

VILNIUS GEDIMINAS TECHNICAL UNIVERSITY

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**NEW METHODS OF THE REGULATION OF THE
MAIN CHARACTERISTICS AND
TECHNOLOGICAL PARAMETERS OF CERAMIC
PRODUCTS**

Summary of Doctoral Dissertation
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VILNIAUS GEDIMINO TECHNIKOS UNIVERSITETAS

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**NAUJI KERAMINIŲ GAMINIŲ SVARBIAUSIŲ
CHARAKTERISTIKŲ IR TECHNOLOGINIŲ
PARAMETRŲ REGULIAVIMO METODAI**

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1. General characteristic of the dissertation

Topicality of the problem. The technological processes are controlled by computer in up-to-date manufacturing although the factories of ceramic products mostly produce specific products using the best materials, drying and burning regimes selected by scientists. However, they could also produce different sort of products for various purposes and desired characteristics having the same materials and devices but using the new methods and regularities determined by them.

Moreover, it would be extremely important to use dateless and at the same time of low conductivity ceramic products of masonry as such the products will reduce the building and exploitation expenses.

Aim and tasks of the work. The aim of the scientific research work was to analyze the works accomplished by Lithuanian and foreign scientists and to develop new methods, on which basis it would be possible to get ceramic products of desired characteristics and different purposes changing only the components of formation mixes, their quantities and the main parameters of drying and/or burning regimes and also to explore the possibilities for obtaining porous ceramics which is resistant to frost when it is being exploited and select the composition of formation mix, the best regimes of drying and burning in order to obtain ceramics which possesses such characteristics.

To achieve the aim of the work such tasks were set:

1. To select the composition of formation mix, drying and burning regimes using local easily fusible clay. To express the stages of drying and burning regimes by conditional square measures, thus estimating in a complex a quantity of heat obtained. To determine the physical-mechanical and structural parameters of ceramic samples. To form the equations of the dependence of the components of formation mix, determined physical-mechanical and structural parameters and vice versa, to show what physical-mechanical and structural parameters are influenced on the components of formation mix and their quantities.
2. To select drying regimes and to form empirical equations which would show how the stages of drying regime which were selected influence the final physical-mechanical and structural parameters of ceramics and vice versa, to show how the final physical-mechanical and structural parameters of ceramic chip influence the selection of drying regime.
3. To select drying regimes and to derive empirical equations which would show the interdependence between ceramics, produced from the local easily fusible clay, physical-mechanical and structural parameters and the stages of burning regime selected and vice versa, to show how the final physical-mechanical and structural parameters of ceramic chip influence the selection of burning regime.

4. To select the best composition of formation mix and drying and burning regimes in order to obtain porous and at the same time exploiting resistant to frost ceramics using the local ilitical easily fusible clay as the main material, which chemical composition can fluctuate within particular limits.

Scientific novelty

1. It was determined after analysis of many works of scientists from Lithuania and other foreign countries that actual and unsolved problem of these days is operative regulation of the parameters of ceramics. It was failed to find any scientific work published which presents the examples of how rapidly one must regulate the properties of ceramics in practice. Only after long search was developed new techniques which enable to regulate the main technological parameters of ceramic products operatively. Using them it is possible to select the best composition of formation mix, drying and/or burning regime according to desired to obtain final parameters of ceramic products and vice versa, it is possible to forecast expeditiously the main parameters of ceramic chip by selecting the composition of formation mix, drying and/or burning regime. The most important thing is that using derived equations it is possible to manage manufacturing of ceramic products operatively.
2. It is obtained porous and exploiting extremely resistant to frost ceramics when using local easily fusible clay. According to resistance to frost these products can be used under harsh conditions.

Methodology of research. The materials used for research include: clay, sand, chip, anthracite, peat, sawdust, ground glass. The physical-mechanical and structural parameters of formed, dried, and burned ceramic samples were determined according to LST EN 771-1+A1, LST 1985:2006 and other known methodologies.

It was performed the phases analysis of powder of burned samples according to method of X-ray examination. Mainly used devise is difraktometer DRON-2, it was used anode of cobalt with the length of wave $\lambda = 0.1792$ nm. Inner composition of some samples was explored using microscope Olympus BX 50.

The experimental data obtained was grouped and prepared with “Microsoft Excel” for research of statistical and regressive analysis and with “Statistica” programs for statistical and regressive analysis.

Practical value. It was proved the possibilities to regulate quality of the articles of potential ceramics which were formed using local easily fusible ilitical clay as the main material and produced according to one technological line, when it is used reciprocal systematic correlation between the components of the composition of formation mix, the stages of drying and burning regimes expressed in conditional square measures (evaluating the amount of warm

obtained) and the characteristics of the final products. It is possible to control technological process by computer using newly developed methods.

Using local materials and waste (chip, ground glass, sawdust) and selecting the best compositions of formation mix, drying and burning regimes, it is obtained ceramics which is porous and exploiting extremely resistant to frost. Such the characteristics of ceramics would enable to produce mural articles (bats, bricks, and blocks), especially designed for bricking facades of buildings in near-shore zone. Moreover, the coefficient of heat conduction of such mural ceramic articles would be quite low.

