Наносистеми, наноматеріали, нанотехнології
Nanosistemi, Nanomateriali, Nanotehnologii
2025, т. 23, № 2, сс. 379–392
https://doi.org/10.15407/nnn.23.02.0379

PACS numbers: 61.46.-w, 61.48.De, 65.80.-g, 66.20.Ej, 83.80.Hj, 83.80.Nb, 91.60.-x

Research on the Influence of Nanoadditives on the Thermodynamic and Hydrodynamic Stability of the Solidification Structure of the Ettringite Phase and Its Composition with Calcium Sulphates

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The paper considers influence of various factors on the stability of the ettringite structure of two forms of formation: *a*—on the base of pure minerals $C_3A + C\acute{S}H_2$, *b*—a mixture of alumina cement (AC-400) and gypsum plaster (G5); besides, a model of hydration process using the example of calcium sulphate hemihydrate is presented. Factors affecting stability of ettringite depending on changes in humidity and pH of the environment, temperature, and operating conditions are established. It is confirmed the hypothesis of the effect caused by the ratio $G = f(CaO/Al_2O_3)$ in the minerals of the $CaO–Al_2O_3–H_2O$ system on the Gibbs surface energy: for $CA_2 = 0.27$, $\Delta G = 24.70$; for $CA = 0.54$, $\Delta G = 51.86$; for $C_{12}A_7 = 0.8$, $\Delta G = 141.00$, for $C_3A = 1.63$, $\Delta G = 145$. It is also determined the dependence of the order of formation of hydrate compounds during hydration of aluminate and sulphoaluminate cements on the Gibbs surface energy $\Delta G = f(\mathrm{Da})$ and the ratio $\Delta G = f(CaO/Al_2O_3)$ for the minerals of the system by means of the average Gibbs energy (for CA_2, 24.70; for CA, 51.86; for $C_{12}A_7$, 141.00; for C_3A, 145) of the system $CaO–Al_2O_3–H_2O$: C_2AH_8, C_4AH_{13}, C_4AH_{10}, CAH_{10}, $Al(OH)_3$. Change in the heat of hydration of pure clinker minerals over time and change in their degree of hydration depend on the value of the coefficients. For example, the coefficient K (mCaO/nAl$_2$O$_3$ ratio) is the largest for C_3A mineral, *i.e.*, 1.63, and the heat of hydration is correspondingly the highest for C_3A mineral and is of 873 J/g on the 28th day; therefore, accordingly, C_3A mineral will enter the hydration reaction as the fastest. An increase in temperature is detected for artificially formed ettringite in the $C_3A + C\acute{S}H_2$ system followed by $CA + C\acute{S}H_2$ and $CA_2 + C\acute{S}H_2$ that confirms the main idea of the experiments.

Stabilization of the ettringite phase is carried out by means of the nanomodification with carbon nanotubes (CNTs), taurite, and silicon dioxide. Results of investigations performed confirm a 4–5-fold increase in strength indicators, a change in the softening factor depending on the type of nanomodifier and systems with different plasticizers and nanotubes.

В роботі розглянуто вплив різних чинників на стабільність структури еттрингіту двох форм утворення: *а*) на основі чистих мінералів $C_3A + C\acute{S}H_2$, *б*) суміші глиноземистого цементу (ГЦ-400) і гіпсу будівельного (Г5); також представлено модель процесу гідратації на прикладі напівводяного сульфату Кальцію. Встановлено чинники впливу на стабільність еттрингіту від зміни вологости та pH середовища, температури, умов експлуатації. Підтверджено гіпотезу впливу співвідношення $G = f(CaO/Al_2O_3)$ в мінералах системи $CaO–Al_2O_3$ на Ґіббсову поверхневу енергію: $CA_2 = 0{,}27$ — $\Delta G = 24{,}70$, $CA = 0{,}54$ — $\Delta G = 51{,}86$, $C_{12}A_7 = 0{,}8$ — $\Delta G = 141{,}00$, $C_3\mathrm{A} = 1{,}63$ — $\Delta G = 145$; визначено залежність порядку формування гідратних сполук за гідратації алюмінатних і сульфоалюмінатних цементів від Ґіббсової поверхневої енергії $\Delta G = f$(а.о.м.) і співвідношення $\Delta G = f(CaO/Al_2O_3)$ для мінералів системи за середньою Ґіббсовою енергією: CA_2 — 24,70, CA — 51,86, $C_{12}A_7$ — 141,00, C_3A — 145 системи $CaO–Al_2O_3–H_2O$: C_2AH_8, C_4AH_{13}, C_4AH_{10}, CAH_{10}, $Al(OH)_3$. Зміна теплоти гідратації чистих клінкерних мінералів у часі та зміна їхнього ступеня гідратації залежать від величини коефіцієнтів. Наприклад, коефіцієнт K (співвідношення mCaO/nAl$_2$O$_3$) є найбільшим для мінералу C_3A — 1,63, а теплота гідратації, відповідно, найвища у мінералу C_3A і становить на 28 добу 873 Дж/г; тому, відповідно, мінерал C_3A буде найшвидше вступати в реакцію гідратації. Виявлено підвищення температури для штучно утвореного еттрингіту в системі $C_3A + C\acute{S}H_2$, далі $CA + C\acute{S}H_2$ і $CA_2 + C\acute{S}H_2$, що підтверджує основну ідею дослідів. Проведено стабілізацію еттрингітової фази за рахунок наномодифікування вуглецевими нанотрубками (ВНТ), тауритом, діоксидом Силіцію. Наведені результати досліджень підтверджують збільшення в 4–5 разів міцнісних показників, зміну коефіцієнта розм’якшення в залежності від виду наномодифікатора та систем з різними пластифікаторами і нанотрубками.

