https://erjm.journals.ekb.eg
ISSN: 1110-1180

DOI: ---------

ERJ
Engineering Research Journal
Faculty of Engineering
Menoufia University

Experimental Study for Gypsum Materials Enhanced by Compound Additives Based on Nano-Silica

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ABSTRACT

Gypsum is naturally a soft mineral with low strength and with high porous. Without adding additives, it may not possess the desired level of strength for certain applications and have alow moisture resistance, low fire resistance, and low overall durability. Nano-silica and active mineral additives can significantly increase the mechanical properties and structure formation of gypsum binder, making them more durable and resistant to cracking or breaking. To improve the properties of the additive, the nanosilica has been subjected to sonication. It was confirmed that the effect of sonication allowed nanosilica to be used as a key component in additives and that there was no relationship between the initial particle size and this effect. Activated Portland cement and nano-silica have a positive effect on the mechanical properties of the material, and this effect is observed from the hydration process to hardening. The optimal range of nano-silica content was established, equal to 0.05 - 0.1%, while the increase in compressive strength reaches 30-40% compared to the control composition, water resistance increases to 0.47.The modified matrix is characterized by greater density and compressive strength. This result is due to the formation of new growths based on hydrated calcium silicate capable of binding to gypsum crystals and filling the pores at the same time. new growths based on hydrated calcium silicate were identified by IR spectra and physicochemical analyzes including differential thermal analysis, scanning electron microscopy and energy dispersive X-ray spectroscopy.

Keywords: *nanosilica, gypsum binder, complex additives, modified matrix and compressive strength*

1. Introduction

Currently, the actual direction of gypsum binder production is to obtain materials with improved physical and technical parameters. To solve this problem, various types of mineral additives are used, including products of technological products such as slag, ash and sludge of various origins [1-3]. Studies [4-6] indicate that the most effective method is to introduce silica containing additives that contribute to the formation of sparingly soluble products in the gypsum stone structure, preventing calcium sulfate from dissolving in water and providing strength and water resistance products. The efficiency of introducing these additives directly depends on the particle size of the modifier. Therefore, the introduction of silica containing additives with particle sizes in the micrometer range contributes to increasing the strength of gypsum composites by 10–30% [7,8] and slightly increasing their water resistance. However, as the specific surface area of the modifier increases, more particles are put into the hydration reaction, greatly increasing the efficiency of the additive. It has been demonstrated that the introduction of additives containing nano-sized particles can significantly increase the strength (up to 60%) and performance of composite materials [9,10]. A new modifier of binder properties is nano-silica, an additive containing micro and nanoparticles of silicon oxide. It has been demonstrated that the introduction of nano-silica into the composition of cement binders and concrete significantly reduces the porosity of the material and increases its strength and corrosion resistance [11-13]. At the same time, the use of nano-silica together with other types of additives (metakaolin, nanotubes) allows for more complete filling of the pore spaces of binders, resulting in high strength concretes and cement [14,15]. It is also known to use nano-silica to modify the properties of asphalt solutions, Increasing thermal stability [16,17]. There are developments aimed to modify gypsum binders with complex additives containing nanosilica and the cementitious component tricalcium aluminate. The co-introduction of these types of additives increases the strength by 68% and also reduces the setting time [18]. Low compressive strength, high porosity, low workability, limited fire resistance, and lack of specific properties are a few potential issues with using gypsum materials without any additional additives, so the study of the effect of Nano-silica and active mineral additives in the creation of gypsum binders is an important direction.

2. Materials and methods

Medium hard gypsum of medium grinding was used as the main binder component. The gypsum used is made of natural gypsum stone and complies with the requirements of GOST-125-79, and ASTM C472-20 specifications [19,20].

2.1. Alkaline component of the complex additive.

Based on the analysis of scientific research literature [21-23], Portland cement grade M400 D0, which meets the requirements of GOST 10178-85, ASTM C 191, ASTM C187, ASTM 184, and ASTM C 188 specifications [24-28] was selected as the alkaline component of the complex modifying additive in an amount of 5% by weight of the binder.

