



Study of the Influence of Hydrothermal Treatment Parameters on the Properties of Lightweight Silicate Bricks Using Aluminosilicate Microspheres and Substandard Clay Rocks

Volodchenko A.A.* ¹ 

¹ Belgorod State Technological University named after V.G. Shukhov, Russia

Abstract. This article investigates the influence of various parameters of hydrothermal treatment on the properties of lightweight silicate bricks obtained using substandard clay raw materials, construction lime and aluminosilicate microspheres. It was found that it is possible to obtain products with the required performance characteristics at a minimum hydrothermal treatment pressure of 0.2 MPa. With an increase in pressure to 0.4 MPa, it is possible to reduce the time of isothermal exposure while maintaining the required properties, which helps to reduce the energy intensity of production. The optimal amount of CaO depends on the specific parameters of hydrothermal treatment. So in order to achieve maximum strength indicators, the content of CaO is 10 wt. % at a pressure of 0.2 MPa and 15 wt. % at a pressure of 0.4 MPa, respectively. The addition of aluminosilicate microspheres makes it possible to significantly reduce the average density and obtain a lightweight silicate brick with this indicator from 930 to 1610 kg /m³. The rational time of isothermal exposure, ensuring the formation of a cementing compound of optimal composition, and as a result, obtaining a material with high physical and mechanical properties at a pressure of 0.2 MPa is 8 hours, and at a pressure of 0.4 MPa is 6 hours. Mathematical models are proposed for the selection and optimization of lightweight silicate brick compositions based on construction lime, substandard clay rocks and aluminosilicate microspheres.

Keywords: silicate bricks, aluminosilicate microspheres, hydrothermal treatment, substandard clay raw materials

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*Corresponding author E-mail: alex-0904@mail.ru

1. INTRODUCTION

In modern construction, wall materials combining both structural and thermal insulation properties are used in the construction of buildings and structures. Such walling materials include widespread high hollow ceramic stones and blocks, hollow silicate products, cellular concrete, etc. [1-4]. All these walling materials have their advantages and disadvantages, and the choice of a particular material depends on the project requirements and construction conditions. However, their common goal is to ensure the strength and thermal insulation of the building, which is an important factor for a comfortable environment and energy efficiency of buildings [5-6]. In the production of such materials, the use of unconventional natural and technogenic raw materials is relevant, which will significantly reduce the cost of production, as well as develop new generation construction composites [7-10].

Silicate products, such as dense blocks and bricks, are made from silica sand and lime-silica binder [11-12]. They have high density and strength, as well as good thermal insulation in the case of multi-hollow products, making them an excellent choice for construction. Increasing the number of voids in silicate products helps to reduce their average density, which simplifies the construction process and improves their thermal performance. Another way to reduce the average density of products is to use lightweight aggregates, such as aluminosilicate microspheres [13-16]. Aluminosilicate microspheres are lightweight and porous materials that can significantly reduce the average density of silicate bricks. The use of aluminosilicate microspheres also contributes to the formation of a strong microstructure of the cementing compound and improves the mechanical properties of the material. To achieve the required properties of silicate materials, it is necessary to subject the product to hydrothermal treatment at high pressures and temperatures. Hydrothermal treatment is a process of heating and holding a material in an atmosphere of saturated water vapor at increased pressure. This process allows to activate the chemical reaction between Ca(OH)_2 and crystalline SiO_2 , which leads to the formation of a strong cementing compound based on oxidized calcium hydrosilicates [17-18]. The degree of their crystallization, as well as their composition can be influenced by the pressure and time of hydrothermal treatment. It should be noted that the use of quartz sand as a raw material determines high energy intensity of production, and lower autoclaving pressure will contribute to obtaining products with low performance indicators. The use of substandard clay rocks instead of the traditionally used quartz sand, characterized by the presence of various mixed-layer clay minerals, fine particles of quartz sand, X-ray amorphous phase allows to obtain products at reduced pressure values of hydrothermal treatment while maintaining the required properties of silicate products based on them [19-20].

The nature of the effect of hydrothermal treatment on the properties of silicate bricks using traditional raw materials is well enough studied. At the same time, the use of other raw materials can change the nature of this influence on the processes of structure formation [21-24]. In this regard, it is necessary to investigate the influence of hydrothermal treatment parameters on the properties of silicate bricks in each specific case, including the use of raw materials with variable composition.

Thus, the study of the influence of hydrothermal treatment parameters on the properties of lightweight silicate bricks produced on the basis of substandard clay rocks and aluminosilicate microspheres is an urgent task, as it will optimize the production process and create a material with the required performance characteristics on the basis of these types of raw materials.

2. METHODS AND MATERIALS

In the experiment to study the effect of hydrothermal treatment parameters on the properties of lightweight silicate bricks we used substandard clay rock of the Kursk Magnetic Anomaly (KMA) region as the main component. To reduce the average density of silicate bricks, hollow aluminosilicate microspheres were used as a light chemically active aggregate. Construction lime of 1st grade was used as a binding agent.

The method of obtaining lightweight silicate bricks consists in preliminary obtaining a raw material mixture by mixing in the required proportions of building lime (CaO), substandard clay rock and aluminosilicate microspheres with their subsequent moistening. The obtained raw material mixture is placed in a sealed chamber in order to quench CaO . After the quenching process, further moistening of

the raw material mixture is carried out to achieve the necessary moisture for molding the samples. In this case, the molding moisture is 10 wt. %. The next step is to fabricate the samples by pressing, in which the raw material mixture is subjected to a pressure of 20 MPa. After that, the formed samples are subjected to hydrothermal treatment, which takes place at certain parameters including:

- water vapor pressure: 0.2 and 0.4 MPa, corresponding to temperatures around 120 and 144 °C, respectively;

- isothermal curing time: 2, 4, 6 and 8 h;

- temperature set and reset time – 1.5 h.

After completion of hydrothermal treatment, the samples are cooled and kept for 3 days in normal conditions. After that they are tested in accordance with the requirements of normative documents (GOST 8462 and GOST 7025).

Substantial analysis of raw components, as well as the composition of the formed cementing substance in the samples was carried out by X-ray phase analysis. The microstructure was studied by scanning electron microscopy.

3. RESULTS AND DISCUSSION

To obtain lightweight silicate bricks at different parameters of hydrothermal synthesis, substandard clay rock from the KMA region was used as the main active component. The used rock has plasticity number 6 and belongs to loamy sand. Its composition is dominated by mixed-layer minerals, finely dispersed quartz, and X-ray amorphous phase, which is confirmed by the data of X-ray phase analysis (Fig. 1). Rocks of similar composition are widespread not only in the KMA region, but also in other regions of Russia.

