ANALYSIS OF CLEANED WASTE GLASS AND UTILISATION IN THE NORMAL STRENGTH CONCRETE

Serelis Evaldas*, Vaitkevicius Vitoldas

Kaunas University of Technology, Faculty of Civil Engineering and Architecture, Studentų str. 48, 51367 Kaunas, Lithuania

Abstract

The article investigates the possibilities to utilize waste glass in normal strength concrete. Waste glass was obtained from a local dumpsite. In the research glass shard were prepared in two ways: before and after cleaning process. Before cleaning process waste glass used as it is and contained all impurities. After the cleaning process, most impurities in waste glass were washed out. Waste glass shard was crushed in a ball mill, then analysed by scanning electron microscopy (SEM) and energy-dispersive x-ray spectroscopy (EDX). Effect of waste glass powder before and after cleaning process on Portland cement hydration process was analysed by the semi-adiabatic calorimetry test method. For further research other properties such as density, compressive strength and porosity of normal strength concrete with different amount of waste glass powder also were investigated. In the experiment was found out, that when 25 % of ordinary sand is replaced by waste glass powder after the cleaning process, can be expected slight gain in compressive strength.

Keywords: *waste of glass powder, semi-adiabatic calorimeter, SEM analysis, hydration process analysis*

1. INTRODUCTION

According to Eurostat data (European Statistics Department ec.europa.eu), every year in Europe produced about 38 million tons of glass products (ceramics, porcelain, jars, bottles, etc.). Although glass is one of the few materials, which can be recycled as many times as you want, however not all glass products can be successfully recycled. Most successfully can be recycled such glass waste, which is thrown into specialized containers, the other types of glass are thrown to specialized dumpsites. In the dumpsites, glass can be mixed with other types of waste materials and can form harmful gasses, compounds and pollute groundwater or contribute to the green gas effect. In the dumpsites thrown glass under natural condition will not start to decompose at least for 900 years [1-3]. Even without waiting that long, glass waste in several decades can become a serious problem if it will not be properly solved.

Depending on the contamination degree, chemical composition, fineness, condition of use, etc., the waste glass, which is thrown to dumpsites, can be used in various fields. Currently, the waste glass is used in the following areas: in the road construction [4], in the asphalt pavement [5], in concrete technology as a filler (instead of coarse aggregate, sand or micro-filter) [6,7], in the manufacture of decorative tiles, bricks, wall panels, countertops [8-10], refractory concrete products [11], in the manufacture of artificial filler [12], glass wool [13], fibreglass fibres, also could be used as abrasive material, in landscaping, in the manufacture of reflective road elements, in agriculture as a soil aerator, in ceramics, etc.

One of the biggest disadvantages of waste glass as raw material is, that chemical composition is very volatile and varies very significantly. Due to these reasons, areas, where waste glass can be properly utilised, is very limited. Concrete technology is one type of area, where waste glass, despite its variety of chemical composition, can be properly utilised. In most cases, the waste glass in concrete is used instead of coarse aggregate, sand or micro-filler. Unfortunately, there is no easy way to properly utilise waste glass in concrete, this is because the waste glass in concrete can react in several different ways, and thus there are a lot of disagreements between various scientists. Glass is amorphous material and has a high amount of SiO_2 (~60%), Al_2O_3 (~3%), CaO (~9%), Na_2O (~10%) [14]. Depending on waste glass chemical composition, the amount of active SiO_2, fineness, environmental condition, the amount

of cement, water to cement ratio, temperature and many other factors, waste glass can act in three different ways: 1) can participate as an initiator for alkali-silica reaction during which expansion compound can form and break down the structure of the concrete from the inside [15]; 2) can participate in a pozzolanic reaction during which reacts with portlandite (a product which forms during Portland cement hydration process) and forms low basicity C-S-H, thus contributes to the additional compressive strength of the concrete [16]; 3) can participate as an inert filler, during which deleterious reactions between the waste glass and the cement paste do not occur, or reactions is so slow, that the glass aggregate can be considered as non-reactive.

Despite the facts, most scientists agree, that if the glass will be crushed to a specific surface area (should be similar to surface area of Portland cement), or in the mixture will be used additional (≥ 10 % by the weight of cement) of silica fume, fly ash or blast furnace slag, the deleterious effect of waste glass will be significantly reduced maybe even eliminated [17]. However, waste glass could contain other types of impurities such as aluminium, organic compounds, sulphates, etc., which even in small amounts can cause deleterious volumetric deformations and completely slow down the hydration process of Portland cement. Therefore, it is very difficult to assess why deleterious effect occurred: due to chemical reactions between amorphous SiO_2 and Portland cement or due to chemical reaction between Portland cement and the unwanted impurities, which were introduced together with waste glass. There is no easy answer to those questions, however, experimental research could help to answer one very important question. Can waste glass from dumpsites be used as filler be used in the manufacture process of concrete? Therefore, this research would aim to investigate the suitability of waste glass which obtained from the local dumpsite, if necessarily, clean it and utilise it in ordinary concrete. There are made a lot of different research on how glass powder can affect properties of different types of concrete, but there are much less research about waste glass which was obtained from dumpsites and used in concrete.

