






## Optimization of the properties of lightweight ceramic products using statistical methods

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### ABSTRACT

**Introduction.** Improving the efficiency of ceramic bricks and blocks involves reducing their average density and corresponding thermal conductivity. One possible approach is to introduce porosity into the ceramic structure using burnable additives or specialized foams during the preparation of the ceramic mixture. Another option is incorporating ultra-lightweight aggregates, particularly crushed foam glass. The aim of this study was to develop a method for determining and optimizing the formulations of lightweight ceramic bricks based on regulatory requirements for strength characteristics, density, and thermal conductivity. **Methods and materials.** The raw materials used included medium-plasticity loam, a fluxing component (finely ground glass waste), and crushed foam glass gravel. The properties of the samples were assessed using standardized methods, while statistical and digital techniques were employed for experimental planning and data processing. **Results.** It was found that the optimal conditions include drying at 80 °C and 55% air humidity, followed by firing at 900–950 °C with the introduction of a fluxing component. The optimal values of the factors were determined as follows: foam glass density: 150 kg/m<sup>3</sup>; foam glass consumption: 30 kg/m<sup>3</sup>; optimal particle size of crushed foam glass: 0.68–0.69 mm; fluxing additive content: 8.3–8.4% of the clay mass. These parameters correspond to the following material properties: Average density: 1320–1360 kg/m<sup>3</sup>; Compressive strength: 15–16 MPa; Thermal conductivity: 0.15–0.16 W/(m·°C); Frost resistance grade: F35. **Discussion.** The use of digital methods revealed the influence of variable factors on the material's properties (strength, density, and thermal conductivity), which was confirmed through model validation in active experiment series and structural analysis of the material. **Conclusion.** This research has established a methodological framework for predicting the properties of ceramic products and optimizing their composition using digital methods. Requirements for optimizing the properties of foam glass based products were determined, including guidelines for the consumption of foam glass and fluxing agents. The resulting materials fully comply with the relevant standards for such products. The lightweight brick exhibits both structural and thermal insulation properties, making it suitable for use as a wall material.

**KEYWORDS:** ceramic brick, crushed foam glass, glass waste, digital optimization, statistical planning

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## Оптимизация свойств легких керамических изделий с применением статистических методов

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### АННОТАЦИЯ

**Введение.** Повышение эффективности керамического кирпича и камней связано со снижением средней плотности изделий и соответствующей теплопроводностью. Одним из возможных путей является поризация структуры керамического изделия с помощью выгорающих добавок или введения специальных пен при приготовлении керамической смеси. Другая возможность связана с введением в структуру особо легких заполнителей и, в частности, дробленого пеностекла. Целью исследований являлась разработка методики определения и оптимизации рецептур легкого керамического кирпича, основанная на нормативных требованиях по прочностным характеристикам, плотности и теплопроводности. **Методы и материалы.** В качестве сырья использовались суглинок средней пластичности и флюсующий компонент (тонкомолотый бой стекла), а также измельченный пеностекольный щебень. Свойства образцов определялись по нормативным методикам, при планировании и обработке результатов эксперимента использовались статистические и цифровые методы. **Результаты.** Установлено, что оптимальными являются сушка при 80 °С и влажности воздуха 55% и обжиг в интервале температур 900–950 °С при условии введения флюсующего компонента. Установлено, что оптимальными являются значения факторов: средняя плотность пеностекла 150 кг/м<sup>3</sup>; расход пеностекла 30 кг/м<sup>3</sup>; оптимальный размер частиц измельченного пеностекла 0,68–0,69 мм; содержание флюсующей добавки (плавня) 8,3–8,4% от массы глины. Этим значениям соответствуют: средняя плотность 1320–1360 кг/м<sup>3</sup>; прочность на сжатие 15–16 МПа; теплопроводность 0,15–0,16 Вт/(м·°С); марка по морозостойкости: F35. **Обсуждение.** Применение цифровых методов позволило установить характер влияния варьируемых факторов на свойства материала: его прочность, плотность и теплопроводность, что было подтверждено при проверке адекватности моделей как в сериях активного эксперимента, так и при исследовании структуры материала. **Заключение.** Разработаны основы методики прогнозирования свойств керамических изделий и подбора его состава с помощью цифровых методов, сформированы требования по оптимизации свойств изделий из пеностекла, а также по расходам пеностекла и флюсующего компонента. Полученные материалы вполне соответствуют требованиям к изделиям. Легкий кирпич является конструктивно-теплоизоляционным и может использоваться как стеновой материал.

