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The influence of an additive in the form of selected nanoparticles on the physical and mechanical characteristics of self-compacting concrete

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Abstract

The influence of nanoparticle additives on the physical and mechanical characteristics of hardened self-compacting concrete (SCC) was studied. Research included SCC concretes modified with different amounts of SiO\textsubscript{2}, TiO\textsubscript{2} and Al\textsubscript{2}O\textsubscript{3} nanoparticle additives and one reference concrete made without nanoparticles. Rheological properties, microstructure and compressive strength were determined. The obtained results showed that SiO\textsubscript{2} and Al\textsubscript{2}O\textsubscript{3} additions worsened the workability while the compressive strength was increased in the case of SiO\textsubscript{2} addition. All studied nanomaterials densified the microstructure of the hydrated binder matrix.

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1. Introduction

For the past several years a significant interest in the use of materials with nanodimensions has been noticeable in the construction industry. The most popular construction material, namely concrete, is also in the area of this interest. In many centers around the world attempts to modify concrete with nano-additives are being undertaken and their main objective is to specify the influence of nanoparticles on the physical and mechanical properties of the

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material which is obtained with their participation. From a review of literature it can be concluded that in most cases for the modification of concrete, nano-SiO$_2$, Al$_2$O$_3$, CuO, TiO$_2$, ZnO$_2$, Fe$_2$O$_3$, and Cr$_2$O$_3$ were used to modify concrete mixes. The used amounts of added nanomaterials varied from 0.2 wt.% to 18 wt.% [1-10].

In a few articles there is information available on the influence of nanoparticles on the rheological parameters of concrete mixes made with their use. In study [1] it was stated, for example, that the addition of nano-Cr$_2$O$_3$ adversely affects the workability of a concrete mix. Analogous results were obtained for the addition of nano-Al$_2$O$_3$ in study [2]. In contrast, there are no studies in literature which show how nanoparticles affect the rheological properties of self-compacting concrete mixes. It is important as self-compacting concrete is more commonly used in the construction industry.

There are only a few studies to date which show the influence of the addition of nanoparticles on the rheological parameters of a concrete mix and also on the physical and mechanical properties of hardened concrete. Studies [3,4] show that the use of the nano-TiO$_2$ additive has a positive effect on the compressive and bending strength of concrete and it also improves its structure by reducing porosity. Similar results were obtained for the addition of CuO nanoparticles [5]. Another study which also indicated the positive influence of nanoparticles as an additive which improves mechanical properties of concrete is the work [6] which showed that thanks to the addition of nano-Al$_2$O$_3$, a greater bending strength of concrete can be obtained. In turn, study [7] proves that the compressive and bending strength of a cement mortar increases due to the addition of using both nano-SiO$_2$ and nano-Fe$_2$O$_3$. Similar results were obtained in the case of nano-SiO$_2$ in studies [8,9].

A number of publications prove a significant improvement in the microstructure of concretes with the inclusion of nanoparticles in their composition. In studies [8,10] it was mentioned that the Al$_2$O$_3$ nano-additive causes a reduction of porosity and a better pore structure.

Based on a review of literature it can be said that research, on both concrete mixes and also hardened concretes which are made with the addition of nanoparticles, is dispersed, selective and incomplete and does not allow clear conclusions regarding whether and to what extent it is advantageous to use nanoparticles as a component of concrete to be drawn. This statement became an inspiration for authors to conduct their own analysis.

The purpose of this paper is to present the results of own studies which show what the influence is of the selected nanoparticles on the rheology of a self-compacting concrete, on the physical and mechanical characteristics of the concrete made of these mixes and also the porosity characteristics of hardened concretes.

2. Experimental setup

The self-compacting concrete mixes which were used in the research were made of the following components: Portland cement CEM I 52.5R; superplasticizer Glenium Sky 600 with a density of 1.06 g/cm$^3$ in an amount of 4% wt.%; granite aggregate with an average density of 2.67 g/cm$^3$ and fractions of 10-5, 5-2, 2-1, 1.2-0.5 and 0.6-0.1 mm; a fraction with a particle size <0.1 mm acting as a fine filler and also tap water. Three types of nanoparticles (nano-additive) were used as additives, such as: SiO$_2$ with a particle size of 10-20 nm in an amount equal to 0.5 wt.%, 2.0 wt.% and 4.0 wt.% of the cement weight; TiO$_2$ with a particle size <25 nm in an amount equal to 0.5 wt.%, 2.0 wt.% and 4.0 wt.% of the cement weight and also Al$_2$O$_3$ with a particle size <50 nm in an amount equal to 0.5 wt.%, 1.0 wt.%, 2.0 wt.% and 3.0 wt.% of the cement weight. The W/C ratio was constant and equaled 0.42.

