

Effect of low-calcium fly ash on sulfate resistance of cement paste under different exposure conditions

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Abstract. Low-calcium fly ash (LCFA) were used to prepare cement/LCFA specimens in this study. The basic physical properties including water demand, fluidity, setting time, soundness and drying shrinkage of cement/LCFA paste were investigated. The effects of curing time, immersion time and wet-dry cycles in 3% Na₂SO₄ solution on the compressive strength and the microstructures of specimens were also discussed. The results show that LCFA increases the water demand, setting time, soundness of cement paste samples. 50% and 60% LCFA replacement ratio decrease the drying shrinkage of hardened cement paste. The compressive strength of plain cement specimens decreases at the later immersion stage in 3% Na₂SO₄ solution. The addition of LCFA can decrease this strength reduction of cement specimens. For all specimens with LCFA, the compressive strength increases with increasing immersion time. During the wet-dry cycles, the compressive strength of plain cement specimens decreases with increasing wet-dry cycles. However, the pores in the specimens with 30% and 40% LCFA at early ages could be large enough for the crystal of sodium sulfate, which leads to the compressive strength increase with the increase of wet-dry cycles in 3% Na₂SO₄ solution. The microstructures of cement/LCFA specimens are in good agreement with the compressive strength.

Keywords: low-calcium fly ash (LCFA); cement paste; sulfate resistance; wet-dry cycles; immersion

1. Introduction

Fly ash is an industrial solid waste from power industries and boiler burning coal. Fly ash has been widely used to improve the properties of concrete (Jindal *et al.* 2017, Paliwal and Maru 2017, Jena and Panda 2018, Shi *et al.* 2015, Zhang *et al.* 2011). Zhang *et al.* (1997) concluded that mortars with large content of high-calcium fly ash exhibited good compressive and flexural strength. Guerrero *et al.* (2009) reported that the flexural strength of mortars immersed in sodium sulfate liquid increased because the second hydration of fly ash decreased the porosity of mortars. Cabrera and Plowman (1988) studied the failure mechanism and the attack rate of cement partially replaced by fly ash under corrosive sulfate condition. Torii *et al.* (1995) found that concrete with high volume fly ash had an excellent sulfate resistance. Xiong *et al.* (2016) observed that an appropriate dosage of fly ash could improve the resistance to chemical sulfate attack, while the addition of fly ash might accelerate the damage caused by the physical sulfate attack. Wang *et al.* (2017) reported that fly ash improved the combined freezing-thawing and sulfate attack resistance of concrete.

Usually, it is defined as low-calcium fly ash (LCFA) when the content of CaO is less than 8%. Papadakis (1999) researched the effect of LCFA on Portland cement system and developed a theoretical model to predict the behavior of

concrete with LCFA. Poon *et al.* (2000) prepared high strength concrete with lower water to binder ratios and large dosage of LCFA. A number of studies have shown that the use of sufficient amount of LCFA can increase the chemical resistance of concrete (Tikalsky and Carrasquillo 1992). Malhotra (1990) observed the long-term performances (including resistance to chloride ion permeability, freeze-thaw cycles, and alkali-aggregate reaction) of concrete mixed with high-volume of LCFA. Roy *et al.* (2001) found that LCFA could increase the acid resistance of concrete and need an alkali activation especially for higher LCFA replacement (Bui *et al.* 2016). Irassar and Batic (1989) found LCFA delayed the cracking phenomenon of mortar in sulfate environment. Ghafoori *et al.* (2015) reported that class F fly ash improved the sulfate resistance of concrete under interrupted and continuously-immersed exposure conditions. Wang and Lee (2013) developed a numerical model to predict the chloride diffusion in concrete containing LCFA. However, in the wet-dry cycles sulfate environment, LCFA did not lead to any significant improvement in performance even when up to 40% of LCFA was used (Stark 2002).

