FOUNDATIONS FOR DESIGNING OF AERATED CERAMIC MIXTURES

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Abstract. The widespread introduction of aerating technology into the production of highly porous ceramics requires the development of principles for the selection of a raw mixture based on clay raw materials from various deposits. The effectiveness of using fiber for the cellular ceramic matrix reinforcement was shown in the article. The utilization efficiency of reinforcement additive based on basaltic fiber incorporated into the aerated ceramic mixture was proved, and the tailored composition was defined. The synergies between the durability of ceramic products and refractoriness temperatures of raw material input were specified. The test method of refractoriness specified the temperature of argillous raw material depending on the content of heat-resistant and fusible oxides in it was given. The correlation between the chemical composition of dry mix and compressive resistance of the aerated ceramic products of average density from 0.4 to 0.8 g/cm^3, which allows assessing volume components of the raw mix for the aerated high-quality ceramic products production, was established. The relative criterion of resistance that allows classifying the aerated ceramic products by category and assigning them to the appropriate group was defined.

Keywords: aerated ceramic products, refractoriness temperatures, chemical composition, fiber reinforcement, relative criterion of resistance

1. Introduction

Clay-based wall ceramics is one of the most popular building materials in industrial and civil construction. The key performance properties of ceramic materials, which determine the widespread use of structures, are durability, architectural expression, environmental friendliness, and affordability. The wide geography of clay deposits in the Russian Federation and the availability of modern ceramic production allow speaking confidently about the need for further development of this industry by reducing fuel costs and material intensity of production, increasing the ratio of structural quality and improving the thermal characteristics of products.

Due to the need to comply with the requirements of SP 50.13330.2012 "Thermal protection of buildings", the modern construction industry focuses mainly on the construction of multi-wall structures, including load-bearing, insulating decorative elements. The currently used ceramic bricks and stones have relatively high thermal conductivity values, so in the construction of buildings with energy efficiency classes B, A, A+, A++, the share of ceramic materials is constantly decreasing: they are replaced by products of cellular concrete. Traditional technologies of existing ceramic enterprises cannot produce products with an average density of less than 680 kg/m^3 and values of the ratio of thermal conductivity below 0.18 W/(m°C).

The following main ways to reduce the average density of ceramic tiles can be identified resulting from an analysis of existing methods of creating porous structures of building ceramic materials: burning out of burning additives; introduction of porous aggregates; gas formation; blowout; foaming; and aerating.

In existing processes for the production of porous ceramic products, there are also combinations of the above methods, allowing to optimize some technological operations and obtain new properties of the finished products. However, mostly two main methods of ceramic product porization are used: burning out of burning additives with mechanical cavitation and the introduction of porous aggregates.

Each of the above methods for reducing the average density of ceramic products has its advantages and disadvantages. The choice of porization technology depends on the quality of clay raw materials of a particular deposit, and the required physical and mechanical parameters of the resulting ceramic products (Belyakov *et al.*, 2021; Saidova *et al.*, 2021).

One of the indicators of building materials, conditionally characterizing their performance is the strength-density ratio (SDR). The higher the SDR of the material used, the higher the efficiency of the material application. To systematize and compare the efficiency data of different methods of reducing the average density of ceramic materials, their efficiency is expressed in SDR (Table 1) (Baravalle *et al.*, 2016; Bennett *et al.*, 2021).

Table 1 shows that the least effective way to reduce density is the burning out of burning additives, the most effective – foaming and aeration. Both methods use in their basis the slicker technology for preparing the clay mass, where one of the most important rheological parameters is the viscosity at the lowest water-soluble ratio.

The technology of aerated wall ceramics with an average density of 400 to 800 kg/m^3 with compressive strength values from 3 to 10 MPa and more is one of the promising directions in the development of the ceramic industry. Aeration is based on the principle of involving air bubbles in the clay slip mass during mixing and allows the use of various additives (reinforcing, plasticizing, fluidizing, etc.) to modify the raw mixture to obtain new properties of the finished products.

