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APPLICATION OF NANOMODIFIED COMPOSITE SULFOALUMINATE CEMENTS WITH ENHANCED FUNCTIONAL PROPERTIES

Introduction. *Ionizing radiation induces defects in the crystalline lattice of calcium hydroxide, leading to radiation-induced shrinkage. Due to shape anisotropy and aggregate deformation, these effects propagate non-uniform stresses throughout the concrete matrix, compromising its structural integrity.*

Problem Statement. *In the context of addressing scientific and technical challenges, the potential to enhance the physical and mechanical properties of* $CaO—Al_2O_3—SO_3$ *binder systems has been investigated. Given the limited availability of raw materials for alumina cement production and the high cost of imported specialty cements (25–35 thousand UAH/t), both theoretical and experimental studies on the development of advanced composite binders are of current importance.*

Purpose. *This study aims to develop theoretical foundations for improving the performance of mortars based on nanomodified composite binders within the* $CaO—Al_2O_3—SO_3$ *system, focusing on stabilization of the ettringite phase and optimization of nanoadditive integration technologies.*

Materials and Methods. *The experimental phase employed modern analytical techniques, including X-ray diffraction (XRD), scanning electron microscopy (SEM), and low-temperature dilatometry, to assess the structural and phase transformations in cementitious systems.*

Results. *The theoretical framework for designing construction-grade mortars with ion-protective properties has been further developed. These mortars are based on composite mineral systems of the* $CaO—Al_2O_3—SO_3—H_2O$ *type, which incorporate up to 42% chemically bound water due to ettringite content. Carbon nanotubes have been successfully used to modify sulfate and sulfoaluminate phases, resulting in improved microstructural stability.*

Conclusions. *For the first time, a theoretical model has been established, and experimentally validated, for stabilizing the ettringite phase in specialty cements through the introduction of functionalized carbon nanotubes (5—25 nm in diameter). The stabilization mechanism involves nanoscale alloying and reinforcement of the ettringite crystal structure, thereby enhancing its durability under operational conditions.*

Keywords: composite binder, mortar, nanoadditive, ettringite, phase stabilization, aluminate cements, sulfoaluminate cements.

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Cements with special properties are widely employed in the construction industry for manufacturing structural products, advanced building materials, and specialized mortars. Aluminate and sulfoaluminate cements represent key categories within this group. Among them, alumina cement — classified as an aluminate cement — is of particular significance. However, domestic production of alumina cement is currently absent, resulting in reliance on imported materials from countries such as Turkey and Poland.

The current market price for alumina cement ranges between UAH 25,000 and 30,000 per metric ton, which highlights the importance of developing cost-effective alternatives. One such approach involves the partial replacement of alumina cement with gypsum, along with the modification of gypsum binders using alumina cement to enhance their performance.

Despite their advantages, these cements exhibit certain limitations, particularly in the recrystallization behavior of hydrosulfoaluminate phases during service life. This issue is not unique to aluminate systems; the stability of ettringite — a key hydration product — is also a known concern in Portland cement-based composites.

Contemporary research on these specialized cements primarily focuses on developing alternative formulations that reduce or eliminate the need for scarce raw materials. While the production cost of such composite materials may exceed that of conventional binders, the enhanced performance characteristics — such as improved durability, chemical resistance, and dimensional stability — justify their use. Cumulative research findings confirm the technical and economic feasibility of advanced cementitious compositions that integrate alumina cement, gypsum, and industrial by-products.

From both theoretical and practical standpoints, the development and study of binder systems within the $CaO—Al_2O_3—SO_3$ composition — particularly those derived from industrial waste streams — are of continued relevance. Investigating the impact of various chemical additives on the hydration kinetics and microstructural evolution of the $CaO—Al_2O_3—SO_3—H_2O$ system offers the potential to regulate internal stresses and improve the functional properties of the hardened matrix.

The hardening of gypsum–cement–slag–pozzolanic and other composite gypsum binders results from complex physicochemical transformations. These processes lead to the formation of new hydrated phases — distinct from those in conventional gypsum binders — which ultimately enhance performance and align these materials more closely with Portland cement in terms of strength and durability [1—6].

