



# The effect of nano-MgO on the setting time, autogenous shrinkage, microstructure and mechanical properties of high performance cement paste and mortar



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## HIGHLIGHTS

- The nano-MgO improves the properties of the cementitious materials over time.
- The nano-MgO increases the hydration heat and compressive strength of mortar.
- The autogenous shrinkage decreases with the increasing of nano-MgO content.
- The nano-MgO developments the microstructure of the mortar.

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## ABSTRACT

Many studies have targeted the application of micro-size MgO to reduce autogenous shrinkage in cement based materials. However, there is little knowledge the effect of nano-MgO on the mechanical, durability and microstructure properties of high-performance cement-based materials. In this paper, the properties of cement pastes and mortars with nano-MgO were experimentally investigated. 2.5%, 5.0% and 7.5% nano-MgO by weight of binder were used in mortar and paste mixtures. While the setting time and the volumetric autogenous shrinkage tests were carried out in paste mixtures, the heat of hydration, 7 and 28 days compressive strength, ultrasonic pulse velocity (UPV) and 28 days linear autogenous shrinkage test were carried out in mortar mixtures. In addition, Scanning Electron Microscopic (SEM) observations were also made to evaluate the effect of nano-MgO on microstructure. It was concluded that nano-MgO had a significant effect on the fresh and hardened properties of cement pastes and mortars. Generally, nano-MgO increased the compressive strength (about up to 8%). Nano-MgO decreased autogenous shrinkage and setting time decreased with an increased in use of nano-MgO. The final hydration heat of the control mixtures and the nano-MgO mixtures are almost the same. Nano-MgO can be used as an expanding additive.

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## 1. Introduction

The use of nanomaterials in cement composites as the most used building material in the world has been investigated over the past few years. The physical and mechanic behavior of cement composites depends on the properties of components and physical and chemical changes that occur at the micro and nano level. CSH gel formed as a result of reactions of cement with water is a natural nanostructure material. Accordingly, nanotechnology can modify the molecular structure improving the concrete's bulk properties

and allows the development of inexpensive, high-strength and durable products of cement-based materials [1,2]. In general, it is now known that nanomaterials improve the fundamental properties of cement composites such as strength, durability, physical, microstructure [3–7].

Nanoparticles, particles of less than 100 nm in diameter, generally shows the properties of electrical, physical, mechanic or chemical that are entirely dissimilar from those showed by the same products with larger dimensions. Nanomaterials with different chemical properties have been used in the production of cement paste and mortar and improved the mechanical, physical, chemical and durability properties of cement composites. The inclusion of nanoparticles into the cementitious materials may be

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as spherical materials (e.g., nano-SiO<sub>2</sub>, nano-Cu<sub>2</sub>O<sub>3</sub>, nano-TiO<sub>2</sub>, nano-Al<sub>2</sub>O<sub>3</sub>, nano-CaCO<sub>3</sub>, nano-Fe<sub>2</sub>O<sub>3</sub>, nano-CuO, nano-Fe<sub>3</sub>O<sub>4</sub>, nano-ZnO<sub>2</sub>, nano-ZrO<sub>2</sub>, nano-ZnO etc.) or as nanotubes or fibers (e.g., carbon nanotubes and carbon nanofibers, respectively) or as nano-clay. In the relevant literature, many researchers have shown that even at small dosages, nano-SiO<sub>2</sub> can develop the mechanical properties of cement composites [2,8–10] and the microstructure becomes more compact [2,11,12]. Morsy et al. [1] showed that the use of single and combination of nano metakaolin and carbon nanotubes increases the compressive strength compared to control mix. Fan et al. [13] stated that a suitable calcined nano-kaolinite clay addition amends the strength of cement based mortar exposed to acid. In the separate study, Farzadnia et al. [14] showed that incorporation of nano-clay in mortars improved the compressive strength by up to 24% in samples with 3% nano-clay. Nano-clay particles better the properties of cement composites showing a pozzolanic reaction with cement and micro-filling effect [15,16]. Noorvand et al. [17] and Nazari and Riahi [18] found that there have been a few studies regarding the functionality of nano-TiO<sub>2</sub> on hardened cement composites. Generally, one of the broadly-studied functions of nanoparticles is their nucleation effect in the cement paste [19]. Farzadnia et al. [20] and Farzadnia et al. [21] showed that nano-titania and nano-alumina used in mortar mixtures increased mechanic properties and energy absorption of mortars. Nano-CaCO<sub>3</sub> particles create a suitable environment for nucleation of hydration products and eventually improve hydration reactions and besides treat as nano-fillers thereby developing the mechanical properties of cement composites [22–24]. Aside from these, Nazari and Riahi [25] and Nazari and Riahi [26] experimentally showed that nano-ZnO<sub>2</sub> and nano-TiO<sub>2</sub> powders improved the mechanical and physical properties of the concrete [27]. There are much more studies on nanomaterials [28,29].