Little studied principles of aeration as a technology for obtaining highly porous ceramic materials and the lack of theoretical foundations for selecting rational compositions of ceramic clay slip are of high scientific and practical interest. Given the flexibility of the technological line for the production of aerated ceramics, promising is the introduction of this technology in the technological chains of existing enterprises for the production of bricks and stones, focused on the method of plastic molding.

Table 1. Characteristics of the effectiveness of various methods of reducing the average density of ceramic materials.

Method name	Average density, kg/m³	Compressive strength, MPa	SDR
Burnout of burning additives	800-1650	0.9-15.0	1.1-9.1
Introduction of porous aggregates	500-600	2.6-4.0	5.2-6.6
Gas formation	300-1000	1.0-6.0	3.3-6.0
Blowout	450-950	2.0-6.4	4.4-6.7
Foaming	400-750	1.5-6.2	3.8-8.3
Aeration	350-860	0.8-7.3	2.3-8.5

2. Method of aerating ceramic masses

Aeration and foaming methods are close technologies for manufacturing products and effectively reduce the density of ceramic materials, allowing to obtain similar external and technical indicators of porous ceramic mass. However, they are fundamentally different in some technological units of the preparation of the raw mix and its porization. In both cases, the most important step is to determine the rational compositions of clay slurries, allowing to obtain porous ceramic mass with the required density, ductility and the dynamics of plastic strength after molding.

Aeration in contrast to the foaming allows the preparation of porous ceramic mass in a single mixing unit, significantly reducing the consumption of air-entraining additive. In addition, as the number of air bubbles increases during aeration, a homogenized cellular structure is created with pore walls evenly filled with solid-phase minerals, increasing the structural strength of the molded ceramic mass (Progulny *et al.*, 2020; Nafikov *et al.*, 2020).

The ceramic matrix is reinforced by different types of fibers (metal, vitreous, etc.) to increase the resistance of ceramic products to the emergence and spread of cracks. The length, diameter and number of fibers and the water-solid ratio of the initial clay slip determine the plastic strength of the molded porous masses. Depending on the fiber type, it is possible to produce products with a controlled average density in a wide range of values, which also allows producing large-sized products with minimal cracking after firing and with sufficient compressive strength. Using fibers with a minimum cross-section increases the bending strength of porous thin-walled ceramic products (plates, shells, etc.) (Dmitriev, 2017; Sokov, 2015; Zhavoronkov *et al.*, 2017; Pukharenko and Morozov, 2013).

Fiber or filament reinforcement strengthens areas of the ceramic material subject to high tensile stresses. However, the positive effect of using fibers with high tensile strength is observed if the reinforcing fibers have a higher elastic modulus than the ceramic matrix. Otherwise, the reinforced ceramic structure will collapse before the tensile forces are transferred to the fibers, especially when their volume content is below the optimal value. Moreover, when selecting the type of reinforcing fibers, it is always necessary to consider the relative elongation during heating, i.e., this indicator of the fibers should not exceed the corresponding indicator of the ceramic material to be reinforced.

It is established that reinforcement of construction composites with fibers of different modularity degrees allows increasing the strength, crack resistance, and fracture toughness of finished products and improving their deformative properties significantly during hardening (Sagyndykov *et al.*, 2015; Behera *et al.*, 2020; Devasia *et al.*, 2021).

The aeration technology allows to effectively use different types of fibers, distributing them evenly throughout the entire volume of the aerated ceramic mass (ACM). Dispersed reinforcement reduces shrinkage strain during drying of aerated raw ceramic (ARC) and also increases their structural strength, allowing to intensify the firing process to obtain aerated ceramic products (ACP) with a minimum of scrap (Lukuttsova *et al.*, 2017; Uthaman *et al.*, 2021). The preliminary tests of aerated ceramic products reinforced with basalt fiber are summarized in Table 2.