Defended propositions

1. Scientists explore in what ways the composition of formation mix as well as the regimes of drying and burning influence the final characteristics of ceramic chip. However, the reciprocal interdependence, which shows that desired values of physical-mechanical, structural parameters of ceramic chip greatly influence the selection of the components of formation mix, their quantities, the intensity and duration of drying and burning regimes, is extremely important.
2. As the statistical analysis are performed and the equations of the reciprocal interdependence are developed it is possible to forecast what properties of ceramics we will obtain when select the components of formation mix, their quantity, the values of stages of drying and burning regimes and vice versa, it is possible to determine what the components of formation mix are needed, their quantities, the values of stages of drying and burning regimes are as we put in the desired values of physical-mechanical and structural parameters of ceramics in order to obtain the best parameters of a ceramic body. It is shown that using developed empirical equations it is possible to regulate the properties of ceramics and technological parameters effectively.
3. Using local materials (clay, sand) and waste (burned crumbled articles of ceramics of low quality, sawdust, ground glass) it is possible to obtain effective (porous) and exploiting extremely resistant to frost ceramics suitable for bricking facades in near-shore zone.

The scope of the scientific work. The work include: a general characteristic of the work, publication list, 7 chapters, general conclusions, literature list. The total scope of the dissertation is 130 pages, 28 tables and 29 figures.

Structure of the work. In the first (introductory) chapter it is analyzed the topicality of the problem, the aim and tasks of the work are stated, the novelty of the scientific work is described, the reports and publications of the author and the structure of the dissertation are presented.

The second chapter is designed for literature analysis. There are presented the dependence of the properties of structural ceramics and technological factors, which was determined by other authors, the processes of agglomeration of ceramics, resistance to frost and porosity. At the end of the chapter the summary of the literature analysis is presented, and the tasks of the dissertation are characterized.

In the third chapter the methodology, the properties of the main material (clay) and the preparation of the samples for the experiments are described.

In the fourth, fifth and sixth chapters the new methods for the regulation of the characteristics of ceramic articles and technological factors (the composition of formation mix, the regimes of drying and burning) according to empirical equations of interdependence are shown.

In the seventh chapter it is shown the possibility to produce exploiting resistant to frost porous ceramics from the local materials and waste.

2. The method to regulate the properties of structural ceramics by the composition of formation mix

The formed samples, which compositions of formation mix are presented in Table 1, were dried in a laboratory for 72 hours at (20 ± 2) °C temperature, later they were dried in the electric stove for 24 hours at (105 ± 5) °C temperature. Dried samples were burned out under such a burning regime: 36 hours, keeping for 3 hours at the maximum temperature. Average values of physical-mechanical parameters of burned ceramic samples are presented in Table 2.

Table 1. The compositions of formation mixes of ceramic samples

Formation mix	Quantity of clay, y_1 , %	Quantity of sand, y_2 , %	Quantity of chip, y_3 , %	Quantity of anthracite, y_4 , %	Quantity of peat, y_5 , %	Quantity of glass, y_6 , %
1	74.5	18.0	6.0	1.5	0.0	0.0
2	80.0	15.0	0.0	0.0	5.0	0.0
3	70.5	12.0	6.0	1.5	0.0	10.0
4	69.5	18.0	6.0	1.5	0.0	5.0
5	59.5	18.0	6.0	1.5	0.0	15.0
6	54.0	30.0	5.0	1.0	0.0	10.0
7	82.5	10.0	5.0	0.0	2.5	0.0
8	82.5	10.0	5.0	2.5	0.0	0.0

From the data presented in Table 2 we see that the final properties of ceramic chip, which influence the quality of ceramic product, greatly depend on the variation of the components of formation mix and their quantities when all the other technological conditions are stable.

In order to determine the influence of the quantity of formation mix on the final physical-mechanical parameters of ceramics more precisely statistical and regression analysis of experimental data were performed.

Table 2. Average values of selected physical-mechanical parameters

Formation mix	Density, x_1 , kg/m^3	Compressive strength, x_2 , MPa	General contraction, x_3 , %
1	1752	24.34	10.43
2	1624	19.35	11.88
3	2022	39.11	12.77
4	1920	29.74	11.24
5	2246	40.99	13.30
6	2170	28.94	10.24
7	1626	27.86	12.33
8	1664	29.20	10.62

It is determined that distribution character of experimental data is normal and Fisher's criterion of derived empirical equations is bigger than the value in the standard table. The values of determination coefficients $R^2 = 0.576-0.992$ indicate that selected linear mathematical model reveals distribution of experimental data respectably enough. Correlation coefficients $R = 0.759-0.996$ indicate strong linear correlation between the components of formation mix and analyzed properties of ceramic chip. The importance of analyzed parameters was determined by Student criteria. The empirical equations of interdependence are derived: Table 3 and 4, according to which it is possible to forecast properties of ceramic body selecting the composition of formation mix, and vice versa, it is shown, that according to desired parameters of ceramic chip one must select the composition of formation mix.

