

## THE DEPENDENCE OF THE VALUE OF CERAMICS RESISTANCE TO FROST ON THE COMPOSITION OF RAW MATERIAL MIXTURE

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Received 21 Oct. 2009; accepted 30 Sept. 2010

**Abstract.** The operational resistance of ceramic products to frost is a persistent problem. In order such products should remain of high quality without losing their initial qualities for a long time, it is necessary to improve the technological process of production. This work is confined to selecting the composition of the formation mix in order to turn attention to the amount of every component of the formation mix that can positively or negatively influence the operational resistance of ceramics to frost. The paper presents 8 formed batches of ceramic samples different in composition while being dried and burned under similar conditions. Operational resistance to frost was determined according to the established standard. The performed analysis of the obtained experimental data allowed working out an empirical equation forecasting operational resistance to frost according to the composition of the formation mix.

**Keywords:** ceramics, resistance to frost, formation mix, properties, statistical analysis.

### 1. Introduction

An increase in the operational resistance of products to frost is a very recurring problem, especially in Lithuania, because the buildings built a few years lose their original appearance (rip off, crumble etc.) and start collapsing. Resistance of a porous body to frost is a physical feature indicating the ability to maintain the change limits of some physical parameters set before the porous body was alternately frozen and thawed after being soaked.

The resistance of material to frost is expressed in the number of freezing and thawing cycles where the product coped without any collapse. The criteria of evaluating resistance to frost are as follows: a decrease in compressive strength, mass change, visible breaks and other abrasions (Mačiulaitis 1996; Мачюлайтис 1997).

We need to regulate the operational resistance of ceramic products to frost just from the initial moment of the technological process that includes the selection and dosage of raw material. Therefore, we need to know how the amount of each component included in the formation mix influences the final properties of ceramics and especially operational resistance to frost.

Some scientists (Petrikaitis 1999; Mačiulaitis *et al.* 1995; Daunoravičiūtė and Petrikaitis 1997; Kizinievič *et al.* 2005) were investigating how the components of the formation mix (the supplements of peat, coal, sawdust, the dust of wood and dolomite) might influence the final properties of ceramics, including resistance to frost. They concluded that a supplement of 1.42% of coal (to 1.5 mm) to the formation mix increased the indicator of the reserve of pore volume to 22% and the operational

resistance of products to frost – to 42 cycles. However, compressive strength decreases about 33%. The quantity of 4% of dry peat and 1.5% of coal in the formation mix increase the indicator of the reserve of pore volume to 37% and the operational resistance of products to frost – to 60 cycles. However, compressive strength also decreases. More than 5% of sawdust in the formation mix increases the values of the reserve of pore volume to 30%, however, it extremely decreases the strength of the products. When decreasing the amount of sawdust to 4% and adding 1% of coal, the values of the reserve of pore volume and mechanical strength increase, however, operational resistance to frost includes only 48 cycles. The supplements of (3–4%) of the dust of wood and (1–1.5%) of coal conclude less reserve pores than the same amount of sawdust and peat, and thus the mechanical strength of products does not increase. Moreover, the preparation of the formation mix with the dust of wood is more complex; therefore, the scientists suggest not using the above-mentioned supplement.

A minor dispersal supplement of dolomite raises the operational resistance of products to frost; however, larger dolomite worsens most of the final properties of ceramics. Chalk increases the mechanical strength of ceramic bricks, however, operational resistance to frost reaches 49 frost-defrost cycles. The work by Mačiulaitis *et al.* (2004) determined the kind of structural indicators that in most cases influenced an increase or a decrease in resistance to frost. The scientists (Kizinievič and Petrikaitis 2005; Kizinievič *et al.* 2006) also were exploring the influence of clay properties, reducing, firing, waste addition, mixing efficiency, the degree of pressure in

formation head, the depth of rare characteristics of the vacuum chamber and the duration of keeping at the highest firing temperature considering resistance to frost. Other scientists (Correia *et al.* 2004a) discovered that poppy-seeds in ceramics formed more closed pores and the density of such samples made about 2500 kg/m<sup>3</sup> as the mix was burned at a temperature of 1570 °C. The valuable results of the performed analyses are described in the work by (Bhattacharjee *et al.* 2007) showing how the porosity of aluminous (alumo-silicates) articles depends on the amount of albumin and starch in the formation mix. Adding albumin to the formation mix allows forming more closed and less open pores, and starch increases in the number of joined pores.