Samples were made based on clay rocks from various deposits: composition No. 1 – Cambrian clay (Leningrad region), 2 – Borovichi-Lubytinsky clay (Novgorod region), 3 – Kaolin clay (Novgorod region), 4 – Shaberdinsky loam (Udmurtian Republic).

Table 2. Preliminary test results of reinforced ACP.

Name of component/ indicator	Component content, % by weight / indicator value							
	1		2		3		4	
Clay	43.25		50.52		48.25		52.87	
Grog	12.43		16.39		6.89		11.50	
Basalt fiber (over 100%)	–	0.36	–	0.23	–	0.22	–	0.29
Liquid glass	0.49		0.76		0.88		0.69	
SAW	0.63		0.63		0.47		0.46	
Water	42.56		31.70		43.28		34.48	
Average density, g/cm^3	0.51	0.45	0.57	0.52	0.78	0.67	0.69	0.63
Total shrinkage, %	12.8	9.7	12.2	9.4	13.4	10.4	12.1	9.5
Compressive strength, MPa	4.02	4.18	3.95	4.32	7.46	7.85	5.52	5.71

It should be noted that the presence of basalt fiber in ACM compositions positively affects the drying properties of ARC, reducing the values of total shrinkage by an average of 22.8% compared to the control compositions.

3. Materials and research methods used

The study aims to develop the relationship between the chemical composition of the ceramic charge and the compressive strength of reinforced ACP with an average density of 0.4 to 0.8 g/cm^3.

The physical and mechanical characteristics of ACP were determined under the current regulatory documents specified based on GOST 530-2012 "Bricks and ceramic stone".

ACM compositions include the following components: clay raw material, water, liquefying, air-entraining, reinforcing, and thinning additives (Smith and Naït-Ali, 2021).

Sodium liquid glass (GOST 13078) is used as a liquefying additive, air-entraining additive – synthetic hydrocarbon blowing agent "PB-Formula 2012" (TU 2481-008-80824910-2012). The reinforcing additive is a basalt fiber (TU 5952-036-05328981), the characteristics of which are presented in Table 3. The chemical composition of the clay raw materials used in the study is shown in Table 4.

Table 3. Characteristics of basalt fiber.

Fiber diameter, mm	0.009
Fiber length, mm	13.0
Density, g/cm^3	2.6
Tensile stretch, %	1.4-3.6
Tensile strength, MPa	1600-3600
Elastic modulus, kg/mm^2	9100-11000

Production of aerated ceramic samples is carried out as follows:

- obtaining a clay slip by mixing the prepared raw components in certain proportions until the homogeneity of the slip and relative viscosity values in the range from 1.49 to 1.78. The air-entraining additive is added last. Thinning additive (grog) and clay are used in a ground state with a maximum particle size of 0.16 mm;
- aeration of the feed mixture using a laboratory propeller stirrer with a blade speed of 1000 – 1500 rpm to obtain ACM of desired density;
- ACM molding into prepared onboard molds with dimensions of 290×140×75 mm;

Table 4. Chemical composition of clay raw materials.

Name of clay raw materials	SiO_2	Al_2O_3+ TiO_2	Fe_2O_3	CaO	MgO	Na_2O+ K_2O	Loss on ignition
Cambrian clay	61.95	18.02	4.82	1.38	3.67	5.43	4.73
Borovichi-Lubytinsky clay	55.34	17.70	7.29	4.53	2.42	4.32	8.40
Kaolin Clay	48.35	33.60	1.24	0.85	0.47	2.13	13.36
Shaberdinsky loam	66.32	13.85	5.36	1.41	2.64	4.45	5.97

Table 5. Strength parameters of optimal ACP compositions.