Key words: nanomodifying, alumina cement, carbon nanotubes, hydration, nanosystem, solidified structure.

Ключові слова: наномодифікування, глиноземистий цемент, вуглецеві нанотрубки, гідратація, наносистема, затвердла структура.

(Received 20 June, 2024; in revised form, 26 June, 2025)

1. PROBLEM STATEMENT

The need for the production of the latest building materials for the repair and construction of high-quality buildings is constantly increasing and requires a solution [1–4].

The rapid pace of construction of buildings and structures requires rapid dismantling of monolithic structures, increasing the reversibility of forms during the factory production of building structures. In monolithic construction, such technologies are possible when using fast-hardening binders, which can gain up to 90–100% strength after 1–3 days. This also applies to the emergence of 3*D*-construction technologies.

In addition, during the war and post-war years, the restoration of construction objects, thermal units of metallurgical plants as well as production of building materials and reconstruction of existing objects are possible only within short periods of time, and such processes may include the use of composites based on alumina cement and gypsum, which, in the early hardening periods, have high indicators of strength properties.

In this regard, a particular attention should be paid to aluminate, sulphate and sulphoaluminate binders [3–6].

One of the problems of their use consists in recrystallization of sulphoaluminates during hardening and insufficient stabilization of the ettringite phase during operation [3, 5].

Therefore, studies of the structure and durability of the ettringite phase and its composition with calcium sulphates are relevant.

2. RESULTS AND DISCUSSION

Based on the analysis of literary sources of Ukrainian and foreign scientists [1–3], it was concluded that there is a need to solve the problem of developing compositions of composite binders based on the $CaO–Al_2O_3–SO_3$ system, surfactants with high structure density and strength indicators by means of stabilizing the state of the ettringite component. This will contribute to the intensification of the construction period and to the formation of a durable concrete strong structure.

After conducting an analysis of possible directions for formation of the concrete structure, a hypothesis was proposed about the possibility of:

- stabilization of the ettringite phase by means of modification with nanoadditives; in order to solve this problem, it is necessary to determine the conditions under which the ettringite phase goes into an unstable state;
- the technology of introducing nanoadditives and studying solutions with the maximum amount of ettringite for the stability of their structure and mineral composition of the $CaO–Al_2O_3–SO_3–H_2O$ system.

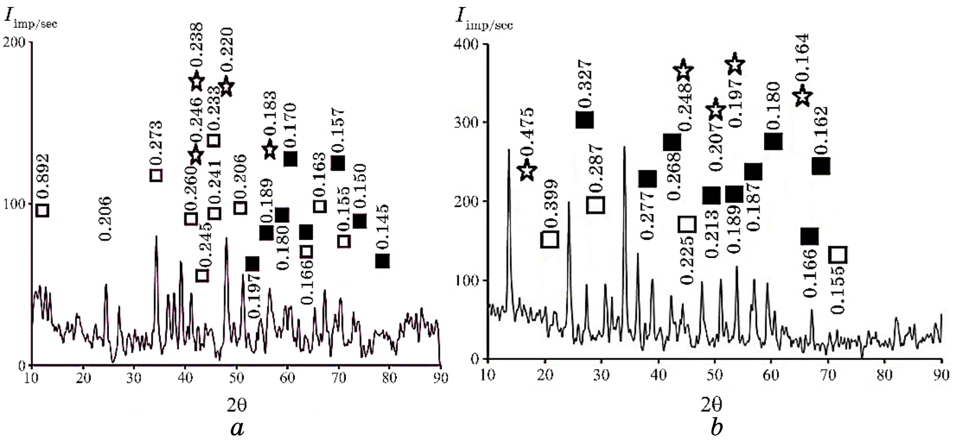


Fig. 1. X-ray diffractograms of samples made of the composition C_3A–$C\dot{S}H_2$ on the 3rd day of hardening (*a*) and on the 28th day of hardening (*b*); ■—ettringite $3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}31H_2O$; □—ettringite $3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}12H_2O$; ★—tetracalcium nineteen-hydroaluminate $3CaO{\cdot}Al_2O_3{\cdot}19H_2O$.