2. Research significance

LCFA has become one of the important components of concrete. However, all or part of concrete material may be subject to sulfate corrosion under different condition, such as wet-dry cycles or complete immersion. The degradation mechanism of concrete with LCFA may be different under different conditions. The objective of this study is to

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Table 1 Chemical compositions of cement and LCFA (%)

	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	R ₂ O*	Loss
Cement	62.28	21.08	5.47	3.96	1.73	2.63	0.80	1.61
Fly ash	2.42	49.83	39.13	4.23	0.58	0.18	0.53	-

*R₂O=Na₂O+0.658K₂OTable 2 Exposure conditions in 3% Na₂SO₄ solution

	Descriptions
Effect of continuous immersion	The specimens were continuous immersed in 3% Na ₂ SO ₄ solution after 3 days curing under the standard condition.
Coupling effect of continuous immersion and curing time	The specimens were continuous immersed in 3% Na ₂ SO ₄ solution after 3, 7, 14 and 28 days curing under the standard condition.
Effect of wet-dry cycles	The specimens were firstly cured for 3 days under standard condition. In one cycle, they were totally immersed in 3% Na ₂ SO ₄ solution for 8 hours and then dried in air for another 8 hours. The temperature and relative humidity of the atmosphere is 20±2°C and 60%.

investigate the effect of LCFA and exposure conditions on the deterioration mechanisms of hardened cement paste subjected to sulfate attack. An experimental program was designed to monitor water demand, paste fluidity, setting time, soundness and drying shrinkage of specimens without sulfate attack, and the compressive strengths and the microstructures of specimens under sulfate exposure conditions were investigated. The results of this research are expected to clearly understand the deterioration mechanism of concrete with LCFA.

3. Experimental procedures

3.1 Materials

42.5 ordinary Portland cement (OPC) set by Chinese national standards GB175-2007 was used. The fly ash was a commercially available LCFA. The chemical compositions of OPC and LCFA are listed in Table 1. The aggressive sulfate was 3% Na₂SO₄ solution (weight by weight).

3.2 Specimens and test program

Cement pastes with standard consistency were prepared with and without LCFA. The replacement ratio of LCFA is 10%, 20%, 30%, 40%, 50% and 60% (by weight of cement). The specimen size is 20 mm×20 mm×20 mm. A number of specimens (20 mm×20 mm×300 mm) were cast to measure the drying shrinkage. The specimens were cured under a standard condition until the age of test. The temperature and relative humidity of the atmosphere is 20±2°C and 90%. There are three sulfate exposure conditions in this study, as shown in Table 2.

The water demand, paste fluidity, setting time and soundness of cement paste without sulfate attack were measured based on Chinese national standards GB/T 1346-2001. The drying shrinkage at early age of cement/LCFA specimens was measured according to Chinese national

standards GB/T 29417-2012 and calculated by Eq. (1). The compressive strength of specimens was recorded by a pavement material strength tester. The microstructures were observed by scanning electron microscope (SEM CS3400 CamScan UK).

$$D = \frac{L_t - L_0}{(L_0 - S_1 - S_2)} \times 10^{-6} - (T_t - T_0) \times \varepsilon \quad (1)$$

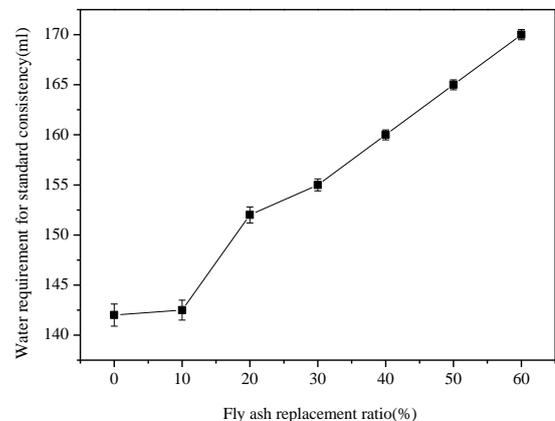
Where D is the drying shrinkage, L_0 , L_t is the initial length and the length at time t (mm), respectively. S_1 , S_2 is the length of the metal test head at both ends of specimen (mm). T_0 , T_t is the initial temperature and the temperature at time t (°C), respectively. ε is the linear expansion coefficient of hardened cement paste (13×10^{-6}).