z_1, kg	z_2, kg	z_3, g/cm³	ρ_{av}, g/cm³	R^1_{comp}, MPa	R^2_{comp}, MPa	R^3_{comp}, MPa	R^4_{comp}, MPa
25.0 – 32.1	0.84 – 0.90	0.494 – 0.502	0.408	3.54	3.02	4.68	3.32
		0.503 – 0.540	0.438	3.88	3.37	5.18	3.74
35.7 – 46.4	0.73 – 0.81	0.541 – 0.572	0.456	4.08	3.61	5.45	3.92
		0.573 – 0.602	0.485	4.49	4.12	5.94	4.28
75.0 – 85.0	1.41 – 1.50	0.603 – 0.628	0.504	4.83	4.29	6.22	4.58
		0.629 – 0.661	0.532	5.15	4.73	6.47	4.92
85.1 – 95.0	1.33 – 1.40	0.662 – 0.684	0.558	5.71	5.09	6.84	5.22
		0.685 – 0.714	0.586	5.92	5.37	7.32	5.71
145.0 – 153.6	1.84 – 1.90	0.715 –0.733	0.604	6.07	5.65	7.61	5.84
		0.734 – 0.759	0.629	6.47	6.14	7.98	6.08
157.9 – 166.4	1.76 – 1.81	0.760 – 0.794	0.661	6.85	6.48	8.29	6.55
		0.795 – 0.828	0.694	7.36	6.84	8.81	7.11
205.0 – 212.1	2.24 – 2.30	0.829 – 0.858	0.718	7.67	7.18	9.15	7.27
		0.859 – 0.877	0.742	7.89	7.56	9.52	7.65
215.7 –222.9	2.16 – 2.21	0.877 – 0.909	0.776	8.51	8.05	9.96	7.99
		0.910 – 0.944	0.789	8.62	8.23	10.16	8.28

- pre-exposure of ACM in natural conditions at a temperature of not more than 30 °C to the humidity of the raw material of 40%;
- unclogging and further reduction of ARC humidity to 5% in the drying cabinet;
- heat treatment of ARC, where the rise time to the firing temperature is 180 minutes, isothermal soaking at 1000 °C – 150 minutes, natural cooling is performed in the furnace chamber;
- machining of cooled ACP to the size of standard bricks 250×120×65 mm (Latif *et al.*, 2019; Liu *et al.*, 2021; Xiaoshan *et al.*, 2021; Urazaeva *et al.*, 2021).

To establish the optimum compositions of different ACP densities, a three-factor experiment with a change in the value of each factor at two levels is used, which allows establishing mathematical equations of dependence of the compressive strength limit on the content of fiber (z_1), grog (z_2) and density of ACP (z_3) in the composition of ACP (Yin *et al.*, 2018; Jie et al., 2021; Chen *et al.*, 2020; Chen *et al.*, 2021).

4. Research results

The complex analysis of the obtained regression equations allowed determining the optimum component composition for each range of values of the average ACP density from 0.4 to 0.8 g/cm³: R^1_{comp}, R^2_{comp}, R^3_{comp}, R^4_{comp} – the compressive strength limits of ACP based on Cambrian, Borovichi-Lubytinsky, kaolin clays and Shaberdinsky loam, respectively, MPa (Table 5). The compressive ACP strength increases in direct proportion to the change in average density over the entire range of its values.

Calculation of the relationship between the chemical composition of the initial clay raw materials and the compressive ACP strength is based on the results of the dissertation work of V.B. Zverev in the field of methods for determining the calculated temperature of refractoriness of ceramic materials using industrial by-products by the gross chemical composition of raw materials (Equations 1-4) and is its logical continuation.

$$T_{ref}^{est} = \left(\frac{2.92}{1 + 1.156 K_{fus}} - 1 \right) \times 798 \pm \Delta t \quad (1)$$

where: T_{ref}^{est} T_{ref}^{est} – estimated refractoriness temperature, °C; K_{fus} – number of fusibility; Δt – temperature correction.

