

KAUNAS UNIVERSITY OF TECHNOLOGY

EVALDAS ŠERELIS

**RESEARCH OF STRUCTURE, PROPERTIES AND TECHNOLOGY OF
ULTRA-HIGH PERFORMANCE CONCRETE**

Summary of doctoral dissertation
Technological Sciences, Civil Engineering (02T)

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INTRODUCTION

Advances in the science of concrete materials have led to the development of a new class of cementitious composites, namely, ultra-high performance concrete (UHPC). The mechanical and durability properties of UHPC make it an ideal candidate for use in various construction elements (Graybeal, 2011). UHPC demonstrates excellent workability with the very low water-to-cement ratio, specifically, $W/C \leq 0.30$. The high compressive strength and workability stems from its excellent homogeneity and packing density (Orange, 1999). However, the packing density and workability are closely interrelated. In order to optimize the mixture with desired characteristics, initially it is important to find the optimal W/C ratio.

According to Ma (2003), the W/C ratio and the workability of self-compacting concrete extremely strongly correlate. A lower W/C ratio reduces the voids between particles and thus the packing density may consequently be increased. The increased packing density maintains a positive effect on the reduced porosity in the cement matrix. A similar conclusion was drawn by Long (2002). He analysed the compactness of binary and ternary compound pastes containing silica fume (SF), pulverized granulated blast furnace slag (PS) and pulverized fly ash (PFA). Long claims that SF is more efficient in improving the packing density of the cement matrix because silica fume exhibits a positive effect on interfiling spaces between larger particles. Sobolev (2004) proposed an innovative method to design and optimize concrete mixture. Experimentally he replaced a part of cement with various mineral additives. According to the research, the workability of UHPC may be modified by incorporating various amounts of a super plasticizer (from 5% up to 10% by the weight of cement) and with different amounts of silica fume (from 5% up to 25% by the weight of cement). However, the particle size distribution also plays an important role. Amen (2011) studied the compressive strength of UHPC development with differing amounts of silica fume. He established that silica fume exerts a positive effect on the early and subsequent compressive strength of concrete. Moghadam (2012) discovered that with the decreased W/C ratio, most mechanical properties will improve and the porosity will subsequently decrease. Similar findings were made by Rahmani (2012) who also observed that with the decreased W/C ratio the increased resistance to abrasion can concurrently be observed.

Microstructural investigations conducted by Reda (1999) with SEM micrographs pinpointed the cause of UHPC being different from the conventional concrete. In fact, it is believed that the mixture containing SF and silica flour (pulverized quartz of a uniform size $<75 \mu\text{m}$) together with the low W/B ratio leads to a very dense and homogeneous microstructure with very low porosity impeding the formation of large crystals of CH (which, in fact, are generally absent in UHPC). It was noticed in some specimens that a relatively

high content of silica fume together with the inclusion of silica flour and the elevated temperature curing regime creates an effective pozzolanic environment which consumes most of the weak CH crystals produced during cement hydration. These crystals were converted into strong C-S-H; hence excellent mechanical properties developed. Alawode (2011) established that in the concrete with a high W/C ratio, CH crystals tend to grow larger if compared with the low(er) W/C ratio mixtures.

The effect of recycled glass powder (GP) on the slump and compressive strength in ultra-high performance fibre reinforced concrete (UHPFRC) was also studied by Kou (2012). In this experiment, cement was partially replaced with glass powder whereas the amount of silica fume was kept constant. The results revealed that glass powder reduced the flow ability of fresh UHPFRC. As a matter of fact, the workability of concrete decreased with an increase in the amount of glass powder. It was also found out that, by replacing cement with GP, the 7-day-compressive tends to decrease while the posterior compressive strength (after 28 days) tends to increase. Similar conclusions were drawn by Khatib (2012). He noticed that the optimal amount of glass powder is when 10% of cement gets replaced with glass powder. Whereas, when the substitution degree exceeds 20%, the compressive strength tends to decrease. Patil (2013) also agrees that the addition of GP increases the strength of concrete. His experiments underline that, in 7 days, glass powder gives less strength, and this is probably due to the low hydration process. Abdelalim (2008) stated that when W/C ratio varies from 0.15 to 0.17, the optimal amount of silica fume ranges between 10% and 15% of the cement mass. However, not all silica reacts chemically with portlandite – some of it actually remains unreacted and fills the empty voids between larger particles. Tavakoli (2013) found that more than 15% of silica fume of the cement mass tends to decrease the workability of concrete.

According to scholarly researches, there is relatively little data suggesting how exactly the W/C ratio influences the mechanical properties of UHPC and how glass powder affects the workability and compressive strength of UHPC. Some authors believe that glass powder may increase the workability and compressive strength of concrete while others disagree with the claim. It is still unclear which amount of glass powder is optimal in a UHPC system. In order to clarify this misconception, four different compositions of ultra-high performance concrete (UHPC) were developed for this study while the W/C ratio varied from 0.25 to 0.33.

The aim of the work

To theoretically and experimentally investigate physical and mechanical properties as well as the overall structure of UHPC, to discover the relationship between properties of concrete and the relevant technological factors and to use that knowledge in order to predict the properties of hardened concrete and design compositions of UHPC mixtures.

Tasks of the work

1. To investigate the effects of silica fume and glass powder on the hardened cement structure and properties.
2. To adapt Brouwer's and Amen's mathematical models so that to predict the properties of hardened cement paste and concrete. To create a UHPC mixture design methodology
3. To research the effect of glass powder on the process of cement hydration.
4. To test the research results under field conditions and to prepare recommendation for UHPC.

Novelty of the research

It was discovered that during cement hydration glass powder acts as a pozzolanic material. The reaction results in the formation of additional low basicity calcium silicate. Glass powder also accelerates hydration of the clinker mineral.

Methods of the research

Cement hydration parameters were investigated with a semi-adiabatic calorimeter. Hardened cement paste was investigated with X-ray and thermographic analyses as well as by employing the ²⁹MAS nuclear magnetic resonance method. The dynamic viscosity of fresh concrete was investigated by applying the falling ball method. A hardened concrete structure was investigated by using the mercury intrusion porosimetry method. Shrinkage of concrete was investigated by measuring its contractions/expansions. Alkali silica reaction data was determined with the accelerated alkali silica reaction method. Resistance to frost damage was investigated by applying surface scaling and freeze-thaw methods.

