Cement-Based Materials Modified with Nanoscale Additives

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Abstract. The most common and reliable material without which modern construction is indispensable is concrete. The development of construction production is pushing for new solutions to improve the quality of concrete mix and concrete. The most demanded and significant indicators of a concrete mixture are the compressive strength and mobility of the concrete mixture. Every year, the volume of research on nanomaterials as modifying components of concrete is significantly increasing, and the results indicate the prospects for their use. Nanoparticles with a large specific surface are distinguished by chemical activity, can accelerate hydration and increase strength characteristics due to nucleation and subsequent formation of C–S–H and compaction of the material microstructure. Sol of nanosilica, which can be used instead of microsilica from industrial enterprises, and carbon nanomaterial have a wide reproduction base. This paper presents studies of these types of nanomaterials and the results of their application in cement concrete. Studies have shown that the effect is also observed with the introduction of an additive containing only one type of nanoparticles. The dependence of the obtained characteristics of cement concretes on the content of these nanomaterials has been established. It has been found that the best results were obtained with an additive in which the above-mentioned nanomaterials were used together. Compressive strength of heavy concrete samples, improved by the complex nanodispersed system, was 78.7 MPa, which exceeds the strength of the sample obtained with an additive in which the above-mentioned nanomaterials were used together. Compressive strength of heavy concrete samples, improved by the complex nanodispersed system, was 78.7 MPa, which exceeds the strength of the sample obtained with an additive in which the above-mentioned nanomaterials were used together. 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Keywords: water sol of nanosilica, carbon nanotubes, super-plasticizer, heavy concrete, strength

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Introduction

Currently, nanoscale materials have found their application in industry, as modifying components. Having very large values of the specific surface, nanosized particles are capable of affecting the physicomechanical characteristics of the new material. Depending on the type of raw material and the production method, the characteristics of the obtained nanomaterials vary widely [1–8]. All this affects the results obtained when using the same concrete mix formulations.

An analysis of the literature shows nanomaterials, such as carbon nanotubes and silica fume, were initially used only as a means to increase the strength characteristics of modified concrete. A little later it became known that the use of these nanomaterials together with plasticizing additives provide an effective reduction in water and an increase in the durability (frost resistance) of the created concrete [9–25]. However, questions regarding the compaction of the structure and the filling of voids between cement particles and even larger sand particles remain open at the moment. The working hypothesis of the study is that a complex nanodispersed system consisting of nanosilica, multilayer carbon nanotubes (MCNT) and a superplasticizer (SP) will allow to obtain a synergistic effect, i. e. summation of the positive properties and qualities of each of the components.

Materials and methods of experimental studies

The following materials were used in the work as nanomaterials: water sol of nanosilica SiO₂, obtained from hydrothermal fluids, with characteristics of a pH = 9.2, density ρ = 1075 g/dm³, solids content of nanosilica SiO₂ = 120 g/dm³, total salinity equal to 1720 mg/dm³ [26–28] and carbon nanotubes obtained in a plasma of a high-voltage atmospheric pressure discharge with the optimal composition of the CH₄ gas mixture: air = 1:(2.4–2.5), followed by chemical treatment with a mass fraction of solids of not less than 37 %, pH = 7.0, ρ = 1.1 g/cm³ [29–36]. Superplasticizer from a series of polycarboxylates highly effective in water-reducing ability in the form of an aqueous solution.

Portland cement CEM I 42.5N was used as a binder as a fine aggregate, quarry sand with a fineness modulus Mf = 3.8 with a true density ρ = 2572 kg/m³ and bulk density ρ = 1540 kg/m³ was used. As a coarse aggregate, the fraction is 5–20 mm thick, bulk density ρ = 1460 kg/m³.

The particle size distribution of water sols of nanosilica was determined using the Analysette 22 laser diffraction device, which is built on the principle of “Inverse Fourier Optics” or a converging laser beam (Fig. 1). Reverse Fourier Optics also allow extremely high resolution measurements. By automatically moving the measuring cell supported by the computer in a converging beam in the NanoTec model, it is possible to create a supermatrix with a max 520 measuring channels for calculating. In this case, the measurement range is from 0.01 to 1000 µm.

The results of determining the particle size of nanosilica were given the following results: the size of the solid particles of nanosilica contained in aqueous ash is less than 10 nm, which is explained by the resolution of the device (from 10 nm to 1000 µm).