The cited results of research into the hydration processes of the pure C_3A–$C\dot{S}H_2$ system showed that three periods of formation are visible in the life cycle of ettringite: primary ettringite (hardening, Fig. 1, *a*), secondary ettringite (Fig. 1, *b*), and recrystallization during operation.

Thus, in the C_3A–$C\dot{S}H_2$ system (Fig. 1, *a*), after 3 days of hardening, the main lines of hydrated ettringite phases appear ($d/n = 0.892$, 0.273, 0.260, 0.245, 0.241, 0.233, 0.197, 0.189, 0.180, 0.170, 0.166, 0.163, 0.150, 0.145 nm), $4CaO{\cdot}13H_2O$ ($d/n = 0.246$, 0.220, 0.183 nm) (Fig. 1).

After 28 days of hardening, there is an increase in the intensity of the main interplanar distances and hydrated phases $C_3AC_3H_{32}$ ($d/n = 0.475$, 0.399, 0.327, 0.2773, 0.2680, 0.225, 0.2130, 0.189, 0.187, 0.180, 0.166, 0,162, 0.155 nm) (Fig. 6, *b*). $4CaO{\cdot}Al_2O_3{\cdot}13H_2O$ lines ($d/n = 0.475$, 0.248, 0.207, 0.197, 0.164 nm) are observed (Fig. 1, *b*).

Further, to determine the factors and conditions of stability of the ettringite phase for compositions based on aluminate and sulphate components and sulphoaluminate cements, studies of the stability of the ettringite phase with changes in humidity and pH of the environment, temperature, and operating conditions were conducted (Figs. 2–4).

The rate of heat release, total heat release, temperature and intensity of reactions of the hydration process depend on the ratio $\Delta G = f(CaO/Al_2O_3)$ of CaO, Al_2O_3 molecular weights. As is customary in the work, this ratio is defined as the coefficient of dependence of the Gibbs surface energy on the molar mass ratio. The higher the mineral coefficient and the Gibbs surface energy, the higher the exothermic rate of hydration and the total heat release (Fig. 2) [18].

The composition with a C_3A mineral content of 70%, 150–200 J/g C_3A has the highest exothermic rate during C_3A hydration in the C_3A–$C\dot{S}H_2$ system with different $C\dot{S}H_2$ content (Fig. 2, *b*). Because its surface energy is 2.8-times higher than that of CA and 5.8-times higher than that of CA_2. Thermodynamic curves characterize the rate of the hydration process and the influence caused by the ettringite formation phase on the structure of the mixture and, accordingly, on its properties.

In addition, the surface energy and specific surface area affect the formation of ettringite crystals, which subsequently creates

