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# Study of the processes of formation of the structure and strength gain of nanomodified cement systems during long-term hardening

O. V. Artamonova, M. A. Shvedova<sup>™</sup>

Voronezh State Technical University, 20 letiya Oktyabrya str., 84, Voronezh 394006, Russian Federation

#### Abstract

The study investigated the influence of nanoscale admixtures on the structure formation and strength characteristics of cement systems during their hardening with a duration of up to ten years. The study of the processes of structure formation of modified cement systems showed that there is a significant acceleration of the process of cement hydration during the early period of hardening, despite the decrease in water content. It was established that phase transformations and changes in phase composition in all systems were observed throughout the entire studied hardening period. At the same time, in the later stages of hardening, the appearance of stable hydrate new formations (xonotlite, afwillite, ettringite), capable of creating a lower-dimensional, dense and homogeneous structure of nanomodified cement stone was observed. This provides nanomodified cement systems with high compressive strength values ( $R_{\rm com}$ ) both during the early and long stages of hardening. At the same time, the highest strength indicators throughout the entire study period ( $R_{\rm com}$  = 85 MPa, with a hardening duration of 28 days and  $R_{\rm com}$  = 157 MPa, with a hardening duration of 10 years) was characteristic for the cement system modified with a complex nanoscale admixture based on SiO<sub>2</sub> particles.

 $\textbf{Keywords:} \ Cement \ systems, SiO_2 \ nanoparticles, Carbon \ nanotubes, Structure \ formation, Long-term \ hardening, Compressive \ strength$ 

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Maria A. Shvedova, e-mail: marishwedowa@mail.ru © Artamonova O. V., Shvedova M. A., 2024



### The designations accepted in the article

C – cement;

W – water;

W/C – water-cement ratio;

CNA - complex nano-additive based on SiO<sub>2</sub> particles;

CNT – carbon nanotubes

### 1. Introduction

Increasing and regulating the long-term strength of cement composites is an urgent problem for domestic and foreign researchers due to the wide practical significance of these materials. Now, this is especially important due to the production and use of new generation cement composites that have high strength and uniformity, and low porosity. Such performance indicators are achieved by nano- and micromodification of the structure of cement composites with various chemical admixtures, which at the same time represent both a component of the cement matrix and a technological method for achieving the required properties. Moreover, the role of chemical admixtures in the technology of modern cement composites increases in accordance with the increase in their influence on the hydration processes and formation of the structure of cement stone. In [1], the authors suggest that the increase in the strength of cement systems over time can be described by a sawtooth curve, which is due to the process of hydrolysis along the Si-O-Si bonds (providing 50–60% of the strength [2]), as a result of which physico-chemical changes in the structure of cement stone changes will occur.

Previously, in the studies of domestic [3–6] and foreign scientists [7–10], it was established that the use of chemical admixtures with a plasticizing effect in the technology of cement composites for structural purposes is the most reasonable way to regulate their phase composition, microstructure, and strength properties. For example, in studies [3, 4] the influence of the method of administration and dosage of the superplasticizer C-3 (based on sulfonated naphthalene formaldehyde polycondensates) on the formation of longterm strength of cement stone for up to 18 years was investigated. It was established that the strength characteristics of the studied cement systems depend on the concentration of the superplasticizer and, in comparison with the reference unmodified system, can be both higher and lower. This is due to the fact that in a hardening cement paste, the simultaneous crystallization of new hydrate formations of various quantities and compositions from the  $(CaO)_x$ – $(SiO_2)_y$ – $(H_2O)_z$  solid solution of implementation, formed in the early stages of cement hydration, occurs.

It should be noted that now modifying admixtures for cement composites are complex, i.e. they contain not only a superplasticizer, but also various micro- and nanoadmixtures of inorganic nature [10–16]. At the same time, establishing the relationship between the parameters of the structural strength of cement composites with the technological dosing procedure and the optimal dosage of the admixture is the main task in the problem of modifying cement systems with modern complex admixtures.

