

## Influence of Assorted Waste on Building Ceramic Properties

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Influence of the combined use of catalytic cracking catalyst and milled glass waste on the properties of building ceramics (density, compressive strength, water absorption, frost resistance) is analysed in the paper. The research showed that under the 1050 °C temperature firing regime, catalyst waste decreases density, compressive strength and frost resistance of ceramic samples, while milled glass increases the values of these parameters. The formation mix without milled glass and with 20 % of catalyst waste had the following values of physical-mechanical properties: density 1590 kg/m<sup>3</sup>, compressive strength 17.5 MPa, frost resistance 195 cycles, and water absorption about 18.0 %. The formation mix without catalyst waste and with 20 % of milled glass had the values of density 1790 kg/m<sup>3</sup>, compressive strength 29.2 MPa, frost resistance more than 300 cycles, and water absorption 12.6 %. Firing samples at 1080 °C temperature, the largest values of density (1980 kg/m<sup>3</sup>), compressive strength (36.1 MPa) and frost resistance (>300 cycles according to LST 1985:2006, unilateral method) were obtained for the formation mix containing the combination of 10 % of catalyst waste and 10 % of milled glass.

*Keywords:* building ceramics, structural, physical, mechanical properties, catalyst and glass waste.

### 1. INTRODUCTION

In recent years, there has been an increasing interest in sustainable development of waste-free manufacture. Different wastes might be used in the production of building ceramics and may even improve the properties of ceramic products.

The waste often applied by researchers is milled glass shivers. The authors [1] have found that the use of glass waste in ceramics enables a sintering process to start at the lowest temperature. Fineness of milled glass has a considerable influence on the formation of liquid phase. While the fineness of glass increases, the temperature of liquid phase formation decreases. Thereby, due to the increased surface area of reaction zone the melting glass reacts with clay more intensively. The maximum degree of glass phase reaction might have influence on the development of new heat resistant materials, it may prolong sintering interval and stabilize the sintered structure [2]. According to the conclusions of these publications, sintering of ceramic body is affected when 10 % of milled glass waste is added to a formation mix. The larger quantity of milled glass in a formation mix decreases the plasticity number of clay, shaping humidity, clay sensitivity and contraction during drying, i. e. glass additive acts as clay thinner. The density of samples with milled glass is higher (approximately 2000 kg/m<sup>3</sup>), compressive strength rises more than twice (about 40 MPa) and water absorption falls to 6 or less percent. The milled glass distributes evenly in a ceramic body, thus decreasing total open porosity very effectively [3–4]. The scientists [5] analysed the influence of three glass

wastes on the properties of ceramic samples. Using these wastes, the sintering temperature was decreased from 1100 °C to 950 °C. The other research [6] investigated the possibilities to use SiC-based waste from polishing glass articles. It has been determined, that with the waste quantities of 5, 10 and 15 % the ceramics both with low density and with especially large porosity can be produced. The scientists [7–8] examined the influence of glass waste (several types), lime and mining residues on the properties of ceramics. It has been determined that using these wastes and firing the samples at a relatively low temperature (880 °C–930 °C) for 1 h–3 h, it is possible to produce ceramic articles with compressive strength of 100 MPa and density of 2600 kg/m<sup>3</sup>. The scientists [9] determined, that highest value of resistance to frost was received for samples that exhibited the highest density, compressive strength, the reserve of pore volume and the lowest water absorption value. These batches were designed based on the largest amount of milled glass.

Large amounts of different catalyst waste come from an oil industry. These catalysts could be milled and used in the manufacture of building products. The wastes from oil industry are designated as potential additives for concrete [10] because they are composed of silicon dioxide and aluminium oxide, and act as pozzolans [11–13]. After the reuse of catalyst waste it has been determined [14], that it can replace 15 %–20 % amount of binding material or 10 % of fine fillers without worsening the qualitative properties of mortar. Due to special chemical composition and convenient characteristics, the catalyst waste may be used in the manufacture of fire resistant [15–16] and ceramic products [17–20]. Moreover, the waste is also suitable as a filler in making asphalt concrete or as a pozzolanic

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**Table 1.** Composition of formation mixes

No of formation mix	Composition of formation mixes, wt. %				Maximum firing temperature, °C
	Clay	Sand	Milled glass (S)	Catalyst (K)	
1	75	5	0	20	1050
					1080
2	75	5	20	0	1050
					1080
3	75	5	10	10	1050
					1080
4	75	5	5	15	1050
					1080
5	75	5	15	5	1050
					1080

