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Influence of Structural Parameters on the Frost Resistance of Clay Masonry Units

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Investigation of ceramic samples is presented in the article. To attain this aim, the samples formed by plastic molding out of local raw materials were dried under standard conditions in the dryer and burned at the temperature of 1000 °C with exposure time of 1 h at the maximum temperature of burning. In the paper these physical and structural parameters of ceramic samples are determined experimentally: density, water absorption, effective porosity, total open porosity, reserve of pore volume, relative wall thickness of the pores and capillaries, degree of structural inhomogeneity, capillary rate of mass flow and all-side and one-side frost resistance.The statistic analysis of data obtained in the study was done and the relationship between various structural parameters was established. It was proved that the structure of ceramic products predetermines the frost resistance of ceramic masonry products.
Keywords: clay masonry units, structural parameters, water absorption, porosity.

1. INTRODUCTION

The structure of burnt ceramic masonry products is subject to chemical and granulometric composition of raw materials and additives, their content in formation mix and various technological parameters [1 – 7].

The structure of ceramic masonry products can be characterized by various structure parameters. Much of work in that field of research has been performed by Lithuanian scientists [7 – 10]. The authors in the publication offer empirical equations defining the interdependence of service frost resistance and physical-mechanical, structural and other parameters.

It is well-known that during freezing of the soaking ceramics products, the water turns into ice. In this case stresses and strains are induced when volume increases by 9 %. The possibility to resist such stresses and strains depends on the structure and phase composition of the material carcass [7, 8]. The frost resistance of samples is measured by surface defects showing up during testing (such as peeling, splitting, cracking).

After A. Sadūnas [7], two types of decomposition dominate during freezing of products: disintegration, crumbling, peeling and splitting into two parts or pieces. The first type is a surface decomposition, characteristic of performance. During decomposition the water absorption of sample is greater than that of sample after vacuum compaction, as in the former case the migration of moisture into surface prevails. The second type of decomposition is not typical to performance (splitting into pieces). Here, during decomposition, the water absorption of sample is smaller than that of the same sample after vacuum compaction. Consequently, the migration of moisture takes place into defective places in the middle or center of sample.

Consequently, as the most simplified physical quantity, the frost resistance of ceramic facade products can also be characterized as the capacity of a porous body to fill it's defective volume with water, that as a results leads to a considerable destruction of the surface, followed by further separate cases of destruction.

The aim of this work is to perform the analysis of structural parameters (water absorption, effective porosity, total open porosity, reserve of pore volume, relative wall thickness of the pores and capillaries, degree of structural inhomogeneity, capillary rate of mass flow), to evaluate the structural parameters influence on frost resistance, to evaluate the relationship between various structural parameters.

2. EXPERIMENTAL

Chemical compositions of the main raw material were determined by the classical methods of chemical analysis for silicate materials [11].

Phase analysis of burned samples was carried out by the method of X-ray diffraction. The diffractometer DRON-2 (Russia) with Cu anti-cathode and Ni filter / Fe anti-cathode and Mn filter was used when $U = 30$ kV, $Ia = 8$ mA and rotation speed of the sample was $1°\text{min}^{-1}$. X-ray diffraction pattern were registered on paper by means of typed and decoded comparing with the data of PDC catalogue and research literature [12].

The fusible hydro – micous clay was selected for investigation (clay C1). Granulometric composition of this clay: amount of sand particles > 0.05 mm is from 0.09 % to 0.77 % amount of dust particles (0.05 – 0.005) mm is from 9.26 % to 21.39 %, and amount of clay particles < 0.005 mm fluctuates from 72.11 % to 96.04 %. Chemical compositions of the clay C1 : SiO_2 – 47.0 %, Al_2O_3+TiO_2 – 17.8 %, Fe_2O_3 – 7.7 %, CaO – 6.3 %, MgO – 3.6 %, K_2O – 4.5 %, Na_2O_3 – 0.5 %, L.o.l. – 11.2 %.

The fusible devonian clay – C2 was selected for the research. Chemical compositions of the clay C2 : SiO_2 – 66.3 %, Al_2O_3+TiO_2 – 15.8 %, Fe_2O_3 – 6.4 %, CaO – 1.8 %, MgO – 2.7 %, K_2O – 1.6 %, L.o.l. – 5.3 %.