Table 3. The empirical equations of interdependence between the components of formation mix and physical-mechanical parameters

Equation	Components	Empirical equations
1	Clay, y_1	$y_1 = 146.3 - 0.05x_1 + 0.44x_2 + 0.51x_3$
2	Sand, y_2	$y_2 = -9.46 + 0.03x_1 - 0.64x_2 - 1.07x_3$
3	Chip, y_3	$y_3 = 9.31 - 0.001x_1 + 0.26x_2 - 0.95x_3$
4	Anthracite, y_4	$y_4 = 5.60 - 0.001x_1 + 0.11x_2 - 0.53x_3$
5	Peat, y_5	$y_5 = -3.94 + 0.001x_1 - 0.22x_2 + 1.15x_3$
6	Glass, y_6	$y_6 = -46.7 + 0.02x_1 + 0.05x_2 + 0.89x_3$

Table 4. The empirical equations of the reciprocal dependence (of physical-mechanical parameters and the components of formation mix)

Equation	Property	Empirical equation
7	Density, x_1	$x_1 = 2552 - 9.482y_1 - 14.64y_5 + 23.75y_6$
8	Compressive strength, x_2	$x_2 = -33.74 + 0.706y_1 + 1.732y_3 + 1.948y_6$
9	General contraction, x_3	$x_3 = 30.34 - 0.207y_1 - 0.323y_2 + 0.416y_5$

It is the example of the usage of the derived equations. Let's say we want to obtain ceramic chip with particular physical-mechanical parameters, e.g., density of 2000 kg/m³, compressive strength of 30 MPa, general contraction of 12 %. Then we put these values into (1)–(6) equations and get such the composition of mix: 66 % of clay, 19% of sand, 4 % of chip, 1 % of anthracite, 5 % of peat, 5 % of glass.

In order to ascertain the reliability of this composition of formation mix, we put the values of these components into (7)–(9) equations and get that the density of the chip is 1972 kg/m³, compressive strength is 29.5 MPa, general contraction is 12.6 %. Consequently the values obtained differ slightly from the set ones and using the set composition it is possible to expect the above mentioned physical-mechanical parameters of products.

3. The method to regulate properties of structural ceramics by drying regimes

The values of drying regimes were expressed in conditional square measures, the largest area equating to 100. In such a way the quantity of warm of samples was evaluated. The drying regime was divided into two stages: drying in a laboratory and in the electric stove keeping at a maximum temperature. Calculated values of drying regimes are presented in Table 5.

The values of compressive strength varied to the greatest degree from approximately 9 MPa (Batch 1) to 22 MPa (Batch 7), when changing drying regime but all the other technological conditions setting stable. The degree of structural inhomogeneity was changing approximately from 0,2 unit (Batch 7) to 1 unit (Batch 1), maximum rate of capillary rise was fluctuating approximately between 17 mm (Batch 7) and 50 mm (Batch 1) depending on the watering front, and the rate of the flow of capillary mass was fluctuating between 0.25 g/cm² (Batch 7) and 1 g/cm² (Batch 1).

Table 5. Calculated values of drying regimes

Batch	Stage of drying in a laboratory, y_1 , unit	Temperature of drying in a laboratory, °C	Stage of drying in the electrical stove, y_2 , unit	Temperature of drying in the electrical stove, °C
1	5.44	20	–	–
2	8.16	20	35.37	65
3	20.95	22	17.69	65
4	41.9	22	28.57	105
5	8.16	20	11.73	105
6	17.14	18	7.14	105
7	57.14	20	42.86	105
8	17.14	18	40.82	150

The results which we obtained show that drying regime influences the final properties of ceramic product very heavily, therefore it is worth to know how the different value of stage of drying regime influences physical-mechanical and structural parameters of ceramic chip. In order to determine the interdependence between stages of drying regime and analyzed parameters and also to regulate properties of ceramic chip the empirical equations of interdependence (the same as in chapter 2) were derived.

By checking the adequacy of experimental results for calculated values according to derived empirical equations it is determined that all the other technological conditions do not change, it is possible to select drying regime according to desired physical-mechanical and structural parameters of a product, and vice versa, it is possible to predict the final physical-mechanical and structural parameters of a ceramic product by selecting the drying regime.

4. The method to regulate properties of structural ceramics by burning regimes

Burning regimes, as well as drying regimes, were expressed in conditional square measures (the example of calculations is presented in Figure 1), the largest area equating to 100 in order to evaluate the quantity of warm of samples which we obtained. The burning regime was divided into three stages: the temperature rise, keeping at the maximum temperature, and the temperature fall to 400 °C as the inner composition of ceramic sample is fully formed to this temperature and the important processes of change of material structure do not proceed. The values of these burning stages are presented in Table 6 according to the batch.

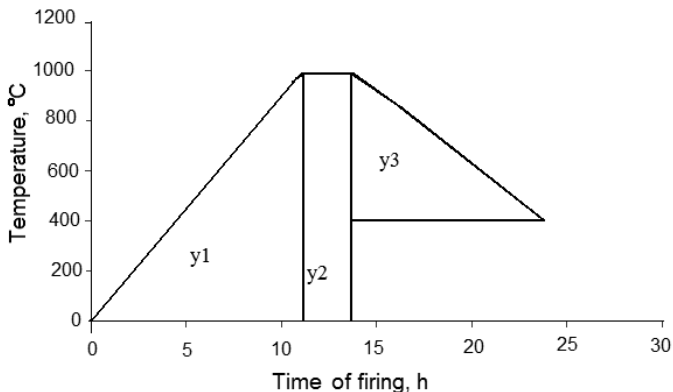


Figure 1. Evaluation of burning area

Table 6. Burning regimes

Batch	Stage of the temperature rise, y_1 , unit	Stage of keeping at the maximum temperature, y_2 , unit	Stage of the temperature fall, y_3 , unit	Maximum burning temperature, °C
1	14.12	7.70	4.28	900
2	38.51	23.11	10.70	900
3	14.91	8.13	4.71	950
4	40.66	24.39	10.70	950
5	47.08	25.68	15.41	1000
6	31.38	17.12	10.27	1000
7	15.91	5.99	6.49	1050
8	44.94	26.96	11.77	1050
9	16.87	9.20	8.19	1075
10	33.74	18.40	11.79	1075
11	15.55	9.33	6.89	1090
12	34.21	18.66	12.80	1090
13	34.21	31.10	25.59	1090
14	34.52	18.83	12.98	1100
15	10.36	9.42	6.99	1100
16	51.78	28.25	19.97	1100

The structural parameters, which average values are presented in Table 7, were determined by burning samples according to set regimes.

