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Influence of Technological Factors on the Structural Parameters of Clay Masonry Units

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In this work the influence of eight technological factors (clay, non-plastic materials, burning out additive, waste materials, mixing effectiveness in a mixer, degree of compression in formation heads, level of vacuuming in a vacuum chamber, duration of keeping at the highest burning temperature) on the determined specific characteristics of structural parameters of ceramic samples: reserve of pore volume, relative wall thickness of the pores and capillaries, capillary rate of mass flow under vacuum respectively in the direction of freezing and perpendicular to this, degree of structural in homogeneity, capillary rate of mass flow in the direction of freezing, is analysed applying fractional factorial design at two levels. On before-mentioned by structural parameters it is possible to estimate frost resistance of clay masonry units. Regression equations are derived enabling to predict the values of specific characteristics of structural parameters properties of clay masonry units or knowing the desirable values of the properties to determine the optimal effect of the technological factors under investigation and thus to produce the clay masonry units of different purpose with the desirable properties within certain limits.

Keywords: product quality, clay masonry units, experimental design, structural parameters, frost resistance.

INTRODUCTION

Production process of the clay masonry units includes many technological operations, each of them influencing the final product quality. To analyse the influence of all technological factors on the product properties and the reciprocity is a complicated, expensive and labour-intensive operation. For that purpose special statistical methods and means may be applied enabling to analyse the reciprocity of technological factors and the influence on qualitative characteristics of products quite clear and objectively, to find their optimal values decreasing the number of experiments considerably [1 – 4].

There is a fair amount of works, where the experimental design was applied for determination of technological factors` influence on product quality [5 – 17]. Advantage of this method over the classical analysis methods is the decrease in a number of experiments and a big amount of statistically proved information from the analysis of investigation results enabling to make the concrete conclusions.

These technological factors were examined: mixing effectiveness in a mixer, level of vacuuming in a vacuum chamber, length of pressure and formation heads, speed of formation band outlet from a formation head, treatment and composition of formation mix, drying and burning regimes of semimanufacturer and organic film applied. Many works have also been performed investigating influence of technological factors on frost resistance of clay masonry units [12, 13, 15, 18, 19]. The authors [20] have analysed in details interdependence among separate components of formation mix and various structural parameters of ceramic body, necessary for forecasting of exploitation frost resistance. The authors [8 – 12] who analysed more technological factors that have an effect on the characteristics of ceramic articles established that besides the influence of formation mix's composition, manufacturing factors also influence the characteristics of the articles significantly. Therefore, a thorough investigation is needed on the influence of technological factors on the characteristics of ceramic articles.

Frost resistance of clay masonry units may be determined by direct methods (all side or one side freezing-thawing), however these investigations take several weeks or even months; also it is possible to determine the frost resistance of clay masonry units by rapid forecasting methods (according to the size and distribution of pores and capillaries, structural and deformation parameters, physical-mechanical and structural characteristics) [13, 21 – 23].

R. Mačiulaitis [21, 22, 24] has offered the equations for prediction of frost resistance of clay masonry units according to the beginning and the end of destruction:

$$F_{R1E}\ (F_{R2E}) = \frac{1}{e^{a}} \times \frac{R_p^b D^c G_1^d G_2^e}{N_H^f g_2^g}\ , \qquad (1)$$

where R_p is the reserve of pore volume, D is the relative wall thickness of the pores and capillaries, G_2 is the capillary rate of mass flow under vacuum respectively in the direction of freezing and G_1 perpendicular to this, N_H is the degree of structural in homogeneity, g_2 is the capillary rate of mass flow in the direction of freezing, *a*, *b*, *c*, *d*, *e*, *f*, *g* – are the coefficients.

The aim of this work is to analyse the significance of the selected technological factors, the influence of significant factors on the determined structural parameters of ceramic samples applying the experimental design and to derive the regression model equations, enabling to predict the structural parameters of clay masonry units or

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knowing the desirable properties to determine the optimal values of the technological factors under investigation.