$$K_{fus} = \frac{a_1 n_1 + a_2 n_2 + a_3 n_3 + a_4 n_4 + a_5 n_5}{b_1 m_1 + b_2 m_2}$$

$$K_{fus} = \frac{a_1 n_1 + a_2 n_2 + a_3 n_3 + a_4 n_4 + a_5 n_5}{b_1 m_1 + b_2 m_2} \quad (2)$$

where: a_1, a_2, a_3, a_4, a_5 a_1, a_2, a_3, a_4, a_5 – melting constants for iron, calcium, magnesium, potassium and sodium oxides respectively ($a_1 = 0{,}8, a_2 = 0{,}5, a_3 = 0{,}6, a_4 = 1, a_5 = 1$); n_1, n_2, n_3, n_4, n_5 – content in the charge of oxides of iron, calcium, magnesium, potassium and sodium respectively, % by mass; b_1, b_2 – melting constants for silicon and aluminum oxides respectively ($b_1 = 1$, $b_2 = 1.2$ $b_1 = 1, b_2 = 1{,}2$); m_1, m_2 m_1, m_2 – content in the charge of silicon and aluminum oxides respectively, % by mass.

$$\Delta t = 833.3e - 100 \quad \Delta t = 833{,}3e - 100 \quad (3)$$

where: e – alumina-alkaline module.

$$e = \frac{M_1}{M_2 + M_3 + M_4} \quad e = \frac{M_1}{M_2 + M_3 + M_4} \quad (4)$$

where: M_1, M_2, M_3, M_4 M_1, M_2, M_3, M_4 – the content of aluminum, silicon, potassium and sodium oxides in the charge, respectively, %/mol.

An example of determining the calculated temperature of refractoriness for Borovichi-Lubytinsky clay is shown below. This indicator is calculated similarly for other clays.

$$K_{fus} = \frac{0.8 \times 7.29 + 0.5 \times 4.53 + 0.6 \times 2.42 + 1 \times 2.15 + 1 \times 2.17}{1 \times 55.34 + 1.2 \times 17.7} = 0.181$$

$$e = \frac{\frac{17.7}{102}}{\frac{55.34}{60} + \frac{2.15}{94.2} + \frac{2.17}{62}} = 0.177$$

$$\Delta t = 833.3 \times 0.177 - 100 \approx 47.5°C$$

$$T_{ref}^{est} = \left(\frac{2.92}{1 + 1.156 \times 0.181} - 1 \right) \times 798 + 47.5 \approx 1177°C$$

$\Delta t = 833{,}3 \times 0{,}177 - 100 \approx 47{,}5$ The calculated temperatures of refractoriness of the studied clays are summarized in Table 6.

Table 6. Calculated refractoriness temperatures of clay raw materials.

Clay	e	K_{fus}	Δt Δt, °C	T_{ref}^{est}, °C
1	0.156	0.146	34.0	1227
2	0.177	0.181	47.5	1177
3	0.395	0.043	230	1651
4	0.116	0.133	– 3.3	1220

There is a certain relationship between the strength of the ceramic product and the chemical composition of the original clay raw materials. Since ceramics acquire their main properties during the firing process, indicators such as the sintering interval and refractoriness temperature are among the main production criteria for obtaining the desired products of a given quality. Control over the content of fusible and refractory oxides in the charge allows regulating the firing regime and the physical and mechanical properties of the finished ACP (Wu and Huang, 2021; Ren *et al.*, 2021; Tiuc *et al.*, 2017).

The compressive strength values of the optimal ACP compositions from Table 5 are used to construct smoothing straight lines using the least-squares method. The resulting equations of dependence and the scale of refractoriness temperature values are presented in the combined diagram (Fig. 1).