4. Results and discussion

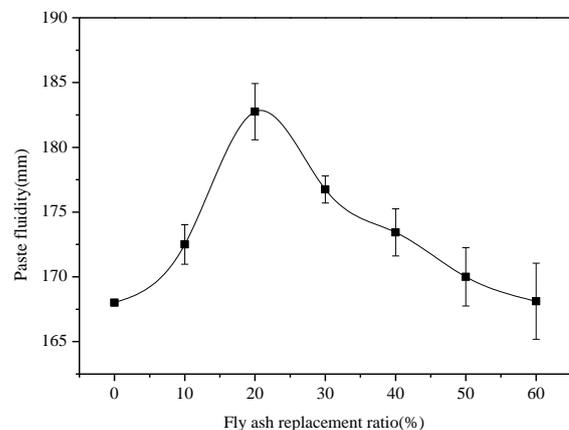
4.1 Basic physical properties of cement/LCFA paste

4.1.1 Water demand and paste fluidity

The water demand under standard consistency and fluidity of cement/LCFA paste are shown in Fig. 1. It is clear that the water demand under standard consistency increases almost linearly with the increase of LCFA replacement ratio. The fineness of fly ash is greater than that of cement.



(a) Water demand



(b) Fluidity

Fig. 1 Water demand and fluidity of cement/LCFA paste

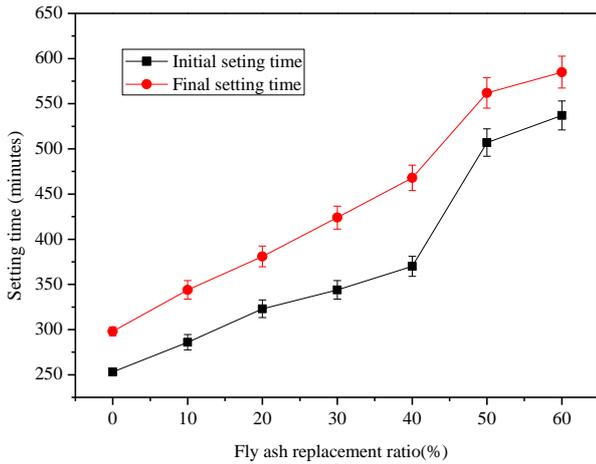


Fig. 2 Setting time of cement/LCFA paste

Fly ash tend to increase the water demand (Supit *et al.* 2014). The paste fluidity increases firstly and then decreases with the increase of LCFA replacement ratio. The maximum fluidity is 180 mm when 20% LCFA is added. LCFA in fresh cement paste has two functions. Firstly, the spherical particles of fly ash can improve the fluidity of cement paste. Therefore, 10% and 20% fly ash increase the fluidity of cement paste. Secondly, the addition of LCFA will increase the water demand under standard consistency. In this case, there may be an intersection (close to 20% in this study) between the two effects, and the fluidity of the cement paste begins to decrease after the intersection, as shown in Fig. 1b).

4.1.2 Setting time

The effect of LCFA on the setting time of cement paste is given in Fig. 2. It can be found that both initial setting time and final setting time of cement paste increase with the increase of LCFA replacement ratio. The setting time of cement is prolonged due to the second hydration of LCFA, which is in good agreement with the traditional theory. Berry *et al.* (1990) proposed a two-step mechanism for pozzolanic reaction between fly ash and Portland cement: (a) depolymerization/ silanolation of the glassy constituents of fly ash by the highly alkaline pore fluids, followed by (b) reaction between solubilized silicate and calcium ions in solution to form C-S-H. However, the rate of the pozzolanic reaction required a prolonged period of moist curing if the full benefits of fly ash were to be realized. The pozzolanic reaction of fly ash in hardened paste cured at 20°C began at the age of 28 day (Hanehara *et al.* 2001). In addition, Lam *et al.* (2000) pointed that a small percentage of the fly ash was reacted at 7 days. At different curing ages, the degree of fly ash reaction depends on the fly ash content and the water to binder ratio of the paste. The paste with high volumes of fly ash undergone a lower degree of fly ash reaction than a paste with less fly ash. Therefore, the second hydration of fly ash affects the setting time.