**КЛЮЧЕВЫЕ СЛОВА:** керамический кирпич, дробленое пеностекло, бой стекла, цифровая оптимизация, статистическое планирование

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## INTRODUCTION

Ceramics have been used in construction for millennia. At the end of the 20<sup>th</sup> century, with the adoption of new thermal engineering standards, ceramic wall paneling met the requirements for heat conductivity and cladding for thermal resistance. Note that the construction of a brick wall in 2–2.5 bricks met the requirements of comfort, but the new regulations were unattainable for walls made of solid brick. In this regard, for most regions, laminated cladding was recommended, which includes effective heat insulation elements: stone cotton-based tiles or extruded polystyrene foam, with the cost of construction increased significantly [1–3].

The adopted direction to improve energy efficiency gave an incentive to develop ceramic products with lower density while maintaining the level of strength and frost resistance of materials: vacuum products and porous wall ceramics. Light pottery products had lower heat conductivity, which increased their thermal performance and reduced weight, reducing the load on the foundation [4–6]. The production of light (heat-insulating and structural) wall materials could be carried out in several ways. First, by increasing the emptiness of the articles to some limit. Second, by polarizing the article during the stages of forming, drying, and firing. The third (combined) method, combining the first two [7–9].

The formation of a high-porous ceramic skull can be carried out by means of burn-out additives, by introducing wood sawdust or sieving polystyrene. The second most important method is to increase the porosity of the ceramic brick by creating a cellular structure as in the gas formation process or mixing with special foams [10–12]. How a variety of this method can be considered for obtaining ceramic aerogels [13, 14]. The difficulties for materials in this group are due to the need to create special foams, as well as soft drying modes, that is, with low temperatures and heat transfer speeds. Otherwise, there was a risk of uneven placement and sedimentation or cracking of the ceramic billet.

The solution to the problem of obtaining light materials while maintaining quality characteristics is possible through the introduction in their composition at the stage of preparation of mixtures of light fillers. For such a light filler, granular mining waste of various types [15, 16] and waste from thermal power plants [17, 18] can be used. Promising results are obtained by using a particularly light microsphere of different nature [19–21]. High-porous aggregates may be used for refractory vermiculite and refractory perlite sand. Perlite sand has high water absorption, which significantly increases the drying time of the semi-processed material. Some researchers have noted the advisability of using heating foam [22–24]. Plate foam is not produced on an industrial scale in the Russian Federation, but it is produced in the form of a foam stone,

the usefulness of which is due to its relatively low water absorption and comparability with the characteristics of a ceramic skull.

The technology of ceramic products includes technological retooling of raw materials, forming, drying and firing [25–27]. The drying duration of articles depends on the properties of the clay mixture, relative humidity of the heat carrier, size, shape and configuration of the article, temperature drying mode, structural features of the drying devices. The drying time of products varies in industrial dryers or dry sheds from 2 to 20 days, and in laboratory drying cabinets from 6 to 48 hours. The temperature regime of burning brick and effective ceramic stones is conditionally divided into four periods: leisure (temperature up to 200 °C), heating (700–800 °C), burning itself (900–1050 °C), cooling (cooling up to 40–50 °C). In the case of use of ground foam, it is important to correct the temperature of the ceramic head sintering, which can be carried out by inserting a thread. Including fine-grained glazing [28–30].

Taking into account data analysis of formulations and technologies of heat-insulating and structural building ceramics, the aim of the research is formulated: development of a methodology for defining and optimizing formulations of light ceramic bricks, which is based on regulatory requirements for the strength, density and thermal conductivity of articles.