In particular, our experimental studies [17–19], as well as the results obtained by other scientists [5–9], allowed to establish that complex admixtures based on nano-sized SiO<sub>2</sub>, as well as nanosized carbon and chrysotile tubes have a positive effect on the structure formation and strength characteristics of cement composites during the initial period of their hardening (up to 28 days).

Studies examining the influence of nanosized particles on the formation of the structure and strength characteristics of cement systems under long-term hardening are practically absent. However, this problem is relevant since it is assumed that at the later stages of hardening, chemically active nano-sized particles in the composition of cement composites can contribute to the occurrence of two differently directed processes - the evolution and self-organization of the structure of composites with a corresponding increase in their strength characteristics, or degradation of the structure, which may be associated with an increase in stresses and deformations over time, ultimately leading to the destruction of the material [20].

The purpose of this study was investigation of the processes of structure formation and strength gain of nanomodified hardened cement paste during its hardening duration of up to 10 years.

### 2. Experimental

The following ingredients were used as initial components for the creation of cement systems: ordinary Portland cement (C) CEM I 42.5 produced by JSC EUROCEMENT GROUP (Russian National State Standard 31108–2016); tap water (B) (Russian National State Standard 23732–2011); carbon nanotubes (CNT) of the fulleroid type Nanocyl-7000 (l = 700–3000 nm, d = 5–35 nm) obtained by chemical vapor deposition; complex nano-sized admixture (CNA), consisting of SiO<sub>2</sub> particles with medium diameter  $d_{av}$  ~ 10 nm and superplasticizer grade  $Sika^*ViscoCrete^*$  20 HE produced by LLC "Zika". The synthesis of CNA was carried out by the sol-gel method, described in detail in [17].

The mixtures were prepared by mixing Portland cement with a liquid mixer containing nano-sized admixtures. The water-cement ratio (W/C) for each system was selected based on the normal density of the cement paste. Three systems were obtained: reference C - W (W/C = 0.45), C - CNA (W/C = 0.27), C - CNT (W/C = 0.27). The mass fraction of nanosized particles of CNA and CNT required for modifying cement systems was determined earlier [18, 19] and amounted to 0.01% by weight of cement. The molded samples were placed in a normal hardening chamber (T = 21 °C, W = 100%), where they were kept for the required time (28 days, 1 year, 5 years, and 10 years).

A study of the degree of hydration  $(D_h)$  and phase composition of cement systems was carried out using a diffractometer *ARL X'TRA* (Cu $K_{\alpha}$  radiation,  $\lambda$  = 1.541788 Å) by powder X-ray diffractometry. Primary results were processed using the software package *PDWin* 4.0 [21].

Calculation of  $D_h$  values of cement systems were carried out according to ratio (1). For this, the content of the alite phase (3CaO·SiO<sub>2</sub> ( $C_3S$ )) in a sample of pure cement clinker and in the studied cement systems [22] were determined:

$$C_{\rm h}(C_3S) = \left(1 - \frac{I_{\rm mod}}{I_0}\right) \cdot 100\%,$$
 (1)

where  $I_{\rm mod}$  – intensity of the maximum phase peak  $C_3S$  (d=2.75 Å) in cement stone samples of different composition and hardening age;  $I_0$  – intensity of the maximum peak of  $C_3S$  phase in the original cement (d=2.75 Å).

Features of the microstructure of cement systems were determined by scanning electron microscopy (SEM) using *Phenom XL* microscope,  $v_{acc} = 15$  kV, P = 0.10 Pa) with a backscattered electron detector. A cement stone chip was used as a sample.

The study of the strength characteristics of cement systems was carried out using *INSTRON Sates* 1500HDS testing machine. For this, we destroyed a series (9–12 samples) of cube samples with the size of  $5\times5\times5$  cm. As a result of tests, it was established that the coefficient of variability within one series of samples was 7–10%.