4.1.3 Soundness

The soundness of cement/LCFA paste is measured

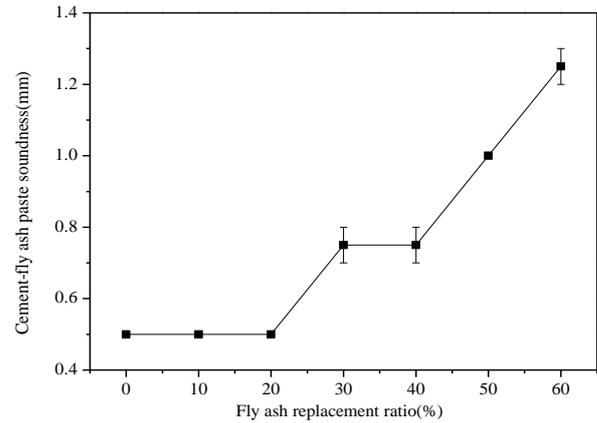


Fig. 3 Soundness of cement/LCFA paste

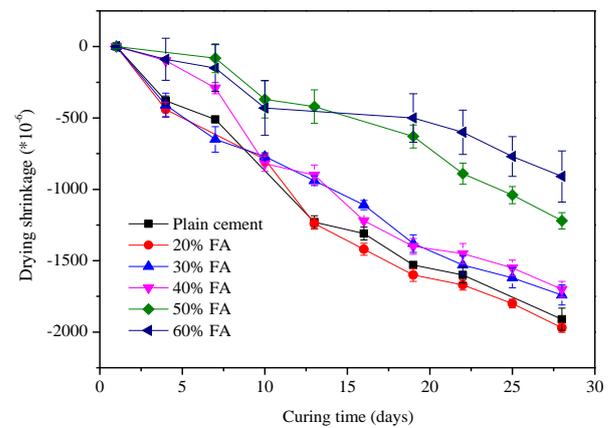


Fig. 4 Drying shrinkage of cement/LCFA specimens

before the sulfate corrosion test. The results are presented in Fig. 3. There is an increasing trend in the soundness value when the LCFA content is more than 20%. The increasing trend is obvious, especially after the LCFA content is more than 40%. That is to say, hardened cement paste with high content of LCFA shows a certain volume instability. Zhang *et al.* (1997) reported the similar results which indicated the appearance of ettringite in paste. However, all measured values are within the standard allowable range.

4.1.4 Drying shrinkage at early age

The drying shrinkage of hardened cement/LCFA paste is measured before the sulfate corrosion test. The results are shown in Fig. 4. The drying shrinkage of cement specimens with 20%, 30% and 40% LCFA is close to that of plain cement specimens. Comparing to the drying shrinkage at 28 days of plain cement, the shrinkage values are reduced by about 10% when 30% and 40% of cement paste were by LCFA. However, all shrinkage value in the measurement period is lower than that of the plain cement and the drying shrinkage at 28 days is reduced by 36% and 52% when the replacements of LCFA are 50% and 60% respectively. This result may be attributed to the slow hydration rate of LCFA. And a large number of non-hydrated LCFA particles have the function of micro aggregate which constrains the early shrinkage of hardened cement paste (Chindaprasirt 2004, Tertnkhajornkit 2005).

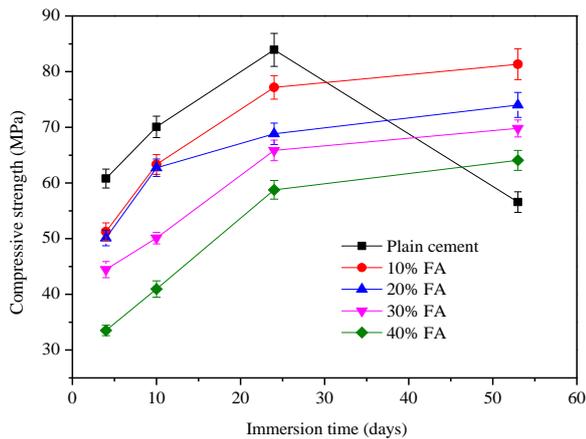


Fig. 5 Compressive strength of cement/LCFA specimens subjected to continuous immersion