2. MATERIALS AND METHODS

2.1. Materials

Cement. Portland cement CEM I 42.5 R was used in the research. The main properties of the material were as follows: specific surface area – 3720 cm^2/kg; the soundness (by Le Chatelier) measures 1.0 mm; the compressive strength (after 2/28 days) was found to be 32.3/63.1 MPa; The mineral composition could be quantitatively expressed: C_3S – 68.70 %; C_2S – 8.70 %; C_3A – 0.20 %; C_4AF – 15.90 %. Chemical composition of Portland cement: SiO_2 – 21.10 %, TiO_2 – 0.22 %, Al_2O_3 – 3.42 %, Fe_2O_3 – 5.23 %, MnO – 0.05 %, MgO – 0.79 %, CaO – 66.40 %, SO_3 – 1.93 %, Na_2O – 0.19 %, K_2O – 0.38 %, P_2O_3 – 0.28 %, Na_2O_{eq} – 0.44 % and loss of ignition – 0.60 %. Particle size distribution can be found in Figure 1.

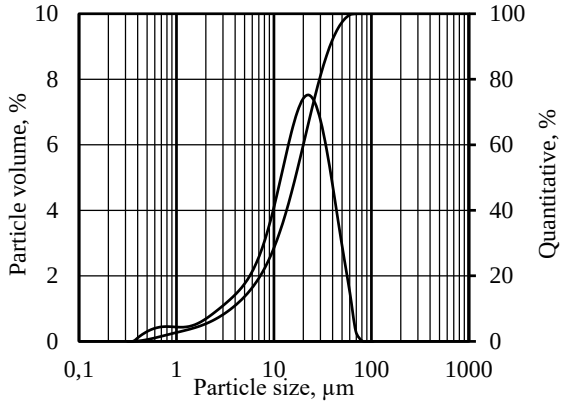

Fig. 1. Particle size distribution of Portland cement CEM I 42.5 R

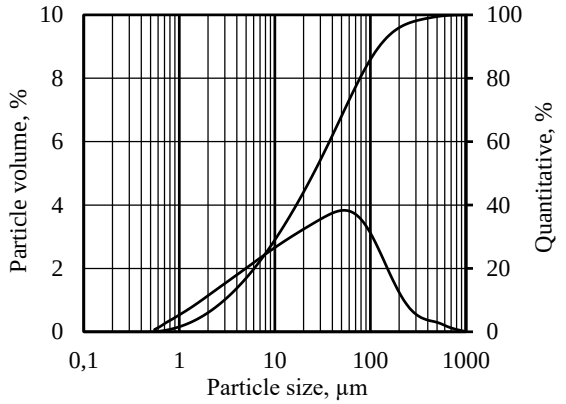

Fig. 2. Particle size distribution of waste glass powder

Glass powder. In the experiment used two types of waste glass shards: 1) waste glass shards before the cleaning process and 2) waste glass shards after the cleaning process. Glass shards were milled in a ball mill, and prepared glass powder used in the experiment. In detail how glass shards were prepared is described in chapter "Methods". Main properties of glass powder before cleaning process: specific density – 2304 kg/m^3, specific surface area – 2380 cm^2/g. Main properties of glass powder after cleaning process: specific density – 2268 kg/m^3; specific surface area – 2316 cm^2/g. Since materials were ground under the same conditions, they have similar properties and similar particle size distribution shown in Figure 2. Elemental analysis is presented in the chapter "Results".

Aluminium powder. In the experiment used aluminium powder (Al). Main properties: assay – 99.5 % trace metal basis; form – powder; auto ignition temp. – 760 ºC; resistivity - 2.6548 μΩ-cm; bp - 2460 °C (lit.); mp - 660.37 °C (lit.); density - 2.7 g/mL at 25 °C (lit.).

Sand. In the experiment 0/2 mm fraction ordinary sand was used. Main properties: specific surface area - 1103 cm^2/g, specific density – 2390 kg/m^3, the average particle size – 691.17 μm.

Gravel. The main properties of gravel used in the research: fraction - 0.063/31.5mm; bulk density - 1582 kg/m^3; Particle density - 2670 kg/m^3; water absorption - 1.39%; resistance to crushing according to the Los Angeles method - 24%. The size distribution of gravel chippings is shown in Figure 3.

NaOH. Sodium hydroxide granules were used in the research, the main properties of which: density - 2.13 g/cm^3; water solubility: 418 g/l (0 ºC); 1110 g/l (20 ° C); 3370 g/l (100 °C); molar mass - 39.9971 g/mol; pure NaOH content ≥99%.