### 3. Results and discussion

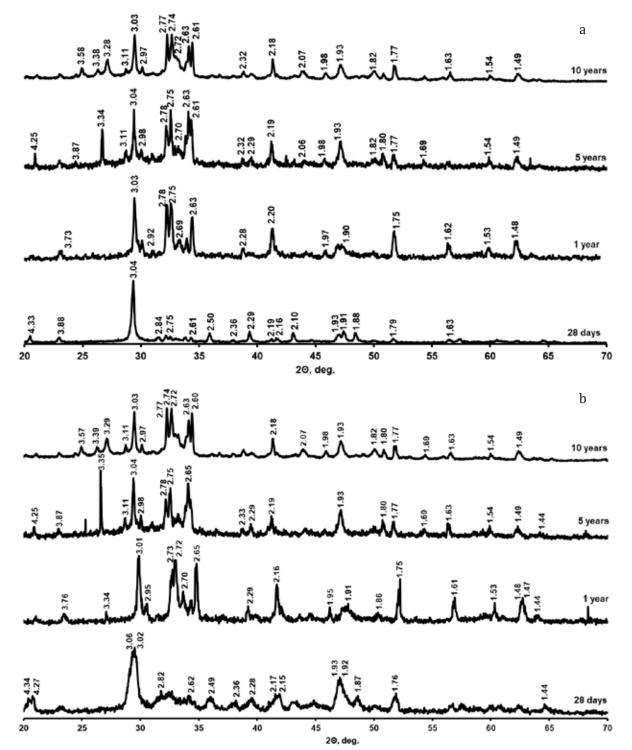
## 3.1. Processes of hydration and structure formation of cement systems

It was established that the intensity of cement hydration processes with the introduction of CNA and CNT increased significantly during the initial period of hardening (Figs. 1, 2). Already at the 28 days in nanomodified cement systems (C - CNA and C - CNT) the  $D_{\rm h}$  index amounted to ~90%. In the reference system without modifiers, such value was recorded only after 10 years.

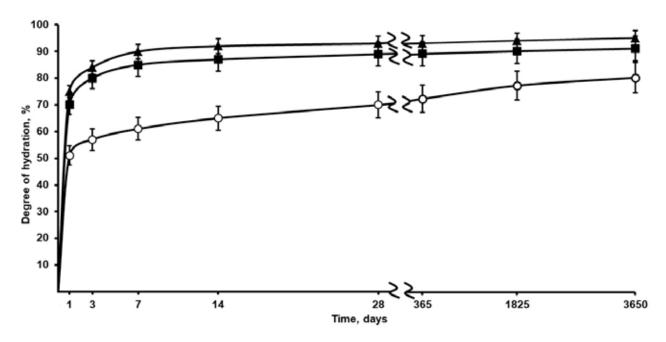
The phase composition of all studied systems (Fig. 1, Table 1) *after 28 days of hardening* was predominantly represented by phases of tobermorite-like (xCaO·SiO $_2$ ·zH $_2$ O), primary (CaO·SiO $_2$ ·H $_2$ O – CSH) and secondary (2CaO·SiO $_2$ ·H $_2$ O – C $_2SH$ ) calcium silicate hydrates.

At this stage of hardening, in addition to the indicated phases in the C-CNA system, an additional formation of a phase of highly basic calcium silicate hydrate  $(3\text{CaO}\cdot\text{SiO}_2\cdot2\text{H}_2\text{O})$  occurred, and in the reference system C – W the presence of a portlandite phase  $(\text{Ca}(\text{OH})_2)$  was revealed.

It can be assumed, that the heterogeneous process of formation of silicate hydrate phases in the reference and modified cement systems proceeds differently. During the initial hardening period of up to 28 days, congruent dissolution and hydration of the main clinker minerals occurs [18]. In this case, a xCaO·ySiO<sub>2</sub>·zH<sub>2</sub>O solid solution of implementation of variable composition is formed, which subsequently undergoes decomposition into several phases. In the reference system C – W, the solid solution of implementation decomposes mainly into



