

FEATURES OF HARDENING AND UTILIZATION OF MODERN CEMENT COMPOSITIONS WITH NANOMODIFYING ADDITIVES FOR REPAIR AND RESTORATION WORKS

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Abstract

The paper presents the results of a study of the peculiarities of structure formation when adding complex silica-carbonate nanomodifying additives to Portland cement. The conducted research indicates that the modification of cement systems with complex organosilicate-carbonate additives leads to the directed formation of low-alkali hydro-silicate phases such as scawite. These phases not only contribute to strength synthesis but also influence the micro-reinforcement characteristics of the cement matrix and the rheological properties of concrete mixes. The obtained high-strength matrices based on Portland cement systems modified with these additives can serve as the foundation for restoration and repair plaster and concrete materials used in emergency and urgent conservation work on cultural heritage sites during times of military conflict.

Keywords: *Portland cement; Complex silica-carbonate; Nanomodifying additives; Microsilica; Nanocarbonate*

Introduction

Repair and restoration works encompass a complex set of actions aimed at the restoration and physical preservation of historical monuments while maintaining their artistic and historical significance. These activities encompass the restoration of architectural monuments, repair elements, conservation measures, as well as adapting structures for new uses. Such measures become particularly relevant during repair, emergency and urgent conservation efforts on cultural heritage sites that have suffered damage due to wartime activities.

In contrast to general construction and repair tasks where technical and economic characteristics of building materials and structures take precedence (such as quality, reliability, durability, modern design etc.), the restoration of historical and architectural landmarks emphasizes the preservation of distinctive elements, features, and characteristics of the objects. Furthermore, it involves ensuring the continued existence of these structures for future generations, reflecting the culture of the people and serving as tangible witnesses to history. This approach necessitates the use of predominantly authentic materials during restoration activities.

On the other hand, in compliance with the principles outlined in the Venice Charter for the Conservation and Restoration of Monuments and Sites, the use of modern advancements in

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the field of construction is permissible for strengthening monuments during their conservation and restoration. Various materials can be employed, but they must resemble, in appearance and properties, the materials from which the original structure was built.

One of the well-established principles of restoration, as documented in international architectural preservation documents and Ukrainian regulatory documents, is the principle of scientific rigor. This principle calls for the incorporation of contemporary scientific advancements in restoration processes and the ability to foresee the consequences of chosen technologies [1-4].

These principles allow for the replication, either partially or entirely, of the original appearance of partially or fully ruined structures, through the combination and interaction of new building materials, constructions, and technologies with authentic elements.

A critical step in carrying out repair, emergency, and urgent conservation work on cultural heritage objects, especially during times of conflict, could be the development of modern modified binder materials for plastering repairs and concrete mixes. These materials should exhibit long time set retardation, necessary viscosity, and the resulting artificial stone should demonstrate significant strength and durability. The use of Portland cement systems modified with complex organo-silica-carbonate nanomodifying additives appears promising.

It is known that the use of calcium carbonate positively influences the hydration processes of Portland cement, enhancing its early-age strength, microstructure, and durability [5]. Consequently, numerous studies have been conducted to elucidate the mechanisms by which calcium carbonate additives affect the properties of cement paste, mortar, and concrete [6, 7].

In 1938, Bessey *et al.* [5] first established the formation of calcium hydroxycarbonate during cement hydration when calcium carbonate was added. Similar results were obtained in subsequent studies [8].

Presently, it is acknowledged that the density and compactness of the cement matrix can be enhanced by introducing calcium carbonate as filler, with its particle size being comparable to or smaller than cement particles. Furthermore, it can act as a chemical reagent, accelerating hydration through nucleation effects [5, 9].

In [10], the influence of particle size of finely-ground limestone on cement hydration was investigated using three types of powders with varying degrees of dispersity.

Compressive and flexural strength decrease with an increase in the content of finely-ground limestone (in quantities exceeding the optimum) [10, 11]. Conversely, when higher amounts of carbonate additives are introduced, the water demand of the concrete mixture increases, resulting in a high effective water-cement ratio and reduced strength.