**Table 2.** Chemical composition of clay from Ukmerge

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub> +TiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Loss on ignition
66.33 %	15.8 %	6.42 %	1.8 %	1.72 %	1.63 %	5.3 %

component in Portland cement [21]. It has been defined that about 10 % of milled catalyst might be applied to a formation mix; larger amount of the waste (20 %) has negative influence on the physical-mechanical properties of ceramic body, even when firing the ceramic body at the highest firing temperature [22]. It has been determined that these technogenic raw materials may be used to make frost resistant products of sintered building ceramics. These products might be fired at higher temperatures compared to the items made of low-melting clay with typical additives. Therefore, the recommendation is to add up to 10 % catalyst waste [23].

The objective of this paper is to determine the combined influence of glass and catalyst waste on the properties of ceramic body.

## 2. MATERIALS AND ANALYSIS METHODS

Ceramic samples with dimensions (70×70×70) mm were shaped manually. Compositions of formation mixes according to weight are shown in Table 1. The composition of formation mixes was chosen according to performed results of previous experiments and generally used by others researchers [5–7, 14–19].

The samples were made of clay from Ukmerge deposit. The chemical and granulometric compositions of this clay were determined by standard methods (LST EN 725-5:2007, LST EN 1071-4:2002 and others) and are presented in Tables 2 and 3 correspondingly. The clay from Ukmerge was passed through a 0.63 mm sieve.

The standard thinner was applied: sand (0 mm–1 mm) from Anyksčiai deposit. Chemical composition of the sand is shown in Table 4.

The main components of catalyst from catalytic cracking reactor are SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>. The complete chemical composition of catalyst is presented in Table 5. Catalyst particles are spherical, varying in size about from 0.04 μm to 75 μm (Fig. 1).

The size of milled glass particles varying from 0.03 μm to 63 μm (Fig. 3). Figure 4 provides the microstructural view of a glass particle.

**Table 3.** Granulometric composition of clay from Ukmerge

Amount of sandy fraction (>0.05 mm particles), %	Amount of dusty fraction (0.05–0.005 mm particles), %	Amount of clayey fraction (<0.005 mm particles), %
23.93	34.03	42.04

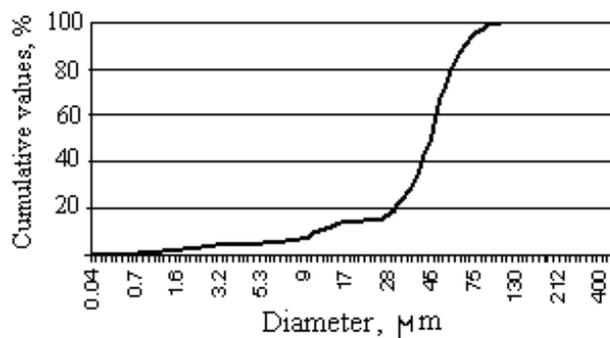
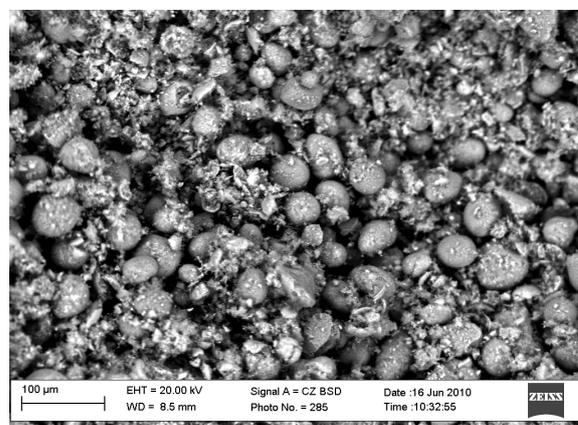
**Fig. 1.** Catalyst particle size distribution

Fig. 2 presents the microstructural view of the particles.

