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INFLUENCE OF MODIFICATION OF THE SOLID COMPONENT ON THE PROPERTIES OF NON-AUTOCLAVED AERATED CONCRETE

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Наведені результати досліджень модифікації твердої складової неавтоклавного газобетону вапняно-карбонатною добавкою, що містить карбонат кальцію (кальцит), гідроксид кальцію (портландит) та добавки пластифікуючої і прискорюючої дії з метою підвищення його міцності. За результатами аналізу літературних джерел висловлено припущення, що властивості ніздрюватих бетонів визначаються характером твердої складової. В якості об'єкту досліджень обрано неавтоклавний газобетон густиною 500 кг/м^3*. Наведено перелік сировинних матеріалів та їх характеристики. Газобетон формували при фіксованій водопотребі, яка відповідала розпливу суміші 220 мм за віскозиметром Суттарда. В процесі експериментальних робіт застосовували як стандартні методи випробувань, так і оригінальні (математико-статистичні методи, рентгенофазовий аналіз, визначення напруженості еквіпотенціального поля поверхні зразків газобетону).*

Отримано неавтоклавний газобетон з модифікованою твердою складовою, який має максимальну міцність при стиску 3,53 МПа, що відповідає класу бетону С2 згідно діючого стандарту. Висока міцність пояснюється, за даними рентгенофазового аналізу, присутністю кристалічних фаз, які представлені стійкими новоутвореннями у вигляді карбонату кальцію та його модифікацій: фатеріту – μ-форма $CaCO_3$*, арагоніту – метастабільної форми* $CaCO_3$ *і тоберморітового гелю.*

На основі отриманих даних побудовані експериментально-статистичні моделі досліджуваних властивостей. Встановлено певний зв'язок між міцністю неавтоклавного газобетону та напруженістю еквіпотенціального поля. Результати досліджень запроваджені на виробництві при виготовленні виробів із неавтоклавного газобетону, які за міцністю не поступаються його автоклавним аналогам

Ключові слова: ніздрюватий бетон, газобетон, тверда складова, кальцит, поліморфні модифікації, портландит

1. Introduction

One of the priority tasks for construction industry is the task on saving natural and energy resources. The existing raw materials base for the production of building materials is almost depleted. Places of their extraction are further away from enterprises that manufacture them. Supply of raw materials that are far away leads to a rise in the cost of materials. The increasing cost of articles is also due to the steady rise in energy resources. Recently, construction industry has been given a series of new regulations aimed at reducing the energy and raw material resources, as well as improving the quality and reliability of construction. That primarily refers to strict requirements to ensuring the thermal resistance of enclosures, enhanced requirements to structures being built in seismic active areas. Meeting the requirements to protecting structures, made from materials such as ceramic and silicate bricks, lightweight concrete on porous aggregates, is challenging due to low thermal-insulating indicators according to DSTU B EN 15217 (EN 15217:2007, IDT). One of the ways to solve this task is a widespread use of effective wall and thermal-insulating materials, including articles made from cellular concretes.

One type of cellular concrete is non-autoclaved aerated concrete. Technical and economic attractiveness of its production, when compared with autoclaved aerated concrete, is due to much lower energy intensity.

Traditionally, dependence of the properties of cellular concretes is associated with the character of their porosity [1]. However, during operation, it is not the porous structure but the solid component that perceives a complex of external loads and actions, resulting in the emergence of magistral cracks in the material, leading to deterioration in the material's properties and, ultimately, to its destruction. Therefore, it is advisable to relate the properties of cellular concretes to the character of a solid component that acts as a carrying frame. Given the constantly increasing energy prices, the cost of a binder steadily grows, which necessitates a search for variants to partially replace it in the composition of concrete with the waste from energy sector.

Therefore, it is a relevant task to address the issues on improving the production technology and on enhancing the construction and operational properties of non-autoclaved aerated concrete. A study into the effect of modification of the solid component, with the use of active mineral additives, would contribute to resolving this task.