Given the above data, the scientific interest in utilizing nanoparticles with grain sizes less than 100 nm as carbonate additives becomes evident [12]. Nano-calcium carbonate, compared to micro-calcium carbonate, possesses smaller particle sizes and higher specific surface areas, thus potentially exerting a greater influence on hydration processes, workability of concrete mixtures, and the physical-mechanical properties and durability of cement composites. The impact of nano-calcium carbonate on cement hydration is contingent on its content, particle size, and crystal structure [13]. Authors examined the influence of nano-calcium carbonate particle content and size on cement hydration.

The crystalline structure of nano-calcium carbonate can also influence cement hydration processes. The impact of nano-calcium carbonate additives of calcite and aragonite composition on the properties of self-compacting concrete has been studied, revealing the greater effectiveness of calcite additives over aragonite [14]. The physico-mechanical properties of cement composites containing nano-carbonate additives primarily depend on their content, whereby strength

indicators initially increase with an optimal additive amount, but diminish with excessive nano-carbonate content [15].

Agglomeration of nano-carbonate particles is also possible, leading to the loss of both physical and chemical activity [16]. Furthermore, a denser matrix formed by the introduction of nano-carbonate may not produce an adequate amount of hydration products of the hydro-silicate type [15]. Hence, introducing micro-silica additives that can foster the formation of necessary quantities of hydro-silicate hydration products is reasonable.

The objective of this study is to investigate the peculiarities of structural formation during the addition of complex organo-silica-carbonate nanomodifying additives to Portland cement. The study aims to determine the optimal quantity of these additives for formation the strength of artificial stone.

Materials and methods

In the conducted research, the following raw materials were used: Portland cement OPC Type I 42.5R-N (according to DSTU B V.2.7-46:2010) produced by CRH company (PJSC "Podilsky Cement"), with a specific surface area of 390m²/kg by Blaine method; fine-dispersed active mineral additives based on nano-calcium carbonate in the form of powder and microsilica.

The mineralogical and chemical compositions of the Portland cement are presented in Table 1 and Table 2, respectively.

Table 1. Mineralogical Composition of Portland Cement Clinker

| Material | Content of Major Minerals, wt. % | | | |
|---|----------------------------------|----------------------------|-------------------------|---------------------------|
| | C ₃ S | C ₂ S | C ₃ A | C ₄ AF |
| Portlandcement clinker by "Podilsky Cement" | 62,46 (Bogue) (69 XRD) | 10,07 (Bogue) (5.5 XRD) | 7,4(Bogue) (XRD 5,2) | 11,3(Bogue) (10,4 XRD) |

For investigating the specific surface area of fine-dispersed active mineral additives, including finely ground microsilica of various types, a Blaine apparatus was employed. During the research, it was determined that products of the trade names Elkem Microsilica Grade 940-U possess a specific surface area $S = 24795\text{cm}^2/\text{g}$.

Table 2. Chemical Composition of Portland Cement Clinker

| Oxide content, mass % | | | | | | | | | |
|-----------------------|--------------------------------|--------------------------------|------------------|------|-------|-------------------|------------------|-----------------|-----------|
| SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | TiO ₂ | MgO | CaO | Na ₂ O | K ₂ O | SO ₃ | Free lime |
| 21,06 | 5,1 | 3,16 | 0,31 | 1,55 | 65,28 | 0,11 | 0,58 | 0,58 | 0,93 |

The phase composition of the raw components was assessed using X-ray diffraction analysis (XRD) (Fig. 1). The mineralogical composition of the Portland cement clinker is primarily dominated by tricalcium silicate 3CaO·SiO₂ ($d = 0.294; 0.274; 0.193; 0.177\text{nm}$), tricalcium aluminate 3CaO·Al₂O₃ ($d = 0.332; 0.304; 0.283; 0.155\text{nm}$), and dicalcium silicate 2CaO·SiO₂ ($d = 0.273; 0.260; 0.218; 0.162\text{nm}$).