According to the above diagram, the dependence of the refractoriness temperature on the compressive strength is expressed by the general Equation 5.

$$R_{comp} = 0.02T_{ref} - 20 \quad R_{comp} = 0{,}02T_{ref} - 20 \tag{5}$$

Values R_{comp} and T_{ref} have an individual nature of change for each component composition of ACM based on clay raw materials of a particular deposit, so Equation 5 is supplemented by a general (k_0) and temperature (k_t) compliance factors, which are determined by Equations 6 and 7, respectively. k_0 and k_t the calculated data for the Cambrian clay are taken as a basis for determining the factors.

$$k_0 = \frac{T_{ref}}{T_{ref}^{est}} = \frac{50R_{fus}+1000}{T_{ref}^{est}} = \frac{50\times(13.42\rho_{av}-1.97)+1000}{1227} = 0.547\rho_{av}+0.735$$

$$k_o = \frac{T_{ref}}{T_{ref}^{est}} = \frac{50R_{fus}+1000}{T_{ref}^{est}} = \frac{50\times(13{,}42\rho_{av}-1{,}97)+1000}{1227} = 0{,}547\rho_{av}+0{,}735 \tag{6}$$

The temperature ratio of compliance reflects the individual dependence of the compressive strength of ACP on the calculated charge refractoriness temperature, which has a linear character of change. In this case, the function $k_t = f(T_{ref}^{est})$ $k_t = f\left(T_{ref}^{est}\right)$ is determined by the least-squares method.

$$k_t = 1.627 - \frac{T_{ref}^{est}}{1955.72} \quad k_t = 1{,}627 - \frac{T_{ref}^{est}}{1955{,}72} \tag{7}$$

The values of temperature correspondence ratios $k_t^1, k_t^2, k_t^3, k_t^4$ for Cambrian, Borovichi-Lubytinsky, kaolin clays, and Shaberdinsky loam, respectively, are summarized in Table 7.

Table 7. Temperature ratios of compliance of the studied clays.

k_t^1	k_t^2	k_t^3	k_t^4
0.9996	1.0252	0.7828	1.0032

We obtain the final general correlation between the chemical composition of the charge and the compressive strength of reinforced ACP with an average density of 0.4 to 0.8 g/cm³ (Equation 8).

$$R_{comp} = 0.02k_t\left(0.547\rho_{av}+0.735\right)T_{ref}^{est} - 20$$

$$R_{comp} = 0{,}02k_t(0{,}547\rho_{av}+0{,}735)T_{ref}^{est} - 20 \tag{8}$$

A comparative analysis of the values of the compressive ACP strength, calculated by Equation 8, and the experimental values from Table 5, found that the average value of the relative error (ΔR_{comp}) is 5.07%.

The variety of ACP types is shown in Fig. 2.

