



# Combined effects of micro-silica and nano-silica on durability of mortar



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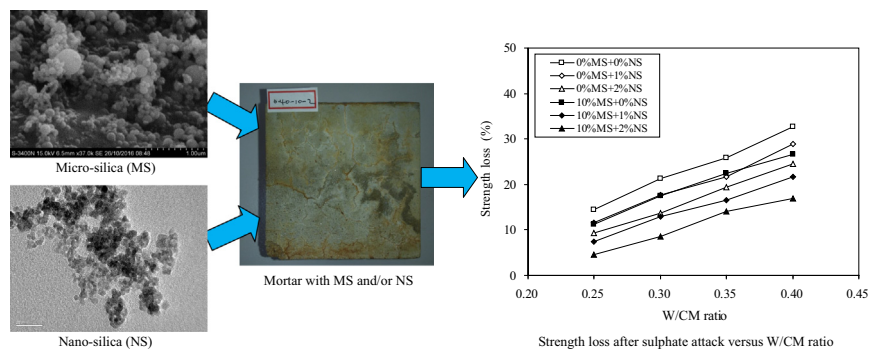
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## HIGHLIGHTS

- Addition of MS (micro-silica) or NS (nano-silica) enhances strength and durability.
- Combined addition of MS and NS offers synergistic effects in sulphate resistance and carbonation resistance.
- Combined addition of MS and NS offers additive effects in chloride resistance and water resistance.
- NS should be added together with MS for production of new generation high-performance concrete.

## GRAPHICAL ABSTRACT



Mortar with MS and/or NS was made for durability tests

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## ABSTRACT

To investigate the combined effects of micro-silica (MS) and nano-silica (NS) on the durability of concrete, an experimental program was launched, in which mortar mixes representing mortar portions of concrete with various water, MS and NS contents but a constant workability were made for strength, sulphate attack, carbonation, rapid chloride permeability and water absorption tests. It was found that the addition of MS and/or NS was effective in improving the durability of mortar. Moreover, for enhancing the strength, sulphate resistance and carbonation resistance, 1% NS was almost as good as 10% MS and the combined addition of MS and NS showed certain synergistic effects in the sense that the combined effects were larger than the respective sums of the individual effects. However, for enhancing the chloride resistance and water resistance, 1%–2% NS was less effective compared to 10% MS probably because the filling effect of 1%–2% NS was not as good as that of 10% MS. These results suggest that NS should not be added alone but should be added together with MS for best overall performance.

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## 1. Introduction

Micro-silica (MS), also called silica fume, is a by-product of the production of silicon or silicon alloys [1]. It contains high purity of silica (>80%) in amorphous form and generally has particle size ranging from 0.1 to 0.5  $\mu\text{m}$  and nitrogen BET surface of about 20,000  $\text{m}^2/\text{kg}$  [2,3]. Throughout the years, MS has become one of the most popular supplementary cementitious materials used in the production of high-strength concrete and high-performance concrete [4–8]. On the other hand, various types of nano-materials have been brought in by the recent advent of nanotechnology, which

is commonly defined as the control of matter based on molecule-by-molecule manipulation [9]. Some of these nano-materials may have good applications in civil engineering, especially concrete materials [10–13]. Among them, nano-silica (NS), which can be produced by the sol–gel process, vaporization method, biological method or precipitation method [14–17] and contains very high purity of silica (>90%) with ultra-high fineness [18], is one of the most attractive nano-materials for further advancement of concrete technology [19–24].

It is well known that MS is a good supplementary cementitious material for improving the durability of concrete structures. For instance, Mardani-Aghabaglou et al. [25] compared the effectiveness of fly ash, metakaolin and MS in enhancing the mechanical properties and durability performance of mortar and pointed out that among these supplementary cementitious materials, MS is the best. Gesoğlu et al. [26] made self-compacting concretes with binary, ternary and quaternary blends of cement, fly ash, slag and MS, and revealed that in terms of durability performance, the ternary blend of cement, slag and MS is the best combination. Farahani et al. [27] applied MS to produce marine concrete and have developed a mathematical model to predict the long-term chloride diffusion in concrete containing MS. Gupta et al. [28] explored the feasibility of using waste rubber fiber in concrete and found that the reductions in strength and water resistance due to the use of waste rubber fiber can be compensated by increasing the MS content.

In recent years, some studies on the effects of NS on concrete durability have been launched. Early in 2005, Ji [29] carried out a preliminary study on the water permeability and microstructure of concrete containing NS and found that addition of NS can improve the water penetration resistance and make the microstructure more uniform and compact. In 2012, Said et al. [30] investigated the effect of colloidal NS on concretes incorporating cement only or cement + fly ash, and revealed that the addition of NS can significantly improve the chloride resistance. In 2013, Beigi et al. [31] added NS and different fibers to self-consolidating concrete and showed that NS has great positive effects on reducing water absorption and chloride penetration of self-consolidating fiber-reinforced concrete. In 2016, Wu et al. [32] used NS in ultra-high strength concrete and pointed out that there exists an optimal NS content of about 1% for minimizing the porosity of concrete.