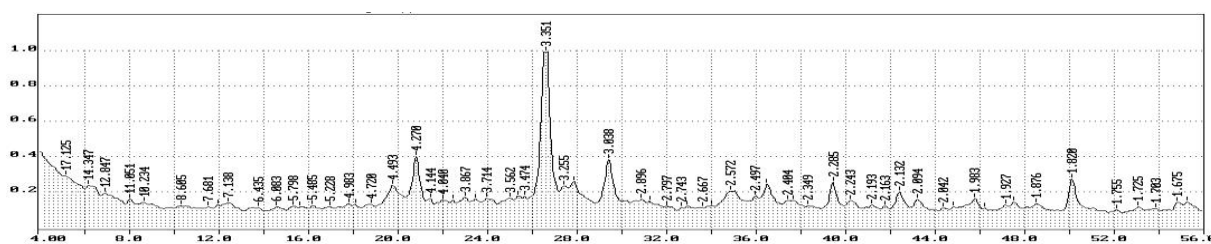


Fig. 1. X-ray pattern of the used substandard clay rock.

Aluminosilicate microspheres (AM) are a chemically active porous aggregate that can significantly affect the properties of lightweight silicate bricks. The AMs used in this work have a bulk density of 350-450 kg/m³ and an average particle radius in the range from 10 to 250 microns. The microstructure of the AM used in the work is shown in Fig. 2.

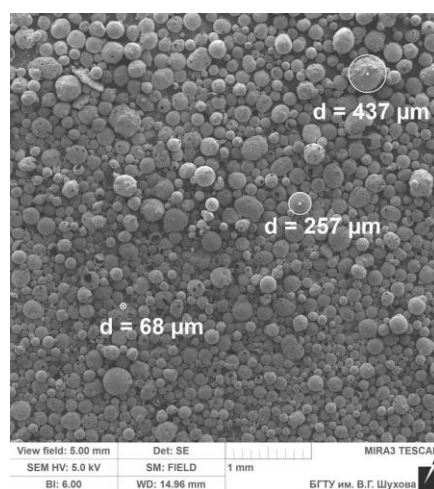


Fig. 2. Microstructure of the used aluminosilicate microspheres.

The objective of this research was to study the parameters of hydrothermal treatment (HT) on the properties of lightweight silicate bricks using substandard clay raw materials, construction lime and aluminosilicate microspheres.

To solve this problem, the compositions of raw mixtures with the content of construction lime (CaO) in the amount of 10-20 wt. % were developed; aluminosilicate microspheres (AM) in the amount of 10-60 wt. %; substandard clay rock – the rest.

It should be noted that different CaO content in the mixture at different parameters of hydrothermal treatment can differently affect the final properties of composites due to different chemical activity of components at different values of pressure and temperature in the process of hydrothermal treatment.

Proceeding from the analysis of the results of the obtained data it is established that at CaO content of 10 wt. % and HT pressure of 0.2 MPa (Fig. 3, a) the samples reach the maximum strength of 10.3-15.7 MPa (AM content of 10-60 wt. %) at isothermal curing time of 8 h (Fig. 3, a, curve 4), and at 6 h of isothermal holding time this index is 9.4-14.9 MPa, which is lower in comparison with the samples obtained at 8 h of isothermal curing time.

Isothermal curing (IC) for 2 and 4 h (Fig. 3, a, curves 1 and 2) provides the lowest strength parameters of the products in comparison with the samples obtained at the time of isothermal curing 6 and 8 h. So the strength of samples at isothermal curing time of 2 h and AM content of 10-60 wt. % is 7.6-11.4 MPa, which is 26-27 % less in comparison with samples obtained at isothermal curing time of 8 h. Samples obtained at isothermal curing time of 4 h and equal AM content have a strength of 8.5-13.8 MPa.

Samples containing 10 wt% CaO and obtained at hydrothermal treatment pressure of 0.4 MPa reach the maximum strength (10.4-21 MPa) at isothermal curing time of 6 h (Fig. 3, b, curve 3). Increasing the time of IC up to 8 h contributes to obtaining samples with lower strength (10.6-16.5 MPa) in comparison with samples obtained at the time of IC 6 h (Fig. 3, b, curve 4). In this case, the decrease in the strength of samples at the time of isothermal curing more than 6 h is probably due to the fact that due to the longer interaction of AM with calcium hydroxide in the microstructure of composites formed a greater number of gel-like new formations in relation to weakly crystallized hydrosilicates, which leads to a decrease in strength at increasing the time of isothermal curing. At the same time, when using lower HT pressure (0.2 MPa), the samples have the highest strength at the time of IC 8 h, which may be due to the lower temperature of hydrothermal synthesis, and as we know temperature and pressure are some of the important factors affecting the chemical reaction and structural changes.

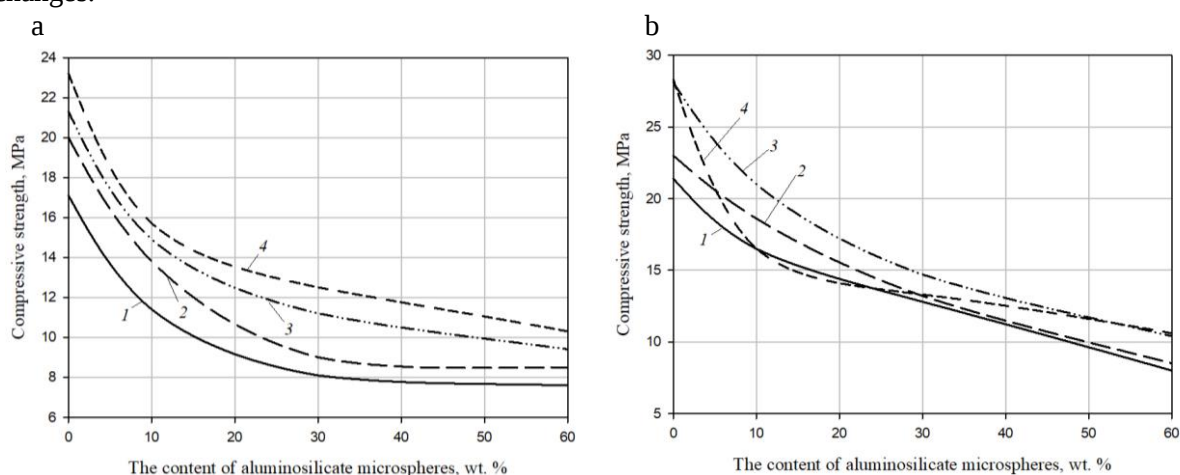


Fig. 3. Change in the compressive strength of samples with different content of aluminosilicate microspheres and CaO content of 10 wt. % depending on the pressure of hydrothermal treatment (a – 0.2 MPa; b – 0.4 MPa) and the time of isothermal curing: 1 – isothermal curing of 2 hours; 2 – isothermal curing of 4 hours; 3 – isothermal curing of 6 hours; 4 – isothermal curing of 8 hours.