Practical relevance of the work

A calculation methodology of the composition of UHPC was presented; preparation techniques of glass powder and other dispersive additives were suggested; the mechanism of cement hydration with glass powder was clarified; the price of UHPC by utilizing glass powder instead of silica fume was reduced; UHPC with a compressive strength up to 230 MPa was developed.

Statements to be defended

1. Cement hydration mechanism and kinetics are altered by the glass powder additive.
2. Glass powder acts as a pozzolanic material during cement hydration, which makes additional calcium silicates. These new hydration products increase the compressive strength of the hardened cement paste.

Scientific approbation of the dissertation

11 articles on the subject of the dissertation have been published in scientific journals: 2 articles have been printed in the database “ISI Web of Science” with citation index; 1 article has been issued in the data base “ISI Web of Science” without citation index; 2 articles have been accepted by international databases; 3 articles have been presented in other peer-reviewed scientific journals; 3 articles have been brought out in journals for science popularization.

1. Used materials

Cement. Portland cement CEM I 52.5 R was used in the experiments. The main properties of the employed cement were as follows: the paste of normal consistency makes up 28.5%; specific surface (by Blaine) equals $4840 \text{ cm}^2/\text{kg}$; the soundness (by Le Chatelier) measures 1.0 mm; the setting time (initial/final) is 110/210 min; the compressive strength (after 2/28 days) was found to be 32.3/63.1 MPa. The mineral composition could be quantitatively expressed as: $\text{C}_3\text{S} - 68.70$; $\text{C}_2\text{S} - 8.70$; $\text{C}_3\text{A} - 0.20$; $\text{C}_4\text{AF} - 15.90$. The 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. Its main properties are: density – 2532 kg/m^3 ; bulk density – 400 kg/m^3 ; pH – 5.3. The particle size distribution is also shown in Fig. 1.

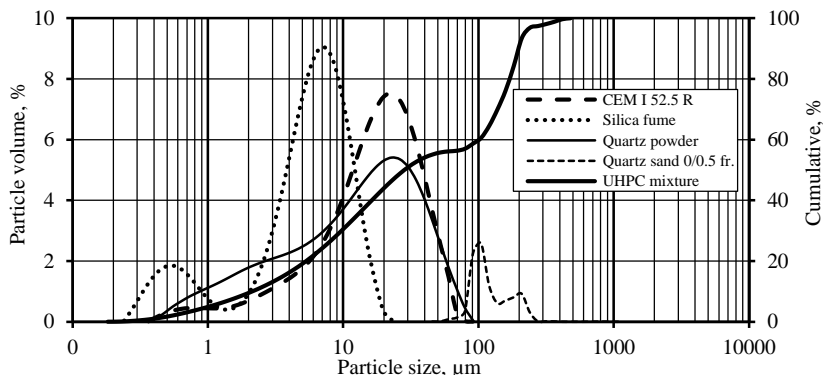


Fig. 1. Particle size distribution of Portland cement, silica fume, quartz powder, 0/0.5 fr. quartz sand and UHPC mixture

Quartz powder. In the experiments, quartz powder was used. The main properties of quartz powder are as follows: the density equals 2671 kg/m^3 ; the bulk density is 900 kg/m^3 ; the average particle size was found to be 18.12 μm ; the specific surface (by Blaine) is $3450 \text{ cm}^2/\text{g}$. The particle size distribution is shown in Fig. 1.

Glass powder. In the experiments, glass powder was used. The main properties of glass powder are: its density is 2528 kg/m^3 ; the average particle size measures 25.80 μm while the specific surface (by Blaine) equals $3350 \text{ cm}^2/\text{g}$.

Quartz sand. In the experiments, quartz sand was used. The main properties of quartz sand are: its fraction equals 0/0.5; its density is recorded as 2650 kg/m^3 ; the specific surface (by Blaine) numerical value is $91 \text{ cm}^2/\text{g}$.

Chemical admixture. In the experiments, superplasticizer (SP) based on polycarboxylic ether (PCE) polymers with the following main properties was used: its appearance was dark brown liquid, its specific gravity (20 C) measured $1.08 \pm 0.02 \text{ g/cm}^3$; the pH value was 7.0 ± 1 ; the viscosity was $128 \pm 30 \text{ Pa}\cdot\text{s}$; it had 65.0% alkali content and 60.1% chloride content .

Micro steel fibres. In the experiment, micro steel fibres were used. The main properties of the fibres were as follows: the length of 13 mm, the diameter of 0.30 mm and the tensile strength of 1000 MPa.

2. Methods

Glass powder preparation. Recycled glass bottles of various colors were used in the experiments. The recycled glass with the splinter size of up 5 cm was crushed with a jaw crusher to an average particle size of 0.5 mm. The crushed glass particles were milled with a vibratory disc mill until a specific surface of $3350 \text{ cm}^2/\text{g}$ was obtained. The main parameters of the vibratory disc mill used in this experiment were the cylindrical container diameter measuring 2171 mm; the thickness of the wall is 5.84 mm while its height is 58.00 mm; the mass of the mill measures 6.246 kg. The materials were ground with 3 smaller rings: the data of the 1st ring is as follows: the diameter of 184.60 mm, the thickness of 14.50 mm, the height measuring 47.55 mm and the mass reaching 2.827 kg; ring 2 had the diameter of 134.05 mm, the thickness of 14.50 mm, the height of 45.50 mm and the mass of 1.878 kg; the diameter of ring 3 was 83.90 mm, its height was 47.05, its mass measured 1.951 kg. The rotation speed is 750–940 rpm.