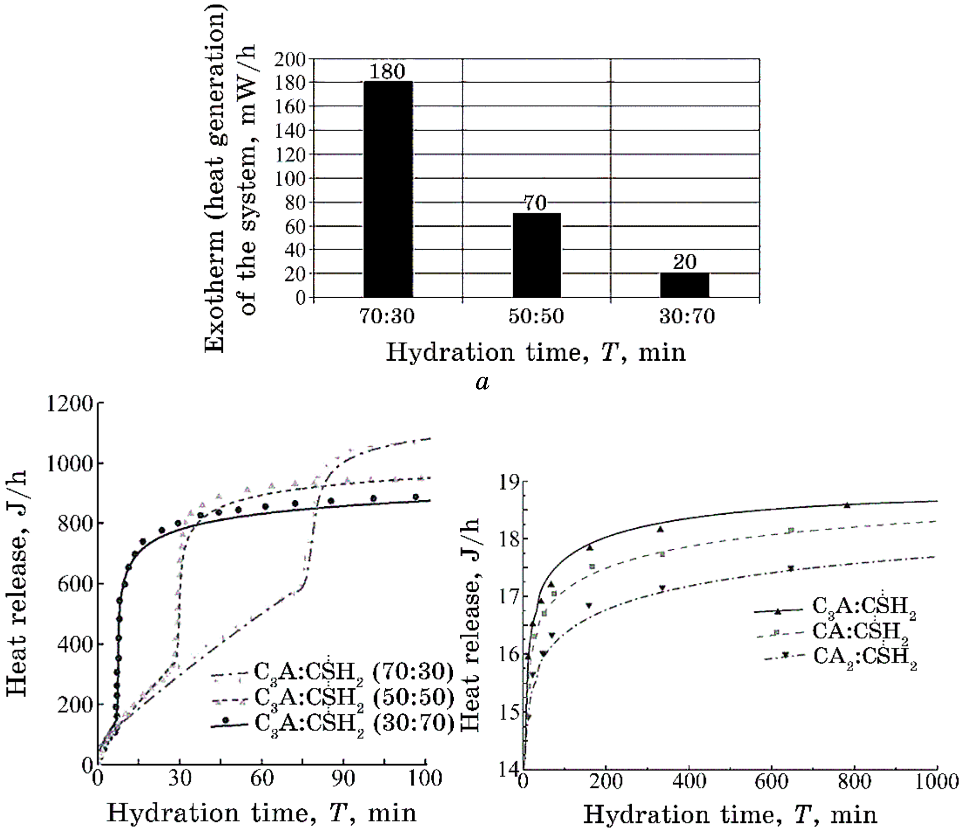
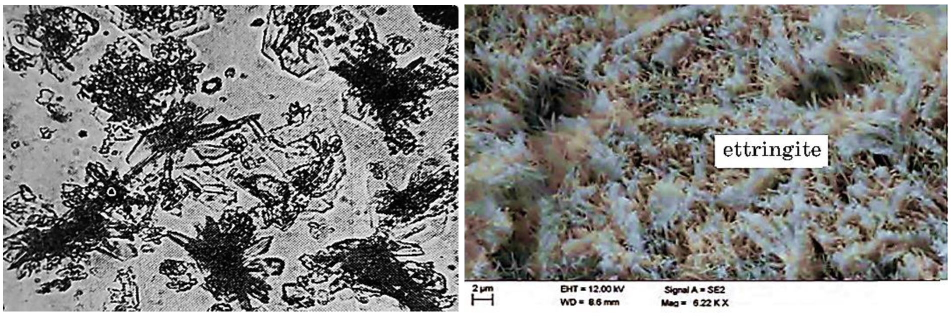


Fig. 2. Thermodynamic characteristics of the $C_3A + C\dot{S}H_2$ system: (*a*) exothermic histograms (mW/h); (*b*) heat release (J/g) [18]; (*c*) hydration temperature of $CA + C\dot{S}H_2$, $CA_2 + C\dot{S}H_2$, $C_3A + C\dot{S}H_2$ systems [18].

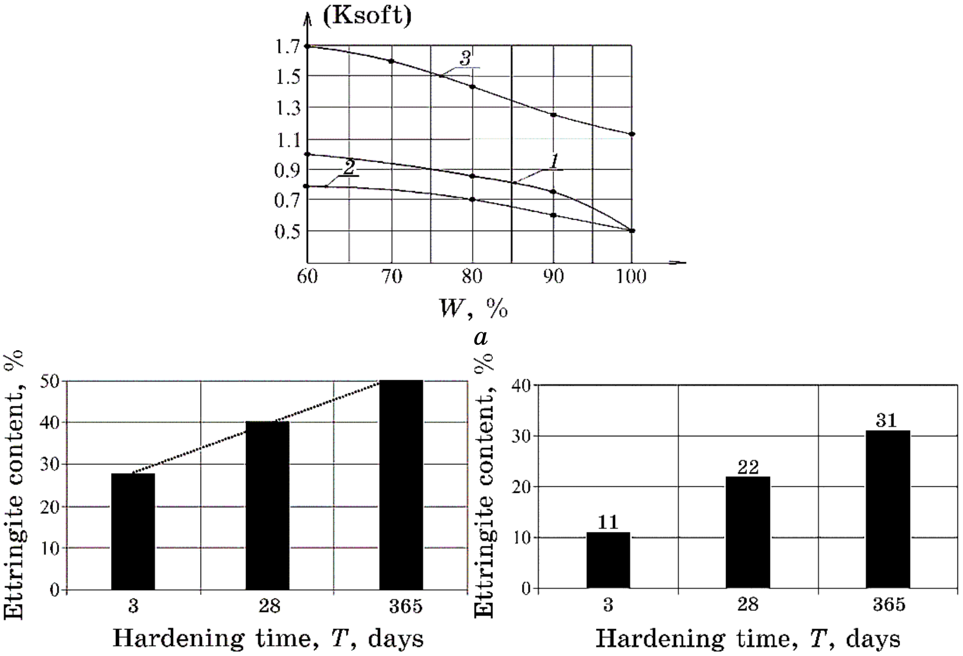
prerequisites for recrystallization in the process of system hardening (Fig. 3) [18].