Therefore, lower HT pressure leads to less intensive chemical interaction between the components of the whole system, which in turn can affect the formation of the microstructure of the final product, and as a consequence, its properties.

The samples at minimum isothermal curing time of 2 h (Fig. 3, b, curve 1) have a compressive strength value of 8-16.5 MPa (AM content 10-60 wt. %).

Thus, the obtained experimental data showed that the samples with the addition of 10 wt% of CaO and obtained at a pressure of 0.4 MPa at all modes of isothermal curing possess the necessary strength indices, which is explained by the formation of the necessary strong bonds in the microstructure of the material.

The analysis of samples containing 15 wt. % CaO shows that at the pressure of 0.2 MPa (Fig. 4, a) the samples have a lower compressive strength index in comparison with the samples containing 10 wt. % CaO (Fig. 3, a), including at different isothermal curing times. The maximum strength of 10.1-14.9 MPa (AM content 10-60 wt. %) is reached by the samples at the duration of isothermal curing 8 h (Fig. 4, b, curve 4). Reducing the time of isothermal curing to 6 hours reduces the strength of the samples depending on the composition by 7-10 %. AM content in composites in the range from 10 to 60 wt. % contributes to obtaining samples of lightweight silicate bricks with compressive strength values up to 8-11.2 MPa. However, the lowest strength values (7.6-11.4 MPa) are observed in the samples obtained by isothermal curing for 2 hours.

At a pressure of 0.4 MPa, samples containing 15 wt. % CaO reach a maximum strength of 12.4-22 MPa at isothermal curing time of 6 h (Fig. 4, b, curve 3). Moreover, this index is slightly higher than that of samples containing 10 wt. % CaO (Fig. 3, b, curve 3). Further increase of isothermal curing time up to 8 h reduces the strength of samples (AM content 10-60 wt. %) by 3-17 % in comparison with samples obtained at isothermal curing time of 6 h.

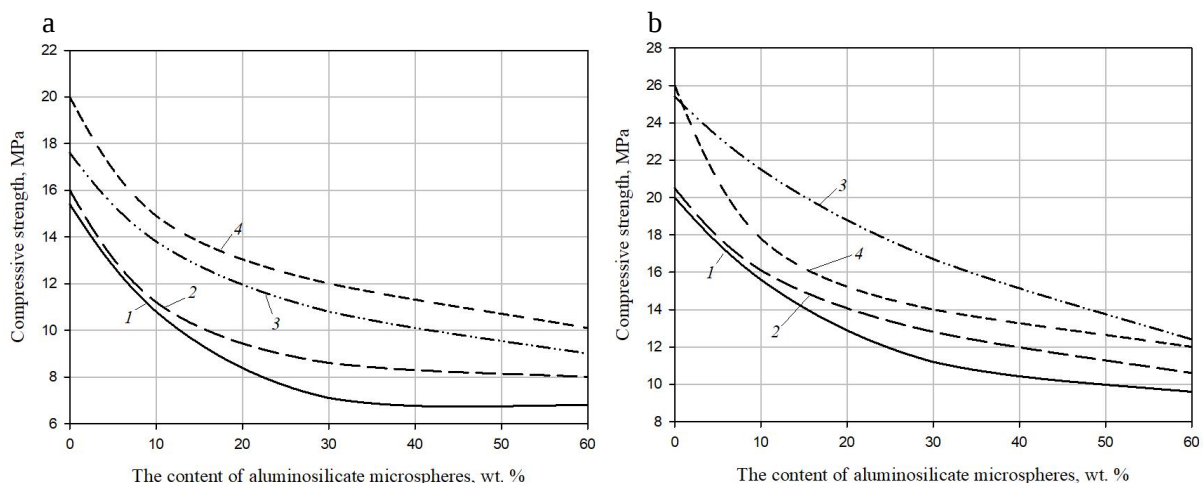


Fig. 4. Change in the compressive strength of samples with different content of aluminosilicate microspheres and CaO content of 15 wt. % depending on the pressure of hydrothermal treatment (a – 0.2 MPa; b – 0.4 MPa) and the time of isothermal curing: 1 – isothermal curing of 2 hours; 2 – isothermal curing of 4 hours; 3 – isothermal curing of 6 hours; 4 – isothermal curing of 8 hours.

It is important to note that when adding 10 wt. % CaO to the raw material mixture and curing for 8 h during hydrothermal treatment, a decrease in the strength of the obtained samples was also observed. The samples obtained at the duration of isothermal curing time of 2 and 4 h have the lowest strength values. In this case, the samples have strengths of 9.6-15.6 MPa and 10.6-16.1 MPa, respectively.

Test results of samples containing CaO in the amount of 20 wt. % are shown in Figure 5. The general nature of the influence of hydrothermal treatment parameters on the properties of samples in this case coincides with samples containing 10 and 15 wt. % CaO.

At HT pressure 0.2 MPa the samples reach the maximum strength at AM content 10-60 wt. % in 9-13.3 MPa (Fig. 5, a, curve 4) at isothermal curing time of 8 h, which is lower in comparison with the samples containing 10 and 15 wt. % CaO by 12-15 % and 10-11 %, respectively (Fig. 3 and 4, a, curve

4). At HT pressure of 0.4 MPa, the samples reach the maximum strength at AM content of 10-60 wt. % at 11.5-17.8 MPa at isothermal curing time of 6 h (Fig. 5, b, curve 3).

The investigated parameters of hydrothermal treatment have not significant influence on the average density of samples. So with increasing isothermal curing time the average density of samples of all compositions increases on average by 1-3 %. Addition of 10 wt. % CaO in the raw material mixture and 10 wt. % AM contribute to the increase in the density of samples. At HT pressure of 0.2 MPa after 2-hour isothermal curing the average density is 1855 kg/m³, and after 8-hour isothermal curing - 1870 kg/m³. At HT pressure of 0.4 MPa, these figures are 1855 and 1880 kg/m³, respectively. A slight increase in the value of average density with increasing duration of isothermal curing is probably due to the formation of a greater number of crystalline new formations, which compact the microstructure and, as a consequence, contribute to the increase in the value of average density.

