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THE REGULATION OF PHYSICAL AND MECHANICAL PARAMETERS OF CERAMIC BRICKS DEPENDING ON THE DRYING REGIME

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Abstract. In the scientific studies the influence of burning regime or composition of formation mix on the final properties of the ceramic bricks is analysed most often. However, drying regime is also of paramount importance in the process of the high quality ceramic production. The formed ceramic samples were dried according to 8 different drying regimes while burning regime was not varied during the investigation. The dried samples were burnt for 24 hours keeping the maximum temperature 1050 ^{o}C for 3 hours. Later on these parameters were determined experimentally: density, general shrinkage, compressive strength and rate of ultrasound spread. As the statistical and regression analyses of data were performed, the empirical equations, showing how the selected stages of drying regime influence the physical and mechanical parameters of ceramics, and vice versa, how the selection of the dimensions of the stages of drying regime depends on the desired values of the ceramics properties, were derived.

Keywords: ceramics, physical and mechanical properties, drying regime, statistical analysis.

1. Introduction

It is known that the quality of structural ceramics is mostly influenced by the selected composition of formation mix and burning regime. Many Lithuanian and foreign scientists work in this field of research. However, a great deal of attention also needs to be paid to the process of ceramics drying, as an inappropriate drying regime can be the main reason for causing first defects in ceramic products.

Drying is the process during which the moisture of the material is evaporated in a thermal way. As the material dries, the process of diffusion happens, during which the moisture is diffusing from the inner layers into the surface, and from it the moisture is evaporating into the atmosphere. The moisture present in material is divided into: free moisture, absorbent moisture and chemically bound moisture. Free moisture can be also named mechanical or capillary moisture, as it is soaked into the cavities of material and bigger capillaries. This moisture is easily removed when drying the material.

Absorbent moisture which is soaked into small capillaries of the material can be also named structural moisture. It is not completely eliminated from the material during drying. The moisture which is unfree chemically (crystalline, hydrate) is not removed when drying the material (Nagrockienė *et al.* 2005).

Defects in the semi-manufactured ceramic appear because the free water (between the particles of clay) is drifting away, the particles of clay move closer to each other, and the product dimensions decrease. During shrinkage mechanical strains appear, which may exceed the allowable limits because of the too fast water drift; consequently a semi-manufacture cracks even though the burning process has not started yet (Ткачев и др. 1999).

The authors (Lewis 2000; Briscoe *et al.* 1998) offer to divide drying process into two parts: linear drying of ceramics (when moisture is driven out from capillaries) and non-linear one, when moisture is driven out from pores in the vapour-diffusion method. These authors have determined that the ceramics drying depends on the relationship between humidity and water, drying temperature, and atmosphere conditions in a sample. Whereas scientists (Krischer 1978; Sadūnas 1997) consider that the burning process of porous bodies can be divided into 3 phases. First phase proceeds because of water migration through the capillaries from the inside to the surface of a sample. This stage particularly depends on temperature, humidity, and spread of air as well as on geometry, and dimensions. During the second stage of material drying the main water evaporation proceeds inside a sample, where the pressure of water vapour is prevailing. The third stage proceeds because of the vapour diffusion in all the spots of material. The first stage is the most risky as a high tension arises inside the material and incorrectly selected burning regime causes material to crack and crumble.

The other scientists (Amoros *et al.* 2003; Barati *et al.* 2003), according to the results of their researches, have shown that ceramics properties (deformation strength) depends on the material moisture and the time of drying. They have determined that the drier is the ma-

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terial, the greater is the mechanical strength. For example, as water absorption equals 5%, strength – 0.5 MPa, and when water absorption is 1%, the strength of analysed ceramics reaches 1.6 MPa.

The authors (Palmero *et al.* 2005) in their publication have shown that drying temperature (dried at 5, 25 and 60 °C temperature) influences the technique of ceramics crystallization and the solidity of final composition. The most solid composition of samples was obtained while drying ceramics at 60 °C temperature. The scientists (Seipel, Nickel 2004) have determined that the critical temperature is 150 °C, as drying samples at a higher temperature causes defects because of the inner tension arising inside the material.