**Fig. 2.** Scanning electron microscopy image of catalyst waste

**Table 4.** Chemical composition of sand from Anyksiai

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	R <sub>2</sub> O	Loss on ignition
88.16 %	3.85 %	0.75 %	3.2 %	0.49 %	1.4 %	2.15 %

**Table 5.** Chemical composition of catalyst

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	La <sub>2</sub> O <sub>3</sub>
55.15 %	40.94 %	0.9 %	1.48 %	0.11 %	1.41 %

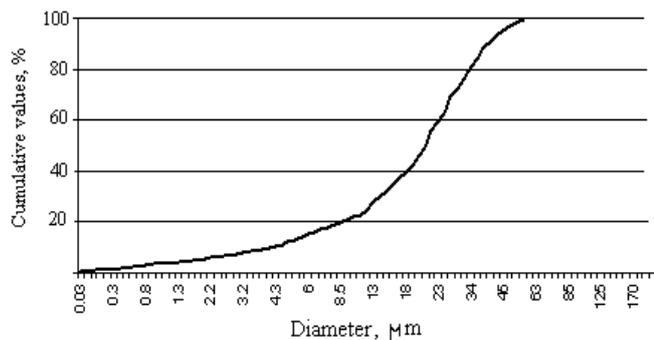
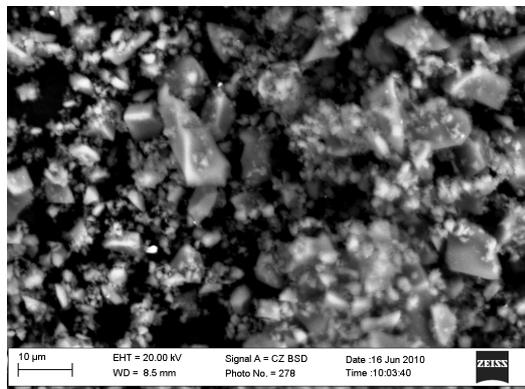
**Fig. 3.** Milled glass particle size distribution

Table 6 shows the chemical composition of milled glass shivers.

**Table 6.** Chemical composition of glass

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	R <sub>2</sub> O
71.18 %	1.64 %	0.21 %	8.21 %	3.26 %	15.5 %

**Fig. 4.** Scanning electron microscopy image of milled glass

The particles of milled glass are very fine and have irregular shape, thus they adhere well with other components in a formation mix.

The samples made of the analysed raw materials were dried to a constant mass; two firing regimes were used. In the first case, the temperature was raised at speed 1.16 °C/min. up to 1050 °C, then the ceramic samples maintained at this temperature 3 h and after that cooled 1.16 °C/min. Otherwise, the temperature was raised at speed 1.19 °C/min. up to 1080 °C, then the samples maintained at this temperature 3 h and cooled 1.19 °C/min.

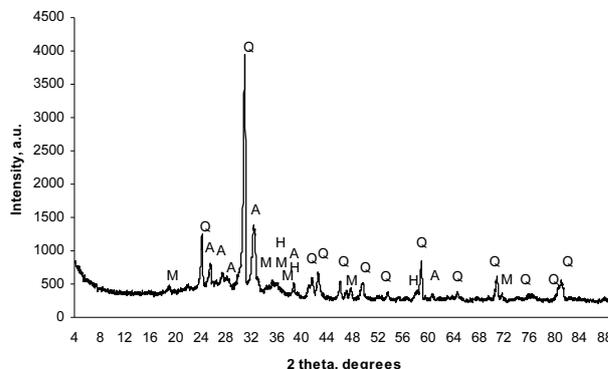
The sintered and cooled samples were used to determine the physical-mechanical and structural parameters. Phase analysis of the powder was performed using XRD. The main instrument used was diffractometer

DRON-7, a cobalt anode was used, (the wave length was  $\lambda = 0.1792$  nm). The diffractograms were decoded according to the characteristic values of distance between planes  $d$  and relative intensity  $I$ ; the values presented in the standard tables [24]. The micro-structural analysis of catalyst and glass waste was performed by a microscope Carl Zeiss Evo LS 25 [25]. The parameters of net dry density and water absorption of ceramic samples were determined in accordance with the standards respectively LST EN 772-13:2003 and LST EN 771-1+A1, compressive strength – LST EN 772-1. The exploitation frost resistance was forecasted according to the methodology of structural parameters [26–27], composed according to LST 1413.12:1998. The parameter of frost resistance was also examined in a laboratory according to the standard LST CEN/TS 772-22:2006.

