

## Research Article

# Influence of Inertia and Low Active Mineral Admixture on Strength and Microstructure of Cement-Based Materials

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Received 10 May 2016; Revised 1 July 2016; Accepted 5 July 2016

Academic Editor: Ana S. Guimarães

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Cement-based materials were investigated by comparing the strength and microstructure of pastes and mortar containing limestone powder or low quality fly ash. The compressive strength of the mortar at 28 and 90 d was examined whose microstructures were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), thermogravimetric analysis, and differential thermal analysis (TG-DTA). The results indicated that the strength of mortar decreased with increasing mineral admixtures. The limestone powder mainly acted as inert filler and hardly took part in the chemical reaction. Low quality fly ash may accelerate the formation of hydration products in samples with more chemically bonded water. This further resulted in a higher degree of cement hydration and denser microstructure, while the overall heat of hydration was reduced. At the early stage of hydration, low quality fly ash can be considered as an inert material whereas its reactivity at the later stage became high, especially for ground low quality fly ash.

## 1. Introduction

Cement is widely used in the production of concrete with its annual consumption close to 2 billion tons [1–3]. The production of cement itself however is quite energy-intensive and responsible for large quantities of CO<sub>2</sub> emissions. In addition, the high amount of alkali in cement might cause cracks in the structure of concrete [4]. As one of the solutions to these, nowadays many mineral admixtures have been used as supplementary cementitious materials (SCMs) [5, 6] to reduce the amount of cement clinker, with other excellent properties such as reduced heat of hydration and improved durability of concrete.

Mineral admixtures can be divided into cementitious materials, pozzolanic active materials, and inert materials according to their chemical activities [7]. Some studies have shown that the inert mineral admixture can enhance the properties of concrete in terms of strength, frost resistance, and so forth [8–11]. This is mainly due to the dilution effect of the inert mineral admixture on cement [11]. It has been reported that equivalent amount of low active or inert mineral

admixture exhibited no difference in the early hydration of cementitious materials [12, 13]. Recently, limestone powder has been applied widely in construction engineering as a new type of concrete admixture [14–16]. The particle size distribution and fineness of limestone powder are similar to those of fly ash, both of which have physical activity, morphological effect, and filling effect. The use of limestone powder as admixture in concrete leads to a better accumulation effect in cement particle skeleton and offers crystalline nucleus to the formation of calcium hydroxide (CH) and calcium silicate hydrate (C-S-H) gel. These synergistic effects can accelerate the process of cement hydration. It is traditionally believed that limestone powder is an inert admixture with high fineness which may be applied in concrete to make up composite cementitious material system that gives full play to its microaggregate effect [17–19]. Fly ash is used in concrete in the building material industry, construction engineering, and road engineering accounts for about 90% of its overall use [20, 21]. Although the progress in fly ash utilization is at the forefront compared to some developed countries, the utilization level is still very low. Research in fly ash is mainly

TABLE 1: Chemical composition of cement, low quality fly ash, and limestone powder/mass, %.

Compositions	SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	SO <sub>3</sub>	LOI
Cement	23.61	3.44	4.06	59.59	2.51	0.86	0.19	—	3.00
Low quality FA	53.41	4.28	25.79	2.60	2.79	1.38	0.42	0.3	3.67
Limestone powder	1.79	0.35	0.56	54.69	0.40	0.14	—	0.03	41.93

TABLE 2: Physical properties of mineral admixtures.

Test item	Residue on sieve of 45 $\mu\text{m}$ /%	Water requirement/%	Specific area/m <sup>2</sup> ·kg <sup>-1</sup>	Density/kg·m <sup>-3</sup>
Cement	—	—	437	3010
Limestone powder	16.0	98.0	780	2670
Raw fly ash	49.8	99.6	278	1880
Ground fly ash	19.7	100.4	794	2340

focused on its physical and chemical properties, but few studies have reported low quality fly ash. In order to promote further use of fly ash, it is therefore critical to say something here about the significance of this work.

In this paper, a comparative system was for the first time established to assess the impacts of limestone powder as an inertia mineral admixture and fly ash as a low active mineral admixture on the strength and microstructure of cement-based materials. This is of great significance in improving the level of low quality fly ash utilization, thereby conserving resources and protecting the environment.

## 2. Experimental

**2.1. Raw Materials.** Ordinary Portland cement P.O 42.5 is supplied by Huaxin Cement Plant. Low quality fly ash is supplied by Guanyinyan Hydropower Project. Table 1 shows the chemical compositions of the cement, fly ash, and limestone powder determined by X-ray Fluorescence (XRF).

Figure 1 is the particle morphology of raw ash and ground ash. The raw low quality fly ash particle is coarse and irregular and its sieve residue of 45  $\mu\text{m}$  reaches as high as 49.8%. The particle microstructure was significantly improved to be smaller and uniform after grinding. Table 2 presents the physical properties of the admixtures. Water demand for the raw fly ash is so high, because there is less free water in raw ash; on the other hand, there are more coarse pore carbon particle in raw ash.

