



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



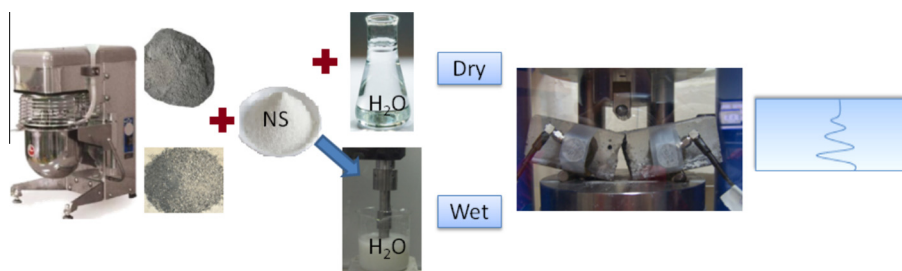
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## HIGHLIGHTS

- 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



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## 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.

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## 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<sub>2</sub> (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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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  $\mu\text{s}/\text{V}$ . 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  $2.75 \text{ kg}/\text{dm}^3$  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:  $60.08 \text{ g}/\text{mol}$ , 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  $\varnothing 22 \text{ mm}$  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  $23 \pm 2 \text{ }^\circ\text{C}$ . 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 ( $40 \times 40 \times 40 \text{ mm}$  for compression and  $40 \times 40 \times 160 \text{ mm}$  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

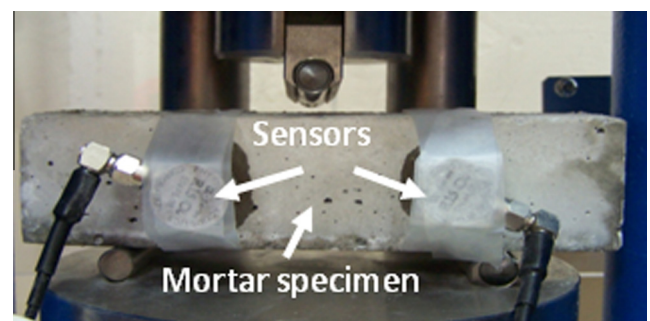


Fig. 1. Experimental set up of the bending tests showing the AE sensors attached on the mortar specimen.

signals were pre-amplified by 40 dB. The nominal number of specimens per type of mortar that were examined by AE was six.

The dynamic elastic modulus,  $E$  was calculated based on the ultrasonic technique applied on the 28 days cured beam specimens prepared for bending (40 8 40 × 160 mm) [44,45]. This methodology has been selected on the basis that it is possible to measure the dynamic modulus of elasticity using stress-wave propagation with good agreement between average values of static and dynamic modulus and lower test variability [46]. The ultrasonic experiments were conducted by the small and sensitive acoustic emission transducers (Pico, PAC), which exhibit a quite wide frequency band. A Tektronix AFG3102 function generator was used to produce an electric pulse of one cycle of 200 kHz, which was fed to the sensor acting as pulser. The received signal was pre-amplified by 40 dB and digitized with a sampling rate of 5 MHz in a PAC PCI-2 board. Noise level was low and wave speed was measured by the first detectable disturbance of the waveform. Considering the applied frequency and the wave speed of mortar (approximately 4000 m/s) the wavelength is calculated at 20 mm, which is considerably shorter than the specimen's thickness of 40 mm. Therefore, no interactions are expected due to other wave or vibrational modes. Measurements were conducted in ten paths through the thickness of the specimens (40 mm) and the results were averaged in order to account for possible in-homogeneity of the material. Though measurements during hydration were conducted, herein the longitudinal wave velocity,  $C_p$ , values of 28 days after mixing are taken into account for elastic modulus,  $E$ , calculation. This is done by Eq. (1):

$$C_p = \{[E(1 - \nu)] / [\rho(1 + \nu)(1 - 2\nu)]\}^{1/2} \quad (1)$$

where  $\rho$  is the density and  $\nu$  is the Poisson ratio of the material. The Poisson's ratio value employed in Eq. (1) was considered 0.2 which is typical for cementitious materials.