The study of the influence of external factors was carried out,



a *b*

Fig. 3. Photomicrographs of the $C_3A + C\dot{S}H_2$ structure depending on the heat release of the system: *a*—$C_3A + C\dot{S}H_2$ (70:30); *b*—*A*C + Gypsum (30:70) [18].



a

b *c*

Fig. 4. Graphs of dependence [18]: coefficient of softening K_{soft} (*a*) on the medium with variable humidity of samples containing: *1*—C_3AH_6 hydrate, *2*—$C_3AC_3H_{32}$ mineral, *3*—*A*C:G; change in ettringite content over time: *b*—$C_3A + C\dot{S}H_2$, *c*—*A*C + Gypsum [18].

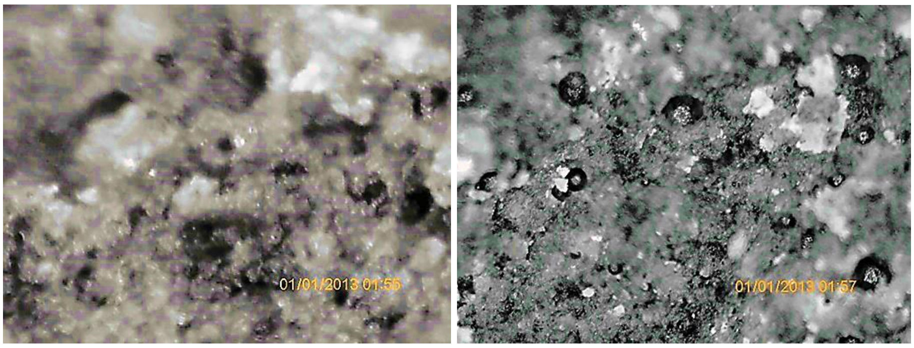
e.g., humidity for 3 days (Fig. 4, *a*). Change of the ettringite phase over time of nanomodified samples under normal conditions (Fig. 4, *b*, *c*).

Due to the high cost of pure minerals, it was decided to conduct further studies of the ettringite phase formed on the basis of the $AC+G$ composition (intended to replace alumina cement 70:30 and modify gypsum binders with the AC:G composition (30:70)).

Stability of ettringite crystals depends on the morphology of the crystals formed under different conditions, for example, on the pH value. The pH range of 11–12 provides a needle-like form of ettringite [7–10]. As the pH value increases, the length and thickness of needle fibres of ettringite decreases. At a pH greater than 13, ettringite has an x-ray amorphous gel-like structure (Fig. 5) [18].

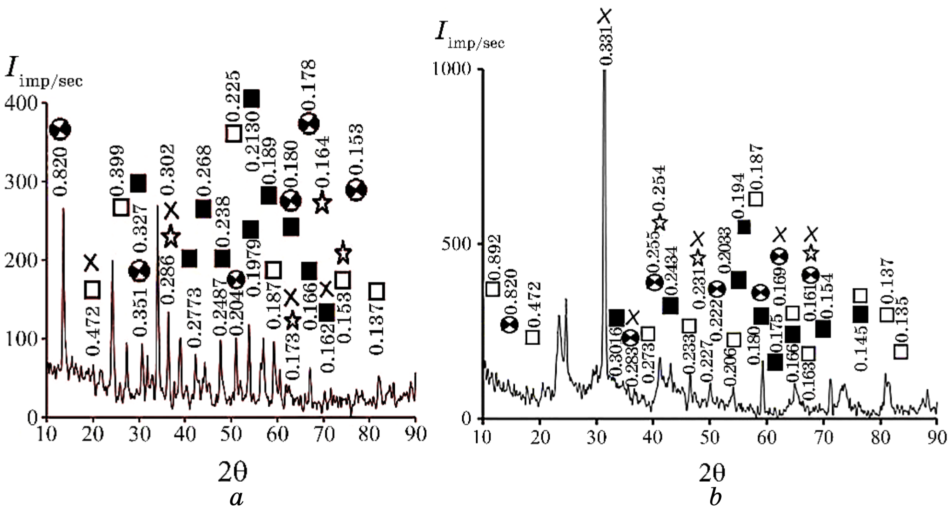
Study of the stability of the ettringite phase of the AC:G (70:30) system. Stabilization was carried out with nanomodifiers—taurite, silicon dioxide, nanotubes, SiC after determining the effect of surfactants. The following additives were used as plasticizers: Sika Viscocrete G, MC Bauhemi 2695, and Stachema STP 156. The smallest reduction in W/T and the best indicators of the main physicomechanical and technological properties are achieved using the Sika Viscocrete G additive: W/T for the AC:G composition (70:30) is of 0.27 compared to 0.32 without plasticizer, compressive and flexural strength is of 19 MPa and 14 MPa, respectively [1].