However, there are very few researches where the influence of drying temperature or other drying factors on the final ceramics properties is analysed. Most authors investigate the kinetics of the processes which occur while drying ceramic bodies under standard drying conditions (Looi *et al.* 2002; Misra *et al.* 2002).

The purpose of our work is to demonstrate how the separate changing of each drying regime stage influences the final properties of ceramics (density, general shrinkage, compressive strength and the rate of ultrasound spread) and vice versa, how the desirable values of physical and mechanical parameters influence the magnitudes of each drying stage while the composition, burning regime, and other technological factors are stable.

2. Characteristics of materials, research methods

Material mix for ceramic samples was formed on the basis of clay from Rokai deposit and such additives were applied: sand, crushed bricks, sawdust of softwood. The average chemical composition of clay from Rokai deposit is presented in Table 1.

The samples were shaped in a plastic way and dosage of components was performed by mass. At first dry materials were mixed manually, later the mix was wetted to the moisture suitable for moulding. The amount of water poured was such that the material mix would be easily moulded and would not stick to hands when squeezed. Such mix was left for 3 days in the medium of (95±5) % relative humidity for moisture evenly spreading in the mix. After 3 days the laboratory samples were shaped into dimensions of 70×70×70 mm. The formed semi manufactures were being dried under 8 different regimes (Table 2), which were expressed by the values of relative area; the maximum area was equated to 100. In this way the sample heat quantity was obtained (the example of calculation is presented in Fig. 1). The drying regime was divided into two stages: drying in a laboratory and drying in the electric stove at the maximum temperature. The burning regime was not varied during our investigation. The dried samples were burned in an experimental chamber oven for 24 hours keeping the maximum temperature 1050 °C for 3 hours. The burned ceramic samples were used to determine the physical and mechanical parameters (according to Nagrockienė *et al.* 2005; Mandeikytė, Šiaučiūnas 1997; LST EN 771-1+A1 2005). The revised methodology for calculation of physical and mechanical parameters is presented in Table 3 and the average values of the above-mentioned parameters are shown in Table 4.

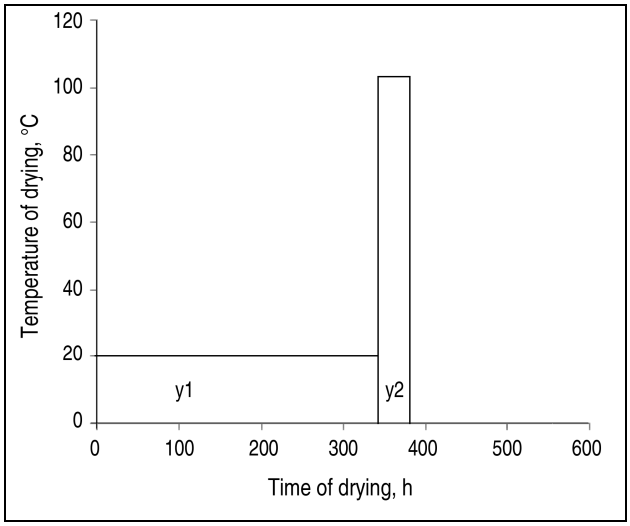

Fig. 1. The evaluation of the relative square measurements of drying regime

Table 2. Drying regimes

No. of drying regime	Stage of drying in a laboratory, y_1 units	Maximum drying temperature in a laboratory, °C	Stage of drying in the electrical stove y_2 units	Maximum drying temperature 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

Table 1. The average chemical composition of clay from Rokai deposit

Chemical composition, %								
SiO_2	Al_2O_3+TiO_2	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	Kaitmenys
47.66	18.32	6.27	8.11	3.04	2.68	0.16	–	12.60

Table 3. The revised (Nagrockienė *et al.* 2005; Mandeikytė, Šiaučiūnas 1997; LST EN 771-1+A1 2005) methodology for calculation of physical and mechanical parameters

