



Improvement of mechanical properties and chloride ion penetration resistance of cement pastes with the addition of pre-dispersed silica fume

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HIGHLIGHTS

- We study the different influence factors on the stability of DSF suspension during ball milling process.
- We add DSF into cement paste to study the effect of DSF on the hydration process of cement.
- The ball milling process reduces the particle size of SF and promotes the uniform dispersion of SF in cement pastes.
- DSF can be applied to significantly improve the mechanical properties and chloride ion penetration resistance of cement pastes.

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ABSTRACT

The addition of pre-dispersed silica fume (DSF) into cement pastes can enormously improve the engineering property of cement pastes. Yet it is hard to evaluate the distribution of DSF and the adsorption process between cement and DSF quantitatively. Therefore, an approach (the titration test) was used to investigate the distribution of DSF and the adsorption process quantitatively in cement pastes. After the examination of the physical and chemical properties of DSF modified cement-based materials, ideal results were obtained in this work. This paper highlights the primary findings in manufacturing and characterizing of DSF modified cement-based materials. For macroscopic performance, the pressure testing machine was used to test the mechanical properties of DSF modified cement-based materials. Meanwhile, Zeta potential analyzer was used to monitor the surface charges of particles in cementitious materials with different DSF contents. From a microcosmic perspective, scanning electron microscope (SEM) was used to capture the microstructure of the compound materials. In addition, to research the influential factors on DSF in the process of ball milling, ball milling time and the parameter of DSF of each ball milling process and other components added into the liquid were taken into account. Finally, the hydration process was simulated. The results show that the DSF can improve the mechanical properties and chloride ion penetration resistance of the sample. The simulated hydration process consists of five stages, which are agglomeration-trigger adsorption (Stage I), initial stable adsorption (Stage II), free-dispersion (Stage III), reagglomeration-trigger adsorption (Stage IV) and secondary adsorption (Stage V). The five stages reflect the corresponding reaction characterization of DSF modified cement-based materials.

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1. Introduction

Silica fume (SF), a by-product obtained from the production of ferro silicon and silicon metal, is noncrystalline silica dust formed

during the oxidation of SiO vapor [1]. Usually the silicon oxide content in SF is more than 90 wt%, in which the reactive silica oxides are above 40 wt%, which allows the high efficiency of pozzolanic activity of SF. The practice of specifying SF in concrete has been widely conducted in the Middle East, where engineers are trying to improve the durability of concrete by adding SF [2].

It is widely accepted that incorporating proper amount of SF as an additive in cement-based composite materials leads to

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mechanical properties enhancement [3], along with the improvement of durability [4], such as, resistance to chloride ion penetration [5], acid corrosion [6], sulfate attack [7], carbonation [8] and freeze-thaw cycling damage [9]. Experiments have shown that compared to neat Portland cement pastes, SF incorporation not only leads to an increase of rheological property [10,11] but also provides the full range of engineering properties [12,13]. SF also has served as a cement replacement material in normal Portland cement concrete so as to obtain a desired compressive strength [14]. Due to pozzolanic effect and particle filling effect of SF, the pore structure of concrete can be significantly reduced and the compactness can be greatly improved, thus improving the mechanical properties and anti-chemical corrosion performance of the sample [15,16].

In order to increase the conductivity of the composite and the dispersion and stabilization of microfibers, some researchers added SF into cement pastes incorporated with carbon microfiber [17–20]. SF is also an ideal component to increase the packing density of the cementitious materials and reduce the quantity of voids which will be filled by water [21]. Kwan and Fung [22] demonstrated that adding condensed SF could increase the packing density and therefore improve the flow-ability of mortar. Zhang [23] proposed a wide and gap-graded particle-size distribution based on the close-packing theory and showed that such particle-size distribution would increase the packing density and reduce the water requirement. Due to the weak apparent density of SF, SF is easy to agglomerate, which will affect the performance of cement concrete [24,25]. Meanwhile, due to the small density of SF, it brings many drawbacks in the storage and transportation, thus, the SF production enterprises apply densifying technology to roll the original SF into small particles. So that the majority of SF used in our country has been densified (SF used in the paper is the densified SF). Therefore, dispersion degree of SF in composite plays a decisive role on physical mechanics and durability.