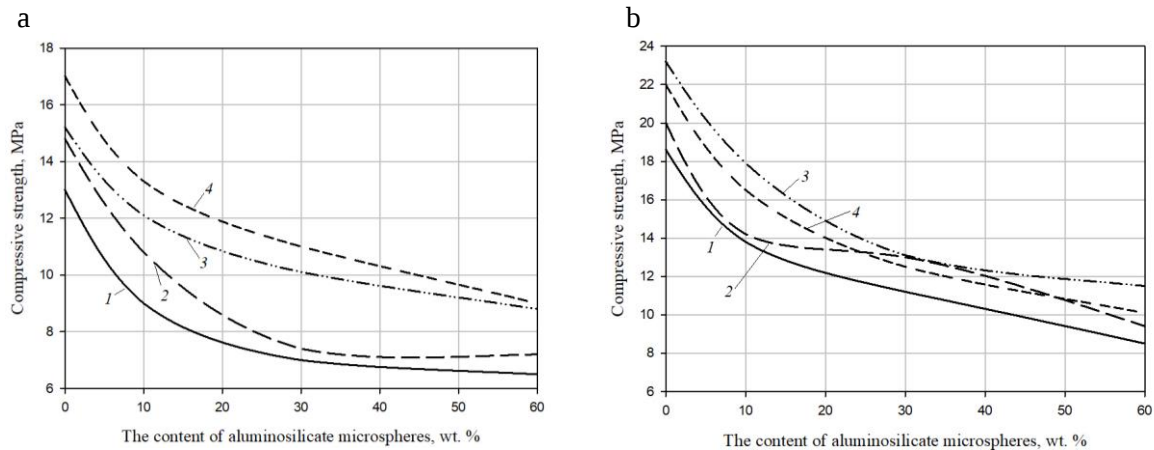


Fig. 5. Change in the compressive strength of samples with different content of aluminosilicate microspheres and CaO content of 20 wt. % depending on the pressure of hydrothermal treatment (a – 0.2 MPa; b – 0.4 MPa) and the time of isothermal curing: 1 – isothermal curing of 2 hours; 2 – isothermal curing of 4 hours; 3 – isothermal curing of 6 hours; 4 – isothermal curing of 8 hours.

Changing the parameters of hydrothermal treatment affects the water resistance index of the samples – softening coefficient. Water resistance of the obtained samples depends on the time of isothermal curing time and pressure of hydrothermal treatment. The best water resistance (softening coefficient 0.68-0.77) possesses the samples obtained at pressure of hydrothermal treatment 0.4 MPa and time of isothermal curing of 6-8 h which are received from raw mixtures containing CaO – 15 wt. % and AM in quantity 10-60 wt. %. Samples of the same composition obtained at hydrothermal treatment pressure of 0.2 MPa have values of softening coefficient in the range of 0.61- 0.71.

Thus, based on the analysis of the obtained data, we can conclude that at a pressure of 0.2 MPa the best water resistance is possessed by the samples obtained at the time of isothermal curing not less than 6 hours. For the samples obtained at a pressure of 0.4 MPa this indicator is not less than 4 h.

According to the results of microstructure analysis of the obtained samples (Fig. 6), it can be concluded that during the curing process under the influence of hydrothermal treatment pressure of 0.2 and 0.4 MPa a crystalline structure is formed, which is low-basic calcium hydrosilicates of CSH (I) type. In addition, there is a uniform distribution of the formed new formations in the composite matrix, which also contributes to the improvement of mechanical properties.

It is worth noting that composites obtained at hydrothermal treatment pressure of 0.4 MPa have better crystallization and formation of stronger joints. At lower pressure (0.2 MPa) weakly crystallized phases are formed.

Using X-ray phase analysis (Fig. 7), it was found that new compounds, mainly represented by low-basic calcium hydrosilicates of the CSH (I) type ($d = 2.8, 3.08, 3.03$ and $2.1 \text{ \AA}$), were formed in lightweight silicate brick samples obtained at different pressures of hydrothermal treatment.

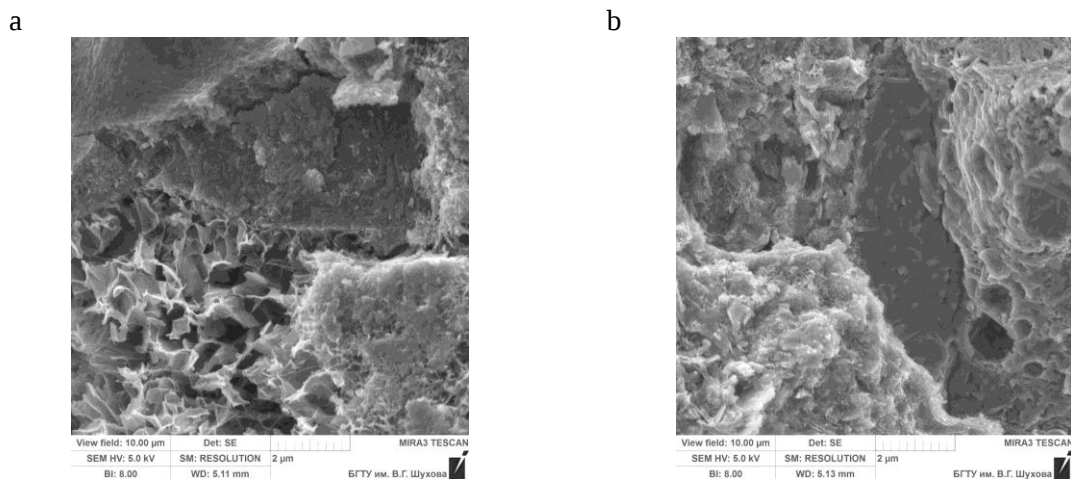


Fig. 6. Effect of hydrothermal treatment pressure on the microstructure of lightweight silicate bricks: a – 0.2 MPa; b – 0.4 MPa

