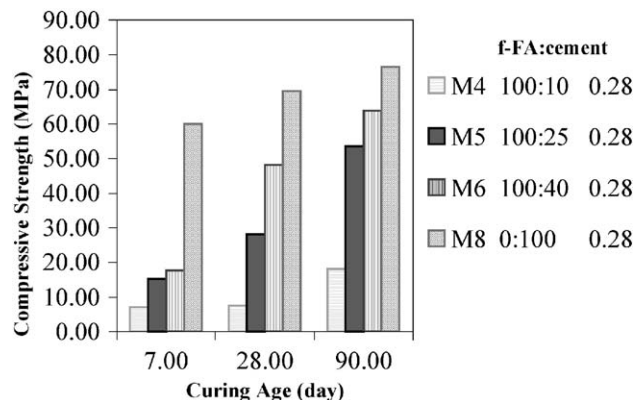


Fig. 3. Compressive strength development of f-FA paste specimens.

generally had higher strength than those with CaCl_2 . These results are different from the results of Shi [9], who showed that Na_2SO_4 increase the early age strength but CaCl_2 increase the later age strength of the f-FA cement blended system. This may be due to a higher pH environment required for r-FA activation.

3.2. Hydration degree of fly ash

The results of the determination of rate of hydration of r-FA and f-FA are shown in Table 4. The results show that at 7 days of hydration, only a small amount of pozzolanic reaction in the fly ash–cement pastes had taken place and the hydration rates of f-FA and r-FA were similar. The data show that reaction between fly ash (both for f-FA and r-FA) and cement had not really started at the early stage of hydration, which is consistent with the findings of Lam et al. [20]. The hydration rates of f-FA increased with the increase in cement content.

Lam et al. [20] reported that the w/b was the principal factor controlling the rate of f-FA reaction. When the w/b increased, the reaction degree also increased and vice versa. In this study, similar results were found for r-FA. From Table 4, it can be seen that as the w/b increased from 0.28 to 0.35 (M2 and M7), the reaction degree of r-FA in paste

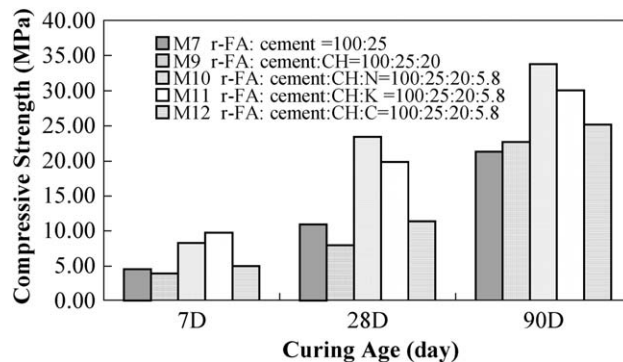


Fig. 4. Compressive strength development of r-FA paste specimens with $\text{Ca}(\text{OH})_2$ and chemical activators (note: CH– $\text{Ca}(\text{OH})_2$, N– Na_2SO_4 , K– K_2SO_4 , and C– CaCl_2).

Table 4
Hydration degree of fly ash in fly ash-cement pastes

	7 days (%)	28 days (%)	90 days (%)
M1	2.50	4.48	14.44
M2	0.81	4.26	10.36
M3	5.08	9.09	20.44
M4	2.84	5.30	13.62
M5	3.64	8.04	13.04
M6	5.52	11.95	17.46
M7	4.40	6.48	19.14
M9	1.91	6.75	14.22
M10	16.13	18.67	22.32
M11	14.72	17.73	21.01
M12	9.59	13.36	21.43

containing 25% cement also increased. The data also show that $\text{Ca}(\text{OH})_2$ only (M9) could not accelerate the hydration rate of r-FA, but the addition of small dosages of Na_2SO_4 , K_2SO_4 , and CaCl_2 dosage (M10, M11, and M12) accelerated the hydration rate significantly. The paste containing Na_2SO_4 showed the fastest hydration rate at 7 days.

