

## INVESTIGATION OF THE PROPERTIES OF LITHIUM-CONTAINING CERAMICS BASED ON LOW-PLASTIC CLAY

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**ABSTRACT:** Nowadays the production of construction materials and items is a multi-tonnage and actively developing production sphere. The regulatory requirements imposed on construction materials and products provide the assessment of their quality and application efficiency in construction or reconstruction of buildings and structures. The constantly increasing regulatory requirements and the limited availability of high-quality natural reserves demand the rapid development of new raw material resources. This paper presents the results of charge composition development based on low-plasticity clay, which has seen limited application due to the poor performance of items produced from it. However, certain functional additives can be introduced for producing high-quality materials. In this work, alongside lithium carbonate, the following additives have been considered: boric acid used as flux and titanium dioxide as a vitreous phase source. The lithium carbonate effect on total and open porosity, density, thermal conductivity, water absorption, frost resistance, compressive and bending strength has been studied. On the one hand lithium carbonate was found to demonstrate a pore-forming effect due to its decomposition during firing. On the other hand, lithium oxide formed during the additive decomposition facilitated the viscosity reduction of the vitreous phase during firing and its strength increase after cooling. Lithium oxide, similar to boric acid, is a flux, which makes further temperature reduction of liquid-phase sintering possible while preserving the surface self-glazing effect and the formation of the closed-pored internal structure of ceramics. The lithium carbonate pore-forming effect prevails over the flux-hardening and therefore, the amount this additive should be limited to obtain high performance properties and the compliance of resulting ceramics with regulatory requirements. The resulting ceramic material can be used for manufacturing products for buildings' and structures' plinth lining.

**ABSTRAK:** Pada masa kini, pengeluaran bahan dan barangan pembinaan adalah dalam gandaan tan dan aktif dibangunkan terutama dalam bidang pengeluaran. Keperluan pengawalseliaan bahan dan produk pembinaan menyediakan aplikasi penilaian kualiti dan kecekapan pembinaan atau pembinaan semula bangunan dan struktur. Peningkatan berterusan terhadap keperluan pengawalseliaan dan ketersediaan rizab semula jadi berkualiti tinggi yang terhad menuntut kepada pembangunan pesat sumber bahan mentah baru berkembang. Kajian ini memperkenalkan hasil pembangunan komposisi caj berdasarkan tanah liat keplastikan rendah, di mana aplikasi terhad kepada keburukan bahan yang dihasilkan, namun bahan tambahan tertentu boleh diperkenalkan bagi menghasilkan bahan berkualiti tinggi.

Menggunakan litium karbonat bersama bahan tambahan berikut: asid borik digunakan sebagai fluks dan titanium dioksida sebagai sumber fasa vitreus, telah diambil kira dalam kajian ini. Kesan litium karbonat pada keliangan, ketumpatan, kekonduksian terma, penyerapan air, rintangan fros, kekuatan mampatan dan lenturan telah dikaji. Litium karbonat didapati menunjukkan kesan pembentukan liang dalam proses penguraian semasa pembakaran. Sebaliknya, litium oksida dibentuk semasa penguraian aditif dan membantu dalam pengurangan kelikatan fasa vitreus semasa pembakaran dan kekuatannya meningkat selepas penyejukan. Litium oksida, serupa dengan asid borik, adalah fluks, berpotensi menyebabkan suhu sinteran terus berkurang pada fasa cecair sambil mengekalkan kesan kilauan pada permukaan kaca dan menutup pembentukan struktur liang dalaman seramik. Kesan pembentukan liang litium karbonat adalah mengatasi pengerasan fluks. Oleh itu, jumlah bahan tambah ini harus dihadkan bagi mencapai piawaian dan prestasi tinggi seramik yang terhasil. Bahan seramik yang terhasil ini boleh digunakan sebagai lapisan plint bangunan dan struktur dalam pembuatan produk.

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**KEYWORDS:** *facing ceramics; low-plasticity clay; self-glazing; fluxing agent; vitreous phase*

## 1. INTRODUCTION

Nowadays, construction refers to one of the largest capacity and actively developing industries. Annually, the range of manufactured building materials and products is expanding, and their production is constantly increasing. The quality, application, and effectiveness of construction materials and products are determined by certain basic physical, mechanical, and operational properties, as well as their numerical values. The reasons for the construction industry's ongoing development of an already wide range of building materials and products are large scale construction and reconstruction, a variety of structural types, and operating conditions for buildings and structures.