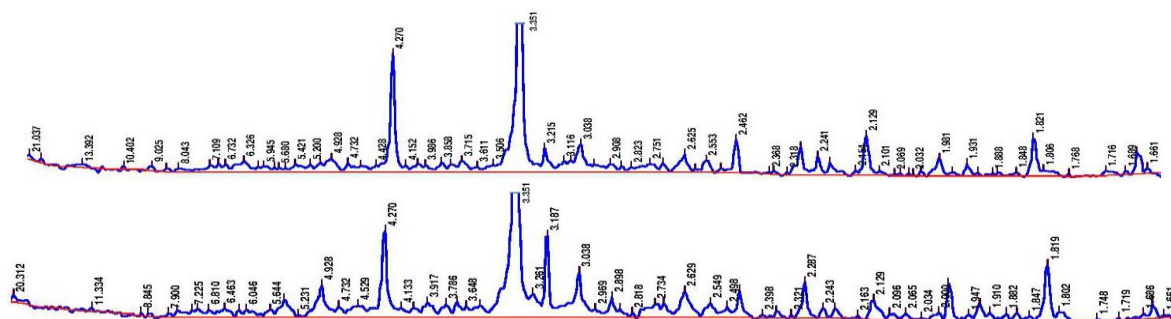


Fig. 7. X-ray images of samples obtained under conditions of hydrothermal synthesis at different pressures: a – 0.2 MPa; b – 0.4 MPa

To obtain lightweight silicate bricks in these studies, three components of the raw material mixture are used in certain proportions and different parameters of hydrothermal synthesis are applied. In the course of the conducted experiment, several factors influencing the final properties of the obtained lightweight silicate bricks (LSB) were changed: the proportions of the components of the raw material mixture and the parameters of hydrothermal treatment. Changing the proportions of the components of the raw material mixture can affect the physical and chemical properties of the resulting LSB. For example, changing the amount of construction lime can affect the strength and average density of the resulting products. The parameters of hydrothermal treatment also affect the properties of LSB. Thus, changing the temperature and time of hydrothermal treatment can affect the microstructure and phase composition of new formations of the obtained LSB, and as a consequence, affect its final properties.

In order to determine the optimal conditions for obtaining the desired properties of lightweight silicate bricks by varying the concentrations of raw material mixture components, pressure and time of isothermal curing during hydrothermal treatment, an experiment was set using the method of mathematical planning of the experiment (Table 1).

In accordance with the planning matrix of the three-factor experiment, a series of laboratory samples were fabricated. The obtained samples were subjected to hydrothermal treatment in a laboratory autoclave at pressures of 0.2 and 0.4 MPa. A series of experiments involving different combinations of these given factors were carried out.

Table 1. Conditions for planning a three-factor experiment.

Factors		Variation levels			Variation interval
Natural form	Coded form	-1	0	+1	
Construction lime (CaO), wt. %	x1	10	15	20	5
Aluminosilicate microspheres, wt. %	x2	0	30	60	30
Isothermal curing time, h	x3	2	5	8	3

The obtained experimental data were subjected to statistical computer processing, which allowed us to obtain mathematical regression equations (1-6). These equations describe the change in the performance indicators of the products, such as compressive strength (R), average density (ρ) and softening coefficient (K). Using these equations, we can carry out the optimization of the lightweight silicate brick production process. Thus, we can determine the influence of each factor on these indicators and find the optimal conditions that will allow us to obtain lightweight silicate bricks with the desired properties.

The regression equations describing the change in performance indicators of the products obtained at hydrothermal treatment pressure (HTP) of 0.2 MPa are as follows:

$$R = 9,56 - 1,405 \cdot x_1 - 4,52 \cdot x_2 + 2,02 \cdot x_3 + 5,503 \cdot 10^{-3} \cdot x_1^2 + 3,23 \cdot x_2^2 + 0,13 \cdot x_3^2 + 0,987 \cdot x_1 \cdot x_2 - 0,287 \cdot x_1 \cdot x_3 - 0,613 \cdot x_2 \cdot x_3 \quad (1)$$

$$\rho = 1237,706 - 41 \cdot x_1 - 396,2 \cdot x_2 + 9 \cdot x_3 + 9,775 \cdot x_1^2 + 143,775 \cdot x_2^2 - 0,225 \cdot x_3^2 + 30 \cdot x_1 \cdot x_2 + 3,75 \cdot x_1 \cdot x_3 + 2,5 \cdot x_2 \cdot x_3 \quad (2)$$

$$K = 0,663 - 0,04 \cdot x_1 + 0,028 \cdot x_3 - 0,013 \cdot x_1^2 + 0,16 \cdot x_2^2 - 0,049 \cdot x_3^2 - 0,02125 \cdot x_1 \cdot x_3 - 0,02125 \cdot x_2 \cdot x_3 \quad (3)$$

Regression equations describing the change in the performance indicators of the products obtained at the hydrothermal treatment pressure (HTP) 0.4 MPa are as follows:

$$R = 14,29 - 1 \cdot x_1 - 6,46 \cdot x_2 + 1,73 \cdot x_3 - 0,423 \cdot x_1^2 + 3,302 \cdot x_2^2 - 1,32 \cdot x_3^2 + 1,13 \cdot x_1 \cdot x_2 - 0,5625 \cdot x_1 \cdot x_3 - 0,762 \cdot x_2 \cdot x_3 \quad (4)$$

$$\rho = 1244,634 - 43,5 \cdot x_1 - 396,75 \cdot x_2 + 10 \cdot x_3 + 8,92 \cdot x_1^2 + 145,1692 \cdot x_2^2 - 3,58 \cdot x_3^2 + 30 \cdot x_1 \cdot x_2 \quad (5)$$

$$K = 0,727 - 0,019 \cdot x_1 + 0,075 \cdot x_2 + 0,04 \cdot x_3 - 0,016 \cdot x_1^2 - 0,006 \cdot x_2^2 - 0,032 \cdot x_3^2 - 0,013 \cdot x_1 \cdot x_2 \quad (6)$$

The analysis of the obtained output parameters using regression equations allows us to evaluate the compliance of the calculated values of the product properties in relation to the experimental data (Table 2). Using the obtained regression equations, the values of product properties were determined based on the used input parameters and then the calculated values were compared with the experimental data. The results show that the calculated values of product properties using the regression equations are quite close to the experimental data. This indicates that the obtained equations are adequate models for describing the dependencies in the process of production of LSBs. Such adequacy of

the models is an important indicator of the success of the study, as it confirms that it is possible to apply the obtained equations to predict the properties of products when the input parameters change in the process of real production. This is of great importance for optimization of production and improvement of product quality.

Table 2. Output parameters.