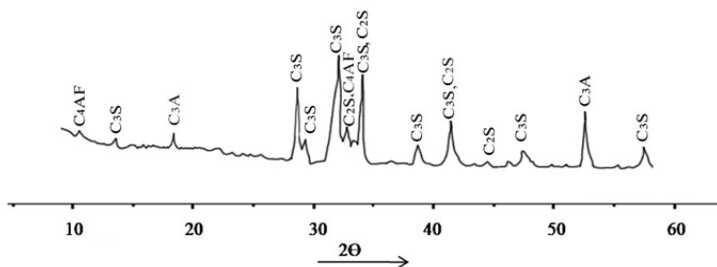


Fig. 1. X-ray diffractogram of OPC CEM I 42.5 R. Legend: C₃A – tricalcium aluminate; C₃S – tricalcium silicate; C₂S – dicalcium silicate; C₄AF – tetracalcium alumino-ferrite.

As the nanocarbonate additive, technical products from the Norwegian company "Nordcalk" in the form of the dispersion "Enrich C 50" were utilized. The key characteristics are presented in Table 3.

Table 3. Characteristics of "Nordcalk" Nanocarbonate Additive "Enrich C 50"

| Technical Parameter | Unit | Values |
|---------------------------------|-------------------|------------------|
| Solid Content | % | 50 |
| Average Density | g/cm ³ | 1.45 |
| pH in Dispersion | | 7...9 |
| External Appearance (at T=25°C) | | White Dispersion |
| Particle Size in Dispersion | | |
| d50% | nm | 130 |
| d90% | nm | 300 |

Identification of the nanocarbonate additive's composition is confirmed by the results of X-ray diffraction analysis (Fig. 2). The diffraction peaks of calcite CaCO₃ are clearly evident on the diffractogram (d=0.389; 0.303; 0.249; 0.227; 0.208; 0.191; 0.187; 0.162; 0.152; 0.144; 0.115nm). The chemical composition of the siliceous additive is presented in the table. 4.

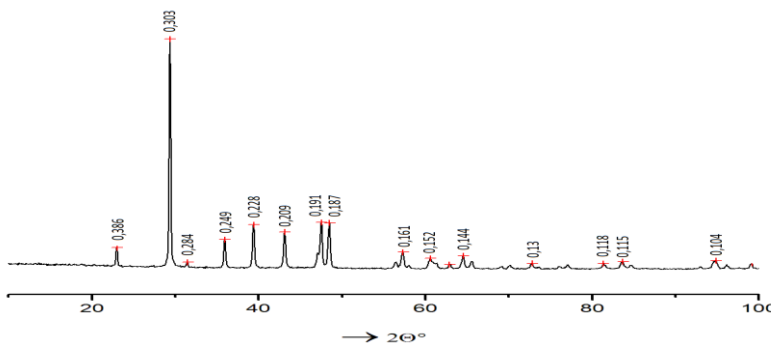


Fig. 2. X-Ray Diffractogram of the Nanocarbonate Additive/

Table 4. Chemical Composition of Silica Additive.

| Names of Micro-Mineral Additives | Oxides, (wt. %) | | | | | | LOI |
|----------------------------------|------------------|--------------------------------|-----|-----|-------------------|-------|------|
| | SiO ₂ | Al ₂ O ₃ | CaO | MgO | Na ₂ O | Other | |
| Microsilica Grade 940-U | 93,8 | - | - | - | 0,27 | 2,84 | 2,85 |

Photographs of the microstructure of carbonate additives of various degrees of dispersion are shown in Figure 3. Phase Composition of Silica Additives Determined by X-Ray Diffraction Analysis results are shown in Figure 4.

Based on X-Ray Diffraction Analysis (XRD) data, the microsilica "Grade 940-U" (Figure 4) contains an amorphous phase with microcrystalline inclusions of α -cristobalite ($d = 0.416; 0.315; 0.252; 0.199\text{nm}$) and residual β -quartz ($d = 0.335; 0.198; 0.154\text{nm}$).