2. Literature review and problem statement

The construction and operating properties of cellular concretes are largely defined by the degree of saturating them with air pores. Taking into consideration the specific properties of cellular concretes and the interdependence between strength and mean density, there are certain difficulties when comparing structural indicators of cellular concretes for density. Most often, the strength and density of cellular concretes are characterized by a coefficient of constructive quality, which is adopted by experts to simplify the comparison of materials [1]. There are also other methods for comparing the quality of cellular concretes of different mean density. For example, authors of [2] suggest using an indicator of the reduced height for comparing the bearing capacity of cellular concretes of different density. Paper [3] reports a procedure for obtaining aerated concrete with predictable durability, which takes into consideration the mean density of aerated concrete in the dry state, as well as the strength of microporous cement stone and its mean density [4]. The above examples are based on the statistical treatment of experimental information and do not make it possible to fully estimate the impact of mean density on a change in the strength of cellular concrete.

Some researchers point to the effect of differential parameters of pores (pore size, shape of the pores, the distribution of pore size in a material's volume) on the properties of cellular concretes [5, 6]. However, the authors note that it is difficult to establish a clear relationship between the strength at compression of control samples and the mean size of macropores. In addition, the above studies did not take into consideration the general character of the structure of cellular concrete.

Recent years have seen a large body of research whose authors attribute the properties of cellular concretes to their structure [7, 8]. Paper [6] proposed a hypothesis on that the properties of cellular concretes are determined by the character of distributions of a solid phase (intraporous partitions). In turn, it is possible to change the character of intraporous partitions in the manufacture of cellular concrete through formulation-technological factors, including the modification of the solid component.

Progress in the materials science of composite materials based on binders with mineral additives contributes to deeper studies into the chemical aspects of hydration of non-autoclaved aerated concretes [9, 10]. From a practical point of view, it can be associated with difficulties related to determining the amount and strength of coagulation and crystallization phase contacts between particles of the solution mixture [11]. The pattern that is associated with the effect of modification of the solid component of non-autoclaved aerated concrete significantly changes the mechanism and kinetics of processes of hydration of clinker minerals.

To overcome this problem, the research examined the influence of modification of the solid component on the properties of non-autoclaved aerated concrete. It is proposed to use, as modifiers of the solid component, a lime-carbonate additive containing calcium carbonate (calcite), calcium hydroxide (portlandite), as well as additives with a plasticizing and accelerating effect. The comprehensive action of the additive is due to the influence of crystallochemical factors – epitaxial merging between $Ca(OH)_2$ and the surface of a limestone filler with the formation of a series of compounds of calcium bicarbonate $CaCO_3{\cdot}Ca(OH)_2{\cdot}H_2O$, which strengthens bonds between crystals of portlandite and calcite [12, 13]. Despite the practical significance of such results, the authors failed to consider in detail the chemical and physical-chemical processes of hydration of the integrated additive. Obviously, this is due to the complexity in determining the indicators for properties of concrete at modification transformations [14, 15].

The expediency of using calcium carbonate as a finely dispersed microfiller is predetermined by the granulometric compatibility with grains of cement. The size of calcite particles can be compared to the size of capillaries in cement stone, and the character of the distribution curve of particle size matches the region of optimal grain composition of fillers in aerated concrete. Therefore, the authors assumed that the introduction of chalk in the amount not exceeding 8 % by weight of cement would contribute to the formation of a strong crystalline contact (frame) of cement stone and to compressing the hardening system [16].

When making cellular concrete, it is appropriate to apply mineral fillers with moderate water demand and elevated surface activity, ensuring a long-term increase in strength, especially at a moderate content of the clinker component [17]. Decisive for quality formation of cellular concrete is the technological stage.

Based on the stagewise formation of the structure of non-autoclaved aerated concrete, we defined the formulation-technological factors whose choice is predetermined by the purpose and objectives of research. Patterns in the process of influence of the solid component of cellular concrete, modified by the lime-carbonate finely dispersed filler additive, as well as additives with a plasticizing and accelerating effect, should be defined by using mathematical-statistical methods of experiment design [18]. A set of constructed mathematical models for the properties of aerated concrete would make it possible to reveal the impact of variable factors on the character of the structure and properties of non-autoclaved aerated concrete. Based on these patterns, it would be advisable to choose the technological methods and modes of production of aerated concrete with the predefined properties. The main part of the current research was conducted with the use of methods of mathematical statistics.

Therefore, there are grounds for conducting research into the influence of modification of the solid component of non-autoclaved aerated concrete on a change in the character of its structure and basic properties.

3. The aim and objectives of the study

The aim of this study is to improve the strength of non-autoclaved aerated concrete by revealing and implementing the patterns that accompany the process of modification of the solid component of non-autoclaved aerated concrete.