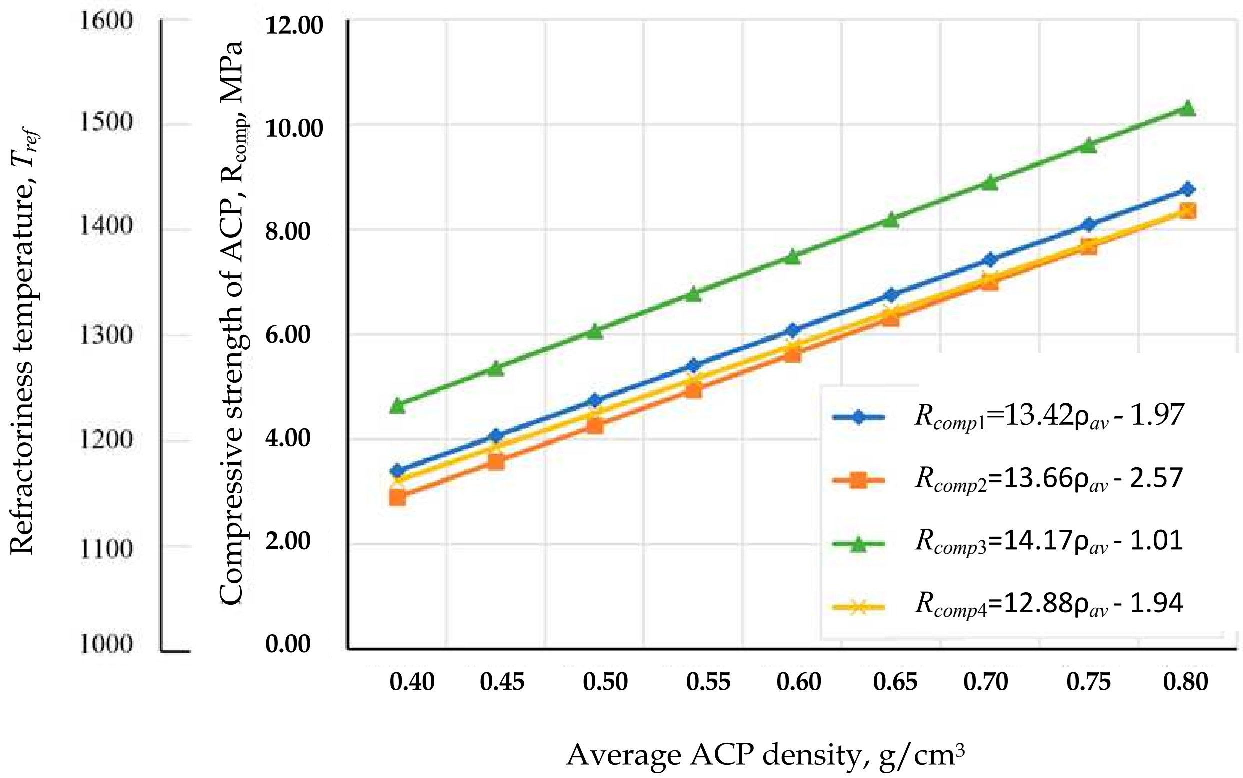

Fig. 1. Combined diagram of the temperature dependence of refractoriness, compressive strength and average density values of ACP.

Fig. 2. Photo of ACP average density of 0.6 g/cm³ in the form of bricks and tiles.

The calculated value of the compressive strength of aerated ceramic products with an average density of 0.5 g/cm³ is denoted by the author as the relative criterion of resistance of ACP (R^{rel}_{comp} R^{rel}_{comp}). Based on this criterion, an assessment of the quality of the raw mix for the production of ceramic products by aerated technology is formed. The ACP classification is shown in Table 8.

Table 8. ACP classification by the relative criterion of resistance.

R^{rel}_{comp}, MPa	Product category	Product group according to strength properties
6.5 and more	R 65	Special strength
5.8 – 6.4	R 60	
5.1 – 5.7	R 55	High-strength
4.5 – 5.1	R 45	
3.8 – 4.4	R 40	Increased strength
3.1 – 3.7	R 35	
2.4 – 3.0	R 25	Standard strength
1.7 – 2.3	R 20	
1.1 – 1.6	R 15	Low strength
1.0 and less	R 10	

Conclusion

The following results were obtained as a result of the study:

1. It was found that fiber reinforcement of basalt fiber in the aerated raw mass can reduce shrinkage and increase the strength of the resulting aerated ceramic tile.

2. The mathematical dependences linking the consumption of fiber and grog, and the density of the raw mass, with the strength of aerated ceramics at a given value of its density, regardless of the clay type were obtained. It was found that the nature and extent of the influence of dispersed reinforcement, grog consumption and the density of the raw mix on the air shrinkage, density and strength of aerated ceramics does not depend on the clay type used.

3. The type of clay used has a decisive influence on the strength value of ceramics of a given density. The dependence between the chemical composition of clay raw materials and the strength characteristics of reinforced high-porous ceramic products has been established, allowing to adjust the characteristics of the finished products and produce aerated ceramics with density from 0.4 to 0.8 g/cm^3, strength from 3 to 10 MPa and the structural quality ratio from 7 to 13.

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