On the other hand, the application of high-performance cement composites is continually augmenting due to its superior durability, mechanical, physical and other properties according to normal cement based composites [30]. However, high-performance cement composites produced with tremendously low w/c ratios is vulnerable autogenous shrinkage and even failure (cracks) [31]. These cracks deteriorate the concrete properties and a lot of research has been done to reduce them [32]. These are internal curing with lightweight aggregates [31,33–37], fibers [38–42], recycle aggregate [43] and expansive reactions [44]. In their studies, the autogenous shrinkage reduced, but the strengths also simultaneously reduction. Cracks in high-performance cement composites occur the formation of nano-micro scales and have a high impact on the durability of high-performance cement composites and these could be controlled by incorporating nanoscale materials [45].

MgO-based expansive additives for the shrinkage reduction was discovered and used for years. MgO reacts with water, and the hydration reaction produces Mg(OH)<sub>2</sub>. Because of the difference in density between MgO (3.58 g/cm<sup>3</sup>) and Mg(OH)<sub>2</sub> (2.36 g/cm<sup>3</sup>), when MgO converts to Mg(OH)<sub>2</sub>, the volume more than doubles. Many studies have been investigated the hydration and expansion of light and dead-burnt MgO [46–52]. As a result, micron-sized MgO generally decreases the autogenous shrinkage but, on the other hand, also decreases the strengths. The particle diameter and calcining temperature influenced the degree of hydration of the MgO expansive agent. Low calcining temperature and smaller particle diameter increased the rate of hydration of MgO expansive agent. The expansion of cement paste decreased with an increase in the calcining temperature of MgO expansive agent. The expansion time was prolonged and it occurred in a later period when the holding time was prolonged. The best calcining system was treatment at 1000 °C for 1 h with the particle diameter of

45–150 μm [53]. Therefore, both the mechanical and durability properties of nano-MgO high-performance cement composites should be carefully examined.

The properties of mortars containing nano-MgO were experimentally studied by Moradpour et al. [54], Polat et al. [55], Ye et al. [56], Kahidan [57] and Hou et al. [58]. Moradpour et al. [54] stated that the strengths increased in mixtures containing nano-MgO particle, nano-MgO behaved as a filler to enhance cement composites internal structure. Ye et al. [56] expressed that nano-MgO used in cementitious materials does not damage the mechanical properties and increases the strength in later age at water curing and that nano-MgO may use as the expansive agent for cement materials. Polat et al. [55] experimentally showed that reduction in autogenous shrinkage due to nano-MgO was higher than that of MgO for same mixture ratios. Kahidan [57] stated the hydration rate of nano-MgO cured as standard was low. Gao et al. [59] investigated the effect of nano-MgO for clay and used different nano-MgO contents (0%, 1%, 2%, 3%, 4%, 5%, 6%) in their study and stated that nano-MgO improved the strength and stability of the clay increasing the cementation and pore filling. Yuan et al. [60] investigated the effect of using nano-MgO in the properties of thermal energy storage materials produced with aluminate cement. The pressure strength of the samples produced with 1% nano-MgO was found to be 30.5% higher than the samples produced with pure cement. Hou et al. [58] investigated the effects of the hydration reactivity of ultrafine MgO on cementitious materials and stated that the initial and final setting times are shortened with using ultrafine MgO, the compressive strengths of samples with ultrafine MgO decreased at the later ages while the compressive strength of samples with ultrafine MgO increased at early ages and the effects of MgO on cement hydration varied considerably with water binder ratio.

As seen in the literature, Moradpour et al. [54] and Ye et al. [56] studied the strength of mortars which water/binder ratio is 0.50 and the strengths also increased for with small amounts of nano-MgO. But, MgO based expansive additives are used for high strength (performance) mortar/concretes that occurred the micro-nano cracks and produced with low water/binder ratio (below than 0.42). Mo et al. [50] expressed that MgO based additives may be more suitable for concrete which has low permeability and insufficiency the external water curing. Thus, it is important the examination of the effect of nano-MgO in cement composites with low water/binder ratio. It is also necessary to investigate the effect of nano-MgO on the properties of the cementitious materials other than the strength and expansion properties.

This research aimed to investigate the performance of nano-MgO on the properties of high-performance cement composites in terms of microstructure, durability and mechanical properties. For this purpose, the effect of nano-MgO as an additive on the durability and mechanical properties of high strength mortars and pastes was investigated. 2.5%, 5.0% and 7.5% nano-MgO as cement replacement were added to high strength mortars and pastes. Compressive strength, linear autogenous shrinkage, UPV, SEM for mortar mixtures and the setting time and volumetric autogenous shrinkage for paste mixtures were also obtained.

## 2. Experimental work

### 2.1. Materials

Ordinary Portland cement (PC) (type 1 – 42.5 MPa) in compliance with ASTM C150/C150M [61], 7% silica fume (SF) a replacement with cement, and natural river sand that has the maximum particle size of 4 mm according to ASTM C33/C33M [62] were used. Table 1 shows the properties of Portland cement and silica fume. Water to binder ratio of 0.30 and 0.5% for paste mixtures and 2% for mortar mixtures naphthalene sulfonate base superplasticizer (SP) were applied. The

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