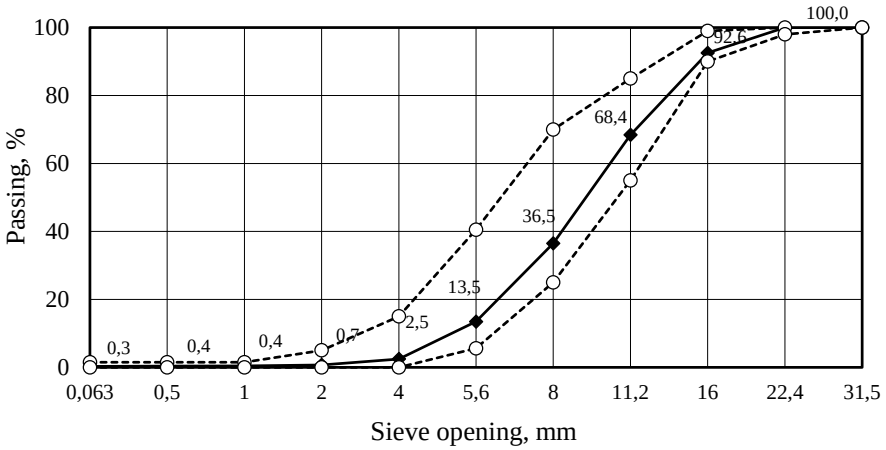

Fig. 3. Particle size distribution of gravel fr. 0.063/31.5

2.2. Methods

Preparation of glass powder. In the experiment were used various glass shards waste, which generated in the local Region Waste Management Centre after compost cleaning. The glass shards waste can contain earth, metal, aluminium, paper, gravel or sand particles and etc. The glass shard waste was prepared in two ways (Figure 4): 1) Untreated glass shard waste was milled in a ball mill and later used for the experiment. Such type of waste is referred to as the waste glass powder before the cleaning process. 2) Glass shard waste was placed on a 1.0 mm sieve and washed under tap water. Waste glass shards waste were washed till colour changed from dark colour till natural glass colour. When the leftovers on the sieve were milled in the ball mill. Such type of glass in the experiment is referred to as the waste glass after the cleaning process. Waste glass shatters before the milling process was additionally crushed with a jaw crusher, and the rest of the glass shatter was milled for 10 minutes in a laboratory ball mill. Main ball mill properties: drum diameter – 70 cm, drum length 52 cm, ball diameter

6.35 cm, the mass of the ball 1.030 kg, the number of balls during milling process – 120 units, per one attempt was milled 3.00 kg of glass shatters.

Specific surface area and particle size distribution. The specific surface area was measured with the Blaine instrument according to EN 196-6:2018 [18]. The particle size distribution was measured with Mastersize 2000 instrument produced by Malvern Instruments Ltd.

Cement hydration analysis. Cement hydration analysis was researched by semi-adiabatic test method. Semi adiabatic test method was performed according to the EN 196-9:2010 [23].

Compressive strength and density. Compressive strength and density were determined after 28 days according to the EN 12390- 4:2019 [19] and EN 12390-7:2019 [20]. Compressive strength and density were calculated from 3 samples (10x10x10 cm cubes) as average value.



Fig. 4. Waste glass powder preparation procedure

SEM analysis. The structures of hardened cement pastes studied with a scanning electron microscope. A high-resolution scanning electron microscope FEI Quanta 200 FEG with a Schottky field emission gun (FEG) was used for the research. Chemical compositions of FCC waste investigated by an energy-dispersive X-ray spectrometer (EDS) with silicon type drift droplet detector.

Porosity of concrete. Open, closed and overall porosity of concrete cubes with and without glass powder calculated according to the ГОСТ 12730.4-2020 [21].

3. RESULTS

3.1. SEM analysis of waste glass

Two types of waste glass powder (before and after the cleaning process) researched by SEM and EDX analysis (Fig.5). From both SEM micrographs can be seen sharp-edged, elongated, various sizes particles, which probably occurred during the milling process. Visually were not detected any additional impurities such as paper, ceramic shards, clay, sand, gravel stones, or etc. Thus, additionally EDX analysis was applied and the main results can be seen in Figure 6.