Table 7. The average values of structural parameters

Batch	Reserve of pore volume, x_4 , %	Degree of structural inhomogeneity, x_5 , unit	Maximum rate of capillary wetting front, x_6 , mm	Capillary rate of mass flow, x_7 , g/cm ²	Water saturation, x_8 , %
1	24.29	0.584	34.4	0.561	27.99
2	25.48	0.541	35.2	0.648	28.06
3	23.80	0.386	36.8	0.811	26.26
4	25.81	0.369	37.0	0.809	25.60
5	23.58	0.358	37.2	0.803	28.56
6	21.36	0.268	42.8	0.931	26.62
7	29.38	0.271	38.4	0.768	25.60
8	31.48	0.249	39.6	0.842	17.42
9	37.02	0.183	43.6	0.770	15.51
10	31.52	0.440	49.4	0.882	13.35
11	42.90	0.189	54.6	1.045	12.77
12	42.22	0.363	51.2	0.725	13.09
13	38.73	0.106	61.2	1.095	14.15
14	44.21	0.212	62.4	1.137	12.05
15	45.12	0.205	63.0	1.154	11.87
16	48.21	0.065	61.8	0.934	11.42

It was performed more thorough analysis of the dependence of each separate value of the stage of burning regime on structural parameters of ceramics because of a great change in quality of ceramic article.

Further on the regression analysis of data was performed and the empirical equations derived. The intensity of interdependence was estimated according to the numerical values of correlation coefficient. Suitability of a model was evaluated by calculations of the determination coefficient. According to the numerical values and signs of regression equations one can judge in which way and how much each of the investigated factors and their interdependence affect the structural properties of ceramic samples. Applying the model of piecewise linear regression with breakpoint the empirical equations of interdependence of structural parameters and firing cycles are obtained (10)–(14). Equations (15)–(17) show the inverse interdependence among separate stages of firing cycles and values of structural parameters.

$$x_4 = (27.46 - 4.909y_1 + 3.153y_2 + 9.421y_3) (x_4 < 34.78) + (41.56 - 0.249y_1 + 2.997y_2 - 3.398y_3) (x_4 \geq 34.78), \quad (10)$$

$$x_5 = (0.100 + 0.003y_1 + 0.013y_2 + 0.011y_3) (x_5 < 0.251) + (0.476 + 0.011y_1 + 0.015y_2 - 0.063y_3) (x_5 \geq 0.251), \quad (11)$$

$$x_6 = (31.20 - 1.008y_1 + 1.256y_2 + 1.358y_3) (x_6 < 48.98) + (54.51 - 0.400y_1 + 1.720y_2 - 1.237y_3) (x_6 \geq 48.98), \quad (12)$$

$$x_7 = (0.623 - 0.033y_1 + 0.046y_2 + 0.036y_3) (x_7 < 0.902) + (1.161 - 0.007y_1 + 0.002y_2 + 0.005y_3) (x_7 \geq 0.902), \quad (13)$$

$$x_8 = (15.11 - 0.213y_1 + 1.152y_2 - 1.218y_3) (x_8 < 18.18) + (27.70 + 0.270y_1 + 0.144y_2 - 1.021y_3) (x_8 \geq 18.18). \quad (14)$$

Hereinafter we have analyzed the empirical equations of inverse interdependence (15)–(17), that enable more accurate analysis of relations between the firing cycles and structural parameters.

$$y_1 = (11.52 + 0.161x_4 - 2.270x_5 - 0.141x_6 + 0.689x_7 + 0.188x_8) (y_1 < 27.28) + (27.60 - 0.549x_4 + 16.78x_5 - 0.332x_6 - 1.690x_7 + 0.559x_8) (y_1 \geq 27.28), \quad (15)$$

$$y_2 = (-14.30 + 0.371x_4 - 6.713x_5 + 0.221x_6 + 1.644x_7 - 0.172x_8) (y_2 < 15.99) + (36.21 - 0.622x_4 - 22.53x_5 + 0.369x_6 - 7.380x_7 + 0.101x_8) (y_2 \geq 15.99), \quad (16)$$

$$y_3 = (3.991 - 0.233x_4 - 2.901x_5 + 0.229x_6 + 5.587x_7 - 0.165x_8) (y_3 < 11.26) + (33.90 - 0.768x_4 - 10.82x_5 + 0.492x_6 - 8.291x_7 - 0.303x_8) (y_3 \geq 11.26). \quad (17)$$

Equations (10)–(17) and information in Table 8 show the strong interdependence among separate stages of firing cycles and values of structural parameters, applying the model of piecewise linear regression with breakpoint. Determination coefficients are higher than 0.7, thus this mathematical model is hand-picked rightly.