Construction material and product properties are primarily determined by the characteristics and properties of the raw materials used for their production. In most cases, high-quality materials and products meeting regulatory requirements for the properties cannot be manufactured using only one raw material, therefore, raw material mixtures are used in the production processes, with one component being the basic raw and the others serving as functional additives [1-4].

The increasing requirements for operational properties and limited reserves of high-quality natural raw materials cause the necessity to expand the raw material base, primarily meaning the finding of new additives for producing high-quality finished products [5,6]. The decision to use materials and substances as functional additives is related to their impact on the finished product properties and structure, its cost, prevalence, and complexity of its processing [7-10].

Herewith, it should be considered that each additive introduction into the composition of the raw material mixture causes an increase in some property values and a decrease in other property values [11-14]. Therefore, the comprehensive study of the additives' effect in the raw material mixture composition on the material and product characteristics, for both construction and any other purpose, is extremely urgent [15-17].

The research authors have previously experimented with charge composition development for the production of self-glazing facing ceramics based on low-plasticity clay. One of the experiments resulted in the composition development including 5 wt. % of boric acid as a flux, 10 wt. % titanium dioxide as a vitreous phase source and up to 7.5 wt. % of lithium carbonate as a pore-forming additive and glassy phase modifier. The developed composition was good

for manufacturing products that meet the Russian State Standard GOST 13996-93 requirements for tiles used in facing plinths in buildings and structures [18].

Considering the new, recently additionally stringent, regulatory requirements of Russian State Standard GOST 13996-2019, the composition, regarding water absorption, can be used to manufacture the products of B1b and B1Ia groups, characterized by 100 cycles of frost resistance, and bending strength of min 30 and 22 MPa, respectively. However, for previously developed ceramics, frost resistance does not exceed 66 cycles, and bending strength does not exceed 13.6 MPa. In this respect, some additional studies were required to specify the charge composition and the compliance of the resulting ceramics to the requirements concerning bricks and stones for facing the plinths of buildings and structures.

The research objectives were to study the dependencies of the main physical, mechanical, and operational properties of the lithium carbonate content in the charge composition on the developed self-glazing facing ceramics and to determine the studied additive content for ensuring high quality and good operational properties of the resulting products.

## 2. MATERIALS AND METHODS

The main charge component for producing the studied ceramics was the clay of the Suvorotskoye deposit of the Vladimir region with the following composition (in wt. %):  $\text{SiO}_2 = 67.5$ ;  $\text{Al}_2\text{O}_3 = 10.75$ ;  $\text{Fe}_2\text{O}_3 = 5.85$ ;  $\text{CaO} = 2.8$ ;  $\text{MgO} = 1.7$ ;  $\text{K}_2\text{O} = 2.4$ ;  $\text{Na}_2\text{O} = 0.7$ . This clay plasticity index, determined by the standard method, is 5.2, and, therefore, it is of low-plasticity in accordance with Russian State Standard GOST 9169-75 [18,19].

Boric acid brand B, 2nd grade by Russian State Standard GOST 18704-78 with the basic substance content of min 98.6 wt. % was used as a flux in the charge composition. According to previous experimental results by this research's authors, boric acid application in the amount of 2.5 to 5 wt. % makes it possible to increase the ceramic's strength and reduce its water absorption due to the liquid-phase sintering intensification and its temperature decrease [18-20].

Titanium dioxide of brand R-02 by Russian State Standard GOST 9808-84 containing min 93 wt.% of the basic substance was used as the glassy phase source in the charge composition. As one of the research authors has previously revealed, titanium dioxide in the amount from 5 to 10 wt. % also makes it possible to increase the ceramic's strength and reduce its water absorption due to the glassy phase formation [18,21-23]. Simultaneously, the combined introduction of titanium dioxide and boric acid enhances each additive effect and allows the obtention of a self-glazing effect of the surface, thus additionally reducing water absorption and increasing frost resistance [18,21,22].