In the waste glass powder before cleaning process were founded such chemical elements: oxygen (O) – 45.99 %, silicon (Si) – 21.33 %, carbon (C) – 12.24 %, sodium (Na) – 5.24 %, calcium (Ca) – 9.01 %, magnesium (Mg) – 0.98 %, aluminium (Al) – 1.92 %, potassium (K) – 0.74 %, sulphur (S) – 0.71 % and iron (Fe) – 1.85 %. After cleaning process: oxygen (O) – 46.14 %, silicon (Si) – 29.79 %, carbon (C) – 9.17 %, sodium (Na) – 7.85 %, calcium (Ca) – 3.17 %, magnesium (Mg) – 1.44 %, aluminium

(Al) – 0.45 %, potassium (K) – 0.15 %, sulphur (S) – 0.10 % and iron (Fe) – 0 %. After the cleaning process element of iron (Fe) was not detected at all. This element does not have any significant influence on the Portland cement hydration process. The most negative elements, that could be found in waste glass is sulphur (S), which usually can be found in the chemical compound SO_3 and elemental aluminium (Al), which could be found from aluminium caps or foil around the glass bottles. Sulphates slow down Portland cement hydration process speed, while aluminium in presence of alkalis such as sodium or potassium can cause undesirable expansions of Portland cement paste. Other elements such as Ca, Mg, which could be found in chemical compounds CaO, MgO mostly affect cement pastes structures stability. And the main element in glass powder is silicon (Si) usually can be found as the chemical compound SiO_2. There are a lot of debates about the SiO_2 chemical compound, which can be found in glass powder. While one type of researches states that SiO_2 found in glass powder can be good pozzolanic material, the other types of researches states, that glass powder can cause an alkali-silica reaction. Despite the fact, finely grounded glass powder should not have any deleterious effect. One of the main disadvantages of EDX analysis is, that this method cannot identify the form of chemical compound (crystalline or amorphous), cannot identify is the chemical element soluble or insoluble.

In order to find out, has waste glass powder deleterious effect or not, a direct test method was applied. 1M NaOH solution was prepared and mixed with both types of waste glass powder (before and after the cleaning process). Additionally the same solution was mixed with ordinary sand (0/2 mm fraction) and aluminium powder. Results can be found in Figure 7.

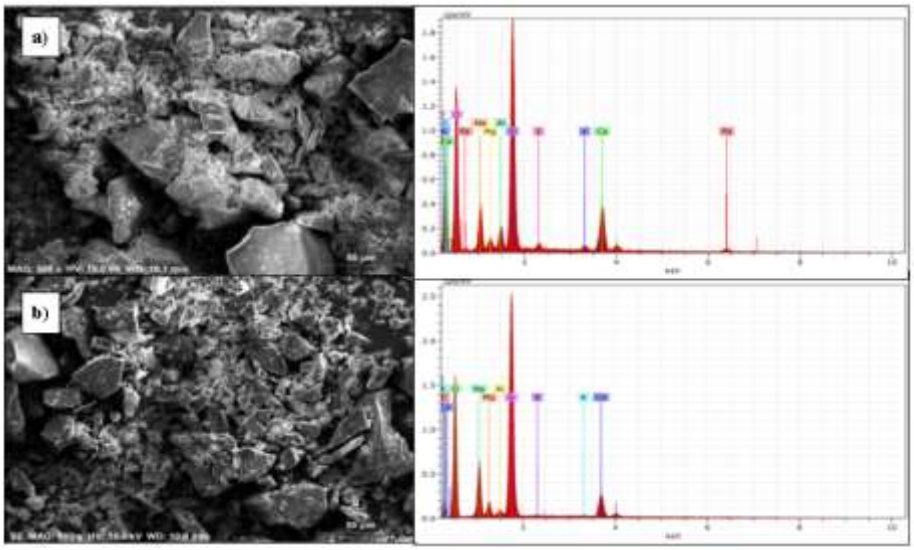

Fig. 5. SEM and elemental analysis micrographs of glass powder: a) before cleaning process and b) after cleaning process

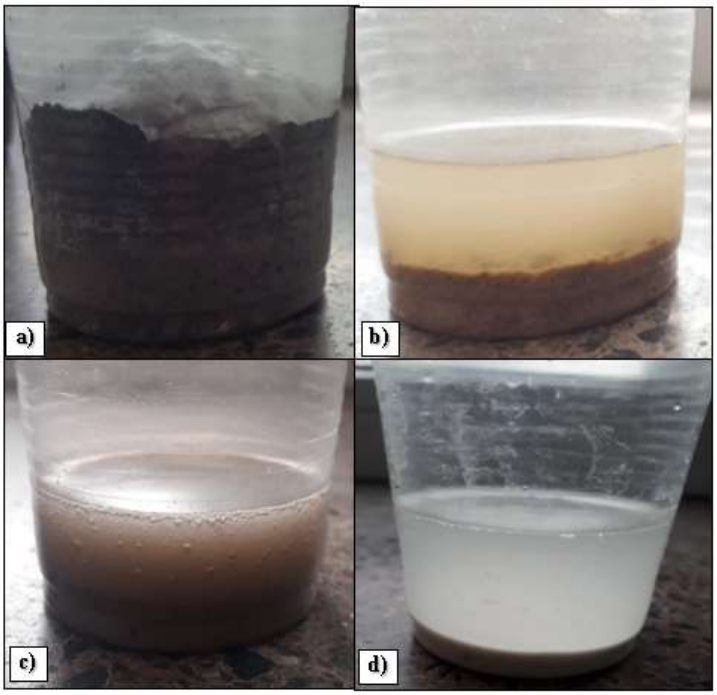

Fig. 7. Effect 1M NaOH solution on: a) aluminium powder, b) ordinary sand, c) waste glass powder before cleaning process, and d) waste glass powder after cleaning process