From the above-mentioned ingredients a total of 11 self-compacting concrete mixes were prepared, from which one mix was prepared without the addition of nanoparticles as a reference. A summary of compositions of the designed mixes per 1m$^3$ is presented in Table 1. These mixes are marked with the symbols S1÷S11.

Homogenization of each mix was carried out in a mechanical mixer in two stages. At first, cement with aggregate and the addition of nanoparticles was stirred for 2 minutes and after the addition of water combined with the superplasticizer, everything was then stirred for a further 2 minutes.

The rheological properties of each mix were assessed using an Abrams cone by measuring the maximum diameter of the slump.

From each mix a series of six cubes having dimensions of 100x100x100 mm were made in order to investigate the compressive strength. These series were marked analogously to the mixes from which they were made, namely S1÷S11. Strength tests were carried out after 28 and 90 days. All samples were cured in a climate chamber at a temperature of 20°C (± 1°C) and with a relative humidity of 95% (± 5%).
Table 1. Summary of compositions of the designed self-compacting concrete mixes per 1m³.

| Number of mix | Type of nano-additive and its amount (%) | Water (kg) | Cement (kg) | Aggregate (kg) | Nano-additive (kg) | SP (kg) |
|---------------|-----------------------------------------|------------|-------------|----------------|-------------------|--------|
| S1            | -                                       | 193,2      | 460,0       | 1640           | 0,0               | 18,4   |
| S2            | SiO₂ - 0,5%                             | 193,2      | 457,7       | 1640           | 2,3               | 18,4   |
| S3            | SiO₂ - 2,0%                             | 193,2      | 450,8       | 1640           | 9,2               | 18,4   |
| S4            | SiO₂ - 4,0%                             | 193,2      | 441,6       | 1640           | 18,4              | 18,4   |
| S5            | TiO₂ - 0,5%                             | 193,2      | 457,7       | 1640           | 2,3               | 18,4   |
| S6            | TiO₂ - 2,0%                             | 193,2      | 450,8       | 1640           | 9,2               | 18,4   |
| S7            | TiO₂ - 4,0%                             | 193,2      | 441,6       | 1640           | 18,4              | 18,4   |
| S8            | Al₂O₃ - 0,5%                            | 193,2      | 457,7       | 1640           | 2,3               | 18,4   |
| S9            | Al₂O₃ - 1,0%                            | 193,2      | 455,4       | 1640           | 4,6               | 18,4   |
| S10           | Al₂O₃ - 2,0%                            | 193,2      | 450,8       | 1640           | 9,2               | 18,4   |
| S11           | Al₂O₃ - 3,0%                            | 193,2      | 446,2       | 1640           | 13,8              | 18,4   |

Two additional cubes were made in order to specify the characteristics of air pores in hardened concretes. To prepare samples a 25 mm thick slices were cut out from each cube and polished. The measurement of air pore characteristics was made by using the microscopic method with a Nikon SMZ800 microscope and also an automatic image analysis computer system which cooperates with a Sony DXC950P camera and Image ProPlus4.1 software with the Scope Pro module. The range of the tested pores included those with a size from 4 to 4000 μm.

The determination of the porosity of the obtained self-compacting concretes in the range of pore size between 5 nm and a pore size greater than 1000 nm was made with the use of the mercury intrusion porosimetry method (MIP). For the purpose of the analysis an additional series of small beams with dimensions of 40 x 40 x 160 mm was executed and in those beams a drill hole was made with a core drill. Next, after collecting the material, all samples were dried in a Memmert 200 dryer at a temperature of approximately 30°C for a minimum 48 hours. Their dryness condition was verified using the gravimetric method with AND weight. The procedure of measuring the porosity was carried out using an AutoPore 9220 mercury intrusion porosimeter.

3. Test results and analysis

Figure 1 summarizes the results obtained from research of the rheological characteristic of the prepared 11 mixes in the form of the maximum diameter of slump in relation to the amount of nano-additive.

