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Advanced mechanical properties and frost damage resistance of ultra-high performance fibre reinforced concrete

Vitoldas Vaitkevičius [a,*], Evaldas Šerelis [a], Danutė Vaičiukynienė [a], Vidas Raudonis [b], Žymantas Rudžionis [a]

[a] Dept. of Building Materials, Kaunas University of Technology, Studentų 48, 51367 Kaunas, Lithuania
[b] Dept. of Automation, Kaunas University of Technology, Studentų 48, 51367 Kaunas, Lithuania

HIGHLIGHTS

- Glass powder decreases porosity of ultra-high performance concrete.
- Glass powder increases hydration process of Portland cement.
- Mixture of ultra-high performance concrete can be prepared with decent technology.
- Addition of micro steel fibres in ultra-high performance concrete do not increase resistance to salt-scaling.

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ABSTRACT

Ultra-high performance concrete (UHPC) mixture with advanced mechanical and durability properties was created using decent mixer. Usually UHPC is made with advanced and sophisticated technology. In experiment UHPC was prepared with high intensity mixer “EIRICH R02”, then mixture was modified and prepared in rotating pan mixer “Zyklos ZZ50HE”. Rotating pan mixer is similar to mixer which has common concrete plants. Experiment results revealed that UHPC with W/C = 0.30 and advanced mechanical and durability properties can be prepared. In experiment tremendous amount of micro steel fibres (up to 147 kg/m^3) were incorporated in UHPC. Concrete with excellent salt scaling resistance and great mechanical properties was obtained. Compressive strength was increased about 30% from 116 MPa to 150 MPa and flexural strength was increased about 5 times from 6.7 to 36.2 MPa. Salt-scaling resistance at 40 cycles in 3% NaCl solution varied from 0.006 kg/m^2 to 0.197 kg/m^2. There were a few attempts to create UHPC and UHPFRC with decent technology, however, unsuccessfully till now. In the world practice this new material is currently used in the construction of bridges and viaducts.

1. Introduction

Recent advantages in concrete and nanotechnology opened new ways to create a strong and long lasting material with advanced properties. There has been made an extensive effort to improve the strength and durability of concrete, however, ordinary type of concrete (30 MPa) has reached certain limits, which nowadays could not meet all necessary requirements for strength, durability, safety, security and low maintenance. Those factors are important for high rise buildings, high span bridges and many other various products made of concrete [1]. Ultra-high performance concrete (UHPC) can easily meet such properties. UHPC according to the EN 206:2013 standard, has compressive strength over 100 MPa [2]. With proper equipment and appropriate composition, compressive strength can be increased up to 250 MPa. Such concrete could easily meet all needed requirements for strength, durability, safety, serviceability and etc.

UHPC, as well as all materials, has its own advantages and disadvantages. The main disadvantages could be: relatively high price; lack of appropriate standards; very brittle failure; challenging mixing process; high autogenous shrinkage; there is a small amount of a long-term research on how it holds up over time in certain conditions. Deeper research is needed to overcome existing problems of current UHPC preparation methods. These methods require rather expensive materials and relatively advanced technology. According to Aldahdooh et al. typical UHPC mixture has 500 kg/m^3–1000 kg/m^3 of Portland cement and up to 250 kg/m^3 of silica fume [3]. Yu et al. tried to improve particle size distribution and reduce Portland cement amount in UHPC. He founded that

* Corresponding author.
E-mail addresses: vitoldas.vaitkevicius@ktu.lt (V. Vaitkevičius), evaldas.serelis@ktu.lt (E. Šerelis), danute.palubinskaite@ktu.lt (D. Vaičiukynienė), vidas.raudonis@ktu.lt (V. Raudonis), zymantas.rudzionis@ktu.lt (Ž. Rudžionis).