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Granulometric composition of clay C2: amount of sand particles > 0.05 mm is 24 % amount of dust particles (0.05 – 0.005) mm is 34.03 %, and amount of clay particles < 0.005 mm fluctuates is 42.0 %.

The X-ray analysis of the clay C3 has shown that there is identified: hidromica (9.90; 4.98; 4.47; 2.56; 1.50 Å), kaolin (7.14; 3.56; 1.50 Å), quartz (4.25; 3.35; 2.45; 2.38; 2.28; 2.24; 2.20; 2.13; 1.99; 1.82; 1.67; 1.66; 1.54; 1.47; 1.45 Å), feldspar (3.24; 3.00; 1.80 Å) and dolomite (3,70; 1,80; 1,79 Å).

Chemical compositions of the clay C3: SiO_2 – 46.0 %, $Al_2O_3 + TiO_2$ – 18.09 %, Fe_2O_3 – 6.84 %, CaO – 8.02 %, MgO – 4.10 %, K_2O – 1.47 %, Na_2O – 0.43 %, SO_3 – 0.22 % L.o.l. – 12.38 %.

Granulometric composition of clay C3: amount of sand particles > 0.05 mm is 0.22 % amount of dust particles (0.05 – 0.005) mm is 16.22 %, and amount of clay particles < 0.005 mm fluctuates is 83.52 %.

The X-ray analysis of the clay C3 has shown that there is identified: hidromica (10.0, 4.98; 4.48; 2.56; 1.98; 1.50 Å), kaolin (7.14; 3.54; 1.98 Å), quartz (4.25; 3.35; 2.44; 2.28; 2.13; 1.82; 1.67; 1.62; 1.60; 1.54 Å), feldspar (3.23; 3.02 Å), dolomite (2.88; 2.39; 2.19; 1.79 Å), calcite (2.48; 2.09; 1.91; 1.87 Å) and chlorite (14.0; 3.54; 1.50 Å).

Non-plastic materials used in the work are: crushed bricks (Cb) and sand (S). Crushed bricks and sand of encaustic samples were passed through 2.5 mm sieve.

The 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 three days in the medium of (95 ±5) % relative humidity for moisture evenly spreading in the mix. After three days the laboratory samples were shaped into the dimensions (70 × 70 × 70) mm.

The formed prefabs were dried at first under natural conditions in a laboratory, later they were dried in the electric stove with temperature (105 ±5) °C. The dried samples were burned out in a laboratory stove. The sample burned temperature 1000 °C and exposure to the highest burning temperature was 1 h.

The formation mixes are presented in Table 1.

Table 1. Composition of forming mixtures

Mixtures	Formation mixes				
	I	II	III	IV	V
C1	20	18	–	–	–
C2	60	52	–	–	18
C3	–	–	74	92	74
S	12	18	18	5	5
Cb	8	12	8	3	3

Density of the burned ceramic samples was determined according to LST 1272–92 “Ceramics bricks. Specifications”. Water absorption of samples, effective and total open porosity, reserve of pore volume, relative wall thickness of the pores and capillaries, degree of structural inhomogeneity, capillary rate of mass flow were determined according to the methodology presented in [8].

The obtained date of the regression analysis was performed with software “STATISTICA 6.0”.

The frost resistance was directly measured by volumetric and one-side methods of freezing-thawing, as well as by heating according to [13].

3. EXPERIMENTAL RESULTS AND DISCUSSION

Density, water absorption and other structural parameters of ceramic bodies are presented in Table 2.

From the data provided in Table 2, one can see that the density of ceramic products under investigation is ranging from 1900 kg/m^3 to 2010 kg/m^3. The density may be affected by types of clay and content of non-plastic additives in formation mix. The greatest density is received in the V th ceramic body where mixture of clay of two types C2 and C3 is used with small amount of non-plastic materials of 8 %. In this ceramic body, the smallest water absorption, 6.4 %, was found out.

Parameter of effective porosity characterizes the open porosity space of ceramic sample in the aspect of macrostructure and microstructure.