It was noticed what the reactions between aluminium powder and 1M NaOH solution, caused the mixture to expand (Fig.7-a). When 1M NaOH solution was mixed with ordinary sand (Fig.7-b), no chemical reaction appeared. When 1 M NaOH solution was mixed with waste glass powder before the cleaning process, suddenly bubbles start forming (hydrogen gass) and rise up (Fig.7-c). The reaction was very similar, however less intensive compared to the reaction found in Figure 7.-a. When 1M NaOH solution was mixed with waste glass after the cleaning process no chemical reaction appeared (Fig.7-d). In order to find out, how waste glass powder affects the hydration process semi-adiabatic calorimetry test method was applied.

3.2. Effect of waste glass powder on hydration process

The effect of waste glass powder on the Portland cement hydration process analysed by the semi-adiabatic calorimetry test method. For the experiment prepared nine different binders (Table 1), where a certain percentage of Portland cement was substituted to waste glass powder. Two types of waste glass powder were used: before and after the cleaning process. When the binder is analysed by the semi-adiabatic calorimetry test method, can be distinguished two characteristic points: the minimum/lowest temperature point (in Figure 8, is marked with number 1) at which starts setting process, and from this point binder goes from liquid to solid-state and the highest peak temperature point (in Figure 8, is marked with number 2) at which setting process is finished and binder is finally converted to solid form. In this experiment analysed only the highest temperature peak, which later was compared with other binders.

Composition	Description
C100G0	100 % of Portland cement CEM I 42.5 R
C80G20 - before cleaning process	80 % of Portland cement CEM I 42.5 and 20 % of waste glass before cleaning process
C60G40 - before cleaning process	60 % of Portland cement CEM I 42.5 R and 40 % of waste glass before cleaning process
C20G40 - before cleaning process	20 % of Portland cement CEM I 42.5 R and 40 % of waste glass before cleaning process
C80G20 - after cleaning process	80 % of Portland cement CEM I 42.5 and 20 % of waste glass after cleaning process
C60G40 - after cleaning process	60 % of Portland cement CEM I 42.5 and 40 % of waste glass after cleaning process
C40G60 - after cleaning process	40 % of Portland cement CEM I 42.5 and 60 % of waste glass after cleaning process
C20G80 - after cleaning process	20 % of Portland cement CEM I 42.5 and 80 % of waste glass after cleaning process

Table 1. Binder compositions used for semi-adiabatic calorimetry test method (W/C=0.45)

In the experiment was noticed, that the initial setting time of binder (C100G0) with 100 % of Portland cement CEM I 42.5 R started at 158 min (Figure 8 and point 1), final setting time finished at 577 min (Figure 8 and point 2) and the final temperature rose to 41.88 ºC. When from 20 % up to 80 % of Portland cement were replaced by waste glass powder (before cleaning process) hydration process was prolonged by 14.48 hours and maximum temperature decreased to laboratory's room temperature. According to the results can be stated, that waste glass powder before the cleaning process has a negative effect on hydration process of the Portland cement. EDX analysis revealed, that waste glass powder before the cleaning process has a higher amount of sulphur (S), which usually is found in the chemical compound SO_3. A higher amount of sulphates can significantly slow down the hydration process of Portland cement. When from 20 % up to 80 % of Portland cement were replaced by waste glass powder (after the cleaning process), the hydration process was prolonged only by 31 min, and the maximum temperature decreased from 50.52 ºC down to 35.68 ºC (Figure 9). According to the EDX analysis, even after the cleaning process in waste glass powder were found some trace of sulphur (S). This probably could be explained, why still waste glass powder even after the cleaning process had a minor negative effect on hydration time. Decreased temperature indicates, that waste glass powder does not possess any cementing or pozzolanic properties or they are very insignificant. Glass powder at rooms temperature acts as inert material and the increase of the temperature from the Portland cement hydration process is insufficient for the glass powder to dissolve. At the laboratory's room temperature could take years for glass powder completely to dissolve however if samples after demoulding could be thermally treated above the 60 ºC temperature, melting process and the pozzolanic reaction of glass powder could be significantly increased. Regardless of the obtained results, for further research were used the waste glass powder after the cleaning process.