### 3. EXPERIMENTAL RESULTS AND ANALYSIS

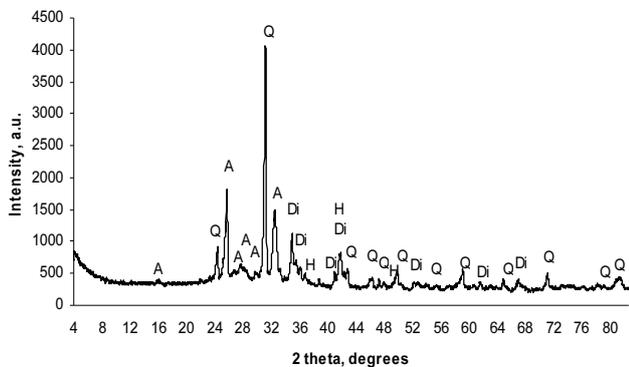
First of all, the XRD analysis of samples was performed. The X-ray diffractograms for the most characteristic batches (containing 20 % of catalyst waste in one case and containing 20 % of milled glass in the other case) are presented in Figures 5 and 6.

Ceramic body with 20 % of catalyst waste contains the following minerals (Fig. 5): A anorthite CaOAl<sub>2</sub>O<sub>3</sub>2SiO<sub>2</sub> (0.472, 0.404, 0.377, 0.368, 0.320, 0.294, 0.252, 0.177) nm, Q quartz SiO<sub>2</sub> (0.426, 0.335, 0.246, 0.228, 0.224, 0.213, 0.198, 0.182, 0.167, 0.154, 0.145, 0.138, 0.137) nm, H haematite Fe<sub>2</sub>O<sub>3</sub> (0.270, 0.251, 0.184, 0.148) nm, M mullite 3Al<sub>2</sub>O<sub>3</sub>2SiO<sub>2</sub> (0.542, 0.270, 0.254, 0.221, 0.153) nm.

**Fig. 5.** XRD pattern of sample containing 20 % of catalyst waste (A – anorthite, Q – quartz, H – haematite, M – mullite)

Ceramic body with 20 % of milled glass additive contains the following minerals (Fig. 6): A anorthite

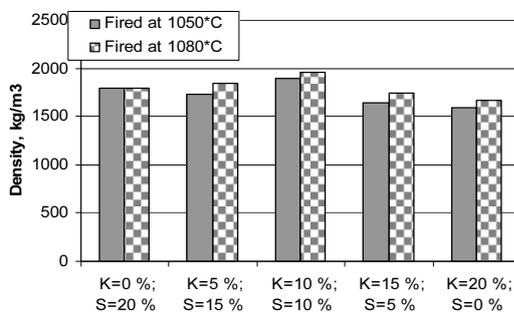
$\text{CaOAl}_2\text{O}_3\text{2SiO}_2$  (0.640, 0.403, 0.386, 0.375, 0.348, 0.319) nm, Q quartz  $\text{SiO}_2$  (0.426, 0.335, 0.246, 0.228, 0.224, 0.213, 0.198, 0.182, 0.167, 0.154, 0.145, 0.138, 0.137) nm, H haematite  $\text{Fe}_2\text{O}_3$  (0.270, 0.251, 0.220) nm, Di diopside  $\text{CaOMgO2SiO}_2$  (0.298, 0.294, 0.289, 0.251, 0.203) nm.



**Fig. 6.** XRD pattern of sample containing 20 % of milled glass

As could be seen from Figures 5 and 6, the main difference of XRD patterns is the different mineral formed: mullite (when catalyst waste is used), and diopside (when glass waste is used). According to the results of authors' research [28–30], diopside, anorthite and mullite are thermodynamically stable minerals that strengthen ceramic body and increase its resistance to frost.

Figure 7 illustrates the dependence of density of ceramic sample on the firing temperature and the amount of waste in a formation mix.



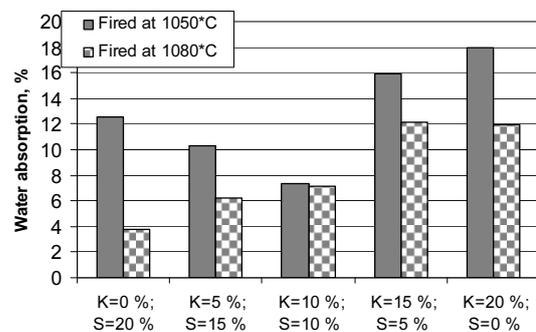
**Fig. 7.** Dependence of density on the firing temperature and quantity of waste in a formation mix

As shown in Figure 7, firing samples at higher temperature leads to higher density. In general, it is possible to state that glass additive increases density, while catalyst waste decreases it. However, the largest density of samples is obtained for formation mixes with 10 % of each waste. This quantity of waste is often considered by researchers to be the optimum [3–4, 21, 23].