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UHPC with W/C = 0.23 and compressive strength up to 160 MPa can be achieved when Portland cement is reduced down to 612 kg/m^3 [4]. Sabet et al. (2013) proposed the idea, that expensive silica fumes could be replaced with natural zeolite or fly ash [5]. Nazari and Riahi founded that ground granulated blast-furnace slag could be used as partial replacement of Portland cement and silica fume [6]. Yoo et al. did a research of how different amount of fibres affects mechanical and fracture properties of UHPC. The results were that incorporating up to 4% (by volume) of steel fibres have positive effect to reduce brittle failure [7]. Wang et al. (2012) in his research offered a simple approach to overcome sophisticated technology to produce UHPC. He proposed a simple method to increase dosage of superplasticizer and to use coarser microfillers [8]. According to proposed idea, UHPC with W/C = 0.18 and compressive strength up to 175 MPa can be prepared. Maruyama and Teramoto did a research and founded that deleterious autogenous shrinkage in ultra-high performance concrete is critical issue, mainly due to pozzolanic reaction. This phenomenon could occur when silica fume consumes water and reacts with portlandite [9]. In research Zhutovsky and Kovler noticed that internal curing has astonishing effect on deleterious autogenous shrinkage [10]. In order to overcome deleterious autogenous shrinkage, Yoo et al. proposed to use shrinkage reducing admixtures or adequate amount of steel fibres [11]. Similar conclusions were proposed by Soliman and Nehdi [12]. Although UHPC has its own disadvantages, various low cost solutions are presented.

The main advantages of ultra-high performance concrete are mechanical strength, durability and workability. Oertel et al. (2014) investigated different types of amorphous silica on hydration in UHPC. During investigation it was noticed, that amorphous silica, such as silica fume, stroeber particles or pyrogenic silica are needed in order to achieve compressive strength over 100 MPa [13]. Yoo et al. investigated effect of micro steel fibres on ultra-high performance concrete and founded that up to 4% (by volume) of micro steel fibres have no significant effect on compressive strength, however, flexural strength can be reached up to 46 MPa [14]. Máca et al. stated that UHPC with up to 2% (by volume) of steel fibre can have workability almost as good as self-compacting concrete, although further increasing of steel fibres has negative effect on workability [15]. Yaz et al. proposed autoclave curing regime. He founded that UHPC without steel fibres can be created with compressive strength up to 270 MPa and flexural strength up to 30 MPa [16]. According to Yi et al. ultra-high performance concrete has up to 8 times greater resistance to accidental impact of blast damage comparing with normal strength concrete (30 MPa) when different amount of steel fibre is incorporated in composition [17]. Teng et al. created various UHPC compositions when W/C ratio varied from 0.28 to 0.35. He founded that even concrete with W/C = 0.35 and Portland cement of 450 kg/m^3 has extremely low chloride diffusivity [18]. Wang et al. investigated UHPFRC durability on progressive aging. Fibres make concrete more porous, however, 74% of its all porosity is smaller than 4 nm which is located in C-S-H [19]. Tayeh et al. stated that UHPC has up to 20 time higher salt-scaling resistance than normal strength concrete [20]. According to literature review UHPC has excellent mechanical and durability properties comparing with ordinary concrete. Most notable characteristics probably are caused by extremely dense microstructure, low porosity ant stronger C-S-H matrix. Although UHPC has outstanding durability, it is important to examine how it was adapted in filed construction. Probably the world's first engineering structure of UHPC was Sherbrooke footbridge (in 1997, USA) [21]. First industrial application was applied in Civaux power plant (France) during 1997 and 1998 when in extremely corrosive cooling towers steel beam were substituted by UHPC beams [22]. Gaertnerplatzbridge Bridge was built in Kassel (Germany) at 2007 [23]. Although, UHPC is relatively new material and a lot of further research is still needed, according to the literature review and worlds practice, ultra-high performance concrete can tolerate very aggressive environment, static or dynamic loads with negligible deterioration.