Specific surface and particle size distribution. The specific surface was measured with the Blaine instrument according to EN 196-6:2010 standard. The particle size distribution was measured with *Mastersize 2000* instrument produced by *Malvern Instruments Ltd.*

Mixing, sample preparation and curing. Fresh concrete mixes were prepared with *EIRICH R02* mixer. The mixtures were prepared from dry aggregates. The cement and aggregates were dosed by weight while water and chemical admixtures were added by volume. Cylinders ($d = 50 \text{ mm}$, $h = 50 \text{ mm}$) were formed for the research in order to determine the properties of concrete. Homogeneous mixes were cast in moulds and stored for 24 h at 20 C/95 RH (without compaction). After 24 h, thermal treatment (1 + 18 + 3) was applied and during the remaining time till the end of the 28 day-day period, the specimens were stored under water at 20 C.

Mercury intrusion porosimetry (MIP) test. After 28 days of testing, cylinders ($d = 50 \text{ mm}$, $h = 50 \text{ mm}$) of each composition were broken into small fragments and placed in isopropanol. Later on, these fragments were dried at 40 C in order to remove all the free water. These dried fragments were stored in sealed containers for MIP tests. The pressure was applied from 0 MPa to 450

MPa. A constant contact angle of 140 degrees and the constant surface tension of mercury of 480 mN/m was 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 *XRD 3003 TT* diffractometer manufactured by *GE Sensing & Inspection Technologies GmbH* with θ - θ configuration und *CuKa* radiation ($\lambda = 1.54 \text{ \AA}$). 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 when using the Rietveld refinement, 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. This allows us to derive the estimation of the amount of non-crystalline phases on the grounds of the Rietveld fitting procedure.

Nuclear magnetic resonance (NMR) analysis. Solid-state NMR was performed with a *Brucker Avance 300* spectrometer (the magnetic field strength of 7.0455 T, the resonance frequency of ^{29}Si is 59.63 MHz). In order to measure the ^{29}Si MAS NMR spectra, the samples were packed in 7 mm zirconia rotors and spun at 5 kHz at an angle of 54°44' (MAS). The chemical shifts were recorded relatively to external tetramethylsilane (TMS). The single pulse technique was applied with a pulse width of 6 μs . Owing to the slow relaxation of the silica fume, a repetition time of 45 s was chosen while the typical number of scans was 2000. Thirty Hertz line broadening was applied to all the spectra prior to deconvolution. The signal patterns of the spectra were deconvoluted with *Bruker WINNMR* software. The interpretation of the ^{29}Si NMR spectra was performed following the paradigm of Schachinger (2008). Because of the high Fe content in the cement, it was necessary to include the first spinning side bands. In order to avoid additional signals, all the mixtures for the NMR investigations were prepared without quartz.

Compressive strength. The compressive strength test was performed after 28 days according to EN 12390-4 Standard. The compressive strength was obtained as the average value of 6 cylinders (d = 50 mm; h = 50 mm).

2. EXPERIMENT RESULTS

2.1 Structure formation and compressive strength development of UHPC

Volumetric ratios of hydration products (ϕ_{hp}), unhydrated cement (ϕ_c) unreacted water (ϕ_w) and chemical shrinkage (ϕ_s) were calculated according to Brouwer's (2005) model (formulas 1-6). The water-to-binder ratio (W/B) was calculated according to formula 1. Here: W stands for the amount of water; C represents the amount of cement; k serves as the activity coefficient of pozzolanic material (k=0.6 for glass powder and k=1.6 for silica fume); x is the amount of pozzolanic material; α shows the hydration degree.

$$\frac{W}{B} = \frac{W}{(C + k \cdot x)}; \quad (1)$$

$$\varphi_{hp} = \frac{(0,639 + 0,013t_c)\alpha}{0,32 + \frac{W}{B}}; \quad (2)$$

$$\varphi_c = \frac{0,320(1-\alpha)}{0,320 + \frac{W}{B}}; \quad (3)$$

$$\varphi_w = \frac{\frac{W}{B} + (0,400 + 0,014t_c)\alpha}{0,320 + \frac{W}{B}}; \quad (4)$$

$$\varphi_s = \frac{(0,081 + 0,001t_c)\alpha}{0,320 + \frac{W}{B}}; \quad (5)$$

$$\varphi_{hp} + \varphi_c + \varphi_w + \varphi_s = 1; \quad (6)$$

According to the Brouwers (2005), model relations between the W/B ratio and the hydration degree were created (Fig.2) and compared to the experiment results (Fig.3). As expected, with the decreased W/B ratio, volumetric ratios of hydration products and the W/B ratio for hydrated cement were presented. The increased volumetric volumes of chemical shrinkage and the required amount of water for the process of hydration were consequently estimated. The economically increased amount of unreacted binder is not desirable. However, at

a lower W/B ratio, the hydrated binder tends to form a greater amount of C-S-H with an increased density. Despite this fact, the model is still incapable of yielding the answer what kind of hydration products, types of C-S-H and portlandite are formed.

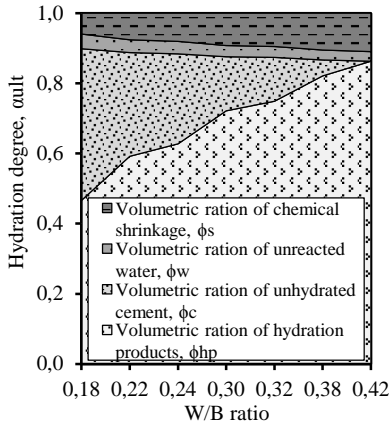


Fig. 2. Influence of W/B ratio and cement hydration degree on the volumetric ration of hydration products (ϕ_{hp}), unhydrated cement (ϕ_c), unreacted water (ϕ_w) and chemical shrinkage (ϕ_s)

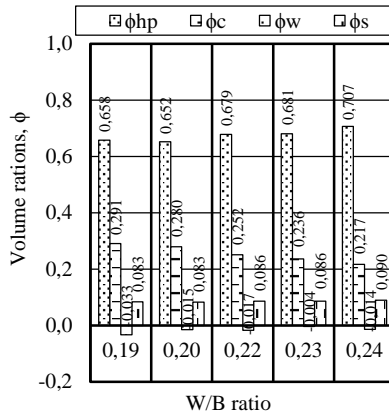


Fig. 3. Influence of W/B ratio on the volumetric ratios of UHPC with silica fume and glass powder (C735+99SiO₂+412MS+5%SP)

