



Effect of sodium sulfate and nano-SiO₂ on hydration and microstructure of cementitious materials containing high volume fly ash under steam curing

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HIGHLIGHTS

- The synergistic effect of NS and SS on hydration of CHVFA system is found.
- NS has much stronger effect on refining aperture and promoting the hydration of PC.
- SS exerts much stronger effect on accelerating the hydration of FA.
- NS and SS co-doped decrease the Ca/Si ratio and the enrichment degree of CH of ITZ.

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ABSTRACT

Generally, high volume fly ash would considerably reduce the early strength of cement-based materials, and because of this disadvantage, the added amount of fly ash in steam-cured precast concrete is very limited. In this study, an attempt to enhance the early strength of steam-cured cement-high volume fly ash (40 wt%, CHVFA) system has been done with addition of nano-SiO₂ (NS) and sodium sulfate (SS). Effect of these two chemicals on hydration, pore structure, and microstructure of interfacial transition zone (ITZ) were investigated. The results show that both NS and SS can improve the compressive strength, and the reason is mainly because these two chemicals can accelerate the hydration of cement minerals and can also hasten the pozzolanic reaction of fly ash. By contrast, because of the high activity of pozzolanic reaction and the filling and nucleation effects, NS has much stronger effect on refining pore structure and promoting the hydration of cement; while SS can exert much stronger effect on accelerating the hydration of fly ash than that of NS. An interesting thing can also be seen that with co-doped NS-SS, the enhancement in compressive strength is much stronger than that of the sum of these single systems with the same dosage, indicating that NS and SS can exert the synergistic effect; this effect is involved in the accelerated hydration of CHVFA, the refined pore structure, and the improved microstructure of ITZ. Such results would be expected to provide guidance on enhancing the early strength of steam-cured precast concrete with high volume fly ash in practical engineering.

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1. Introduction

Coal is the main energy source in China, and in current and coming decades, it still occupies the dominant position in primary energy structure. Fly ash (FA) is a by-product of coal-fired power plant, and every two tons of coal consumption can produce one

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ton of FA. FA output in China was reached 620 million tons in 2015 according to estimates, being the highest in the world [1]. The mass stack of industrial solid waste not only pollutes the ecological environment but also does great harm to the human beings, and squanders resources as well, not in agreement with the requirements of sustainable development. So effective utilization of FA has great environment and economic benefits.

In past decades, FA has been widely used in concrete industry as one of the most popular supplementary cementitious materials (SCMs), not only for environment and economic purpose but also

for the purpose to improve the performance of cement-based material. The mechanism behind the improvement can be revealed: Firstly, FA microspheres are conducive to optimize the fluidity of the cement paste and significantly reduce water demand to achieve the necessary workability of concrete [2]. Secondly, the pozzolanic reaction of FA consumes calcium hydroxide to induce the hydration of cement clinker, and also benefits to disperse cement particles to increase the contact area between water and cement clinker, all of which would increase the hydration degree of cement [3]. Thirdly, the unhydrated part of FA can exert filling effect as micro-aggregates to refine the pores, further improving the density of concrete [4]. Moreover, the hydration heat and the hydration exothermic rate can be markedly reduced by the addition of FA, which lowers the concrete temperature rise to avoid cracks [5–9].

However, a widely accepted disadvantage is that the high volume FA can seriously reduce the early strength, and because of this disadvantage, the use of FA in precast concrete is very limited. The main reason for this is that compressive strength is compulsory to reach more than 20 MPa to ensure enough mechanical strength for the demoulding process. In order to promote the production efficiency and the use efficiency of mould, steaming curing is often employed. In spite of this, the early compressive strength is still hard to reach compulsory level if high volume FA used. In most case, the added dosage is less than 15%. As a consequence, to improve the early strength of steam-cured cement-FA system is of great importance for high volume FA system in precast concrete.

In order to enhance the early strength of cement-FA system, many attempts have been done by incorporating nanoparticles. The addition of nano-material can promote the early hydration of both cement and FA, contributing to the early strength. As reported, this contribution could be obtained with addition of nano-SiO₂ [10–12], nano-TiO₂ [13] or carbon nanofibers [14]. Nano-SiO₂ has attracted significant attention in research area for its superior properties. It has been proven that nano-SiO₂ not only possesses good pozzolanic reactivity and provides nucleation sites for hydration products at early ages, but also acts as the pore filler to modify the microstructure and interface transition zone (ITZ) to make the paste become denser [10–12]. The study of Gao et al. [15] showed that, compressive strength of lightweight aggregate concrete (LWAC) was continuously improved with the increasing content of NS, and 2% NS increased the strength of LWAC containing 18% FA by 21.4%. Shaikh et al. [16] also evaluated the effect of NS on compressive strength of high volume FA system. It was observed that with the addition of 2% NS in mortars containing 40% and 50% FA, compressive strength at 7 days was increased by 5% and 7%, respectively. Hou et al. [17,18] studied the effect of colloidal NS on compressive strength of mortar with high volume FA, and the results showed that 5% colloidal NS could increase the compressive strength of mortar containing 60% FA by approximately 80%. Based on the discussion above, it could be concluded that addition of nanoparticles can promote hydration of the cement-FA system, with an obvious increase in compressive strength.

