



## Study of clay raw materials with Korolek technogenic waste for the production of ceramic bricks

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**Abstract.** The use of industrial waste in the production of building materials is an urgent task at present. Studies on the use of mineral wool waste in the manufacture of ceramic bricks have been carried out. For the preparation of experimental samples of ceramic bricks used clay deposit, as heaters and intensifiers of sintering – waste production of mineral wool. Chemical composition of investigated components is given. The study of mineralogical and phase composition of raw materials was carried out by petrographic, X-ray phase, IR-spectroscopic, electron-microscopic methods of analysis and DTA. During the experiment such properties of samples as cracking temperature; end shrinkage moisture; relative shrinkage; drying time to residual moisture content of 8%; mechanical strength in compression were tested. As a result of the tests, the influence of Korolek on the drying properties of ceramic samples was established. Thus, an increase in the content of Korolek in the samples, improved the crack resistance, relative shrinkage, drying time to residual moisture. The results of the research showed a non-linear dependence of physical and mechanical properties of the samples on the content of Korolek. The best values of mechanical strength were achieved when the Korolek content was 40% of the total mass of the manufactured sample. The proposed composition corresponds to the properties of ceramic bricks above grade M150-175.

**Keywords:** clay, ceramic bricks, waste, mineral wool, strength, shrinkage, cracking, chemical composition.

### 1. Introduction

The authors have conducted research on the development of technology for the production of ceramic bricks from clay raw materials using technogenic waste from the production of mineral wool called “Korolek”. The use of mining and metallurgical industry waste in the production of building materials solves two problems: industrial waste utilization and environmental protection. For the production of ceramic bricks, mineral wool waste, glass waste, high alumina fly ash, and ceramic tile polishing waste are often chosen as a leaning agent [1-2]. Mineral wool waste is used as an additive to Portland cement [3], in the development of ceramic building materials [2], [3], [4], in the production of masonry mortar [5].

The following objectives were set in the study:

- to study the theoretical basis of the use of clay raw materials with technogenic waste «Korolek» for the production of ceramic bricks;
- to carry out an experimental study of the relationship between the content of "Korolek" and the main physical and mechanical properties of ceramic bricks;
- to develop a resource-saving technology for the production of bricks from clay raw materials with technogenic waste «Korolek».

The object of the study – ceramic brick with improved physical and mechanical properties and low cost. Subject of the study – composition and properties of ceramic bricks from clay raw materials and Korolek. The validity of the results of the study is proved experimentally using modern methods and verified instruments.

## 2. Methods

Experimental work was carried out in the Center of competence and technology transfer in the field of construction of the D. Serikbayev East Kazakhstan Technical University (Ust-Kamenogorsk, Kazakhstan).

At the first stage of the study technological samples were taken and properties of clay raw materials and technogenic wastes – Korolek were investigated. Sampling, research of initial clay raw materials and technogenic wastes was conducted in accordance with the guidelines of generally accepted methods and requirements of regulatory documents [6], [7], and recommendations of [8].

The following raw materials were used for the preparation of ceramic brick prototypes:

- as clay raw materials – clay from the Temirtau deposit, clays from the Moisky and Petropavlovsk deposits were used for comparison;
- as heaters and intensifiers of sintering – wastes of mineral wool production in "ISOTERM" LLP (Glubokoe settlement of East Kazakhstan region).

All investigated clay raw materials are yellow-brown in color, dusty to the touch.

The humidity of the soil at receipt – 3.2%, the degree of boiling of the sample of clay raw material in the interaction with 10% hydrochloric acid solution – rapid boiling (Figure 1). In the material provided for the study of large inclusions, which represents quartz, limestone, calcium carbonate, granite, tarmac, plant residues. Clayey raw material was determined by the behavior of the working consistency dough.



Figure 1 – Sampling of clay raw material with 10% hydrochloric acid solution

Clay raw materials contain an admixture of  $\text{CaCO}_3$ . When using clays for the production of ceramic bricks, the raw materials must be passed through mills of less than 1 mm to prevent the bricks from cracking. In the milled state,  $\text{CaCO}_3$  has no harmful effect on the properties of the products. For this purpose, an arc was made, large cracks appeared at the top of the arc when bending. A ball was rolled, which as cracking at the edges when crushed. The study showed that when rolled, the clay dough is able to roll into long cords (Figure 2). A characteristic crunch is heard when cutting.