4.2 Effect of immersion time on compressive strength

The Compressive strengths of cement/LCFA samples cured for 3 days and exposed in 3% Na_2SO_4 solution for 4, 11, 25 and 53 days are investigated. The results are shown in Fig. 5. It can be seen that the compressive strengths of plain cement samples firstly increase with the increase of the immersion time and then rapidly decrease when the immersion time is 53 days. The early strength increase may be due to the rapid hydration of cement and the promoting effect of sodium sulfate solution on the strength development of cement. The final strength of plain cement specimen is even lower than that of specimens cured for 3 days and exposed in 3% Na_2SO_4 solution for 4 days. That is to say, there is an obvious strength reduction at the later stage. The sodium sulfate solution will penetrate into the pores of the hardened cement paste and crystallize out in the capillary pores. Crystallization pressure will occur after the capillary pores are completely filled by the sodium sulfate crystal, which will lead to cracking and strength reduction of hardened cement paste. Micro-cracks are also observed in plain cement specimen cured for 3 days and exposed to Na_2SO_4 solution at the time of 53 days, as shown in Fig. 6a).

Comparing to plain cement specimens, the compressive strength of all specimens with LCFA increase with the increase of the immersion time and no strength reduction at the later stage is found. Shane *et al.* (2013), Lee *et al.* (2003) reported that Na_2SO_4 as an activator for high volume fly ash cement could shorten the induction period and increase the early compressive strength. However, the strength at the same age decreases with the increase of LCFA content and is lower than that of plain cement when the immersion time is less than 25 days. This result could also be explained by the slow second hydration of LCFA. The addition of fly ash increase the porosity of hardened Cement paste (Chindaprasirt *et al.* 2005) at early age because of the slow secondary hydration rate of fly ash. The sodium sulfate solution penetrates into the pores. A crystal pressure will also generate after the capillary pores are completely filled by the sodium sulfate crystal, which will cause a decrease in strength. However, this process may

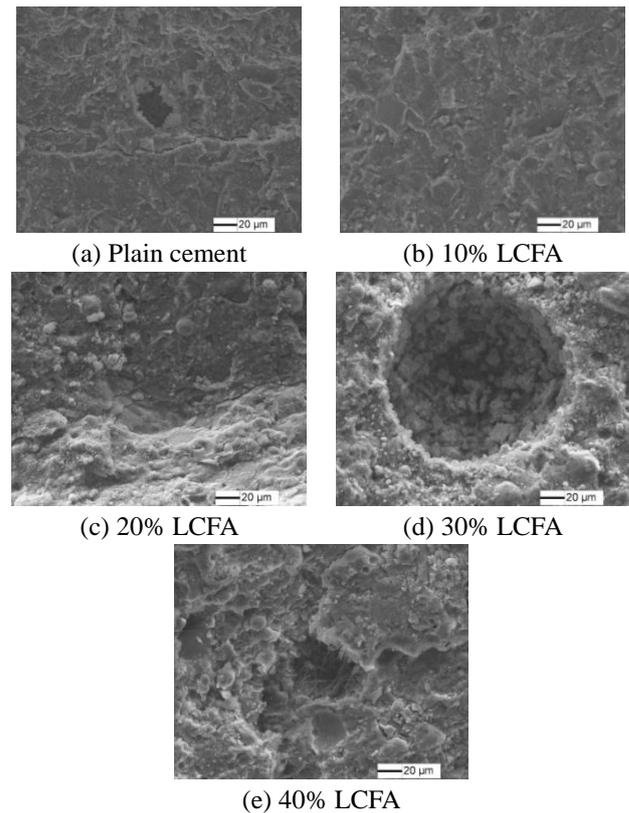


Fig. 6 Microstructure of cement/LCFA specimen cured for 3 days and exposed to Na_2SO_4 for 53 days

take a longer period of time.

The microstructures of cement/LCFA specimen cured for 3 days and exposed to Na_2SO_4 solution for 53 days are shown in Fig. 6. A large number of non-hydrated LCFA particles are still observed in the specimens with 10% and 20% LCFA, as shown in Fig. 6 (b) and (c). The new hydration products of non-hydrated LCFA particles and the sodium sulfate crystals can still be filled in cement pores when the replacement ratio of LCFA is 30% and 40%, as shown in Fig. 6 (d) and (e). Therefore, LCFA particles can continue to hydrate as long as the reaction conditions are supplied, and the specimen strength can also continue to increase until all cement pores are completely filled.