## METHODS AND MATERIALS

The main components of a mixture for making light ceramic bricks are: suckling, crushed foam glass, ground glazing; for correcting the temperature of the sintering, fine-grained glass waste (fluxous component), which is similar in composition to the raw material for the production of foam glass. Raw material preparation was carried out according to the generally accepted technology. Ceramic brick forming was carried out in a plastic manner, with a 22–24% molded moisture content.

Loam has a highly dispersed structure, a commotion texture. It is easy to crush and soaks well in the water. Loam belongs to the group of moderately plastic raw materials (plasticity number 12–19), which are not very dry. Loam is a poly-mineral raw material. The content of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> oxides is 52–53% and 11–12%, respectively; losses at calcination are 7–8%. The clay substance is represented by kaolinite (6–15 %), montmorillonite (10–18 %), hydromica (5–12 %) and quartz as an impurity.

Foam glass is a heating material with an average density of 80 kg/m<sup>3</sup> to 200 kg/m<sup>3</sup>. For the production of heat-insulating foam glass, glassware is most often used; its chemical composition is similar to that of aluminium window glass. These glasses contain mass % of: SiO<sub>2</sub> 72–73; Al<sub>2</sub>O<sub>3</sub> 0.5–1; MgO 3,5–4; CaO 6–6,5; Na<sub>2</sub>O 15,2–15,5; SO<sub>3</sub> 0.3–0.5. Glass thicknesses can be spun in tempera-

ture ranges from 600 to 1000 °C. Bulk density of foamed pebble 130–170 kg/m<sup>3</sup>; true density – 2180 kg/m<sup>3</sup>; water absorption by volume 2.7–4.4%. The heat conductivity in dry state is 0.08 W/(m °C); the increase of heat conductivity by 1% humidity – 0.001 W/(m °C%).

The achievement of the goal was based on the following tasks. First, it was necessary to study the patterns of drying of the designed products. Secondly, using statistical methods, to study the effect of formulation parameters on product properties. Third, using digital methods, develop a methodology for optimizing the composition of light ceramic brick.

Theoretically, the drying process is characterized by three periods: heating, constant and falling droughts. In real products, due to the geometric factor and under conditions of contact of a heat carrier with a product in five planes (one stretcher plane is adjacent to the pallet), the constant drying rate period may be weakly pronounced.

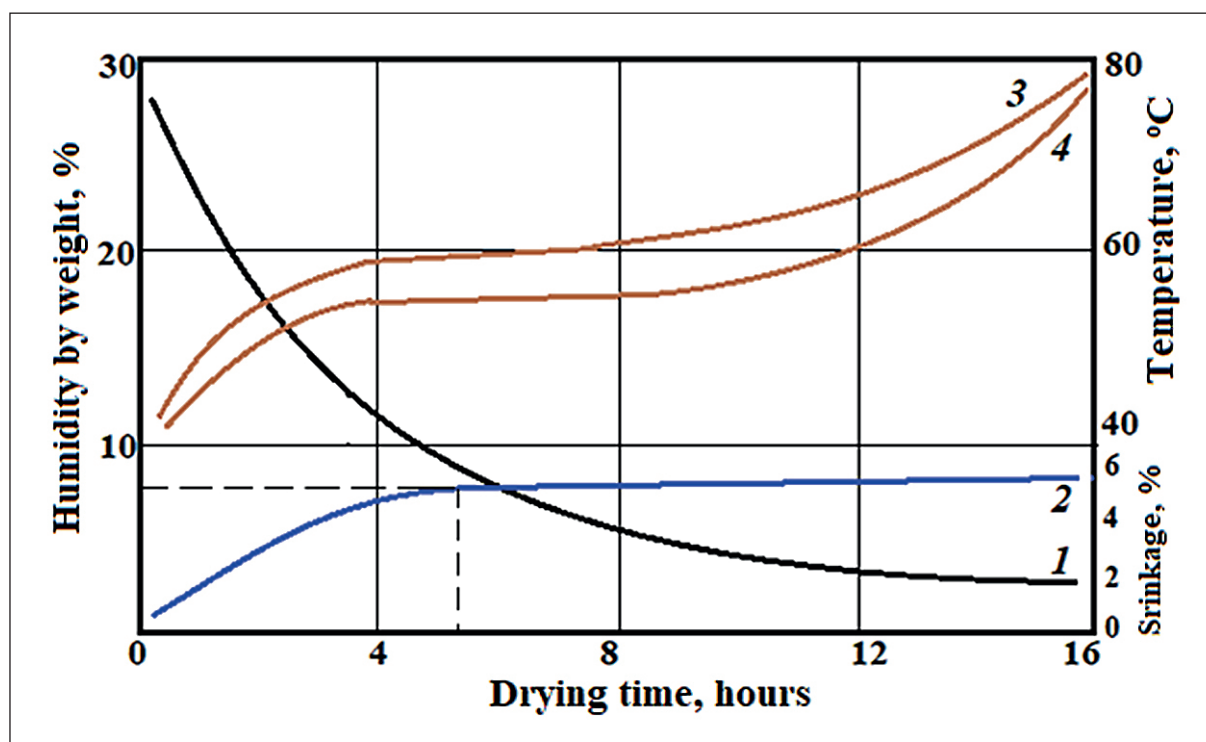
The overall dynamics of the drying process are shown in Fig. 1. The shrinkage of articles in length and width is not large, so it should be assumed that the articles have limited sensitivity to drying. The dependence of drying time on temperature and speed of heat transfer allows for harsher regimes compared to foam ceramics. The initial temperature of the heat carrier on the one hand intensifies the drying process, and on the other has an impact on the quality of the products. At a heat carrier temperature of