Table 8. Correlation R and determination R^2 coefficients and an average standard deflection s_e of equations (10)–(17)

No. of equation	R	R^2	s_e
5.10	0.961	0.924	2.38 %
5.11	0.881	0.777	0.08 unit
5.12	0.892	0.795	4.63 mm
5.13	0.848	0.719	0.10 g/cm ²
5.14	0.922	0.849	2.53 %
5.15	0.939	0.883	4.11 unit
5.16	0.906	0.822	3.17 unit
5.17	0.888	0.789	2.54 unit

It is the example of application of the equations which were developed to practice. We offer one example of how to apply these (10)–(17) empirical equations to practice. The separate stages of firing cycles were being varied, but other technological specifications were being unvaried (one batch of samples was tested practically).

Example. Let's say ceramic products with such structural parameters should be produced: reserve of pore volume 45 %, degree of structural inhomogeneity 0.1 units, maximum rate of capillary wetting front 60 mm, capillary rate of mass flow 1 g/cm², water saturation 15 %. In this case the values should be put into Esq. (15)–(17) and the following parameters of firing cycles are obtained: the area of temperature increase – 13.5 unit; the area of keeping at the maximum temperature – 13.9 unit and the area of temperature decrease – 10.2 unit.

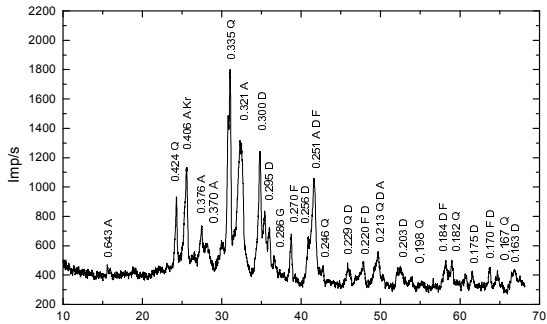
Then these values are put in Esq. (10)–(14) and the parameters are calculated: the expected reserve of pore volume – 45 %, degree of structural inhomogeneity – 0.1 unit, maximum rate of capillary wetting front – 60 mm, capillary rate of mass flow – 1 g/cm², water saturation – 16 %. So the values which we have calculated differ very slightly from those given in the task.

5. The technique of producing exploiting resistant to frost porous ceramics and its properties

Formed prefabs were dried and burned under the best regimes of time and temperature when selecting such a formation mix (clay 59.2–72.8 %, sand 7.2–10.8 %, chip 3.2–4.8 %, sawdust 10.4–15.6 % and ground glass 6.4–9.6 %). In this way it was obtained the porous and exploiting extremely resistant to frost ceramics which total open porosity reaches 40 %, and exploiting resistance to frost, which is estimated according to LST 1985:2006, is higher than 300 cycles. The coefficient of warm conduction, which is calculated according to LST EN 1745:2002, fluctuates between 0.37 W/mK and 0.42 W/mK.

The sawdust and ground glass are the main components of the formation mix which create the desirable effect. More defects arise and the resistance to frost begins to decrease when putting in the more than 16 % of sawdust, and it is not obtained the product of desired porosity by putting in less than 10 % of sawdust. It is impossible to obtain exploiting resistant to frost ceramics by reducing the quantity of ground glass to 6 % and more in the formation mix and by increasing the quantity of glass to 10 % and more; the porous ceramic chip is not produced. Moreover, when we exceed the quotas of glass in the formation mix, the mass of glass begins to diffuse into the surface and the strength of the product decreases.

When X-ray examination was performed it was determined these minerals: silica *Q*, hematite *F*, anortite *A*, diopside *D* (Figure 2).



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Figure 2. X-ray of the sample of the resistant to frost porous ceramics obtained

General conclusions

1. There were analyzed scientific works in which it was determined the dependence of various kinds of clay and components of formation mix, maximum drying and burning temperature on the final properties of ceramics. However, there is presented the influence of drying or burning temperature and duration of separate component of formation mix on particular properties of ceramics which restrict the possibilities to use empirical equations in practice in these works. We have also failed to find the techniques of producing exploiting resistant to frost and at the same time effective (porous) ceramics in the literature.
2. There were developed the new and original methods, which enable to regulate the final properties of ceramic articles by three factors: the composition of formation mix, the regimes of drying and burning, evaluating the temperature and the duration of keeping at it in stages.
3. It is determined that the final physical-mechanical and structural properties of ceramics, which evaluate the quality of ceramic articles, are highly influenced by the components of formation mix and their quantities, the selection of stages of drying and burning:
 - 3.1 It is shown in the examples that changing the components of formation mix and their quantities while other technological factors are stable it is possible to obtain ceramics with different properties: porous (impregnation of water determined after 72 hours reaches 12.7 %, and density is 1610 kg/m³) or parched (impregnation of water determined after 72 hours is not higher than 3.0 %, and density reaches 2280 kg/m³).