Particle sizes of cement, limestone, raw ash, and ground fly ash were analyzed by OMEC lazer particle size analyzer LS-C (III) and Figure 2 shows the cumulative diameter percentage and the differential curve. It can be seen that the particle size distributions of limestone powder and cement were very similar with the former being slightly smaller.

**2.2. Testing Methods.** The quantitative analysis of the influence of limestone powder and low quality fly ash on the strength and microstructure of cement-based materials was based on the comparison of pure cement paste with the content of 20%, 40%, and 60%. Water-binder ratio is 0.5. Table 3 lists the paste mix proportions.

TABLE 3: Mix proportion of composite cementitious materials/mass, %.

Sample	Cement	Limestone powder	Raw fly ash	Ground fly ash
C	100	—	—	—
L2	80	20	—	—
L4	60	40	—	—
L6	40	60	—	—
R2	80	—	20	—
R4	60	—	40	—
R6	40	—	60	—
G2	80	—	—	20
G4	60	—	—	40
G6	40	—	—	60

Paste specimens with 40 mm  $\times$  40 mm  $\times$  40 mm dimensions and a 0.4-water-binder ratio (W/C) were molded and cured with higher than 90% relative humidity and 20  $\pm$  2 $^{\circ}$ C temperature until the stipulated age of 3, 7, 28, and 90 days. Determining the compressive strength of mortar specimens of different ages was conducted by WAY-2000 (i.e., a battery solution type compressive testing machine). Clean, bean-size samples were taken from the center of broken specimens and packed into ampere bottles filled up with absolute ethanol to terminate the hydration process before the XRD, TG-DTA, and SEM microcosmic analysis. Sample pieces were held in a dry environment at 60 $^{\circ}$ C for 2 or 3 hours before SEM tests. The other samples were ground in an agate mortar and dried at 60 $^{\circ}$ C for 2 hours to reduce carbonation before the XRD test and then dried in vacuum condition before the TG-DTA test.

The XRD analysis was made by X-ray diffraction, using a copper target and a continuous scan machine produced by RIGAKU, a Japanese company. The TG-DTA in an N<sub>2</sub>-atmosphere up to a temperature of 1200 $^{\circ}$ C adopted the diamond TG/DTA analysis produced by Perkin Elmer Instruments Plant, and the morphology of the products was investigated using a scanning electron microscopy (JSM-5610LV, Japan).

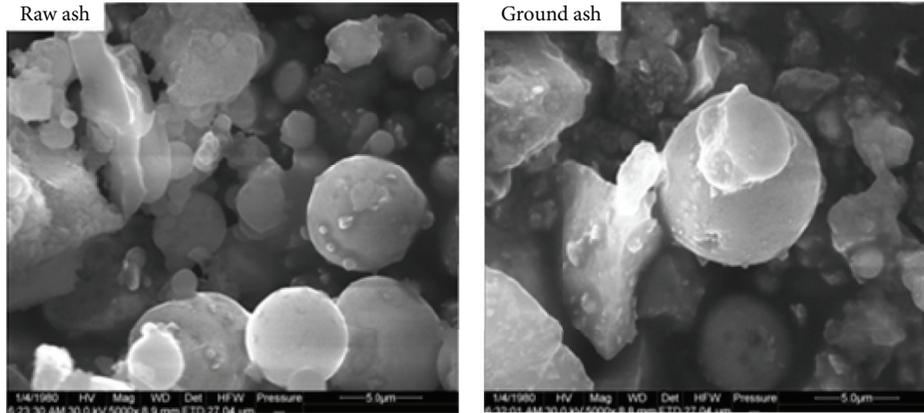


FIGURE 1: Particle morphology of raw ash and ground ash.

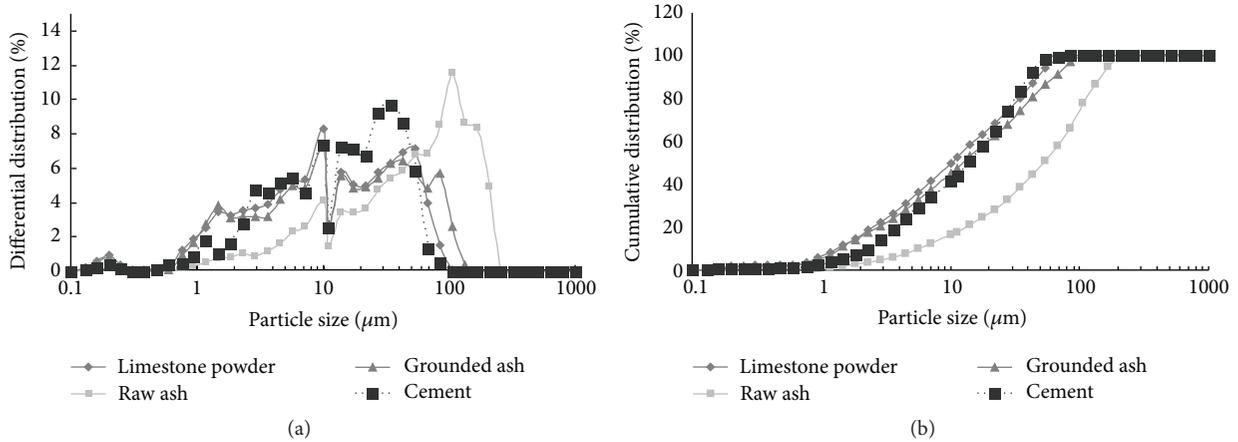


FIGURE 2: (a) Differential and (b) cumulative particle size distribution of the cementitious materials.