![Figure 1](image-url)
It can be noted that with an increase of the amount of SiO\(_2\) and Al\(_2\)O\(_3\) nanoparticle additives the fluidity of the mix which is assessed on the basis of the measurement of the maximum diameter of slump deteriorates. This could be explained by the fact that of a high specific surface area of the used nanoparticles and thus increased water demand. Similar trends were observed elsewhere [11]. Furthermore, the observed deterioration of the fluidity of mixes with the SiO\(_2\) and Al\(_2\)O\(_3\) nano-additives could be affected by their high chemical reactivity which is manifested in e.g. the pozzolanic reaction, cannot be omitted. In turn, the influence of the TiO\(_2\) nano-additive on the fluidity of concrete mixes was negligible. It should be noted that all the tested mixes, except that of the S3, S4 and S11 mixes, met the requirements for the self-compacting concrete which are presented in [12].

The compressive strength values were determined after 28 and 90 days of curing and are shown in Figure 2. The obtained test results revealed that 28 days compressive strength values of all samples containing the addition of nano-SiO\(_2\) (series of concrete S2, S3 and S4) were higher in comparison with the reference concrete of series S1.

Enhanced compressive strength was also observed with the addition of nano-TiO\(_2\) in an amount of 2 wt.% (series S6) and nano-Al\(_2\)O\(_3\) in an amount of 0.5 wt.% and 2 wt.% (series S8 and S10) of the cement weight. However, when analysing the results of the compressive strength after 90 days it can be stated that a higher compressive strength of concrete was obtained for the series of concrete S2, S4, S6 and S8. The phenomenon which explains the increase of concrete strength parameters due to the use of nano-additives was presented, among others, in study [7]. A high chemical reactivity of nanoparticles could enhance formation of the C-S-H by creating additional nucleation sites and thus increasing the ultimate compressive strength. Furthermore, some of the used nanoparticles could initiate a pozzolanic activity and thus would cause transformation of a part of the portlandite into additional C-S-H. At the same time nanoparticles reduce the growth of Ca(OH)\(_2\) crystals. The particle packing density of the mix was also increased due to the presence of nano-additives which additionally could enhance the strength.

![Figure 2. The compressive strength f\(_{cm}\) of the tested series of self-compacting concrete after (a) 28 days of curing; (b) 90 days of curing.](image-url)

The characteristics of air pores as a total content of air A in the hardened 90-day concrete, as well as the content of micropores with a diameter of less than 300 \(\mu\)m \(A_{300}\) in the tested 90-day concretes, is presented in Table 2. Only in the case of 0.5 wt.% of nanoparticle participation (series S2) for concretes with the SiO\(_2\) nano-additive, was the total air content in concrete lower than in the comparative concrete of series S1.

However, in the case of TiO\(_2\) nano-additive, concretes with 0.5 wt.% and 2.0 wt.% of this additive were characterized by a lower porosity. The use of 0.5 wt.% and 1.0 wt.% of Al\(_2\)O\(_3\) nano-additive also improved parameter A. However, it is very significant that the fact that all the tested series of concrete, except series S2 and S9, were characterized by lower porosity in relation to micropores of up to 300 \(\mu\)m. The reduction of the porosity, and in this case the reduction of pore diameter, can be explained by the filler effect and creation of additional nucleation sites by nanoparticles for the growth of cement hydration products [7,13].
Table 2. Characteristics of air pores in the studied series S1÷S11 of self-compacting concretes.

| Symbol of concrete series | Total air content A in the hardened concrete (%) | Content of micropores with a diameter below 300 μm A<sub>300</sub> (%) |
|---------------------------|-----------------------------------------------|---------------------------------------------------------------|
| S1                        | 1,29                                          | 0,24                                                          |
| S2                        | 0,95                                          | 0,25                                                          |
| S3                        | 2,11                                          | 0,14                                                          |
| S4                        | 2,39                                          | 0,09                                                          |
| S5                        | 0,58                                          | 0,13                                                          |
| S6                        | 0,66                                          | 0,24                                                          |
| S7                        | 1,37                                          | 0,22                                                          |
| S8                        | 0,66                                          | 0,09                                                          |
| S9                        | 1,25                                          | 0,30                                                          |
| S10                       | 1,49                                          | 0,12                                                          |
| S11                       | 1,75                                          | 0,19                                                          |

The porosimetry test results of concrete series S1÷S11 are summarized in Table 3. The results are presented as the percentage participation of pores in concrete according to the assumed ranges of pore radius. It can be seen that, regarding pores with a size of up to 50 nm, the majority of tested concrete series which were modified with the addition of nanoparticles are characterized by a smaller percentage participation of pores when compared with the reference concrete of series S1. Only concrete series S2 and S9, and to a lesser extent series S5, showed a slightly higher amounts of pores in this range. In the other intervals of pore radius it is difficult to observe a clear relation regarding the influence of nano-additives which were used to prepare the tested concretes on the percentage decrease in pore participation in the considered ranges.