The effective porosity of ceramic bodies ranges from 13.7 % to 23.8 %. One can see the relationship between the water absorption and effective porosity of ceramic bodies. The greater the effective porosity, the more considerable is the water absorption. The same relationship may be observed between water absorption and total open porosity. The greatest water absorption of 10.8 % is

Table 2. Density, water absorption and other structure parameters of ceramic bodies

Formation mixes	Description of the structural parameter and units of measurement							
	Density, kg/m^3	Water absorption, %	Effective porosity, %	Total open porosity, %	Reserve of pore volume, %	Relative wall thickness of the pores and capillaries	Degree of structural inhomogeneity	Capillary rate of mass flow, g/cm^2·h
I	1900	9.0	17.0	24.0	30.0	3.0	1.08	0.14
II	1965	10.0	21.0	26.0	26.0	2.7	0.57	0.19
III	1950	10.8	23.8	25.2	5.7	3.1	2.50	0.30
IV	1960	10.3	22.6	26.1	13.4	2.8	1.56	0.44
V	2010	6.4	13.7	19.7	30.5	4.1	3.79	0.46

measured in the III-rd ceramic body. This body also has the greatest effective porosity, 23.8 %, and a great total open porosity of 25.2 %. We suppose that along with increase of water absorption, the frost resistance should decrease. Therefore, one can predict a small frost resistance of this ceramic body.

The effective porosity of ceramic bodies ranges from 13.7 % to 23.8 %. One can see the relationship between the water absorption and effective porosity of ceramic bodies. The greater the effective porosity, the more considerable is the water absorption. The same relationship may be observed between water absorption and total open porosity. The greatest water absorption of 10.8 % is measured in the III-rd ceramic body. This body also has the greatest effective porosity, 23.8 %, and a great total open porosity of 25.2 %. We suppose that along with increase of water absorption, the frost resistance should decrease. Therefore, one can predict a small frost resistance of this ceramic body.

The water absorption – effective porosity relationship in all ceramic bodies under investigation is illustrated in Fig. 1.

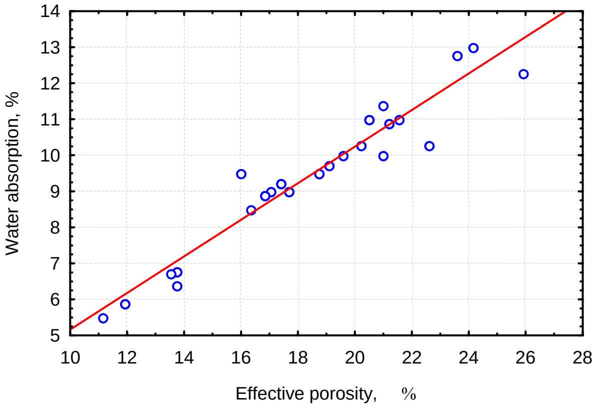

Fig. 1. Ceramic bodies dependence of water absorption on the effective porosity

The dependence may be described by the empirical equation of regression:

$$W = 0.083 + 0.5078 \cdot W_e, \% \quad (1)$$

with the coefficient of determination $R^2 = 0.926$, where W is the water absorption, %; W_e is effective porosity, %.

Parameter of porous volume reserve is an important feature having large influence on frost resistance of the ceramic samples, because it determines the part of ceramic body porous space that is initially unfilled with water, but fills up gradually under cyclic freezing of sample.

The reserve of pore volume determined in the ceramic samples ranges within limits of 5.7 % – 30.5 %. The greatest values of reserve of pore volume are found out in the samples of I st and V th ceramic bodies. These ceramic bodies also have the smallest values of effective porosity. Therefore, supposedly, the frost resistance of these ceramic bodies measured both by one-side and all-side methods will be sufficiently great.

The authors [7, 8, 14, 15] presume that the greater is the reserve of pore volume and relative wall thickness of the pores and capillaries, the more considerable will be the performance frost resistance of products, regardless of values of effective porosity.

The relative wall thickness of the pores and capillaries increases when the used formation mix is well-homogenized and has as low as possible moisture. The biggest values of relative wall thickness of the pores and capillaries are determined for the samples of the V formation mixture. The relationship between relative wall thickness of the pores and capillaries and total porosity in all ceramic bodies under investigation is illustrated in Fig. 2.