MATERIALS AND INVESTIGATION METHODS

For formation of samples these materials from Lithuanian mines were used: clay A and the thinner clay B, sand, crushed brick, burning out additive – anthracite, waste materials – glass, catalyst from the catalyst-cracking reactor. The sand was sieved through the 1 mm aperture sieve, the crushed stone was crumbled by alligator and sieved through the 2.5 mm sieve, for the anthracite the sieve with 2 mm apertures was used, the glass was ground in the disintegrator and sieved through the 1 mm apertures, the catalyst was applied in the form of granules (1.3 mm – 1.5 mm). Components of the formation mix were dosed by mass, mixed by the two-roll screw mixer of Z type, moistened to the humidity necessary for formation and left to lie for 4 days. The tiles with dimensions (120×60×40) mm and 14 cavities were formed by the band vacuum press. The tiles were dried in natural conditions. The dried tiles were burned in the electric semi-industrial stove at the highest clay A burning temperature.

Ceramic samples were produced according to the fractional factorial design at two levels. Eight technological factors were selected, changing in two level (Table 1), that are conditionally encoded: –1.0 (conditionally bad) and 1.0 (conditionally good). In the case of full plan the number of experiments allowing to investigate the interaction of all factors would be $N = 2^8 = 256$, and applying the partial plan $2^{(k–p)} = 2^{(8–4)}$ [1, 2], the number of experiments was decreased to 16 tests.

Table 1. Table of factors` encoding

Notation	Factor	Conditionally bad		Conditionally good	
x_1	Clay (A or A+B)	–1	A (P* = 19.1)	1	A+B (20 % of clay A mass) (P* = 11.14)
x_2	Non-plastic materials (sand + crushed stone)	–1	8 % (5 % sand + + 3 % crushed stone)	1	26 % (18 % sand + + 8 % crushed stone)
x_3	Burning out additive (anthracite)	–1	0 %	1	1.2 %
x_4	Waste materials (catalyst + glass)	–1	0 %	1	22.8 % (6 % catalyst + + 16.8 % glass)
x_5	Mixing effectiveness	–1	5 min	1	15 min
x_6	Level of vacuuming	–1	0.65 MPa	1	0.9 MPa
x_7	Degree of compression	–1	Short formation head	1	Long formation head
x_8	Burning duration	–1	Keeping at the highest burning temperature for 1 h	1	Keeping at the highest burning temperature for 5 h

*P – average plasticity number.

According to the developed randomising experiment matrix, 16 batches of ceramic samples were produced and their structural parameters determined. Reserve of pore volume ($R_{\text{p.exp.}}$), relative wall thickness of the pores and capillaries ($D_{\text{exp.}}$), capillary rate of mass flow under vacuum respectively in the direction of freezing ($G_{2.\text{exp.}}$) and perpendicular to this ($G_{1.\text{exp.}}$), degree of structural in homogeneity ($N_{\text{H.exp.}}$), capillary rate of mass flow in the direction of freezing ($g_{2.\text{exp.}}$) was determined according to special methodology [21, 22].

Experimental results were processed by the method of random balance [4] and the software of experimental design [25].

RESULTS AND ANALYSIS

Performing the analysis of reserve of pore volume values we have determined that the technological factors: x_3 (burning out additive), x_4 (waste materials), x_5 (mixing effectiveness), x_6 (level of vacuuming), x_7 (degree of compression), x_8 (burning duration) and interdependence among technological factors x_2x_3 (non-plastic materials by burning out additive), x_3x_4 (burning out additive by waste materials), x_3x_5 (burning out additive by mixing effective-ness), x_3x_6 (burning out additive by level of vacuuming), x_4x_5 (waste materials by mixing effectiveness), x_5x_8 (mixing effectiveness by burning duration), x_6x_7 (level of vacuuming by degree of compression) have the significant influence on the reserve of pore volume of ceramic samples, other factors have insignificant influence. Conditional influence of technological factors effect on reserve of pore volume is presented in Fig. 1. Conditional values of effects are interpreted so: we decide on the magnitude of influence from the numerical value, and we decide on the positive or negative influence of technological factor on the property under investigation from the sign. Statistical significance $p = 0.05$ shows the possibility of error, i.e. the possibility to fall into error 5 %.

