










## Research Article

# Effects of dry particle coating with nano- and microparticles on early compressive strength of portland cement pastes

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## ABSTRACT

It is known that nano-and microparticles have been very popular in recent years since their advantages. However, due to the very small size of such materials, they have very high tendency to agglomeration particularly for nanoparticles. Therefore, it is critical that they are properly distributed in the system to which they are added. This paper investigated the effects of dry particle coating with nano-and microparticles to solve the agglomeration problem. For a clear evaluation, paste samples were preferred to determine the compressive strength. Nano-SiO<sub>2</sub> and nano-CaCO<sub>3</sub>, micro-CaCO<sub>3</sub> and micro-SiO<sub>2</sub>, also known as silica fume, were selected as particulate additives. It was studied by the addition of various percentages (0.3, 0.7, 1, 2, 3 and 5%) of nano-and microparticles in cementitious systems, replacing cement by weight with and without dry particle coating. Dry particle coating was made by using a high-speed paddle mixer. Portland cement and additive particles were mixed at 1500 rpm for 30 seconds in high-speed powder mixer designed for this purpose. The 3-day compressive strength of the cement-based samples to which particles were added at the specified rates was determined and the effect of the dry particle coating on the early strength was investigated. According to the results, it was observed that the production of paste with the dry particle coating technique gave higher compressive strength compared to the production of paste directly in early period. Especially with dry particle coating, compressive strength increased more than 100% in paste samples containing 0.3% nano-SiO<sub>2</sub> compared to direct addition without coating.

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## 1. Introduction

Cement, which is the main component of concrete, is commonly used in the civil engineering area due to its advantages such as high compressive strength, simple preparation, economical and ease to use (Han et al., 2017). On the other hand, Portland cement is a material with high environmental effect due to high amount of CO<sub>2</sub> released to nature and high energy consumption during production of cement (Ouyang et al., 2017). The carbon emission released to the nature during Portland

cement production constitutes approximately 8% of the CO<sub>2</sub> emission in the World (Atış et al., 2015; Durak et al., 2021) Due to this negative effect of Portland cement, new systems were investigated.

To obtain an eco-friendly system, some amount of cement can be replaced with supplementary cementitious materials like fly ash and silica fume (Camiletti et al., 2013). The use of mineral additives such as micro silica, also known as silica fume, in cementitious systems has positive effects both environmentally and economically, as well as filling voids and showing partial binding effect

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(Oltulu and Şahin, 2013). It has been reported that micro-CaCO<sub>3</sub> acts as an inert filler and forms a denser microstructure and reduce the setting time (Camiletti et al., 2013).

Nanotechnology can be defined as an emerging research area with potential impact on every field of science and technology (Singh et al., 2013). Nanoparticles have a size between 10 to 1000 nanometers (Mohanraj and Chen, 2007).

According to the literature, nano-SiO<sub>2</sub> and nano-CaCO<sub>3</sub> can enhance the early strength of cementitious systems. In addition, nano-CaCO<sub>3</sub> has been used by many scientists to develop the pore structure and early strength of cementitious systems (Meng et al., 2017; Ren et al., 2021). Besides, it was observed that as the nano-CaCO<sub>3</sub> content increased, the flowability of the system decreased and it has been reported that increasing nano-CaCO<sub>3</sub> content up to a certain value increases the compressive strength (Liu et al., 2012).

Nano-SiO<sub>2</sub>, one of the most studied nanoparticles, has three major effects in cementitious materials. These are the filling effect, nucleation effect and pozzolanic effect (Ren et al., 2021). Xu et al. (2016) reported that the inclusion of nano-SiO<sub>2</sub> can optimize the pore structure and accelerate the early hydration. Nano-SiO<sub>2</sub> reacts with calcium hydroxide, which is formed during the cement hydration and does not contribute much to strength development, and additional C-S-H is produced (Singh et al., 2013).

The surface area to volume ratio of nanoparticles is quite high. Nanoparticles with a diameter of 4 nm are very reactive because they have more than half of their atoms on their surface. Chemical reactions at the interface and the agglomeration tendency of materials are key factors to influencing their behavior. This agglomeration affects the rheological properties of systems including nanomaterials that are more difficult to disperse (Senff et al., 2012). In order to observe positive proper-

ties of nanoparticles, particles must be dispersed without agglomeration (Tsuzuki and McCormick, 2004). Cementitious materials containing well-dispersed nanoparticles form dense microstructure, but if the nanoparticles are not properly dispersed, it can cause voids and weak zone formation (Li et al., 2004).

In this study, the effect of dry particle coating on early age of cement-based materials containing micro and nano-sized SiO<sub>2</sub> and CaCO<sub>3</sub> were investigated. It is aimed to overcome this agglomeration problem with the dry particle coating.