In the compositions after three days of hardening, in the compositions of alumina cement AC-40 gypsum grade G-5, after 3 days of hardening, the main interplanar distances and intensities of hydrated phases $C_3AC_3H_{32}$ ($d/n = 0.973$, 0.561, 0.388, 0.348, 0.256 nm) appear



a *b*

Fig. 5. The influence of the environment temperature on the structure and morphology of samples made of the composition of AC:G (70:30) on the 3^{rd} day of hardening under normal conditions in running water (*a*) and on the 3^{rd} day of hardening at an elevated temperature of 40–50°C (*b*) [18].



a *b*

Fig. 6. X-ray diffractograms of samples made of the composition *A*C:G (70:30) on the 3rd day of hardening (*a*) and on the 28th day of hardening (*b*); ■—ettringite $3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}31H_2O$; □—ettringite $3CaO{\cdot}Al_2O_3{\cdot}3CaSO_4{\cdot}12H_2O$; ⊗—hydroaluminate $Ca_4O{\cdot}Al_2(OH)_{14}{\cdot}6H_2O$; ★—tetracalcium nineteen-hydroaluminate $4CaO{\cdot}Al_2O_3{\cdot}19H_2O$; —tetracalcium thirteen-hydroaluminate $4CaO{\cdot}Al_2O_3{\cdot}13H_2O$.

(Fig. 10). The observed lines are as follow: $4CaO{\cdot}Al_2O_3{\cdot}13H_2O$ ($d/n = 0.423, 0.266, 0.246, 0.238, 0.212, 0.168$ nm), $4CaO{\cdot}Al_2O_3{\cdot}19H_2O$ ($d/n = 0.331, 0.238, 0.151$ nm), $Ca_4Al_2(OH)_{14}{\cdot}6H_2O$ ($d/n = 0.463, 0.255, 0.176, 0.151$ nm) (Fig. 6).

In the process of hardening, after 3 days, intensity of ettringite and calcium hydroxide lines appears and increases, which confirms the fact of the intensified hydration process (Fig. 6, *b*).

When modified with nanoadditives, the structure of samples with increased surface energy of the solid phase is formed as a result of an increase in the number of crystallization centres, and as a result, the structure is formed more quickly.

During the hardening process, the structure will have the property to deform [10–17]. At the same time, the tubes play a reinforcing role between the blocks [6].

Nanotubes and nanoadditives are entered into the crystal structure and alloying it.

As a result of modification with nanoadditives, an increase in the strength indicators of composite materials was achieved: for *A*C:G (70:30)% + 0.18% nanotubes + 0.4% Sika, up to 70.2 MPa compared to 14.67 MPa of the reference composition *A*C:G (70:30)%; for *A*C:G (70:30)% + 0.75% taurite + 0.4% Sika, up to 66.40 MPa; for *A*C:G (70:30)% + 1.0% silicon dioxide + 0.4% Sika, up to 60.73 MPa.

The softening coefficients increase accordingly: when *A*C:G (70:30)% is modified with nanotubes, up to 1.16; when it is modified with taurite, up to 1.02; when it is modified with silicon dioxide, up to 1.07 (Fig. 7).

Research on the compressive strength limit of samples modified with nanotubes over time showed that the composition with the optimal content of ettringite *A*C:G (70:30) at the age of three days has a compressive strength of 70.2 MPa, while, for the base composition, it is 14.7 MPa (Fig. 8).

The issue of the proposed hypothesis of increasing the stability of the ettringite phase due to modification with nanoadditives was considered.

The developed model of the hydration processes of mineral binders (on the example of gypsum binders) is presented in the form of

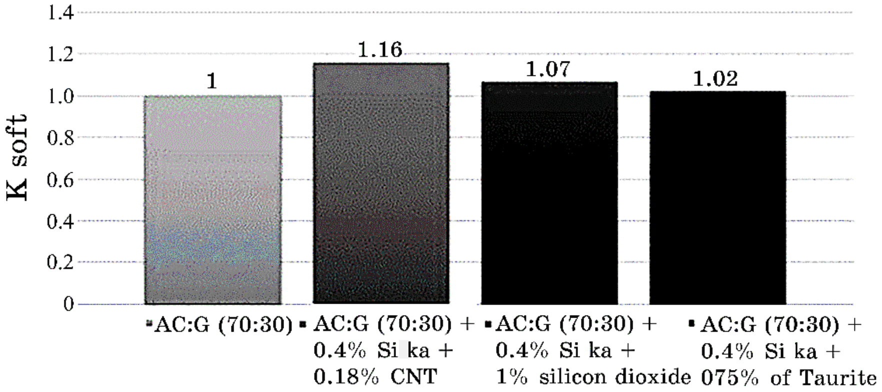


Fig. 7. Dependence of the softening coefficient for the *A*C:G (70:30) + Sika system on the type of nanomodifier.