- 3.2 It is demonstrated that careful selection of values of stages of drying regime as all the other technological conditions are stable it is possible to increase or decrease the values of physical-mechanical and structural parameters. For example, the values of compressive strength were fluctuating between 49 MPa and 18.42 MPa, the degree of structural inhomogeneity of composition were fluctuating between 0.262 unit and 1.017 unit, maximum rate of capillary increase according to watering front were fluctuating between 17.7 mm and 49.3 mm.
- 3.3 It is determined that only changing the values of stages of burning regime when the composition of the formation mix and drying regime are stable, it is also obtained the ceramics of extremely different properties. The impregnation of water of such a ceramics determined after 72 hours fluctuates between 11 % (burning under the intensive regime at 1100 °C temperature) and 30 % (burning under the shortest regime at 900 °C temperature), density varies from 1290 kg/m³ to 1630 kg/m³, the reserve of porous space is from 21.4 % to 48.2 %.
4. It is determined, after considering the examples if the experimental results match the values calculated according to empirical equations when all the other technological conditions do not change, that it is possible to select the composition of formation mix or drying or burning regimes by desired physical-mechanical and structural parameters of a product, and vice versa, it is possible to forecast the final physical-mechanical and structural parameters of a ceramic product. On this account, there are proved the potential possibilities of preparing the new methods to control and regulate technological processes of the production of ceramic articles according to suggested tendencies of mutual cohesion, in order to obtain products of the desired properties.
5. It is obtained exploiting resistant to frost (exploiting resistance to frost is higher than 300 cycles) porous ceramics (total open porosity reaches 40 %, calculated coefficient of warm conduction fluctuates between 0.37 and 0.42 W/mK) using local stock and waste (the composition of the formation mix is as follows: clay 59.2–72.8 %, sand 7.2–10.8 %, chip 3.2–4.8 %, sawdust 10.4–15.6 % and ground glass 6.4–9.6 %).

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NAUJI KERAMINIŲ GAMINIŲ SVARBIAUSIŲ CHARAKTERISTIKŲ IR TECHNOLOGINIŲ PARAMETRŲ REGULIAVIMO METODAI

Mokslo problemos aktualumas. Šiuolaikinėje gamyboje technologiniai procesai valdomi kompiuterizuotai, tačiau keraminių dirbinių gamybos įmonės dažniausiai gamina vienos konkrečios rūšies produktus, naudodamos mokslininkų parinktas geriausias žaliavas, džiovinimo ir degimo režimus. Tačiau, turėdamos tas pačias žaliavas ir tuos pačius įrenginius, bet naudodamos naujus metodus, galėtų gaminti ir keliolikos rūšių bei įvairios paskirties, norimų charakteristikų produkciją.

Be to, šiuolaikinėje statyboje labai svarbu būtų naudoti ilgaamžius ir kartu mažai laidžius šilumai keraminius mūro gaminius, nes tokie dirbiniai sumažintų pastato statybos ir eksploatacijos išlaidas.

Darbo tikslai ir uždaviniai. Darbo tikslas buvo išnagrinėti atliktus Lietuvos ir užsienio mokslininkų darbus ir sukurti naujus metodus, kurių pagrindu būtų galima, turint tas pačias žaliavas, tik keičiant formavimo mišinių komponentus ir jų kiekius ir svarbiausių džiovinimo, degimo režimų etapų parametrus, gauti norimų savybių ir skirtingos paskirties keraminius dirbinius. Taip pat išnagrinėti eksploatuojant atsparios šalčiui ir poringos keramikos gavimo galimybes bei parinkti formavimo masės sudėtį, geriausius džiovinimo ir degimo režimus tokių savybių keramikai gauti.

Darbo tikslui pasiekti, buvo suformuluoti šie uždaviniai:

1. Naudojant vietinį ilitinį lengvai lydų molį, parinkti keramikos formavimo masės sudėtis, džiovinimo ir degimo režimus. Džiovinimo ir degimo režimų etapus išreikšti per sąlyginius ploto vienetus, taip kompleksiskai įvertinant gautą bandinių šilumos kiekį. Nustatyti keramikos bandinių fizikines-mechanines ir struktūrines savybes. Sudaryti formavimo masės komponentų ir nustatytų fizikinių-mechaninių ir struktūrinių rodiklių priklausomybės empirines lygtis. Ir atvirkščiai, parodyti, kokie fizikiniai-mechaniniai ir struktūriniai rodikliai yra įtakojami formavimo masės komponentų ir jų kiekių.
2. Parinkti džiovinimo režimus ir sudaryti empirines lygtis, kurios parodytų, kaip pasirinkti džiovinimo režimo etapai daro įtaką galutiniams keramikos fizikiniams-mechaniniams bei struktūriniams rodikliams. Ir atvirkščiai, parodyti, kaip galutiniai keraminės šukės fizikiniai-mechaniniai ir struktūriniai rodikliai lemia džiovinimo režimo parinkimą.
3. Parinkti degimo režimus ir sudaryti empirines lygtis, kurios parodytų priklausomybę tarp keramikos, pagamintos iš vietinio ilitinio lengvai lydaus molio, fizikinių-mechaninių ir struktūrinių rodiklių ir pasirinktų degimo režimo etapų. Ir atvirkščiai, parodyti, kaip galutiniai keraminės šukės fizikiniai-mechaniniai ir struktūriniai rodikliai lemia degimo režimo parinkimą.
4. Kaip pagrindinę žaliavą naudojant vietinį ilitinį lengvai lydų molį, kurio cheminė sudėtis gali kisti tam tikrose ribose, parinkti geriausią formavimo masės sudėtį, džiovinimo ir degimo režimus poringai bei tuo pačiu metu eksploatuojant atspariai šalčiui keramikai gauti.

