Construction and Building Materials 125 (2016) 546–552

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Optimization of nano-silica's addition in cement mortars and assessment of the failure process using acoustic emission monitoring

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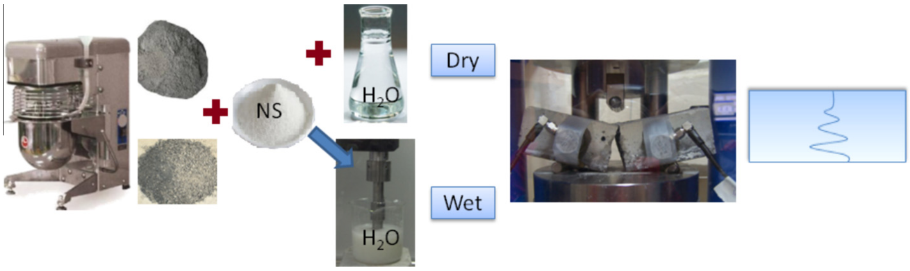
HIGHLIGHTS

- Nano-silica's addition results in enhanced mechanical properties and AE activity.
- "Wet" is more effective process and offers stable properties at limited mixing time.
- "Dry" mixing procedure can be used only in conjunction with superplasticizers.
- A more tortuous path of the crack is found after the addition of nano-silica.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 16 November 2015
Received in revised form 7 June 2016
Accepted 17 August 2016

Keywords:
Nano-silica
Mortar
Superplasticizer
Sonication
Acoustic emission

ABSTRACT

The objective of this work was to optimize nano-silica's addition in cement mortars using different mixing procedures. Nano-silica was either dispersed in water using sonication or directly added into cement and mixed using a rotary mixer. The fresh and hardened properties of nano-silica modified mortars were defined while acoustic emission (AE) activity was monitored and correlated with the failure processes. The addition of nano-silica resulted in 25–35% increase of the mechanical properties accompanied with simultaneous influence on the failure process as indicated by AE. Sonication was beneficial leading in stable mechanical properties at limited mixing duration.

1. Introduction

It is well established that cement-based materials offer high compressive strength in constructions however their whispered use is limited by their poor post-cracking durability. Therefore extensive work is being performed to improve the toughness and bending strength of these materials using an additional phase, such as fibres and fillers [1–5]. Nano-particles are now in the forefront of material research as filler materials to improve the mechanical properties and add functionalities to the cementitious mixes [6–8]. Among different nano-particles, nano-SiO_2 (nano-silica) has drawn considerable attention due its pore-filling effect, ability to accelerate the cement hydration and pozzolanic effect [9–12]. These mechanisms contribute to increased compressive and flexural strength, decreased porosity and improved durability of the nano-silica modified cement composites [11,13–16]. However, due to their very high specific surface area and surface energy nano-silica particles tend to agglomerate and aggregate, even in the well-dispersed colloidal suspensions [17], which limits their efficiency to act as fillers at the nano-scale. Therefore very recent studies focus on the assessment of the effect of agglomeration on microstructure and properties of fresh and hardened cement based materials [18–20]. The conclusion from these studies is that the

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http://dx.doi.org/10.1016/j.conbuildmat.2016.08.055

cement hydration, microstructure and property improvement after the addition of nano-silica is controlled by the final agglomerates no matter if nano-silica was in powder or colloidal state [19].

As highlighted achieving effective, uniform dispersion, especially at large quantities, is very critical and remains a challenge. The use of surfactants and/or functionalization of the nanoparticles provide solution towards dispersion issues in organic matrices [21], however, when used in cement based composites these additives interfere with the hydration reaction altering the hardening process [22]. For optimal dispersion nano-particles have been added in amounts up to 5 wt.% of cement [23–25], while mixing with flat beater [6,26], has been preferred so far due to its convenience. Another commonly used approach is using ultrasound dispersion in nano-particle's suspensions [25,27]. Apart from its reinforcing role and its participation in the hydration process, nano-silica is expected to alter the failure mechanism during fracture [6,23,25–27].

Acoustic emission (AE) is a sensitive technique for monitoring of processes like damage development in materials. Elastic waves are emitted when crack nucleation and propagation events occur within a material, recorded by piezoelectric sensors. AE allows monitoring very early signs of micro-cracking, while no signs of damage are visible, until the last moment of ultimate failure [28], which classifies AE as an appropriate technique for structural health monitoring in different fields [29,30]. Next to that, one can relate the dominant fracture modes, such as tensile cracking under bending test [31–33], pullout [34], compression [35] and splitting tests [36] with specific AE features. Important parameters of an AE waveform are the amplitude (A) and the duration (Dur), which is the delay between the first and last threshold crossings. In addition, rise time (RT) is the time between the onset of the waveform and the point of maximum amplitude. Another basic feature, which is sensitive to the cracking mode is RA which is calculated as RT over A, and is measured in μs/V. The frequency content can be either assessed simply in time domain by the ratio of threshold crossings over duration (average frequency, AF) or after FFT (central frequency CF in kHz). Several studies indicate that any shift from tensile to shear damage mechanisms is escorted by certain increase in RA and inversely a decrease in AF [37–39].