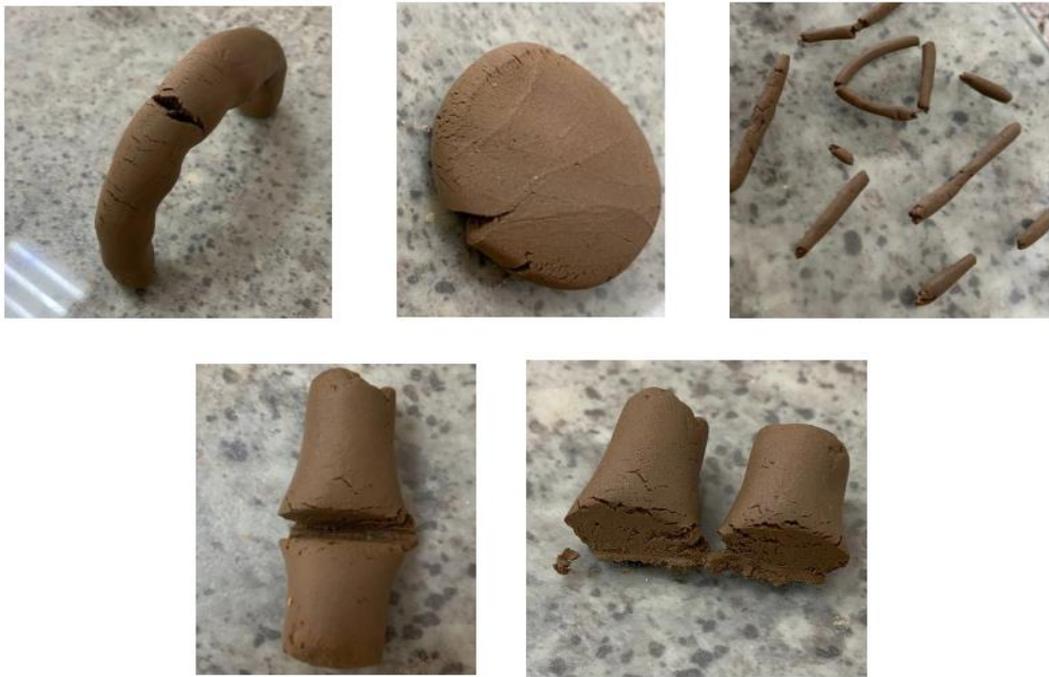


Figure 2 – Investigation of the behavior of clay dough of working consistency

The study of the granulometric composition of the studied clays according to the Rutkovsky method is presented in Figure 3.



Figure 3 – Study of the granulometric composition of clays using the Rutkovsky method

According to the triple diagram of Okhotin [9-10] showing the granulometric composition of the studied soil, determined by the Rutkovsky method, the provided material is a loamy clay.

Grain composition of gravel-sand particles (sieve analysis) was determined after washing out. There were detected the stony inclusions with fractions of 10 mm, 2 mm, 1 mm presented by tarmacs, organics, and quartz sands. The maximum grain size was 30 mm (Figure 4).



Figure 4 – Analysis of granulometric composition of clays

The study of the porous structure of ceramic samples is carried out using a mercury porometer 2000 «Carlo Erba»; the study of the microstructure of ceramic materials is carried out using an electron scanning microscope Phillips 525M.

The formation of the porosity structure of ceramic samples was investigated using the method of small-angle diffuse X-ray diffraction (XRD). Microanalysis of localized areas of mullitized glass of ceramic material was carried out on the installation with microprobe of «Samebax» company.

The study of mineralogical and phase compositions of raw materials is carried out by petrographic, X-ray phase, IR spectroscopic, electron-microscopic methods of analysis and DTA. Powder X-ray diffractograms are obtained on diffractometer DRON-2 under the conditions of imaging: angle range from 6 to 70° using CuK $\alpha$ -radiation. IR-absorption spectra were obtained on a Spectrocard 75IR spectrograph. To analyze the particle size in the raw materials, metallographic analysis was carried out on a MIM-8M microscope.

In order to obtain complete information about the structure formation in ceramic materials, the microstructure is studied using an electron microscope EMV-100B. Dilatometric studies are carried out on a DKV-5A dilatometer in the temperature range of 20-700 °C [11].