In particular, the hardening of gypsum–cement–pozzolanic and gypsum–cement–slag–pozzolanic binders relies on the chemical transformation of highly basic calcium hydroaluminates (and ferrites) present in the initial mixture. Through the introduction of reactive acidic mineral additives — such as pozzolans or hydraulic components — these phases are converted into lower-basicity compounds. Furthermore, favorable conditions are established for the transformation of ettringite into its low-sulfate, more stable forms.

Upon interaction with water, the semi-aqueous gypsum binder undergoes hydration and hardening. This process leads to the formation of dihydrate gypsum crystals, which create the primary structural framework. Simultaneously, hydration of the cement clinker minerals is initiated, accompanied by the release of free calcium hydroxide [7—8].

An essential component of these binders is an active mineral additive, such as *trepel* or *opoka*, which plays a regulatory role in maintaining the alkalinity of the system. These additives react with calcium hydroxide, reducing its concentration in the liquid phase to levels at which highly basic calcium hydroaluminates become unstable. This creates favorable conditions for their transformation into more stable, low-basicity forms. As a result, aluminum oxide (Al_2O_3) is rapidly bound into a low-crystallinity, calcium hydrosulfoaluminate phase of the trisulfate type — ettringite — during the early stages of hardening. Over time, ettringite may partially or completely convert into the monosulfate form ($3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 12H_2O$).

In parallel, other hydration products such as dihydrate gypsum, hydrogelenite ($2CaO \cdot Al_2O_3 \times \times SiO_2 \cdot 8H_2O$), hydrogarnets ($3CaO \cdot Al_2O_3 \cdot xSiO_2 \times \times (6-2x)H_2O$), hydrosilicoaluminates ($3CaO \times \times Al_2O_3 \cdot CaSiO_3 \cdot 12H_2O$), and their solid solutions may also form. These phases can pose long-term risks to the structural stability of the hardened binder matrix [2—4].

Cementitious calcium silicates, namely alite and belite, are partially hydrolyzed during hydration and produce gel-like calcium hydrosilicates with a general composition of $CaO \cdot SiO_2 \cdot nH_2O$. Similar hydrosilicate phases are also formed from the interaction of calcium hydroxide with the active mineral additives. These newly formed gel phases act as a cementing medium that binds the larger dihydrate gypsum crystals developed in the early stages of setting, effectively shielding them from subsequent dissolution in water. This mechanism significantly enhances the water resistance of gypsum–cement–pozzolanic and gypsum–cement–slag–pozzolanic binders, in contrast to conventional gypsum binders [8—12].

Returning to the problem of developing fast-hardening special cements, it should be noted that the direction of their creation based on aluminates and sulfates is quite promising. Such composite binders are presented in the sphere of modern construction in the form of special cements (expanding, non-shrinking).

It should be noted that hydrophobization of calcium sulfates with Portland cement or alumina cement provokes the problem of ettringite formation (especially secondary one). This process is present both when modified with Portland cement and when modified with alumina cement. The influence of operational factors leads to the recrystallization of the highly sulfated form of ettringite into the low sulfate form and vice versa. As a result of these processes, there arises the problem of stabilizing the structure of the highly sulfated form of ettringite [7].

Purpose of the article is to develop theoretical provisions for improving the special properties of mortars based on nanomodified composite binders of the $CaO—Al_2O_3—SO_3$ system, stabilization of the ettringite phase $3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot nH_2O$ (calcium trisulphato aluminate hydrate — CtSAH) and $3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 12H_2O$ (calcium monosulphato aluminate hydrate — CmSAH), and the technology of introduction of nanoadditives.

The solution preparation technology includes: a) production of composite binders, according to which pre-dosed components of the solution are mixed in the given proportions of the optimal composition of the X-ray protective coating solution: –1 : 2.5 : 1.6 for AC-40 : G5 : BC-3 (barite concentrate) and 0.8% by mass of binder for Sika plasticizer; b) production of concentrated CNT nanoadditives: the technology of introducing nanoparticles was developed by the authors and involves dispersing a nanoadditive in a water–plasticizer medium for 4.5—6.0 minutes with the subsequent introduction of the grouting fluid during mortar or concrete preparation; c) preparation of the solution according to the developed compositions (Table 1); d) industrial production of sulfate-based products.