Extensive literature review has been made, and it is clear, that UHPC has impressive mechanical and durability properties. The main aim of this research is to create UHPC composition which could be prepared with decent mixer and without usage of advanced technology. New composition was prepared with different amount of micro steel fibres. Main properties in experiment were determined by mercury intrusion porosimetry, XRD analysis, compressive strength, flexural strength and salt-scaling. Proposed composition could be used as innovative material for various elements made of concrete which could be prepared with decent mixer.

2. Used materials

Cement. Portland cement CEM I 52.5 R was used in experiment. Main properties: paste of normal consistency – 28.5%; specific surface (by Blaine) – 4840 cm^2/kg; soundness (by Le Chatielier) – 1.0 mm; setting time (initial/final) – 110/210 min; compressive strength (after 2/28 days) – 32.3/63.1 MPa. Mineral composition: C_3S – 68.70; C_2S – 8.70; C_3A – 0.20; C_4AF – 15.90. Particle size distribution is shown in Fig. 1.

Silica fume. Silica fume, also known as microsilica (MS) or condensed silica fume is a by-product of the production of silicon metal or ferrosilicon alloys. Main properties: density – 2532 kg/m^3; bulk density – 400 kg/m^3; pH – 5.3. Particle size distribution is shown in Fig. 1.

Quartz powder. Quartz powder was used in the experiment. Main properties: density 2671 kg/m^3; bulk density – 900 kg/m^3; average particle size – 18.12 μm; specific surface (by Blaine) – 4423 cm^2/g. Particle size distribution is shown in Fig. 1.

Glass powder. Glass powder was used in the experiment. Main properties: density 2528 kg/m^3; average particle size – 25.80 μm; specific surface (by Blaine) – 3350 cm^2/g. Particle size distribution is shown in Fig. 2.

Quartz sand. Quartz sand was used in the experiment. Main properties: fraction: 0/0.5; density 2650 kg/m^3; specific surface (by Blaine) – 91 cm^2/g.

Chemical admixture. Superplasticizer (SP), based on polycarboxylic ether (PCE) polymers, was used in the experiment. Main properties: appearance: dark brown liquid, specific gravity

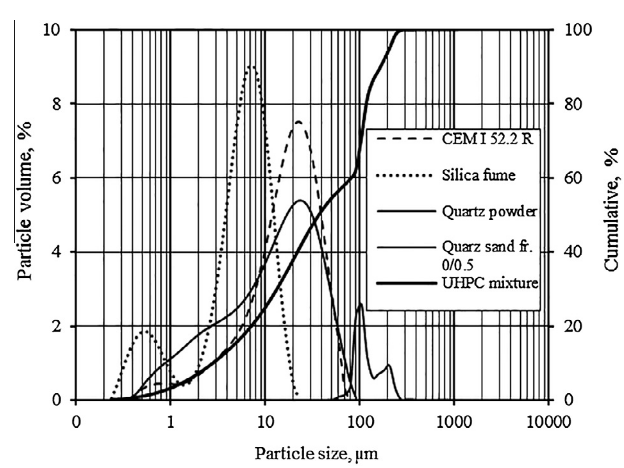

Fig. 1. Particle size distribution of Portland cement, silica fume, quartz powder and 0/0.5 mm fr. quarts sand.

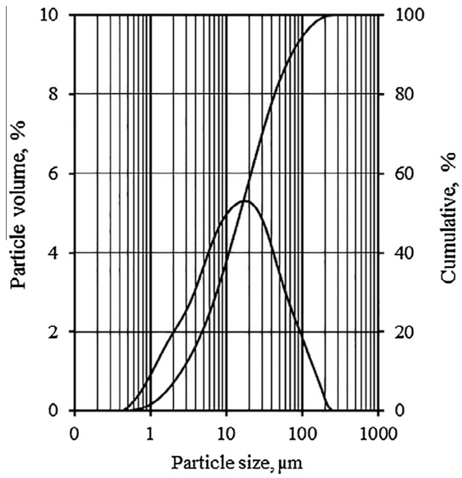

Fig. 2. Particle size distribution of glass powder.