Some scientists (Gregorová and Pabst 2007; Dondi *et al.* 2003; Correia *et al.* 2004b) derived empirical equations with a possibility of forecasting the values of shrinking ceramics and water absorption in accordance with the composition of the formation mix (clay, feldspar and quartz). It is found that when increasing the amount of feldspar in the formation mix up to 65% we would obtain water absorption of ceramic crushed bricks close to zero and when increasing the amount of quartz in the formation mix up to 65% we would highly decrease the firing shrinkage of the samples (from 11.35% to 3.32%). However, these scientists did not define how the components of the formation mix would influence the operational resistance of ceramics to frost. The scientists (Raimondo *et al.* 2009) also researched the durability of clay roofing tiles having influence on phase composition. It was established that quartz, plagioclase and pyroxene made the most positive influence on the resistance of clay roofing tiles to frost.

The aim of this work is to find out the components of the formation mix having the most positive and negative influence on the operational resistance of ceramics to frost. Besides, the equation forecasting the operational resistance of ceramics to frost in accordance with the amounts of the components of the formation mix was determined.

**2. Characteristics of Materials. Research Methods**

The samples were formed based on plastic shaping from raw materials as follows: clay from the Girininkai deposit, sand from the Daugeliai deposit, crushed bricks (milled waste of ceramic bricks) from Rokai factory, peat

from the Rekyvai deposit, anthracite from Archangelsk region and milled glass. The X-ray pattern (Mačiulaitis and Žurauskienė 2007) of the main raw material – clay from the Girininkai deposit is presented in Fig. 1 and chemical and granulometric (Mačiulaitis *et al.* 2008; Mačiulaitis and Malaiškienė 2009; Mandeikytė and Šiaučiūnas 1997) compositions are accordingly shown in Tables 1 and 2.

According to the analysis of the X-ray pattern of clay from Girininkai (Mačiulaitis and Žurauskienė 2007) (Fig. 1), the minerals of clay are as follows: hydromica *H* (0.990, 0.498, 0.448, 0.256, 0.199) nm, kaolinite *K* (0.710, 0.335, 0.199) nm, chlorite *X* (1.410, 0.710, 0.355) nm, quartz *Q* (0.425, 0.335, 0.245, 0.228, 0.224, 0.213) nm, dolomite *Do* (0.288, 0.240, 0.219) nm, calcite *Ca* (0.304, 0.249, 0.209, 0.191, 0.188) nm and some feldspar *Ls* (0.324 nm). Based on the fired process of the initial clay minerals from Girininkai, the phases are formed as follows: hematite *F* (Fe<sub>2</sub>O<sub>3</sub>), gelenite *G* (2CaO·Al<sub>2</sub>O<sub>3</sub>·SiO<sub>2</sub>), anortite *A* (CaO·Al<sub>2</sub>O<sub>3</sub>·2SiO<sub>2</sub>), diopside *D* (CaO·MgO·2SiO<sub>2</sub>), cristobalite *Kr* (SiO<sub>2</sub>) and glass phase.

According to the chemical composition, clay mixture from Girininkai is half-acid with a great amount of iron oxide.

In addition, it includes a great amount of CaO + MgO (more than 10%), which contracts a sintering interval, and therefore, conditions for firing ceramic articles become worse and the obtained products are less firm and not resistant to frost.

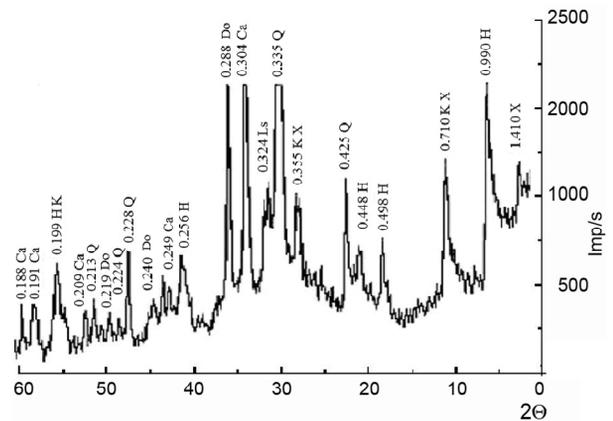


Fig. 1. The X-ray pattern of clay from the Girininkai deposit

Table 1. The average chemical composition of clay

Chemical composition, wt %								
SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub> +TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	SO <sub>3</sub>	L.O.I
47.66	18.32	6.27	8.11	3.04	2.68	0.16	–	12.60