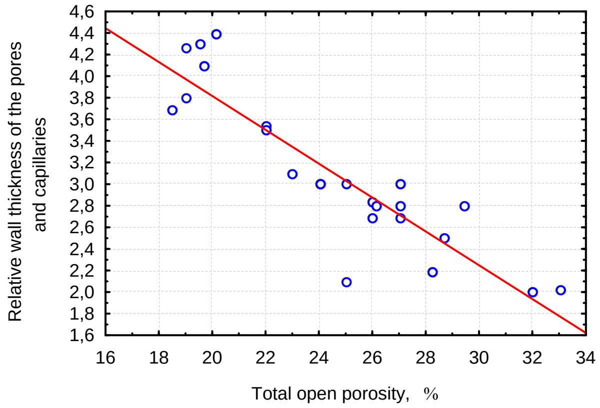

Fig. 2. Ceramic bodies dependence of relative wall thickness of the pores and capillaries on the total open porosity

The dependence may be described by the empirical equation of regression:

$$D = 6.9541 - 0.1569 \cdot W_t \quad (2)$$

with coefficient of determination $R^2 = 0.806$, where D is the relative wall thickness of the pores and capillaries water, W_t is total open porosity, %.

Degree of structural inhomogeneity, allows evaluating the unevenness of effective capillary structure according to their reciprocal length. The relationship between water absorption and degree of structural inhomogeneity of ceramic bodies under investigation is illustrated in Fig. 3.

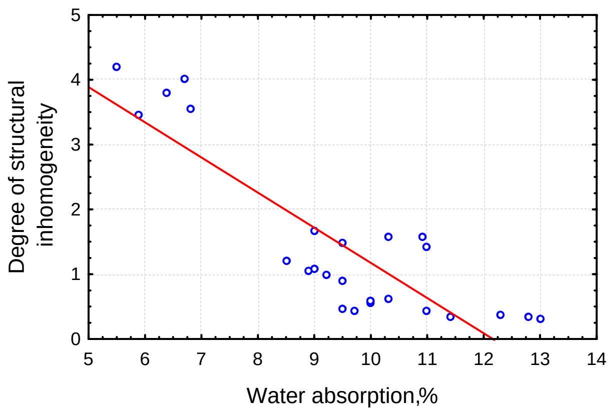

Fig. 3. Ceramic bodies dependence of water absorption on the degree of structural inhomogeneity

The dependence may be described by the empirical equation of regression:

$$N = 6.5964 - 0.5421 \cdot W \quad (3)$$

with coefficient of determination $R^2 = 0.739$, where W is the water absorption, %; N is the degree of structural inhomogeneity.

Capillary rate of mass flow shows the equivalent diameter of these capillaries. When capillaries with small radius are predominant in the ceramic body, the probability of water freezing in it is slight, and obviously the moisture transport will mainly be in the direction of the heat flow. The least values of the capillary rate of mass flow are determined for the samples of the I formation mixture. These ceramic bodies have a small effective porosity, but rather a great relative wall thickness of the pores and capillaries.

The results of frost resistance of the ceramic bodies under investigation received by one-side and all-side methods of freezing-thawing are provided in Fig. 4.

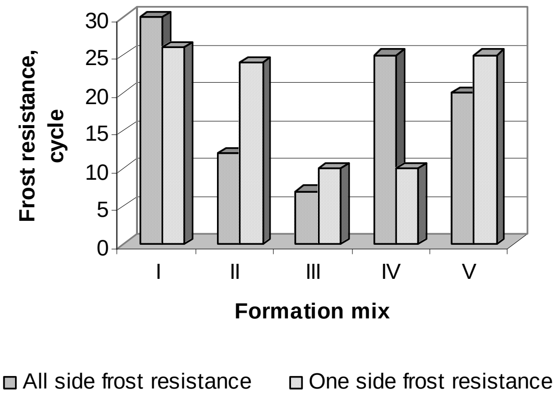

Fig. 4. Frost resistance of ceramic bodies measured by all-side and one-side method of freezing-thawing

Frost resistance by one-side methods presented in Fig. 5.

Fig. 5. Determination of frost resistance by one-side method of freezing-thawing

From the data provided in Fig. 4, one can see that the greatest frost resistance measured both by all-side and one-side method was found out, as predicted, for samples of I-rst formation mix. The frost resistance of this ceramic body measured by all-side method of freezing-thawing amounts to 29 cycles, and that by one-side method to 26 cycles. The effective porosity and capillary rate of mass flow are small and the reserve of pore volume is great.