№	Compressive strength, MPa				Average density, kg/m ³				Softening coefficient			
	Experimental		Calculated		Experimental		Calculated		Experimental		Calculated	
	HTP – 0.2 MPa	HTP – 0.4 MPa	HTP – 0.2 MPa	HTP – 0.4 MPa	HTP – 0.2 MPa	HTP – 0.4 MPa	HTP – 0.2 MPa	HTP – 0.4 MPa	HTP – 0.2 MPa	HTP – 0.4 MPa	HTP – 0.2 MPa	HTP – 0.4 MPa
1	9	10.1	9.11	9.94	1000	1000	999	995	0.58	0.6	0.56	0.6
2	6.5	8.5	6.87	9.13	960	965	969	975	0.58	0.55	0.59	0.52
3	17	22	17.4	22.1	1725	1725	1727	1729	0.7	0.78	0.69	0.78
4	13	18.6	12.7	18.24	1710	1715	1706	1708	0.64	0.7	0.63	0.7
5	10.3	10.6	10.5	10.79	1010	1015	1014	1022	0.61	0.68	0.59	0.67
6	7.6	8	7.13	7.73	1000	1005	998	1002	0.55	0.6	0.54	0.59
7	23.2	28.3	22.7	27.5	1870	1880	1861	1875	0.72	0.82	0.73	0.79
8	17.1	21.4	16.9	21.39	1855	1855	1856	1855	0.55	0.68	0.59	0.71
9	8.75	1305	8.16	12.87	1210	1210	1207	1210	0.65	0.67	0.65	0.69
10	10.1	13.95	10.9	14.87	1280	1295	1289	1297	0.65	0.71	0.65	0.73
11	8.5	11.5	8.27	11.14	993	1000	985	993	0.6	0.6	0.63	0.64
12	16.8	22.95	17.3	24.04	1765	1778	1778	1787	0.76	0.8	0.72	0.79
13	12	14	11.7	14.7	1240	1250	1247	1251	0.61	0.7	0.64	0.73
14	7.1	11.2	7.67	11.23	1230	1230	1229	1231	0.62	0.65	0.59	0.65
15	9.7	14.75	9.56	14.3	1235	1240	1238	1245	0.67	0.75	0.66	0.73
16	9.6	14.8	9.56	14.3	1234	1241	1238	1245	0.66	0.74	0.66	0.73
17	9.8	14.6	9.56	14.3	1236	1239	1238	1245	0.65	0.76	0.66	0.73

Using the results obtained in the course of mathematical planning of the experiment, the influence of the studied factors was analyzed at the hydrothermal treatment pressure of 0.2 and 0.4 MPa, which is significantly lower than in the traditionally used technology of autoclaved silicate brick production, where the pressure of about 1 MPa is used. This was made possible by replacing the commonly used quartz sand with a non-traditional clay raw material. Thus, the optimal choice of hydrothermal treatment pressure will depend on the required performance characteristics of the material under study. If high compressive strength is required, it is recommended to use high-compressed steam (0.4 MPa), as it leads to the formation of new formations of higher degree of crystallization, and as a consequence, contributes to the strength of samples. However, if lower strength and other properties are required, hydrothermal treatment pressure of 0.2 MPa can be used. In practice, it is possible to use HTP more than 0.4 MPa, which will also lead to an increase in the strength of samples. In this research the study of the properties of samples obtained at minimum values of HTP was carried out, in order to optimize the composition of the raw material mixture to achieve the best characteristics of products at minimum HTP, which in the conditions of real production will lead to a reduction in energy intensity of production, including in small and medium-sized businesses, where there is no opportunity to purchase expensive autoclaves.

The isothermal curing time also influences the desired properties of the samples. Thus, the optimum isothermal curing time depends on other factors such as the mass content of CaO and AM. For example, when using a higher hydrothermal treatment pressure and a certain CaO and AM content in the samples, the highest strength is achieved after 6 hours of isothermal curing time (IC). However, a further increase in the IC time may lead to a decrease in the strength of the products. Thus, the results

indicate the need to determine the optimum isothermal curing time for each individual case to achieve the maximum strength of the samples, which can be achieved using the obtained regression equations.

It is important to note that in addition to pressure and curing time, the change in the composition of the raw material mixture also plays an important role in shaping the performance characteristics of lightweight silicate bricks. Therefore, rational selection of the composition and optimization of technological parameters are also an integral part of the production process of such materials.

Using the obtained regression equations, nomograms of dependence of the properties of lightened silicate bricks when changing the studied factors were constructed (Fig. 8).