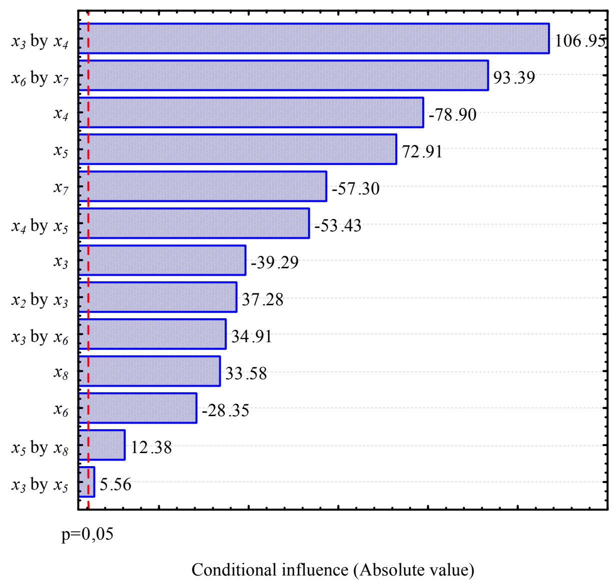

Fig. 1. Conditional influence of technological factors on reserve of pore volume

Substituting the obtained values of regression coefficients to the incomplete quadratic regression model equation 2 we have obtained regression model equation for the reserve of pore volume (equation 3):

$$A = b_0+b_1x_1+b_2x_2+...+b_mx_m+...+b_{12}x_1x_2+...+b_{mn}x_mx_n \quad (2)$$

where A is the property, b_0 is the overall average coefficient, b_1, b_2, ...,b_m – are the coefficients of the first set effects; b_{12}, b_{23}, ..., b_{mn} – are the coefficients of the second set effects; x_1, x_2, ..., x_m (x_n) – are the correspondingly the equation of technological factor (–1 or 1).

$$R_{\text{p.calc.}} = 35.19 - 2.16x_3 - 4.28x_4 + 3.47x_5 - 1.19x_6 - 2.85x_7 + 2.52x_8 + 2.87x_2x_3 + 4.25x_3x_4 + 0.29x_3x_5 + 2.71x_3x_6 - 3.89x_4x_5 + 1.33x_5x_8 + 4.38x_6x_7 . \quad (3)$$

Comparing the adequacy of experimental and calculated data according to Fisher criteria when the significance level is $\alpha = 5$ %, we confirm that equations of models are adequate.

Comparison of the values of porous volume reserve calculated by the derived regression model equation 3 and the experimental values is presented in Fig. 2. We can see that the model obtained is valid for the received experimental values.

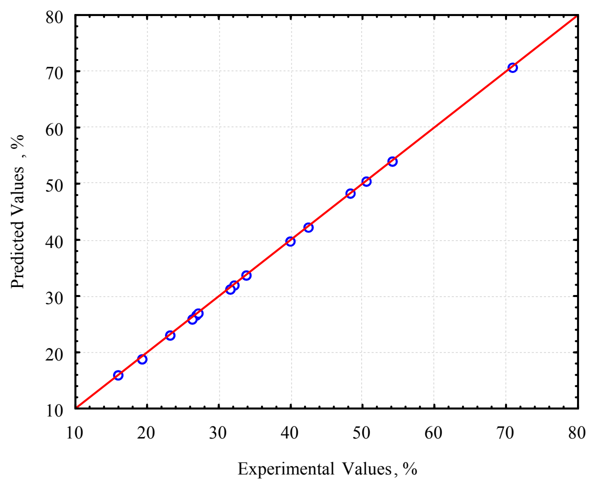

Fig. 2. Comparison of the experimental and the calculated values of porous volume reserve

Analogously the analysis of values of relative wall thickness of the pores and capillaries, capillary rate of mass flow under vacuum respectively in the direction of freezing and perpendicular to this, degree of structural in homogeneity, capillary rate of mass flow in the direction of freezing was performed. Conditional influence of technological factors effects is presented correspondingly in Figures 3, 5, 7, 9, 11. Comparison of the experimental and the calculated values is presented in Figures 4, 6, 8, 10, 12.