2.3. Pozzolanic component of the complex additive.

To ensure a stable increase in the strength characteristics of the gypsum binder, a chemically precipitated nanosilica was used in the composition of the complex additive, the content of which varied in the range from 0% to 0.5% by weight of the binder. The nano-silica used in the study, obtained by chemical treatment of liquid glass with hydrochloric acid, is a spherical fine particle with more than 90% SiO2 (Table 1).

The study used the addition of four selections, characterized by different granulometric compositions. An analysis of the particle distribution of the additive, presented in Figure 1, showed that the nano-silica has a polysized dispersed phase, represented by micro and nanosized particles in a wide range from 0.025 to 43.5 µm.

To determine the possibility of using nanosilica as a modifier, regardless of the changing technological modes of production, 4 samples of the additive were considered.

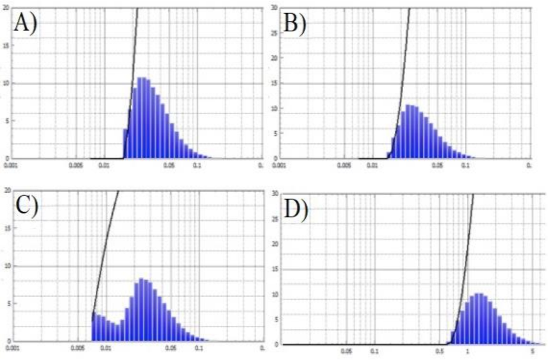


Figure 1. Granulometric composition of the dispersed phase of nanosilicas: A) first selection; B) second selection; C) the third selection; D) the fourth selection.

The powders were preliminarily subjected to sonication to activate them and to average the granulometric composition. To establish the possibility of using the additive, the effect of nano-silicic powders was studied immediately after activation and after holding the resulting suspensions for a month. The results of the analysis of the variance of the additive before and after ultrasonic treatment are presented in Table 2.

The dispersion analysis of nano-silica before and after activation made it possible to establish that the ultrasonic treatment of nano-silica powders of all selections leads to grinding of the largest phase of the additive, which generally contributes to the averaging of the granulometric composition of the powders. At the same time, it should be noted that the resulting suspensions are close in dispersed composition, regardless of the initial particle size in the composition of subdisperse powders. The average particle size in suspensions immediately after treatment is 0.031-0.036 µm, while the sizes of the most active particles are in the range of 0.022-0.036 µm. The overall stability of the resulting suspensions was also established, which characterizes the preservation of the concentration of the most active dispersed phase during storage.

Table 1. Chemical composition of nano-silica

Component	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**CaO**	**MgO**	**Na_2O**	**K_2O**	**SO_3**	**MnO**
Content,%	96,12	0,86	0,34	0,39	0,53	0,21	1,05	0,36	0,04

Table 2. Comparative dispersion analysis of nanosilica powders

Selection	Average size (µm) and particle content				Size range with the largest partial residues (µm) and their content
	≤ 50%	≤ 75%	≤ 90%	≤ 100%	
Powders					
NC-1	0,031	0,044	0,061	1,875	0,022-0,045 (50%)
NC-2	0,025	0,037	0,054	1,047	0,022-0,036 (39%)
NC-3	0,031	0,043	0,060	0,585	0,022-0,036 (50%)
NC-4	1,429	1,972	2,758	43,510	23,285-78,953 (57%)
After ultrasound processing					
NC-1	0,031	0,043	0,060	0,738	0,022-0,036 (51%)
NC-2	0,030	0,043	0,059	0,830	0,022-0,036 (50%)
NC-3	0,031	0,043	0,059	0,585	0,022-0,036 (51%)
NC-4	0,031	0,044	0,061	0,585	0,022-0,036 (49%)
One month after ultrasound treatment					
NC-1	0,031	0,043	0,059	0,738	0,022-0,036 (50%)
NC-2	0,036	0,053	0,078	1,177	0,025-0,040 (42%)
NC-3	0,031	0,043	0,059	0,738	0,022-0,036 (50%)
NC-4	0,031	0,043	0,059	0,585	0,022-0,036 (50%)

2.4 Mixtures proportions

To study the effect of a nano-silica suspension on the physical and mechanical properties of a gypsum-based composite material, nine types of compositions were prepared. The content of nanosilica in the composition of the composite material varied from 0.05 to 0.1% by weight of the binder, while the C-3 plasticizer was introduced at a concentration of 0.0125-0.025% by weight of the nanosilica, respectively. The content of the alkaline activator Portland cement was fixed at the level of 5% for all the compositions under consideration. The optimum water binding ratio for all formulations is set at 65%.