Following Amen's (2011) model, the relationship scheme between the hydration degree and the compressive strength at various W/B ratios was created (Fig.4) and consequently compared to the experiment results. The compressive strength was calculated according to Formula 7. Here f_c represents compressive strength, a stands for the coefficient estimating the average compressive strength of concrete (140-230) while b denotes the coefficient estimating the angle of the inclination of the curve (3-4).

$$f_c = a \left(\frac{0,68\alpha}{0,32\alpha + \frac{W}{B}} \right)^b ; \quad (7)$$

Amen's (2011) model is suitable for the prediction of UHPC compressive strength at various W/B ratios; however, the accuracy of the model mainly depends on a and b coefficients. The values of a and b coefficients tend to

increase with the decreasing W/B ratio and with the increasing amount of pozzolanic material. The model is thus suitable for concrete with and without thermal treatment. The disadvantage of the model is that in calculations the influence of porosity is not estimated. However, porosity is indirectly estimated by employing coefficient a (i.e. the average compressive strength of concrete).

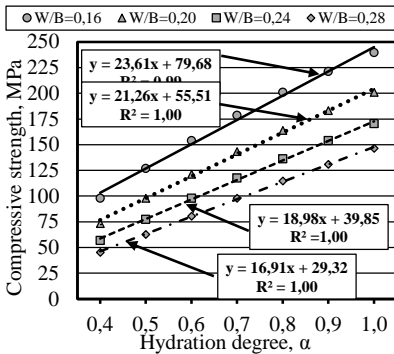


Fig. 4. The influence of W/B ratio and the hydration degree on the compressive strength

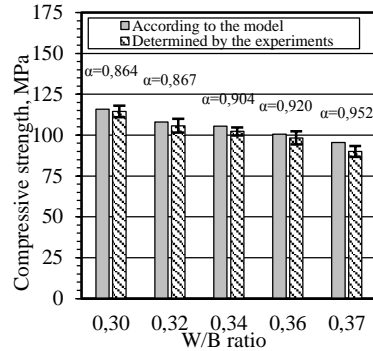


Fig. 5. The influence of W/B ratio and the hydration degree on the compressive strength. The investigated composition of UHPC contained silica fume (C735+99SiO₂+0MS+5%SP)

2.2. Mix design for ultra-high performance concrete

The mix design for ultra-high performance concrete was performed according to Funk (1994) method. The optimal particle size distribution was estimated according to Formula 8. Here d represents the particle size, d_{min} and d_{max} show the minimum and maximum size(s) of the particle while q stands for the particle distribution coefficient.

Table 1. Compositions of Ultra-high performance concrete.

Constituents	Composition			
	QP/GP0	QP/GP100	QP/GP100SF/GP100	SF/GP100
Water, l	186			
Cement, kg/m ³	735			
W/C	0.25			
Silica fume, kg/m ³	99		-	-
Quarz powder, kg/m ³	412	-		412
Glass powder, kg/m ³	-	391	489	99
Quarz sand 0/0.5, kg/m ³	962			
superplasticizer, l	30.65			

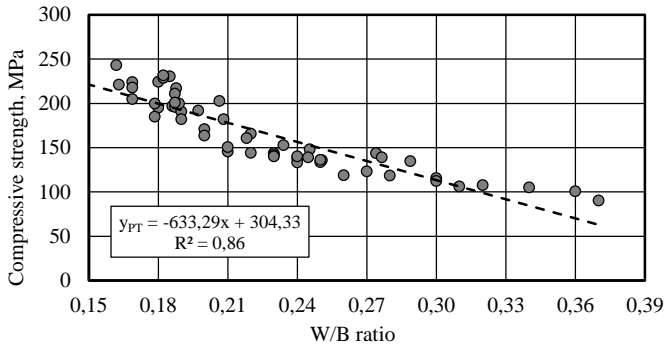


Fig. 6. Influence of the water binder ratio on compressive strength

The relationship between W/B ratio and the compressive strength is shown in Fig. 6. This Relationship of more than 70 points is created. Each point was calculated as the average value of 6 compressed cylinders. The quantities of the employed materials were calculated on the grounds of the absolute volume method and later corrected by using Funk (1994) method. The recommended particle size distribution of the UHPC mix is presented in Fig.7.

$$P(d) = \frac{d^q - d_{\min}^q}{d_{\max}^q - d_{\min}^q} \cdot 100\%; \quad (8)$$

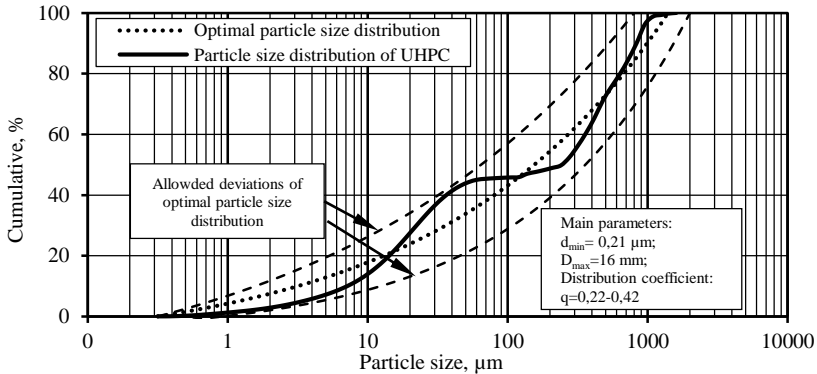


Fig. 7. Recommended particle size distribution for UHPC

2.3. Structure analysis of UHPC

Four compositions of UHPC with different amounts of glass powder were created (Table 1). QP/GP0 serves as the reference composition; QP/GP100 refers

to a case when quartz powder was substituted by 100% glass powder. In QP/GP100SF/GP100, quartz powder and silica fume were substituted by 100% glass powder while in SF/GP100, silica fume was substituted by 100% glass powder.

For scholarly investigation, the following research techniques were applied: mercury intrusion porosimetry (MIP), qualitative and quantitative XRD analysis, NMR and compressive strength tests methods.