**Fig. 1.** X-Ray diffraction patterns of the nanomodified cement systems. Designated: a) C – CNA; b) C – CNT (CaO)  $_{x}$ SiO  $_{2}$ ·zH $_{2}$ O (d = 3.34; 3.05; 2.93; 2.80; 2.31; 1.83); CaO·SiO  $_{2}$ ·H $_{2}$ O (d = 4.24; 3.01; 2.78; 2.50; 2.23; 1.77); 2CaO·SiO  $_{2}$ ·H $_{2}$ O (d = 3.34; 3.01; 2.92; 2.25; 1.96; 1.81); 3CaO·SiO  $_{2}$ ·2H $_{2}$ O (d = 3.33; 3.04; 2.92; 2.84; 1.88; 1.77); 3(2CaO·SiO  $_{2}$ ·H $_{2}$ O (d = 3.07; 2.97; 2.72; 2.62; 2.22; 1.74); 2CaO·3SiO  $_{2}$ ·H $_{2}$ O (d = 4.24; 3.84; 3.36; 3.15; 2.85; 2.25); 3CaO·2SiO  $_{2}$ ·3H $_{2}$ O (d = 3.05, 2.74, 2.31, 2.21, 1.92; 1.68); 2CaO·SiO  $_{2}$ ·0.5H $_{2}$ O (d = 2.99; 2.77; 2.67; 2.25; 1.80; 1.61); 6CaO·6SiO  $_{2}$ ·2H $_{2}$ O (d = 3.65; 3.23; 3.07; 2.83; 2.04; 1.95); CaO·Al  $_{2}$ O  $_{3}$ ·8.5H $_{2}$ O (d = 3.57; 3.13; 2.75; 2.58; 2.34; 1.79); 4CaO·Al  $_{2}$ O  $_{3}$ ·19H $_{2}$ O (d = 2.88; 2.78; 2.67; 2.35; 1.93; 1.82); 3CaO·Al  $_{2}$ O  $_{3}$ ·SiO  $_{2}$ ·4H $_{2}$ O (d = 3.07; 2.74; 2.24; 1.99; 1.70; 1.64); CaO·Al  $_{2}$ O  $_{3}$ ·2SiO  $_{2}$ ·4H $_{2}$ O (d = 3.34; 3.19; 2.74; 2.70; 2.66; 1.81); 3CaO·Fe  $_{2}$ O  $_{3}$ ·CaSO  $_{4}$ ·16H $_{2}$ O (d = 3.40; 2.93; 2.55); 3CaO·Al  $_{2}$ O  $_{3}$ ·3CaSO  $_{4}$ ·32H $_{2}$ O (d = 4.90, 3.02, 2.79, 1.62, 1.54; 1.50); Ca(OH)  $_{2}$ (d = 3.11; 2.63; 1.93; 1.79; 1.69; 1.49) [19]



**Fig. 2.** Kinetics of hydration of cement systems with a duration of their hardening of up to 10 years. Designated: o - C - W;  $\triangle - C - CNA$ ;  $\blacksquare - C - CNT$ 