Mokslinis naujumas

1. Išnagrinėjus daugelį Lietuvos ir užsienio mokslininkų darbų, buvo nustatyta, kad aktuali ir neišspręsta šių dienų problema yra operatyvus keramikos savybių reguliavimas. Nepavyko rasti publikuotų mokslinių darbų, kuriuose būtų pateikti pavyzdžiai, kaip sparčiai reguliuoti keramikos savybes praktikoje. Tik po ilgų ieškojimų buvo sukurti nauji būdai, įgalinantys

- operatyviai reguliuoti keramikos dirbinių svarbiausius technologinius parametrus. Naudojant juos galima parinkti geriausią formavimo mišinio sudėtį, džiovinimo ir degimo režimus pagal norimus gauti galutinius keraminių gaminių rodiklius. Ir atvirkščiai, parinkus formavimo masės sudėtį, džiovinimo ir degimo režimus, galima sparčiai prognozuoti pagrindines keraminės šukės charakteristikas. Svarbiausia, kad naudojant gautas lygtis, galima kompiuterizuotai operatyviai valdyti keraminės produkcijos gamybą.
2. Naudojant vietinį lengvai lydų ilitinį molį, gauta poringa ir eksploatuojant labai atspari šalčiui keramika. Pagal atsparumą šalčiui šie gaminiai gali būti naudojami agresyviausiomis eksploatacijos sąlygomis.

Tyrimų metodika. Tyrimams buvo naudojamos tokios žaliavos: molis, smėlis, skaldelė, antracitas, durpės, pjuvenos ir maltas stiklas. Suformuotų, išdžiovintų ir išdegtų keraminių bandinių fizikiniai-mechaniniai ir struktūriniai rodikliai buvo nustatyti pagal LST EN 771-1+A1, LST 1985:2006 bei kitas žinomas metodikas.

Rentgenofazinių tyrimų metodu buvo atlikta išdegtų bandinių miltelių fazinė analizė. Pagrindinis naudotas prietaisas – difraktometras DRON–2, kai naudotas kobalto anodas, o bangos ilgis $\lambda = 0,1792$ nm. Kai kurių bandinių vidinė struktūra buvo iširta naudojant mikroskopą Olympus BX 50.

Gautų eksperimentinių duomenų grupavimas, paruošimas statistinės ir regresinės analizės tyrimui buvo atliekamas su „Microsoft Excel“, o statistinė ir regresinė analizės – su „Statistica“ programomis.

Praktinė vertė. Įrodytos potencialios keramikos dirbinių, suformuotų, kaip pagrindinę žaliavą naudojant vietinį lengvai lydų ilitinį molį, gaminamos viena technologine linija, kokybės reguliavimo galimybės, kai taikoma abipusė sisteminė sąryšio tarp formavimo mišinio sudėties komponentų, džiovinimo ir degimo režimo etapų, išreikštų per sąlyginius ploto vienetus (įvertinant gautą bandinių šilumos kiekį) ir gatavos produkcijos savybių priklausomybę. Pagal naujai sukurtus metodus galima kompiuterizuotai valdyti vykdomą technologinį procesą.

Iš vietinių žaliavų ir atliekų (skaldelės, stiklo duženų, pjuvenų), parinkus geriausius formavimo masės sudėtis, džiovinimo ir degimo režimus, gauta poringa eksploatuojant labai atspari šalčiui keramika. Iš tokių savybių keraminės šukės būtų galima gaminti sieninius dirbinius (pusplytes, plytas, blokus), ypač skirtus pajūrio zonos pastatų fasadams mūryti. Be to, tokių sieninių keraminių dirbinių šilumos laidumo koeficientas būtų gana mažas (< 0.42 W/mK).

Ginamieji teiginiai

1. Mokslininkai tiria, kaip formavimo masės sudėtis, taip pat džiovinimo ir degimo režimai lemia galutines keraminės šukės charakteristikas, tačiau labai svarbi yra ir atvirkštinė priklausomybė parodanti, kad nuo norimų gauti keraminės šukės fizikinių-mechaninių ir struktūrinių rodiklių reikšmių labai

- priklauso formavimo masės komponentų ir jų kiekių bei džiovinimo ir degimo režimų intensyvumo ir trukmės parinkimas.
2. Atlikus statistinę analizę ir sudarius abipusės priklausomybės empirines lygtis galima, pasirinkus formavimo masės komponentus ir jų kiekius, džiovinimo ir degimo režimų etapų dydžius, prognozuoti, kokių savybių keramiką gausime. Ir atvirksčiai, imant norimas gauti keramikos fizikinių-mechaninių ir struktūrinių rodiklių reikšmes, galima nustatyti, kokie yra reikalingi formavimo masės komponentai ir jų kiekiai, džiovinimo ir degimo režimų etapų parametrai, norint gauti geriausius keraminio kūno rodiklius. Parodyta, kad naudojant sudarytas empirines lygtis, galima sparčiai ir efektyviai reguliuoti keramikos savybes ir technologinius parametrus.
 3. Naudojant vietines žaliavas (molių, smėlių) ir atliekas (sutrupintus išdegtus nekokybiškus keramikos dirbinius, pjuvenas, maltą stiklą), galima gauti efektyvią (poringą) ir eksploatuojant labai atsparią šalčiui keramiką, tinkamą ir pajūrio zonos fasadams mūryti.

Darbo apimtis. Darbą sudaro bendra darbo charakteristika, publikacijų sąrašas, 7 skyriai, bendrosios išvados, literatūros sąrašas. Bendra disertacijos apimtis – 130 puslapių, 28 lentelės ir 29 paveikslai.

Darbo struktūra. Pirmajame (įvadiniame) skyriuje nagrinėjamas problemos aktualumas, formuluojamas darbo tikslas bei uždaviniai, aprašomas mokslinis darbo naujumas, pristatomi autoriaus pranešimai ir publikacijos, disertacijos struktūra.