2.3.1. Pore size distribution

The compositions with glass powder (QP/GP100 and QP/GP100SF/GP100) exhibited a significantly lower measured porosity and had no macroporosity (Figs. 8 and 9).

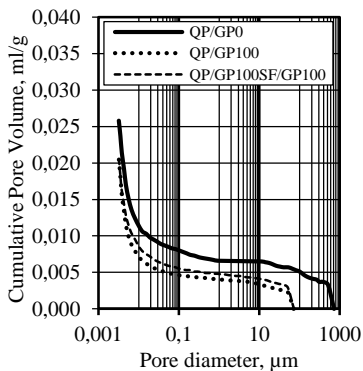


Fig. 8. MIP pore diameter versus the cumulative pore volume

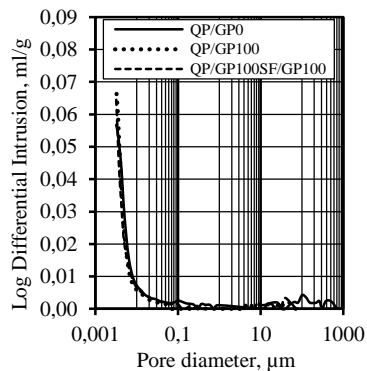


Fig. 9. MIP pore diameter versus the log differential intrusion

The composition without glass powder (QP/GP0) had a continuous pore size distribution up to 700 μm while the compositions with glass powder (QP/GP100 and QP/GP100SF/GP100) measured up to 70 μm . Reduced macroporosity significantly affects the mechanical properties of UHPC. The most significantly reduced porosity was observed in the composition containing silica fume and glass powder (QP/GP100). Another interesting fact was observed that all the compositions had a higher concentration of pores at nanoscale $\leq 0.1 \mu\text{m}$. These types of pores do not influence the mechanical properties but mostly affect the shrinkage and creep of concrete. XRD analysis was applied in order to find out why UHPC with glass powder showed a lowered pore size distribution.

2.3.2. XRD analysis

Qualitative and quantitative XRD analysis was applied in order to obtain information on the phase compositions of the cement pastes. Fig. 10 illustrates the XRD patterns of the four hardened cement pastes with different amounts of glass powder. CH phase was found at d equalling 0.3042; 0.2789 and 0.1924 nm. Evidently the crystalline phase of CH decreased with an increase of the amount 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. It was observed that whenever the amount of glass powder increases, the intensities of C_2S and C_3S tend to decrease. The decreased C_2S and C_3S peaks are probably related with a better solubility of the clinker phase. The pozzolanic reaction of portlandite with silica fume and glass powder probably demonstrated the most prominent influence on the decreased intensities of CH peaks.

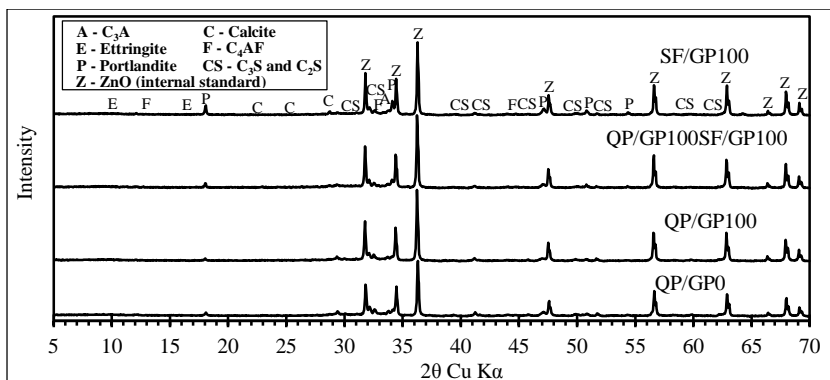


Fig. 10. XRD patterns of hardened cement pastes with different amounts of glass powder

The results of X-ray diffraction measurements were analysed by using the Rietveld method (Table 2 and Fig. 11). The experiment results revealed that the clinker phases containing glass powder reacted more intensively if compared with the reference mixture (QP/GP0). Glass powder and silica fume may react with portlandite so that to form additional C-S-H phases. However, most probably, glass powder drastically increased the solubility of clinker phases due to the high amount of alkalis. The best performance was shown by composition (QP/GP100) when 100% of quartz powder was substituted with glass powder: $C_2S + C_3S$ decreased by about 12 percentage points from 45.1% to 33.1%; C_2S decreased by about 9 percentage points from 34.2% to 25.4; C_3S decreased by about 3 percentage points from 10.9% to 7.7%; C_4F decreased by about 2.5

percentage points from 7.0% to 4.5%; the amount of portlandite decreased by about 3 percentage points from 7.0% to 3.8%. The best clinker solubility performance was obtained with the composition (QP/GP100SF/GP100) when 100% of quartz powder and 100% of silica fume were substituted with glass powder: $C_2S + C_3S$ decreased by about 16 percentage points from 45.1% to 29.18%. Even when 100% of silica fume was substituted with glass powder, the solubility of clinker drastically increased: $C_2S + C_3S$ decreased by about 13 percentage points from 45.1% to 32.3%. However, glass powder is not as effective a pozzolanic material as silica fume is since glass powder leaves high amounts of unreacted portlandite (Table 2).

Table 2. Mineralogical composition of the binder for UHPC with different amounts of glass powder

Phases	QP/GP0		QP/GP100		QP/GP100SF/GP100		SF/GP100	
	wt.%	sd	wt.%	sd	wt.%	sd	wt.%	sd
Amorphous	38.0	3.9	56.4	3.0	58.9	2.8	50.2	2.6
C_2S	34.2	3.9	25.4	3.3	24.1	2.7	27.2	2.5
C_3S	10.9	1.1	7.7	0.8	5.1	0.9	5.1	0.8
C_4AF	7.0	1.1	4.5	0.8	3.9	0.9	5.4	0.8
Calcite	2.8	0.6	2.2	0.4	2.0	0.5	1.6	0.5
Portlandite	7.0	0.6	3.8	0.5	6.1	0.7	10.5	0.7
Total	100.0	-	100.0	-	100.0	-	100.0	-
C_2S+C_3S	45.1	-	33.1	-	29.8	-	32.3	-

Portlandite constitutes a non-homogeneous microstructure exerting a negative effect on the compressive strength of UHPC. Experiment results revealed that glass powder can bind about 5 times less portlandite if compared with silica fume. However, the particle size distribution, the fineness of the glass powder and the pH value of the pore solution also maintain very important roles. Another interesting fact was observed that cement pastes with glass powder showed an increased amount of amorphous phase. Although the increased amount of amorphous phase could be attributed to the increased amount of C–S–H phases, however to this phase unreacted silica fume and glass powder. In order to clarify the experimental data, another method was applied, and a newly obtained sample of data was researched.