4.3 Coupling effect of curing time and immersion time on compressive strength

The specimens cured for 3, 7, 14 and 28 days were immersed into 3% Na_2SO_4 solution for 53, 49, 42 and 28 days, respectively. The total curing time and immersion time is 56 days. The compressive strengths are measured immediately after the immersing process, as shown in Fig. 7. It can be found that the coupling action of the curing time and the immersion time has little influence on the strength development of plain cement specimens. That is to say, the strength development speed of hardened cement paste in 3% Na_2SO_4 solution or under the curing condition is relatively close. However, the compressive strength of specimens with LCFA increases with increasing immersion time, which indicates that a longer immersion time in 3%

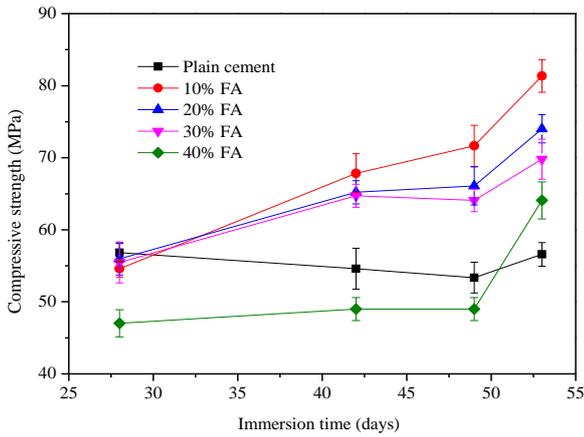


Fig. 7 Compressive strength of cement/LCFA specimens cured for different time and subjected to continuous immersion

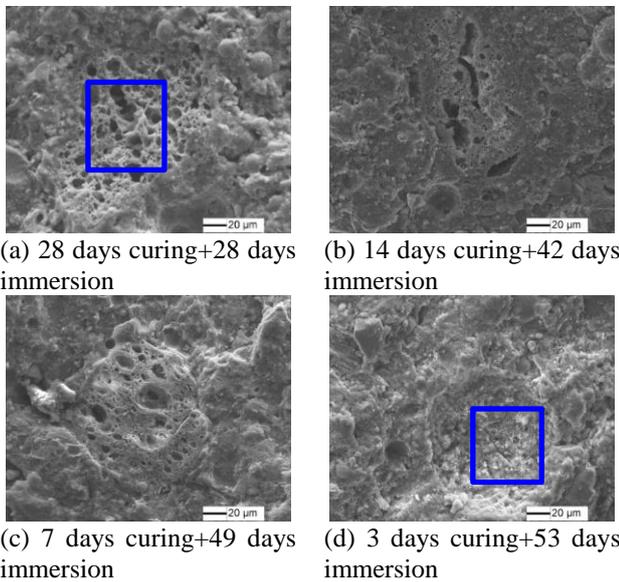


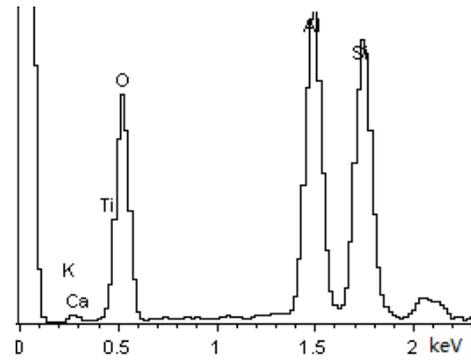
Fig. 8 microstructure of cement specimen with 40% LCFA

Na_2SO_4 solution is more beneficial to increase the early compressive strength of specimens with LCFA.

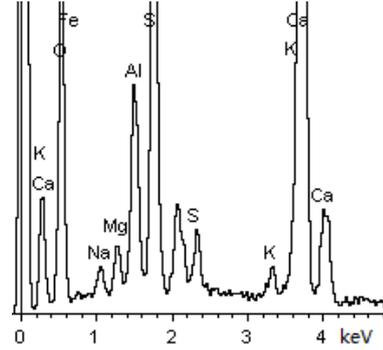
For the specimens with 40% LCFA, the compressive strength did not increase significantly until the specimens were immersed in 3% Na_2SO_4 solution for 49 days. The reason for the slow increase in the early strength may be attributed to the slow hydration of LCFA, especially for the specimens with higher LCFA content.