Compressive strength is a significant property of cementitious materials (Joshaghani et al., 2020). In order to observe the effect of dry particle coating, compressive strength test was performed on the paste samples on the 3rd day and early strength was investigated.

## 2. Materials and Method

The ordinary Portland cement (CEM I 42.5R) is used for the cement based pastes investigated in this study. In this research, as a particle to be used in cementitious based paste samples; nano calcite (NC), nano silica (NS), micro calcite (MC) and micro silica (MS) have been selected. Their chemical compositions are presented in Table 1.

Nano and micro sized particles were added to the cementitious system in various percentages (0.3, 0.7, 1, 2, 3 and 5%) by replacing the cement weight. But, the surface area of NS is much higher than other particles, hence the maximum dose was determined as 2% for NS.

While applying the dry particle coating, cement and particles were mixed in the high speed paddle mixer, which is shown in Fig. 1, at 1500 rpm for 30 seconds. As a result of the preliminary studies, 30 seconds was found to be the optimum time for dry particle coating. Longer mixing was not preferred due to the possibility of having a grinding effect on cement particles.

**Table 1.** Chemical composition of materials.

	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	SO <sub>3</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	MgO	P <sub>2</sub> O <sub>5</sub>	TiO <sub>2</sub>	CO <sub>2</sub>	LOI
Cement (PC)	19.18	4.92	3.35	61.50	3.09	0.25	0.55	2.80	-	-	-	3.58
NC*	0.54	0.1	0.01	99.03	-	-	-	0.30	-	-	-	-
NS	99.48	0.1	0.03	0.11	-	0.04	0.02	-	0.03	0.05	-	0.12
MC	0.05	0.15	0.06	55.4	-	-	-	0.68	-	-	41.2	0.18
MS	93.80	0.21	0.65	0.95	0.41	0.74	1.18	1.02	0.09	-	-	0.13

\*After ignition

The paste mixtures that have been prepared were made into 21x21x21 mm molds. The water/binder ratio was used with a constant value of 0.40 for all paste samples. The paste mixing procedure given in the same standard was followed. Firstly, water was added to the binder material and it was waited for 30 seconds to be fully absorbed. Then, mixing at the low speed for 30 s. After his step, the mixer was stopped and the mortar was allowed to stand for 15 s. Finally, it was mixed at high

speed for a further 60 s. For better compaction of the paste samples, two layers were cast into the molds and after each casting, they were tamped 25 times with a syringe needle. The climate cabinet was set at 99% humidity and 22°C and the samples were cured here for 3 days. The samples taken out of the climate cabinet after 3 days are shown in Fig. 2. The paste samples prepared for compressive strength test by removing from the mold with the utility knife are shown in Fig. 3. A universal testing

machine was used to determine the compressive strength of the pastes. Loading speed of 0.2 kN/s was selected for the compressive strength testing. Compressive strength tests were performed on three samples from each mixture and the average values were taken as a result.

### 3. Results and Discussion

The compressive strength of the samples was tested at the 3 days. The compressive strength of the samples increased with the dry particle coating. The 3-day compressive strength of Portland cement paste was found to be 20.5 MPa. Figs. 4-7 illustrated the 3-day compressive strength of paste samples with and without dry particle coating.



Fig. 1. High-speed paddle mixer.



Fig. 2. Paste samples removed from the climate cabinet after 3 days.



Fig. 3. Samples taken out from the molds and made ready for the experiment.

Fig. 4 shows that the compressive strength of coated paste samples with 0.3, 0.7, 1, 2, 3 and 5% micro- $\text{CaCO}_3$  (MC-C) have 20, 17, 19, 34, 18 and 3% higher at early age (3 day) compared to uncoated samples (MC), respectively. According to these results, the sample which the dry particle coating has the most significant effect is 2% MC-C. In addition, the highest strength was obtained from paste made with 2% MC-C which is 30.6 MPa.

Fig. 5 illustrates that the compressive strength of coated specimens with the addition of MS percentage as 0.3, 0.7, 1, 2, 3 and 5% are 16, 10, 11, 11, 1 and 7% higher, respectively, than uncoated samples. This results imply

that the dry particle coating has a little positive effect on the compressive strength of cement-based materials with MS. The reason for this situation may be that the dry particle coating is not applied very well on the samples with MS added.

Based on Fig. 6, the application of dry particle coating significantly increased the strength gain early age for 2%NC-C samples. The addition of 2% NC with dry particle coating improved the compressive strength of the paste by more than 40% compared to the uncoated sample. Fig. 7 demonstrates that the compressive strength of coated specimens with the incorporation of NS percentage as 0.3, 0.7, 1 and 2 % are 113, 8, 1 and 26% higher,

respectively, compared to uncoated samples. The effect of the dry particle coating is remarkable for cementitious materials with 0.3% NS. The compressive strength val-

ues for the coated cementitious material with 0.3% NS (NS-C) is 32 MPa while uncoated cement-based material with 0.3% NS is 15 MPa.