To accomplish the aim, the following tasks have been set:

– to determine the water demand of the mortar component, which would ensure its maximum strength and contribute to obtaining a stable homogenous structure of aerated concrete;

– to define patterns in the influence of the solid component of non-autoclaved aerated concrete, modified with a lime-carbonate finely dispersed filler, as well as additives with a plasticizing and accelerating effect on the basic properties of non-autoclaved aerated concrete using the mathematical-statistical methods of research.

4. Materials and methods to study the influence of modification of the solid component of non-autoclaved aerated concrete

4. 1. Characteristics of raw materials used in experimental study into the influence of modification of the solid component

Portland cement, grade PCI-500; fly ash; blast furnace, granulated (specific surface – 1,500 cm^2/g by the Blaine device; chalk, natural, milled, enriched, brand MMC-2, residue on sieve 014-0.0047 %; hydrated lime (dry lime). The chemical additives used are: superplasticizer of organic origin, brand SP-1 ("Polyplast", Russia); calcium chloride, technical ($CaCl_2$); sodium hydrate, technical (NaOH); gas-forming additive, brand GPB-1.

4. 2. Procedure for conducting experiments and tests of aerated concrete

The object of research selected was a mortar component and non-autoclaved aerated concrete with a density of 500 kg/m^3. The aerated concrete was made in a blade stirrer (volume 5 liters) for the preparation of a cement mortar according to DSTU EN 196-1. To stir the aerated concrete, the stirrer's blades rotation was set to low speed. The blade of the stirrer that rotates around its own axis was set, using an electric motor, into planetary motion around the axis of the plate. The speed of the blade rotation around its own axis was 140±5 min^{-1}; the planetary movement around the axis of the plate was 62±5 min^{-1}. Both motion directions are opposite. A mortar component was prepared providing for its "strength" at the technological stage of 220 mm, by a Suttard viscometer. The method implies, according to DSTU B V2.7-82, determining the amount of water needed for the manufacture of a mortar component of standard consistency; it flows from the cylinder when it is elevated to a height of not less than 100 mm, and spreads on the glass. The mixing water temperature was not less than 35 °C. Upon obtaining the required consistency, the mortar component was molded into molds in the form of prisms with a cross-section of 40×40 mm and a length of 160 mm, according to DSTU EN 196-1. A second part of the mortar, at a fixed water demand, as used for the preparation of aerated concrete with the addition of a gas-forming additive. The total cycle of aerated concrete preparation took five minutes; it was followed by pouring it into molds in the form of cubes with dimensions of sides 100 mm. Based on the matrix of experiment design, according to the standard plan by Box-Benkin of type B-3, we fabricated six samples for the mortar component and aerated concrete in accordance with [18]. The samples were kept under normal humidity conditions for 7 and 28 days. The main indicators for the properties of aerated concrete that were determined in our experiment were the following: water demand of the mortar component, the mean density of aerated concrete (ρ) and its compressive strength ($f_{c,cube}$). The indicators for the properties of concrete were determined according to the requirements from normative documents: mean density according to DSTU B V.2.7-170, compressive strength according to DSTU B V.2.7-214 [19, 20].

To study the influence of modification of the solid component on the properties of non-autoclaved aerated concrete, we carried out a three-factor experiment substantiating the selected variable factors and the levels in their variance. By changing the factors affecting the properties of cellular concrete within the predefined limits, we conducted experiments and processed the results obtained, which characterized its properties. When processing the results, we calculated the regression coefficients values, based on which we established the analytical dependence between variable factors and the examined properties of non-autoclaved aerated concrete. At the technological stage, we set the task on finding an opportunity to improve the strength of non-autoclaved aerated concrete.

Complete data on variable factors, their levels, and intervals of variance, are given in Table 1.