Meanwhile, some researchers investigated the effects of combined addition of MS and NS. For instance, in 2012, Jalal et al. [33] tried the addition of MS only, NS only and blended MS + NS, and found that the blending of 10% MS + 2% NS would lead to the highest strength and lowest water absorption, capillary absorption and chloride penetration. In 2014, Zapata-Ordúz et al. [34] made concrete containing cement, fly ash, MS and NS in binary, ternary and quaternary mixing for split tensile strength test, and applied Weibull distribution to analyze the tensile strength results. In 2016, Shaikh et al. [35] studied the effects of MS and NS on bond behaviour of steel and polypropylene fiber reinforced mortar with high volume of fly ash, and showed that the combined use of 10% MS + 2% NS would improve the pull-out strength to higher than those with only 10% MS added or only 2% NS added. In 2017, Li et al. [36] proved that the addition of both MS and NS has synergistic effects on strength and microstructure of mortar. Nevertheless, comprehensive research on the combined effects of MS and NS on the durability performance of various kinds of concrete is still lacking.

In the study presented herein, in order to investigate the combined effects of MS and NS on the durability of mortar or the mortar portion of concrete, a number of mortar mixes with various water, MS and NS contents were produced for strength, sulphate resistance, carbonation resistance, chloride resistance and water

resistance tests. For each mortar mix, the superplasticizer dosage was adjusted to achieve a constant workability. Based on the results so obtained, the effects of adding MS alone, NS alone, and MS and NS together on durability are evaluated, and the possible synergistic effects of combined addition of MS and NS are studied.

## 2. Materials and test methods

### 2.1. Materials

In this study, cement, MS and NS were used as the cementitious materials. The cement used was an ordinary Portland cement (OPC) of strength class 52.5N, which had been tested to comply with BS EN 197-1: 2011. The MS used was a condensed silica fume imported from Europe, which had been tested by the supplier to comply with ASTM C1240-15. The NS used was a powder form nano-silica manufactured in China with particle size ranging from 5 to 20 nm and purity higher than 99.6%. Scanning electron microscope image of the MS particles and transmission electron microscopy image of the NS particles are presented in Fig. 1(a) and (b), respectively, from which the particle sizes and shapes can be clearly seen. Regarding the fine aggregate, natural river sand with maximum size of 1.18 mm, moisture content of 0.21% and water absorption of 1.1% was used. The relative densities of the OPC, MS, NS and fine aggregate were measured as 3.11, 2.20, 1.94, and 2.58, respectively. A laser diffraction particle size analyzer was used to measure the particle size distributions of the OPC and MS, while the mechanical sieving method was used to measure the particle size distribution of the fine aggregate. The particle size distributions so obtained are plotted in Fig. 2.

On the other hand, the superplasticizer (SP) added to the mortar mixes was a polycarboxylate-based type with a solid mass content of 20% and a relative density of 1.03. It is a commonly used SP for production of high-performance concrete.

### 2.2. Mix proportions

To investigate the effects of MS and NS, an experimental program was launched, in which mortar mixes with different water to cementitious materials (W/CM) ratios, MS and NS contents (each expressed as a percentage by mass of total cementitious materials) and SP dosages (each in terms of liquid mass of SP expressed as a percentage by mass of total cementitious materials) were tested. The cementitious paste volume (solid volume of cementitious materials + liquid volume of water, expressed as a percentage of total volume of mortar) was fixed at 60%, while the W/CM ratio by mass was varied among 0.25, 0.30, 0.35, and 0.40, the MS content was varied between 0% and 10%, and the NS content was varied among 0%, 1%, and 2%. As there were four different W/CM ratios, two different MS contents and three different NS contents, a total of  $4 \times 2 \times 3 = 24$  mortar mixes were produced for testing.

Each mortar mix was assigned an identification code of X-Y-Z, in which X denotes the W/CM ratio, Y denotes the MS content (%) and Z denotes the NS content (%), as listed in the first columns of Tables 1 and 2. Unlike the other mix parameters, the SP dosage in each mortar mix was not fixed but determined by trial mixing, during which the SP was added to the mortar mix in small increments until a flow spread of at least 200 mm was achieved (such workability should be good enough for proper mixing and compaction of the concrete produced).

### 2.3. Mini slump flow test

The workability of each mortar mix was measured in terms of flow spread by the mini slump flow test for mortar, which may

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