2.3.3. ^{29}Si MAS NMR analysis and compressive strength

Peak assignments were made by using the standard Q^n nomenclature. Because of the overlapping signals, the following regime for deconvolution was applied: first, the spectra of the pure powder and the mixture QP/GP0 having less overlap were deconvoluted. The signal of the glass powder could be fitted against the curves contributing to Q^3 and Q^4 at a ratio of about 4:1.

The spectrum of QP/GP0 delivered parameter for the unhydrated cement phases C₃S and C₂S, the C–S–H and the remaining silica fume (Fig. 13). The resulting peak positions and widths were used as fixed values for the deconvolution of the other more overlapping spectra. ²⁹Si MAS NMR spectra of the different mixtures are shown in Fig. 14. The resulting distributions of the silicate phases and the calculated values before hydration are shown in Table 3.

As expected, the silicate phases of the glass powder showed more or less no pozzolanic reaction after that short time of hydration. Nevertheless glass powder increases solubility of Portland cement phases confirming XRD analysis data. The composition when 100% of quartz powder were substituted by glass powder had about 4% increased amount of reacted cement; the composition when 100% of quartz powder and 100% of silica fume were substituted by glass powder had about 23% increased amount of reacted cement; even composition when 100% of silica fume was substituted by glass powder had about 18% increased amount of reacted cement.

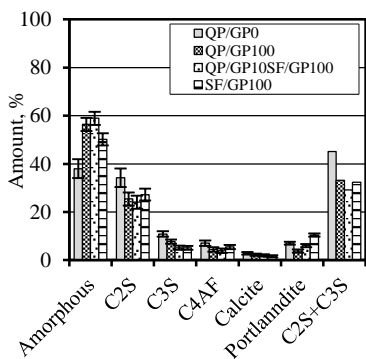


Fig. 11. Mineralogical composition of the binder with different amounts of glass powder

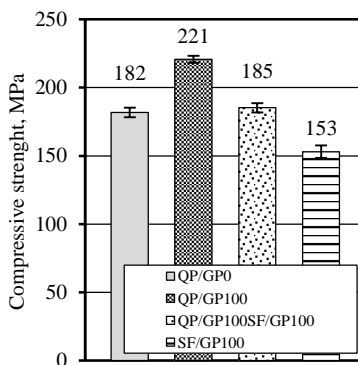


Fig. 12. Compressive strength of UHPC with different amounts of glass powder

The accelerating effect does not correlate with the amount of glass powder. The highest hydration degrees were determined to occur when silica fume was substituted. However, all the compositions had the mean C–S–H chain featuring the length of ~5.00. The highest compressive strength was obtained in composition (QP/GP100) with silica fume where 100% of quartz powder was substituted by glass powder. The compressive strength increased by about 40 MPa from 182 MPa to 221 MPa (Fig. 12). When 100% of quartz powder and 100% of silica fume were substituted with glass powder (QP/GP100SF/GP100), the compressive strength remained almost the same.

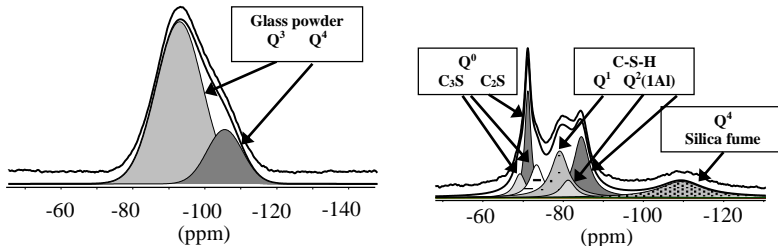


Fig. 13. ²⁹Si MAS NMR spectra with interpretation of the glass powder (left) and QP/GP0 (right).

When 100% of silica fume was substituted with glass powder (SF/GP100), the compressive strength started decreasing and confirmed the assumption that glass powder is not as good a pozzolanic material as silica fume is. When 100% of silica fume was substituted with glass powder (SF/GP100), the compressive strength started decreasing and confirmed the assumption that glass powder is not as good a pozzolanic material as silica fume is.

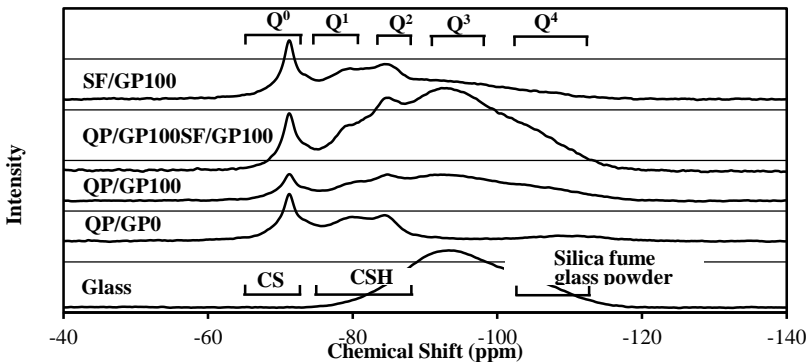


Fig. 14. ²⁹Si MAS NMR spectra of the binder with different amounts of glass powder.

An extensive experiment was carried out in order to devise the optimal composition of UHPC. The research revealed that glass powder can be successfully incorporated into UHPC without any loss of compressive strength. Milled glass can certainly substitute silica fume or quartz powder and even reduce the amount of Portland cement. Thus the cost of UHPC can be drastically reduced.