Table 3. The percentage participation of pores in the tested self-compacting concretes in the range of pore radius from 5 nm to > 1000 nm.

| Symbol of concrete series | 5-15 (nm) | 15-50 (nm) | 50-80 (nm) | 80-150 (nm) | 150-200 (nm) | 200-250 (nm) | 250-500 (nm) | 500-750 (nm) | 750-1000 (nm) | > 1000 (nm) |
|---------------------------|-----------|------------|------------|-------------|--------------|--------------|--------------|--------------|---------------|-------------|
| S1                        | 30,58     | 36,35      | 9,57       | 3,89        | 0,98         | 3,29         | 3,68         | 1,91         | 0,44          | 9,31        |
| S2                        | 33,40     | 38,98      | 9,45       | 3,56        | 0,65         | 3,01         | 3,14         | 1,74         | 0,29          | 5,78        |
| S3                        | 27,89     | 33,57      | 10,60      | 4,36        | 1,35         | 3,64         | 4,42         | 2,02         | 0,68          | 11,47       |
| S4                        | 26,29     | 32,53      | 11,03      | 5,29        | 2,01         | 4,45         | 5,64         | 2,29         | 0,71          | 9,76        |
| S5                        | 30,76     | 36,90      | 10,97      | 4,17        | 0,99         | 3,24         | 3,62         | 1,84         | 0,42          | 7,09        |
| S6                        | 27,93     | 33,29      | 10,60      | 4,36        | 1,29         | 3,44         | 4,19         | 1,93         | 0,65          | 12,31       |
| S7                        | 29,40     | 34,83      | 10,65      | 4,09        | 1,05         | 3,19         | 3,66         | 1,77         | 0,52          | 10,85       |
| S8                        | 30,57     | 36,55      | 10,38      | 3,82        | 0,81         | 3,04         | 3,42         | 1,69         | 0,39          | 9,33        |
| S9                        | 31,03     | 37,29      | 11,32      | 4,18        | 0,90         | 3,11         | 3,28         | 1,69         | 0,35          | 6,84        |
| S10                       | 29,98     | 36,22      | 11,41      | 4,39        | 1,02         | 3,26         | 3,64         | 1,80         | 0,42          | 7,86        |
| S11                       | 29,00     | 35,04      | 11,30      | 4,29        | 1,24         | 3,43         | 3,94         | 1,84         | 0,50          | 9,42        |

4. Summary

Based on the conducted rheological research of the 11 self-compacting concrete mixes it can be concluded that, depending on the amount of SiO₂ and Al₂O₃ nanoparticle additives which were used to make the concrete, the fluidity of the studied SCC concrete mixes deteriorated and in contrast, in the case of the addition of TiO₂, it remains almost unchanged. Eight out of eleven studied concrete mixes met the requirements for self-compacting concrete mixes.

Tests of the compressive strength of self-compacting concretes made from the designed mixes showed that the SiO₂ nano-additive which was used for making concrete in an amount ranging from 0.5 wt.% to 4 wt.% of cement weight, clearly and beneficially enhanced the 28-day compressive strength when compared to the reference concrete. In the case of concretes containing TiO₂ and Al₂O₃ nano-additives the compressive strength appeared to be unaffected.
The MIP test results showed that most tested series of concretes modified with the addition of SiO$_2$, TiO$_2$ and Al$_2$O$_3$ nanoparticles were characterized by finer porosity in the range of pores with a size of up to 50 nm, when compared to the reference concrete series S1.

Similarly, porosity below pore size of 300 $\mu$m appeared to be lower in concretes containing nanomaterials except concrete series S2 and S9 containing respectively 0.5 wt.% of SiO$_2$ and 1 wt.% of Al$_2$O$_3$.

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