The values of water absorption, determined after 72 h, are presented in Figure 8.

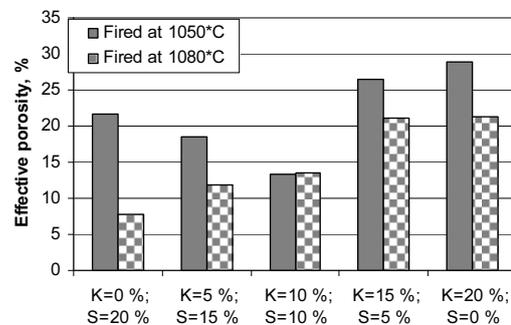
As can be seen from Figure 8, catalyst waste increases water absorption, while glass waste decreases it. The milled glass melts at high temperatures; hard phase sintering takes place based on a liquid-phase. The motive power of this sintering is the surface strain of a melt, which causes negative pressure in a closed pore. Under the negative pressure the pores of ceramic material are filled

with melt and the grains approach each other. The more liquid phase form, the finer is the glass, and the more intensive diffusion process in a sample persists. Due this process, the material grains regroup, the numbers of open pores with irregular shape diminish, the closer, smaller and more regular shape pores appear. As a result, water absorption decreases and density of products rises. The catalyst waste [22–23] contains a significant amount of thinners, which usually increase water absorption and decrease density, as they still do not melt at 1080 °C temperature.



**Fig. 8.** Dependence of water absorption on the firing temperature and amount of waste in a formation mix

The values of porosity are presented in Figure 9. According to the value of effective porosity, the samples are divided into two groups [26]: effective porosity larger than 26 % and smaller than 26 %. According to this division, different equations for the forecasting of frost resistance are selected.



**Fig. 9.** Dependence of effective porosity on firing temperature and amount of waste in a formation mix

Effective porosity is larger than 26 % when catalyst waste in a formation mix constitutes 15 % and 20 %. In other cases the values of effective porosity are remarkably less than 26 %. Effective porosity is significantly smaller for batches with glass additive as it accelerates the sintering process; the samples acquire less open adjoining pores and capillaries.

To forecast the exploitation frost resistance, the structural parameters of ceramic samples were determined. The values of these parameters are presented in Table 7.

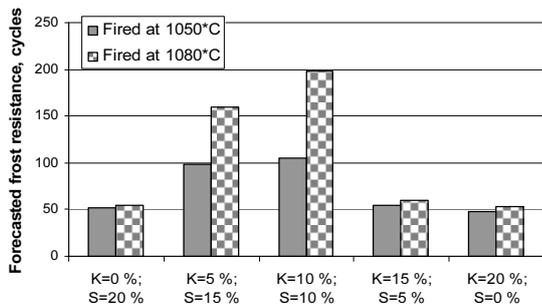
Figure 10 provides values of the forecasted frost resistance depending on the amount of waste in a mix and the firing temperature.

The largest forecasted frost resistance is obtained for the formation mix containing 10 % of each waste. The

**Table 7.** Structural parameters according to batches

Composition, %				Firing temperature, °C	Structural parameters				
Clay	Catalyst waste	Milled glass	Sand		$R_p$ , %	$D$ , %	$G_1$ , g/cm <sup>2</sup>	$g$ , g/cm <sup>2</sup>	$N$ , units
75	20	0	5	1050	23.37	1.65	0.97	0.97	0.88
				1080	33.37	2.40	0.96	0.92	1.24
75	0	20	5	1050	34.75	2.00	0.86	0.65	0.43
				1080	62.51	3.97	0.47	0.44	0.78
75	10	10	5	1050	48.06	2.91	0.37	0.25	0.89
				1080	45.06	3.05	0.29	0.12	1.50
75	15	5	5	1050	23.22	1.90	0.94	0.89	0.20
				1080	37.67	2.37	0.51	0.47	1.23
75	5	15	5	1050	36.75	2.40	0.62	0.50	0.64
				1080	53.10	3.16	0.41	0.30	0.45

Notes:  $R_p$  – reserve of porous volume,  $D$  – relative wall thickness of pores and capillaries,  $G_1$  – rate of capillary mass flow in vacuum in the direction of freezing,  $g$  – rate of capillary mass flow in normal conditions,  $N$  – degree of structural inhomogeneity.