In accordance with the technological scheme, the pre-dosed components — alumina cement (AC), gypsum (G5), and barium sulfate ($BaSO_4$) — are delivered from waste hoppers equipped with dispensers to the mixer drum via a skip elevator. The mixing duration in the mortar mixer is 2 min.

Nanoadditives and water are introduced into the mixer and then transferred to an ultrasonic treatment unit for homogenization, which lasts 4.5—6.0 min. Afterward, the remaining components are added to the concentrated suspension, and the working mortar is prepared.

Table 1. **Physical and Mechanical Properties of Radiation Protective Coatings**

Composition of mortars	Properties		
	R_{compr}, MPa	R_{bend}, MPa	ρ_0, kg/m^3
AC-40 : G5(70:30) : BC-3	3.58	2.16	2450
AC-40 : G5(50:50) : BC-3	3.26	1.93	2250
AC-40 : G5(70:30) : BC-3:CNT	2.66	1.53	2310

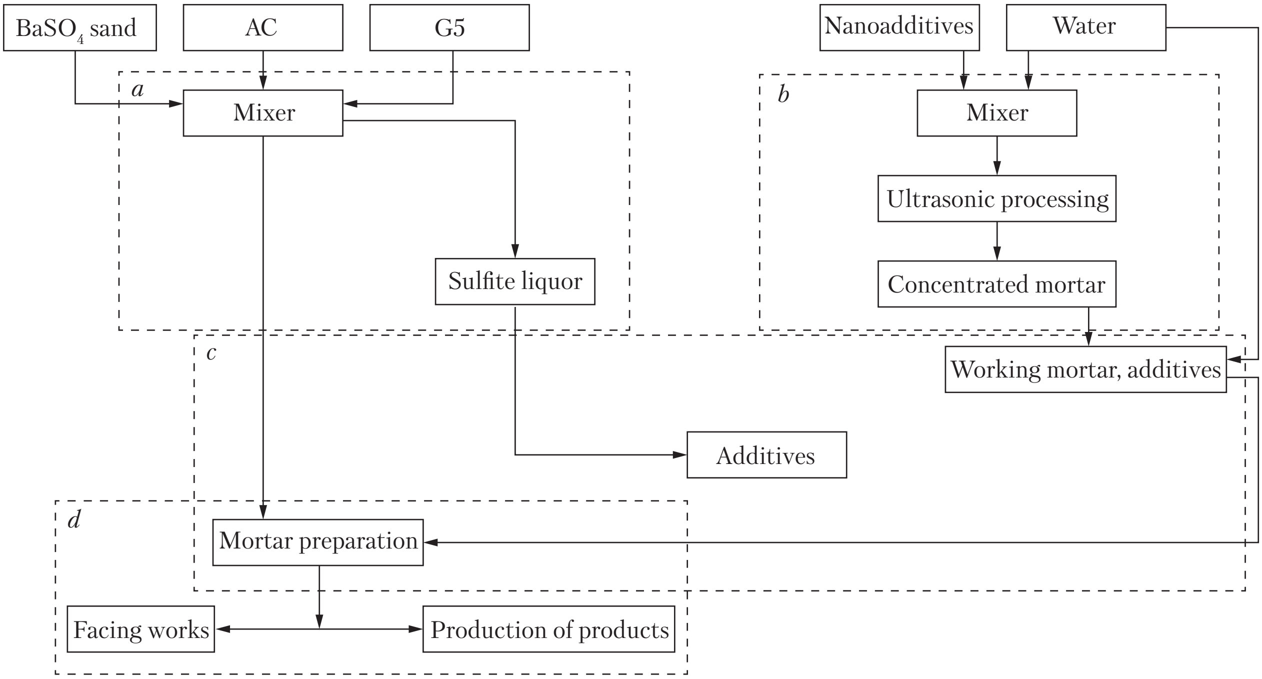

Fig. 1. Technological scheme of production: *a* — composite binders of the $CaO—Al_2O_3—SO_3$ system; *b* — concentrated nanoadditives CNT; *c* — preparation of the mortar; *d* — industrial production of sulfate products

The yield coefficient of the concrete mortar is 0.8. A mortar mixer with a 325 L capacity produces 33 batches/h. Assuming the mixer operates in two shifts over 300 working days/year, and applying the mortar output ratio of 0.8, the annual production capacity of the mortar mixer is 18,000 m^3/year. The calculation of annual capacity also takes into account a time utilization factor of 70% for the mortar mixer.