At 28 days, the rate of hydration of r-FA still remained lower than 5% except mix M3 and M7. Nevertheless, the degrees of f-FA reaction exceeded 5%. According to Berry et al. [24] and Lam et al. [20], the hydration degree of the fly ash in M1, M2, and M4 was still in the initial alkali attack stage. Once again, it showed that the reactivity of r-FA was low. Aimin and Sarkar [10] and Berry et al. [24] reported that from 7 to 28 days, the glass phase of many fly ash particles showed signs of etching and $\text{Ca}(\text{OH})_2$ began to redissolve and join the reaction. Evidence of fly ash reaction at 28 days was also shown in M3, M5, and M6 in which the degree of hydration of fly ash was nearly doubled (Table 4). Compared with the f-FA pastes, the hydration degree of r-FA only increased slightly except for the mix containing 40% cement.

Compared with those without any chemical activators, the pastes containing Na_2SO_4 , K_2SO_4 , and CaCl_2 (M10, M11, and M12) still had the higher hydration rates although the rates of increase were lower at 28 days. The hydration degree of the paste activated only by $\text{Ca}(\text{OH})_2$ was a little higher than those without it.

At 90 days, the measured hydration degree of f-FA increased to about two to four times of that at 7 days. Similar results were obtained by Lam et al. [20]. However, the increase in the hydration degree of r-FA was higher than that of f-FA. According to Aimin and Sarkar [10] and Berry et al. [24], for f-FA, etched fly ash surfaces and hydrated rims with broken fly ash particles are quite common after 90 days of hydration. Compared with the f-FA, the pozzolanic reaction of the r-FA particles only started at much later curing ages.

Although the hydration degrees of the pastes without chemical activators doubled at 90 days, they are still lower than those activated by Na_2SO_4 , K_2SO_4 , and CaCl_2 . This result shows that the pozzolanic reaction of r-FA is slow even with the addition of $\text{Ca}(\text{OH})_2$.

3.3. SEM observations

3.3.1. 7 Days

The microstructure of the fractured surfaces of M2, M3, M5, M7, and M9–M12 were studied by SEM. Fig. 5 shows the micrographs of the pastes cured for 7 days. It can be found that a number of particles of f-FA (Fig. 5d) had already been etched although smooth ones still existed. Similar results were reported by Zhang [3]. Compared with f-FA paste, the surfaces of r-FA particles (Fig. 5a–c) were smoother and rounder. This suggested that r-FA has not yet entered into an active state of reaction at this age. It can also be noticed that the needlelike crystals around the paste with a high w/b (Fig. 5b) were more abundant than those pastes with a low w/b (Fig. 5a), which were consistent with the results of the degree of hydration study.

The r-FA particles also remained smooth in the paste activated only by $\text{Ca}(\text{OH})_2$ (Fig. 6a) but with more platy crystals and gel-like products surrounding the r-FA particles. Some needlelike crystals were also observed. The r-FA particles in the paste containing Na_2SO_4 were etched badly (Fig. 6b) and were coated by a dense layer of gel-like products. No obvious platy crystal was observed. The morphology of the paste containing K_2SO_4 was similar to that containing Na_2SO_4 (Fig. 6c). As for the pastes with CaCl_2 , the r-FA particles were etched sharply and appeared to have reacted partly (Fig. 6d).

3.3.2. 28 Days

At 28 days, it was found that the surface of f-FA particles (Fig. 7d) appeared to be broken and reacted significantly. Fig. 7a and b show that the r-FA particles had not reacted except with some deposits, but the ash particles in the sample with the higher w/b were surrounded by more gel-like and needlelike products. It can also be noticed that the r-FA particles in the sample with higher cement content had only been etched slightly at this age (Fig. 7c).

The particles in the paste activated only by $\text{Ca}(\text{OH})_2$ were etched sharply at 28 days (Fig. 8a). For the pastes activated by Na_2SO_4 or K_2SO_4 (Fig. 8b and c), the boundary between r-FA particle and the other hydration products cannot be distinguished easily. As for the pastes activated by CaCl_2 , the r-FA particles were also etched badly although the boundary still remained (Fig. 8d).