Table 2. The average granulometric composition (mm) of clay

Particle size distribution, wt %							
more than 0.5	from 0.5 to 0.2	from 0.2 to 0.09	from 0.09 to 0.06	from 0.06 to 0.01	from 0.01 to 0.005	from 0.005 to 0.001	less than 0.001
0.13	0.07	0.10	0.08	4.58	9.28	24.28	61.48

**Table 3.** The compositions of formation mixes

Formation mix	The amount of clay $x_1$ , %	The amount of sand $x_2$ , %	The amount of glass $x_3$ , %	The amount of crushed bricks $x_4$ , %	The amount of anthracite $x_5$ , %	The amount of peat $x_6$ , %
1	74.5	18.0	0.0	6.0	1.5	0.0
2	80.0	15.0	0.0	0.0	0.0	5.0
3	70.5	12.0	10.0	6.0	1.5	0.0
4	69.5	18.0	5.0	6.0	1.5	0.0
5	59.5	18.0	15.0	6.0	1.5	0.0
6	54.0	30.0	10.0	5.0	1.0	0.0
7	82.5	10.0	0.0	5.0	0.0	2.5
8	82.5	10.0	0.0	5.0	2.5	0.0

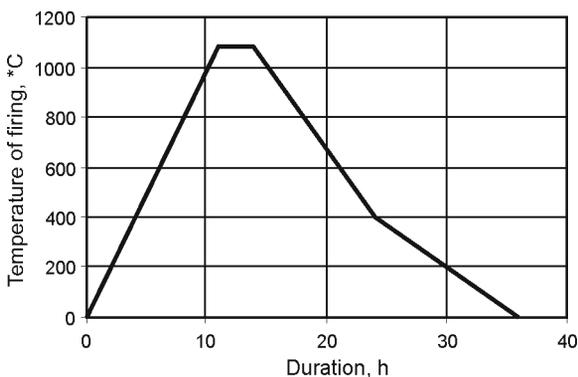
Particularly damaging are insertions larger than 1 mm of  $\text{CaCO}_3$ , because firing semi-manufactures produce CaO able to slake in a humid environment which destroys the products.  $\text{K}_2\text{O}$  and  $\text{Na}_2\text{O}$  make the melting temperature of clay lower, accelerate sintering clay and extend the sintering interval. However, the quantity of these oxides is sufficiently low (about 2%). L.O.I. is composed of organic impurities, water from clay minerals and  $\text{CO}_2$ , which is split from carbonates. According to the quantity of clayey particles, this clay is very dispersive (Malaiškienė 2008).

The compositions of the formation mixtures selected to form ceramic samples are presented in Table 3.

Selecting the composition of formation mixes, it was taken into account that an additive of milled glass was effective when came up to 20% in a formation mix. Also, this addition must be dispersive enough, because during the firing process, larger particles of milled glass may diffuse into the surface of a product (Paulaitis and Vyšniauskas 1995).

The dosage of components was performed according to mass. First, a dry formation mix was stirred manually, later it was irrigated to moisture appropriate to form. Such a mass is left for three days in an environment of  $(95 \pm 5\%)$  of humidity in order to distribute moisture in the formation mix evenly. In three days time, the ceramic samples were formed and obtained dimensions of  $70 \times 70 \times 70$  mm.

First, the formed semi-manufactures were dried in a laboratory under natural conditions, and at a later stage – in the electrical stove. Consequently, the dried samples were fired in the electrical stove under an appropriate regime (Fig. 2).

**Fig. 2.** The graph of firing

Five samples were selected from each batch in order to determine resistance to frost that was determined following LST 1272-92 standard (LST 1272-92 1993) and applying the one-sided freezing and thawing method.

The samples had been soaked for three days before the experimental fragment was formed.

Then, the fired samples were frozen for 8 hours from minus 15 °C to minus 20 °C. Next, the samples underwent the thawing procedure for 8 hours at a temperature of  $(20-25\text{ }^\circ\text{C})$  (LST 1272-92 1993). The destruction of ceramics was rated according to its cycle of disintegration starting using any criteria of the collapse of a cold surface: stratification, crumbling, cracking and cleaving. The physical-mechanical and structural parameters of formed, dried and burned ceramic samples were determined following LST EN 771-1+A1 and other known methodologies (Kičaitė *et al.* 2010; Malaiškienė 2008; Mandeikytė and Šiaučiūnas 1997).