The results of clay research are summarized in Tables 1-3. The chemical composition of the investigated components is summarized in Table 1.

Table 1 – Chemical compositions of raw materials

Components	Oxide content, %							Impurities
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Fe <sub>2</sub> O <sub>3</sub>	R <sub>2</sub> O	SO <sub>3</sub>	
	Clays							
Temirtau	56.08	13.40	2.02	1.24	5.14	3.90	1.03	3.87
Moiskoe	68.97	16.00	1.37	1.47	2.50	4.28	0.50	3.87
Petropavlovskoe	75.68	11.50	2.05	0.27	2.67	2.55	0.20	4.22
	Mineral wool production waste							
Korolek	43.20	7.30	23.60	14.60	6.72	2.79	0.90	0.80
WPR mineral wool flue gas cleaning product	15.30	7.98	31.20	7.60	10.60	6.79	0.98	19.30

Mineralogical compositions and technological properties of clays are shown in Tables 2 and 3. In addition to clays, the properties of mineral wool production wastes were investigated. In the process of mineral wool production not all melt drops have time to stretch into threads. A part of them takes the form of balls, flagella, etc. Such inclusions are called Korolek.

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Table 2 – Mineralogical composition of clays

Deposit	Mass fraction of mineral in clays, %						
	Hydro mica	Quartz	Gypsum	Feldspar	Kaolinite	Beidel lith	Iron oxides
Temirtau	5-10	20-25	2-3	10-15	3-5	35-45	5-7
Moiskoe	25-30	25-30	5-7	10-15	10-15	–	4-7
Petropavlovskoe	–	10-20	2-4	20-30	45-50	–	1-3

Table 3 – Technological properties of clays

Deposit	Plasticity number	Content of clay particles smaller than 0.005	Refractoriness refractoriness °C	Sinterability without deformation distortions
Temirtau	15-24	40-55	1320-1350	sinter
Moiskoe	7-9	15-25	1100-1200	sinter
Petropavlovskoe	10-15	30-35	1520-1550	sintered

The product of flue gas purification from the furnace during melt production in the production of mineral wool (WPR mineral wool) is also a waste and is disposed of in the production of mineral wool in separate receivers [12].

Figure 5 shows images of Korolek and the product of purification of flue gases from flue gases of mineral wool WPR, made on the Phillips 525M scanning electron microscope - according to E. Vdovina [12].