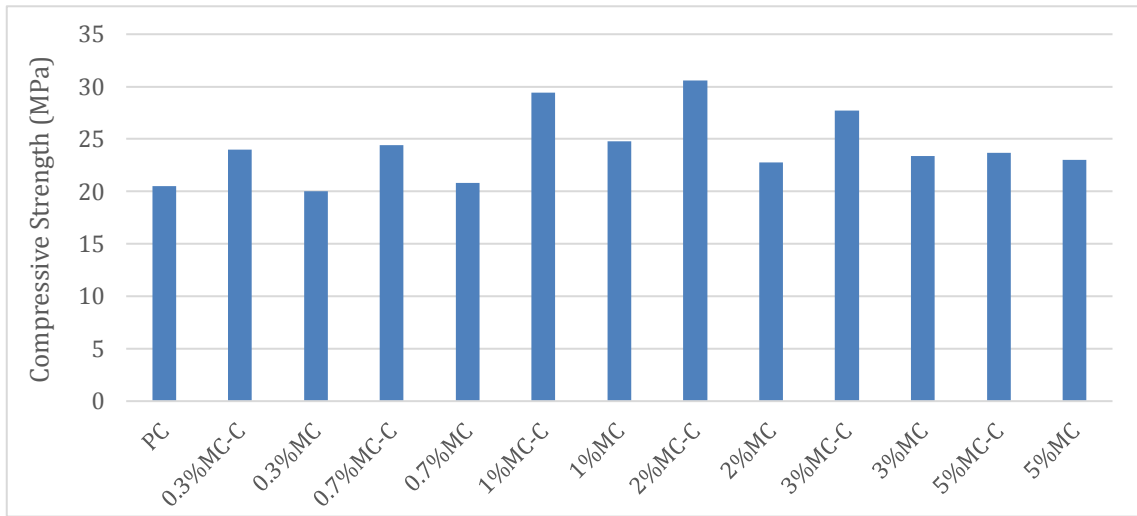


Fig. 4. 3-day compressive strength of pastes with coated and uncoated systems containing MC.

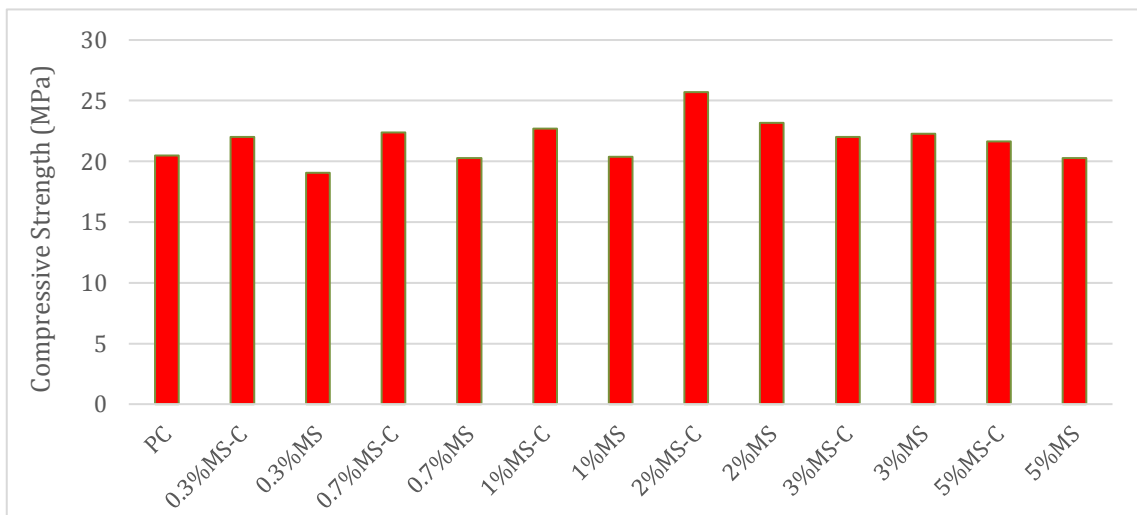


Fig. 5. 3-day compressive strength of pastes with coated and uncoated systems containing MS.