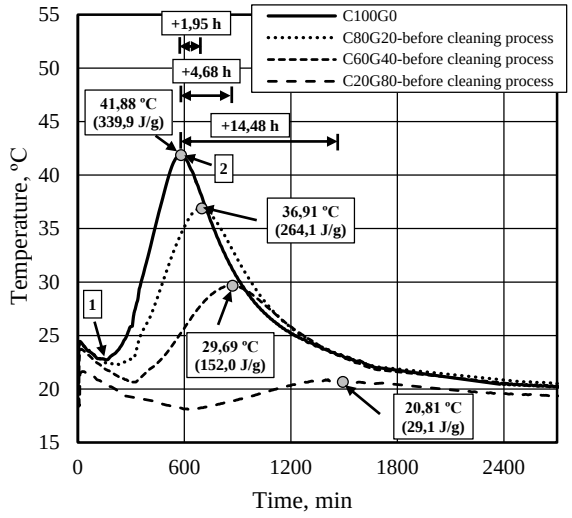

Fig. 8. Effect of contaminated with harmful impurities glass powder on Portland cement (CEM I 42.5 R) hydration process (W/C=0.45)

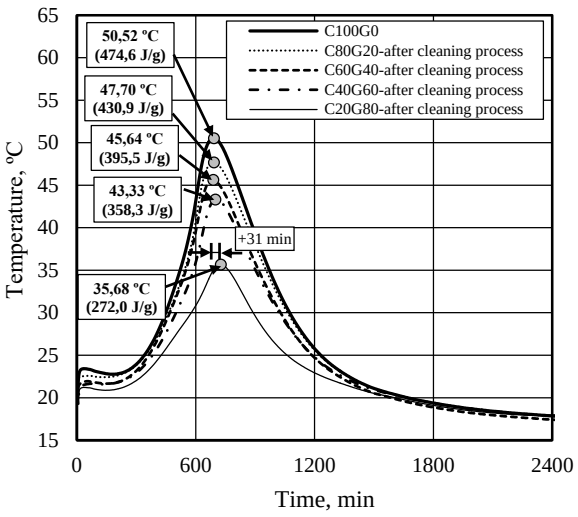

Fig. 9. Effect of cleaned (without harmful impurities) glass powder on Portland cement (CEM I 42.5 R) hydration process (W/C=0.45)

3.3. Effect of cleaned glass powder on density and compressive strength on properties of normal strength concrete

Normal strength concrete mixture prepared with a concrete mixer "Zyklos ZZ50HE". Were prepared four different mixes (Table 2), where some portion of ordinary sand (fr. 0/2 mm) substituted to waste glass powder after the cleaning process, where the amount of Portland cement was kept constant. The main properties of hardened concrete with waste glass powder after the cleaning process could be found in Figures 10 and Figure 11.

Amount of glass powder (from total amount of cement), %	Water, l/m^3	Cement, kg/m^3	W/C	Sand (fr. 0/2 mm), kg/m^3	Gravel (fr. 0.063/31.5)	Waste glass powder, kg/m^3
0	164	340	0,45	907	978	0
25	164	340	0,45	816	978	90,7
37	164	340	0,45	771	978	136,1
74	164	340	0,45	635	978	272,1

Table 2. Composition of normal strength concrete with glass powder

According to the obtained results, could be noticed, that regardless of the amount of waste glass powder in the normal concrete mixture, the densities after 7 and 28 days was very similar (~2300 kg/m^3). However, the compressive strength results strongly depended on the amount of waste glass powder. It was observed, that when ordinary sand (fr. 0/2 mm) was replaced from 0 % up to 74 % early age (after 7 days) compressive strength decreased from 38.6 MPa down to 35.7 MPa. When ordinary sand (fr. 0/2 mm) was substituted from 0 % up to 74 % to waste glass powder, 28 days compressive strength decreased from 45.3 MPa down to 41.1 MPa. However, when ordinary sand (fr.0/2 mm) was substituted from 0 % up to 25% into waste glass powder, the compressive strength slightly increased. According to the results, can be stated, that waste glass powder at the laboratory's room temperature do not have any pozzolanic or cementing properties and in the hydration process participates as an inert micro filler. A

small deviation of compressive strength results probably could be attributed due to the different particle size distribution of the mix. Natural dissolution of waste glass powder is a very slow process that can take up to several years, but with thermal curing of waste glass powder could dissolute much quicker. Despite this fact, the effect of thermal treatment in this research was not pursued.