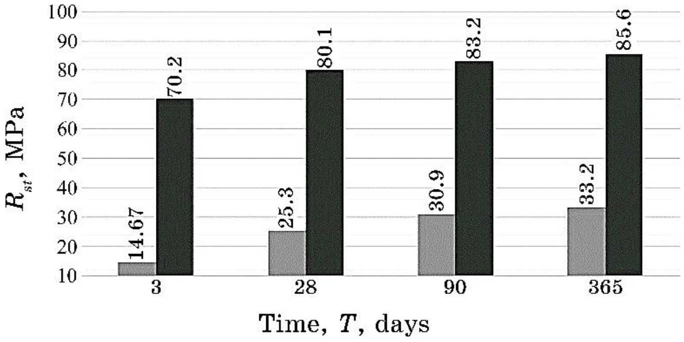
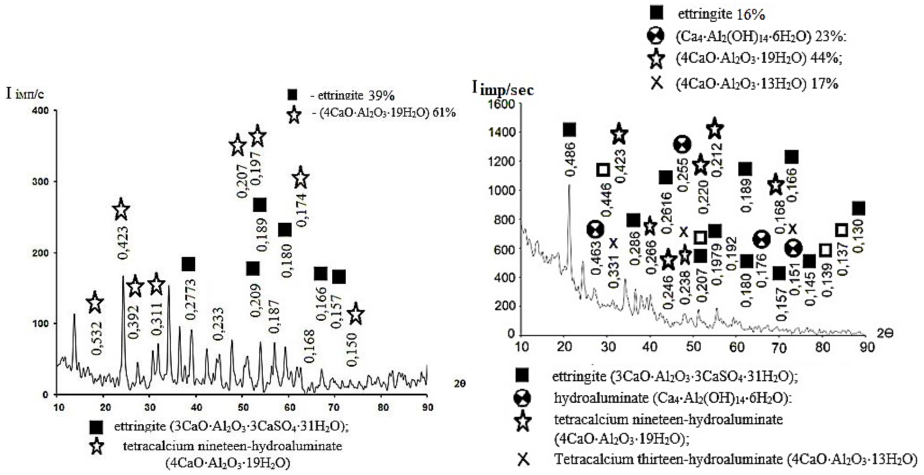


Fig. 8. Change in the compressive strength limit for the composition *A*C:G (70:30)% + 0.4% Sika + 0.18% CNT.



a *b*

Fig. 9. The amount of the formed ettringite phase for the composition $C_3A + C\dot{S}H_2$ (*a*) (■—ettringite 28%, □—ettringite 11%, ★—tetracalcium nineteen-hydroaluminate $3CaO{\cdot}Al_2O_3{\cdot}19H_2O$) and *A*C:G (70:30)% (*b*) (■—ettringite 11%, □—ettringite 5%, ⊗—hydroaluminate $Ca_4O{\cdot}Al_2(OH)_{14}{\cdot}6H_2O$ 23%, ★—tetracalcium nineteen-hydroaluminate $4CaO{\cdot}Al_2O_3{\cdot}19H_2O$, —tetracalcium thirteen-hydroaluminate $4CaO{\cdot}Al_2O_3{\cdot}13H_2O$) for 3 days during hardening under normal conditions.

a system that changes over time and passes through the stages of the physical state of macro–micro–nano–micro–macro. The transition from macro-/micro- to nanosystem with formation of a dispersed medium is presented in the form of a surface (Fig. 10), consisting of nanoparticles in a multidimensional phase space [3].

The model represents a three-phase medium binder → solution → → stone and is separated with two surfaces. The surface is the in-

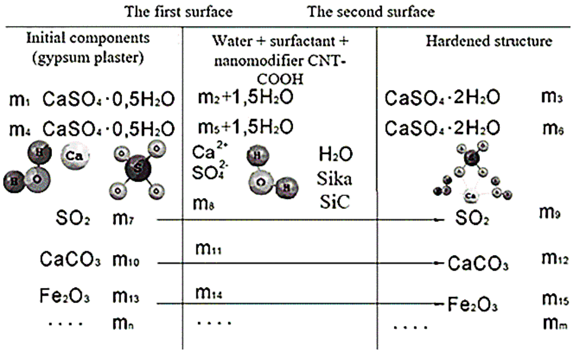


Fig. 10. Model of the hydration process on the example of hemihydrate.

terface between structural elements and the dispersed medium. The second surface is the interface between the dispersed medium and the solidified structure. At the interface, there is a partial transition of the macro- and microsystem into a nanosystem, as well as topochemical reactions of the transition of hemihydrate of calcium sulphate into calcium sulphate dihydrate.