**Table 1.** Phase composition and morphology of cement systems with a hardening duration of up to 10 years

	Phase composition of system / morphology of crystals						
System	28 days	1 year	5 years	10 years			
$\begin{array}{c} C-W \\ (W/C=0.45) \end{array}$	xCaO·SiO <sub>2</sub> ·zH2O / Plate 2CaO·SiO <sub>2</sub> ·H <sub>2</sub> O / Plate CaO·SiO <sub>2</sub> ·H <sub>2</sub> O / Needle Ca(OH) <sub>2</sub> / Plate- prismatic	$CaO \cdot SiO_2 \cdot H_2O / Needle$ $xCaO \cdot SiO_2 \cdot zH_2O / Plate$ $2CaO \cdot SiO_2 \cdot H_2O / Plate$ $Ca(OH)_2 / Plate$ prismatic	2CaO·SiO <sub>2</sub> ·H <sub>2</sub> O / Plate (CaO) <sub>x</sub> ·SiO <sub>2</sub> ·zH <sub>2</sub> O / Plate CaO·Al <sub>2</sub> O <sub>3</sub> ·8.5H <sub>2</sub> O / Hexagonal plate Ca(OH) <sub>2</sub> / Plate- prismatic	2CaO·SiO <sub>2</sub> ·0.5H <sub>2</sub> O / Plate 3CaO·Al <sub>2</sub> O <sub>3</sub> ·SiO <sub>2</sub> ·4H <sub>2</sub> O / Hexagonal plate 3CaO·Fe <sub>2</sub> O <sub>3</sub> ·CaSO <sub>2</sub> ·16H <sub>2</sub> O / Hexagonal plate Ca(OH) <sub>2</sub> / Plate-prismatic			
C - CNA $(W/C = 0.27)$	xCaO·SiO <sub>2</sub> ·zH <sub>2</sub> O / Plate 3CaO·SiO <sub>2</sub> ·2H <sub>2</sub> O / Hexagonal plate CaO·SiO <sub>2</sub> ·H <sub>2</sub> O / Needle	CaO·SiO <sub>2</sub> ·H <sub>2</sub> O / Needle 4CaO·Al <sub>2</sub> O <sub>3</sub> ·19H <sub>2</sub> O / Hexagonal plate 3(2CaO·SiO <sub>2</sub> )·2H <sub>2</sub> O / Hexagonal plate	CaO·SiO <sub>2</sub> ·H <sub>2</sub> O / Needle CaO·Al <sub>2</sub> O <sub>3</sub> ·2SiO <sub>2</sub> ·4H <sub>2</sub> O / Hexagonal plate 2CaO·3SiO <sub>2</sub> ·H <sub>2</sub> O / Hexagonal plate	6CaO·6SiO₂·H₂O / Fibrous and Needle 3CaO·2SiO₂·3H₂O / Fibrous and Needle 2CaO·SiO₂·H₂O / Plate			
C - CNT $(W/C = 0.27)$	$x$ CaO·SiO $_2$ · $z$ H $_2$ O / Plate 2CaO·SiO $_2$ ·H $_2$ O / Plate CaO·SiO $_2$ ·H $_2$ O / Needle	CaO·SiO <sub>2</sub> ·H <sub>2</sub> O / Needle 3CaO·Al <sub>2</sub> O <sub>3</sub> ·SiO <sub>2</sub> ·4H <sub>2</sub> O / Hexagonal plate 3(2CaO·SiO <sub>2</sub> )·2H <sub>2</sub> O / Hexagonal plate	CaO·SiO <sub>2</sub> ·H <sub>2</sub> O / Needle 4CaO·Al <sub>2</sub> O <sub>2</sub> ·19H <sub>2</sub> O / Hexagonal plate 3CaO·2SiO <sub>2</sub> ·3H <sub>2</sub> O / Fibrous and Needle	6CaO·6SiO <sub>2</sub> ·H <sub>2</sub> O / Fibrous and Needle 3CaO·Al <sub>2</sub> O <sub>3</sub> ·3CaSO <sub>4</sub> ·32H <sub>2</sub> O / Fibrous and Needle 2CaO·SiO <sub>2</sub> ·0.5H <sub>2</sub> O / Plate			

tobermorite and the  $C_2SH$  phase, while in modified systems, under the influence of active nano-sized particles, the formation of CSH calcium silicate hydrate phases with x = 0.8-1.5 predominantly occurs.

It should be noted that over 10 years, phase transformations and changes in phase composition have been observed in all systems.

After 1 year of hardening the phase composition of the reference unmodified system practically did not change, but in nanomodified systems it changed significantly: the tobermorite-like phase transformed into highly basic calcium silicate hydrate (3(2CaO·SiO\_2)·2H\_2O); at the same time, a phase of highly basic hydrated calcium aluminate (4CaO·Al\_2O\_3·19H\_2O) is formed in the system with the addition of CNA , and the phase of calcium aluminosilicate hydrate (3CaO·Al\_2O\_3·SiO\_2·4H\_2O) is formed in the system with the addition of CNT.

After 5 years of hardening in the reference system, low-basic hydrated calcium aluminate (CaO·Al<sub>2</sub>O<sub>3</sub>·8.5H<sub>2</sub>O) starts to form, which was typical for 1 year of hardening in nanomodified systems. An increased content of low-basic calcium aluminosilicate hydrates (CaO·Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>·4H<sub>2</sub>O) and calcium silicate hydrates (2CaO·3SiO<sub>2</sub>·H<sub>2</sub>O) was observed in the modified C – CNA system. This may be due to the introduction of nanosized SiO<sub>2</sub> particles into the system, which are related in their crystal chemical structure to the minerals of cement stone, and are capable of chemical interaction with clinker minerals of cement, which leads to a decrease in the basicity of the resulting silicate hydrate phases.