2.5 Mixing procedure and curing

Composite material samples were prepared as follows: nano-silica powder was activated by ultrasonication in an aqueous medium in the presence of superplasticizer C-3 in a ratio of 1:4. Activation was carried out on a Hielscher UP 200 ht device at a power of 150 W and amplitude of 19.5 kHz for 3 minutes. Portland cement, an activated suspension of nano-silica, and a gypsum binder were sequentially introduced into the mixing water with continuous stirring. At the same time, taking into account the water used to prepare the suspension, the water-binding ratio was corrected for each composition. The mixing of the components was carried out until the visual homogenization of the mixture, but not more than a minute after mixing the binder. The resulting mixture was poured into metal molds with geometric dimensions of prism samples 40x40x160 mm.

2.6 hydrophysical properties.

The determination of water resistance and water absorption of the composite material was carried out in accordance with the methods described in GOST-125-79, and ASTM C472-20 specifications [19-20]. The tests were carried out on a series consisting of 2 beams of the same composition, at the age of 28 days. The samples were weighed in a dry state and kept in water in a horizontal position in for 4 hours until they reach constant weight. At the same time, the beams were first filled with water up to half and kept for 2 hours, then they were poured water completely and kept for another 2 hours, after which samples were reweighed.

2.7 Physical and mechanical properties.

The strength characteristics of the studied compositions were determined according to standard methods in accordance with the requirements of GOST-125-79, and ASTM C472-20 specifications [19,20]. For testing, specimen beams with dimensions of 40x40x160 mm were made. Demoulding of the samples was carried out no earlier than 20 minutes after molding. Storage of samples until the moment of testing was carried out at T=20±2°, and relative air humidity of 60-70%. Tests of samples for tension in bending and compression were carried out at the age of 7 and 28 days on the press PGM-100MG4. SEM, IR-ray spectroscopy, differential thermal analysis, and dispersion analysis of materials were used to determine the change in physical and chemical properties and substantiate the processes leading to a change in physical and technical characteristics.

2.8 S.E.M.

The study of the features of the microstructure of a composite material based on gypsum was carried out at the Udmurt federal research center of the ural branch of the russian academy of sciences on a fisher scientific quattro S scanning electron microscope. Microstructure imaging parameters: pressure in a vacuum chamber 50 Pa; accelerating voltage HV 30 kV; beam current 16 A (Pa).

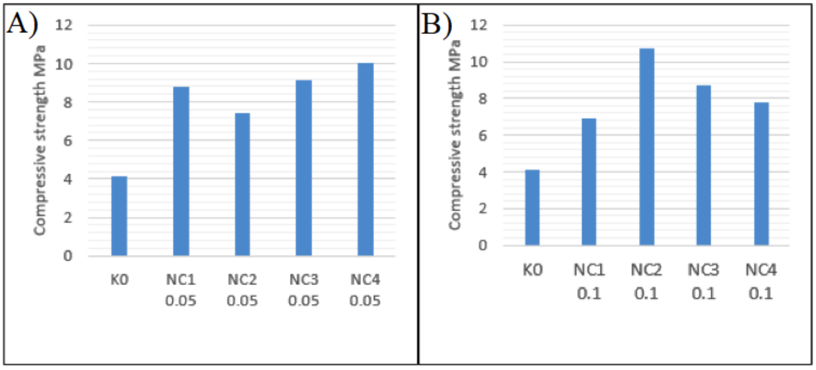


Fig. 2. Strength properties of gypsum binder with the introduction of ultrasonic treated nano-silica and cement for 7 days of hardening at a concentration of: A) 0.05%; B) 0.1%

2.9 IR-ray diffraction.

Infrared spectroscopy of a finely dispersed powder obtained by grinding samples of the control and modified composition was carried out on an IR-Fourier spectrophotometer "IRAffinity-1".

2.10 Differential thermal analysis.

Recording of DTA spectra obtained by heating (or cooling) the sample and reference at a given rate while maintaining their temperatures the same by controlling the heating power and measuring the compensating heat flux that maintains the sample temperature within the specified program was carried out on a TGA/DSC1 thermogravimetric analyzer.