### 3. Research Results

The average values of some physical-mechanical and structural properties are presented in Table 4 showing that maximum density, compressive strength, the reserve of pore volume and minimum water absorption have samples providing a maximum amount of milled glass (formation mixes 3, 5, 6). The sample indicating a maximum amount of peat (formation mix 2) has minimum density, compressive strength, the reserve of pore volume and maximum water absorption. With reference to the obtained results, it can be predicted that the samples of

**Table 4.** The average values of physical-mechanical and structural properties:  $\rho$  – density,  $R_{gp}$  – compressive strength,  $R_p$  – reserve of pore volume,  $W_h$  – water absorption following 72 h

Formation mix	$P$ , $\text{kg/m}^3$	$R_{gp}$ , MPa	$R_p$ , %	$W_h$ , %
1	1752	24.34	43.64	9.31
2	1624	19.35	39.56	12.72
3	2022	39.11	65.75	2.90
4	1920	29.74	49.15	5.75
5	2246	40.99	71.15	2.34
6	2170	28.94	48.62	4.34
7	1626	27.86	48.43	8.80
8	1664	29.20	47.28	8.21

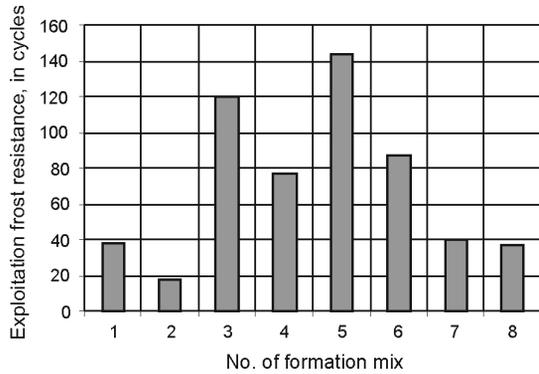


Fig. 3. The average values of operational resistance to frost (y)

formation mixes 3, 5 and 6 will have the highest value of resistance to frost, whereas the samples of formation mix 2 – will have the lowest one.

The average experimentally determined values of operational resistance to frost are presented in Fig. 3. The view of the samples followed the one-sided freezing and thawing procedure is shown in Fig. 4.

Fig. 3 shows that the maximum values of operational resistance to frost are derived from the samples of batches 3, 4, 5 and 6 in which milled glass was used as an additive. In other cases, the values of operational resistance to frost are significantly lower.

Fig. 4 indicates that following one-sided freezing and thawing ceramic samples start flaking off.

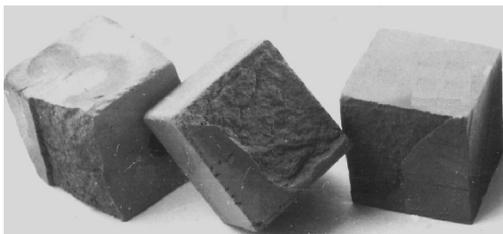


Fig. 4. Samples following one-sided freezing and thawing (from formation mixes 3, 4, 5)

The X-ray pattern of the most characteristic third formation mix (Table 1) is presented in Fig. 5.

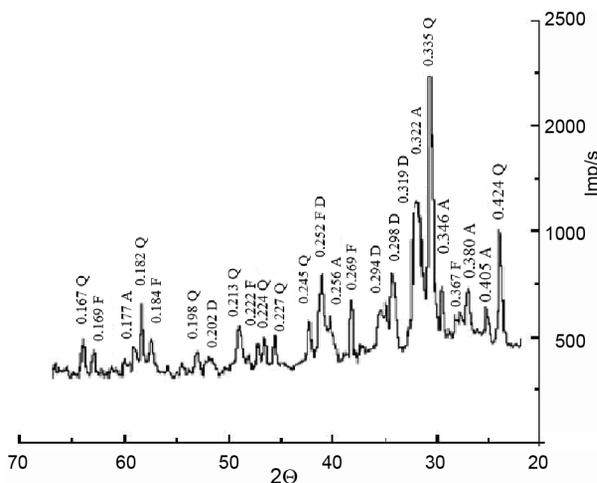


Fig. 5. The X-ray pattern of the sample from the third formation mix (Table 1)