Table 1

Factors, levels, and intervals in their variance

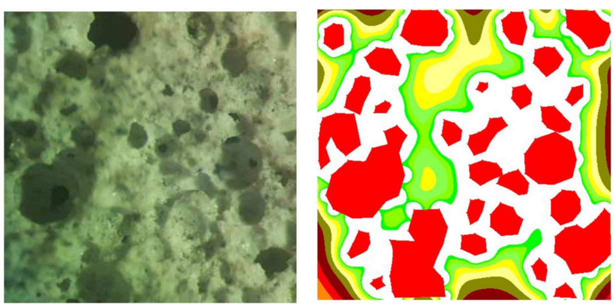
Factor's natural form	Factor code	Measurement unit	Variance levels			Variance interval
			–1	0	+1	
X_1-content of calcium carbonate (calcite)	C	%	5	6.5	8	1.5
X_2-content of calcium hydroxide (portlandite)	P	%	0	2	4	2
X_3-content of calcium chloride	CC	%	0	0.30	0.60	0.30

Constant factors: mean density of aerated concrete – 500±10 kg/m^3, diameter of the spread of a mortar component based on a Suttard viscometer – 220 mm.

4. 3. Procedure for processing the results of experiments

To solve the task on determining the influence of the formulation-technological factors on the physical and mechanical properties of non-autoclaved aerated concrete, we applied statistical methods of experiment design processing the results by using the computing graphical software Microsoft Excel [18]. We built mathematical models and the single-factor dependences of water-solid ratio of the mortar component, the equipotential field tension and compressive strength of aerated concrete in the system COMPEX. 3D charts of isosurfaces mapped their mutual influence. To study polymorphic transformations in cellular concrete based on the modified solid component, we used X-ray phase analysis in line with DSTU B A.1.1-8. X-ray phase analysis is based on acquring and studying a diffraction pattern, which occurs as a result of interference of x-rays scattered by electrons of atoms in the irradiated object. Polymorphic transformations were determined by using the diffractograms of powder samples of cellular concrete and registered at the diffractometer DRON-4-07 under conditions of nickel-filtered Cu K_α (λ=1.5418A) irradiation and at angular interval 2Θ from 5 to 80°. A quantitative indicator for the structure of aerated concrete was chosen to be the equipotential field strength. This characteristic predicts the emergence of the inner surface of boundary between two adjacent pores. To calculate the equipotential field strength, we selected samples of aerated concrete in each line of the experiment, cleaned the surface, and, by using a camera and a microscope, we photographically registered the structure of aerated concrete [21–23].

The sequence of processing an image of the surface of aerated concrete and its computer processing are shown in Fig. 1.



a *b*

Fig. 1. Computer processing of the surface of aerated concrete samples: *a* — photograph of a sample surface; *b* —equipotential fields

First, a photograph of the sample is converted to a monochrome form for removing its defects and to facilitate its software processing. Second, by using the software we determine the position of the internal interfaces in the solid phase of aerated concrete. Finally, the equipotential fields are constructed and the intensities are defined (Fig. 1, *b*). A given characteristic makes it possible to quantify the character of a material's structure.

The accepted raw materials and procedures for processing results from the experiment have made it possible to solve the set tasks and to achieve the goal of the current research.

5. Results of experimental study into the influence of modification of the solid component on the properties of non-autoclaved aerated concrete

At the initial stage of research, we determined the impact of water demand by a mortar mixture on a change in the strength of the solidified mortar component and in the character of structure of the aerated concrete mixture. In the formation of structure of aerated concrete, it is necessary to ensure the synchronous progress of processes for gas evolution and acquiring structural strength. To meet the first condition, it is necessary to ensure that the reaction of gas production should proceed maximally effectively, which is enabled by the defined rheologic characteristic for the mortar component and the alkalinity of environment. As regards the second condition, it is necessary to accelerate hydration of the mortar component by forming the structural strength of aerated concrete.

To study the influence of water demand by a mortar mixture on its strength, we conducted, according to the research objective, an experiment with different W/P ratios, from 0.40 to 0.48. The results showed that on day 28 of hardening the largest strength of the solidified mortar component is 33.5 MPa at W/P 0.42–0.44 (220 mm by a Suttard viscometer). At such a water demand by the mortar component aerated concrete demonstrates a stable homogeneous structure. Therefore, in a subsequent study, the water demand by the mortar mixture was fixed at a constant level and amounted to 220 mm by a Suttard viscometer. At the technological stage, we determined the following physical-mechanical characteristics: strength at compression of aerated concrete on days 7 and 28. We also defined influence of the examined factors on a change in the character of structure of cellular concrete. The chosen structure parameter was the equipotential field strength. The result of our experiment is the built mathematical models of the investigated properties:

$$\frac{W}{P}=0{,}39+0{,}02X_2-0.03X_3-0.02X_{11}^2+0.01X_{33}^2,$$

$$R^{28}=3{,}0+0{,}16X_1-0.06X_2+0.08X_3+0.07X_{12}-$$
$$-0.15X_{13}+0.05X_{23}-0.03X_{11}^2+0.07X_{22}^2+0.2X_{33}^2,$$

$$T=77-5.4X_1-3.4X_2-3.6X_3+9X_{12}+$$
$$+4.5X_{13}+9.5X_{23}+3X_{11}^2-3X_{22}^2+X_{33}^2.$$

Mathematical models are adequate by the Fisher criterion at a 5 % error in the experiment. Based on mathematical models, graphic dependences have been constructed.

The reported single-factor dependences reflect the impact of variable factors on the strength at compression of aerated concrete. In Fig. 2, the upper line reflects the influence of factors on the properties of concrete in the region of maximum values, and the bottom line – minimal. Dependences testify to that the introduction of calcium carbonate (calcite), variable factor X_1, exerts the greatest influence on the strength of aerated concrete. Increasing the content of calcium carbonate linearly leads to a higher strength (Fig. 2, *a*). The introduction of calcium chloride $CaCl_2$, factor X_3, in the amount of 0.3 % by weight of cement leads to a decrease in the strength of cellular concrete (in the region of maximum values). When adding a larger amount of the hardening accelerator to the structure of concrete, strength is growing slowly (Fig. 2, *b*). The introduction of calcium hydroxide (portlandite) $Ca(OH)_2$, factor X_2, in the region of maximum values for strength almost does not affect the strength of aerated concrete. In the region of minimum values, there is a slight decrease in strength (Fig. 2, *c*).

Fig. 3 shows in a three-dimensional formulation field the isosurfaces of compressive strength of aerated concrete. When analyzing the obtained results, it should be noted that the strength of aerated concrete increases to 3.47 MPa when using the maximum amount of calcite (up to 8 %) regardless of the presence of portlandite across the entire range of its consumption (0–4 %). At the maximum amount of 0.6 % of the hardening accelerator in aerated concrete, its compressive strength grows to 3.37 MPa provided a total absence of portlandite, or when adding it in the maximum amount of 4 %. At the content of calcite of 8 % without the presence of a hardening accelerator the strength of aerated concrete also increases to 3.53 MPa. A similar event regarding an increase in strength occurs when the content of a hardening accelerator in aerated concrete is 0.6 % and that of calcite is 5 %.

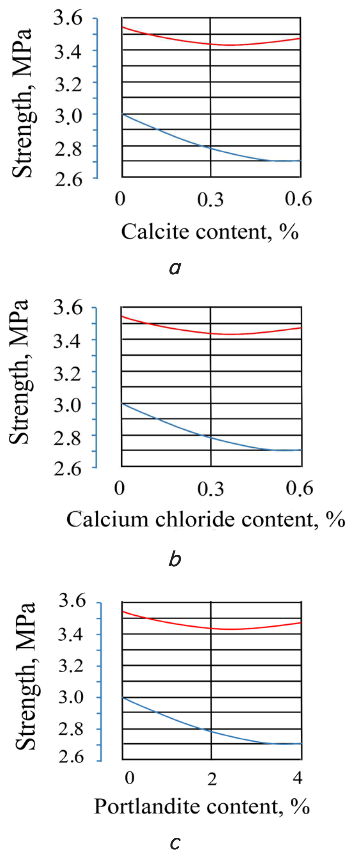
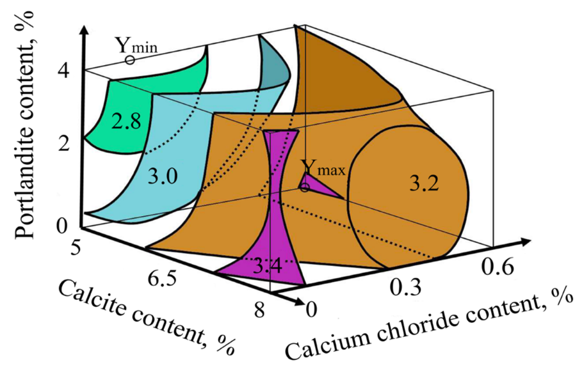


Fig. 2. Influence of formulation factors on strength of aerated concrete: *a* — calcium carbonate; *b* — calcium chloride; *c* — calcium hydroxide

Thus, the greatest strength at compression of 3.53 MPa is demonstrated by the composition of aerated concrete with a maximum content of calcite and portlandite in the absence of a hardening accelerator. The resulting aerated concrete, in terms of the mean density of 500 kg/m^3, corresponds to the class of cellular concrete C2.