### 3. Results and discussion

#### 3.1. Properties of nano-silica modified fresh mortar

The effect of nano-silica's addition on the air content of fresh mortar is illustrated in Fig. 2 as a function of the mixing procedure, duration and use of superplasticizer. As shown, nano-silica modified specimens presented a considerable increase in their air content values, which was more pronounced in the "wet" mixing procedure. Mixing duration had an opposite effect in the "dry"

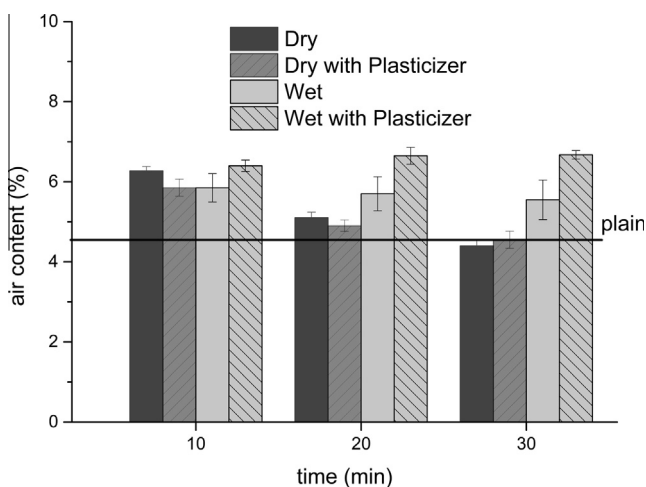


Fig. 2. Air content as a function of mixing time and procedure for plain and nano-silica modified specimens.

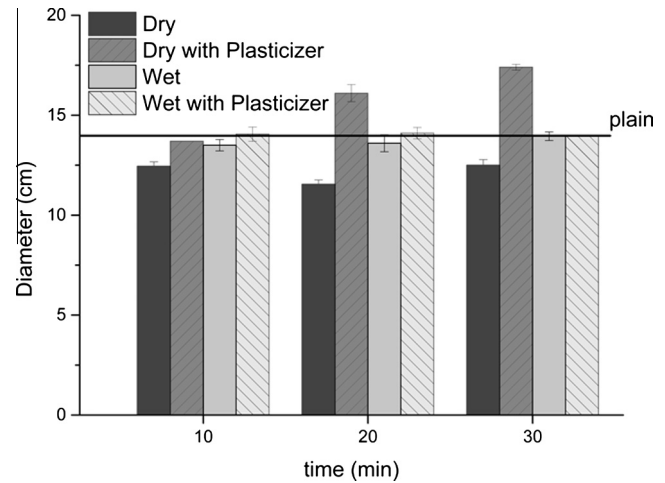


Fig. 3. Workability as a function of mixing time and procedure for plain and nano-silica modified specimens.

and "wet" procedures. As the mixing progressed the air content stabilized to values close to those of the plain mortar when the "dry" mixing procedure was applied. On the other hand in the case of "wet" mixing the increased values of the air content remained unaffected by the sonication duration in specimens without superplasticizer. The combined use of superplasticizer and sonication resulted in the highest air content with values as high as 6.7% after 30 min of sonication. Since nano-silica particles are in a powder form, the "dry" mixing procedure is very similar to the process followed in the plain mortar in terms of air entrapment. Higher mixing duration resulted apparently in more homogenous distribution of the nano-silica particles in the mix avoiding the development of agglomerates. On the other hand the sonication of nano-silica with water/superplasticizer increased the amount of air entrapped in the modified systems suggesting that superplasticizer functioned like a surfactant in the mix.

In Fig. 3 the workability results of the nano-silica modified specimens along with that of the plain mortar are presented. In the "dry" mixing procedure the addition of nano-silica without superplasticizer resulted in considerable reduction (up to 17%) of the workability. This is due to the high specific surface of nano-silica particles, giving an increase in water demand or requiring addition of superplasticizer to maintain certain flowability as confirmed by the results of the "dry" mix with superplasticizer. Mixing duration seems to play an important role in the "dry" procedure when combined with superplasticizer. As seen, higher mixing duration improved the workability results suggesting better distribution of the superplasticizer and improvement of the flowability of the modified mortars. Almost no variation was found in the workability values of specimens prepared via the "wet" method, independent to the application of superplasticizer with all specimens presenting values close to those of the plain specimen. This implies nano-silica where successfully wetted during sonication and did not absorb excess amount of water which is normally contributing to the flowability of the mortar.