The compositions and properties of the solutions are given in Tables 1.

The physical and mechanical properties of the facing radiation protective coating are presented in Table 1. Molecular weight 233.4 g/mol, $\rho =$ $= 4500$ kg/m^3. The mobility index of the solution, determined by the immersion depth of the cone, for the coating is 7 cm.

Approbation of protective properties of coatings against ionizing radiation has shown its effectiveness.

Materials containing chemically bound water are effective materials for biological protection against ionizing radiation, especially against neutron radiation. Such materials include concrete and mortars based on aluminate binders of the $CaO—Al_2O_3—SO_3$ system.

Alumina cement, the main minerals of which are CA, $C_{12}A_7$, C_3A and others, are particularly effective radiation protection materials for absorbing and weakening neutrons. Thus, the linear attenuation coefficient γ — of protons, cm^{-1} (the energy of 0.3—1 MeV is equal to 0.096—0.319). When interacting with water, a dicalcium hydroaluminate crystal hydrate is formed, in which the number of water molecules reaches the level of eight molecules. The total amount of chemically bound water is within 25—35%.

Due to the difficulty of determining the linear attenuation coefficient in the work, its calculation was carried out by comparing the mortars according to the content of chemically bound water in them. At $E \geqslant 1.0$ MeV for plastering mortars $PC + BaSO_4$ with a composition ratio of 1 : 3, the coefficient $\mu = 0.324\ cm^{-1}$, the amount of water of hydrated binder Portland cement is approximately equal to 100 l. The arithmetic mean contribution of chemically bound moisture to the linear attenuation coefficient of ionizing radiation is $\mu = 0.09\ cm^{-1}$.

As a result of industrial tests, it was confirmed that the modification of the CNT mortar due to the high specific surface area (80—120 m^2/g) of the additive leads to a decrease in the coefficient of linear expansion and an increase in the absorption properties of particles with high energy. That is, the use of nano-sized materials leads to an up to 1.5-time increase in the absorption coefficient of neutrons as well as to an increase in the scattering coefficient of gamma rays by 30—40%.

Modification of the compositions of radiation protective solutions made it possible to reduce the coefficient of linear expansion to 0.8% while increasing the strength by an average of 8—12%.

Mortar based on AC + G5 + $BaSO_4$ + CNT has a content of chemically bound water that is 10—15% higher than formulations based on Portland cement due to the formation of the ettringite component.

At the same time, the average arithmetic composition of chemically bound moisture of the linear attenuation coefficient of ionizing radiation increases by 0.0088—0.009 cm^{-1}. And then the total coefficient can reach 0.354 cm^{-1} and more, which makes it possible to reduce the equivalent (14.6 cm^{-1}) thickness of the radiation protective layer by 1—1.5 mm.

The results of the approval of special solutions have been also obtained (Table 2).

In the process of testing, it was confirmed that the maximum content of ettringite has the following composition of the solution: 17.1% alumina cement, 7.32% gypsum, 0.4% plasticizer and 0.18% nanotubes and 75% sand, providing a system strength of 39.5 MPa. The mobility of the mortar is 8 cm.

For samples with white carbon (its optimal content is 1%) the strength of the system of 38.6 Mpa includes 16.52% of alumina cement, 8.48% gypsum, 0.4% plasticizer and 75% sand.

From the economic point of view, depending on the customer's requirements, it is possible to regulate the consumption of alumina cement and gypsum binder according to developed recomendations.

As a result of the conducted research, a model of a solidified system was obtained, with a certain structure and with characteristic properties of the developed composition of composite binder materials through the use of nanosystems, which ensure reduction in energy consumption of production.