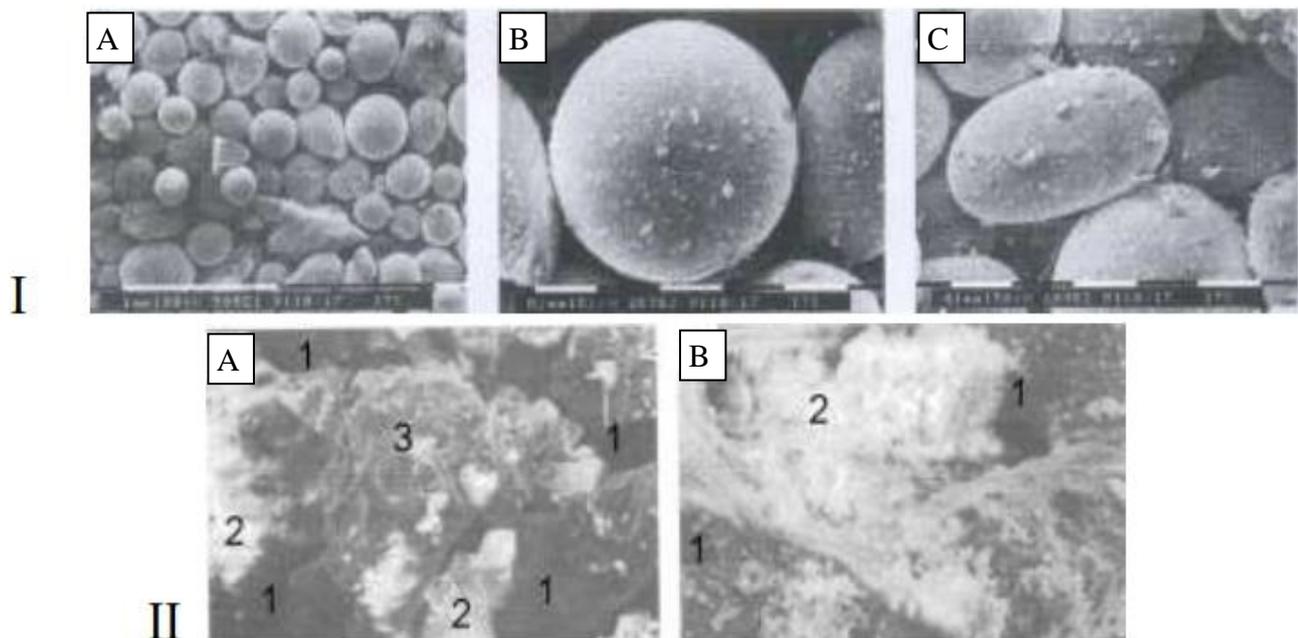


Figure 5 – Korolek (I) and WPR (II) at magnifications of IA 50x, IB 200x, IC 200x, IIA 100x, IIB 1000x: 1 – organics; 2 – glass phase; 3 – hematite

Studies of clay raw materials have shown that in the clay of the Temirtau deposit, unlike the clays of the Moisky and Petropavlovsk deposits, the presence of swelling mineral beidellite contributes to more dramatic changes in viscosity and increased moisture content. Therefore, to obtain ceramic bricks based on clay of the Temirtau deposit, it is necessary to introduce heaters into the composition of ceramic masses. As a heater in the experiment was used technogenic waste Korolek.

Thus, at the first stage analytical and laboratory studies of clay raw materials and technogenic waste – Korolek were carried out, primary information about chemical and material composition of raw materials for preparation of clay mass and experimental samples from it was obtained.

For the preparation of test samples were taken clay of the Temirtau deposit (Figure 6) and Korolek – waste products of mineral wool production of «ISOTERM» LLP.



Figure 6 – Clay of Temirtau deposit and mineral wool waste Korolek for preparation of prototypes

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The samples were molded by plastic method at charge moisture content of 20-25%. Samples of the following shapes and sizes were made:

- 50x50x10 mm tiles – for studying air and fire shrinkage and research of clay sinterability;
- cubes 25x25x25 mm – to determine the coefficient of sensitivity to drying and to study the change of compressive strength depending on the firing temperature;
- 15x50x10 mm plates – to determine the bending strength [13].

To investigate the influence of Korolek on the drying properties of ceramic bricks, three compositions were studied, presented in Table 5.

Table 5 – Composition of prototypes

Component	Mass fraction, %		
	Sample 1	Sample 2	Sample 3
Clay from the Temirtau deposit	70	60	50
Korolek	30	40	50

The following properties of the specimens were tested during the experiment:

- temperature at which cracks appear;
- moisture content at the end of shrinkage;
- relative shrinkage;
- drying time up to residual moisture content of 8%;
- mechanical strength in compression.

Methods of research of drying properties of samples are carried out according to [6].

The results of determining the drying properties of experimental samples are summarized in Table 6.

Table 6 – Drying properties of ceramic brick prototypes

Indicator	Sample number		
	Sample 1	Sample 2	Sample 3
Temperature at which cracks appear, °C	130	145	155
Moisture at the end of shrinkage, %	6	7	10
Relative shrinkage, %	5.8	3.4	2.0
Drying time to residual moisture content of 8%, hours	72	68	48
Mechanical compressive strength to residual moisture 7-8%, MPa	8.6	7.8	5.5

### 3. Results and Discussion

In order to study the effect of Korolek on the physical and mechanical properties of ceramic bricks, eight compositions were prepared (Table 7).

Table 7 – Compositions of ceramic samples – series 2

Component	Mass fraction, %							
	1	2	3	4	5	6	7	8
Clay	100	80	75	70	65	60	55	50
Korolek	0	20	25	30	35	40	45	50

The compositions were also prepared by plastic method at a charge moisture content of 20-25%. The molded specimens, dried to a residual moisture content of not more than 7-8%, were fired at 1050°C. The samples were tested for frost resistance according to [14]. Tests for mechanical compressive strength were carried out according to [15].

The results of testing the samples for frost resistance and mechanical compressive strength are summarized in Table 8.