The microstructures of cement specimen with 40% LCFA are shown in Fig. 8. A number of unfilled pores are still observed in the matrix exposed to 3% Na_2SO_4 solution for 28, 42 and 49 days, as shown in Fig. 8 (a) to (c).

From the energy spectrum analysis in Fig. 9(a), it is known that the material at the pores is the cement hydration product. However, the pores have been filled by the hydration products when the specimen is exposed to 3% Na_2SO_4 solution for 53 days, as shown in Fig. 8(d). Although Na_2SO_4 solution can accelerate the early hydration of cement/LCFA system, the effect only contributes to the compressive strength when the pores are



(a) 28 days curing+28 days immersion



(b) 3 days curing+53 days immersion

Fig. 9 Energy spectrum images

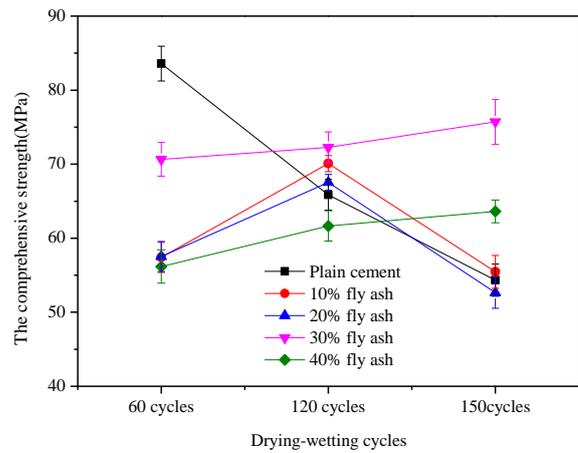


Fig. 10 Compressive strength of cement/LCFA specimens subjected to wet-dry cycles

almost completely filled the hydration products. Sulfur element is found in the pore product (as shown in Fig. 9(b)), which indicates that Na_2SO_4 solution has penetrated into the pore.

4.4 Effect of wet-dry cycles on compressive strength

The Compressive strengths of cement/LCFA specimens cured for 3 days and subjected to wet-dry cycles in 3% Na_2SO_4 solution are shown in Fig. 10. It is clear that the compressive strength of plain cement specimens decreases with the increase of wet-dry cycles. This result can be explained by the traditional theory of the chemical and physical action of sulfate to hardened cement paste (Stark

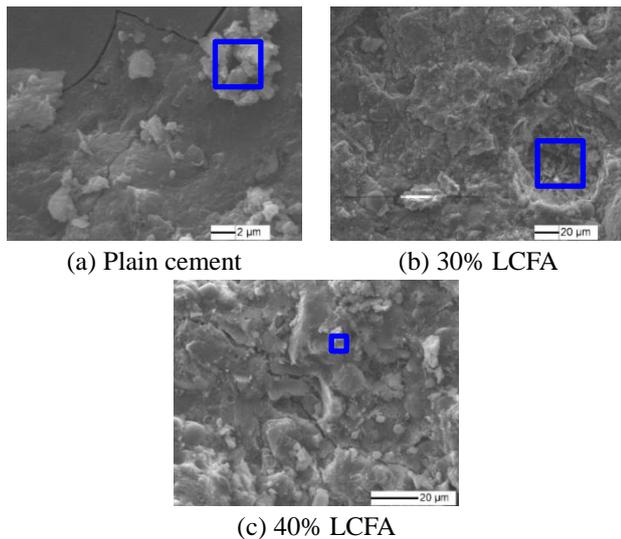


Fig. 11 Microstructures of cement/LCFA specimens subjected to 150 wet-dry cycles

2002). A number of micro-cracks in the microstructure of plain cement with 150 wet-dry cycles in 3% Na₂SO₄ solution further verify the reasons for the decrease of compressive strength, as shown in Fig. 11(a). The compressive strengths of cement specimens with 10% and 20% LCFA firstly increase with the increase of wet-dry cycles and then decrease when the wet-dry cycles are 150. In the early wet-dry cycles, the crystal of sodium sulfate has a filling action to the pores in cement paste. However, too many crystals formed in the later wet-dry cycles will produce expansive stress to hardened cement paste, which will lead to the formation of micro-cracks and the decrease of compressive strength (Djuric *et al.* 1996).

