

A new dispersing method on silica fume and its influence on the performance of cement-based materials



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HIGHLIGHTS

- The zeta potential of the modified silica fume is much higher than the commercial silica fume.
- The higher fluidity of S2 paste mainly benefits from improvement of dispersion of the modified silica fume.
- The modified silica fume increases the hydration rate and hydration degree of cementitious material.
- The micrographs show that the modified silica fume particles are homogeneously dispersed in S2.
- The mean value of Ca/Si atom ratio of the C-S-H gel in S2 with modified SF is lower than in S1 with commercial SF.

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ABSTRACT

Because the agglomerated silica fume can't realize its filling effect, hydration nucleation effect and pozzolanic effect in cement-based composites, a new dispersing method on silica fume aiming to dispersing it fully is introduced. The influence of physical and chemical properties of the modified silica fume on performance of cement-based materials was investigated. In view of the presence of silanol groups and negative charges of silica fume surface, the zeta potential of silica fume surface becomes positive after reaction of silanol groups on silica fume surface and calcium ions in modification solution. The change of zeta potential generates stronger adsorption force to anionic polycarboxylate copolymer. This paper discusses in detail the effect of the modified solution with different concentrations and pH values on adsorption amount of calcium ions on the surface of silica fume, thereby the optimal modified system was determined. The optimized dispersion of the modified silica fume was characterized by infrared spectra and comparing with the result of the commercial silica fume. The performance of modified silica fume in the simulated cement pore solution was evaluated by zeta potential and adsorption amount of polycarboxylate copolymers on silica fume particles surface. Besides, laser particle distribution and SEM photos help us clarify the difference between the particle size distribution and microstructure of the modified silica fume and the commercial silica fume. Results show that zeta potential, adsorption amount of polycarboxylate copolymers on modified silica fume surface increases obviously, the homogeneous dispersion fraction of the modified silica fume in the simulated cement pore solution increases significantly. Furthermore, influence of modified silica fume on the performance of cement-based materials was evaluated. Results showed that the effects of modified silica fume on fluidity, mechanical properties, hydration heat, non-evaporable water, hydration products, pore structure and morphology of cement-based materials are optimizer than the commercial silica fume. And the Ca/Si ratio of the C-S-H gel of cement-based material containing modified silica fume is lower than that of cement-based material containing commercial silica fume.

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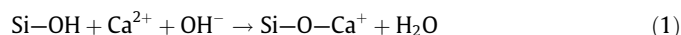
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1. Introduction

Silica fume as a kind of mineral admixture with a high mineral reactivity has been widely used in cement-based materials. The reason why silica fume has a high mineral reactivity is that on the one hand silica fume contains a high SiO_2 active ingredient, which is able to cause a hydration reaction with $\text{Ca}(\text{OH})_2$ produced by cement hydration, on the other hand silica fume is a kind of ultra-fine powder containing a large of nano-particles, it can play a good filling effect and nucleation effect when it is used in cement-based materials [1,2]. But, the surface energy of silica fume containing nano-particles is very high. When silica fume presents in the form of individual particles, they are able to attract each other to close and reunite spontaneously. However, this phenomenon is easy to cause surfactivity of silica fume to decrease, and weaken the practical performance of silica fume [3,4]. Therefore, how to eliminate the agglomeration effect of silica fume particles has been a hot research. Currently dispersion method of silica fume is mainly divided into physical and chemical methods. Physical dispersion methods include ordinary mechanical stirring dispersion, grinding and stirring dispersion in a high speed, ultrasonic dispersion etc. Chemical dispersion methods are complied by the use of different types of anionic dispersing agents, which include various types of anionic superplasticizer, lignin sulfonate and its derivatives, hydroxy acids (salts), organic and inorganic phosphoric acid (salts), and various types of anionic polymer etc [5]. However, it may have a significant negative impact on the performance of cement-based materials when the amount of dispersing agents is excessive, like introduction of a large number of bubbles, retarding the setting time of cement paste, rapidly degradation in high alkali pore solution of cement paste and a significant reduction in strengths of hardened cement-based composites [6].

This study presents a new dispersing method to prepare a superior dispersion suspension of silica fume, which can stable exist in high alkali cement paste. There are a large number of silanol groups on the surface of silica fume particles, these groups are expected to act like a weak acid ($K_1 = 10^{-9.8}$) [7]. The weak acidic bonds are capable of conducting an acid-base reaction with $\text{Ca}(\text{OH})_2$ in alkaline pore solution of cement paste, thereby the Ca^{2+} ions are able to be adsorbed on the surface silica fume. The acid-base reaction (electron transfer reaction) is shown in Eq. (1):