The concentration and solids content of nanosilica SiO₂ in the sol was determined by evaporation of the initial water sol with a mass of m_initial = 100 cm³ in an oven at a temperature of t = 150 °C.

Weight reduction according to studies:
1 day – m₁ = 233 ml/g;
2 days – m₂ = 166 ml/g;
3 days – \( m_3 = 146 \text{ ml/g} \);
4 days – \( m_4 = 145 \text{ ml/g} \);
8 days – \( m_8 = 145 \text{ ml/g} \).

This indicates that the SiO\(_2\) = 120 g/dm\(^3\) approximating value for the indicated solid particle content is solid; the mass from the 4\(^{th}\) day remained unchanged due to the room humidity. Upon evaporation, the sol particles stuck together.

The results of measuring the particle size distribution of the dispersed composition of carbon nanomaterial are presented in Fig. 2.

In order to study the effect of nanomaterials on the basic properties of cement concrete, an additive was made and a series of laboratory studies was carried out. The amount of added additive in all studied compositions was 0.8 % by weight of cement.

To determine the effect of an aqueous sol of SiO\(_2\) (NS) silica on the strength characteristics of concrete, it was preliminarily mixed with mixing water, uniformly distributed throughout the volume, after which water was introduced into the concrete mixture. The mixture filled the cube-shaped nests (100×100 mm), which was mounted on a vibrating table and compacted. After manufacture, the samples were unformed and stored in bathtubs with water until they reached a certain age. Testing the samples for compressive strength was carried out on the 1\(^{st}\), 7\(^{th}\), 14\(^{th}\) and 28\(^{th}\) days.

The data obtained showed that when the sol content of nanosilica SiO\(_2\) in the amount of 0.1 wt. % in cement (NS 0.47) the increment in compressive strength compared to the control (Control) was +30 % on the first day, +20 % on the 28\(^{th}\) (Fig. 3).

It was found that an increase in the concentration of this nanosupplement NS 4.7 leads to a decrease in strength by 1.2 times, which indicates the advisability of using SP in this system.

A method of preparing a plasticizing additive used hereinafter consists in dispersing a superplasticizer and nanomaterial (nanosilica or nanocarbon) in a mixer for several minutes. After that, concrete samples were manufactured and tested according to the above methodology.

Tests of a plasticizing additive, including a nanosilica sol paired with a superplasticizer, were carried out at W/C values from 0.2 to 0.3 (Fig. 4).

An additive containing a nanosilica sol with a concentration of solid SiO\(_2\) equal to 0.47 g/kg, introduced into the superplasticizer in amounts of 8, 4, and 1 g/t when determining the compressive strength, showed the best result of 73.7 MPa when solid SiO\(_2\) 4 g was added per 1 t of superplasticizer.

At the same time, the introduction of NS increased the strength of concrete to 30 % compared to concrete containing only SP and to 40 % compared to the control sample.

The effect of a complex additive containing MCNT (MLCNT), NS and SP on the strength characteristics of concrete are presented in Fig. 5.

The compressive strength of samples of heavy concrete, improved by an integrated nanodisperse system, was 78.7 MPa, which exceeds the strength of a sample containing an MLCNT additive paired with superplasticizer by 27 %.
It can be assumed that the mechanism of action of the presented complex additive is as follows: the introduction of a nanocarbon material into the cement system leads to the packing density of the particles of the system, and when an even smaller nanosilica is introduced, additional crystallization centers of hydrated neoplasms are created. Therefore, nanosilica acts as a nanofiller of this system, which more actively affects the concentration of Ca\(^{2+}\) and OH\(^{-}\) and reduces the number of pores.

**CONCLUSION**

Based on the analysis of the test results, it can be concluded that a complex nanodispersed system, including multilayer carbon nanotubes, nanosilica and superplasticizer, helps to bring together particles, densify the structure and form inter-growth contacts, which effectively affects the structure of heavy concrete. Thus, this effect is achieved by directed structural modification of the main components of the cement stone – calcium hydroxysilicates relative to the composition and morphology of neoplasms.

**List of accepted symbols and abbreviations**

\(f_c\) – compressive strength of concrete, MPa;  
\(m_{1...8}\) – weight reduction, ml/g;  
\(\text{pH}\) – pH value;  
\(M_f\) – size module;  
\(\rho\) – density, kg/m\(^3\);  
indexes:  
\(c\) – compression;  
\(f\) – large-ness.

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