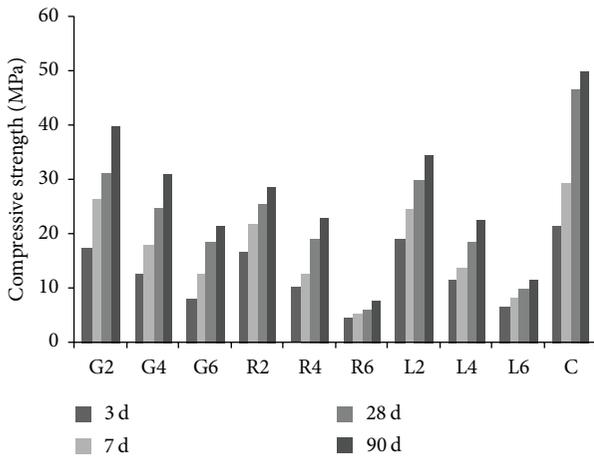


FIGURE 3: Compressive strength of mortars with water-binder ratio of 0.5.

### 3. Results and Discussion

3.1. *Strength.* Figures 3 and 4 present the compressive strength of mortar at different water-binder ratios of 0.3 and 0.5. The figures showed that the strength of mortar

decreased with increasing incorporation of admixture at the same water-binder ratio. The mortar containing low content of limestone powder showed higher compressive strength than that containing ground fly ash at the early stage of hydration. However, with the onset of hydration, the strength of the mortar containing ground fly ash improved rapidly and exceeded the one with limestone at the later age, which may be linked to the higher pozzolanic activity of fly ash at the later period.

The water-binder ratio had little influence on the strength developing law of mortar containing limestone powder or ground fly ash. Therefore, reducing the water-binder ratio could significantly improve the strength of mortar specimen containing high content of ground fly ash or low content (20%) raw fly ash to higher strength than those containing the same content of limestone powder.

3.2. *Hydration Heat Evolution Process.* Figure 5 shows the hydration heat produced and the rate of heat evolution for the four selected mixtures within the first 72 h. The hydration heat curves indicated that the influence of incorporating different admixtures in the composite cementitious materials varied the hydration heat. The addition of fly ash exhibited little effect on the five stages of cement hydration but reduced

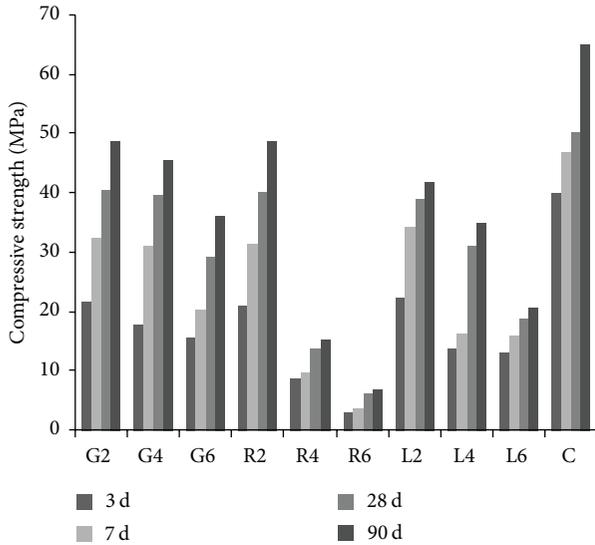


FIGURE 4: Compressive strength of mortars with water-binder ratio of 0.3.

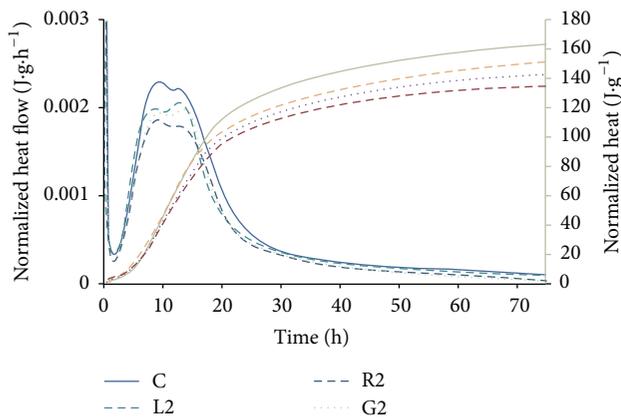


FIGURE 5: Hydration heat and rate of the cementitious materials.

the hydration rate and the total heat compared to those of the pastes C. The third exothermic peak, caused by the conversion of AFt to AFm as a result of the consumption of gypsum, was much higher than the second one in paste G2 whereas the opposite response was observed in paste R2. This suggested that fine-grinding leads to a quicker hydration reaction and both raw ash and ground ash as mineral admixtures reduce the heat of hydration which is beneficial to mass concrete construction. The rate of heat evolution of pastes L2 exceeded that of G2 after the induction period but became lower during the deceleration period. It is proposed that the transformation from ettringite to a more steady monocarbon aluminate contributed to the third exothermic peak.