The regression model equations are obtained for: relative wall thickness of the pores and capillaries (equation 4), capillary rate of mass flow under vacuum respectively in the direction of freezing and perpendicular to this (equation 5 and 6), degree of structural in homogeneity (equation 7), capillary rate of mass flow in the direction of freezing (equation 8):

$$D_{\text{calc.}} = 5.58 - 0.41x_1 - 4.14x_2 - 4.03x_4 + 1.50x_2x_4 + 1.31x_2x_5 + 1.90x_2x_6 + 2.19x_2x_8 + 2.07x_4x_5 + 2.25x_5x_6 + 1.61x_5x_8 - 3.82x_6x_7 - 3.79x_7x_8 ; \quad (4)$$

$$G_{\text{1.calc.}} = 0.34 + 0.11x_1 + 0.15x_2 + 0.12x_4 - 0.02x_5 + 0.08x_6 - 0.02x_7 + 0.01x_1x_5 - 0.08x_5x_6 + 0.03x_5x_7 - 0.06x_6x_7 + 0.09x_6x_8 - 0.04x_7x_8 ; \quad (5)$$

$$G_{\text{2. calc.}} = 0.18 + 0.03x_1 + 0.08x_2 + 0.09x_3 + 0.06x_4 + 0.01x_6 - 0.04x_7 + 0.02x_1x_2 - 0.02x_2x_8 ; \quad (6)$$

$$N_{\text{H.calc.}} = 0.48 - 0.19x_1 - 0.75x_2 + 0.39x_3 - 0.37x_4 + 0.13x_5 - 0.26x_6 - 0.22x_8 - 0.07x_3x_7 + 0.64x_5x_6 + 0.10x_5x_7 - 0.11x_5x_8 - 0.27x_6x_7 - 0.44x_6x_8 - 0.14x_7x_8 ; \quad (7)$$

$$g_{\text{2.calc.}} = 0.33 + 0.31x_2 - 0.19x_3 + 0.16x_4 - 0.05x_5 + 0.11x_6 + 0.03x_7 - 0.07x_8 - 0.04x_2x_5 + 0.002x_2x_7 - 0.22x_5x_6 - 0.06x_5x_7 + 0.07x_6x_7 + 0.18x_6x_8 . \quad (8)$$

Knowing the effectiveness of influence of technological factors under consideration on the structural parameters and the regression model equations, we can design manufacturing conditions. Substituting the quantitative magnitudes expressed in natural values to the obtained regression model equations 3, 4, 5, 6, 7 and 8 we have derived the following equations:

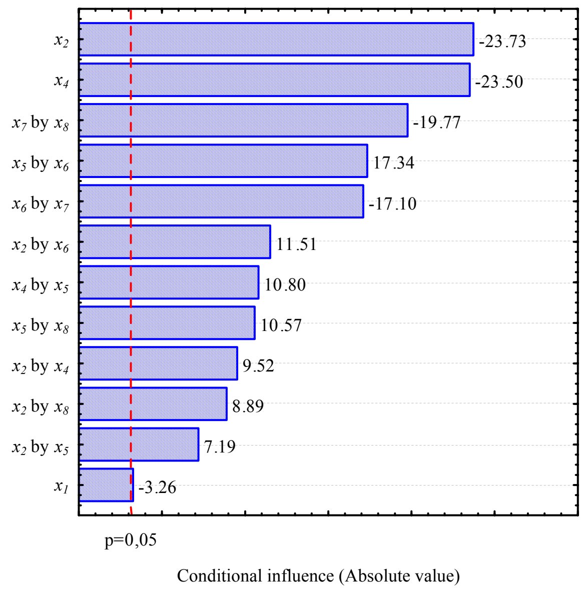

Fig. 3. Conditional influence of technological factors on relative wall thickness of the pores and capillaries