Lithium carbonate brand CH by TU 6-09-3728-83, containing min 98 wt.% of the basic substance, was additionally introduced into the charge composition. As one of the research authors has previously found, lithium carbonate introduction along other specified additives allows the production of ceramics with a self-glazing effect of the surface and closed porosity in the depth, which facilitates the decrease in thermal conductivity while maintaining low water absorption and high frost resistance [18]. Lithium carbonate is worth noting to serve as a pore-forming additive because lithium oxide formed during firing is more reactive compared to calcium, magnesium, potassium and sodium oxides, and therefore belongs to stronger fluxes, increasing the effect of boric acid and titanium dioxide. In addition, lithium oxide reduces the vitreous phase viscosity, contributing to its uniform distribution in the material depth and penetration into the small pores and voids, and also increases its strength [18,24,25].

The studied ceramic samples were produced applying semi-dry pressing technology. Prior to the application, the clay was crushed to reach the max particle size fraction of 0.63 mm and dried to a constant mass. Then, the charge components were dry mixed at the specified ratios at the beginning, and then with water addition of 8 wt. % to reach a homogeneous mass, from which ceramic samples were produced at 15 MPa pressing pressure and maximum firing temperature of 1000 °C.

The samples were cube-shaped with 50 mm sides and were made in series of three samples for each charge composition, followed by averaging of the study results for each series. For the determination of bending strength, the samples were made in parallelepiped shape of 10x10x70 mm.

The density  $\rho$  (kg/m<sup>3</sup>) has been determined applying the sample mass ratio in the air-dry state to its volume. Compressive strength  $\sigma_{\text{cmp}}$  (MPa) has been determined by continuous and uniform load impact on the sample until its destruction with maximum load fixation. When determining the bending strength  $\sigma_{\text{bnd}}$  (MPa), the sample was mounted on the supports on both sides at 25 mm from the center. The bending load was applied continuously and evenly through the third support installed in the sample middle. To determine the strength characteristics, a hydraulic press P6326B (JSC "Gidropress", Russia) was used.

Water absorption was determined by increasing the dry sample mass after its saturation with water at the temperature of 20±1 °C at atmospheric pressure for 48 hours. The sample frost resistance was determined after the water absorption test. For this purpose, the water-saturated sample was exposed at temperatures ranging from -15 °C to -20 °C for 4 hours, and then placed in ambient water temperature for 2 hours and examined for cracks. If no cracks were observed, a new freeze-thaw cycle was performed.

To determine the total porosity, the true density  $\rho_{\text{true}}$  (kg/m<sup>3</sup>) was determined by the pycnometric method, and then calculated according to the formula:

$$P_{\text{totl}} = (\rho_{\text{true}} - \rho) \cdot 100 / \rho_{\text{true}}$$

Open porosity was calculated using the formula:

$$P_{\text{opn}} = W \cdot \rho$$

Thermal conductivity  $\lambda$  (W/(m·°C)) was determined using an MIT-1 mobile thermal conductivity meter (NPP Interpribor LLC, Russia). For this purpose, 4 cubic samples with 50 mm sides were taken and placed on top of each other. The samples' touching surfaces were thoroughly polished, and then a hole was drilled through the centers of the samples. A measuring probe was placed into the hole, the expected range of thermal conductivity was set and the device was left for 2 hours to set the heating power and stabilize the heat flow. After that, the device readings were recorded, taking into account that the ambient temperature range should be from 20 to 30 °C.

The developed ceramic material composition was studied using a powder diffractometer D8 ADVANCE (Bruker, Germany), a macrostructure applying microscope MBS-10 (JSC Lytkarinsky Optical Glass Plant, Russia), and a microstructure using scanning electron microscope Quanta 200 3D (Systems for Microscopy and Analysis, LLC, Russia).

### 3. RESULTS AND DISCUSSION

Figure 1 demonstrates the dependences of the ceramic's total and open porosity resulting from experimental studies on lithium carbonate in the charge composition, containing 5 wt. % of boric acid and 10 wt. % of titanium dioxide.

The resulting data proves that the material porosity increases along with an increase in the lithium carbonate amount as during firing, this additive decomposes at temperatures above 730 °C and forms lithium oxide and carbon dioxide [18,26]. Meanwhile carbon dioxide acts as a pore-forming agent, and lithium oxide together with boric acid act as a flux. According to the research data concerning the material macrostructure (see Fig. 2), the flux contribute to the material's liquid-phase sintering and predominantly forms the material's closed-pore structure.