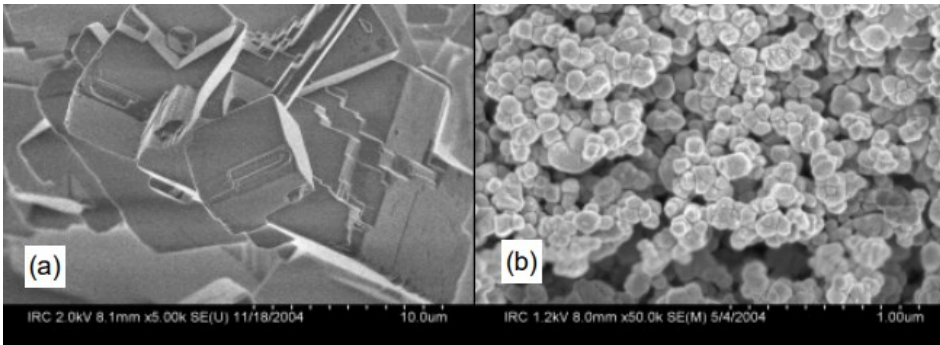


Fig. 3. SEM Microstructure Images of Original Additives: Micro- CaCO_3 (a) and Nano- CaCO_3 (b)

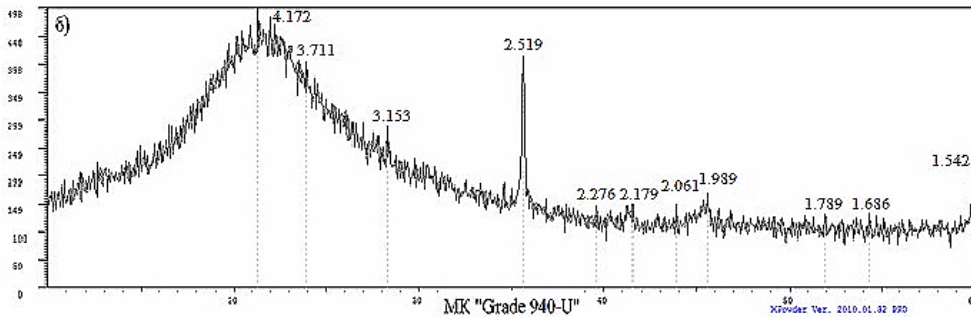


Fig. 4. X-Ray Diffractograms of Microsilica Additive "Grade 940-U"

With the aim of tailored structure development in concrete and enhancement of their physical and mechanical characteristics, various types and grades of superplasticizers based on polycarboxylates were utilized in the study.

Modification of the composition of cementitious composites was carried out using a 2-factor experimental design method. The dosage of the polycarboxylate-based superplasticizer was set at 1.25%. The ranges of variable factors are presented in Table 5.

Table 5. Ranges of Variation and Values of Variable Factors

| Types of Factors | Coded | Range of Variation | | | Variation Interval |
|-------------------------------------|-------|--------------------|------|------|--------------------|
| | | High | Mid | Low | |
| Nanocarbonate additive dosage, wt.% | X_1 | 4,5 | 3, 5 | 2, 5 | 1,0 |
| Silica Fume dosage, wt.% | X_2 | 15 | 10 | 5 | 5 |

The results of the experimental data are recorded in Table 6, according to which quadratic regression equations for the compressive strength of samples tested after 3, 7, 28 and 90 days of curing were obtained, and diagrams depicting the change in strength were constructed (Fig. 5). The regression equations for evaluating the compressive strength of samples after curing with the modification of the superplasticizer "MC PowerFlow 3100" and a comprehensive mineral additive that includes nano carbonate dispersion and microcrystalline silica are provided below, taking into account the regression coefficients:

$$\begin{aligned}
 fck(3 \text{ days}) &= 81,3 - 1,5 x_1 + 5,417 x_2 - 11 \cdot x_1^2 - 7,85 \cdot x_2^2 - 0,75 \cdot x_1 \cdot x_2; \\
 fck(7 \text{ days}) &= 74,12 - 0,65 \cdot x_1 + 7,183 \cdot x_2 - 4,283 \cdot x_1^2 + 2,517 \cdot x_2^2 - 0,025 \cdot x_1 \cdot x_2; \\
 fck(28 \text{ days}) &= 141,367 - 3,35 \cdot x_1 + 7,133 \cdot x_2 - 7,75 \cdot x_1^2 - 8,1 \cdot x_2^2 - 4,05 x_1 \cdot x_2; \\
 fck(90 \text{ days}) &= 144,7 - 2,933 \cdot x_1 + 5,083 x_2 - 5,6 \cdot x_1^2 - 6,95 \cdot x_2^2 - 3,45 \cdot x_1 \cdot x_2.
 \end{aligned}$$

Table 6. Experimental Design Matrix

| № | Types of variable factors | | | | Change in compressive strength of cement stone samples, MPa, after curing for days | | | |
|---|---------------------------|----------------|---------------------|-------------------------------|--|------|-------|-------|
| | Coded | | Dosage of SF, wt. % | Dosage of nanoadditive, wt. % | 3 | 7 | 28 | 90 |
| | X ₁ | X ₂ | | | | | | |
| 1 | 1 | 1 | 15 | 4,5 | 69,1 | 79,5 | 125,2 | 130,1 |
| 2 | 1 | 0 | 15 | 3,5 | 62,3 | 68,9 | 130,1 | 136,2 |
| 3 | 1 | -1 | 15 | 2,5 | 59,3 | 64,2 | 119,3 | 128,3 |
| 4 | 0 | 1 | 10 | 4,5 | 72,3 | 82,9 | 140,2 | 143,5 |
| 5 | 0 | 0 | 10 | 3,5 | 67,1 | 74,0 | 142,3 | 146,3 |
| 6 | 0 | -1 | 10 | 2,5 | 62,4 | 70,5 | 125,4 | 130,4 |
| 7 | -1 | 1 | 5 | 4,5 | 73,2 | 80,5 | 140,3 | 143,7 |
| 8 | -1 | 0 | 5 | 3,5 | 66,1 | 70,9 | 136,2 | 140,4 |
| 9 | -1 | -1 | 5 | 2,5 | 60,4 | 65,1 | 118,2 | 128,1 |

The study of the interaction processes between organic and mineral additives when simultaneously introduced into the composition of Portland cement was determined through the kinetics of plastic strength change over time or by the use of plastometry methods. The beginning and end of setting were determined using a Vicat apparatus. The method is based on determining the parameters of cone penetration under the influence of a constant load F, which provides a conditional rheological characteristic showing the relationship between the rate of dh/dt and the tangential stress Pm during shearing. This characteristic gradually decreases during penetration due to the increased contact area of the cone with the mixture.

The value of Pm is determined by the maximum penetration depth of the cone under the action of the load F. It is assumed that during cone penetration, there is movement of the mixture along the lateral surface of the cone. This condition is achieved in sufficiently plastic systems, hence the stress Pm during shearing, which induces this flow, is determined by the projection of the force F acting on the cone onto the tangent l of the cone, normalized by the contact area S between the cone and the medium (Fig. 5).

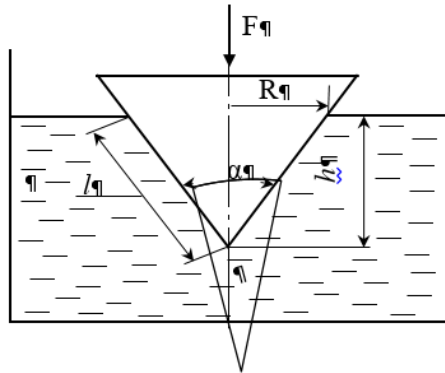


Fig. 5. Schematic representation of cone penetration under the action of load during the determination of plastic strength.

The shear stress limit is determined using the formula:

$$P_m = K_\alpha (F/h^2),$$

where K_α is the cone constant, dependent on the angle at its apex; F is the load on the cone in grams (g); h is the depth of cone penetration in centimeters (cm).