In addition, we determined the dependence of equipotential field intensity, which is an indicator for the character of its structure, on strength of the resulting aerated concrete (Fig. 4).

The physical-chemical methods that were used to investigate the phase composition of aerated concrete included an X-ray phase analysis, the results of which established the polymorphic transformations of aerated concrete with a modified solid component. Based on a diffraction pattern, we managed to establish the arrangement of atoms in space – the structure of crystals, to decipher the structure of complex compounds, which were recorded in the form of radiographs shown in Fig. 5.



Y_{min}=2,7

X_1= –1; X_2=1; X_3= –0,66

Y_{max}=3,43

X_1= –1; X_2=1; X_3=1

Fig. 3. Mutual influence of variable factors on compressive strength of aerated concrete

Fig. 5 shows the radiographs of compositions of aerated concrete based on a lime-carbonate finely dispersed filler (No. 4) with the addition of a hardening accelerator (No. 5). For comparison, there is also the radiograph of aerated concrete with the addition of crushed blast furnace slag (No. 8).

Characteristic of the crystalline phases of non-autoclaved aerated concrete that contains a lime-carbonate filler (composition No. 4) is represented by calcium carbonate ($CaCO_3$), vaterite – μ-form $CaCO_3$, and aragonite – a metastable form of $CaCO_3$, as well as new structures in the form of a tobermorite gel and the increased amount of an amorphous component and a small amount of unidentified phase (phases) [24, 25].

The non-autoclaved hardened aerated concrete was industrially implemented at the production facilities of LLC "NEW Building Materials". in Kyiv oblast, Obukhyv region, village Krasne-Pershe (Ukraine).

The resulting structural thermal-insulating aerated concrete of non-autoclaved hardening with a mean density of 500 kg/m^3 meets the requirements from DSTU B V.2.7-45 in terms of physical and mechanical properties.

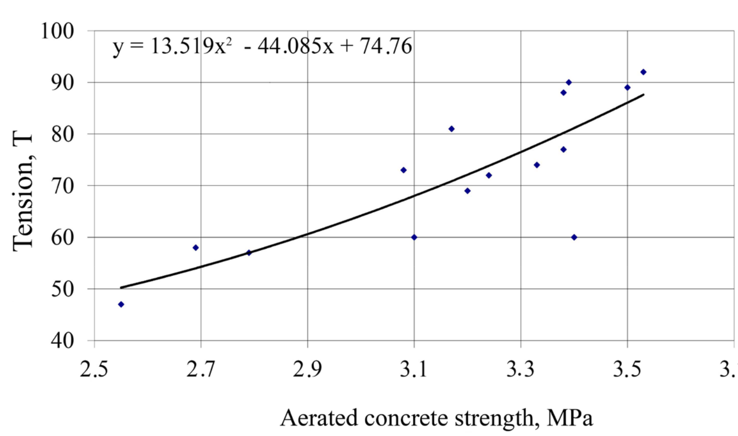


Fig. 4. Dependence of equipotential field intensity on strength of non-autoclaved aerated concrete