Table 8 – Test results of samples for frost resistance and mechanical compressive strength

Component	Sample number							
	1	2	3	4	5	6	7	8
Frost resistance, cycles	67	85	91	103	105	108	98	82
Mechanical compressive strength, MPa	17,3	19,3	20,9	22,7	23,8	24,8	21,4	18,9

Thus, during the experiment the properties of clay raw materials were tested, ceramic samples were tested for drying properties (end shrinkage moisture, relative shrinkage, drying time to residual moisture content of 8%, etc.) and physical and mechanical properties (frost resistance and mechanical compressive strength).

To reveal the dependence of crack formation temperature on the content of Korolek on the experimental data (Table 6), a correlation graph was plotted (Figure 7). Further we investigated the dependence of the moisture content of the end of shrinkage (in %) on the content of Korolek in the laboratory samples. The dependence of the moisture content of the end of shrinkage (in %) on the content of «Korolek» in the experimental sample is shown in Figure 8.

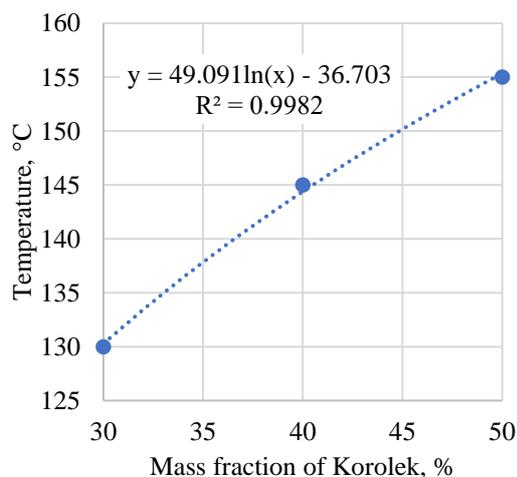


Figure 7 – Dependence of cracking temperature on the content of Korolek

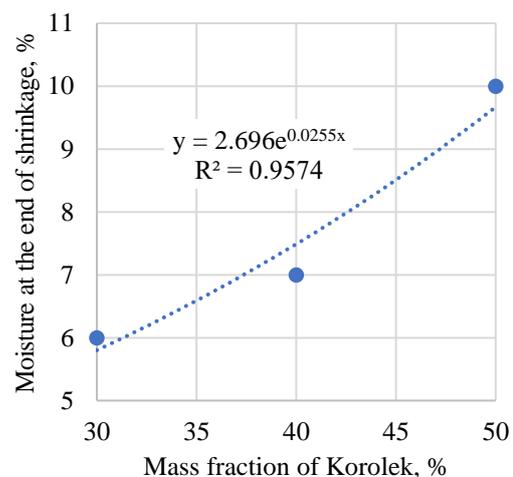


Figure 8 – Dependence of shrinkage on the moisture of Korolek

From the data presented in Figure 7, it can be seen that as Korolek increases, the cracking temperature of the laboratory samples increases. Thus, it is obvious that the addition of waste

Korolek to the clay raw material increases the crack resistance of ceramic products. Analysis of the data presented in Figure 8 indicates: increasing the content of Korolek increases the moisture content of the end of shrinkage.

To reveal the influence of anthropogenic waste on the relative shrinkage of test samples, a graph is plotted (Figure 9).

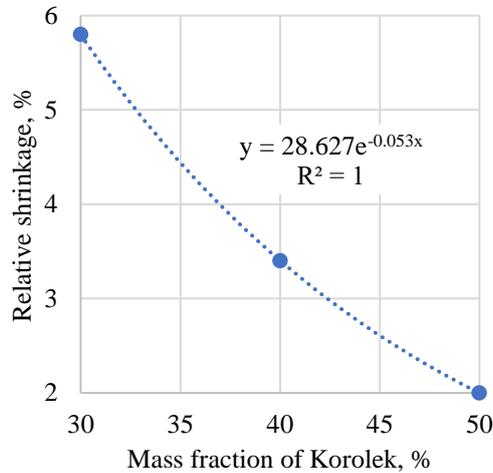


Figure 9 – Effect of Korolek content on the relative shrinkage

From the data presented in Figure 9, it can be seen that the more Korolek is introduced into the clay mass, the less relative shrinkage of the samples. Thus, when introducing into the clay 30% of man-made waste Korolek, the relative shrinkage of the sample is 5.8%, and when introducing 50% of Korolek, the relative shrinkage is 2.0%. Hence, the drying and technological parameters of the experimental samples are significantly improved by increasing the content of Korolek.