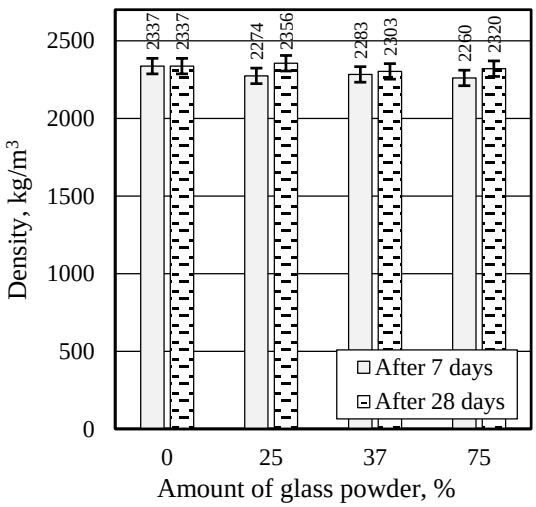

Fig. 10. Effect of glass powder on density of normal strength concrete (W/C=0.45)

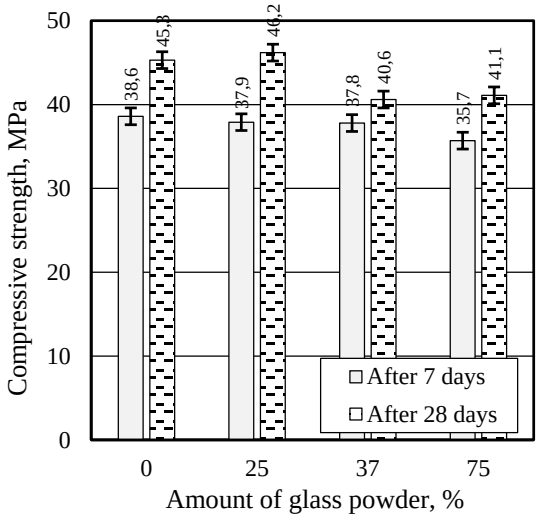

Fig. 11. Effect of glass powder on compressive strength of normal strength concrete (W/C=0.45)

3.4. Microstructure analysis of normal strength concrete with glass powder

The interfacial transition zone between waste glass powder and cement paste was analysed by optical microscope "CETI Steddy-D". For the experiment were used the remaining samples after the compressive strength test method. The main results of microstructure analysis could be found in Figure 12. It was observed, that usually in concrete structure can be found sharply irregularly shaped glass particles, which is probably formed during milling or mixing process. The surfaces of glass particles also could have small micro-cracks, where could form sodium silicate gel. Sodium silicate gel could have two different effects. It can react with moisture, certain alkalis, amorphous SiO_2 and form swelling products which can cause micro-cracks in the concrete. This phenomenon is also known as the alkali-silica reaction. However if glass shards are very finely grounded, glass powder dissolves, forms sodium silicate gel and homogeneously distributes in cement matrix. However this time, sodium silicate works similarly as the hydration accelerator and accelerates the hydration process of Portland cement. This mechanism in more detailed researched by V. Vaitkevicius [22]. Despite the fact, in the interfacial transition zone between waste glass powder and cement paste were not observed positive or deleterious reaction. This confirms the previous assumptions that waste glass powder more likely acts as an inert micro filler and at laboratory's room temperature do not possess any cementing or pozzolanic properties, or those properties are very negligible.

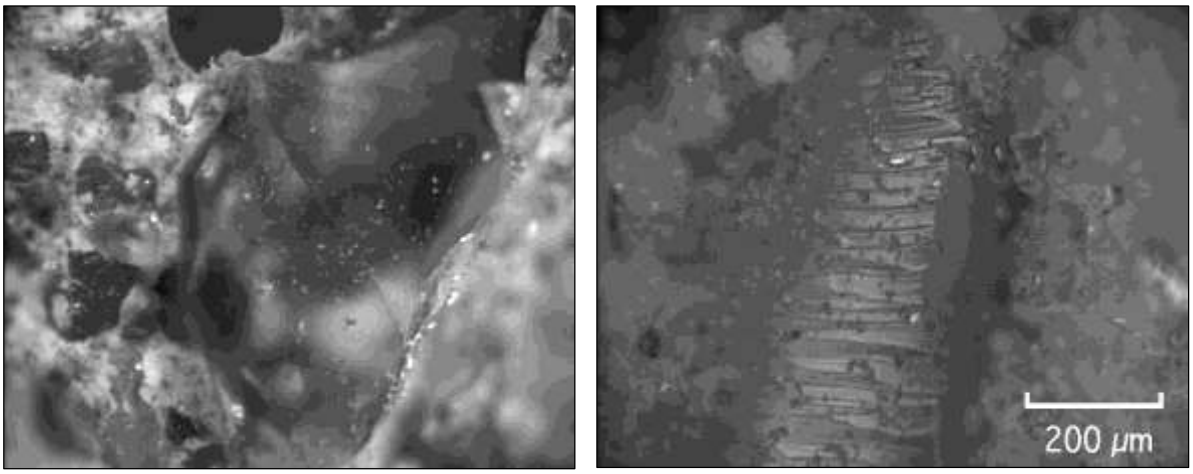

Fig. 12. Interfacial transition zone between waste glass powder and cement paste

3.5. Porosity analysis of normal strength concrete with waste glass powder

Porosity analysis of normal strength concrete with different amount of glass powder was performed according to the ГОСТ 12730.4-2020 [21]. The porosity parameters were determined using the water absorption kinetics method. For the experiment used 10x10x10 cm cubes, which were kept for 28 days in water at 20 ºC. After 28 days, samples were dried for two days at 105 ºC and water absorption kinetics were performed. The main results of the experiment could be found in Figure 13 and the composition of the mixtures with different amount of waste glass powder could be found in Table 2.