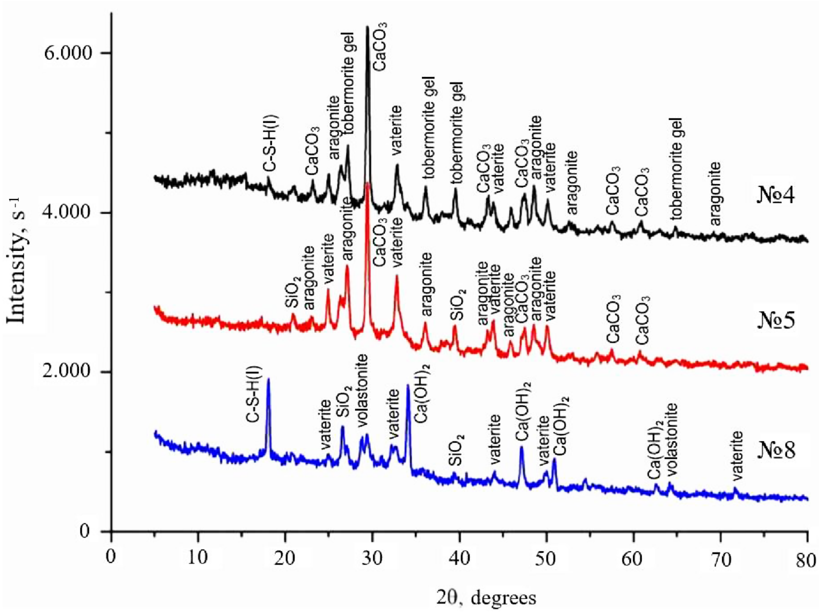


Fig. 5. Radiographs of cellular concrete compositions

6. Discussion of results of studying the influence of modification of the solid component of non-autoclaved aerated concrete on its properties

Our research has found that obtaining aerated concrete of improved strength is predetermined by a change in the character of structure of the solid phase. The change in structure occurs under the influence of crystallochemical factors – the epitaxial merging between $Ca(OH)_2$ and the surface of a limestone filler. This is accompanied by the formation of compounds of calcium bicarbonate $CaCO_3{\cdot}Ca(OH)_2{\cdot}H_2O$, which strengthens bonds between crystals of portlandite and calcite. This is confirmed by a set of physical and chemical methods of research into the phase composition of aerated concrete using an x-ray diffractometer. The results from identification have shown the presence of a considerable number of stable new structures in non-autoclaved aerated concrete: calcite ($CaCO_3$), and its polymorphic modifications in the form of vaterite – μ-form $CaCO_3$, aragonite and tobermorite gel.

The study was carried out according to a scientific hypothesis on that the properties of cellular concretes depend on the character of structure of the solid phase, that is, intraporous partitions. This hypothesis is different from conventional views that link the dependence of the properties of cellular concretes to the character of porosity. We believe this idea defies logic, since a pore is essentially a void and cannot affect the strength of frame of cellular concrete. The proposed approach necessitates introduction of relevant characteristics for the solid component of cellular concrete and the construction of methods to estimate them. We accepted the equipotential field strength as such a characteristic.

The obtained results of our research have certain limitations in practical use. It is possible to achieve the quantitative indicators for the properties of non-autoclaved aerated concrete, predicted by mathematical models, when using materials with such characteristics that were applied in the process of experimentation. However, this limitation does not apply to the character of influence of variable factors on properties of aerated concrete.

Such constraints are caused by the fact that the current study mostly employed the so-called method of a "black box". When this method is applied, one examines the response of the system to the influence of external factors. More valuable information can be obtained in the framework of a systematic approach. This methodological approach considers an object as a system that consists of a totality of interconnected structural elements. A more profound application of the systematic approach could have established a causal relationship between the character of a material's structure and its properties. Therefore, the further research should employ the framework of a systemic approach, in particular, take into consideration the phenomena of self-organization in the establishment of systems. However, such an approach requires, first and foremost, the selection of structural elements in the system, as well as connections among them, thereby establishing appropriate characteristics and parameters for them. Following this, there is a need to develop methods for quantitative estimation of these parameters (as an example, the intensity of equipotential field).

7. Conclusions

1. We have conducted an experimental research aimed at investigating the impact of water demand by a mortar component on its strength. It was determined that the water/solid ratio of the mortar should not exceed 0.42 to ensure its maximum strength and to obtain aerated concrete with a stable homogeneous structure to avoid the subsidence of the mortar, as well as destructive phenomena in the formation of samples. That was a reason to fix the spread of the mortar component at a constant level of 220 mm in a subsequent study into obtaining non-autoclaved aerated concrete.

2. We have theoretically substantiated and experie mentally confirmed the possibility to improve strength of non-autoclaved aerated concrete through the modification of the solid component. The result from applying the mathematical-statistical methods of research is the obtained non-autoclaved aerated concrete of elevated strength R^{28}=3.53 MPa, which corresponds to the class of cellular concrete C2. The physical and mechanical indicators for aerated concrete were improved by modifying its solid component, whose composition includes a lime-carbonate finely dispersed filler, resulting in the formation of polymorphic modifications of calcium carbonate ($CaCO_3$) and hydro- silicates from a tobermorite group.

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