Figures 10 and 11 show the changes in physical and mechanical properties of the samples at different contents of Korolek.

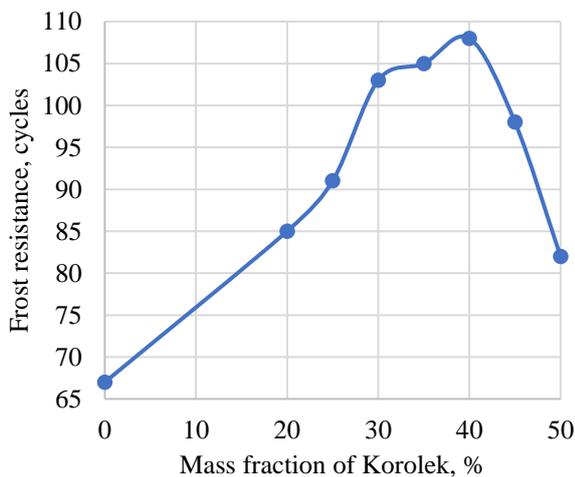


Figure 10 – Changes in frost resistance

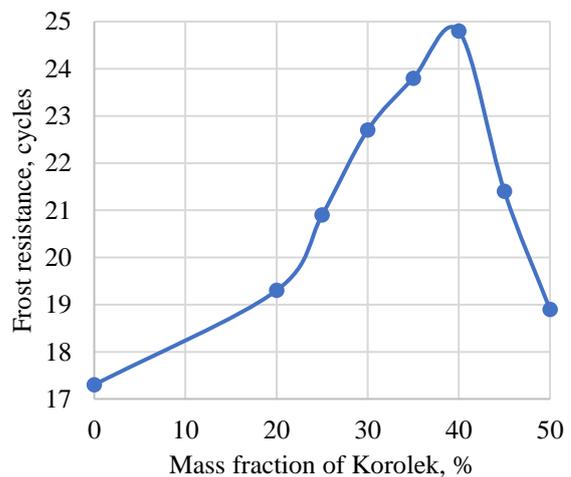


Figure 11 – Changes in mechanical compressive strength

From the above it is clear that as the proportion of Korolek in the dried sample increases, the mechanical compressive strength of the sample dried to a residual moisture content of 7-8% decreases.

The constancy of the effect is obvious: an increase in crack resistance is accompanied by an increase in shrinkage end moisture content. It follows from this that optimization of the composition of ceramic samples by the content of Korolek will be required due to the ambiguous influence of

the waste on important drying parameters and mechanical strength. Thus, the dependence of physical and mechanical properties of the samples on the content of Korolek is nonlinear. The analysis shows that the mechanical compressive strength nonlinearly depends on the content of Korolek waste in the samples. It can be stated that the best values of mechanical strength can be achieved at the Korolek mass fraction of 40%. The chemical nature of the process is explained by the fact that if the content of Korolek in the charge, in which CaO is 23.6%, exceeds 35%, the sintering at a temperature of 1050°C begins to deteriorate, but very slightly up to 40%, and then sharply. This is explained by the fact that the increase in the content of CaO in the ceramic mass, significantly intensifies the crystallization of anorthite, which prevents sintering [17].

Thus, the analysis shows that the optimal composition of the experimental sample is the ratio of the Korolek:clay of 40:60. Compliance of laboratory samples with the requirements of [16] is presented in Table 9.

Table 9 – Compliance of the best prototype with requirements [16]

Indicators	Full-body ceramic bricks		
	Korolek:clay of 40:60	Brick of grade M150	Brick of grade M175
Compressive strength, MPa	18.8	16.8	17.5
Bending strength, MPa	3.2	2.8	3.1
Brick density, kg/m <sup>3</sup>	1800	1400	1400
Frost resistance, cycles	>50	35	45
Water absorption, %	8	10	8

Thus, the proposed composition corresponds to the properties of ceramic bricks above grades of M150-175 according to [16].

#### 4. Conclusions

The following main conclusions can be formulated based on the results of the study:

- Increase in the content of Korolek waste improves the crack resistance, relative shrinkage, drying time to residual moisture, as well as worsens the end shrinkage moisture content and mechanical compressive strength of the samples dried to residual moisture content of 7-8%;
- Addition of Korolek waste to the clay in the ratio of 40:60 significantly improves the physical and mechanical properties of ceramic bricks.

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