**Research Paper** 

# Effect of Nano-MgO Additive on Compressive Strength of Concrete Fabricated by Different Processing Methods

#### Eisa Mahmoudsaleh<sup>1</sup>, Ali Heidari<sup>1&2\*</sup>, Farshid Fathi<sup>1</sup>, Seyed Ali Hassanzadeh-Tabrizi<sup>3</sup>

1. Department of Civil Engineering, Najafabad Branch, Islamic Azad University, Najafabad, Iran.

2. Department of Civil Engineering, University of Shahrekord, Shahrekord, Iran.

3. Advanced Materials Research Center, Department of Materials Engineering, Najafabad Branch, Islamic Azad University, Najafabad, Iran.

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# ABSTRACT

The effects of adding different nano-MgO dosages (0, 1, 2, 3, and 4) wt.% with respect to cement) on microstructural, compressive strength, and phase evaluation of concrete were investigated. Two different post-treatment conditions with water and CO<sub>2</sub> gas were used to study the processing method on the samples. The specimens were characterized via SEM and XRD analysis. The mechanical properties of the samples were also investigated. The results showed that compressive strength significantly improved after the addition of magnesium oxide nanoparticles. However, this improvement was more remarkable in the case of post-treatment with CO<sub>2</sub> compared to the samples fabricated with water. SEM results showed that the samples treated under CO<sub>2</sub> gas had irregular and needle-like morphology. The samples prepared by normal processing had CaCO<sub>3</sub> and SiO<sub>2</sub> phases, whereas the ones fabricated under CO<sub>2</sub> gas contained CaCO<sub>3</sub>, SiO<sub>2</sub>, and Ca(OH)<sub>2</sub>. With the addition of nano-MgO, the density of concrete decreases in the samples post-treated with water, whereas it increases for the samples post-treatment with CO<sub>2</sub> gas. Adding 4 wt.% nano-MgO to concrete and further posttreatment with  $CO_2$  for 45 days could increase the mechanical properties from  $\sim 23$  MPa to  $\sim 55$  MPa.

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\* Corresponding Author

E-mail Address: aliheydarisku6@gmail.com

# 1. Introduction

Nano dimension materials like nanoparticles, nanobelts, nanosheets, etc., have attracted much attention in the last 100 years [1]. In this dimension, the mechanical and physical properties of materials could be changed. This phenomenon has been extensively used in recent years in many industries like textile, chemical, plastic, cosmetics, and so on [2–4]. One of the essential subjects is to use of nanomaterials for improving the raw materials in the construction industry [5,6].

Portland cement may be a widely used building material in the world. Because of chemical, drying, and autogenous shrinkage caused by temperature stress, this material cracks quickly [7]. These cracks compromise building safety, diminish service life, and result in significant economic and societal losses. In addition, the formed cracks allow acidic chemicals to enter the concrete, causing carbonation, corrosion, and unwanted reactions. It could also destroy the steel in the reinforced concrete [8]. These events reduce the strengths of the structure [9]. Nanomaterials could be added to the concrete to improve the mechanical properties and inhibit these drawbacks. For example, some researchers added carbon nanotube [10,11] to concrete and showed that the nanomaterials ameliorate the properties of the structure. Promising results have also been reported with the addition of graphene [12–14]. Magnesium oxide (MgO) is a fascinating basic oxide ceramic with numerous applications [15]. This oxide ceramic can be used as an absorbent of toxic chemicals, refractories, catalyst support, optic instrument, etc. [16–20]. Volume expansion happens in this material when it reacts with water due to the formation of Mg(OH)<sub>2</sub>. By adding MgO to concrete, this hydration and expansion could compensate for the contraction of concrete and may reduce the chance of crack formation [21,22].

This study aimed to add nano-MgO to concrete processed by two different methods (post-treated with water and carbon dioxide) to investigate its effects on the mechanical properties, phase evaluation, and microstructure. Although the MgO addition to concrete has been studied before, in the present work, the effect of nano-MgO was investigated based on the post-treatment processing of concrete which is a new approach.

Table 1. Chemical composition of Fortuald cement												
Chemical Composition	CaO	SiO <sub>2</sub>	$Al_2O_3$	Fe <sub>2</sub> O <sub>3</sub>	MgO	SO <sub>3</sub>	K <sub>2</sub> O	Na <sub>2</sub> O	L.O.I			
Weight percent	53.27	21.89	5.3	3.34	6.45	3.67	0.98	0.18	3.21			

Table 1. Chemical composition of Portland cement

Curing Condition	Samula Cada	Composition weight (g)							
	Sample Code	Cement	Stone Powder	Grit	Sand	Water	Nano-MgO		
Water	0% MgO-N	333	167	625	708	120	0		
	1% MgO-N	314.7	167	625	708	120	18.3		
	2% MgO-N	296.4	167	625	708	120	36.6		
	3% MgO-N	287	167	625	708	120	55		
	4% MgO-N	260	167	625	708	120	73.32		
CO <sub>2</sub>	0% MgO-C	333	167	625	708	120	0		
	1% MgO-C	314.7	167	625	708	120	18.3		
	2% MgO-C	296.4	167	625	708	120	36.6		
	3% MgO-C	287	167	625	708	120	55		
	4% MgO-C	260	167	625	708	120	73.32		