**Fig. 10.** Dependence of forecasted frost resistance on firing temperature and amount of waste in a mix

frost resistance is approximately 70 % larger for samples fired at 1080 °C temperature compared with the samples fired at 1050 °C. Consequently, 1050 °C temperature is too low for new crystal bonds to form.

The frost resistance determined in a laboratory according LST CEN/TS 772-22:2006 is larger than 300 cycles for almost all batches. The freezing was performed up to 300 cycles as this number is sufficient for ceramic products to be used in the especially aggressive environmental conditions [31].

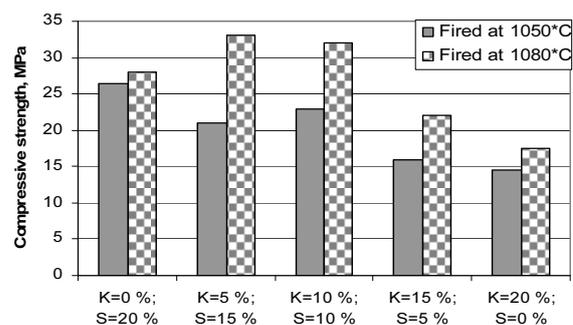
The frost resistance of less than 300 freezing-thawing cycles was obtained for the batches with more than 15 % of catalyst waste. The average frost resistance of samples fired at 1050 °C temperature was 195 cycles (in case when 20 % of catalyst waste was used) and 256 cycles (when 15 % of catalyst waste was used). For the samples fired at 1080 °C temperature, the values of average frost resistance were 283 cycles (20 % of catalyst waste) and 300 cycles (15% of catalyst waste).

The forecasted frost resistance was smaller, because the forecasting methodology was derived from a different standard LST 1413.12:1998. According to this standard, the freezing-thawing cycle takes up to 24 hours, when the testing side of sample 7 times is freezing (to  $-16\text{ °C} \pm 2\text{ °C}$ )

and partially thawing 6 times (to  $0\text{ °C} \pm 4\text{ °C}$ ) and 24 hours on thawed period the testing side of sample is wetting (1–3) minutes then slowly thawed in an enclosed chamber ( $16\text{ h} \pm 2\text{ h}$ ) and then irrigated with water ( $6\text{ h} \pm 2\text{ h}$ ). According to LST CEN/TS 772-22:2006 standard the first period of freezing is 6 h and every further freezing-thawing cycles are 120 min. The thawing period with warm air is 20 min. In the later case, the moistening duration is 120 s.

It might be possible that samples are not saturated to the extent that ice could start to decay the surface; thereby the parameter of frost resistance is significantly larger.

The determined compressive strength of samples is presented in Figure 11.



**Fig. 11.** Dependence of compressive strength on firing temperature and amount of waste in a formation mix

It is apparent from the Figure 11, that glass increases compressive strength of ceramic samples, while catalyst waste decreases it. However, one of the largest values of compressive strength was obtained for the batch with the 10 % of each waste added and fired at 1080 °C temperature.

The samples of this batch also have the highest frost resistance. Taking into account the values of structural parameters, this might be caused by the smallest rate of capillary mass flow and the largest relative wall thickness

of pores and capillaries for this batch. According to the mentioned parameters, it is possible to state that ceramic samples with 10 % of each waste have the smallest diameter of pores and capillaries and the thickest walls.

Thus, in this case a bigger number of fine closed pores could have been formed, which could not be reached by water. In addition, the harder frame (from clay and glass) of a product was received, enabling to bear larger compressive loads.

## CONCLUSIONS

- The performed XRD analysis showed that adding 20 % of catalyst waste to a formation mix leads to a new mineral formed – mullite, while adding 20 % of milled glass forms diopside.
- The results indicate that the additive of milled glass increases frost resistance and compressive strength of ceramic products and decreases water absorption; while the catalyst waste decreases compressive strength, frost resistance and increases water absorption. However, applying 10 % of each catalyst and glass waste in a formation mix leads to the improved compressive strength, density and frost resistance of ceramic products, while water absorption and porosity decrease.
- It has been determined that frost resistant ceramic items might be produced by adding 10 % of catalyst and 10 % of glass waste to a formation mix and these semi-products should be fired at 1080 °C temperature. The ceramics produced has frost resistance of more than 300 freezing-thawing cycles (determined according to LST 1985: 2006). Moreover, ceramic samples of that batch have sufficiently high 31 MPa compressive strength.

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