The ternary system of cement, glass powder and silica fume exerts a positive effect on the maximal compressive strength (QP/GP100). Compressive strength of up to 220 MPa may be achieved. However, the maximal economic benefit can be achieved when glass powder is used instead of silica fume and

quartz powder (QP/GP100SF/GP100). The results of the experiment revealed that all compositions contain enormous amounts of unreacted cement. It may be further reduced, the economic benefit can be increased even further; however additional research is needed to confirm this assumption.

Table 3. Hydration degrees of the silicate phases of admixtures

Composition	Hydration degree, mol% Si			Mean C–S–H chain length
	Cement	Silica fume	Glass powder	
QP/GP0	47	64	-	4.6
QP/GP100	51	66	0	4.4
QP/GP100SF/GP100	70	-	5	5.2
SF/GP100	65	-	0	4.6

2.3.4. Compressive strength, flexure strength and salt scaling

During the research, it was unexpectedly observed that whenever glass powder was incorporated in UHPC composition, with the substitution of up to 100% of quartz powder with glass powder, the compressive strength increased by about 40 MPa from 182 MPa (GP/GP0) to 221 MPa (GP/GP100); however, the flexure strength remained almost the same at the level of about 20 MPa (Fig. 15).

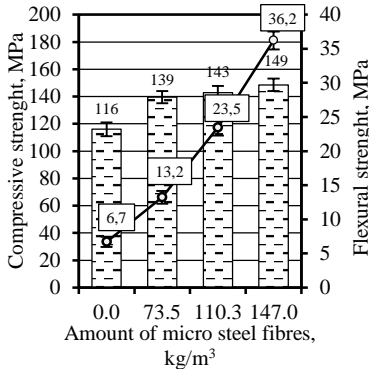


Fig. 15. The compressive and flexural strength of UHPC with various amounts of micro steel fibres (W/C=0.30)

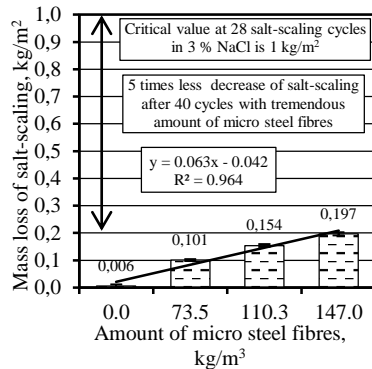


Fig. 16. Salt-scaling of UHPC with various amounts of micro steel fibres at 40 cycles (W/C=0.30)

Such enormous compressive and flexure strength could be obtained in each plant manufacturing concrete as long as it is equipped with advanced and sophisticated technology; however most producers unfortunately cannot afford such equipment. In order to prepare UHPC with standard mixers, the concrete particle size distribution was modified according to Yu et al. (2014) and Nguyen

et al. (2014). The water-to-cement ratio was increased up to 0.30. What is more, the modified mixture was prepared with various amounts of micro steel fibres.

Another interesting fact was observed (Fig. 15) that with the increase of up to 147 kg/m^3 of micro steel fibres, the compressive strength increased by about 30% from 116 MPa (QP/GP0-F0) to 149 MPa (QP/GP0-F147) whereas the flexural strength increased more than 5 times from 6.7 MPa (QP/GP0-F0) to 36.2 MPa (QP/GP0-F147). The experiment results prove that micro steel fibres exert a positive effect on the compressive and flexural strength on UHPC. However, most authors highlight that with excessive amounts of steel fibres, the durability of concrete could drastically decrease. This postulate may potentially reduce the applicability of UHPC. In order to verify this postulate, an alternative test method was applied.

In cold climate zones, the main issue with durability is insufficient resistance to frost damage. In order to find out how various amounts of micro steel fibres affect the resistance against frost damage, salt-scaling test methods were applied (Fig 16). It was demonstrated that with an increase of up to 147 kg/m^3 of micro steel fibres salt-scaling in 3 % NaCl solution at 40 cycles decreased from 0.006 kg/m^2 (QP/GP0-F0) to 0.197 kg/m^2 (QP/GP0-F147). Critical losses of mass at 28 cycles reach 1 kg/m^2 according to CEN/TS 12390-9:2006 Standard. Michta (2013) investigated how different water-to-binder ratios affect salt-scaling. The research yielded the data that a composition with $W/B=0.38$ and the compressive strength of up to 70 MPa after 56 cycles of salt-scaling in 3% NaCl solution reaches up to 6 kg/m^2 . While experimenting with different air entraining agents, Heede et al. (2013) improved the normal strength concrete (30 MPa) and after 28 salt-scaling cycles in 3% NaCl solution, 0.5 kg/m^2 mass loss was observed. Hacha et al. (2010) discovered that UHPC after 70 cycles of salt-scaling in 3% NaCl measured $\leq 0.012 \text{ kg/m}^2$. Similar studies were conducted by Vaitkevičius et al. (2012). Although micro steel fibres negatively affect salt-scaling, however, UHPC still exhibits advanced resistance to frost damage. Pre-prepared compositions could be used for various elements made of concrete with excellent durability and mechanical properties.

2.3.5. Hydration mechanism of cement with glass powder

The gain in the mechanical strength of UHPC appears to be the outcome of the use of glass powder. Glass powder actually serves as a chemical activator rather than a pozzolanic material; however, it still possesses both characteristics. In the early hydration stage, glass powder acts as an inert material. In some cases glass powder can reduce the amount of water for the normal consistency mixture while in other cases it acts in the opposite way. This probably depends on the particle size distribution, on the specific surface or on defects of the glass powder. When the pore solution reaches the value $pH \geq 13.0$, the dissolution rate

of glass powder rapidly increases. The dissolution of glass powder is an endothermic process; thus heat treatment increases the dissolution rate. The dissolution of glass releases a high amount of alkali which acts as a catalyst for the decomposition of Portland cement, silica fume and glass powder. In a high-alkaline environment, glass powder undergoes the alkali-silica reaction. If the particle size of glass powder is small enough, for example, 0.25 mm, the formed alkali silica gel does not exhibit the negative expansion effect and could be treated as sodium silicate also known as water glass. The further process of hydration is influenced by the sodium content and by the silica modulus (M_s). The higher is the amount of Na_2O and M_s the higher hydration level of cement will be consequently obtained. However, the optimal values depend on the type of the concrete, on the water-to-cement ratio, on the parameters of thermal treatment, etc. As long as water glass is homogeneously distributed in all the composition, it dramatically decreases the porosity of UHPC, which is beneficial for mechanical and durability properties. The increased solubility of Portland cement also increases the amount of portlandite. The amorphous silica in glass powder may react with CH and thus form low basicity C-S-H during the later stage of hydration. The alkali will be incorporated into the newly formed hydration phases, mostly in low basicity C-S-H. Thus the microstructure of concrete and its compressive strength could still be significantly increased and UHPC with superior mechanical properties could be prepared.