(20 °C) – 1.08 ± 0.02 g/cm^3; pH – 7.0 ± 1; viscosity – 1.28 ± 30 mPa·s; alkali content ⩽5.0%, chloride content ⩽0.1%.

Micro steel fibres. In experiment were used micro steel fibres with hooked ends. Main properties: length – 30 mm, diameter – 0.30 mm; tensile strength – 1000 MPa.

3. Methods

Specific surface and particle size distribution. Specific surface was measured with Blaine instrument according to EN 196-6:2010 standard [24]. Particle size distribution was measured with "Mastersize 2000" instrument produced by Malvern Instruments Ltd.

Mixing, sample preparation and curing. Some fresh concrete mixes were prepared with "EIRICH R02" mixer and some with "Zyklos ZZ50HE" mixer. Main properties of "EIRICH R02" mixer were: capacity – 10 L; mixing pan – worm gear motor for 2 speeds (0.55 and 0.88 kW); rotor – by V-beld and multi-step pulley with frequency converter (0.55/1.10 or 1.4/1.8 kW). Main properties of "Zyklos ZZ50HE" were: capacity – 50 L; high-speed whirler 1500 rpm (1.5/4 kW); power mixing star – 2.2 kW. During this experiment it was noticed how mixing time depends on the mixer type. It took about 8 min of time to prepare UHPC mixture with "EIRICH R02" mixer and about 12 min with "Zyklos ZZ50HE" mixer. Despite the fact, that properties of created mixture or hardened concrete were the same, however the energy consumption differed a lot. Mixtures were prepared from dry aggregates. Cement, aggregates and chemical admixtures were dosed by weight. Quartz powder and silica fume to glass powder were substituted by volume. Cylinders (d = 50 mm, h = 50 mm) were formed for the research to determine concrete properties. Homogeneous mixes were cast in moulds (without compaction) and kept for 28 days at 20 °C/95 RH.

Mercury Intrusion Porosimetry (MIP) test. At age of 28 days cylinder (d = 50 mm, h = 50 mm) of each composition was broken into small fragments ant then placed in isopropanol. Later these fragments were dried in the oven at 40 °C to remove all free water. All dried fragments were stored in sealed containers for mercury porosimetry tests. The pressure was applied from zero to 450 MPa. A constant contact angle of 140° and a constant surface tension of mercury of 480 mN/m were assumed for the pore size calculation.

X-ray diffraction (XRD) analysis. Hardened cement pastes were used for XRD analysis. The XRD measurements were performed with a XRD 3003 TT diffractometer of GE Sensing & Inspection Technologies GmbH with θ-θ configuration und CuKα radiation (λ = 1.54 Å). The angular range was from 5 to 70° 2 Theta with a step width of 0.02° and a measuring time of 6 s/step. For XRD quantitative phase analysis the samples were mixed with 20 wt% ZnO (a standard material widely used in XRD analysis) as an internal standard and stored in argon atmosphere until measurement using the Rietveld refinement. This permits the estimation of the amount of non-crystalline phases by the Rietveld fitting procedure.

Compressive and flexural strength. Compressive and flexural strength were performed after 28 days according to EN 12390-4:2000 and EN 12390-5:2009 standard respectively [25,26]. Compressive strength was obtained from 6 cylinders (d = 50 mm; h = 50 mm) and flexural strength was obtained from 3 prisms (40 × 40 × 160 mm) as an average value.

Salt-scaling. Salt-scaling of concrete was performed according to CEN/TS 12390-9:2006 standard [27]. 3 prisms (7 × 7 × 21 cm) were used for experiment.