up to 80 °C, the sample reduction does not exceed 6%, and the products maintain high quality.

The samples were made in size 50×50×50 mm. Drying was carried out in the laboratory drying cabinet at a temperature of 80 °C and relative humidity 50–55 %; drying time – 12 hours. The firing of items was carried out in the furnace at a calculated temperature of 900–950 °C. The firing mode: raising the temperature from 18 to 100 °C – 15 minutes, holding at 100 °C – 10 minutes, raising the temperature to 650 °C – 120 minutes, holding at 650 °C – 60 minutes, holding the temperature up to 120 minutes, Storage at the calculated temperature – 120 min; natural cooling in the furnace.

The basis of the methodology for studying the influence of the characteristics of foam glass and fluxous component on the properties of products were digital methods of planning and processing experimental results [31, 32], as well as a method of analytical optimization, Developed at MSUCE and tested for many types of construction materials in the formulation and technology applications [33, 34].

The characteristics of the shredded foam glass are taken as variable factors: average density ( $X_1$ ), flow rate ( $X_2$ ) and average particle size ( $X_3$ ), as well as the flow rate of the thin-grain glass as a fluxing component fluxing component ( $X_4$ ). The response functions are the strength of the article under compression ( $Y_1$ , MPa), its average density ( $Y_2$ , kg/m<sup>3</sup>) and heat conductivity in dry con-



**Fig. 1.** Drying of ceramic samples on foam (heat transfer agent characteristics: temperature 80 °C, relative humidity 40%): 1 – curve drying; 2 – drying shrinkage; 3, 4 – top and middle temperature of the sample

ditions ( $Y_3 = \lambda \cdot 10^{-3} \text{ t}/(\text{m} \cdot ^\circ\text{C})$ ). The values of response functions were determined according to GOST 530-2012.

After conducting preliminary series of experiments, the optimal heat treatment conditions were determined and intervals for varying factors were established. The conditions of the experiment are presented in Table 1. A complete quadratic matrix D-optimal plane was adopted in the experiment. In each line of the plan, the experience was repeated 5 times ( $n = 5$ ). Randomization of the sequence of experiments was used to reduce the likelihood of interference and unaccounted factors.

Regression equations derived from experimental data processing in the Statistika program. The significance of the coefficients was checked at confidence intervals for strength and material density. The size of the confidence interval was determined by the Student criterion for 5 experiments and the dispersion of parallel experiments. The confidence intervals ( $\Delta b$ ) were for strength – 0.2 MPa, mean density – 9 kg/m<sup>3</sup>, thermal conductivity – 9 kg/m<sup>3</sup>, and thermal conductivity – 0.004 W/(m °C). The coefficients of the regression equations, which are smaller in absolute value than the respective confidence intervals, were considered as non-significant and equated to 0.