Thence, the zeta potential of the silica fume is turned into positive potential. Then the silica fume adsorbing Ca^{2+} ions is able to adsorb different types of anionic dispersing agents to achieve the dispersion of silica fume [8,9]. But this process will take some time and was achieved in pace with the cement hydration process, plus the ion species are very complex in the pore solution of cement paste, dispersion effect of silica fume is limited [10]. In order to achieve a better dispersion on silica fume, this research put forward a pre-treatment method on silica fume, which was able to turn the zeta potential of silica fume into be positive in advance, then the modified silica fume and anion superplasticizer were together incorporated in cement-based materials to achieve better dispersion for silica fume. In order to explain the influence mechanism of the modified silica fume on dispersion effect, this paper studies the effect of the modified solution with different concentrations and pH values on adsorption amount of calcium ions on the surface of silica fume. The performance of modified silica fume was evaluated by measuring zeta potential and adsorption amount of polycarboxylate copolymers on silica fume particles surface in cement pore solution. Meanwhile, we compared the differences of particle size distributions and morphology (characterized by SEM pictures) between the modified silica fume and the

commercial silica fume. Furthermore, the influence of modified silica fume on the fluidity, mechanical properties, hydration heat, non-evaporable water, hydration products, pore structure and morphology, Ca/Si ratio of the C-S-H gel of cement-based materials were evaluated as well.

2. Experiments

2.1. Materials

The Portland cement used was P-II42.5 in accordance with the relevant Chinese standard. Dried SF powder was obtained from Elkem Carbon Co., Ltd, China. The compositions of the cement and SF are shown in Table 1, the XRD pattern of SF is shown in Fig. 1, the amorphous structure of SF was revealed by the XRD pattern (illustrated in Fig. 1). The polycarboxylate superplasticizer was from Sika Co., Ltd, China, the water-reducing ratio was more than 20%. Water used in the experiments was tap water.

A synthetic pore solution of cement paste was prepared by dissolving per 9.72 g $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ in 148.5 mL water, pH of $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ solution was adjusted by NaOH, showing a pH of 12.2, was employed as mixing water to mimic Ca^{2+} ion content and pH value of cement paste pore solution.

2.2. Modification of silica fume

Firstly, saturated $\text{Ca}(\text{OH})_2$ solution was prepared, then $\text{Ca}(\text{OH})_2$ aqueous solution was prepared by the volume ratio of 1:2 between saturated $\text{Ca}(\text{OH})_2$ solution with water. The SF paste was prepared by the mass ratio of 1:10 between silica fume with the prepared $\text{Ca}(\text{OH})_2$ aqueous solution after stirred for 30 min. Then the prepared silica fume paste was filtrated after being stewed in room temperature for 24 h. And the filtration bottle was placed on electronic scale simultaneously, the filtration was stopped when the mass ratio between silica fume with $\text{Ca}(\text{OH})_2$ aqueous solution was 1:1 by calculation, at this time the silica fume was modified successfully, and the remaining silica fume paste (denoted as F) was seen as a unified suspension for casting cement-based materials. The reason why the modified silica fume paste was seen as a unified suspension is to ensure the dispersion stability of the modified silica fume. Water usage of cement-based material contained the water contained in F. However, some researchers might to question whether $\text{Ca}(\text{OH})_2$ modifying solution can react with silica fume to generate C-S-H gel so much so that not conducive to the dispersion of silica fume. So, in order to prevent emergence of this adverse result, influence of different contents of $\text{Ca}(\text{OH})_2$ in modified solution and different modified time on dispersion of silica fume was studied, thereby a kind of rational modifying regulation was determined. The improvement of space dispersion of modified silica fume will be confirmed by FTIR spectra, SEM photos and hydration heat of cement-based materials.

2.3. Mixture proportion of cement-based materials and preparation of samples

Two mixtures were designed in this paper. The recipe of the cement-based materials is listed in Table 2. Cement was partly replaced by SF, the w/b was 0.29, the dosage of superplasticizer was 1% (incorporated additionally) of total cement-based binder. When the cement-based material containing the modified silica fume was prepared, the water usage involved the water contained in modified silica paste (F). The cement-based material containing the commercial silica fume was denoted as S1, another containing the modified silica fume was denoted as S2.

To prepare the S1 and S2 samples, the superplasticizer was dissolved in water and then the SF was incorporated and mixed into the cement paste for 2 min. After that, the cement was put into the mixer and mixed for 3 min. Afterwards, the fresh mixtures were cast into steel moulds and compacted via a standard vibrating table. The specimens were demoulded after 24 h and cured under standard conditions ($20^\circ\text{C} \pm 2^\circ\text{C}$, RH > 90%) for designated ages (3 d, 7 d, 28 d) before testing.

2.4. Test methods

The pH values of the modifying solution and the adsorption amount of calcium ions on the surface of modified silica fume can be determined by the ion meter, pH electrode and calcium ion electrode was employed respectively. Calculation formula of adsorption amount of calcium ions is by Eq. (2):

$$\Gamma_{\text{Ca}}^{2+} = V(C^\circ - C)/m \quad (2)$$

where Γ_{Ca}^{2+} (mol/g) is the adsorption amount of calcium ions, V (mL) is the volume of modifying solution, C° (mol/mL) and C (mol/mL) is the calcium ion concentration of modifying solution before and after adding in silica fume respectively, m is the mass of silica fume.

When zeta potential of commercial silica fume was tested, 1 g of the dried commercial silica fume was mixed in 500 mL simulating cement pore solution, and was stirred for 5 min with a glass rod. Then, a small amount of silica fume solution was used to test the zeta potential with a zeta potential analyzer. However, when zeta

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