The rather great frost resistance measured by all-side and one-side method of freezing-thawing was found out also for samples of the V-th formation mix. The density and reserve of pore volume of this ceramic body are the greatest and the water absorption – the smallest.

The frost resistance of III-rd formation mix measured by both all-side and one-side method is the smallest. The result is expected, since the reserve of pore volume of this formation mix is 5.7 % only, water absorption 10.8 % and effective porosity is the greatest, 23.8 %.

It should be mentioned that the character of decomposition in all samples being frozen both by all-side and one-side methods was similar, i. e. splitting into two parts or pieces. In the authors [7] opinion, such decomposition is not typical to performance.

The general tendency of relationship between reserve of pore volume, water absorption and one-side frost resistance is graphically depicted in Fig. 6.

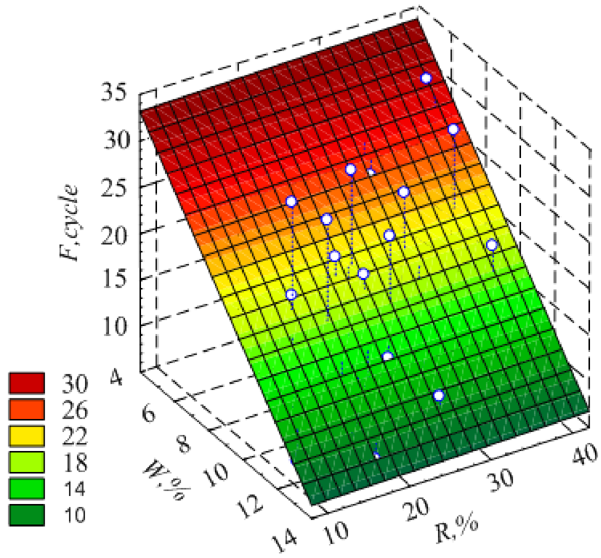

Fig. 6. Tendency of relationship between reserve of pore volume, water absorption and frost resistance determined by one-side method of freezing-thawing

One can see from this diagram that along with increase of reserve of pore volume and decrease of water absorption, the frost resistance, determined by one-side method of freezing-thawing, is increasing. The same tendency in relationship may be observed while investigating the reserve of pore volume, water absorption and frost resistance determined by all-side method of freezing-thawing.

The dependence may be described by the empirical equation of regression:

$$F = 43.8237 - 2.6787 \cdot W + 0.0105 \cdot R \quad (4)$$

were F is one side frost resistance, cycle, W is the water absorption, %; R is the parameter of porous volume reserve, %.

4. CONCLUSIONS

The various parameters of structure of ceramic bodies (water absorption, effective porosity, total open porosity, reserve of pore volume, relative wall thickness of the pores and capillaries, degree of structural inhomogeneity, capillary rate of mass flow) exert influence on frost resistance determined by all-side and one-side method of freezing-thawing.

It was established that along with decrease in water absorption, capillary rate of mass flow, effective and total open porosity and along with increase in reserve of pore volume, relative wall thickness of the pores and capillaries, the frost resistance determined both by all-side and one-side methods should increase.

The relationships between various structural parameters (water absorption and effective porosity, relative wall thickness of the pores and capillaries and total open porosity, degree of structural inhomogeneity and water absorption) provided in the study are described by empirical equations. The values of the multiple determination coefficients describing the interdependence are $R^2 = 0.739 - 0.926$. It was found out that along with increase in effective porosity, the water absorption increases, while along with decrease in total open porosity, the relative wall thickness of the pores and capillaries increase, with decrease in water absorption, the degree of structural inhomogeneity grows.

It was established that the frost resistance is the greatest for those samples of formation mix, which is formed out of 70 % low-melting clays of two types (C1 + C2) and 30 % non-plastic materials (S + Cb) burnt at temperature of 1000 °C and kept at the maximal burning temperature for 1 hour. The frost resistance of samples measured by all-side method of freezing-thawing amounts to 29 cycles, while that measured by one-side method to 26 cycles. In the samples of this formation mix, the frost resistance, measured both by all-side and one-side methods, is predetermined by rather a great reserve of pore volume, 30.0 %, and small effective porosity, 17.0 %, and capillary rate of mass flow, 0.14 %.

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