The presented dependencies (see Fig. 1) reveal that the greatest porosity increase is observed at the introduction of up to 5 wt. % of the studied additive, and further increase of lithium carbonate amount only increased the proportion of open pores relative to the total material porosity, which grew less significantly. The research data dealing with the ceramic's microstructure (see Fig. 3) demonstrates that this amount of additive causes the fluxing effect of lithium oxide to compensate for the pore-forming effect of the additive to a greater extent. It is manifested in the vitreous phase increase in the ceramic's depth, filling some of the pores and voids in the material volume and reducing the intensity of the total porosity growth.

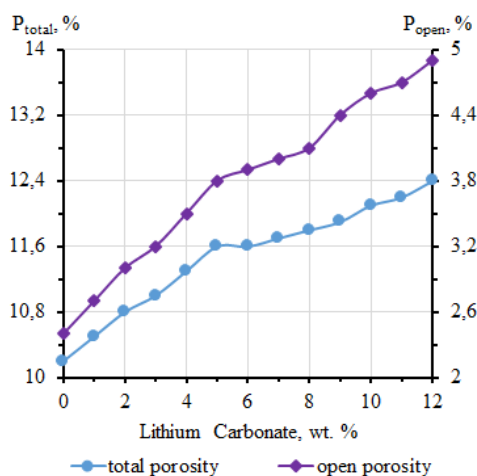


Fig. 1: The effect of lithium carbonate content on the developed ceramic's porosity.



Fig. 2: The developed ceramic's macrostructure with 5 wt. % lithium carbonate introduced into the charge.

Figure 4 presents the dependences of density and thermal conductivity on the quantity of lithium carbonate in the charge composition. The resulting data proved that both considered properties decreased with the increase in the quantity of lithium carbonate. Such dependence



naturally occurs as the pores formed during the lithium carbonate decomposition reduce ceramic density and thermal conductivity. At the same time, the numerical density values decreased from high to medium for construction wall and facing ceramic materials. Thermal conductivity, in turn, decreased from the values typical for clinker products (0.8-1.6 W/(m·°C)) down to the values typical for full-bodied ceramic bricks (0.5-0.65 W/(m·°C)).

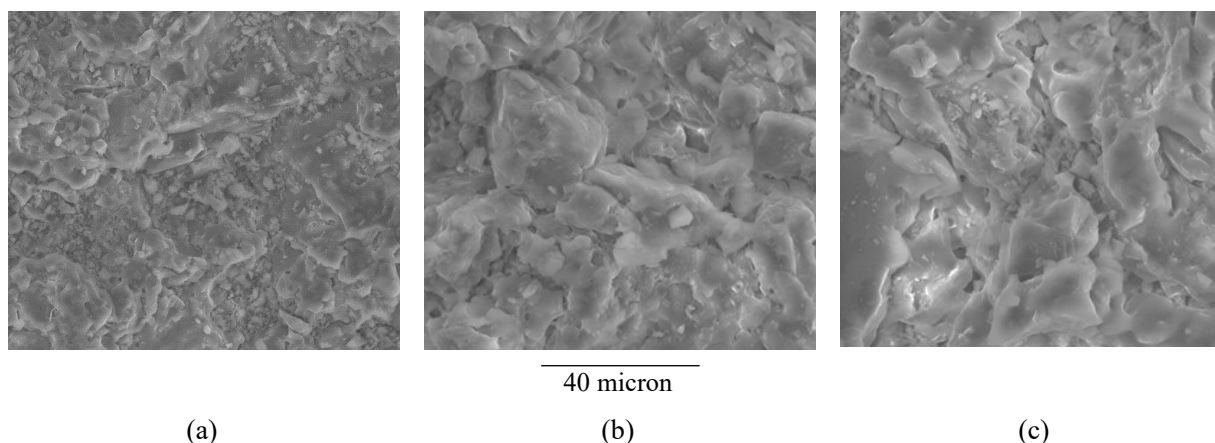


Fig. 3: Microstructure of developed ceramics at different lithium carbonate content in the charge composition: (a) 0 wt. %; (b) 2.5 wt. %; (c) 5 wt. %.