For cement specimens with 30% and 40% LCFA, there are a number of pores in the matrix at the early age because of the slow hydration of LCFA, as shown in Fig. 11 (b) and (c). Therefore, the specimens with high volume LCFA could provide enough growth space for the crystal of sodium sulfate, which is also the reason that the compressive strength increases with the increase of wet-dry cycles. However, if the wet-dry cycles continue to increase, the expansive stress may also lead to micro-cracks and strength reduction when the volume of sodium sulfate crystal is more than that of the pores in hardened cement paste.

The energy spectrum analysis is shown in Fig. 12. It is clearly that sulfur element is found in the specimens after 150 wet-dry cycles 3% Na₂SO₄ solution. Sulfur element may be present in the sulfate crystals. The salt crystallization is a physical phenomenon which could be the cause of faster deterioration, especially in wet-dry cycles. A common form of physical sulfate attack occurs when sodium sulfate crystallizes in the pore structure of cement, the typical consequence of physical sulfate attack of specimens is surface erosion. Sodium sulfates may interconvert between the 10 hydrate and the anhydrous salt. Thenardite has been reported to be capable of producing pore pressures of 400-5000 psi while mirabilite Na₂SO₄·10H₂O produces pore pressures of 1000-1200 psi (Brown 2002).

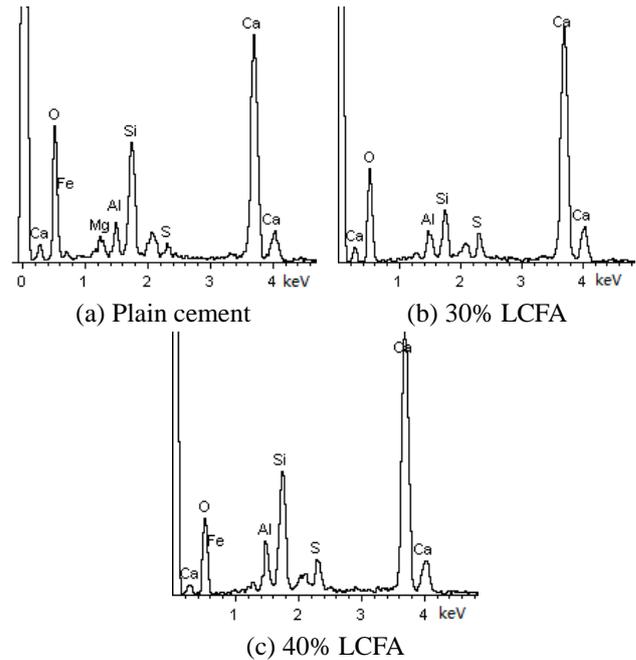


Fig. 12 Energy spectrum images

5. Conclusions

- 1) The addition of LCFA increases the water requirement, setting time, soundness of cement paste samples. 50% and 60% LCFA decrease the drying shrinkage of hardened cement paste.
- 2) During continuous immersion after 3 days curing, the compressive strength of plain cement samples firstly increases with the increase of the immersion time and then rapidly decreases when the immersion time is 53 days. For all specimens with LCFA, the compressive strength increases with the increase of the immersion time.
- 3) The coupling actions of the curing time and the immersion time have little influence on the strength development of plain cement specimens. The compressive strength of specimens with LCFA increases with the increase of immersion time.
- 4) The compressive strength of plain cement specimens decreases with the increase of wet-dry cycles. For specimens with 10% and 20% LCFA, the compressive strength firstly increases with the increase of wet-dry cycles and then decreases when the wet-dry cycles are 150.
- 5) The early compressive strength of the specimens with 30% and 40% LCFA increases with the increase of wet-dry cycles in 3% Na₂SO₄ solution.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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