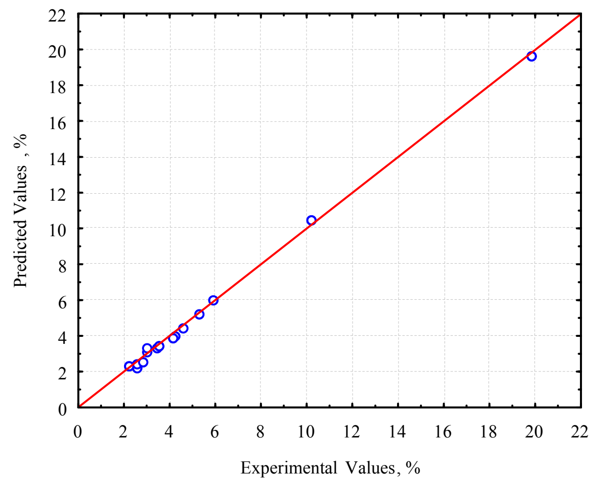

Fig. 4. Comparison of the experimental and the calculated values of relative wall thickness of the pores and capillaries

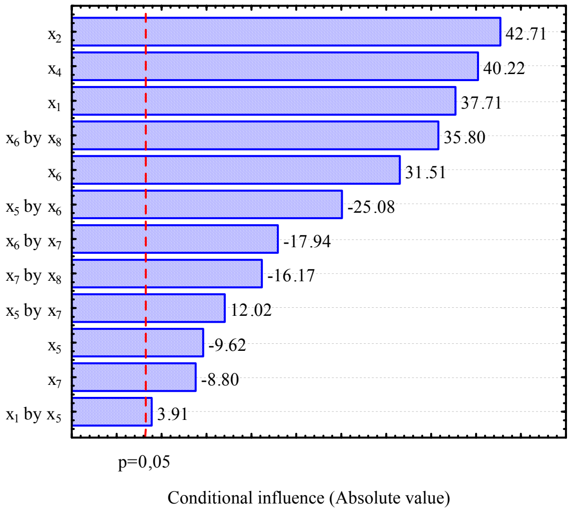

Fig. 5. Conditional influence of technological factors on capillary rate of mass flow under vacuum in a direction perpendicular to freezing

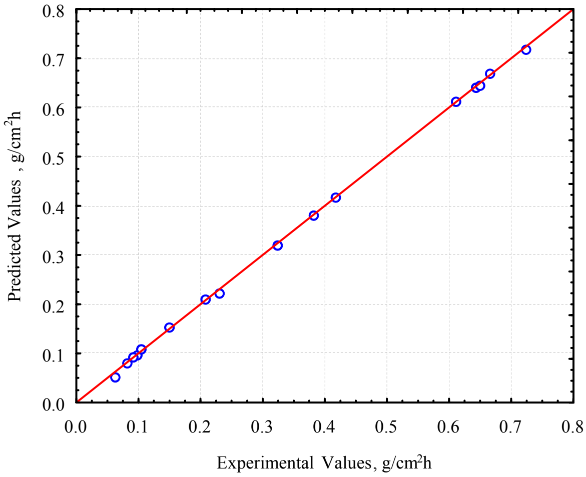

Fig. 6. Comparison of the experimental and the calculated values of capillary rate of mass flow under vacuum in a direction perpendicular to freezing