Furthermore, many studies have shown that chemical activator can also promote the activity of FA as well as the hydration of cement minerals at early ages, and a lot of progress has been made all over the world. Sodium sulfate (Na₂SO₄, SS) has been reported as a kind of ideal activator, with the dual excitation effects of alkali and sulfate [19,20]. On the one hand, the ionized sulfate can react with AlO₂⁻ in the liquid phase to promote the formation of ettringite (Aft) [21], which can hasten the dissolution of FA to compensate the consumption of AlO₂⁻, thereby accelerating the pozzolanic reaction of FA. Since the compactness of Aft layer is lower than that of C-S-H, Ca²⁺ in solution is much easier to diffuse into interior of FA particles and react with the active SiO₂ and Al₂O₃, showing

higher reaction activity of FA [19–27]. On the other hand, the addition of Na₂SO₄ increases the alkalinity of the system, which can also promote the dissolution of SiO₂ and Al₂O₃ into liquid phase to form C-S-H gel and C-A-S-H gel, thereby hastening the reaction activity of FA [19–27].

Although the effects of single system of nano-SiO₂ and Na₂SO₄ on the hydration process of cement-FA system have been extensively studied, the results of combined use of these two chemicals are very limited, especially in high volume FA system under steam curing. Thus, in this study, the influence of the binary system of nano-SiO₂ and Na₂SO₄ on the hydration and microstructure of steam-cured cement-high volume FA (40 wt%, CHVFA) system at the early age was investigated. The compressive strength was measured to evaluate the mechanical performance. Pore structure and microstructure of interfacial transition zone (ITZ) were studied with MIP and SEM-EDS, and the effect of these two chemicals on hydration was discussed with XRD, DSC-TG ²⁹Si NMR and SEM. Based on these discussions, the mechanism behind the improvement in compressive strength was revealed in terms of hydration, ITZ and pore structure. Such results would be expected to provide guidance on promoting the use of FA in precast concrete.

2. Experimental

2.1. Materials

Portland cement (PC) CEM I 42.5 produced by Huarun cement Co., Ltd. was used in all mixes. Fly ash (FA) produced by Yangluo power plant in Wuhan of China was used as SCMs, in accordance with the Chinese standard GB1596-2005 [28]. Nano silica (nano-SiO₂, NS) with average particle size of 15 nm produced by Aladdin Biological Technology Co., Ltd. was used. The reagent-grade sodium sulfate (SS) produced by Sinopharm Chemistry Reagent Co., Ltd. was used as activator. Polycarboxylate superplasticizer (SP) with a solid content of 40.0% was added to ensure the same fluidity level with the same water/binder ratio. The chemical composition of PC and FA, obtained with X-ray fluorescence spectrometer (XRF, Axios advanced), are shown in Table 1, and the physical properties of PC are shown in Table 2.

The particle size distribution of cement and FA was tested with a Mastersizer 2000 laser particle size distribution instrument, and the results are shown in Fig. 1. The phase constitution and microstructure were studied with XRD, SEM and TEM, respectively, as shown in Figs. 2 and 3. XRD patterns indicate that the NS is amorphous material and is relatively less crystal than PC and FA. SEM images show that FA particles are perfectly spherical shape and PC particles are somewhat irregular. TEM image confirms that the particle size of NS is about 15 nm.

Standard sand produced in Xia'men of China, in accordance with GB/T14684-2011 [29], was used as aggregates for mortars. The deionized water was used throughout the experiment.

Table 1
Chemical composition of PC and FA.

Chemical analysis	PC (wt%)	FA (wt%)
CaO	56.04	11.00
SiO ₂	22.82	47.00
Al ₂ O ₃	7.59	24.23
Fe ₂ O ₃	3.20	4.18
MgO	2.72	1.10
Na ₂ O	0.13	0.59
K ₂ O	0.55	1.84
SO ₃	2.73	1.59
Loss on ignition (LOI)	3.18	6.65
Temperature used to calculate LOI (°C)	1000	1000
Time used to calculate LOI (min)	60	60

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