Results and Discussions

The obtained data indicate that at early strength gain times (3 and 7 days), the zone of optimal solutions for evaluating the compressive strength of artificial stone is achieved with a microsilica content of around 10% and the strength values increase proportionally with the nano-carbonate additive content (Fig. 6).

At later setting times (28 and 90 days), a change in the distribution of isoparametric curves occurs, and the zone of optimal compositions becomes more distinct (Fig 6). In other words, the maximum strength of the artificial stone is achieved with a microsilica content of around 10% and a nano-carbonate additive content of 3.5 to 4.5%.

As demonstrated by previous research [17, 18], the introduction of nano-carbonate additives not only promotes the nucleation effect but also leads to a reduction in the alkalinity of the hydro-silicate phases compared to compositions undergoing hydration in the presence of only polycarboxylate superplasticizer (Fig. 7a). According to X-ray phase analysis data after 28 days of curing, the newly formed compounds in the composition include portlandite ($d=0.493; 0.263; 0.193; 0.149$ nm), tobermorite 11.3\AA ($d = 0.307; 0.297; 0.228; 0.193; 0.176$ nm), $C_3A \cdot CaCO_3 \cdot 12 H_2O$ ($d = 0.493; 0.304; 0.273$ nm), as well as possible microcrystalline inclusions of scawtite $Ca_7(Si_6O_{18})(CO_3) 2 H_2O$ (Fig. 7a). Upon introducing a complex nano-additive with a carbonate-silicate composition, the rate of crystallization of the hydro-silicate phase changes, and the formation of more stable phases of hydro-carbosilicates, such as scawtite ($d = 0.303; 0.301; 0.277; 0.228; 0.207; 0.193; 0.163; 0.148$ nm), occurs (Fig. 7b). This phenomenon is expected to lead to full binding of free portlandite and the formation of artificial stone with stable operational characteristics, especially in later curing stages.