## RESULTS AND DISCUSSION

The results of the active experiment give basic regression equations. The significance of the coefficients was checked at confidence intervals for strength and material density. The size of the confidence interval was determined by the t-criterion for 5 experiments ( $n = 5$ ) and the dispersion of parallel experiments. The confidence intervals ( $\Delta b$ ) were for strength – 0,2 MPa, mean density – 9 kg/m<sup>3</sup>, thermal conductivity – 9 kg/m<sup>3</sup>, and thermal conductivity – 0,004 W/(m °C). The coefficients of the regression equations, which are smaller in absolute value than the respective confidence intervals, were considered as non-significant and equated to 0.

The basic regression equations are obtained at intervals of varying factors (Table 1) for strength and mean density and heat conductivity:

$$Y_1 = 13,6 + 0,8X_1 - 1,7X_2 + 0,7X_3 + 0,5X_4 + 0,6X_2X_3 - 0,4X_3^2 - 0,3X_4^2; \quad (1)$$

**Table 1.** Conditions of the three-factor experiment

Factor name	Symbol, $X_i$	Average factor, $\bar{X}_i$	Range, $\Delta X_i$	Factor values in levels	
				-1	+1
Average density of foam glass, kg/m <sup>3</sup> ( $\rho_n$ )	$X_1$	120	40	80	160
Foam glass consumption, kg/m <sup>3</sup> ( $P_n$ )	$X_2$	36	8	28	44
Average size of foam glass particles, mm ( $d_n$ )	$X_3$	0.5	0.2	0.3	0.7
The Content of the fluid component, % ( $C_\phi$ )	$X_4$	10	2	8	12

$$Y_2 = 1378 + 18X_1 - 51X_2 + 14X_3 + 8X_2X_3; \quad (2)$$

$$Y_3 = 144 + 14X_1 - 7X_2 + 4X_1X_2. \quad (3)$$

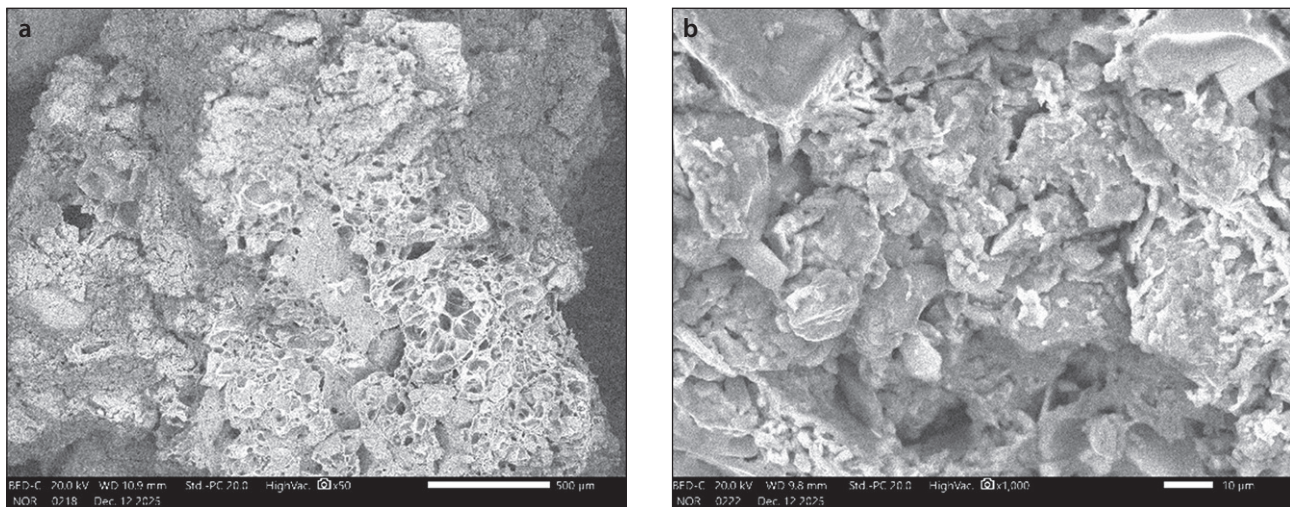
The assessment of adequacy at the initial level was carried out by comparing calculated and experimental values of the Fischer criterion (F-criterion) and further as a result of the implementation of the control series of the active experiment.