For further analysis, Figure 6 presents a plot of the induction period against the acceleration period in the hydration process. The addition of fly ash prolonged the induction period but shortened the acceleration period compared with pastes C, which demonstrated that fly ash had little effect

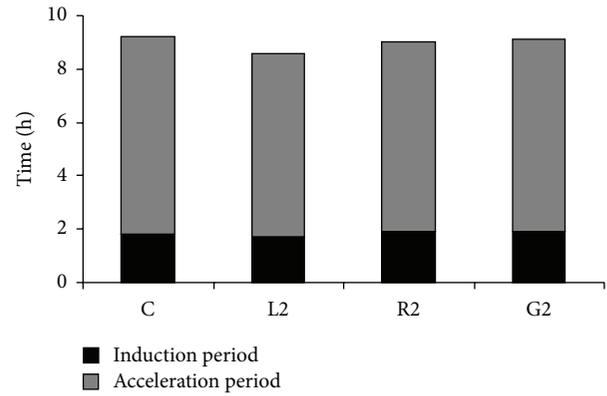


FIGURE 6: Induction period and acceleration period of the cementitious materials.

on the appearance of the second exothermic peak. There are two reasons for the extension of the induction period. On one hand, the increase of effective water reduced the concentration of  $\text{Ca}^{2+}$  in the pore solution and extended the induction period by increasing the time required for  $\text{Ca}^{2+}$  saturation. On the other hand, ground fly ash sphere particles may adsorb  $\text{Ca}^{2+}$  in solution, which inhibited the increase of  $\text{Ca}^{2+}$  concentration through adsorption in the first few hours. This also delayed the crystallization nucleation of CH and C-S-H and accordingly cements hydration. In the accelerated phase, more water came into contact with the new exposed surface and accelerates cement hydration because of the increasing effective water. In summary, the nucleation effect of the ground fly ash may also accelerate cement hydration.

The difference between pure cement and cement-based materials is that the cement-based material containing limestone powder had three special points. These special points included a shortened induction period, an accelerated period, and a significantly higher third exothermic peak at around 13 hours. It accounted for the assertion that limestone powder does not take part in hydration due to its inertia but acts as a filler and contributes to the nucleation of  $\text{C}_3\text{S}$  and  $\text{C}_2\text{S}$  [22].

### 3.3. Microstructure

**3.3.1. Hydration Products.** Figures 7 and 8 show the XRD patterns of the hydration products of the selected pastes at 7 and 90 d, respectively. The main peaks in pastes C are those of calcium hydroxide (CH) and ettringite. The peaks of mullite (M) and quartz (Q) in the specimen containing fly ash are clearly evident, indicating that fly ash did not influence the types of cement hydration products. This was because the main chemical composition and component were the same. In addition to the noticeable diffraction peaks of calcium carbonate, there were also obvious single carbon calcium aluminate diffraction peaks in pastes L2. This was due to the calcium carbonate which reacted with  $\text{C}_3\text{A}$  to produce single carbon hydrated calcium aluminate as the main ingredient of limestone powder.

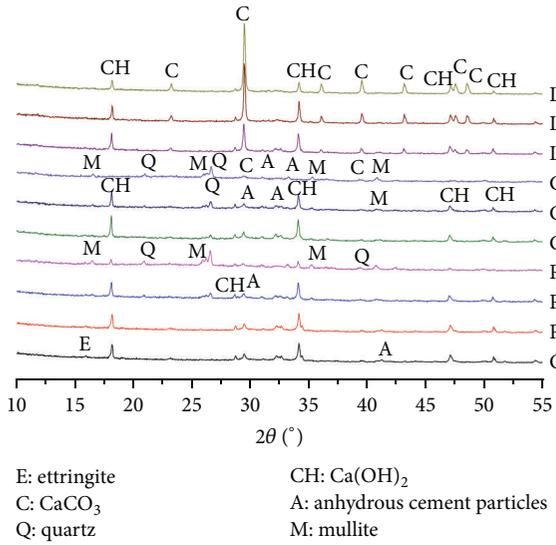


FIGURE 7: XRD patterns of specimens at 7 d.