 Table 2. The composition, code name, and applied curing condition of the samples

# 2. Experimental procedure

### 2.1. Materials and Methods

In the present study, Portland cement, Stone Powder, Grit, Sand, and water with the chemical composition shown in Table 1 were used. Nano-MgO was purchased from US-nano. Based on the compositions shown in Table 2, the raw materials were mixed. The MgO-mixed cement was made by adding different amounts of nano magnesium oxide to the Portland cement and other compounds mixture. The raw materials were mixed based on the ASTM C 305 [23]. The method includes the dispersion of nano-MgO in water by ultrasonic for 15 min in order to deagglomerate MgO nanoparticles. All the samples were produced with a water-to-cement ratio of 0.45 and produced in a laboratory mixer. For the construction of blocks, H2O was added to the mixed samples for 3 min under stirring. The fresh pastes were cast in steel molds. The samples were covered with a polyethylene sheet to prevent water evaporation. After 24 h, the samples were demolded without slump. Then, the samples were subjected to CO<sub>2</sub> gas or immersed in water for 7, 30, and 45 days to investigate the effect of curing on the samples. The purity of CO<sub>2</sub> gas was 98%. Four samples were produced for each mixture and an average value of measurements with error bars was reported. The name of the samples was shown in Table 2.

#### 2.2. Characterizations

The phase evaluation of the samples was analyzed by X-ray diffraction (Philips PW3040 diffractometer) with copper radiation. SEM images were taken with a VEGA/TESCAN Mira 3-XMU microscope. The compressive strength of the fabricated specimens was measured by uniaxial loading of 55 kN/min using a Toni Technik Baustoffprüfsysteme machine. The density ( $\rho$ ) of samples was measured according to their size and weights based on equation 1.

 $\rho = \frac{Mass of sample after post-tratment}{Volume of sample after post-tratment} \quad (Eq. 1)$ 

# 3. Results and discussion

Fig. 1 shows the XRD result and SEM image of the nano-MgO used for addition to the concrete. According to the XRD pattern of the sample (Fig. 1a), all of the peaks are related to the conventional cubic MgO reflections (JCPDS 45-0946). The (111), (200), (220), (311), and (222) MgO crystal planes correspond to the diffraction peaks at 36.6°, 42.5°, 61.8°, 74.0°, and 78.3°. SEM micrograph (Fig. 1b) shows that the powders have homogeneous spherical particles with an average particle size of about 30 nm. The nanopowders contain some aggregates and agglomerates. The high surface energy of the nanopowders causes the production of these agglomerates. As a result, nanoparticles tend to clump together in order to reduce their surface energy [24-27].



Fig. 1. (a) XRD pattern and (b) SEM image of the MgO.

Fig. 2 displays the XRD patterns of samples with different amounts of MgO post-treated with water and  $CO_2$  gas for 45 days. As can be seen, in the sample post-treated with water,  $CaCO_3$ ,  $SiO_2$ , and  $Ca(OH)_2$  phases are observed. The formation of calcium hydroxide is due to a reaction between calcium oxide and water during post-treatment according to equation 2 [28].

 $CaO + H_2O \rightarrow Ca(OH)_2$  $\Delta H=-104.2 \text{ kJ mol}^{-1}$  (Eq. 2)

Due to variations in the specific volumes of  $Ca(OH)_2$ (0.45 cm<sup>3</sup>/g) and CaO (0.29 cm<sup>3</sup>/g) [29], the sample expands during the reaction. The nano-MgO may also react with water and form magnesium hydroxide, which accompanies expansion. Due to the intrinsic contraction of concrete during reaction and drying, cracks form, which is harmful and reduces the mechanical properties. The expansion of formed hydrated phases after post-treatment compensates for the contraction of the concrete and reduces the chance of crack creation. The peaks related to magnesium oxide could not be detected in the samples, which may be due to the low amount of MgO in the samples. The samples with  $CO_2$  posttreatment show  $CaCO_3$  and  $SiO_2$  components. Processing with carbon dioxide prevented the formation of hydrated phases. In this condition, the carbonate phases of calcium and magnesium may be formed.



Fig. 2. XRD patterns of the fabricated samples with different nano MgO post-treated with (a) water and (b) CO<sub>2</sub>.

The density of the samples with different amounts of MgO post-treated with water and CO<sub>2</sub> is shown in Fig. 3. As can be seen, nano-MgO has a different effect on the density of the samples. In the samples post-treated with water, by increasing MgO content, the density of concrete decreases. This reduction is due to the hydration of magnesium oxide ( $\rho$ =~3.58 g cm<sup>-3</sup>) and the formation of Mg(OH)<sub>2</sub> ( $\rho$ =~2.34 g cm<sup>-3</sup>). For the samples post-treated with CO<sub>2</sub>, the density of samples increases by adding nano magnesium oxide. This increase may be accomplished by the creation of magnesium carbonate ( $\rho$ =~2.96 g cm<sup>-3</sup>)

from magnesium hydroxide ( $\rho = \sim 2.34$  g cm<sup>-3</sup>) according to equation 3 [30].