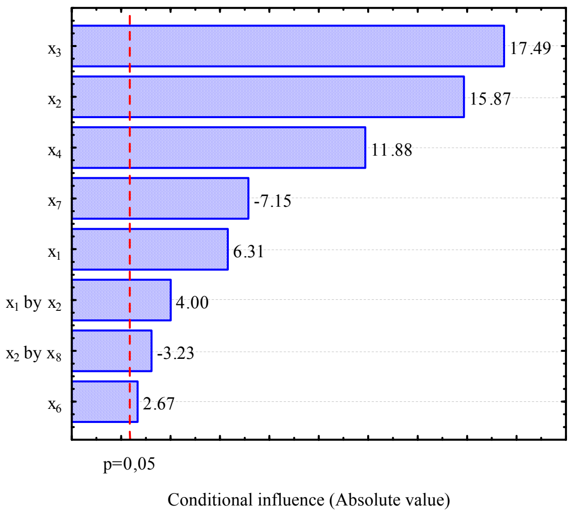

Fig. 7. Conditional influence of technological factors on capillary rate of the mass flow under vacuum in the direction of freezing

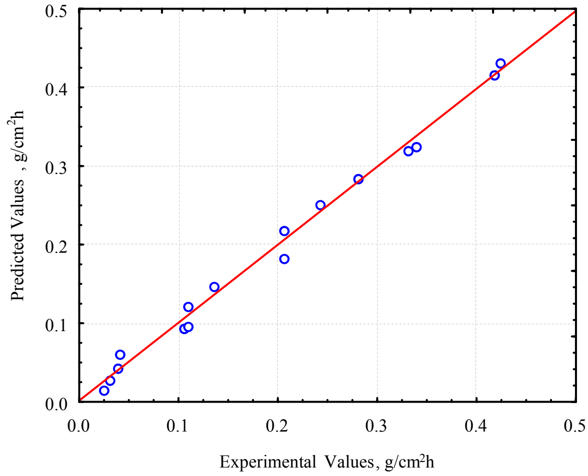

Fig. 8. Comparison of the experimental and the calculated values of capillary rate of the mass flow under vacuum in the direction of freezing

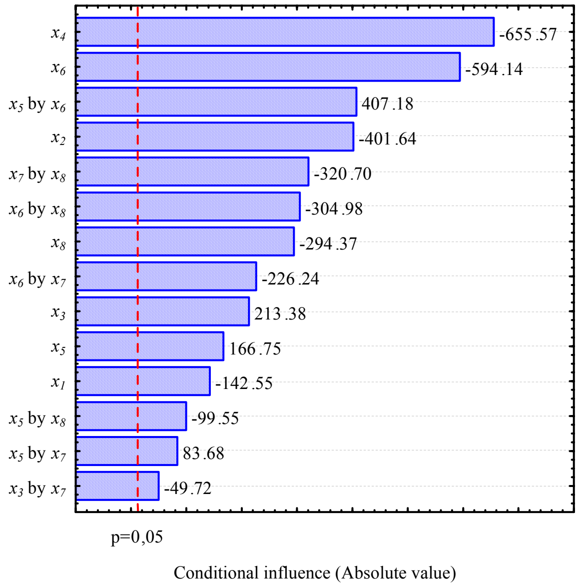

Fig. 9. Conditional influence of technological factors on degree of structural inhomogeneity

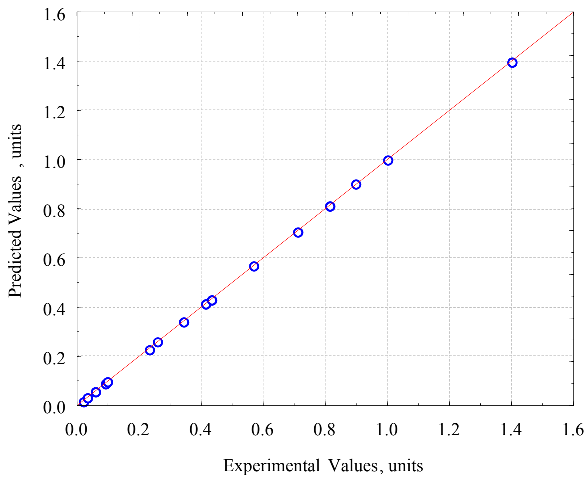

Fig. 10. Comparison of the experimental and the calculated values of degree of structural inhomogeneity