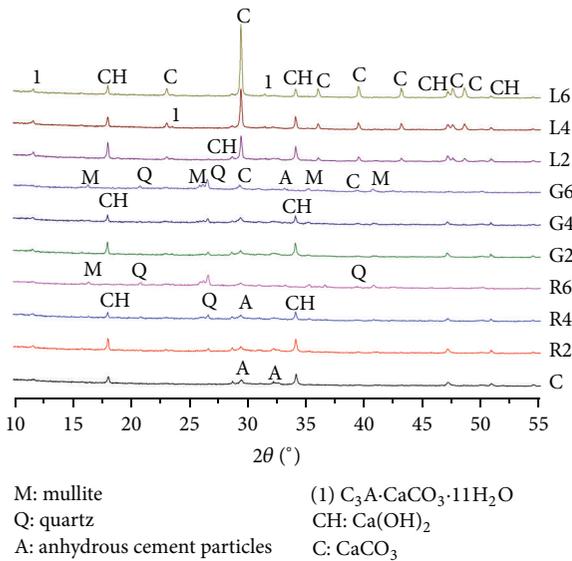


FIGURE 8: XRD patterns of specimens at 90 d.

Figures 9 and 10 also showed that the CH content in pastes R2 was lower at 90 d, indicating that the raw ash consumed some CH at 7 d. As for pastes G2, grinding may promote the activity, which made much higher consumption of CH at the secondary hydration, causing CH content at 90 d to be far less than that at 7 d. In addition, due to the consumption of cement clinker with hydration process, the characteristic peak of clinker was also reduced, which indicated exactly that fly ash exhibited a filling effect in promoting the hydration of cement to form CH. However, the ground one worked better. The CH content in pastes R2 was slightly higher at 90 d, suggesting that the raw ash was less active than the ground ash for the coarse particle, contributed to a relatively loose mortar structure. The strength of pastes R2 was lower.

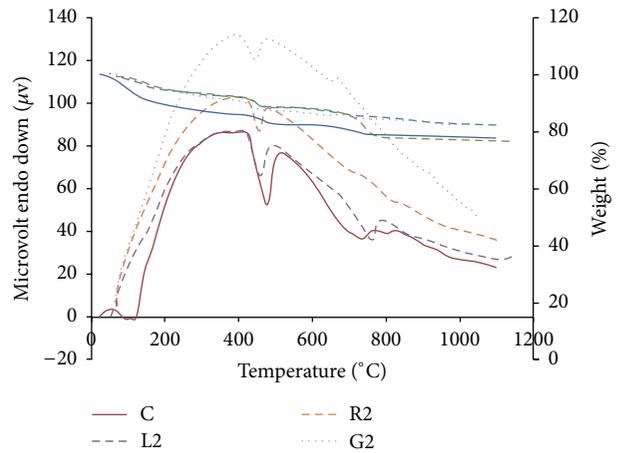


FIGURE 9: TG-DTA curves of C, L2, R2, and G2 at 7 d.

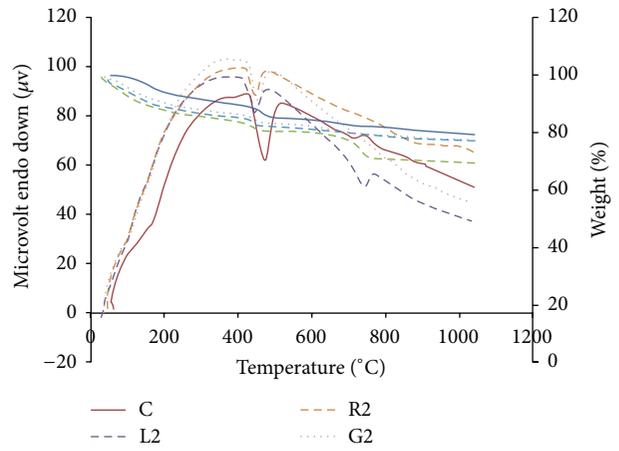


FIGURE 10: TG-DTA curves of C, L2, R2, and G2 at 90 d.

When the content of fly ash was up to 40%, the calcium hydroxide content was lower than that of the cement paste at the age of 90 d and was significantly lower than that of the sample at the age of 7 d. As a result, the early filling effect of fly ash promoted the cement hydration; however, the late pozzolanic reaction was gradual and consumed part of the calcium hydroxide. Comparing each group with admixture content of 20%, the amount of calcium hydroxide decreased at the age of 90 d for two main reasons. The first reason was the relative decrease in the cement content and the amount of calcium hydroxide produced. The other reason was that the pozzolanic activity of ash was more evident with increasing ash. Moreover, from the slurry map of ash mixed with raw fly ash, the content of calcium hydroxide changed similarly compared with that of ground fly ash indicating that the raw fly ash had pozzolanic activity.

When the content of fly ash was up to 60% (high volume), the diffraction peak of calcium hydroxide was significantly lower than that of the pure cement hydration at the age of 7 d and 90 d, respectively. The content of CH was less or even disappeared at the age of 90 d compared with that at the age of 7 d because the CH content produced by the cement

TABLE 4: Nonevaporating water content and hydration degree/mass, %.

Sample	Nonevaporating water	
	7 d	90 d
C	13.88	16.62
L2	11.5	12.02
R2	12.34	13.35
G2	12.91	14.89
L4	8.51	10.26
R4	9.33	11.53
G4	11.01	12.31
L6	2.89	3.42
R6	3.35	4.83
G6	4.16	5.19

hydration failed to meet the needs of the pozzolanic reaction of ash.