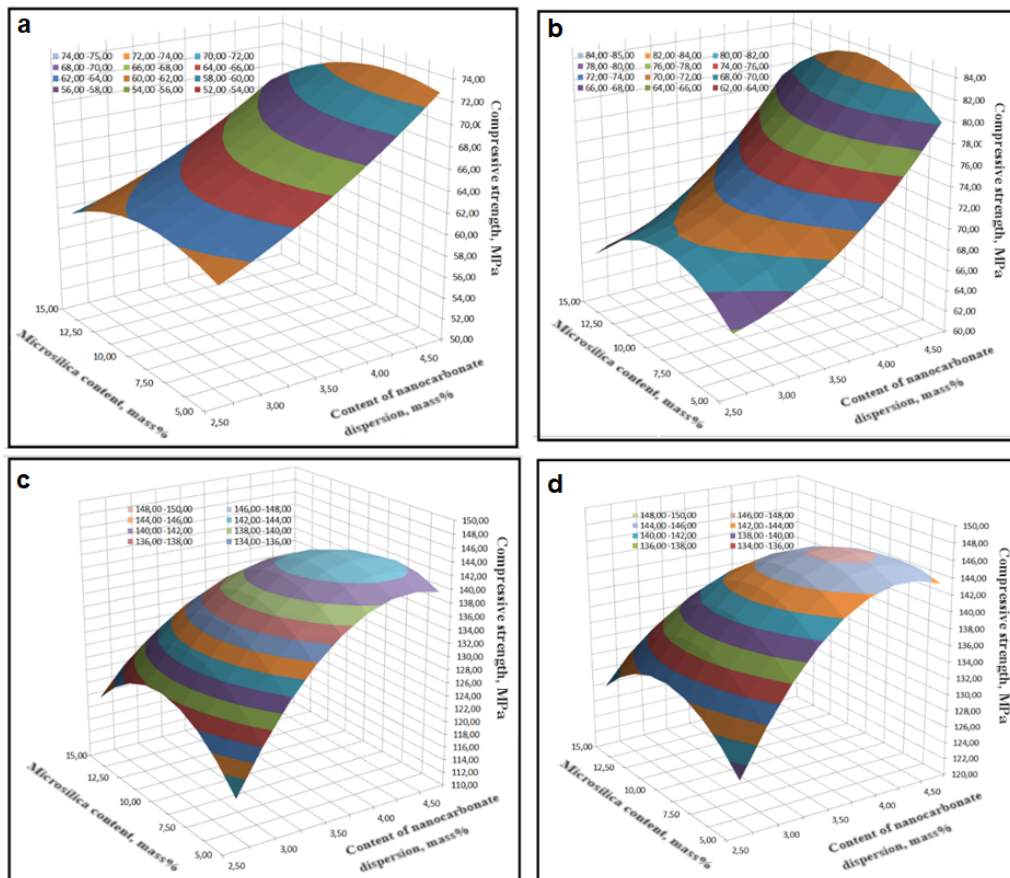


Fig. 6. Isoparametric diagrams showing the strength variation of cementitious compositions modified with polycarboxylate superplasticizer and a complex mineral additive containing microsilica and nano-carbonate dispersion after 3 days (a), 7 days (b), 28 days (c), and 90 days (d).

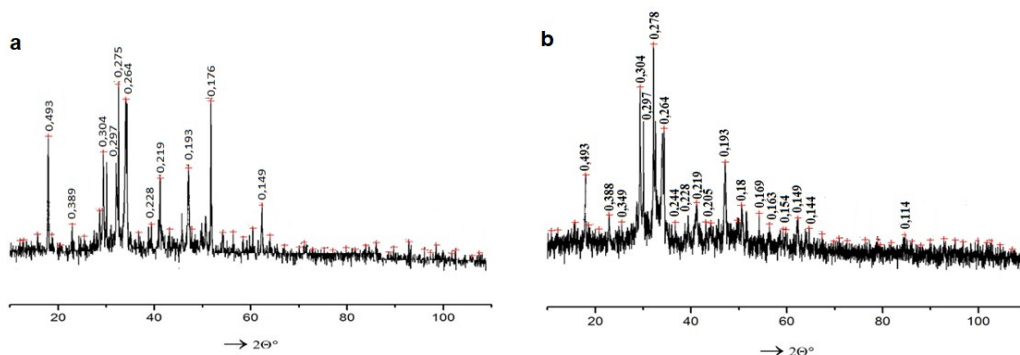


Fig. 7. X-ray diffraction pattern of hydration products after 28 days for compositions based on Portland cement, modified with nanocarbon additive (a), and complex nanocarbon-silicate additive (b) in the presence of polycarboxylate plasticizer

The change in the nature of nano-level structural formation processes and the manifestation of the nucleation effect explain the rapid strength development of the artificial stone, reaching 125...140MPa at 28 days and 130...146MPa at 90 days, which corresponds to an increase of 64...89% in strength compared to unmodified artificial stone.

Investigation of the microstructure of artificial stone (Fig. 8) based on Portland cement systems indicates that their modification with complex nanocarbon-silicate additives results in hydration products with a finer crystalline structure compared to systems modified only with nanocarbon additives.