The analysis of the basic regression equations shows that the most important influences on the strength of the foam glass are its characteristics (ratios at  $X_1$  and  $X_2$ ) and its flow rate (ratio at  $X_3$ ), as well as the pair effect of the flow rate and particle size (ratio at  $X_2X_3$ ). Also significant is the content of the fluent component (coefficient at  $X_4$ ). The nature of this influence can be explained by the structure of the resulting material (Fig. 2). The corrective action of malting glass as a fluxing component that lowers the temperature of the ceramic skull, which makes it possible to spin foam glass particles from the ceramic matrix without breaking (melting) the particles of foam glass and their preservation of the porous structure of the burned material. The average density of the material and its thermal conductivity are determined by the characteristics and flow rate of the crushed foam (polynomials 2 and 3).

The values of the coefficients of polynomial 1 allow to deduce the extreme influence of the particle size of the foam glass and the flow rate of the fluid component on the strength of the product (coefficients at  $X_3^2$  and  $X_4^2$  are equal to  $-0,4$  and  $-0,3$ , respectively). Taking into account that testing of polynomials (1-3) according to the F-criterion has shown their adequacy both as statistical polynomials and as algebraic models, it is possible to apply methods of algebraic analysis of nonlinear functions of four variables for their study. This makes it possible and useful to use the analytical optimization method.

### Factor $X_3$ optimization (average particle size of fosterol)

1. Determine the private derivative function  $Y_1(X_1, X_2, X_3, X_4)$  by variable  $X_3$ , equate the result with 0 and find the optimal value of the mean particle size of the foam glass ( $d_n$ ):



**Fig. 2.** Structure of material (a) and ceramic skull (b) when enlarged: a – 50 times; b – 1000 times

$$\partial Y_1 / \partial X_3 = 0.7 - 0.8X_3 \Rightarrow X_3 = 7/8 = 0.88.$$

Determine the natural value of the optimal size of a part of the foam glass by using the data from table 1:

$$d_{\text{н}} = 0.5 + 0.88 \times 0.2 = 0.68 - 0.69 \text{ mm.}$$

2. Determine the private derivative function  $Y_1(X_1, X_2, X_3)$  by variable  $X_4$ , equate the result with 0 and find the optimal flow rate of the fluid component ( $P_{\phi}$ ):

$$\partial Y_1 / \partial X_4 = 0.5 - 0.6X_4 \Rightarrow X_4 = 5/6 = 0.83.$$

The natural value of the flow rate of the fluxing component (fluxing agent) is determined using the data from Table 1:

$$P_{\phi\kappa} = 10 + 0.83 \times 2 = 8.3 - 8.4 \% \text{ from the mass of clay.}$$

3. We optimize algebraic polynomials (1–3) according to the optimal particle size of the foam glass and the fluxing component content, and we obtain optimized models:

$$Y_1 = 14,1 + 0,8X_1 - 1,2X_2; \quad (4)$$

$$Y_2 = 1390 + 18X_1 - 37X_2; \quad (5)$$

$$Y_3 = 144 - 14X_1 - 7X_2 - 4X_1X_2. \quad (6)$$

Dependencies (Formulas 4–6) became the basis for forming a nomogram (Fig. 2). Variable factors are mean density and foam flow rate; mean particle size of the foam and fluid content have fixed optimization values. In the nomogram sectors, a relationship is established between the response function and varying factors: in