2.11 Dispersion analysis of materials.

The laser analyzer makes it possible to determine the particle size distribution over a wide range. For the dispersion analysis of additives, a SALD-7500 laser analyzer was used, the measurement range was from 7 nm to 800 µm.

3. Results and discussion

Earlier studies were carried out [29,30] on the effect of nano-silica on the strength characteristics of gypsum when it is introduced in an amount of 0.05; 0.1 and 0.5%, as a result of which it was found that the introduction of nanosilica due to its high dispersion in the amount of 0.5% is not effective.

To determine the optimal content of ultrasonicated nanosilica in the composite material, its percentage was varied from 0% to 0.1%, with a constant content of Portland cement. The results of the study of the physical and mechanical properties of the developed compositions of the composite material based on gypsum are shown in Figures 2, 3.

When assessing the strength of the material during early crystallization, the increase in strength reaches 110-140%. It should be noted that already on the 7th day the strength of the modified material is comparable to the strength of the control samples at the age of 28 days. Thus, the modification of gypsum with the developed complex additive makes it possible to obtain the effect of "dried stone". It should be noted that the increase in strength characteristics with an increase in the content of HC-2 in the composition of the complex additive is due to the fact that the dispersion of this additive in terms of distribution as a whole is somewhat shifted towards larger particles compared to additives of other selections [7,8].

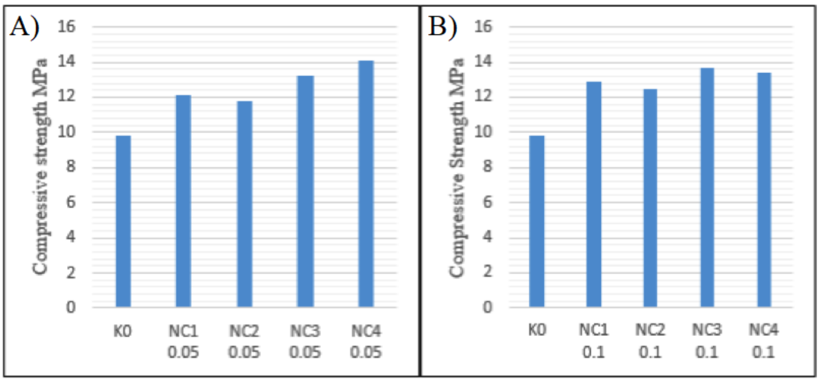
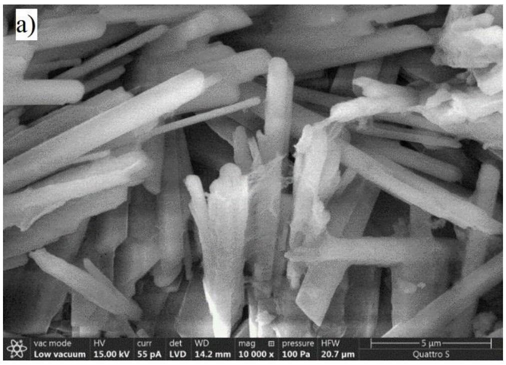


Fig. 3. Strength properties of a gypsum binder with the introduction of ultrasonic treated nanosilica and cement on the 28th day of hardening at a concentration of: A) 0.05%; B)0.1%

An analysis of the results obtained made it possible to establish that the optimal content of nanosilica in the composition of the composite material is from 0.05 to 0.1%. At the same time, the increase in the strength of the modified compositions on the 28th day compared to the control averages 30-40%,[7,8] depending on the average particle size in the suspension, which is probably due to the appearance of compaction zones in the structure of the modified gypsum stone. The results obtained in the course of physical and mechanical testing of samples indicate that the modification of a gypsum binder with a complex additive based on activated nanosilica is possible regardless of the initial dispersion of the deposited nano-silica [9,10].

The greatest increase in strength is observed with the introduction of nano-silica suspensions, which are characterized by high dispersion with an average particle size of 0.031 µm. In particular, samples containing suspensions of nanosilicas of the third and fourth selections showed high results in the increase in strength.