3.3.3. 90 Days

At 90 days, more broken particles of the f-FA could be seen and the system became much denser (Fig. 9d), which was due to an increase in bonding between f-FA particles and the surrounding paste at this age [10]. Fig. 9a shows that the r-FA particles with a w/b of 0.28 began to be etched and react with the hydration products. But the r-FA particles in the paste with a w/b of 0.35 were already at an advanced hydration state (Fig. 9b) and the system was very dense. The r-FA particles in the paste with 40% cement had a similar morphology (Fig. 9c). As for the pastes containing $\text{Ca}(\text{OH})_2$

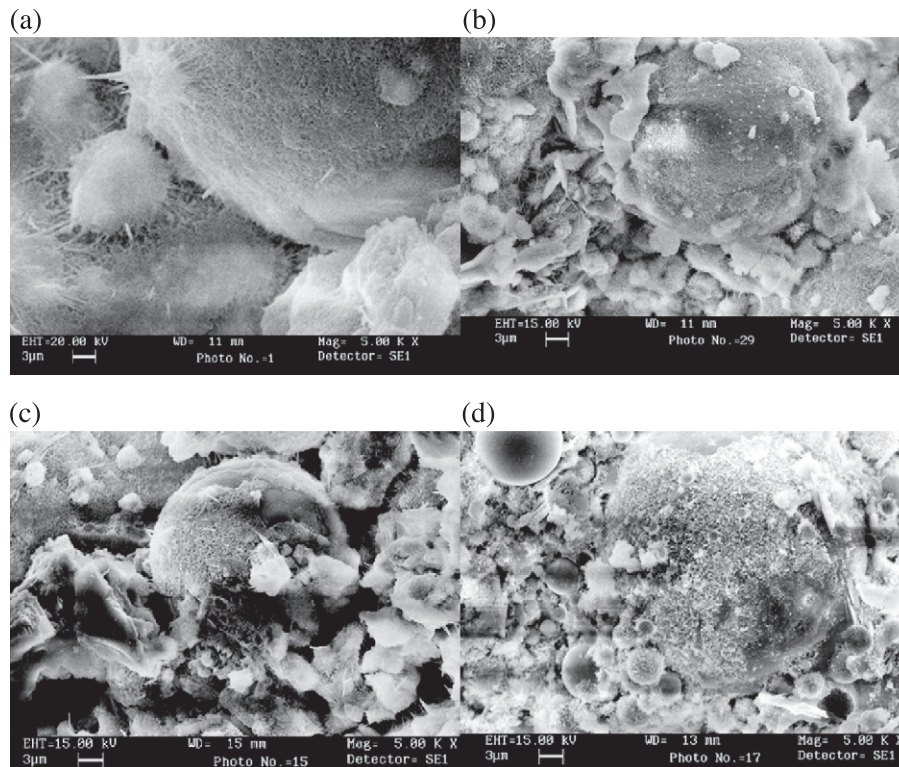


Fig. 5. Morphology of hydration products at 7 days. (a) M2 (r-FA-cement = 100:25; w/b = 0.28), (b) M7 (r-FA-cement = 100:25; w/b = 0.35), (c) M3 (r-FA-cement = 100:40; w/b = 0.28), (d) M5 (f-FA-cement = 100:25; w/b = 0.28).