#### 3.2. Properties of nano-silica modified hardened mortar

The variation of the modulus of elasticity,  $E$  as a function of mixing time and procedure for plain and nano-silica modified specimens is depicted in Fig. 4. A pronounced enhancement in the  $E$  modulus is being observed after the addition of nano-silica, with an increase up to app. 30% compared to plain mortar. As indicated in Fig. 4,  $E$  Modulus presented fluctuations with mixing time

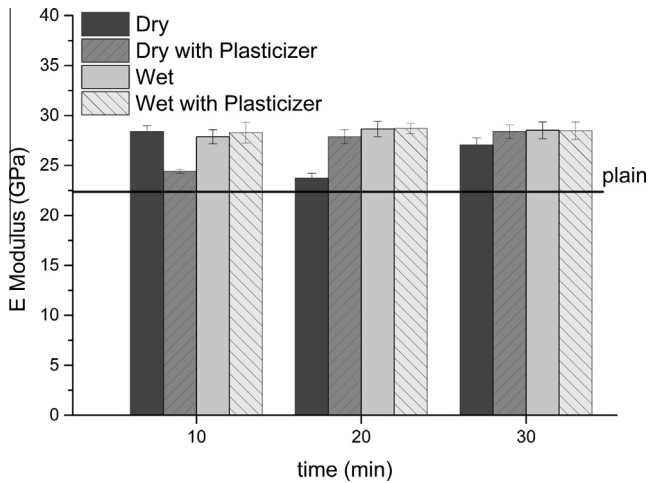


Fig. 4. Modulus of Elasticity (E) as a function of mixing time and procedure for plain and nano-silica modified specimens.

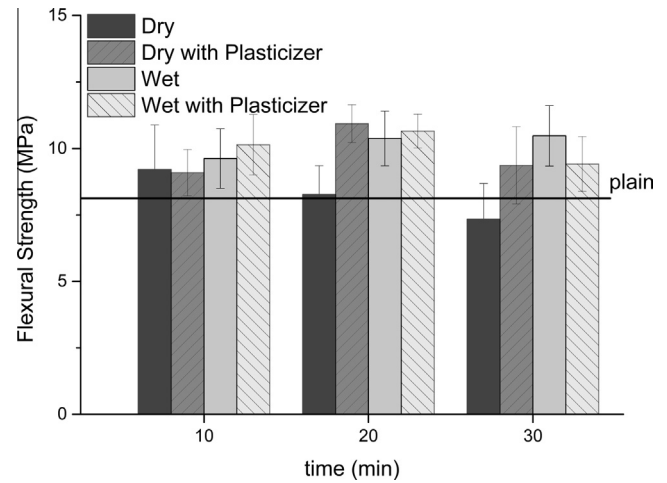


Fig. 6. Flexural strength as a function of mixing time and procedure for plain and nano-silica modified specimens.

in the “dry” mixing procedure and highest values were obtained after 30 min of mixing while more stable values over mixing time were obtained when the “wet” mixing procedure was applied. This suggests that the sonication resulted in better dispersion of the nano-silica particles.

The compressive properties after 28 days of curing of the plain and nano-silica modified specimens are presented in Fig. 5. As seen, there is a pronounced difference in the response of the specimens depending on the mixing procedure. The “dry” procedure resulted in app. 35% reduction of the compressive strength for the specimens without superplasticizer. Specimens with superplasticizer presented an initial reduction in the compressive strength followed by an increase as the mixing time developed. The maximum compressive strength after 20 and 30 min is in the range of 70 MPa (app. 30% increase compared to the plain mortar). Thus, it can be suggested that the use of superplasticizer was crucial for the dispersion of the nano-silica and the wetting of the cement matrix in the “dry” procedure. On the other hand mixing with ultrasound was sufficient and the use of superplasticizer did not result in any changes in the compressive properties. As in the case of Young’s modulus an app. 30% increase was observed in the compressive strength. This increase was obtained after 10 min of sonication and was stable thereafter. Although these specimens presented an increase in the air content values, the

elastic properties of the nano-silica in conjunction with the appropriate dispersion resulted in the observed enhancement. It is well known that compressive strength is a matrix dominated property. Thus, it can be suggested that as seen from the workability results, appropriate wetting of the nano-silica particles and cement matrix, either via sonication or via the introduction of superplasticizer was crucial for the resultant compressive properties. These results are in agreement with previous studies where well-dispersed nano-particles resulted in improved compressive properties [6,7,23,27,47,48].