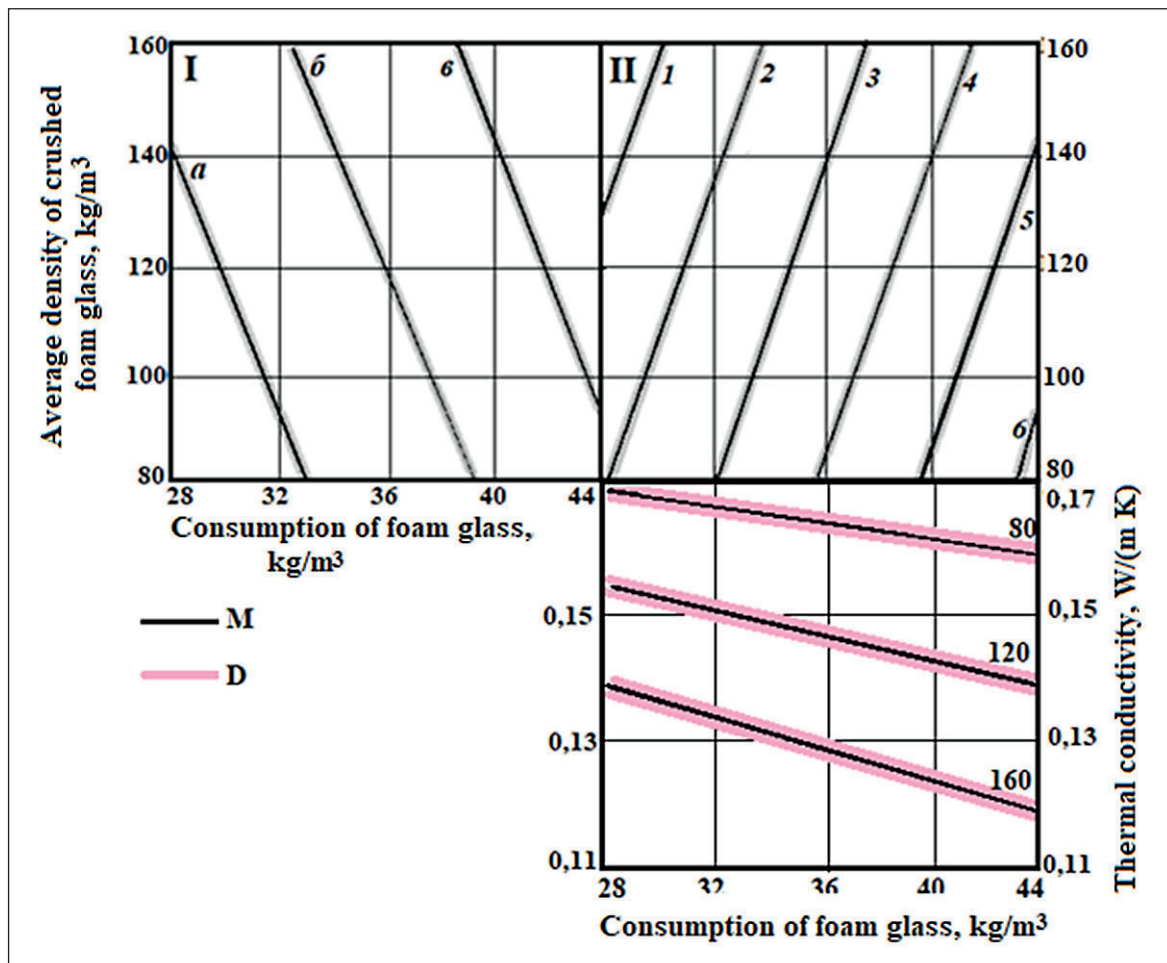
sector I – for medium density of burnt ceramics; in sector II – for strength to compression; in sector III – for heat conductivity. Average particle size of the foam glass 0.68–0.69 mm; concentration of the fluxing component = 8.3–8.4% of clay mass.

The mean value of the result (mathematical expectation) was determined, and deviation from the mean (spread of results or discount) was taken to be closer to the normal distribution (Gauss distribution). Using a nomogram (Fig. 3), the direct task of digital modeling (predicting product properties by specified values of variable factors) is solved graphically. The inverse problem (determination of optimal values of factors) is solved analytically and as a result of the implementation of the control series of the experiment.

According to product properties predictions derived from the nomogram (Fig. 3). Control formulae (active control experiment) were carried out, the results of which are presented in the Table 2. The average density and the flow rate of foam glass varied; the particle size of the foam glass and the content of the fluxing agent were taken at an optimal level. The compressive strength of samples was taken as a parameter for evaluation.