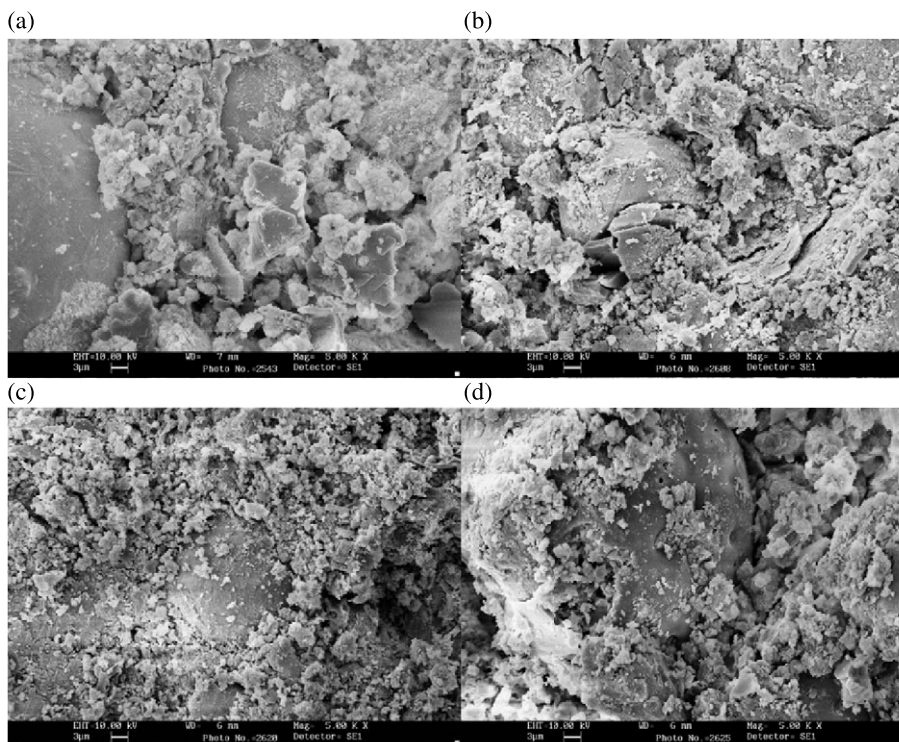


Fig. 6. Morphology of hydration products at 7 days. (a) M9 [r-FA-cement-Ca(OH)₂ = 100:25:20], (b) M10 [r-FA-cement-Ca(OH)₂-Na₂SO₄ = 100:25:20:5.8], (c) M11 [r-FA-cement-Ca(OH)₂-K₂SO₄ = 100:25:20:5.8], (d) M12 [r-FA-cement-Ca(OH)₂-CaCl₂ = 100:25:20:5.8].

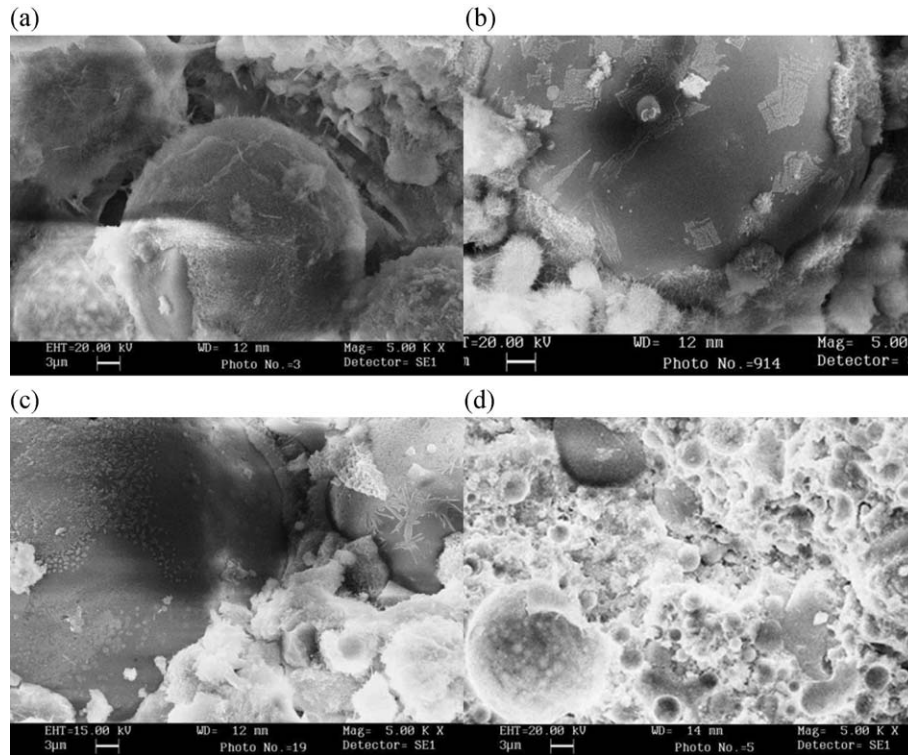


Fig. 7. Morphology of hydration products at 28 days. (a) M2 (r-FA–cement = 100:25, w/b = 0.28), (b) M7 (r-FA–cement = 100:25, w/b = 0.35), (c) M3 (r-FA–cement = 100:40, w/b = 0.28), (d) M5 (f-FA–cement = 100:25, w/b = 0.28).

