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Effect of the particle size of nanosilica on the compressive strength and the optimum replacement content of cement mortar containing nano-SiO₂

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HIGHLIGHTS

- We present effect of nanosilica particle size on compressive strength.
- We investigate effect of nanosilica particle size on optimum content.
- We found that particle size affected directly on the compressive strength.
- Microstructure results support compressive strength and optimum content results.

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ABSTRACT

This research presents the compressive strengths and the microstructure photographs of cement mortars containing nanosilica (NS) with various sizes of 12, 20 and 40 nm and then compared with cement mortar with silica fume (SF). Tested results indicated that NS significantly improved compressive strength of cement mortar and the strength improvement was also dependent on the NS particle size. Cement mortar containing NS 40 nm gave higher compressive strength compared with NS 12 and 20 nm due to their agglomeration and ineffective dispersion. By varying the replacement contents of 3%, 6%, 9% and 12% NS by weight of cement, the optimum replacement content was 9% for all NS particle sizes and SF. These results indicate that the particle size of nanosilica affected only the compressive strength of cement mortar, but it had no effect on the optimum replacement content. The results of microstructure photographs are also supported the compressive strength and optimum replacement content results. NS particles presented high pozzolanic activity and could fill up pores, resulting in homogeneous, dense and compact

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1. Introduction

In the past, silica fume (SF), or microsilica, appeared to be a good pozzolanic material because it presented a small particle size with high packing ability. Moreover, SF particles consist essentially of silica in non-crystalline form (amorphous) with a high specific surface, and thus exhibit great pozzolanic activity. SF has been applied to improve the properties of hardened cement paste in many applications, and it was found that great success could be achieved [1–5]. In recent years, new pozzolanic material containing nanosilica (NS) particles has been available on the market and has been utilized in many applications of concrete technology [6–19]. It is produced synthetically, in the form of a water emulsion of ultrafine amorphous colloidal silica (UFACS). Therefore, it appears to have the potential to replace SF because of its very high percentage

of amorphous silica (>99%) and the very small spherical particle size (1–50 nm) [6]. Also, the pozzolanic activity of NS is possibly higher than that of SF and other pozzolanic materials.

Existing reports in the literature have described the incorporation of small-sized particles of nano-SiO₂, nano-Fe₂O₃, nano-TiO₂, and carbon nanotubes into cement for high-strength concrete applications – especially NS particles [7]. In the works of Li et al. [8,9], nano-SiO₂, and nano-Fe₂O₃ with particle sizes of 10 nm were added to cement mortar; it was found that these nanoparticles could increase the compressive strength of cement mortar by about 1.26 times. Moreover, nanoparticles in concrete can be used for improving abrasion resistance and the performance of pavement under a cyclic load (fatigue properties) [10,11]. The effect of NS on cement mortar properties – e.g. flowability, setting time, and accelerated heat and hydration – were reported in the works of Senff et al. [12] and Mondal et al. [13]. These reports suggested that the addition of NS to cement mortar and concrete improved their short-term strength, long-term strength, and durability. Other







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interesting results were presented in the work of Jo et al. [14]. This study investigated cement mortar containing NS particle size of 40 nm; they reported that the strength of cement mortar developed significantly, about 2.7 times that of the control mortar and 2 times that of cement mortar with SF. This work also described that the NS particle behaved as a nucleus to tighten the bonding of hydrated cement. A stable gel structure could be formed which improved the mechanical properties of hardened cement paste. Chen and Ye [15] also reported on the strength improvement of cement mortar with NS particles compared with cement mortar containing SF. They also found that nanoparticles improved the compressive strength to a greater extent than SF. Tang et al. [16] found that the microstructure, as analyzed by X-ray diffraction (XRD) and SEM, as well as the mechanical properties of cement mortar were better improved by the addition of NS and SF compared with cement mortar without nanoparticles. Lin and Tsai [17] investigated the influence of nanomaterials on the microstructure of sludge ash cement paste. They found that the degree of crystallization in the hydrates was increased by adding quantities of nanoparticles to the cement mortar. Higher content of nanoparticles in the cement mortar also resulted in denser crystallization, smaller pore sizes, and a decreased number of pores. Shih et al. [18] found that NS could fill up the pores, and had the characteristics of pozzolanic activity. The microstructures of cement mortars mixed with nanoparticles were improved significantly, corresponding with their compressive strength and permeability [19].

As mentioned previously, the strength improvement of cement mortar containing NS with particle size of 40 nm was significantly greater than that of mortar containing NS with particle size of 10 nm. Therefore, nanosilica particle size has a possible influence on the properties of cement mortar. Several previous research reports have investigated the properties of cement mortar containing nanoparticles of a single size compared with cement mortar with SF. However, published articles focusing on the influence of nanoparticles of different sizes are rarely found. Therefore, the emphasis of this study is placed on the effect of nanoparticle size on the compressive strength of cement mortar and on the optimum replacement content. Nano-SiO₂ particles of various sizes were applied. The obtained results were compared with the control cement mortar without nanosilica and with cement mortar containing SF. The major concern was to study how the particle sizes of NS and SF influence the properties of cement mortar. Microstructure photographs analyzed by SEM [16-19] were also included to support the results of compressive strength tests.