The test series of the in-situ experiment allowed the following conclusions to be drawn. First, the mean error, that is, the difference between calculated and experimental values does not exceed 1.6%, which confirms the model's adequacy and the validity of the results obtained. Second, the best strength values correspond to the conditions of experiment No. 7; calculated strength from equation (4) – 15.9 MPa, experimental – 16.1 MPa;  $\Delta = 1.3\%$ .

The optimal values of factors are as follows. Variable in the experiment: average density of foam glass 150 kg/m<sup>3</sup>; flow rate of foam glass 30 kg/m<sup>3</sup>; optimal particle size of ground foam glass 0.68–0.69 mm; fluid content (melt) 8.3–8.4% of clay mass. The following sample character-



**Fig. 3.** A nomogram for predicting parameters and selecting the composition of products with an optimal particle size of 0.68–0.69 mm foam and a melt content of 8.3–8.4% of clay mass. Mean density, kg/m<sup>3</sup>: a – 1320; b – 1360; in – 1400. Compressive strength, MPa: 1 – 18; 2 – 17; 3 – 16; 4 – 15; 5 – 14; 6 – 13; mean density of the foam, kg/m<sup>3</sup>: 80, 120, 160; M – mean result (mathematical expectation); D – deviation from mean value

**Table 2.** Test results

Experience No.	Conditions of the experiment		Compressive strength, MPa		Error Δ, %
	Average density of foam glass, kg/m <sup>3</sup>	Foam glass consumption, kg/m <sup>3</sup>	Calculated	Experimental	
1	150	42	13.7	13.5	1.5
2	120	42	13.0	13.2	1.5
3	90	429	12.3	12.5	1.6
4	150	36	14.8	14.6	1.4
5	120	36	14.1	14.4	2.2
6	90	36	13.4	13.6	1.5
7	150	30	15.9	16.1	1.3
8	120	30	15.2	15.0	1.4
9	90	30	14.5	14.2	2.1
Average Δ, %					1.6

istics correspond to these values: average density 1320–1360 kg/m<sup>3</sup>; compressive strength 15–16 MPa; thermal conductivity 0.15–0.16 W/(m °C). Separately, the brand was determined by frost resistance: F35.

The laboratory-generated samples have properties that are quite suitable for structural and thermal insulation materials. The next stage of research will be testing in the conditions of existing production, as well as developing recommendations for the use of light ceramics and exploring the possibility of heat-insulating and structural ceramic products of lower density. Light bricks can be used in wall cladding, but are not recommended for foundations. It is possible to make ceramic products both full-length and with voids, as well as the production of modular ceramic bricks and large-format ceramic stones.

## CONCLUSION

The use of digital methods in the design and processing of an experiment can significantly reduce the amount

of material needed and the volume of research without compromising on results. It is mandatory to check the data obtained in a control experiment.

The following factors have been accepted as optimal in the results of the studies. Variable in the experiment: average density of foam glass 150 kg/m<sup>3</sup>; flow rate of foam glass 30 kg/m<sup>3</sup>; optimal particle size of ground foam glass 0.68–0.69 mm; fluid content (melt) 8.3–8.4% of clay mass. The following sample characteristics correspond to these values: average density 1320–1360 kg/m<sup>3</sup>; compressive strength 15–16 MPa; thermal conductivity 0.15–0.16 W/(m °C); frost resistance mark: F35.

As a result of the research, the foundations of methods for predicting properties of ceramic articles have been developed, requirements for optimizing properties of foam glass, as well as for costs of foam glass and fluxing component have been formed. The resulting materials fully meet the requirements for structural and thermal insulation products. Lightweight foam glass based brick can be used in thermal efficient wall cladding. The use of light bricks in fundamentals is not recommended.

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## ADDITIONAL INFORMATION

The authors declare that no generative artificial intelligence or AI-based technologies were used in the preparation of the article.

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**Petr M. Zhuk** – literary review, intermediate conclusions, adaptation of the method to the objectives of the study;

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