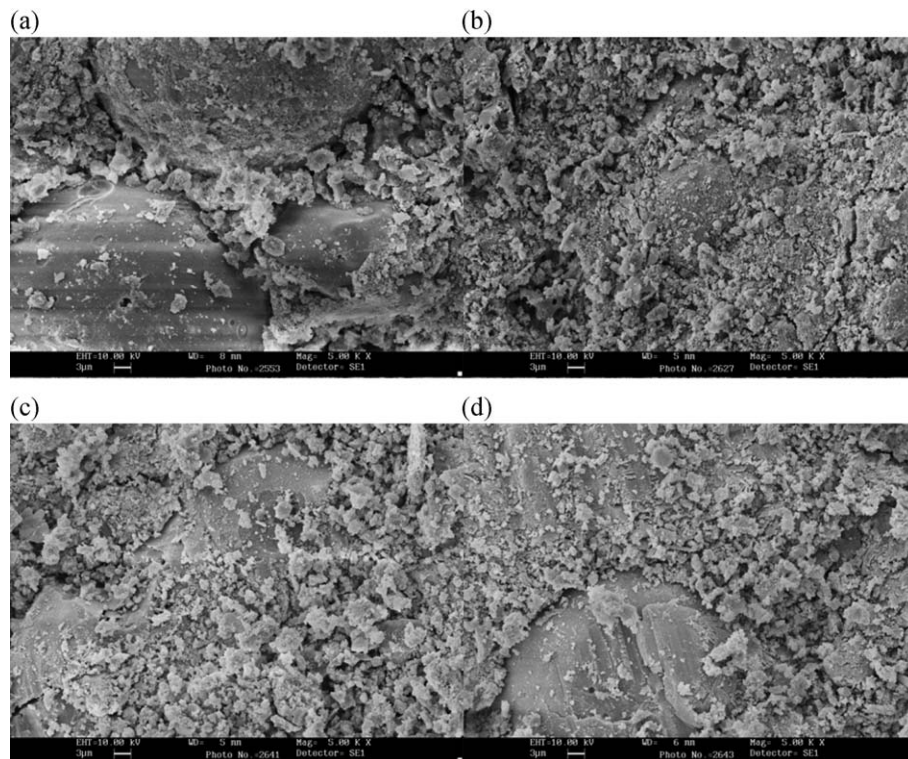


Fig. 8. Morphology of hydration products at 28 days. (a) M9 [r-FA–cement–Ca(OH)₂ = 100:25:20], (b) M10 [r-FA–cement–Ca(OH)₂–Na₂SO₄ = 100:25:20:5.8], (c) M11 [r-FA–cement–Ca(OH)₂–K₂SO₄ = 100:25:20:5.8], (d) M12 [r-FA–cement–Ca(OH)₂–CaCl₂ = 100:25:20:5.8].

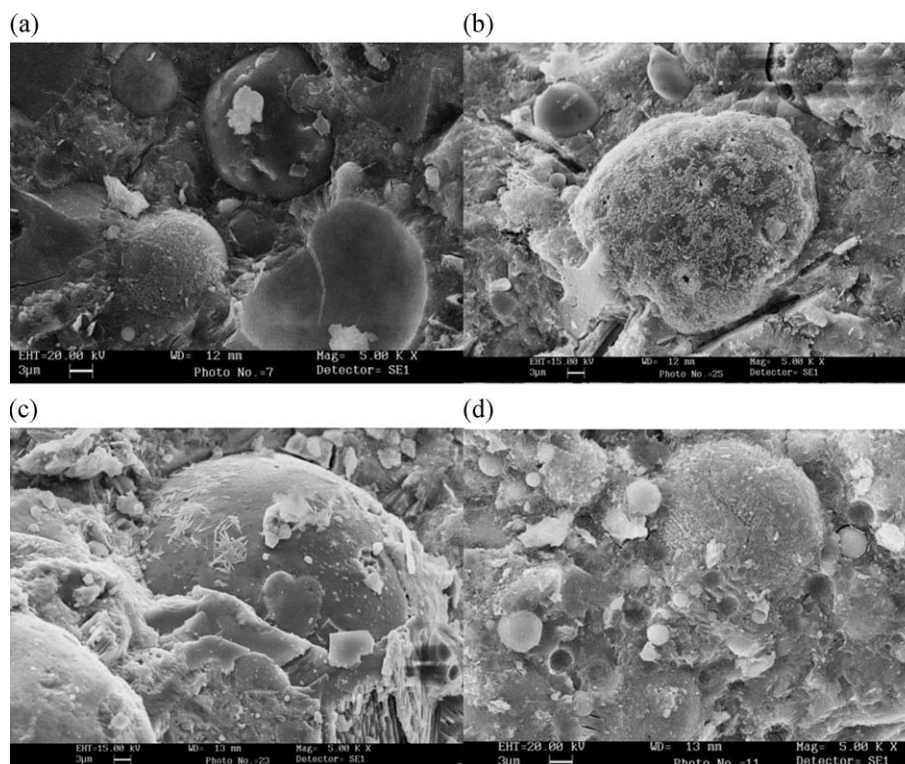


Fig. 9. Morphology of hydration products at 90 days. (a) M2 (r-FA–cement = 100:25, w/b = 0.28), (b) M7 (r-FA–cement = 100:25, w/b = 0.35), (c) M3 (r-FA–cement = 100:40, w/b = 0.28), (d) M5 (f-FA–cement = 100:25, w/b = 0.28).

or other chemical activators, they showed a very dense microstructure in which the individual phases were very difficult to be identified (micrographs not shown).