According to the obtained results, when ordinary sand (fr. 0/2 mm) was substituted from 0 % up to 75 % into waste glass powder after the cleaning process, the overall and open (capillary) porosity decreased about 2%. Decreased overall porosity has a positive effect on the compressive strength of concrete specimens. Decreased open (capillary) porosity also has a positive effect, because most harmful substances are most likely to enter the deeper layers of concrete through capillary pores. The decrease of overall and open (capillary) porosity can be explained in two ways: 1) the waste glass powder started to dissolve and upon dissolution formed sodium silicate gel, which clogged the capillary pores; 2) The particle size distribution of the concrete mixture improved, whereas well decreased empty gaps between particles and relative density of the mix increased. Despite the fact, decreased capillary porosity has a positive effect on durability properties of normal strength concrete, especially has a higher resistance to frost damage. In general, concrete with close porosity content between 3.5 % and 5 % have sufficiently good resistance to frost damage.

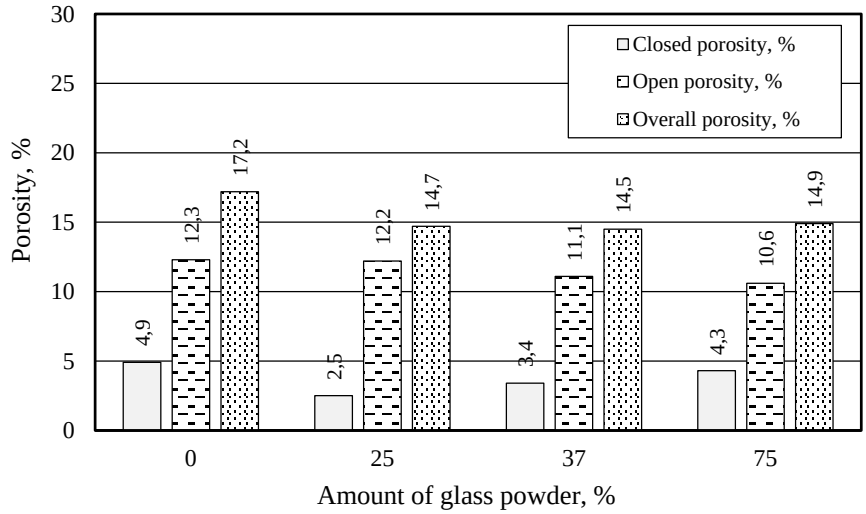

Fig. 13. Effect of glass powder on porosity of normal strength concrete (W/C=0.45)

It is worth mentioning, that the porosity test method probably is more suited for concrete that does not contain any glass powder or other mineral additives. The main disadvantage of ГОСТ 12730.4-2020 method [21] is, that the samples must be dried in an oven at 105 ºC before water absorption procedure. For the normal concrete without glass powder, this drying process does not significantly affect the microstructure. However, when in the concrete mixture is added any type of mineral filler (in our case waste glass powder), the drying process performs a very similar function to thermal curing, in which waste glass powder dissolves and forms sodium silicate gel. The formed sodium silicate gel clogs capillary porosity thus overall and opens porosity decreases. This assumption would explain, why the use of the higher amount of waste glass powder reduced total porosity, but the compressive strength on previous results, despite the used amount of waste glass remained very similar. Therefore, the porosity on uncured concrete with waste glass powder should be very similar to the composition, which does not had waste glass powder at all.

4. CONCLUSIONS

1. After the cleaning process of waste glass powder, the amount of silicon (Si) increased from 21.33 % up to 29.79 %, the amount of sulphur (S) decreased from 0.71 % down to 0.10 %, the amount of aluminium (Al) decreased from 1.92 % down to0.45 %, the amount of iron (Fe) decreased from 1.85 % down to 0,00 %.
2. The negative effect of waste glass on the Portland cement hydration process after the cleaning process was eliminated.
3. When ordinary sand is replaced to waste glass after the cleaning process, the compressive strength remained almost constant. The insignificant gain on the compressive strength can be attributed due to better particle size distribution of normal strength concrete mixture.
4. Research of the normal strength concrete microstructure revealed that waste glass powder after the cleaning process does not have any deleterious effect and acts as inert micro filler.
5. The overall and open porosity of normal strength concrete decreased with an increased amount of waste glass after the cleaning process. Decreased amount of capillary porosity has a positive effect on the durability properties of normal strength concrete.

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