The effect of the nano-silica addition on the flexural properties of the nano-modified specimens is depicted in Fig. 6. When using the “dry” mixing procedure specimens without superplasticizer presented a variation in their flexural properties, with values below/above those of the plain specimen, dependant on the mixing time. The presence of superplasticizer was beneficial for the flexural strength, although some fluctuations as a function of the mixing time were also present. On the other hand the use of sonication resulted in app. 25% increase in the flexural strength. Mixing duration played an important role since 30 min of mixing led to slight reduction of the flexural strength of all tested specimens apart from those prepared following the wet process without superplasticizer. It can be suggested that rigorous mixing either with a flat beater or with sonication resulted in finer nano-silica particle sizes, reducing its reinforcing efficiency. In the dry mixing process, together with the nano-silica particles, cement was also intensively mixed, which may have altered its grain size affecting the resultant flexural properties.

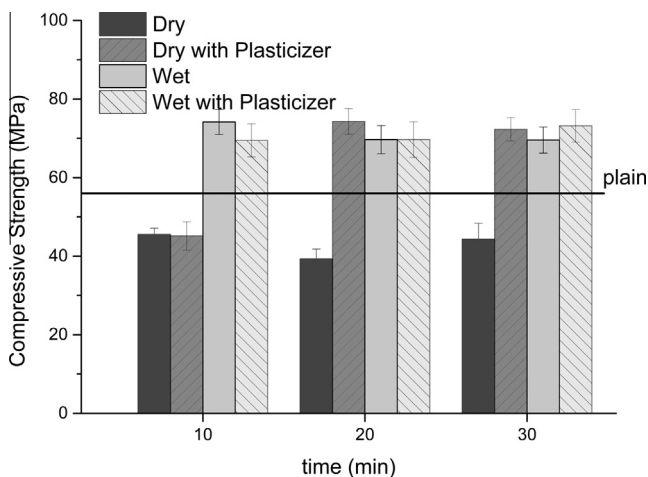


Fig. 5. Compressive strength as a function of mixing time and procedure for plain and nano-silica modified specimens.

### 3.3. Acoustic emission

Nano-modified material exhibited in general higher number of accumulated AE hits as can be seen in the indicative cases of Fig. 7. The specimens presented in this figure were prepared using the “wet” procedure with superplasticizer. The higher number of AE activity reveals a higher number of fracturing events within the material, suggesting also a change in the tortuosity of the crack path. The activity of mortar with nano-silica was more than three times higher than the plain mortar in average values. This does not necessarily imply differences in the total fracture energy; only that the energy is distributed to many incidents of lower level. The pattern of the AE curve shows that after an initial phase of micro-crack nucleation and development (around 5–7 s in Fig. 7) there is a small period of silence for another 5 s during which no energy is



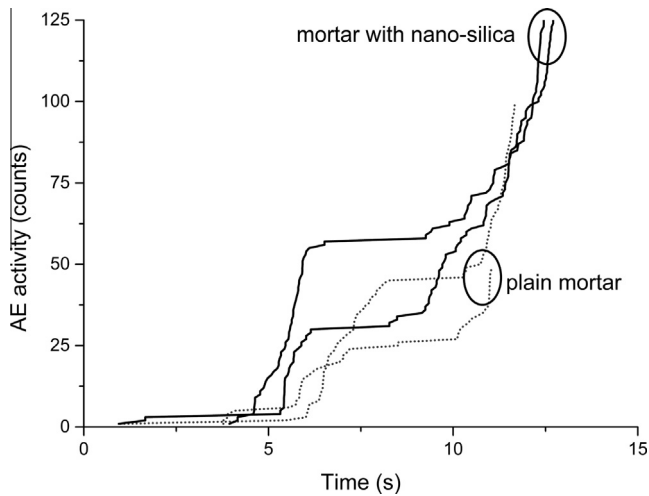


Fig. 7. Effect of nano-silica addition on the cumulative AE activity of mortar specimens prepared using the “wet” procedure with superplasticizer.

emitted while the material is elastically deformed. However, at a certain point these micro-cracks lead to coalescence and the rate of AE is again increased until the macroscopic fracture of the specimens. It is mentioned that the specimens failed catastrophically right after reaching their maximum load. Most of the specimens were split in two parts and the test was terminated as there was no “softening branch” to follow.