In the view of the above, the current research aims in investigating the effect of the nano-silica addition on the physical and mechanical properties of cement mortars in order to define appropriate mixing conditions for optimized properties. For this purpose nano-silica powder is either directly added to the cement powder or dispersed with ultrasounds in water. The use of superplasticizer has been also assessed in conjunction with the aforementioned procedures. Apart from the influence of nano-silica on the mechanical and physical properties, this study investigates its influence on the failure process using AE monitoring. Up to the authors' knowledge it is the first time that cementitious material with nano-modified matrix is monitored by acoustic emission, while preliminary results from specific specimens were published in a earlier short study [40].

2. Material and methods

2.1. Materials

The materials used to produce the mortars were tap water, cement, nano-silica and sand. The cement used was Portland cement II, crushed sand with a specific gravity of 2.75 kg/dm^3 was used as fine aggregate. The mortar matrix was modified using nano-silica particles (silicon dioxide nanopowder, 5–15 nm, 99.5% metal basis, molecular weight: 60.08 g/mol, Sigma Aldrich) at 0.5 wt% of cement. Some mixes contained Glenium SKY 645 superplasticizer at 0.15 wt% of cement (BASF Hellas).

2.2. Specimen preparation

For the specimen preparation two different procedures were followed and evaluated. In the first procedure, designated as "dry" hereafter, nano-silica particles were directly added into the cement powder and mixed using a rotary mixer with a flat beater. In the second procedure, designated as "wet" hereafter, nano-silica particles were first dispersed in water and sonicated to make a uniformly dispersed suspension. Sonication was performed using a Hielscher UP400S 24 kHz device (Hielscher Ultrasonics GmbH, Teltow, Germany), power capacity of 400 W, equipped with a Ø22 mm cylindrical sonotrode. Output power was regulated by means of manual adjustment of wave amplitude at 75% of the device's maximum capacity. The key variables used in the current study were: (a) "dry" or "wet" mixing procedure, (b) mixing duration, i.e. 10, 20 and 30 min and (c) use of superplasticizer in combination with the "dry" or "wet" procedure. For the preparation of the samples sand was further mixed with cement/water/nano-silica for 3 min with a rotary mixer. Superplasticier was added in the mixing stage in the "dry" procedure, while in the "wet" one it was introduced in the water/nano-silica suspension before sonication. After pouring the mixing product into oiled moulds a vibrating table was used to ensure good compaction. All specimens were demoulded 1 day after casting and cured in water saturated with calcium hydroxide at 23 ± 2 °C. The total curing time was 28 days.

2.3. Testing procedures

The air content and the workability of the fresh mortar were measured according to the BS EN 12350–7:2000 [41] and ASTM C230/C230M-03 [42], respectively. Workability results were obtained using the flow table method estimating the fresh mortar diameter.

Compression and 3 point bending tests were performed on the cured specimens (40 × 40 × 40 mm for compression and 40 × 40 × 160 mm for bending) using a testing machine specially designed for mortar specimens (MATEST) with a loadcell of 15 kN for the bending and of 250 kN, for the compression. Three to six specimens were tested for each mixing condition. All tests were performed following the BS EN 196–1:2005 [43]. Loading rate in compression was 2400 N/s, while bending tests were carried out at 50 N/s. The bending test was monitored by two AE sensors on each specimen aiming to record the cracking nucleation and development, as seen in the photograph of Fig. 1. The sensors had a resonance peak 150 kHz (type R15 of Mistras). Before being acquired in the board PCI-2 of Mistras with a sampling rate of 5 MHz, the

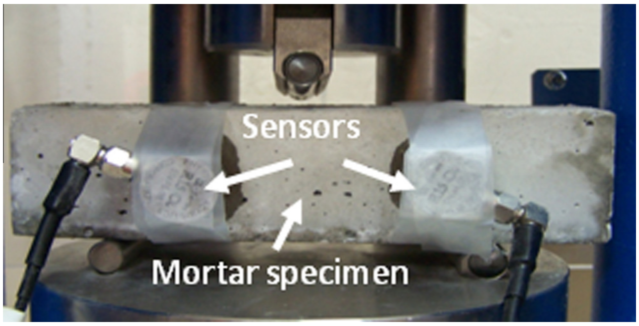

Fig. 1. Experimental set up of the bending tests showing the AE sensors attached on the mortar specimen.