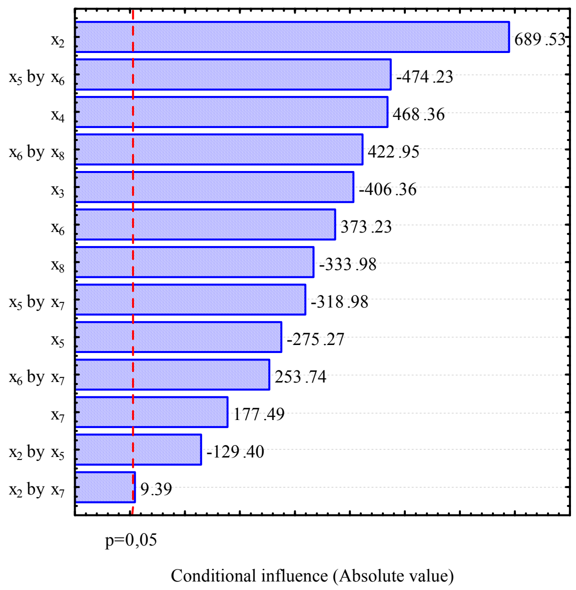

Fig. 11. Conditional influence of technological factors on capillary rate of the mass flow in the direction of freezing

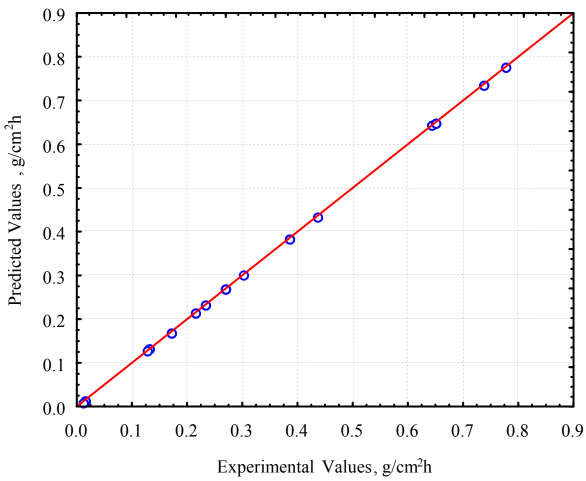

Fig. 12. Comparison of the experimental and the calculated values of capillary rate of the mass flow in the direction of freezing

$$R_{p.calc.} = 61.56 - 0.32X_2 - 48.65X_3 - 0.07X_4 + 1.01X_5 - 31.20X_6 - 30.01X_7 - 0.07X_8 + 0.53X_2X_3 + 0.62X_3X_4 + 0.10X_3X_5 + 36.13X_3X_6 - 0.07X_4X_5 + 0.13X_5X_8 + 35.04X_6X_7\,; \quad (9)$$

$$D_{calc.} = 90.48 - 0.41X_1 - 2.59X_2 - 0.96X_4 - 4.18X_5 - 64.73X_6 + 29.36X_7 - 3.65X_8 + 0.02X_2X_4 + 0.03X_2X_5 + 1.69X_2X_6 + 0.12X_2X_8 + 0.04X_4X_5 + 3.60X_5X_6 + 0.16X_5X_8 - 30.56X_6X_7 - 1.89X_7X_8\,; \quad (10)$$

$$G_{1\,calc.} = -0.67 + 0.10X_1 + 0.02X_2 + 0.01X_4 + 0.10X_5 + 0.84X_6 + 0.35X_7 - 0.28X_8 + 0.002X_1X_5 - 0.13X_5X_6 + 0.01X_5X_7 - 0.48X_6X_7 + 0.36X_6X_8 - 0.02X_7X_8\,; \quad (11)$$

$$G_{2\,calc.} = -0.24 - 0.01X_1 + 0.012X_2 + 0.15X_3 + 0.005X_4 + 0.08X_6 - 0.04X_7 + 0.02X_8 + 0.0022X_1X_2 - 0.001X_2X_8\,; \quad (12)$$