The significant differences between the pastes fully demonstrated that limestone powder promoted cement hydration with growth in age. Raw ash exhibited a filling effect in promoting cement hydration for its coarse particle and low activity. Grinding improved the activity of fly ash and was also conducive to its pozzolanic activity.

The content of nonevaporable water mainly depends on the quantity of hydration products, which relates to the hydration degree. Therefore, the content of nonevaporating water can be used to characterize the degree of cement hydration. The content of nonevaporating water is calculated as follows:

$$H = w_{105} - w_{1050} - \gamma, \quad (1)$$

$$\gamma = p_f r_f + p_c r_c,$$

where  $w$  is the mass fraction measured by TG at corresponding temperature,  $\gamma$  is the loss of ignition,  $p_f$  is the mass fraction of admixture,  $p_c$  is the mass fraction of cement and,  $r_f$  and  $r_c$  are the corresponding losses of ignition. Table 4 summarizes the results obtained from Figures 9 and 10.

Fly ash carries out the secondary pozzolanic reaction and participates in the hydration and excitation of calcium hydroxide as hydration product of cement clinker, contributing to strength. So the amount of CH consumed can indirectly reflect the hydration degree and the TG method can accurately determine the content of CH. The calcium hydroxide content is calculated according to the following equation and results given in Table 5:

$$\text{CH} = (w_{\text{initial}} - w_{\text{finish}}) \cdot \frac{74}{18}. \quad (2)$$

Table 5 lists the CH content calculated according to TG curves.

At 7 d, the nonevaporating water in the group with admixture incorporated was lower than that in group C, which showed that admixtures promoted cement hydration to varying degrees. The nonevaporable water content of

TABLE 5: CH content of the hydration products/mass, %.

Sample	Seven days	90 days
C	13.46	16.39
L2	13.36	14.81
R2	12.53	11.35
G2	12.18	11.04
L4	12.01	10.88
R4	11.98	10.65
G4	9.59	9.37
L6	4.13	3.99
R6	2.92	0.85
G6	1.23	0.00

groups G2 and R2 did not appear to be much different from each other and was just slightly higher than that of group L2. This was due to the low content of cement and the higher activity of G2 than that of R2, which accounted for the large number of nonevaporating hydrogels. In contrast, the limestone powder only promoted cement hydration. At 90 d, the content of nonevaporating water of each group differed a lot and increased with age. The content of nonevaporating water of group L2 remained stable and low, which means that the later products of L2 contained less bounded water. Meanwhile, the nonevaporating water of the pastes containing fly increased significantly, indicating that hydration products between 7 and 90 d have a rapid growth and form gel containing more nonevaporating water.

Table 5 indicated that CH content of group C and L2 pastes increased as hydration degree increased gradually, whereas that of the pastes containing fly ash decreased. From our previous analysis, it can be seen that the nonevaporating water of pastes C and the pastes containing fly ash increased greatly while that of pastes L2 tended to be stable. Since limestone powder primarily played the filling role, the content of CH consumed by fly ash in the two reactions was larger than that produced by the cement hydration. Thus, the nonevaporating water of C-S-H gel was much higher. The CH content of group R2 was significantly less than that of group L2 and obviously decreased further in group G2, which is consistent with the XRD results (i.e., remind us very briefly of what XRD says). The phenomenon indicated that grinding stimulated pozzolanic activity of fly ash effectively. With the content of limestone powder increase and cement content decrease, hydration product CH is from cement so that the CH content decreases for L4 and L6 samples. The CH content decreases with hydration age (because of the decrease of cement) and limestone content. As for R4, raw fly ash content is 40% which would decrease the hydration product CH. Otherwise, as the secondary hydration between fly ash and CH, the pozzolanic activity of raw fly ash is not as good as ground fly ash; the values of sample G4 were so low.

3.3.2. *Morphology of Hydration Products.* As shown in Figure 11, at 28 d, a large number of fibrous C-S-H gels grew as clusters of radiation and overlapped in group C. The microstructure of the pastes containing raw fly ash was