The trend is similar for the cumulative energy (“MARSE”, Measured Area under the Rectified Signal Envelope) shown in Fig. 8. The interesting point of the energy behaviour comes after the plateau of silence; plain specimens fracture with a sudden increase of AE energy while for the nano-modified ones there is a gradual build up of energy (approximately 10–13 s) leading to the final burst of AE energy and macroscopic fracture. However, the total AE energy recorded does not systematically differ. Although the AE energy cannot be regarded as an absolute measure of the fracture energy, considering that AE is anyway a part of the fracture energy and that the experimental conditions were identical in all tests, it can be implied by the present tests that the fracture energy is not significantly altered—positively or negatively—by the addition of nano-silica. For specific characterization of the fracture energy, dedicated experiments should be conducted. As aforementioned, the clearer trend is that the energy is divided to more fracture incidents.

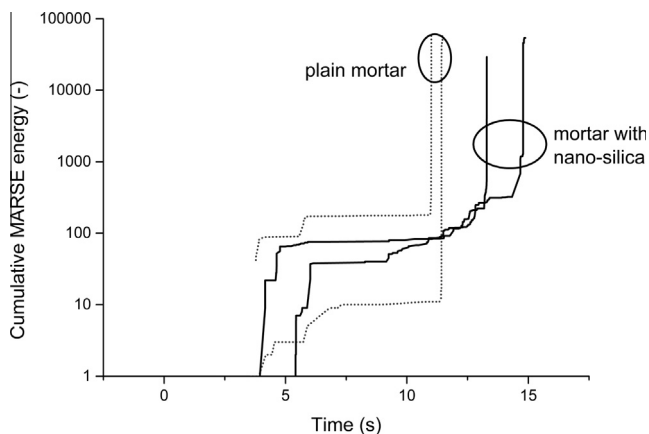


Fig. 8. Effect of nano-silica addition on the cumulative MARSE energy of mortar specimens prepared using the “wet” procedure with superplasticizer.

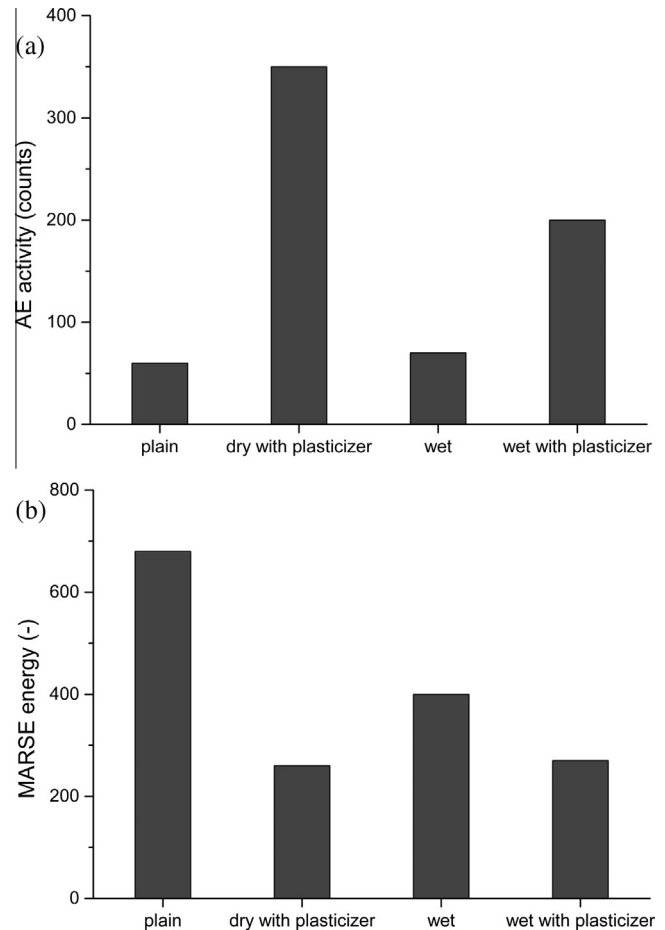
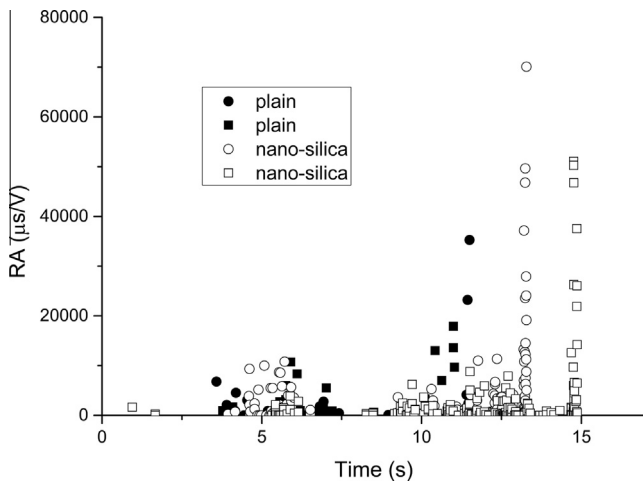