4. Results and discussion

The main aim of the research was to prepare UHPC with advanced properties, which could be applied to decent concrete plant. Eight different compositions of UHPC were created in the experiment (Table 1). High intensive mixer "EIRICH R02" was used for compositions QP/GP0 and QP/GP100. QP/GP was used as a reference composition with W/C = 0.25, QP/GP100 with the same W/C ratio when quartz powder was substituted by 100% of glass powder. Up to 147 kg/m^3 of micro steel fibres were used for compositions from QP/GP100-F0 to QP/GP100-F147.0. Mixtures were prepared in rotating pan mixer "Zyklos ZZ50HE". Main properties of microstructure were characterised by mercury intrusion porosimetry and XRD analysis during the research. Main mechanical properties were characterised by flexural and compressive strength. Resistance to frost damage was characterised by salt-scaling test method in 3% NaCl solution.

4.1. Pore size distribution

Experiment results revealed, that glass powder has positive effect on microstructure improvement of UHPC (Figs. 3 and 4).

Table 1
Compositions of ultra-high performance concrete.

Composition	Water, l	Cement, kg/m^3	W/C	Micro filler, kg/m^3			Quarz sand, kg/m^3	SP, l	Micro fibres, kg/m^3
				Silica fume	Quarz powder	Glass powder			
QP/GP0	186	735	0.25	99	412	–	962	36.76	–
QP/GP100					–	412			
QP/GP100-F0	221	735	0.3	99	–	412	962	36.76	0
QP/GP100-F40									40
QP/GP100-F60									60
QP/GP100-F73.5									73.5
QP/GP100-F110.3									110.3
QP/GP100-F147.0									147.0

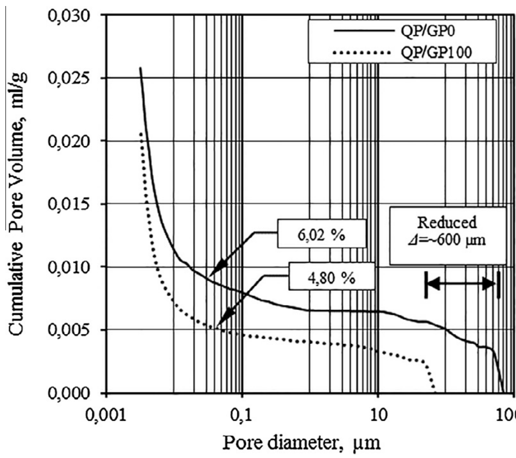

Fig. 3. MIP pore diameter versus cumulative pore volume (W/C = 0.25).

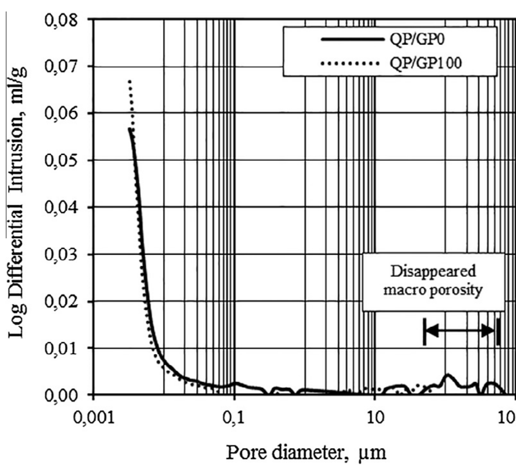

Fig. 4. MIP pore diameter versus log differential intrusion (W/C = 0.25).

Significantly lowered porosity was obtained by composition (QP/GP100) with glass powder. Composition without glass powder (QP/GP0) had continuous pore distribution up to 700 µm, while pore distribution of composition with glass powder (QP/GP100) was smaller than 70 µm. Reduced macro porosity significantly affects mechanical properties of UHPC. Another interesting fact was observed, all compositions had higher concentration of porosity at Nano scale ⩽0.1 µm. These types of pores do not influence mechanical properties, they mostly affect the shrinkage and creep of concrete. XRD analysis was applied in order to find out why composition with glass powder showed lowered pore size distribution.