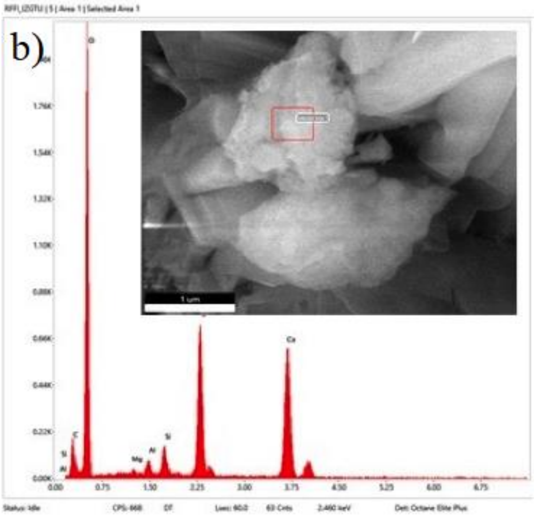


Fig. 4. Microstructure of the surface of a gypsum matrix modified with activated nanosilica and Portland cement: a) view at 10000-fold magnification; b) X-ray microanalysis of the surface of a gypsum matrix modified with nanosilica treated with ultrasound.

This confirms the previously put forward assumption that the more finely dispersed the nanocomponent of the modifier is, the greater the effect it has on the properties of the material, and the optimal effect is achieved when the modifier is introduced at low concentrations. An increase in the fineness of the modifier particles probably contributes to the formation of a larger volume of poorly soluble amorphous material uniformly distributed in the matrix structure, which improves the physico-mechanical parameters of the composite.

The formation of amorphous new growths in the composition of the modified gypsum stone can be indirectly confirmed by an increase in water resistance, which was determined in previous studies [29, 30], as well as an increase in the density of the material from 1.1 g/cm3 for the control composition to 1.2-1 .3 g/cm3, determined for the modified samples, and a change in the nature of the microstructure of the samples (Fig. 4).

An analysis of the microstructure of the modified gypsum matrix showed a compaction of the crystalline matrix, a change in the morphology of crystalline hydrates with the formation of blocks of differently oriented crystals interconnected by the products of the interaction of nano-silica and cement minerals. In the structure of the material, there is also a local intergrowth of gypsum individuals, which are covered over the entire surface with a gel-like material based on calcium hydrosilicates, which contributes to an overall increase in the strength and water resistance of the gypsum matrix which is also confirmed by the results of the microanalysis [11-13].

When analyzing the surface of the phase components of the material (Fig. 4b), on the spectrum in the structure of the modified gypsum matrix, in addition to the atoms of calcium, oxygen, and sulfur that form gypsum crystals, elements such as silicon, aluminum, and magnesium, which are part of calcium hydrosilicates and hydro aluminosilicates, were registered.

To establish the composition of the hydration products of the composite binder, physicochemical studies of samples of optimal compositions were carried out. The samples were studied on an IR-Fourier spectrometer IRAffinity-1 manufactured by Shimadzu (Japan) in the frequency range from 400 to 4000 cm-1.

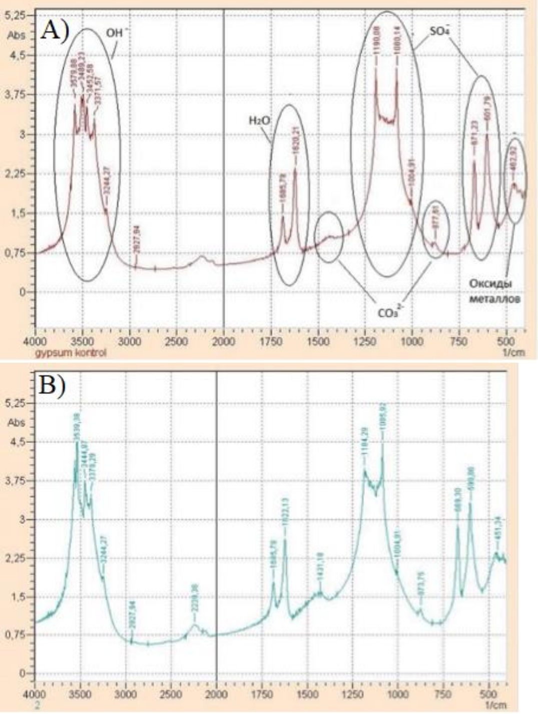


Fig. 5. IR spectra of the gypsum matrix: A) control composition; B) compositions with the introduction of 5% cement and 0.05% nanosilica after ultrasonic treatment