Fig. 9. (a) AE activity (counts) and (b) MARSE energy as a function of mixing procedure for plain and nano-silica modified specimens.

Concerning the average AE activity from all specimens, results are shown in Fig. 9 where plain mortar exhibits the lowest number of acoustic emissions and hence the smaller number of fracturing events (see Fig. 9a). All mixes with nano-silica exhibited higher number, especially those with superplasticizer. Correspondingly, the typical signal from mortar with nano-silica exhibited much lower energy since the fracture energy is divided to a large number of events (see Fig. 9b). The results imply that chemical admixtures help to distribute the energy to higher number of events possibly due to better dispersion of nano-silica. It is noted that “dry” mix without superplasticizer was not tested by the same set of AE sensors and hence is not included in the same graphs for reasons of consistency.

Apart from the total AE activity and the emitted energy, there are other waveform parameters that have been used for the characterization of the fracture process and specifically the mode. As aforementioned the parameter RA which measures the inverse of the initial slope of the waveform has been recently successfully employed in several fracture studies where different modes co-exist [35,37]. Fig. 10 presents the behaviour of RA values for the specimens, the energy of which was presented in Fig. 8. Each point is the RA value of one recorded AE waveform. In all cases low RA values were exhibited during the initial stages of loading, while they strongly increased as the material approached final failure. However, the AE signals of the plain mortar specimens were constrained up to 30 ms/V even for the last events at the moment of final failure and only very few signals exhibited higher values. On the other hand, nano-modified specimens exhibit higher RA values (close to 70 ms/V) while the population of signals with high



**Fig. 10.** Effect of nano-silica addition on the RA value of mortar specimens prepared using the “wet” procedure with superplasticizer.

RA is much larger than the plain specimens. In similar studies the AF (average frequency) of the signals is also examined [33,38]. However, in the specific case the change in AF was much smaller and therefore, results are not presented. Unlike RA, the AF has decreasing trends during the fracture. Therefore, in the cases of mortar of this study, the trends are limited by the zero point, unlike RA the maximum of which can indicate differences between mixtures.

Even though the test setup and specimen geometry is kept constant, the microstructure of the material may well impose variations in the stress field compared to a perfectly homogeneous material. It seems that the addition and good dispersion of nano-silica influences the microstructure and the way the crack path is developed. These preliminary trends concerning AE activity of the nano-modified mortars should be thoroughly investigated, since they give the possibility to characterize the contribution of nano-phase in the fracture process.

#### 4. Conclusions

Physical and mechanical (compressive and flexural strength) properties of nano-silica modified mortars were investigated as a function of the mixing procedure and duration. AE monitoring was simultaneously performed in order to correlate the AE activity with the failure processes of the nano-silica modified specimens. It can be concluded that:

- Nano-silica's addition resulted in enhanced mechanical properties, i.e. Young's modulus, compressive and bending strengths.
- The use of sonication was beneficial for the uniform dispersion of the nano-silica particle, since it resulted in stable properties at limited mixing time.
- The “dry” mixing procedure can be used only in conjunction with superplasticizers. There is a need of higher mixing times (20–30 min) to obtain enhanced properties.
- Higher AE activity was recorded for nano-modified material compared to plain mortar. This corresponded to a larger number of fracturing incidents which distributed the total energy in more and smaller events without significant changes in the total received energy.
- Qualitative AE features like the RA value implied that the fracturing of the modified matrix exhibited different fracture characteristics at the moment of failure from plain mortar, something attributed to a more tortuous path of the crack.

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