Notation	Description of the basic parameter and units of measurement	Physical meaning of parameters	Formulas for determination	Description of the partial values and units of measurement
x_1	Density ρ, kg/m^3	Density – mass of unit volume of the material under natural state, expressed by the ratio of mass per volume	$\rho = \frac{m_{bs}}{V_b}$	m_{bs} – mass of dry sample, kg V_b – volume of sample, m^3
x_2	General shrinkage S_B, %	General shrinkage – decrease of the formed sample in volume during drying and burning. At the same time the magnitude of shrinkage indicates how larger products should be formed to obtain necessary dimensions after burning	$S_B = \frac{l_0 - l_1}{l_0} \cdot 100$	l_0 – distance between stamps marked in the undried sample, mm; l_1 – distance between marked stamps in the burned sample, mm
x_3	Compressive strength R_{gn}, MPa	Compressive strength, limit dynamical load, the ceramic body was still capable to withstand	$R_{gn} = \frac{P}{S} \cdot 0.85$	P – maximum load determined by experiment, MN; S – compressive area of sample, m^2
x_4	Rate of ultrasound spread, v	Rate of ultrasound spread – it is the parameter indicating the defects of material composition. Rate of ultrasound spread depends to a great extent on material strength and can be used as indirect method in determining the strength of product	$v = \frac{l}{t \cdot 10^{-6}}$	l – length of sample, m; t – time, the ultrasound spreads through sample, s

Table 4. Average values of physical and mechanical parameters

No. of batch of samples	ρ (x_1), kg/m^3	S_B (x_2), %	R_{gn} (x_3), MPa	v (x_4), m/s
1	1528	5.32	7.49	2318
2	1572	7.47	17.68	3460
3	1591	7.47	17.66	3323
4	1579	8.08	15.02	2832
5	1572	5.58	9.78	2768
6	1573	6.01	12.46	3199
7	1592	8.53	18.42	3567
8	1586	8.15	11.37	3096

Table 5. The double correlative matrix of the analysed physical and mechanical parameters and selected drying parameters

Parameters	ρ (x_1)	S_B (x_2)	R_{gn} (x_3)	v (x_4)
ρ (x_1)	1.00	0.64*	0.23	0.41*
S_B (x_2)	–	1.00	0.42*	0.58*
R_{gn} (x_3)	–	–	1.00	0.65*
v (x_4)	–	–	–	1.00
y_1	0,28	0,67*	0,34	0,38
y_2	0,65*	0,88*	0,46*	0,54*

Note: * – indicates that double correlation between parameters is significant

3. Statistical analysis of data discussion

We have determined the influence of selected drying stages on the values of physical and mechanical parameters by performing a statistical analysis (Gatti 2005; Lindsey 2004; Mees 2001).

It is determined that the distribution character of the experimental values of the analysed physical and mechanical parameters is normal, so it is possible to derive adequate empirical equations.

Table 5 presents the double correlative matrix of the analysed physical and mechanical parameters and selected drying parameters, which show that the interdependence of the most parameters is strong and significant, thus one has to keep this in mind when deriving empirical equations.

The reciprocal regression analysis (Malaiškienė, Mačiulaitis 2004; Mačiulaitis, Malaiškienė 2007) of data was performed in order to evaluate the dependence of the analysed parameters on the drying regime more precisely and to use the equations in practice. The adequacy of the derived equations was verified applying Fisher's criteria. If these criteria of the derived equation are higher than one found in the tables, the equation is considered to be suitable for presenting experimental data. The strength of correlation is estimated according to the values of the multidimensional coefficient of correlation. The closer the coefficient is to 1, the stronger is the correlation between the parameters. The adequacy of the model is verified by calculating the coefficient of determination. If the values of this coefficient are higher than 0.7, the selected mathematical model demonstrates the distribution of experimental data very well (Gatti 2005; Lindsey 2004; Mees 2001).