4.2. XRD analysis

XRD qualitative and quantitative analysis were applied in order to obtain information on phase composition of cement pastes. Fig. 5 illustrates the XRD patterns of two hardened cement pastes with different amount of glass powder. CH phase was found at *d* equalling 0.3042; 0.2789 and 0.1924 nm. Evidently the crystalline phase of CH was decreased with an increase of glass powder. C_2S was found at the following levels of *d*: 0.2790; 0.2783; 0.2745; 0.2645; 0.2610; 0.2189 nm while C_3S phases were found at *d* equalling 0.3036; 0.2773; 0.2748; 0.2604; 0.2181 nm. Decreased C_2S and C_3S peaks are probably related with better solubility of clinker phase. Decreased CH peaks are probably related with the consumption by pozolanic reaction of silica fume and glass powder.

The results of X-ray diffraction measurements were analysed using the Rietveld refinement (Fig. 6). Experiment results revealed that clinker phases with glass powder reacted more intensively comparing to reference mixture (QP/GP0). Glass powder and silica fume reacted with portlandite to form additional C-S-H phases. However, glass powder drastically increased solubility of clinker phases probably due to high amount of alkalis.

The best performance showed composition (QP/GP100) when 100% of quartz powder was substituted by glass powder: $C_2S + C_3S$ decreased about 12% from 45.1% to 33.1%; C_2S decreased about 9% from 34.2% to 25.4%; C_3S decreased about 3% from 10.9% to 7.7%; C_4F decreased about 2.5% from 7.0% to 4.5%; portlandite decreased about 4% from 7.0% to 3.8%. Portlandite makes inhomogeneous microstructure, which has negative effect on compressive strength of UHPC. Another interesting fact was noticed, that cement pastes with glass powder had increased amount of amorphous phase (Fig. 6). Although increased amount of amorphous phase could be attributed to increased amount of C-S-H phases, to this phase also could be attributed unreacted silica fume and glass powder.

4.3. Compressive and flexural strength

Interesting fact was noticed, when glass powder was incorporated in UHPC composition. With substitution up to 100% of quartz

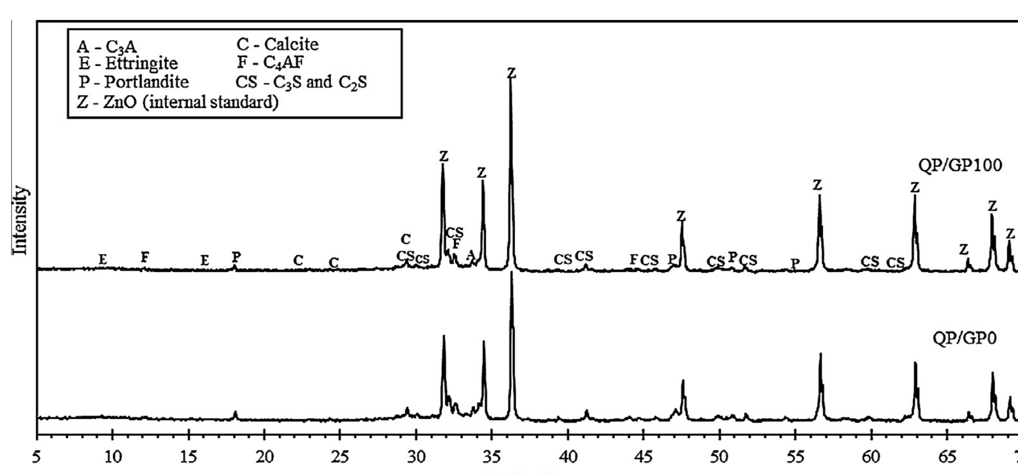

Fig. 5. XRD patterns of hardened cement pastes with different amount of glass powder (W/C = 0.25).

powder by glass powder compressive strength increased about 40 MPa from 182 MPa (GP/GP0) to 221 MPa (GP/GP100), however, flexure strength remained almost the same – 20 MPa (Fig. 7). Such enormous compressive and flexure strength could be obtained in every concrete plant with advanced and sophisticated technology, however, most producers cannot afford such equipment. In order to prepare UHPC with decent mixers, concrete particle size distribution were modified according to Yu et al. [4] and Nguyen et al. [28]. Water to cement ratio was increased up to 0.30. Modified mixture (Table 1) was prepared with different amount of micro steel fibres.