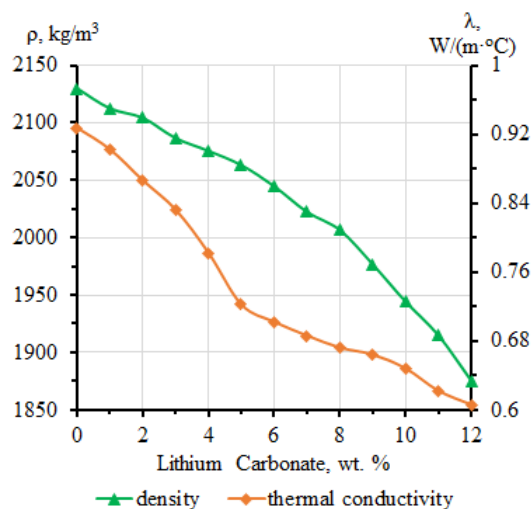


Fig. 4: The effect of lithium carbonate content on the developed ceramic's density and thermal conductivity.

Figure 5 shows the dependences of water absorption and frost resistance of the developed ceramics on lithium carbonate amount in the charge composition. The resulting data demonstrated that the increase in the open porosity share along with the increase in the lithium carbonate amount (see Fig. 1) caused a water absorption increase, which in turn caused frost resistance reduction. The frost resistance reduction depends on the fact that water, penetrating into the open pores, freezes and increases in volume by about 9% when the temperature drops to negative values, causing stress inside the material leading to its destruction during temperature changes.

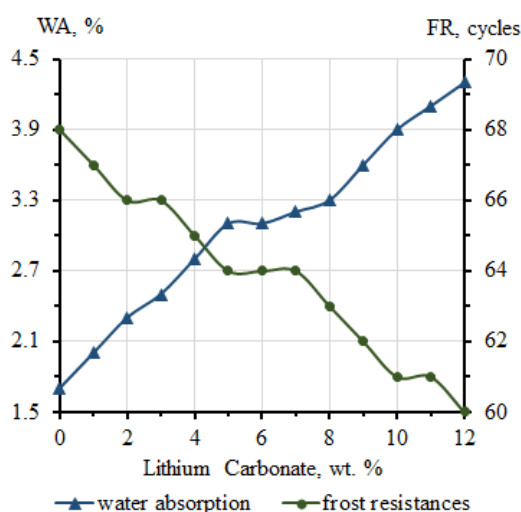


Fig. 5: The effect of lithium carbonate amount on water absorption and frost resistance of the developed ceramics.

The dependence of compressive and bending strength on lithium carbonate amount in the charge is shown in Fig. 6. According to the received data, lithium carbonate significantly reduces the resulting ceramic's strength, which can be explained by the additive pore-forming effect, which, as it was found in previous experiments, was predominant over the fluxing effect of lithium oxide and is associated with a large amount of carbon dioxide formed during the decomposition of lithium carbonate [18].