 $Mg (OH)_2 + CO_2 \rightarrow MgCO_3 + H_2O$  $\Delta H = -30.2 \text{ kJ mol}^{-1}$ (Eq. 3)

In addition to MgCO<sub>3</sub>, the formation of the hydrated phase of magnesium carbonate has also been reported. In other words, the reaction between water and MgCO<sub>3</sub> can create nesquehonite (MgCO<sub>3</sub>.3H<sub>2</sub>O), dypingite (Mg<sub>5</sub>(CO<sub>3</sub>)<sub>4</sub>(OH)<sub>2</sub>.5H<sub>2</sub>O), and artinite (Mg<sub>2</sub>(OH)<sub>2</sub>CO<sub>3</sub>.3H<sub>2</sub>O) [31,32]. Among these hydrated phases of magnesium carbonate, nesquehonite is more common in the CO<sub>2</sub> post-

treatment [30]. It was reported that the formation of these hydrated phases could create a densified structure with high binding power, therefore improving the mechanical characteristics of the construction [33,34]. However, the formation of the prominent phase in the final products strongly depends on many factors such as temperature,  $CO_2$  pressure, the humidity of  $CO_2$  gas, etc. [35–38].



Fig. 3. The density of the samples with different amounts of MgO post-treated with (a) water and (b) CO<sub>2</sub>.

Fig. 4 shows SEM micrographs of the samples with different amounts of MgO post-treated for 45 days with water and  $CO_2$ . As can be seen, the samples post-treated with water have particles with irregular and spherical morphologies, whereas the samples post-treated with  $CO_2$  exhibit irregular and needle-like morphologies. The needle-like shape of particles in the carbon dioxide post-treated samples may be due to the formation of carbonate phases. There are some cracks and pores in the pure concrete, which

could be attributed to the contraction of concrete during the drying. By increasing the nano magnesium oxide in the concreate, the number of cracks and pores is reduced. This reduction of the defects in the nano-MgO added samples is due to the volume changes during the formation of hydrated and carbonated phases during the post-treatment process. In other words, the expansion due to the formed phases reduces the contraction effect of the concrete [39,40].



Fig. 4. SEM micrographs of the samples with different amounts of MgO post-treated with (a) water and (b) CO<sub>2</sub>.

Fig. 5 represents the compressive strength of the samples with different amounts of MgO and post-treated with water and  $CO_2$  on different days. It is clear that in all samples, with the increase of post-treatment days from 7 days to 45 days, the compressive strengths of the samples improve. This improvement could be attributed to the formation of hydrated and carbonated phases during post-treatment, which results in the reduction of the formed stress in

the concrete. In addition, the addition of MgO leads to an improvement in the mechanical properties of the samples. The SEM results showed that adding MgO reduces the number of defects like cracks and pores, which may explain why nano-MgO ameliorates the compressive strength. In addition, it was reported that the improvement in the mechanical properties of samples by adding MgO could be due to its high activity and the seeding effect of these nanoparticles [41]. It seems that post-treatment with  $CO_2$  has a better effect on improving compressive strength. For instance, the 4% MgO-C sample reached a compressive strength of about 55 MPa, but the 4% MgO-N sample had a compressive strength of about 51 MPa after 45 days post-treatment. It is noteworthy that the uniform distribution of nano-size MgO in the concrete matrix is critical. It is because, during the hydration and carbonation of magnesiumoxide, uniform stress would create in the microstructure, which results in the filling of pores and cracks. Therefore, it may cause

better improvement in the mechanical properties of final products. It is noteworthy that high amounts of MgO or large-scale MgO may have an adverse effect on the mechanical properties of samples. For example, It was reported that high amounts of large-size magnesium oxide in the cement could cause extreme expansion, which results in the formation of cracks and reduction of mechanical properties. In the nanosized scale, the formation of these local stressed is reduced, which may improve these destructing expansion effects [42,43].



Fig. 5. Compressive strength of the samples with different amounts of MgO post-treated with (a) water and (b) CO<sub>2</sub>.

#### 4. Conclusions

The effect of two different post-treatment with water and  $CO_2$  on the mechanical properties of the MgOadded concrete was investigated. The samples were studied via X-ray diffraction and Scanning electron microscopy. The XRD results showed that CaCO<sub>3</sub>, SiO<sub>2</sub>, and Ca(OH)<sub>2</sub> phases are formed in the posttreated samples with H<sub>2</sub>O, whereas the hydrated phases are not observed in the post-treated specimens with  $CO_2$ . Compressive strength improved by adding nano-MgO and increasing the time of post-treatment. The samples post-treated in  $CO_2$  had irregular and needle-like morphologies. The density of the samples post-treated with water decreases with adding nano-MgO, whereas the density of the ones post-treated with carbon dioxide increases by increasing magnesium oxide. Post-treatment of concrete with  $CO_2$  had a better effect on the ameliorating of mechanical properties than processing with water.

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