Another interesting fact was noticed (Fig. 8), that with increase up to 147 kg/m^3 of micro steel fibres, compressive strength increased about 30% from 116 MPa (QP/GP100-F0) to 149 MPa (QP/GP100-F147), flexural strength increased more than 5 times from 6.7 MPa (QP/GP100-F0) to 36.2 MPa (QP/GP100-F147). According to the experiment results, micro steel fibres has positive effect on compressive ant flexural strength on UHPC. Despite the fact that optimal amount of micro steel fibres is about 40 kg/m^3, further increase up to 73.5 kg/m^3 has negligible effect on both flexural and compressive strength. Even further increase of micro steel fibres (up to 147 kg/m^3) has negligible effect on compressive strength and has positive effect on flexural strength. However, with extreme amount of steel fibres could be drastically reduced workability and durability of concrete. In order to denote this postulate another test method was applied.

4.4. Salt-scaling

In cold climate zones the main durability problem is insufficient resistance to frost damage. In order to find out how different amount of micro steel fibres affects resistance to frost damage, salt-scaling test methods was applied (Fig. 9). It was noticed that with increase up to 147 kg/m^3 of micro steel fibres after 40 cycles in 3% NaCl solution salt-scaling increased from 0.006 kg/m^2 (QP/GP100-F0) to 0.197 kg/m^2 (QP/GP100-F147). Critical losses of mass

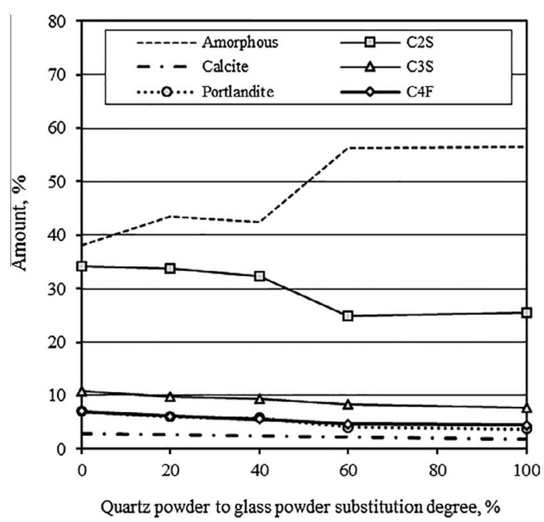

Fig. 6. Mineralogical composition of the binder with different amount of glass powder (W/C = 0.25).

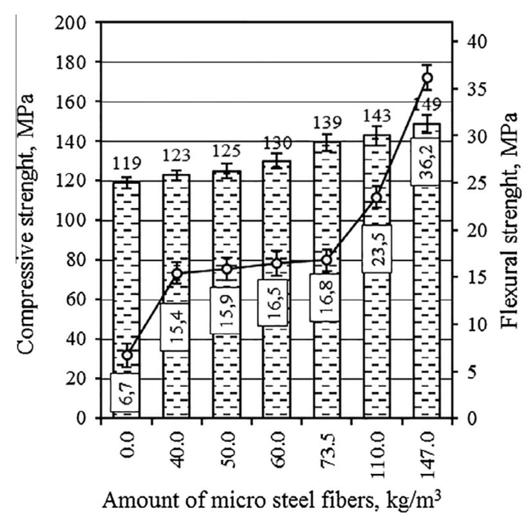

Fig. 8. Compressive and flexural strength of UHPC with different amount of micro steel fibres (W/C = 0.30).