For the C – CNT system, on the contrary, the formation of stable highly basic hydrated calcium aluminate (4CaO·Al<sub>2</sub>O<sub>3</sub>·19H<sub>2</sub>O) and calcium silicate hydrate (3CaO·2SiO<sub>2</sub>·3H<sub>2</sub>O) was observed.

After 10 years of hardening in the reference system, highly basic silicate hydrates and calciumaluminosilicate hydrates start to form, as well as the low-sulphate form of calcium hydrosulfoferrite ( $3\text{CaO}\cdot\text{Fe}_2\text{O}_3\cdot\text{CaSO}_4\cdot16\text{H}_2\text{O}$ ).

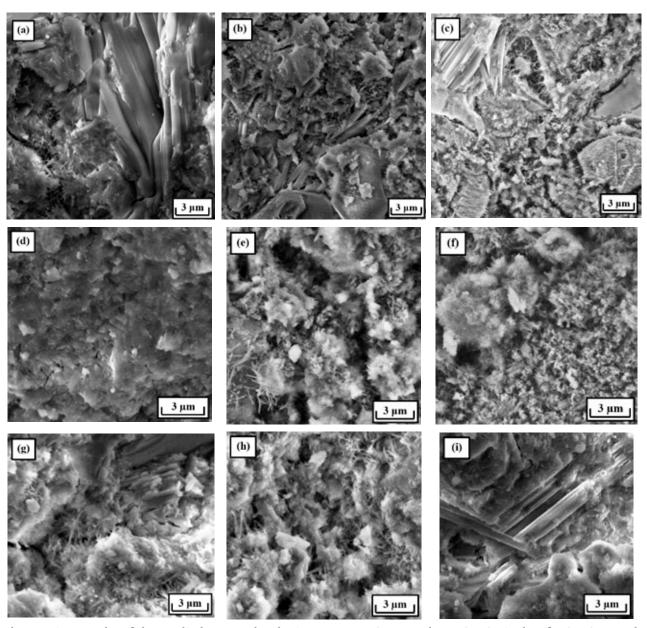
It is worth noting that in the reference system C – W, the presence of the portlandite phase was recorded throughout the entire hardening process, in contrast to modified systems in which this phase was absent.

Thus, the phase composition of nanomodified systems during prolonged hardening was characterized by the formation of stable xonotlite minerals (6CaO·6SiO<sub>2</sub>·H<sub>2</sub>O), while in the C – CNA system, afwillite was additionally formed (3CaO·2SiO<sub>2</sub>·3H<sub>2</sub>O), and in the system C – CNT, ettringite (3CaO·Al<sub>2</sub>O<sub>3</sub>·3CaSO<sub>4</sub>·32H<sub>2</sub>O) was formed. The data obtained are consistent with the results presented in [3].

The X-ray phase analysis data correlate well with the SEM results (Fig. 3). It was established that the morphology of particles of unmodified and nanomodified hardened cement paste differs throughout the entire studied hardening period. In the reference system C – W, with a hardening duration of 28 days (Fig. 3a), a heterogeneous structure of cement stone was formed, consisting mainly of an amorphous-crystalline silicate hydrate gel and large lamellar crystals of portlandite.

In nanomodified systems, during a similar period of hardening (Fig. 3d, g), the formation of nanosized crystallites with a small grain size occurs, forming a more uniform structure. At the same time, in the studied systems, the presence of a weakly crystallized gel, characteristic of primary low-basic calcium silicate hydrates, as well as fiber and needle-like new formations, probably formed by secondary high-basic calcium silicate hydrates was observed. It should be noted that crystallites form a large number of adjacent and intergrowth contacts with each other, which allows nanomodified systems to achieve high-performance  $R_{\rm com}$  already in the early stages of hardening.