Firstly, the empirical equations are derived and they show how the values of physical and mechanical parameters change depending on the selected measures of the drying regime stage:

$$x_1 = (1536 + 0.161y_1 + 0.901y_2)(x_1 < 1582) + (1579 - 1.234y_1 + 2.229y_2)(x_1 \geq 1582), \quad (1)$$

$$x_2 = (4.294 + 0.066y_1 + 0.064y_2)(x_2 < 6.989) + (6.813 + 0.017y_1 + 0.019y_2)(x_2 \geq 6.989), \quad (2)$$

$$x_3 = (7.957 - 0.192y_1 + 0.170y_2)(x_3 < 13.40) + (15.49 - 0.022y_1 + 0.083y_2)(x_3 \geq 13.40), \quad (3)$$

$$x_4 = (2484 - 14.99y_1 + 19.21y_2)(x_4 < 3067) + (3237 + 1.533y_1 + 3.197y_2)(x_4 \geq 3067). \quad (4)$$

The multidimensional coefficients of correlation and determination and the average standard deviation of the empirical equations (1)–(4) are presented in Table 6. The multidimensional coefficients of correlation of equations (1)–(4) (Table 5) are close to 1; consequently, we state that the correlation between the analysed parameters is strong; the coefficients of determination are higher than 0.7, so the model is selected properly. The average standard rate of deviation is low (the highest is approximately 5%, determined to the value of the rate of ultrasound spread). Therefore the actual values and the values counted according to the empirical equations will differ slightly.

Table 6. The coefficients of correlation (R), determination (R^2) and standard deflection (s_e) of (1)–(4) empirical equations

No. of Eq.	Parameters	R	R^2	s_e
1	Density, x_1	0.939	0.881	9.61 kg/m^3
2	General shrinkage, x_2	0.955	0.911	0.32%
3	Compressive strength, x_3	0.917	0.841	1.63 MPa
4	Rate of ultrasound spread, x_4	0.880	0.774	179.5 m/s

It is shown in the empirical equation (1) how the selected measures of the drying regime stages influence the values of the ceramic body density. The stage of drying in the electric stove influences density positively, i.e. in order to get the higher values of density, the higher value of drying in the electric stove must be selected. This occurs because of the fact, that the more gradually and better material is dried, the more samples shrink; the size of pores and the capacity of the semi-manufacture decrease while the density increases. The stage of drying in the electric stove influences the density differently below and above the turning point (1582 kg/m^3, Eq 1). In order to obtain a higher density than 1582 kg/m^3 and to increase the value of drying in the electric stove, the lower values of drying in a laboratory must be selected. It is possible to reduce the duration of drying in a laboratory to a minimum (in the case of our performed research 8.16 units (Table 2), i.e. 72 h at 20 °C); otherwise the samples will dry according to an extremely intensive regime and the free moisture will not remove from the inner layers of material in time; consequently, the inner strains will emerge and the samples will crack.

It is possible to state from the empirical equation (2) that the values of general shrinkage are highly influenced by the measures of drying stages in the electric stove and drying in a laboratory as the tendencies for change remain similar below and above the turning point. The higher the values of drying in a laboratory and drying in the electric stove we select, the higher general shrinkage we obtain. That is because the semi-manufactured ceramics get larger amount of heat energy due to which the larger amount of water evaporates, the particles of material move closer to one another and the product shrinks.

The empirical equation (3) shows that the stages of drying regime have influence on the values of compressive strength with the same tendency below and above the turning point (13.40 MPa). If we like to obtain the values of compressive strength higher than 13.40 MPa, we have to increase the drying stage in the electric stove for decreasing the stage of drying in a laboratory. It is because the most intensive evaporation of the free and absorbent moisture from the inner layers is on the stage of drying in the electric stove. If drying lasts quite long, water will evaporate from the sample (not exceeding the allowable inner strains), less defects will be caused and the compressive strength will be higher. The minimal stage of drying in a laboratory will suffice for this case (according to the results of our research 8.16 units Table 2, i.e. 72 h at 20 °C). If the stage of drying in the electric stove is too low, the articles will have lower compressive strength because the moisture will not evaporate in time, and consequently the strains will emerge in the sample (Fig. 2).