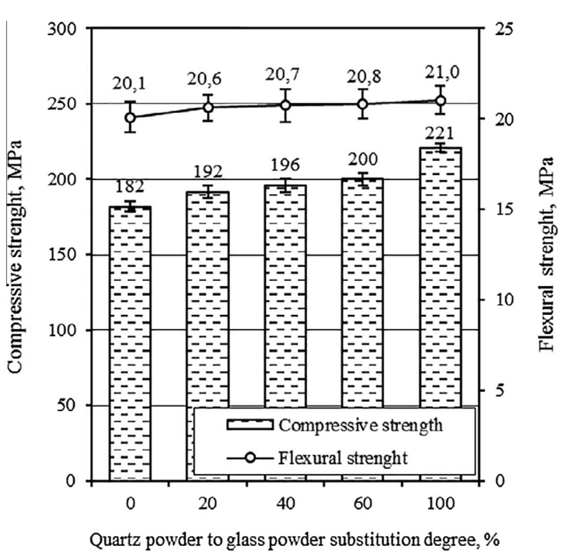

Fig. 7. Compressive and flexural strength of UHPC with different amount of glass powder (W/C = 0.25).

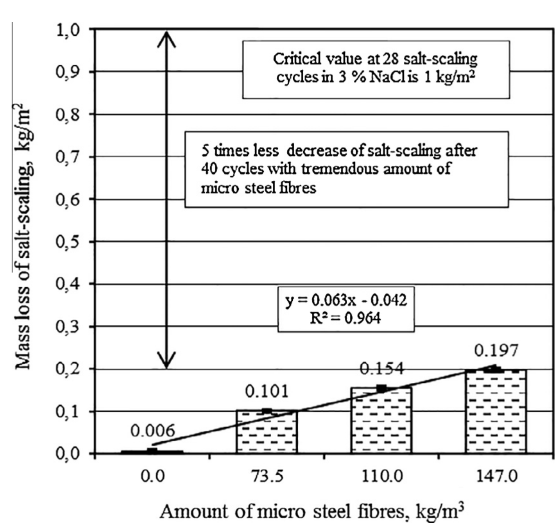

Fig. 9. Salt-scaling of UHPC with different amount of micro steel fibres at 40 cycles (W/C = 0.30).

at 28 cycles are 1 kg/m^2 according to CEN/TS 12390-9:2006 standard [27]. Michta researched how different water to binder ratio affects salt-scaling [29]. On research it was founded, that composition with W/B = 0.38 and compressive strength up to 70 MPa after 56 salt-scaling in 3% NaCl solution is up to 6 kg/m^2. Heede et al. in experiment with different air entraining agents improved normal strength concrete (30 MPa) and after 28 salt-scaling cycles in 3% NaCl solution observed 0.5 kg/m^2 mass loss [30]. Hacha et al. and founded that UHPC after 70 cycles of salt-scaling in 3% NaCl had $\leqslant$0.012 kg/m^2 [31] Similar studies were performed and the authors Vaitkevicius et al. [32,33]. Although micro steel fibres has negative effect on salt-scaling, UHPC still has advanced resistance to frost damage.

5. Conclusions

1. XRD quantitative and qualitative analysis revealed, that glass powder increases Portland cement hydration process. Due to increased hydration process macro porosity of UHPC were eliminated. Largest porosity was observed at micro-scale ($\leqslant$70 μm). All compositions of UHPC had highest concentration of pore at Nano-scale ($\leqslant$0.1 μm
2. During experiment, it was founded that advanced mechanical properties of UHPC with decent mixer can be obtained. Compressive strength can be increased by up to 30% – from 116 MPa (without micro steel fibres) up to 149 MPa (with 147 kg/m^3 micro steel fibres). Flexural strength can be increased more than 5 times – from 6.7 MPa (without micro steel fibres) up to 36.2 MPa (with 147 kg/m^3).
3. After 40 cycles, mass losses of researched compositions of salt-scaling were between 0.006 kg/m^2 to 0.197 kg/m^2. Highest mass losses were noticed in composition with 147 kg/m^3 of micro steel fibres.

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