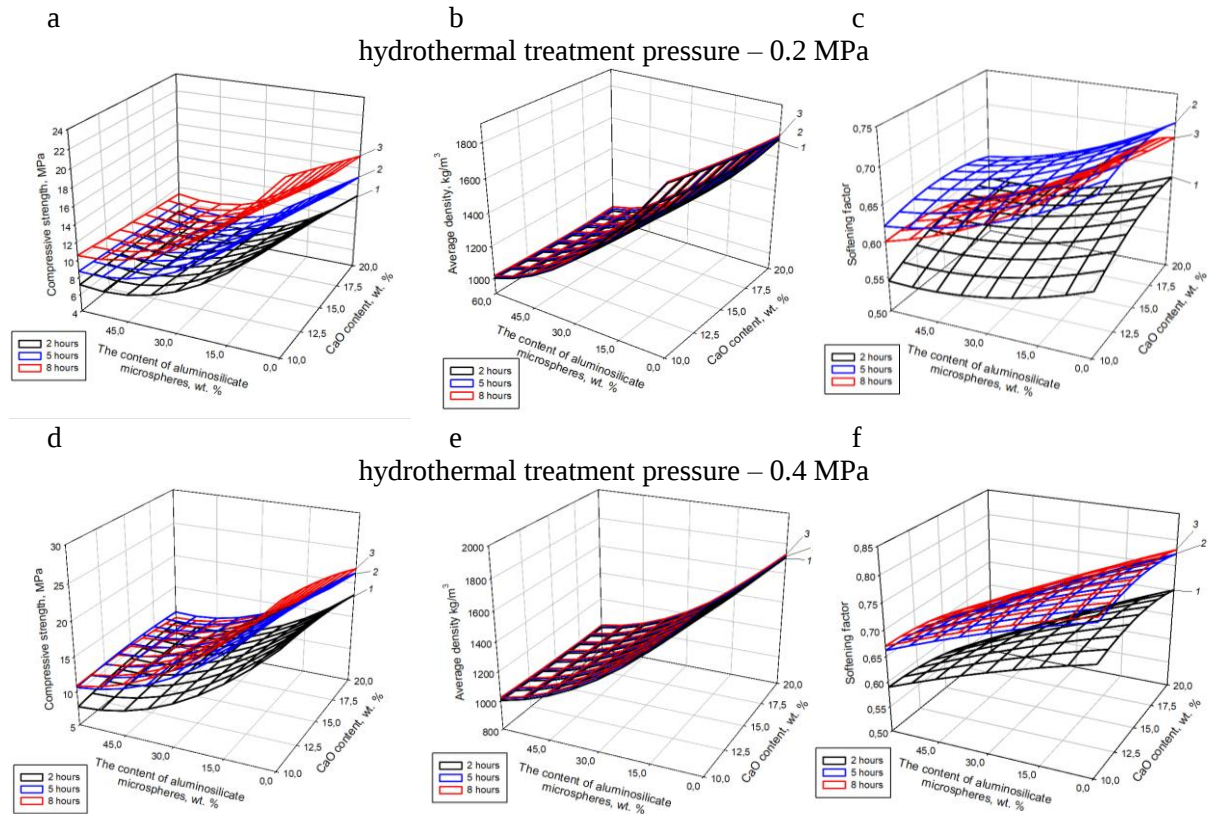


Fig. 8. Nomograms of the dependence of the properties of lightweight silicate bricks (hydrothermal treatment pressure – 0.2 MPa) on the content of lime, aluminosilicate microspheres and the time of isothermal curing (1 – 2 hours; 2 – 5 hours; 3 – 8 hours): a – compressive strength; b – average density; c – softening coefficient.

The obtained nomograms allow us to visually evaluate the influence of hydrothermal treatment pressure and isothermal curing time, as well as the content of CaO and AM on the properties of the samples. At a pressure of 0.2 MPa, the compressive strength of the samples increases with increasing isothermal curing time from 2 to 8 hours (Fig. 8, a). This indicates that longer curing at this pressure contributes to the strength of the samples. The average density does not change significantly depending on the change of input parameters (Fig. 8, b). The greatest water resistance of the samples is achieved at the following parameters of hydrothermal treatment: pressure 0.2 MPa and isothermal curing time of 5 hours (Fig. 8, c).

When the hydrothermal treatment pressure is increased up to 0.4 MPa, the effect of isothermal curing time on the properties of samples changes. The maximum strength of samples at this pressure is reached at isothermal curing time of 6 hours (Fig. 8, d). This confirms the results obtained in this paper and indicates the need to take pressure into account when determining the optimum processing time to achieve the maximum strength of the material, or other properties.

Thus, the analysis of nomograms allows a more detailed understanding of the relationship between hydrothermal treatment pressure, isothermal curing time, CaO content, AM and sample properties.

The obtained regression equations and nomograms are a tool for prediction and optimization of the hydrothermal treatment process to achieve the desired material properties.

4. CONCLUSIONS

Thus, the effect of hydrothermal treatment parameters on the properties of lightweight silicate bricks using substandard clay raw materials, construction lime and aluminosilicate microspheres (AM) has been studied.

It is revealed that it is possible to obtain products with the required performance characteristics under conditions of minimum hydrothermal treatment pressure (0.2 MPa). When increasing the hydrothermal treatment pressure up to 0.4 MPa it is possible to reduce the time of isothermal curing of products, which will reduce the energy intensity of production. Thus, it is possible to obtain lightweight silicate bricks using energy-efficient technology.

The optimum CaO content providing achievement of maximum strength values for samples obtained at hydrothermal treatment pressure of 0.2 MPa is 10 wt. %, and at hydrothermal treatment pressure of 0.4 MPa - 15 wt. %. The content of aluminosilicate microspheres in the range from 10 to 60 wt. % allows to reduce significantly the average density index up to 45%. Thus lightweight silicate bricks will have average density of 930 to 1610 kg/m³.

It was found that the process of formation of cementing compound, and as a consequence, the optimal microstructure of samples is achieved due to good chemical interaction between the initial components at low hydrothermal treatment pressure of 0.2 MPa. Thus at higher pressure (0.4 MPa) better crystallization and formation of stronger compounds occurs. However, at lower pressure (0.2 MPa) weakly crystallized phases are formed. The used aluminosilicate microspheres have high surface activity and as a consequence, the ability to interact with other components of the system. The use of AM, as chemically active porous aggregates, plays an important role in the process of formation of a strong microstructure of cementing compound in the system CaO-SiO₂-Al₂O₃-H₂O, represented by lime and components of substandard clay rock, which consists in the concentration around aluminosilicate microspheres of new formations formed due to the interaction of AM with Ca(OH)₂ that act as centers of crystallization, and predetermine the formation of the microstructure as a whole. This allows to improve the mechanical properties of materials.

Isothermal curing time is also important as it allows to achieve a more complete reaction between CaO and components of the used unconventional clay rock. The optimal time of isothermal curing time, providing the formation of cementing compound of optimal composition, and as a consequence, obtaining a material with high physical and mechanical properties at a pressure of 0.2 MPa is 8 hours, and at a pressure of 0.4 MPa 6 hours. The choice of hydrothermal treatment mode in real production conditions will depend on the needs of the enterprise. If autoclaves have exhausted their resource, the reduce of the hydrothermal treatment pressure will significantly extend their service life, which is especially important in the conditions of import substitution, since most of the operating autoclaves in the country are imported.

Mathematical models for selection and optimization of compositions of lightweight silicate bricks based on construction lime, substandard clay rocks and aluminosilicate microspheres are proposed. With the use of these mathematical models it is possible to select the composition of raw material mixtures to obtain lightweight silicate bricks meeting the requirements of GOST 379-2015 with an average density class of 1.4-1.8 and strength grade M100-150.