Antrasis skyrius skirtas literatūros analizei. Jame išnagrinėtos kitų autorių nustatytos statybinės keramikos savybių ir technologinių faktorių priklausomybės, keramikos sukepimo procesai, atsparumas šalčiui ir poringumas. Skyriaus pabaigoje pateiktas literatūrinės analizės apibendrinimas ir konkretizuojami disertacijos uždaviniai.

Trečiajame skyriuje aprašyta tyrimų metodika, pagrindinės žaliavos (molio) savybės ir bandinių paruošimas eksperimentams. Ketvirtajame, penktajame ir šeštajame skyriuose parodyti nauji keramikos dirbinių charakteristikų ir technologinių faktorių (formavimo masės sudėties, džiovinimo ir degimo režimų) reguliavimo metodai pagal abipusės priklausomybės empirines lygtis. Septintajame skyriuje nustatyta, kaip iš vietinių žaliavų ir atliekų, galima gaminti eksploatuojant atsparią šalčiui poringą keramiką.

Bendrosios išvados

1. Išnagrinėti moksliniai darbai, kuriuose buvo nustatyta įvairių molių ir formavimo mišinio priedų, maksimalios džiovinimo ir degimo temperatūros įtaka galutinėms keramikos savybėms. Tačiau, šiuose darbuose pateikta kiekvieno atskiro formavimo masės komponento, džiovinimo arba degimo temperatūros ir trukmės įtaka konkrečioms keramikos charakteristikoms, kuri riboja empirinių lygčių praktinio panaudojimo galimybes. Taip pat

- literatūroje nepavyko rasti ir eksploatuojant atsparios šalčiui ir kartu efektyvios (poringos) keramikos gavimo būdų.
2. Sukurti nauji ir originalūs metodai, įgalinantys reguliuoti galutines gaminamų keraminių dirbinių charakteristikas pagal tris technologinius parametrus: formavimo masės sudėtį, džiovinimo ir degimo režimus, etapais įvertinant temperatūros ir išlaikymo joje trukmę.
 3. Nustatyta, kad formavimo masės komponentai ir jų kiekiai, džiovinimo ir degimo režimo etapų dydžiai daro įtaką galutinėms keramikos fizikinėms-mechaninėms ir struktūrinėms charakteristikoms, rodančioms keraminių dirbinių kokybę:
 - 3.1. Pavyzdžiais parodyta, kad keičiant tik formavimo masės komponentus ir jų kiekius, o kitus technologinius faktorius paliekant pastovius, galima gauti skirtingų savybių keramiką: poringąją (vandens įmirksis, nustatytas po 72 valandų, siekia 12,7 %, o tankis – 1610 kg/m³) arba sukepusiąją (vandens įmirksis, nustatytas po 72 h, nedidesnis kaip 3,0 %, o tankis siekia 2280 kg/m³).
 - 3.2. Įrodyta, kad tinkamai parinkus džiovinimo režimo etapų dydžius, kai visos kitos technologinės sąlygos nekinta, galima padidinti arba sumažinti nagrinėjamų fizikinių-mechaninių ir struktūrinių rodiklių reikšmes. Pavyzdžiui, gniuždomojo stiprio reikšmės kito nuo 8,80 MPa iki 21,67 MPa, struktūros nevienalytiškumo laipsnis kito nuo 0,262 vnt iki 1,017 vnt, maksimalus kapiliarinio pakilimo pagal drėkinimo frontą greitis kito nuo 17,7 mm iki 49,3 mm.
 - 3.3. Nustatyta, kad tik keičiant degimo režimo etapų dydžius, kai formavimo mišinio sudėtis ir džiovinimo režimas – pastovūs, taip pat gaunama labai skirtingų savybių keramika. Tokios keramikos vandens įmirksis, nustatytas po 72 h, gautas nuo 11 % (degant intensyviausiu režimu esant 1100 °C temperatūrai) iki 30 % (degant trumpiausiu režimu esant 900 °C temperatūrai), tankis – nuo 1290 kg/m³ iki 1630 kg/m³, poringos erdvės rezervas – nuo 21,4 % iki 48,2 %.
 4. Pavyzdžiais patikrinus eksperimentinių rezultatų atitikimą pagal empirines lygtis apskaičiuotoms reikšmėms, nustatyta, kad kai visos kitos technologinės sąlygos nekinta, pagal norimus gauti gaminio fizikinius-mechaninius ir struktūrinius rodiklius, galima parinkti formavimo masės sudėtį arba džiovinimo, ar degimo režimus. Ir atvirkščiai, parinkus formavimo masės sudėtį arba džiovinimo, ar degimo režimus, galima prognozuoti galutinius keraminio gaminio fizikinius-mechaninius ir struktūrinius rodiklius. Tokiu pagrindu įrodytos galimybės parengti naujus keraminių dirbinių gamybos technologinių procesų kontrolės ir reguliavimo metodus pagal siūlomas abipusio sąryšio tendencijas, siekiant gauti geidžiamų savybių produkciją.
 5. Naudojant vietines žaliavas ir atliekas (formavimo mišinio sudėtis yra tokia – vietinis molis 59,2–72,8 %, smėlis 7,2–10,8 %, skaldelė 3,2–4,8 %, pjuvenos

10,4–15,6 % ir maltas stiklas 6,4–9,6 %), gauta eksploatuojant atspari šalčiui (eksploatacinis atsparumas šalčiui yra didesnis kaip 300 ciklų) poringa keramika (bendrasis atviras poringumas siekia 40 %, apskaičiuotas šilumos laidumo koeficientas nuo 0,37 W/mK iki 0,42 W/mK).

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