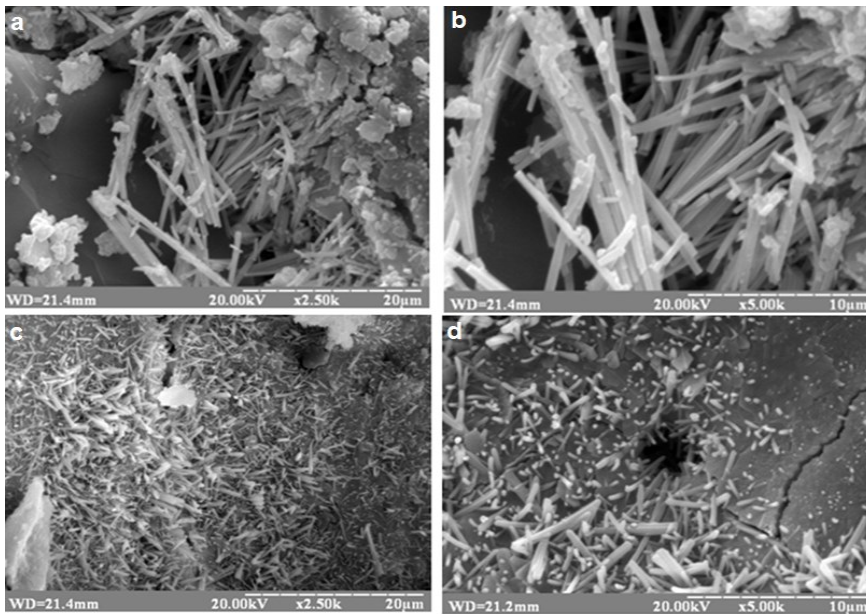


Fig. 8. Microstructure of artificial stone based on Portland cement, modified with nanocarbon dispersion (a, b), nanocarbon dispersion and microsilica additive (c, d), in the presence of polycarboxylate plasticizer after 28 days of curing; magnification x2500 (a, c); x5000 (b, d).

The change in the shape and size of newly formed substances contributes to the formation of a denser artificial stone with improved mechanical and special properties.

It should be noted that the introduction of microsilica into the investigated system: (Portland cement + plasticizer + nanocarbon additive) in an amount of 5...15% leads to not only the directed formation of hydrosilicate phases that contribute to strength synthesis, but also has a certain impact on the rheological properties of the concrete mixture.

The results of determining the influence of organo-carbonate and organo-carbonate-silicate additives on the change in the plastic strength of cement paste over time are presented in Table 7.

Analysis of the presented data allows us to indicate that by adjusting the proportion of carbonate and silicate components in the composition of the complex additive, it is possible to influence the rate of plastic strength development, and consequently, regulate both the rheological characteristics of materials based on such binding compositions and the rate of strength gain during the early stages of setting. This will have a significant impact on the operational characteristics of the resulting materials in the future.

Table 7. Determination of the influence of organo-carbonate and organo-carbonate-silicate additives on the change in plastic strength of cement paste over time

| Mix # | OPC Cem I 42.5, wt.% | Superplasticizer + additive | | Graphical interpretation of the dependence of plastic strength on the amount of additive |
|-------|----------------------|--|-------|--|
| | | Name | wt.,% | |
| 1 | 100 | - | - | |
| 2 | 100 | MC PF 3100 | 0,5 | |
| 3 | 100 | MC PF 3100 | 1 | |
| 4 | 100 | MC PF 3100 | 1,25 | |
| 5 | 100 | MC PF 3100 | 1,5 | |
| 6 | 95-85 | 1,25% MC PF 3100+ finely ground limestone | 5 | |
| 7 | | | 10 | |
| 8 | | | 15 | |
| 9 | | | 2,5 | |
| 10 | 95,5-97,5 | 1,25% MC PF 3100 + nanocarbo nate additi- ve | 3,5 | |
| 11 | | | 4,5 | |
| 12 | | | 5 | |
| 13 | 14 | 1,25% MC PF 3100+ 3,5% nanocarbo nate additi- ve + silica fume | 10 | |
| 14 | | | 15 | |

Conclusions

At the early stages of setting (3 and 7 days), the optimal usage of microsilica is around 10%, and the strength characteristics increase proportionally with the consumption of nanocarbo nate additive. In later setting stages (28 and 90 days), the maximum strength of artificial stone is achieved with a consumption of microsilica at -10% and nanocarbo nate additive at 3.5...4.5%.

The conducted research indicates that the modification of cement systems with complex organosilicate-carbonate additives leads to the directed formation of low-alkali hydro-silicate phases such as scawtite. These phases not only contribute to strength synthesis but also influence the micro-reinforcement characteristics of the cement matrix and the rheological properties of concrete mixes.

The obtained high-strength matrices based on Portland cement systems modified with these additives can serve as the foundation for restoration and repair plaster and concrete materials used in emergency and urgent conservation work on cultural heritage sites during times of military conflict. These materials are required to possess specific characteristics in terms of plastic strength and early strength development. The stability of the phase composition of the newly formed compounds in the developed compositions will ensure the preservation of operational properties of the artificial stone over time.

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