With a hardening duration of 5 years, the reference system (Fig. 3b) was characterized by a heterogeneous morphology of the cement stone, with predominantly lamellar crystals of different sizes; in the modified systems C – CNA and C – CNT (Fig. 3e, h), a predominantly fiber-plate and fiber-needle morphology of the resulting crystals was observed, respectively. After 10 years (Fig. 3f, i), the structure of the nanomodified cement stone can be characterized as dense, formed by crystallites of a predominantly fiber and lamellar structure, forming adjacent and intergrowth contacts with each other and other hydrate new formations.



**Fig. 3.** Micrographs of the studied cement hardening systems: C – W (a, b, c); C – CNA (d, e, f); C – CNT (g, h, i) at different curing times (28 days – a, d, g; 5 years – b, e, h; 10 years – c, f, i)

### 3.2. Strength characteristics of cement systems

The results of determining the compressive strength of the studied cement systems are presented in Table 2. It was found that after 28 days the  $R_{\rm com}$  in the C – CNA system reached 85 MPa, and in the C – CNT system it was 78 MPa. A similar value (80 MPa) in the reference system without admixtures was achieved only after 10 years of hardening. Subsequently, during the entire hardening period for cement systems, a gradual increase in strength characteristics

was observed. Maximum value of the  $R_{\rm com}$  was achieved after 10 years in the C – CNA system and composed 157 MPa.

The values of age-strength relation over time  $(\beta_t)$ , which was calculated as the ratio of the actual compressive strength  $(R_t)$  of cement stone at a given age t to compressive strength  $(R_{28})$  at 28 days are shown in Table 2. It should be noted that higher  $\beta_t$  values were obtained for cement systems with CNA and CNT admixtures compared to the reference system throughout the entire studied hardening period.

Table 2. Kinetics of strength of cement systems with a duration of their hardening up to 10 years

System	W/C	Compressive strength ( $R_{\text{com}}$ ), MPa / Coefficient of strength increase ( $\beta_t$ ), rel. units.			
		28 days	1 year	5 years	10 years
C – W	0.45	53 / 1	68 / 1.28	74 / 1.39	80 / 1.51
C – CNA	0.27	85 / 1	111 / 1.31	132 / 1.55	157 / 1.85
C – CNT	0.27	78 / 1	103 / 1.32	112 / 1.44	121 / 1.55

#### 4. Conclusions

The positive effect of nanoscale admixtures on the processes of hydration and structure formation of cement systems with a hardening duration of up to 10 years has been established. Research results show that in nanomodified cement systems high degrees of hydration are achieved both during the early and long-term stages of hardening. In this case, the formation of stable hydrate new growths of various compositions, crystallizing mainly in the form of fibers and plates, characterized by a large number of adjacent and intergrowth contacts between themselves and other hydrate new growths occurs.

It was established that, in comparison with the reference system, cement systems with admixtures of nanosized particles were characterized by high values of compressive strength throughout the entire studied hardening time. The highest  $R_{\rm com}$  both for 28th day of hardening (85 MPa) and with a hardening duration of 10 years (157 MPa) was characteristic for the cement system modified by CNA based on SiO<sub>2</sub> particles.

### **Author contributions**

Artamonova O. V. – scientific leadership, research concept, methodology development, text writing, final conclusions.

Shvedova M. A. – conducting research, review writing and editing.

### **Conflict of interests**

The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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  - \* Translated by author of the article

### Information about the authors

*Olga V. Artamonova*, Dr. Sci. (Tech.), Associate Professor, Professor at the Department of Chemistry and Chemical Technology of Materials, Voronezh State Technical University (Voronezh, Russian Federation).

https://orcid.org/0000-0001-9157-527X ol\_artam@rambler.ru

Maria A. Shvedova, Cand. Sci. (Tech.), Research Fellow at the Higher School of Building Materials Science (Academy of Building Complex Development), Voronezh State Technical University (Voronezh, Russian Federation).

https://orcid.org/0000-0002-6484-8719 marishwedowa@mail.ru

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