The spectrum of the modified sample shows a change in the nature of the peaks corresponding to the OH group and sulfates (SO4-). With the introduction of a complex additive, the peak of the OH group bifurcates and equalizes, as well as an increase in the intensity and expansion of the base of this peak, which indicates an increase in the hydroxides formed in the composition. When analyzing the peak corresponding to the sulfate group, one can note the appearance of additional peaks between the two main ones in the range of 1000-1200 cm^{-1} on the spectrum corresponding to the sample modified with a complex additive, which indicates the formation of a silicate group (O-Si-O) in the composition as well.

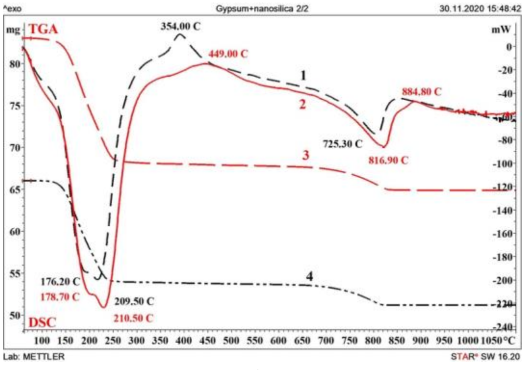


Fig. 6. TGA analysis of the gypsum matrix: 1, 4 control composition; 2, 3 compositions with the introduction of 5% cement and 0.05% nano-silica after activation by ultrasound

On the derivatograms of samples modified with cement and nanosilica, one can note changes in the nature of the peaks of temperature effects:

- in the temperature range of 150-220 °C, gypsum dehydrates. A characteristic feature of this peak is an increase in its intensity compared to the control spectrum, as well as a shift towards higher temperatures.

- in the temperature range of 350-400 °C, anhydrite recrystallizes, however, the introduction of additives leads to a shift in the effect and a decrease in intensity, which indicates a change in the composition of the hydration products of the material.

- in the temperature range of 800-900 °C, dissociation of calcium carbonates and sulfates is observed, and with the introduction of mineral additives, the mass loss increases, which indicates the dehydration reaction of calcium hydrosilicates.

- in the range from 850 °C to 950 °C, a mildly pronounced endoeffect is observed on the spectrum of the modified composition, which is probably caused by the recrystallization of calcium silicates.

Analysis of the data showed that the weight loss in the temperature range of 650–900°C with the addition of non-activated nanosilica as part of a complex additive is 6.6% [30], and with the activation of nanosilica by ultrasound it is 4.2%. The decrease in weight loss indicates the formation of more low-basic calcium hydrosilicates in the composition of the modified gypsum matrix.

4. Conclusions

The influence of a complex additive based on activated nanosilica and Portland cement on the structure formation of a gypsum matrix was analyzed. As a result, it was found:

1. The stability of suspensions obtained by sonication has been proven. At the same time, it was found that ultrasound makes it possible to average the parameters of suspensions and, regardless of the presence of imperfections in the technological line for the production of nanosilica, contributes to the production of an effective modifier.

2. Modification of the gypsum binder with a complex additive based on activated nanosilica improves the mechanical strength of the material, increases the mechanical strength depending on the percentage in the mineral matrix. The optimal range of nano-silica content was established, equal to 0.05 - 0.1%, while the increase in compressive strength reaches 30-40% compared to the control composition, water resistance increases to 0.47.

3. The improvement of the mechanical characteristics of the gypsum binder modified with a complex additive based on activated nanosilica is associated with the formation of new products based on calcium hydrosilicates, the formation of which is confirmed by the results of physicochemical methods of analysis, such as DSC, IR spectroscopy, and X-ray microanalysis.

4. The addition of nano-silica to gypsum-based materials improves their mechanical properties, enhance resistance to environmental factors and improving the overall energy efficiency of construction. This leads to a more sustainable construction process by saving energy and reducing greenhouse gas emissions and decrease in carbon footprint by reducing the amount of cement required in the construction process.

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