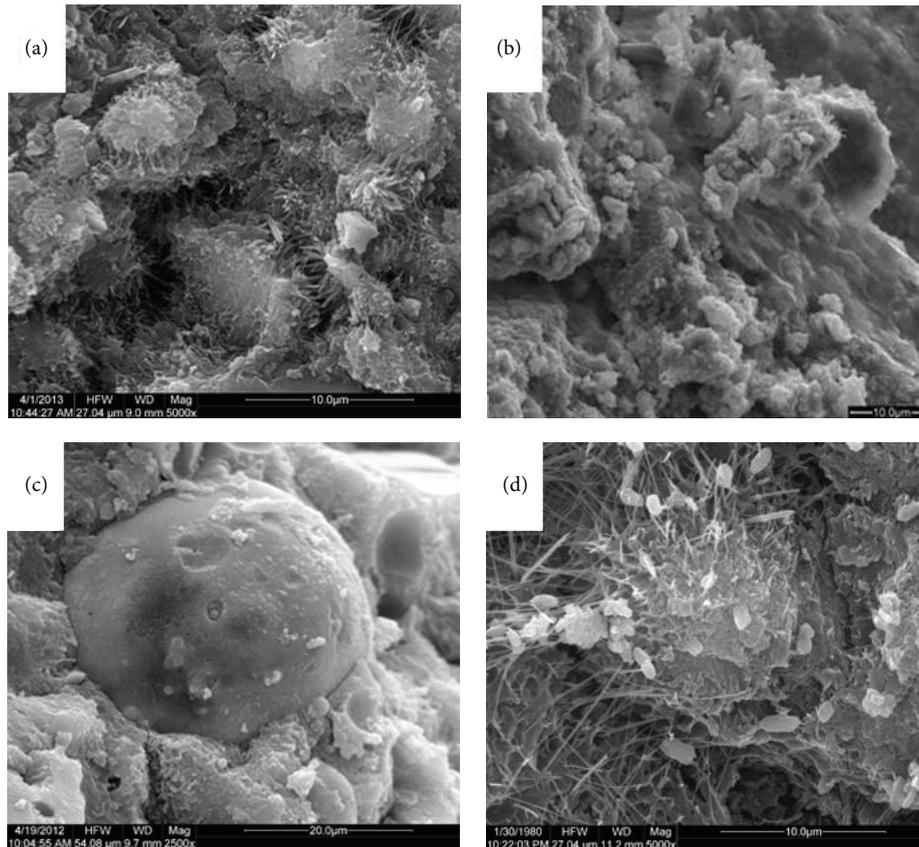


FIGURE 11: Microstructure of hydration products of (a) C, (b) L2, (c) R2, and (d) G2 at 28 d.

loose and the particle surface was substantially smooth. The clubbed C-S-H gel and irregular gel as growing hydration products attached to the particles can be observed. The observation means that fly ash mainly played the role of filling at an early stage of hydration. A large number of C-S-H gels were produced in group G2 and made the structure denser. The main hydration products of group L2 were CH, ettringite crystal, and C-S-H gel of type II netlike and type III granular; at that time, limestone powder particles were already wrapped by a lot of dense gel.

C-S-H gel and thin layered CH staggered (see Figure 12); hence the structure was denser in group C at 90 d. Moreover, a large amount of layered CH could be observed in group R2; fly ash particles were wrapped in lots of hydration products, which showed that fly ash had been involved in the hydration limitedly. This is basically the same as group G2, but the C-S-H gel which wrapped the fly ash particles at the later stage was denser. It could also be observed that the surface of the limestone powder particle had been eroded and the structure of the reaction product was relatively denser, indicating that the limestone powder had taken part in the hydration process.

Due to the thick ash, the surface of the glass beads was substantially smooth at the age of 28 d (see Figure 13). Also it was observed that increased hydration products presented in the surroundings and are attached to the surface of the structure that were relatively loose, illustrating that the fly ash primarily acted as microaggregate filler. The fly ash particles

appeared as etching and a dense layer of hydration products covered their surfaces at the age of 90 d. Again, a large amount of tufted C-S-H became very compact and dense and grew to intermingle with a laminar sheet of CH forming denser microstructure dense. Although the pozzolanic reaction of fly ash had been clearly evident, the consumption of CH was not much and the flaky CH crystals were still about. Even in the slurry sample with up to 60% of fly ash, a large number of laminated growth of CH crystals existed next to the fly ash particles with a considerable degree of reaction. This observation indicated that the consumption of CH in fly ash reaction was small, where more of the CH crystals were still in the slurry cementitious material with high volume of fly ash to maintain the internal alkaline slurry at a higher level.

#### 4. Conclusions

The results from this study support the following.

The strength of mortar decreased with increasing limestone powder content. The water-binder ratio had little influence on the development rule of the strength of mortar containing limestone powder. Limestone powder mainly played roles in promoting cement hydration and it hardly participated in hydration reaction. However, at the later period (90 d), limestone powder showed weak chemical activity. It may react with  $C_3A$  to generate monocarboaluminates.