Research and industrial production and technical and economic indicators of the production of special solutions with taurite and nanotubes.

For the implementation of the research, it is planned to purchase materials, namely cement, brand M400, alumina cement, M400, gypsum G-5 H II, tricalcium aluminate powder, granite crushed stone, fraction 0—40 mm, river sand, graphite nanotubes, 10—20 nm, plasticizer Sika BV 3M.

The cost of materials, equipment, and inventory is calculated based on the prices existing in Ukraine at that time. The price of materials includes the cost of delivery and unloading to the storage place.

When analyzing production of individual parts and products in different areas, it can be conclu-

Table 2. **Experimental Compositions and Test Results of Cements Based on CNT (1) and Taurite (2)**

Compositions of binders and their properties	Ratio of components, in wt.% and indicators of properties	
	1	2
Alumina cement	17.1	16.7
Gypsum	7.32	7.15
Plasticizer	0.4	0.4
Nanotubes	0.18	—
Taurite	—	0.75
Sand	75	75
Water-solid ratio	0.25	0.25
Compression strength, MPa		16.7
3 days	39.5	30.5
28 days	46.8	42.1

ded that stabilization of the ettringite phase is promising, which allows controlling the expansion of cement stone, which is associated with formation of this ettringite phase.

Solving the problems of providing the population with housing, restoring social objects is possible through the development and use of new technologies, one of which is presented as 3D printing technology, through creation of concrete, mortars, and composite materials that would meet regulatory requirements in the sphere of construction.

Therefore, it is promising to develop a binding material from production waste based on the $CaO—Al_2O_3—Fe_2O_3—SO_3$ system.

To develop composition of the binder, the following materials are used: slag, red mud, and semiaqueous gypsum.

Raw samples with a loose structure have a compressive strength of 1.6 MPa, with a gap — 8.2 MPa.

The developed $CaO—Al_2O_3—Fe_2O_3—SO_3$ binder system has been tested with the following composition: 55% slag, 17.6% red mud, and 27.4% gypsum (G-5). The system has demonstrated a dense fine-pore structure and high early compressive strength of 55 MPa, with an average density of 1,750 kg/m^3.

The optimal firing temperature and duration have been set at 1150 °C and 60 min, respectively. An increase in the average density of samples fired at 1150 °C for 60 min, ground, and mixed with water has been observed — 500 kg/m^3 higher compared to samples fired at 950 °C for the same duration.

Based on full-factor experimental design methods and conducted experimental studies, a series of experimental-statistical models has been developed. Multiple optimal mortar compositions have been identified based on components of alumina and gypsum binders containing a maximum ettringite phase. One such composition includes 17.1% alumina cement, 7.32% gypsum, 0.4% plasticizer, 0.18% carbon nanotubes (CNTs), 75% sand, and achieves a compressive strength of 40.2 MPa. Another optimized composition, which provides 37.8 MPa, consists of 16.7% alumina cement, 7.15% gypsum, 0.4% plasticizer, 0.75% taurite, and 75% sand. A further variant with 1% silicon dioxide yields the highest strength of 42.8 MPa and comprises 16.52% alumina cement, 8.48% gypsum, 0.4% plasticizer, 1% taurite, and 75% sand.

Special X-ray protective mortars have been developed based on binders of the $CaO—Al_2O_3—SO_3—H_2O$ system for use as cladding coatings in rooms exposed to ionizing radiation. Due to their ettringite content and the modification of sulfate and sulfoaluminate phases with CNTs (Fp = 80—120 m^2/g), these compositions contain up to 42% chemically bound water. This characteristic enables a reduction of the radiation shielding layer's equivalent thickness (14.6 mm) by 1—1.5 mm, achieved by lowering the linear expansion coefficient and increasing the gamma-ray scattering coefficient by 30—40%, as well as raising the linear attenuation coefficient of ionizing radiation by 0.008—0.009 cm^{-1} to 0.364 cm^{-1}.