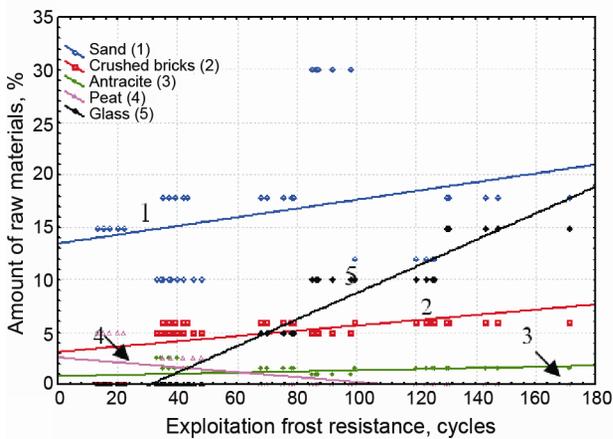
Phases as: quartz *Q* (0.153, 0.167, 0.182, 0.198, 0.213, 0.224, 0.227, 0.245, 0.334, 0.424) nm, hematite *F* (0.169, 0.184, 0.222, 0.252, 0.269, 0.367) nm, diopside *D* (0.202, 0.252, 0.294, 0.298, 0.319) nm and anortite *A* (0.177, 0.256, 0.322, 0.346, 0.380, 0.405) nm were identified.

#### 4. Statistical Data Analysis

Statistical data analysis was performed in order to determine how the value of the operational resistance of ceramic products to frost depended on the content of the components of the formation mix. Grouping data and preparation for research were performed applying “Microsoft Excel” and “Statistica” programs. Statistical analyses were made according to available literature (Čekanaavičius and Murauskas 2002; Манита 2001; Ostle *et al.* 1996; Huang and Hsueh 2007). In order to determine mathematical interdependence, the function having multivariate correlation and determination coefficients closest to one was selected. Regression analysis also finds useful to know the average standard deviation measure from the regression graph. The average standard deviation measure is defined as the square root of the fixed square sum of the deviation of errors (Kleinbaum *et al.* 1998; Graybill *et al.* 1994). It was verified in case the distribution of experimental results was normal using Kolmogorov-Smirnov criterion (Манита 2001). If the value of the introduced criterion is lower than that presented in the statistical table (selected according to the number of samples and the level of importance (in our case it makes 0.05), it is considered a normal distribution of data. For example, as we analyze the values of 40 samples at a significance level of 0.05, the value presented in Kolmogorov-Smirnov statistical table makes 0.210 (Манита 2001). The adequacy of the derived equations was checked using Fisher criterion. If the above mentioned indicator of an equation is higher than that presented in the reference table, the equation is considered adequate and appropriate to describe experimental data. For example, when studying 40 samples at a significance level of 0.05, the value presented in the table of Fisher criterion is equal to 2.44 (Манита 2001). The significance of the variables of the equation was determined applying the Stjudent criterion. If the value of the indicator is higher than that presented in the reference table (when studying 40 samples at a significance level of 0.05, the value presented in the statistical table of the Stjudent criterion is equal to 1.96), it is considered a significant indicator (Gatti 2005).

First, the diagram showing how each supplement of the formation mix influences operational resistance to frost was drawn (Fig. 6).

Fig. 6 displays that the operational resistance of ceramics to frost is most positively influenced by the amount of milled glass in the formation mix. Also, the operational resistance of ceramics to frost is positively influenced by the amount of crushed bricks, sand and anthracite. The amount of peat in the formation mix decreases operational resistance to frost.



**Fig. 6.** Dependence of operational resistance to frost on the amount of sand, crushed bricks, anthracite, peat and glass

The regression Eq. (1) evaluating the influence of the components of the formation mix on the indicator of operational resistance to frost ( $y$ ) was formed according to the direct progressive forward stepwise method ensuring the progressive insertion of independent variables having the highest coefficients of partial correlation with the dependent variable in calculating the regression equation (i.e. the indicators are connected progressively in order the sum of deviations is the smallest). The values of correlation, determination and average standard deviation as well as the Student criterion of the derived empirical equation are presented in Table 5.

$$y = -82.08 + 1.23x_1 + 8.48x_3 + 4.48x_4. \quad (1)$$

Table 5 shows a strong linear relationship between operational resistance to frost and the components of the formation mix because the correlation coefficient of 0.984 is very close to one. According to the signs of empirical equation (1) and the values of the Student criterion of the components of the formation mix presented in Table 5, the operational resistance of ceramics to frost is most positively influenced by the amounts of milled glass, crushed bricks and clay materials in the formation mix (the value of the Student criterion in statistical Table is 1.96). Other components of the formation mix do not have such a big influence on operational resistance to frost.