Fig. 2. The sample of batch 1 after burning (Table 2)

It can be seen from the empirical equation (4), how the rate of ultrasound spread is influenced by the selection of drying regime. The index of ultrasound spread rate is one of the most important parameters contributing to the evaluation of the level of defects in the sample‘s structure. The drying duration in a laboratory influences the index of ultrasound spread rate below and above the turning point (3067 m/s) differently. The higher the drying stages in the electric stove and in the laboratory, the bigger are the values of ceramics ultrasound spread rates. It may be explained by the fact that higher values of drying stages provide conditions for free moisture to be removed more gradually from the material cavities and large capillaries. Also, under higher values of drying stages, the absorbent moisture is removed more gradually from small pores and capillaries later on. For these rea-

sons the large inner strains and defects will not emerge. That is proved by the 5th and 6th empirical equations of the reciprocal subordination.

Now we will analyse the empirical equations of reciprocal subordination in order to examine the validity of the above-mentioned statements and to use them in practice.

$$y_1 = \begin{pmatrix} -118.8 + 0.076x_1 + 0.443x_2 \\ -0.100x_3 + 0.003x_4 \end{pmatrix} (y_1 < 21.77) + \begin{pmatrix} -188.1 + 0.088x_1 + 8.210x_2 + \\ 1.754x_3 + 0.0005x_4 \end{pmatrix} (y_1 \geq 21.77), \quad (5)$$

$$y_2 = \begin{pmatrix} -107.7 + 0.059x_1 + 4.295x_2 + \\ 0.045x_3 - 0.0006x_4 \end{pmatrix} (y_2 < 23.02) + \begin{pmatrix} -229.4 + 0.140x_1 + 1.791x_2 + \\ 0.809x_3 + 0.005x_4 \end{pmatrix} (y_2 \geq 23.02). \quad (6)$$

The multidimensional coefficients of correlation, determination and standard deflection of (5)–(6) empirical equations are presented in Table 7. The coefficients of correlation show that there is an extremely strong interdependence between the stages of drying regime and physical and mechanical parameters as the coefficient of determination is higher than 0.7, thus the mathematical model is selected properly. The values of the average rate of standard deflection are low, so the actual values obtained according to the empirical equations differ slightly.

Table 7. The coefficients of correlation (R), determination (R^2) and standard deflection (s_e) of empirical equations (5)–(6)

No. of Eq.	Parameters	R	R^2	s_e
5	The stage of drying in a laboratory, y_1	0.969	0.940	3.26 units
6	The stage of drying in the electric stove, y_2	0.985	0.971	2.07 units

The empirical equation (5) shows, how the duration of the drying stage in a laboratory influences the values of physical and mechanical parameters below and above the turning point 21.77 units. When the drying stage in a laboratory is selected higher than 21.77 units, we will get such results: the higher density (more than 1582 kg/m^3, Eq 1), the higher general shrinkage (more than 6,989%, Eq 2), the higher compressive strength (more than 13,40 MPa, Eq 3) and the higher rate of ultrasound spread (more than 3067 m/s, Eq 4). If we select the drying duration in a laboratory lower than 21.77 units (Eq 5), it is likely that the values of compressive strength will decrease. The duration of this stage must be not lower than 8.16 units (72 h at 20 °C); otherwise, the free moisture will evaporate too quickly and the strains will emerge.

We see from the empirical equation (6) that the drying duration in the electric stove influences the values of density, general shrinkage and compressive strength equally below and above the turning point of 23.02 units. The rate of ultrasound spread varies according to the value of the turning point (23.02 units, Eq 6). The higher stage of drying in the electric stove we select, the higher density, general shrinkage, compressive strength, and the rate of ultrasound spread we obtain. That is because of the fact that when we heat the material, the moisture evaporates gradually, particles move closer to each other, the semi-products shrink, and a new stronger inner frame of the material is established. When selecting the values of drying in the electric stove lower than 23.02 units, the rate of ultrasound spread can begin to decrease as the sample can dry inadequately and more open pores and capillaries appear when burning. Thus, the equations of reciprocal subordination confirm previously formed statements.