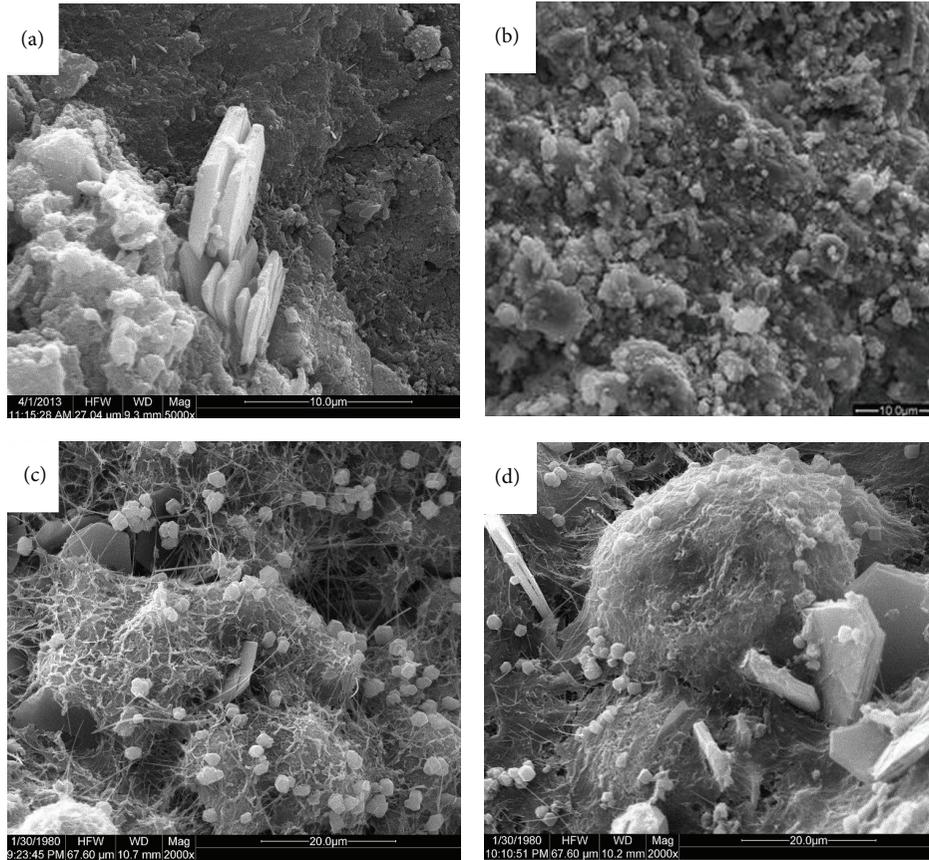


FIGURE 12: Microstructure of hydration products of (a) C, (b) L2, (c) R2, and (d) G2 at 90 d.

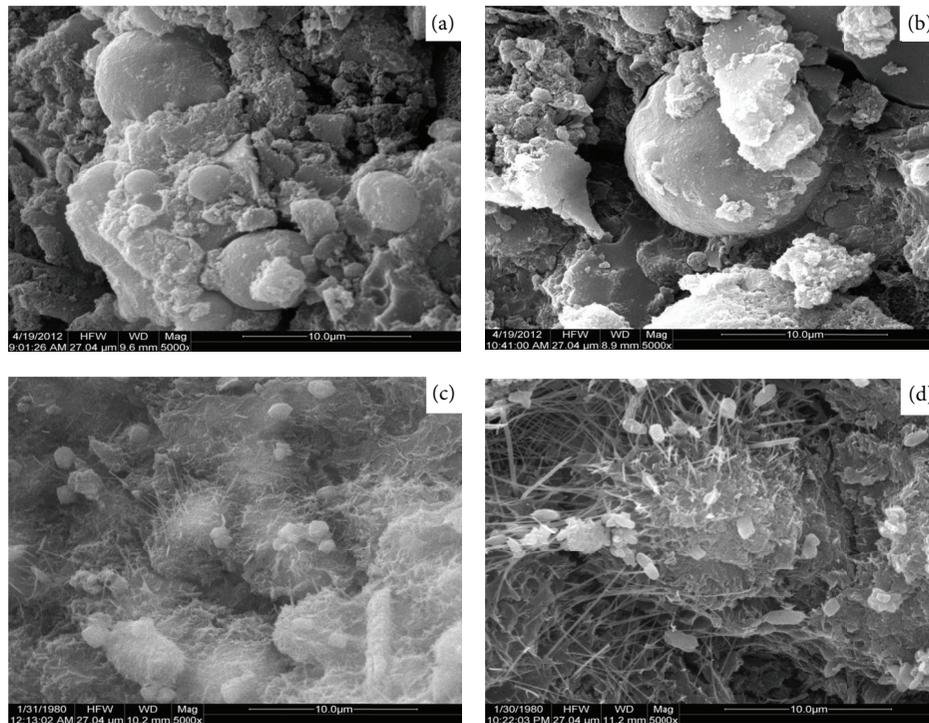


FIGURE 13: Microstructure of hydration products of ((a) and (b)) R6 and G6 at 28 days and ((c) and (d)) R6 and G6 at 90 d.

Compared with limestone powder, low quality fly ash may improve the mechanical properties of cement-based materials and ameliorate the hydration and microstructure. In addition, low quality fly ash showed better mechanical property at a low water-binder ratio. The early hydration heat of the binder containing low quality fly ash was lower than that of the binder containing limestone powder.

Grinding did not only affect the physical properties but also improve the hydration of fly ash in cement-based materials. At the early age, the activity of low quality fly ash was low; but, at the later stage, it exhibits improvements, especially after grinding. Furthermore, low quality fly ash delayed the induction period, shortened the accelerator period, and hence had little effect on the second exothermic peak.

### Competing Interests

The authors declare that they have no competing interests.

### Acknowledgments

This material is based upon work supported by the Central Nonprofit Scientific Research Fund for Institutes (no. CKSF2016003/CL); National Natural Science Foundation of China (51208391 and 51139001), China Postdoctoral Science Foundation (no. 2015M582213) and National Basic Research Program of China (973 Program, 2013CB035901).

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