2. Experimental program

2.1. Materials

Materials used in this investigation were: ordinary Portland cement type I (OPC); dry densified silica fume (SF) grade 920 D, with average particle size of 0.1 μ m (100 nm), containing 88.3% SiO₂; and nanosilica (NS) containing 99.9% SiO₂. Since the effect of nanosilica particle size was the focus of this study, three particle sizes of NS, with average diameters of 12 nm (AEROSIL 200), 20 nm (AERO-SIL 90) and 40 nm (AEROSIL 0X 50), were used. The chemical compositions of these materials are given in Table 1. Tap water was used in the mixtures together with superplasticizer (ADVA Cast 207) in order to achieve the desired flowability of cement mortar and better dispersion of nanoparticles. Standard natural river sand with specific gravity of 2.50 g/cm³ was used.

2.2. Mix proportions

Details of mix proportions of the cement mortars are given in Table 2. The first line is the mix proportion for the control mortar with OPC (without nanoparticles). The second group (lines 2–5) consists of cement mortars with cement partially replaced with SF 3%, 6%, 9% and 12% by weight of cement, respectively. The third group (lines 6–9) consists of cement mortars with cement partially replaced with NS 3%, 6%, 9% and 12% by weight of cement, respectively. Since the nanoparticle sizes are varied in this investigation, the third group of mixtures (lines 6–9) was used, but the NS particle size was changed from 12 nm to 20 nm and 40 nm, respectively.

tively. The water/binder ratio was fixed at 0.65 of all mixtures, while the sand/binder ratio of all mixtures was 2.75. The sand was graded in accordance with ASTM C778 [20]. For all mix proportions, a sufficient amount of superplasticizer was added to achieve the desired properties of bleeding, segregation, and flowability.

2.3. Test methods

Cement mortars containing SF and NS were prepared. The flow capacity was controlled by adjusting the quantities of superplasticizer. Mixing procedures were carried out in a rotary mixer according to the method presented in a previous work [14], as follows:

- 1. NS particles were mixed with water at high speed (285 rpm) for about 1 min.
- Cement and SF (if applicable) were added to the rotary mixer, and the mixer was allowed to run at medium speed (140 rpm) for another 30 s.
- 3. Sand was gradually added while the mixer was running at medium speed.
- 4. Superplasticizer was added and stirred at high speed for 30 s.
- 5. The mixture was allowed to rest for 90 s and then mixed continuously for 1 min at high speed.

After mixing, the cement mortar samples were tested for flowability based on ASTM C1437 [21]. For each mixture, specimens of $50 \times 50 \times 50$ mm were prepared for compressive strength testing in accordance with ASTM C109/C109M [22]. The test specimens were demolded after 24 h and then cured in water. Nine cubic test specimens were made from each mixture, covering three different ages of 1, 7 and 28 days. Their compressive strengths were tested using a hydraulic compression machine under a load control rate of 0.20 MPa/s. Some cement pastes were corrected from the crushed part of the compressive strength test specimen, and were then delivered to the laboratory for SEM investigation of the microstructure of cement mortars containing nanoparticles.

3. Results and discussion

3.1. Compressive strength

3.1.1. Effect of nanosilica particle size on compressive strength

The compressive strengths of cement mortar with OPC (without NS), mortar with SF, and mortar with NS at a mortar age of 7 days are shown in Table 3. It can be seen that the compressive strength of cement mortar with NS was higher than that of cement mortar with OPC, and gradually increased with increments of NS content. By using NS content of 6% and a water/binder ratio of 0.65, the compressive strengths of cement mortars with NS particle sizes of 12, 20 and 40 nm were increased, compared with cement mortar with OPC, by about 1.25, 1.52 and 1.65 times, respectively. However, better strength improvement was obtained with a NS replacement content of 9%, which resulted in strengths of 1.32, 1.67 and 1.74 times that of OPC, respectively. However, the strength improvement obtained in this study cannot be compared with the results presented in the works of Jo et al. [14] and Sadrmomtazi et al. [7], since the water/binder ratio and the mixture proportions used are different.

In the case of cement mortar containing SF, the compressive strength was slightly improved: by 1.03 and 1.06 times for replacement contents of 6% and 9%, respectively. The lower compressive strength improvement of cement mortars with SF compared with cement mortars with NS can be explained by the fact that SF has lower SiO₂ content, which produces less pozzolanic activity than that of NS. Moreover, SF particle size is larger than that of NS [10–12], therefore giving it less packing ability than that of NS. In other words, a very small particle of NS can more easily fill in the pores of cement paste, thus increasing the compressive strength of cement mortar compared with SF.

Fig. 1 presents the compressive strengths of cement mortar with different NS particle sizes in comparison with the control cement mortar without nanoparticles and cement mortar with SF, at a mortar age of 7 days. The effect of NS particle size on the compressive strength by varying the nanosilica content is also presented. For all NS contents, higher compressive strength was clearly obtained when larger-sized NS particles were added into the mixtures. There-

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