$$N_{H\,calc.} = 7.08 - 0.19X_1 - 0.08X_2 + 0.65X_3 - 0.03X_4 - 0.73X_5 - 7.04X_6 + 1.75X_7 + 1.36X_8 - 0.12X_3X_7 + 1.02X_5X_6 + 0.02X_5X_7 - 0.01X_5X_8 - 2.16X_6X_7 - 1.76X_6X_8 - 0.07X_7X_8\,, \quad (13)$$

$$g_{2\,calc.} = -1.90 + 0.04X_2 - 0.32X_3 + 0.01X_4 + 0.28X_5 + 2.24X_6 - 0.28X_7 - 0.59X_8 - 0.0009X_2X_5 + 0.0002X_2X_7 - 0.35X_5X_6 - 0.01X_5X_7 + 0.56X_6X_7 + 0.72X_6X_8\,, \quad (14)$$

where X_1 is clay ($X_1 = -1$ (Clay A) or $X_1 = 1$ (Clay A+B); X_2 is the amount of non-plastic materials, %; X_3 is the amount of burning out additives, %; X_4 is the amount of waste materials, %; X_5 is mixing effectiveness, min; X_6 is level of vacuuming, MPa; X_7 – degree of compression ($X_7 = -1$ (short formation head) arba $X_7 = 1$ (long formation head); X_8 is keeping at the highest burning temperature, h.

According to these equations the values of structural parameters of clay masonry units may be predicted. For example, applying the clay A with 8 % of non-plastic materials, without waste materials and burning-out additives, mixing for 15 minutes, forming with a short formation head, level of vacuuming in a vacuum chamber being 0.9 MPa and keeping at the highest burning temperature for 5 h we obtain that $R_{p.calc.} = 54.0$ %, $D_{calc.} = 18.9$ %, $G_{1\,calc.} = 0.35$ g/cm^{2}h, $G_{2\,calc.} = 0.03$ g/cm^{2}h, $N_{H\,calc.} = 1.01$ and $g_{2\,exp.} = 0.001$ g/cm^{2}h.

Determining the necessary values of structural parameters and evaluating the possible variation limits and solving the system of equations (9 – 14) we can obtain the optimal values of technological factors.

From the equation (1) it is seen that in order to increase the frost resistance of clay masonry units it is necessary to increase the values of structural parameters R_p, D, G_1, G_2 and decrease the values of N_H, g_2. Certainly, these parameters can be increased or decreased within certain limits, however the cases are possible when the increase of one parameter diminishes the other magnitude that actually should be increased and vice versa. Therefore an optimal ratio of these parameters should be defined that the biggest forecasted value of frost resistance would be obtained. The biggest forecasted frost resistance calculated by the Equation (1) within the variation limits of technological factors under consideration is obtained for 4 and 7 sample batches. The formation mix of the fourth batch was made of clay A+B with 8 % of non-plastic materials, without burning out and waste additives, the proportioned components being mixed 15 min in a mixer. The samples were formed in a band vacuum press with a short formation head under the pressure in a vacuum chamber being 0.65 MPa. Semimanufacturers were burned out in a chamber furnace keeping at the highest burning temperature for 5 h. The formation mix of the seventh batch was made of clay A with 8 % of non-plastic materials, without burning out and waste additives, the proportioned components being mixed 15 min in a mixer. The samples were formed in a band vacuum press with a short formation head under the pressure in a vacuum chamber being 0.90 MPa. Semimanufacturers were burned out in a chamber furnace keeping at the highest burning temperature for 5 h.

CONCLUSIONS

Applying the method of experimental design, we have derived the statistical-experimental regression model equations evaluating influence of technological factors and their interdependence with structural parameters of clay masonry units.

Applying the derived regression model equations we can forecast the frost resistance values of clay masonry units and to regulate technological manufacturing process so that the clay masonry units of the highest quality could be produced.

Setting the desirable frost resistance values we can also solve the reciprocal task, i.e. to find the optimal values of technological factors.

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