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REFERENCES

- [1] Amran M., Onaizi A.M., Fediuk R., Danish A., Vatin N.I., Murali G., Abdelgader H.S., Mosaberpanah M.A. Cecchin D., Azevedo A. An ultra-lightweight cellular concrete for geotechnical applications – A review. *Case Studies in Construction Materials*. 2022. 16. e01096. DOI: <https://doi.org/10.1016/j.cscm.2022.e01096>
- [2] Santa A.C., Gómez M.A., Castaño J.G., Tamayo J.A., Baena L. M. Atmospheric deterioration of ceramic building materials and future trends in the field: a review. *Heliyon*. 9 (4). 2023. e15028. DOI: <https://doi.org/10.1016/j.heliyon.2023.e15028>
- [3] Murugesan P., Partheeban P., Manimuthu Sh., Jegadeesan V., Christopher Ch.G. Multi-criteria decision analysis for optimum selection of different construction bricks. *Journal of Building Engineering*. 2023. 71. 106440. DOI: <https://doi.org/10.1016/j.jobbe.2023.106440>
- [4] Soleymani A., Najafgholipour M.A., Johari A. An experimental study on the mechanical properties of solid clay brick masonry with traditional mortars. *Journal of Building Engineering*. 2022. 58. 105057. DOI: <https://doi.org/10.1016/j.jobbe.2022.105057>
- [5] Lesovik V.S., Fomina E.V. The new paradigm of designing construction composites to protect the human environment. *Bulletin of MSUACE*. 2019. 14 (10). P. 1241 – 1257. DOI: 10.22227/1997-0935.2019.10.1241-1257
- [6] He Yi., Chu Y., Song Ye., Liu M., Shi Sh., Chen X. Analysis of design strategy of energy efficient buildings based on databases by using data mining and statistical metrics approach. *Energy and Buildings*. 2022. 258. 111811. DOI: <https://doi.org/10.1016/j.enbuild.2021.111811>
- [7] Makhortov D.S., Zagorodnyuk L.H., Sumskoy D.A., Al Mamouri Saad. Obtaining binder compositions of optimal compositions based on portland cement and ceramic brick waste. *Bulletin of BGTU named after V.G. Shuhov*. 2022. 7. P. 19 – 30. DOI: 10.34031/2071-7318-2022-7-7-19-30
- [8] Alfimova N.I., Pirieva S.Yu., Titenko A.A. Utilization of gypsum-bearing wastes in materials of the construction industry and other areas. *Construction Materials and Products*. 2021. 4 (1). P. 5 – 17. DOI: 10.34031/2618-7183-2021-4-1-5-17
- [9] Tolstoy A., Lesovik V., Fediuk R., Amran M., Gunasekaran M., Vatin N., Vasilev Y. Production of greener high-strength concrete using Russian quartz sandstone mine waste Aggregates. *Materials*. 2020. 13. 5575. DOI: <https://doi.org/10.3390/ma13235575>
- [10] Klyuev A.V., Kashapov N.F., Klyuev S.V., Lesovik R.V., Ageeva M.S., Fomina E.V. Development of alkali-activated binders based on technogenic fibrous materials. *Construction Materials and Products*. 2023. 6 (1). P. 60 – 73. DOI: 10.58224/2618-7183-2023-6-1-60-73
- [11] Da Silva R.B., Matoski A., Junior A.N., Kostrzewa-Demczuk P. Study of compressive strength of sand-lime bricks produced with coal tailings using mixture design. *Construction and Building Materials*. 2022. 344. 127986. DOI: <https://doi.org/10.1016/j.conbuildmat.2022.127986>
- [12] Nelubova V.V., Strokova V.V. Technology of silicate pressed materials. review of innovations for the development of production. *Construction Materials*. 2019. 8. P. 6 – 13. DOI: 10.31659/0585-430X-2019-773-8-6-13
- [13] Kotlyar V.D., Kozlov A.V., Zhivotkov O.I., Kozlov G.A. Calcium-silicate brick on the basis of microspheres and lime. *Construction Materials*. 2018. 9. P. 17 – 21. DOI: <https://doi.org/10.31659/0585-430X-2018-763-9-17-21>
- [14] Steshenko, A.B., Kudryakov, A.I. Cement based foam concrete with aluminosilicate microspheres for monolithic construction. *Magazine of Civil Engineering*. 2018. 84 (8). P. 86 – 96. DOI: 10.18720/MCE.84.9
- [15] Chen J.J., Ng P.L., Li L.G., Kwan A.K.H.. Production of High-performance Concrete by Addition of Fly Ash Microsphere and Condensed Silica Fume. *Procedia Engineering*. 2017. 172. P. 165 – 171 DOI: <https://doi.org/10.1016/j.proeng.2017.02.045>

- [16] Scott N.R., Stoddard D.L., Nelms M.D., Wallace Z., Turner I., Turner L., Croom M., Franklin K., Sandifer S., Ali Al-fahdi M.S., Butler T., Rajendran A.M. Experimental and computational characterization of glass microsphere-cementitious composites. *Cement and Concrete Research*. 2022. 152. 106671. DOI: <https://doi.org/10.1016/j.cemconres.2021.106671>
- [17] Dachowski R., Komisarczyk K. Determination of Microstructure and Phase Composition of Sand-Lime Brick after Autoclaving Process. *Procedia Engineering*. 2016. 161. P. 747 – 753. DOI: <https://doi.org/10.1016/j.proeng.2016.08.762>
- [18] Kostrzewa-Demczuk P., Stepień A., Dachowski R., Krugielka A. The use of basalt powder in autoclaved brick as a method of production waste management. *Journal of Cleaner Production*. 2021. 320. 128900. DOI: <https://doi.org/10.1016/j.jclepro.2021.128900>
- [19] Volodchenko A.N., Strokova V.V. Development of scientific bases for production of silicate autoclave materials using clay raw materials. *Construction Materials*. 2018. 9. P. 25 – 31. DOI: https://doi.org/10.31659/0585_430X-2018-763-9-25-31
- [20] Volodchenko A.A., Lesovik V.S., Cherepanova I.A., Volodchenko A.N., Zagorodnjuk L.H., Elistratkin M.Y. Peculiarities of non-autoclaved lime wall materials production using clays. *IOP Conf. Series: Materials Science and Engineering*. 2018. 022021. DOI: 10.1088/1757-899X/327/2/022021
- [21] Volodchenko A.N. Reactivity of magnesia clay with lime under hydrothermal conditions. *International Research Journal*. 2014. 10-2 (29). P. 7 – 10.
- [22] Lesovik V., Volodchenko A., Fediuk R., Timokhin R., Mugahed Amran Y.H. Enhancing performances of clay masonry materials based on nanosize mine waste. *Construction and Building Materials*. 2021. 269. 121333 DOI: 10.1016/j.conbuildmat.2020.121333
- [23] Nourredine A., Jauberthie R., Nourredine Ch., Molez L. Formation of C-S-H in calcium hydroxide-blast furnace slag-Quartz-water system In autoclaving conditions. *Advances in Cement Research*. 2014. 27 (3). P. 153 – 162. DOI: 10.1680/adcr.13.00069
- [24] Chen M., Lu L., Wang S., Zhao P., Zhang W., Zhang S. Investigation on the formation of tobermorite in calcium silicate board and its influence factors under autoclaved curing. *Construction and Building Materials*. 2017. 143. P. 280 – 288. DOI: <https://doi.org/10.1016/j.conbuildmat.2017.03.143>

INFORMATION ABOUT THE AUTHOR

Volodchenko A.A., e-mail: susannagrants@mail.ru, tel.: +7(4722) 30-99-42, ORCID ID: 0000-0001-8919-7185, SCOPUS: <https://www.scopus.com/authid/detail.uri?authorId=56450953600>, Belgorod State Technological University named after V.G. Shukhov, Department of Building Materials Science, Products and Constructions, Candidate of Engineering Sciences (Ph.D.), Associate Professor