4. The example of the use of empirical equations

The example of empirical equations usage in practice is presented when we vary only the values of drying regime parameters, and all other technological conditions remain constant. The formation mix of ceramic body was prepared using 77% of clay, 10% of sand, 4.5% of chip (the carcass of encaustic ceramics) and 8.5% of cuttings of coniferous trees. It was burnt for 24 h keeping at the maximum temperature of 1050 °C for 3 hours.

When selecting the desirable values of ceramics, it is essential to consider the tendencies of cohesion between the special parameters and the parameters of burning regime (Table 4).

Example. Let us suppose, we want to get a ceramic body with special physical and mechanical parameters, e.g. density 1600 kg/m^3, general shrinkage 7%, compressive strength 17 MPa, the rate of ultrasound spread 3300 m/s. Then we insert these values into (5)–(6) equations (the part of the equation with a lower energy input is used) and obtain these tentative parameters of drying regime: the drying stage duration in a laboratory 14.8 units and the drying duration in the electric stove is 15.6 units.

To ascertain reliability of these drying parameters, we insert their values into (1)–(4) equations and obtain that the density of ceramic density equals 1596 kg/m^3, general shrinkage is 7%, compressive strength is 16.5 MPa, and the rate of ultrasound spread is 3310 m/s. Consequently, the values differ only slightly from the desirable values and it is possible to expect the mentioned physical and mechanical parameters of final products, when applying the parameters of drying regime.

5. Conclusions

1. A strong interdependence between the selected stages of drying regime and analysed physical and mechanical parameters has been determined. The values of multidimensional coefficients of correlation characterizing the interdependence are R = 0.880...0.985.

2. It has been confirmed that a properly selected drying regime can improve the properties of the final ceramic product, while other technological factors are constant. For example, the values of compressive strength can increase up to 88.3% and more.

3. An example of how the derived empirical equations can be applied in practice has been provided. When checking the equivalence of experimental results to the calculated values according to empirical equations, it is determined that all other technological conditions do not vary, and it is possible to select the drying regime according to the physical and mechanical parameters; and, conversely, it is possible to forecast the final physical and mechanical parameters of a ceramic product.

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KERAMINIŲ PLYTŲ FIZIKINIŲ IR MECHANINIŲ SAVYBIŲ REGULIAVIMAS DŽIOVINIMO REŽIMU

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S a n t r a u k a

Statybinės keramikos kokybę labai lemia formavimo masės komponentų ir jų kiekių bei degimo režimo parinkimas. Tačiau taip pat labai svarbus yra ir teisingas keraminio pusgaminio džiovinimo režimo parinkimas, nes netinkamas keraminių pusgaminių džiovinimo intensyvumas gali būti pagrindinė keramikos dirbinių defektų atsiradimo priežastis. Defektų keramikos bandiniuose atsiranda todėl, kad per intensyviai džiovinant bandinius pusgaminyje atsiranda vidinių įtempių, kurie dėl per greito laisvo vandens pasišalinimo viršija leistinus ir pusgaminis trūkinėja net nepradėjus degimo proceso. Todėl, norint gauti kokybiškus keraminius dirbinius, yra svarbu nustatyti, kaip parinkti džiovinimo etapų dydžiai daro įtaką galutinėms keraminės šukės fizikinių-mechaninių rodiklių reikšmėms, taip pat būtų aktualu išnagrinėti džiovinimo režimo reguliavimo galimybes praktiškai.

Tikslui pasiekti buvo suformuoti vienodos sudėties keraminiai bandiniai, kurie buvo išdžiovinti pagal 8 skirtingus džiovinimo režimus ir išdegti pagal vieną pasirinktą režimą. Vėliau eksperimentiškai buvo nustatyti pasirinkti fizikiniai-mechaniniai rodikliai. Atlikta statistinė ir regresinė analizė, gautos empirinės lygtys, kurias naudojant galima parinkti džiovinimo režimą pagal norimus gauti fizikinius-mechaninius rodiklius, ir, atvirkščiai, parinkus džiovinimo režimą galima prognozuoti, kokios keraminės šukės fizikinių-mechaninių rodiklių reikšmės bus gautos.

Reikšminiai žodžiai: keramika, fizikinės ir mechaninės savybės, džiovinimo režimas, statistinė analizė.

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