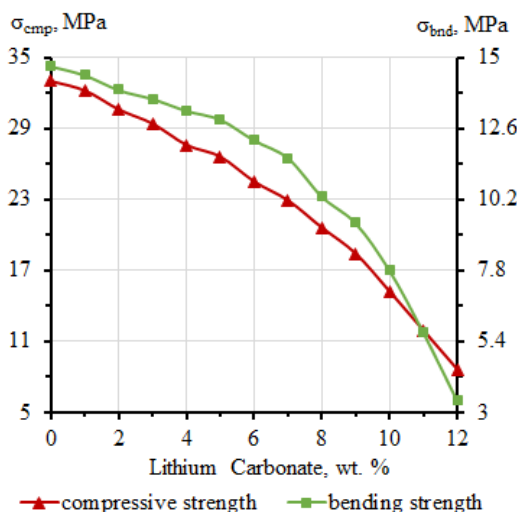
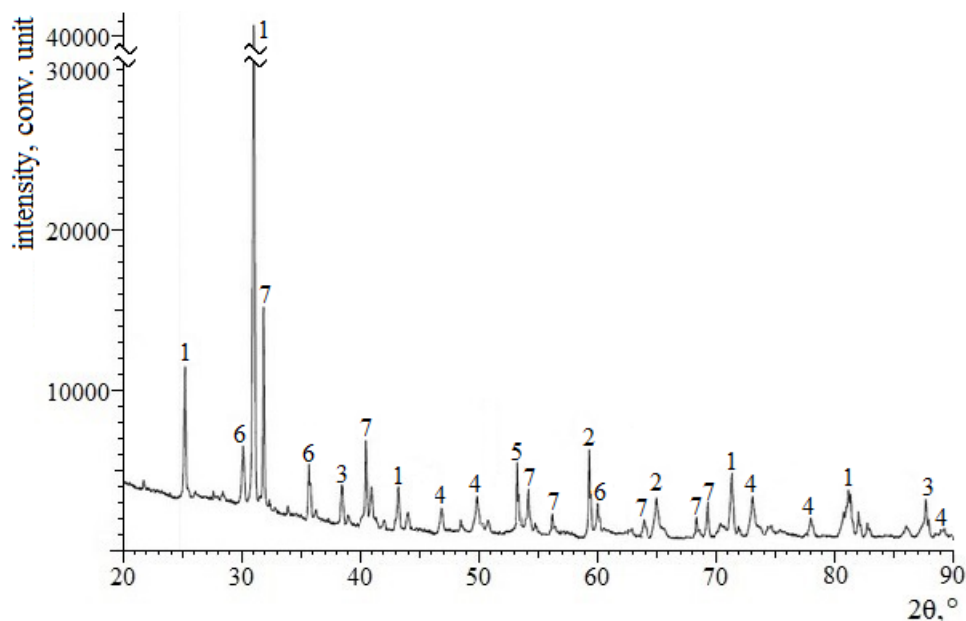
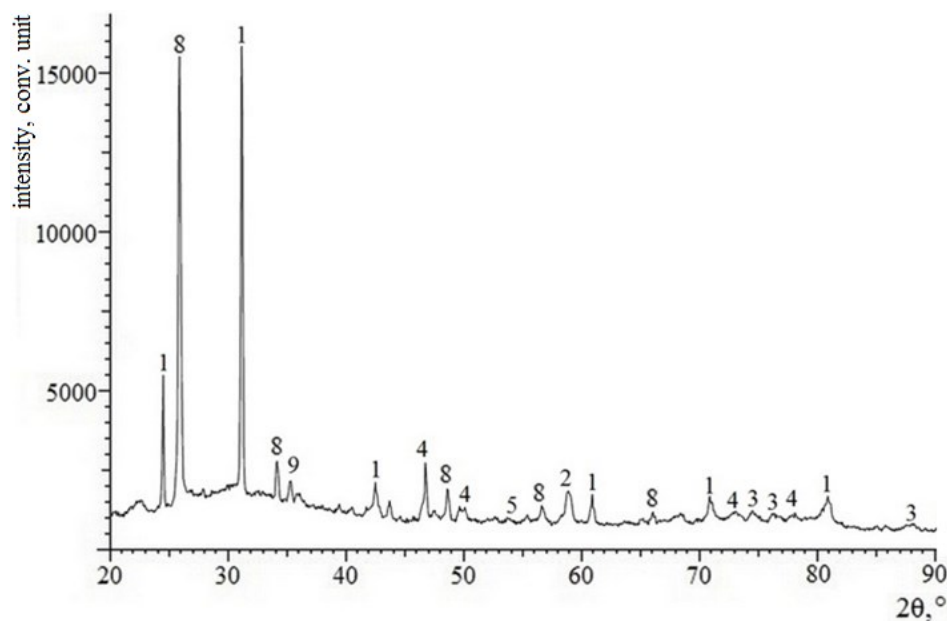


Fig. 6: The effect of the lithium carbonate amount on the developed ceramic's strength.

The resulting dependence was also confirmed by the X-ray phase analysis data (see Fig. 7), which proved that when lithium carbonate was introduced into the charge composition, a considerable reduction in the number of crystalline phases occurred in the developed ceramics. Simultaneously, a significant amount of spodumene appeared, comparable to the silicon oxide amount, as well as a small amount of lithium titanate instead of rutile and iron titanate.