4. Discussion

According to Aimin and Sarkar [10], the possibility of fly ash activation mainly lies in the breaking down of its glass phase and the fly ash can react with $\text{Ca}(\text{OH})_2$ in the fly ash–cement system. From Figs. 2 and 3, it can be noticed that the 28-day strength developments (rate of strength gain) of r-FA and f-FA pastes increased with the increase of cement content. It may be because more $\text{Ca}(\text{OH})_2$ was produced during the hydration of cement, and the solution between the particles reached a higher pH value with the increase in cement content. This is supported by the results of the rate of hydration of the fly ash–cement system, which shows that the hydration degrees of both r-FA and f-FA increase with the increase in cement content (Table 4). Compared with f-FA cement system, it was also found that only the r-FA particles of the paste containing 40% cement started showing some etching at 28 days. This hints that the r-FA particles require a higher pH value to be activated than f-FA particles.

The paste with a higher w/b (0.35) had higher strength values than a similar paste prepared with a lower w/b (0.28) (Fig. 2). This may be because at w/b of 0.28, insufficient

amount of water was available for the hydration of the r-FA particles, which tended to absorb a larger amount of water when compared with the f-FA particles. This is consistent with the results of the SEM observations, which showed more hydration products and more dense microstructure for the paste prepared with a w/b of 0.35 than that with a w/b of 0.28 (shown in Figs. 5a and b, 7a and b, and 9a)). The results of the rate of hydration also indicate that a higher w/b is useful for the reaction of r-FA. It means that a higher but not excessive w/b can enhance the reactivity of r-FA.

Fraay et al. [23] reported that in the presence of aqueous lime, the pH value required to dissolve the alumina and silica in fly ash was about 13.3 or higher. But the pH value of a saturated solution of $\text{Ca}(\text{OH})_2$ was 12.45 at 25 °C [25]. So the addition of $\text{Ca}(\text{OH})_2$ did not have obvious effect on the r-FA cement system (Table 4). The SEM observations also showed that the addition of $\text{Ca}(\text{OH})_2$ induced no obvious changes in the amount of hydration products produced (Fig. 6a). However, the addition of Na_2SO_4 and K_2SO_4 was very effective in accelerating the hydration rate of r-FA–cement– $\text{Ca}(\text{OH})_2$ system, especially at 7 days (Table 4). This may be attributed to the presence of Na^+ and K^+ ions, which can increase the pH value of the solution in the pastes and accelerate the dissolution of glass phase in the r-FA. This is supported by the SEM observations (shown in Figs. 6b and c and 8b and c). With the increase in the amount of hydration products, the compressive strength increased as well (Fig. 4).

On an equal weight basis, the number of ions of Na_2SO_4 in solution was larger than that of K_2SO_4 , this means more Na^+ in the solution than K^+ , and hence a higher pH value in the pastes containing Na_2SO_4 . So the pastes activated by Na_2SO_4 had a higher rate of hydration than those containing K_2SO_4 (Table 4).

5. Conclusions

1. Under the same conditions, r-FA was difficult to react than f-FA and requires a higher pH value to be activated. A higher but not excessive w/b also benefits the reaction of r-FA.
2. The addition of $\text{Ca}(\text{OH})_2$ only shows little use for r-FA cement system. This may attribute to the relatively low pH value (12.45 at 25 °C) of the saturated solution of $\text{Ca}(\text{OH})_2$.
3. Na_2SO_4 and K_2SO_4 are very efficient in accelerating the reaction of r-FA in the r-FA–cement– $\text{Ca}(\text{OH})_2$ system. This may be because Na^+ and K^+ can increase the pH value of the solution in the pastes and accelerate the dissolution of glass phase in the r-FA.
4. Little reaction occurred between r-FA and cement at 7 days of hydration. Significant amounts of reaction only appeared after 28 days of hydration.

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