It is possible to explain a positive influence of milled glass on operational resistance to frost referring to the fact that this component of the formation mix partially

melts at a lower temperature than clay composition forming an aggressive liquid phase. The driving force of this firing process is the tension of a melt surface because negative pressure in closed pore is formed. In such an action, the pores of ceramic material are filled with melt and particles draw closer to one another.

When more liquid phase is composed and smaller particles of glass and clay are present, the diffusion process in a sample goes more intensively. The particles of material regroup because of this process, the quantity of open pores with irregular shape decreases and the pores of a closer, smaller and more regular shape are formed. Therefore, the reserve of porous volume particularly increases and mostly affects a rise in the value of operational resistance to frost (Mačiulaitis *et al.* 2004). A positive influence of the crushed bricks on the value of ceramics resistance to frost could be explained by the fact that the already fired particles of the crushed bricks have an irregular shape and stimulate the sintering process providing the product with a stronger inner carcass thus simultaneously making clay thinner. A negative influence of the peat additive (Fig. 6) to the value of operational resistance to frost could be explained by the fact that this burning out additive composed ceramic systems with open pores and capillaries allowing water migration. Water expands while freezing and destroys these products more rapidly.

## 5. Conclusions

The highest value of resistance to frost was received for samples that exhibited the highest density, compressive strength, the reserve of pore volume and the lowest water absorption value. These batches were designed based on the largest amount of milled glass. The minimum resistance to frost value was received for samples that exhibited the lowest density, compressive strength, the reserve of pore volume and the highest value of water absorption. In these batches, the largest amount of peat was used while the glass component was absent.

Regression analysis was performed and the influence of the amount of each component of the formation mix on operational resistance to frost was evaluated. It was determined that operational resistance to frost could be highly increased by the presence of the milled glass component and crushed bricks in the designed mix. Operational resistance to frost is mostly reduced by an increase in peat amount in the mixture.

**Table 5.** The values of correlation  $R$ , determination  $R^2$ , average standard deviation  $s_e$  and the Student criterion of empirical, Eq (1)

R	$R^2$	$s_e$ , in cycles	The values of Student criterion		
			The amount of clay ( $x_1$ )	The amount of glass ( $x_3$ )	The amount of crushed bricks ( $x_4$ )
0.984	0.968	7.98	4.88	18.7	6.15

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**KERAMIKOS ATSPARUMO ŠALČIUI PRIKLAUSOMYBĖ NUO FORMAVIMO MIŠINIO SUDĖTIES****R. Mačiulaitis, J. Malaiškienė**

Santrauka

Keraminių gaminių eksploatacinis atsparumas šalčiui yra svarbi šių dienų problema. Kad keraminiai mūro gaminiai ilgai išliktų kokybiški, nepraradę savo pradinių savybių, reikia tobulinti technologinį jų gamybos procesą. Šiame darbe buvo nustatyta, kaip kiekvienas formavimo mišinio komponento kiekis gali lemti keraminių bandinių eksploatacinį atsparumą šalčiui. Atliekant darbą, buvo suformuotos aštuonios keraminių bandinių partijos. Jos buvo sudarytos iš tokių formavimo mišinio komponentų, kaip molis, smėlis, maltas stiklas, skaldelė, durpės ir antracitas. Bandiniai buvo džiovunami ir degami vienodomis sąlygomis (didžiausia degimo temperatūra – 1080 °C). Eksploatacinis atsparumas šalčiui buvo nustatytas pagal tuo metu galiojusio LST 1272-92 standarto 3.5.3 p. Atlikus regresinę gautų eksperimentinių duomenų analizę, buvo sudaryta empirinė lygtis, kuria remiantis galima prognozuoti eksploatacinį atsparumą šalčiui pagal formavimo mišinio sudėtį.

**Reikšminiai žodžiai:** keramika, atsparumas šalčiui, formavimo mišinio sudėtis, savybės, statistinė analizė.

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