(a)



(b)

Fig. 7: Diffractograms of the developed ceramics based on the charge excluding lithium carbonate (a) and including lithium carbonate (b):

- 1 – quartz  $\text{SiO}_2$ ; 2 – anorthite  $\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ ; 3 – hematite  $\text{Fe}_2\text{O}_3$ ; 4 – diopside  $\text{CaO}\cdot\text{MgO}\cdot 2\text{SiO}_2$ ;
- 5 – spinel  $\text{MgO}\cdot\text{Al}_2\text{O}_3$ ; 6 – iron titanate  $\text{FeTiO}_3$ ; 7 – rutile  $\text{TiO}_2$ ; 8 – spodumene  $\text{Li}_2\text{O}\cdot\text{Al}_2\text{O}_3\cdot 4\text{SiO}_2$ ;
- 9 – lithium titanate  $\text{Li}_2\text{O}\cdot\text{TiO}_2$ .

The decrease in the crystalline phase number, on the one hand, indicates that their aluminosilicate component was involved in the spodumene formation. On the other hand, these phases turn into an amorphous component of the developed ceramics, which in excess can cause increased brittleness and further reduction of the ceramic's strength characteristics, especially bending strength. The phase composition proves that titanium oxide almost completely passed into the composition of phases containing lithium oxide, including lithium



titanate. The significant amount of spodumene, possessing lower mechanical properties than silicon oxide, also contributed to the ceramic's strength decrease.

Since the developed material is planned for the production of bricks and stones, the main properties determining the choice of lithium carbonate amount comprise compressive strength, water absorption, and frost resistance. During the experiments, it was revealed that frost resistance in the entire considered range of changes in lithium carbonate amount in the charge remained constant allowing the application of this material for external cladding (> 50 cycles for face products in compliance with Russian State Standard GOST 530-2012).

The minimum value of water absorption in compliance with this Russian State Standard GOST for facial products is 6%, which exceeds the received values for the developed ceramics. Simultaneously, water absorption for clinker bricks should not exceed 6%, and frost resistance should be min 75 cycles. The compressive strength at 10 wt. of lithium carbonate amounted to 15.2 MPa, thus meeting the requirements for M150 bricks grade, and at 10.8% of lithium carbonate it reached 12.6 MPa, thus meeting the requirements for M125 bricks grade. Facing bricks of these grades are widely used for exterior finishing of buildings and structures having different numbers of stories. The further increase in the amount of the additive causes the decrease in strength, and, consequently, bricks of lower strength grades can be produced, thus limiting the material application scope.

#### 4. CONCLUSIONS

The conducted study's results revealed that the charge composition, based on a low-plasticity clay containing 5 wt. % of boric acid and 10 wt. % of titanium dioxide, can additionally contain up to 10.8 wt. % of lithium carbonate to reduce ceramic thermal conductivity, which increases its energy efficiency maintaining the main operational properties at a level that meets the requirements of Russian State Standard GOST 530-2012 for facing bricks and stones. The resulting products can be considered as an alternative to clinker bricks, although they are somewhat inferior in frost resistance.

Lithium carbonate produces complex effect on the resulting ceramics technology and properties. Firstly, this pore-forming additive allows a reduction in the density and thermal conductivity of the resulting ceramic, which reduces the load on the foundation and supporting structures when using the products and increases their energy efficiency. On the other hand, during lithium carbonate decomposition at firing, lithium oxide was formed, which, alongside boric acid is a flux, and it increased the glassy phase amount during firing and reduced the liquid-phase sintering temperature, which facilitated reduction in the maximum firing temperature from 1050 °C to 1000 °C. Herewith the vitreous phase viscosity decreased, and made it possible to achieve its uniform distribution in the product depth thus increasing its strength.

The pore-forming effect of lithium carbonate prevailed over the fluxing effect of the additive, so its separate introduction reduced the quality of the developed ceramics, based on low-plasticity clay, and the effective application of lithium carbonate was possible only in combination with other functional additives in limited quantities. The combined application of boric acid, titanium dioxide, and lithium carbonate made it possible to produce ceramics with predominantly closed pores and a self-glazing effect of the surface, that is, it reduced density and thermal conductivity while maintaining low water absorption and high frost resistance. At the same time, the material strength met the regulatory requirements for basement facing tiles.

Thus, lithium carbonate in the developed charge composition facilitated the expansion of the raw material base for construction materials through the low-plasticity clay application, which is not in great demand in ceramic production, but is widespread in the manufacture of high-quality products for external cladding of facades and plinths of buildings and structures.

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