An optimal X-ray protective mortar composition has been determined as: AC-40 : G5 : barite concentrate (KB-3) = 1 : 2.5 : 1.6, with Sika plasticizer at 0.8% by binder mass. The mineral composition and fundamental properties of this mix have been established. Notably, a binder ratio of AC-40 : G5 = 50 : 50% results in an expansion coefficient of 2%, which leads to sample degradation under normal conditions. It has been shown that modification with CNTs effectively reduces the linear expansion coefficient and enhances the gamma-ray scattering coefficient by 30—40%, owing to the additive's high specific surface area (80—120 m^2/g).

Mortars based on AC + G5 + $BaSO_4$ + CNTs demonstrate a 10—15% increase in chemically bound water compared to Portland cement-based systems, attributed to the enhanced formation of the ettringite phase. As a result, the average linear attenuation coefficient of ionizing radiation increases by 0.008—0.009 cm^{-1}, potentially reaching a total coefficient of 0.354 cm^{-1} or higher, allowing for a reduction of the protective layer thickness by 1—1.5 mm.

A novel method for improving the dispersion and stability of mortars using CNTs has been developed. This is achieved by reducing particle settling with Sika plasticizer and treating the mixtures with ultrasound (15—20 kHz), followed by additional mixing with water to form stable suspensions. This innovation opens new prospects for incorporating nanomodification technologies in construction mortars.

Theoretical principles have been developed and experimentally validated for the mechanism of ettringite phase stabilization through the introduction of functional CNTs (5—25 nm in diameter). This mechanism includes alloying and nanoreinforcement of the ettringite structure, ensuring stability during the operational life cycle of the products.

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ПРАКТИКА ВИКОРИСТАННЯ НАНОМОДИФІКОВАНИХ КОМПОЗИЦІЙНИХ СУЛЬФОАЛЮМІНАТНИХ ЦЕМЕНТІВ З ПОКРАЩЕНИМИ ФУНКЦІОНАЛЬНИМИ ВЛАСТИВОСТЯМИ

Вступ. При дії іонізуючого випромінювання відбувається утворення дефектів у решітці кристалів гідрооксиду кальцію, що спричиняє радіаційну усадку. У результаті анізотропії форми і деформацій заповнювачів нерівномірні деформації передаються на скелет бетону.

Проблематика. У межах розв'язання науково-технічних проблем проведено аналіз потенційних резервів фізико-механічних властивостей в'яжучих систем $CaO—Al_2O_3—SO_3$. Враховуючи відсутність сировини для виробництва глиноземистого цементу та вартість імпортних цементів спеціального призначення (25—35 тис. грн/т) виконання теоретичних і експериментальних досліджень щодо розробки теоретичних положень і композиційних в'яжучих є актуальним.

Мета. Розробка теоретичних положень покращення спеціальних властивостей розчинів на основі наномодифікованих композиційнивх в'яжучих речовин системи $CaO—Al_2O_3—SO_3$, стабілізації етрингітової фази та технології введення нонодобавок.

Матеріали й методи. Для реалізації експериментальної частини було застосовано сучасний набір методик, зокрема рентгенівську дифрактометрію, растрову електронну мікроскопію, низькотемпературну дилатометрію.

Результати. Отримали подальший розвиток теоретичні положення розробки будівельних (спеціальних) розчинів для іонозахисних покриттів на основі мінеральних композиційних речовин системи $CaO—Al_2O_3—SO_3—H_2O$, які містять у своєму складі підвищену кількість хімічно-зв'язаної води (до 42 %) за рахунок вмісту етрингіту та шляхом модифікації сульфатних і сульфоалюмінатних фаз вуглецевими нанотрубками.

Висновки. Вперше розроблено теоретичні положення і підтверджено експериментальними дослідженнями механізм стабілізації етрингітової фази цементів спеціального призначення за рахунок введення функціональних нанотрубок діаметром 5—25 нм. Механізм передбачає легування та наноармування структури етрингітової фази і забезпечує її стабільність у процесі експлуатації виробів.

Ключові слова: композиційне в'яжуче, розчин, нанодобавка, етрингіт, стабілізація етрингітової фази, алюмінатні цементи, сульфоалюмінатні цементи.