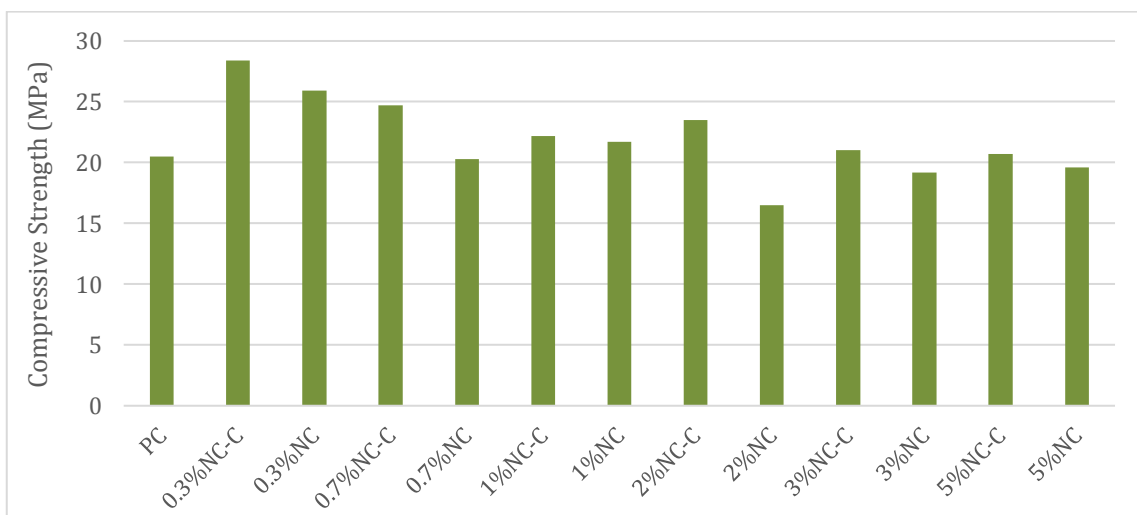


Fig. 6. 3-day compressive strength of pastes with coated and uncoated systems containing NC.

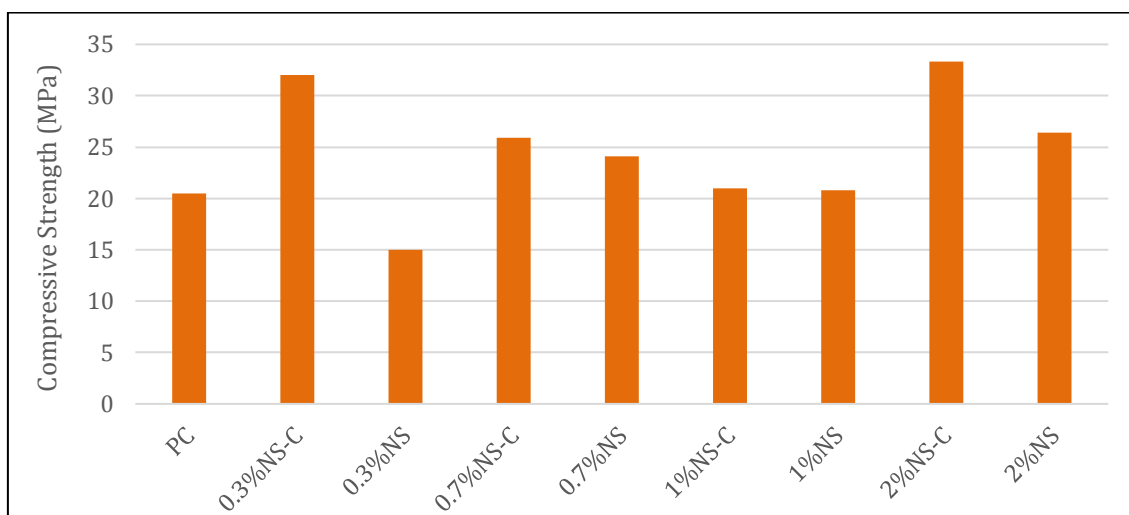


Figure 7. 3-day compressive strength of pastes with coated and uncoated systems containing NS.

#### 4. Conclusions

In this publication, the dry particle coating on cement-based materials with the addition of micro- and nano-sized  $\text{SiO}_2$  and  $\text{CaCO}_3$  was investigated. The following conclusions can be made from the results of this research:

- Experimental results indicate that the cementitious system with the addition of 0.3% NS has the highest effect on the compressive strength of the dry particle coating among all paste mixtures.
- Dry particle coating has not a significant effect on the compressive strength of cementitious materials incorporating with MS.
- Among the samples containing NC, the dry particle coating improved the compressive strength of the sample which is containing 2% NC by 42%.
- The most significant effect of the dry particle coating on the MC added samples was observed at 2% MC. 34% increase in compressive strength was observed in the coated 2% MC compared to the uncoated samples.
- It has been observed that dry particle coating has a positive effect on the mixtures prepared by adding nano- and micro scale calcite and silica to cementitious systems.

More studies can be conducted on the effect of dry particle coating on the mechanical and physical properties of systems containing nanoparticles in the later stage in future.

#### Publication Note

This research has previously been presented at the International Congress on Art and Design Research and Exhibition held in Niğde, Turkey, on June 21-22, 2021. Extended version of the research has been submitted to Challenge Journal of Concrete Research Letters and has been peer-reviewed prior to the publication.

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