Although SF and other additives are used together to improve the properties of cementitious materials, researchers have not qualitatively and quantitatively studied the influence of pre-treated additive. Therefore, it has a nice significance on investigating methods for the pre-treated SF for qualitative and quantitative determination of absorption and dispersion process in cement pastes. In the paper, by using the titration test, the authors qualitatively and quantitatively studied the performance of adsorption and distribution of SF in cement pastes. Meanwhile the physical and chemical properties of SF modified cement-based composite materials were examined and the microstructures and absorption process of compound materials were analyzed. Finally, the simulated hydration process was established to provide the basis for evaluating the absorption performance between pre-treated SF and cement hydration products in composite materials.

2. Methods

2.1. Materials

This research used ordinary Portland cement whose properties are shown in Table 1, abiding by the Chinese standard (GB 175-2007) [26]. Properties of SF are shown in Table 2, meeting with the Chinese standard (GB/T 27690-2011) [27]. The polycarboxylate high performance water reducing agent (H-WRA) was used and its properties are shown in Table 3, complied with the Chinese standards (GB 8076-1997) [28] and (JG/T 223-2007) [29]. Reagents utilized in this study mainly include nitric acid, NaOH analysis reagent, silver nitrate solution, ferrovandium indicator, sulphur cyanogen potassium acid solution, dibutyl phthalate, sodium methyl silicate (SMS) and deionized water.

2.2. Specimen preparation

2.2.1. Pre-dispersed SF

As is shown in Table 4, a certain quantity of the deionized water (quantity is determined according to the water-to-binder ratio), SF (quantity is determined according to the amount of cement replacement), H-WRA (quantity is determined according to the percentage of cementitious material) and SMS (quantity is determined according to the percentage of cementitious material) were joined into a grinding jar as the preparation for the liquid of dispersed-mixtures. The pH value is adjusted to neutral by NaOH. The speed of the grinding machine is 80r/min and the ball milling time is 24 h. Eight experimental schemes with the same water-to-binder ratio and additional proportion of H-WRA and different dosage of DSF and SMS are illustrated in Table 4. The cement and the liquid of dispersed-mixture were later mixed for the DSF modified cement-based materials' preparation.

2.2.2. Samples for mechanical test and titration test

Standard specimens of $40 \times 40 \times 160$ mm cuboid binders of eight groups (each group contains three standard specimens) were made according to the proportion in Table 4 and then standard moist curing for 28 days. These eight groups were prepared for the test of mechanical properties.

In the same way, another eight groups were subsequently prepared for the titration test. The process of making samples for the titration test is as follows: After 28 days' standard moist curing in a curing box, the surface of the specimens was cleaned by deionized water, dried naturally, sanded and then sealed by paraffin wax. The face of the 40×40 mm cuboid specimen was kept unsealed to be the penetration surface for the subsequent penetration test. Completed specimens were soaked into 3.5 wt% NaCl solutions for 30 days. At the same time, another same eight groups treated in the same process were soaked into 3.5 wt% NaCl solutions for 60 days. After reaching the curing time and then being removed from the solution, specimens of the first eight groups (soaked 30 days) were sliced on the face of 40×160 mm cuboid from the direction of the penetration surface (the unsealed face) for 4 mm in width (the cutting face was in parallel with the penetration surface) until the length of 16 mm (the length obtained by experiment) to get the penetration samples of chloride ion which would be used in the later titration test.

Table 3
Properties of H-WRA.

Water reducing rate (%)	Solid content (%)	Sodium sulphate content (%)	Chloride ion content (%)
>30	30.5	1.1	0.13

Table 1
Properties of ordinary Portland cement.

Specific surface (m^2/kg)	Fineness (80 μm) (%)	Setting time (min)		Compressive strength (MPa)		Flexural strength (MPa)		Soundness
		Initial	Final	3 days	28 days	3 days	28 days	
330	7.8	125	320	20.5	45.1	4.1	7.2	Qualified

Table 2
Properties of SF.

Appearance	Bulk density (kg/m^3)	Specific surface (m^2/g)	pH value	Fe_2O_3 content (%)	SiO_2 content (%)
Gray	557	17	7	2.13	89

Note: SF is densified.

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