The measure of the change in the characteristic function at constant parameters and masses (concentrations) of all substances (except for the mass (concentration) of the component with a changing amount in the system) is presented as the chemical potential, which allows creating a structure and framework of the highest strength by adjusting the dimensions of the solid surface and the number of crystallization centres. Creation of a frame with the highest strength can be achieved by adjusting the size of the solid surface and centres of crystallization, which affect the primary spatial structure.

The idea is that a frame of the highest strength can be created by adjusting the dimensions of the solid surface and the number of crystallization centres that affect formation of the spatial structure. Internal stresses, which lead to the weakening of the not yet formed structure, do not arise as a result of the fact that the joining of blocks takes place in free space.

The entire transformation of the system is reduced to obtaining a solidified structure with certain properties.

In the system of macro-/micro- and nanoevolution, each of its elements passes from one permitted state to another.

Calculation of the state of the nanosystem consists in the calculation of the external and internal components and means $\psi(\vec{\xi},\tau)Z_i(m,\bar{\xi}_i)$ introduction of the elementary density, which can be calculated by solving the Schrödinger equation.

According to the proposed model at the starting point, the specific surface of solid components consists of particles in contact with the solution:

$$\sum M_{вих} = m_1 + m_4 + m_7 + m_{10} + m_{13} + \ldots;$$

m_1 $CaSO_4 \cdot 0.5H_2O$ (mass of hemihydrate conversion to dihydrate due to dissolution); m_4 $CaSO_4 \cdot 0.5H_2O$ (topochemical reaction); m_7, m_{10}, m_{13}, $mn-2$ $CaCO_3$, Fe_2O_3, and other, respectively.

Taking the structure of hardened binder or materials based on it as the final criterion (initial factor), it is necessary to determine the main parameters. Such parameters include the spatial framework, shape of the blocks, and morphology of the crystals.

A decisive role for formation of these parameters belongs to solutions of a certain concentration, presence of a solid surface of the site (crystallization of centres), (water), temperature, pressure, and the mechanism of transition of calcium sulphate hemihydrate to calcium sulphate dihydrate.

The sequence of changes in the concentration of the solution determines the moving force of the interaction, which varies from 0 to 1. The maximum concentration of $C_2 = 0.008$ g/l contributes to creation of crystallization centres, growth and formation of blocks, as well as to filling of voids.

Therefore, by means of changing conditions for introduction of various additives into the solution system, it is possible to influence the shape, size, number of crystals, and, accordingly, formation of a spatial structure for the purpose of obtaining a product with certain properties.

4. CONCLUSIONS

Studies of the structure and durability of the ettringite phase and its composition with calcium sulphates were carried out. Raw material compositions based on fast-hardening binder systems CaO–Al_2O_3–SO_3–H_2O AC:G (70:30) for obtaining concrete grades 300–400 were developed, which makes it possible to obtain fast-hardening binders in monolithic construction that are capable to gain up to 90–100% of their strength within 1–3 days and increase the reversibility of forms in case of factory production of building structures.

The influence of the Gibbs surface energy on the order of formation of hydrated compounds during the hydration of minerals of alumina cements was established theoretically and confirmed experimentally: for CA_2, 24.70; for CA, 51.86; for $C_{12}A_7$, 141.00; for C_3A, 145 of the system CaO–Al_2O_3–H_2O: C_2AH_8, C_4AH_{13}, C_4AH_{10}, CAH_{10}, $Al(OH)_3$, which makes it possible to determine the list of stable hydrate compounds and structures, which affect recrystallization in multicomponent systems.

The theoretical position of influence of ΔG during hydration of aluminate cements in the presence of gypsum received further development; the frequencies of chemical reactions CaO–Al_2O_3–SO_3–H_2O can be divided into two parts: *a*) CaO–Al_2O_3–H_2O; *b*) CaO–Al_2O_3–SO_3–H_2O, and, as a result, C_3AH_6, CAH_{10}, C_2AH_8, C_4AH_{13}, $Ca_2(OH)_2$ are formed in the first one, and trisulphate C–A–S–H and monosulphate C–A–S–H are formed in the second one. As a result, it is possible to determine the tendency of hydrate compounds to interact with other components and creates conditions for recrystallization of hardened stone.

It was theoretically established and experimentally confirmed that the optimal content of calcium sulphate in AC + G5 compositions is, according to the calculation, in the range from 28% to

38% of the mass of the aluminate binder and contributes to the increase in the formation of ettringite and allows obtaining the structure of cement stone with special characteristics.

Formulations of special solutions for the production of prestressed products (road, airfield slabs, expanding, waterproofing concrete) were developed that makes it possible to satisfy the need for the production of the latest building materials for the repair and construction of high-quality structures.

For the first time, a theoretical model of the hydration process of mineral binders using ultra- and nanoadditives, surfactants was proposed. The developed model of the hydration process can be used in various systems, including those presented earlier.

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