2.4. UHPC mixture preparation on factory conditions

Adhering to the factory advisory, the UHPC mixture was made in a planetary counter current mixer *HPGM 1125*. The main parameters of the mixer are as follows: the volume is 1125 litres; the effective mixing volume is 750 litres, the engine power is 30 kW and the mass of the mixer is 3800 kg.

The UHPC mix consisted of cement CEM I 52.2R (735 kg/m^3), silica fume (99 kg/m^3), quartz powder (412 kg/m^3), #0.5 mm fraction quartz sand (962 kg/m^3), water (199 kg/m^3), superplasticizer (30.65 kg/m^3) and micro steel fibres (80 kg/m^3). During the experiment, the mixing time was increased from 8 min to 12 min. The prepared mixture exhibited very low viscosity; that is why after pouring the mixture into moulds, additional vibration was not needed. The prepared beams and cubes were left in the laboratory environment with the temperature being set at $20 \text{ }^\circ\text{C}$ for 28 days. After 28 days, the main properties of UHPC were determined: the density level was 2360-2410 kg/m^3 while the compressive strength was 115-120 MPa and the static modulus of elasticity reached 45-48 GPa.

CONCLUSIONS

1. On the grounds of qualitative and quantitative X-ray analysis, thermographic analysis and ^{29}Si MAS nuclear magnetic resonance analysis:

1.1. Silica fume can reduce up to 23% of Portlandite produced by the cement hydration process and increase the compressive strength up to 20%.

1.2. When using glass powder instead of silica fume, the unreacted amount of clinker phase could be reduced from 50% to 25%. As a result, compressive strength additionally increases up to 27%.

1.3. At ambient temperature (20 °C), the pozzolanic reaction of silica fume is about 3 times more intensive in comparison with glass powder. By applying thermal treatment (3+18+5) at 80 °C temperature, the pozzolanic reactivity of glass powder increases. Glass powder also starts to work as a hydration accelerator of clinker.

2. Brouwer's and Amen's mathematical models were adapted to predict the main cement hydration parameters (hydration products, the unreacted amount of cement, the unreacted amount of water and chemical shrinkage) and the compressive strength development.

2.1. The experimentally determined hydration degree of the binder was about 7% higher whereas the amount of unreacted cement was about 7% lower in comparison to mathematical calculations.

2.2. The experimentally determined compressive strength was about 8% lower if compared with mathematical calculations.

2.3. The presented mixture design methodology is suitable for a concrete mixture with a compressive strength of 130-230 MPa.

3. It was discovered that glass powder is more appropriate to be used in UHPC instead of silica fume because glass powder (1) reduces the water demand more efficiently; (2) does not reduce the density of concrete; (3) increases flexural and compressive strength (up to 25 MPa and 230 MPa, accordingly); (4) increases resistance to salt-scaling up to 7 times (after 40 cycles of salt-scaling while without glass powder it measures 0.0249 kg/m² yet with glass powder the numerical value is 0.0034 kg/m²); (5) it only insignificantly affects or has no effect on the final shrinkage of concrete.

4. The designed concreted mixture is suitable for use in field conditions; however, the overall mixing time should be increased. Ultra-high performance concrete created under field conditions showed about 12% lower compressive strength if compared to mathematical calculations

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MOKSLINIŲ PUBLIKACIJŲ DISERTACIJOS TEMA SĄRAŠAS

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Padėka

Norėčiau nuoširdžiai padėkoti mokslinio darbo vadovui prof. dr. Vitoldui Vaitkevičiui už paramą ir vadovavimą doktorantūros studijų metu. Taip pat visam KTU statybinių medžiagų katedros kolektyvui už vertingas pastabas ir pagalbą disertacijos rengimo metu. Dėkoju šeimai už supratingumą ir palaikymą ruošiant disertaciją.

Reziუმэ

Disertacijoje nagrinėjamos ypač stipraus betono (gniuždymo stipris >100 MPa) savybės panaudojant vietines žaliavas. Kaip pucolaninė medžiaga sudėtyje buvo panaudotas malto stiklo priedas ir silicio mikrodulkės. Naudojant vietines žaliavas silicio mikrodulkes ir malto stiklo priedą sukurta nauja sudėtis, su kuria galima pasiekti didesnę nei 200 MPa gniuždymo stiprį betoną. Patobulintoje sudėtyje buvo panaudotos mikro plieninės fibros ir pasiektas 36 MPa lenkimo stipris. Sudėtis buvo patikrinta ir patobulinta Miuncheno technikos universitete. Ypač stipraus betono bandinių struktūra buvo nagrinėta gyvsidabrio porometrijos, XRD kiekybinės ir kokybinės analizės metodu bei magnetinio branduolių rezonanso metodu. Atlikus ilgaamžiškumo tyrimus nebuvo pastebėtas joks žalingas destruktivinis poveikis dėl malto stiklo priedo. Priešingai su malto stiklo priedu pagaminti bandiniai pasižymėjo didesniu gniuždymo stipriu, tankesnę struktūra ir mažesniu poringumu. Atlikus šarminės korozijos tyrimus nebuvo aptikti, jokie reikšmingi struktūriniai pokyčiai. Atlikus tūrinį ir paviršinį šaldymo ir atšaldymo metodą 3 % NaCl druskos tirpale, bandiniai pasižymėjo ypač dideliu atsparumu šalčio poveikiui. Po 200 pagreitintų tūrinio šaldymo ir atšildymo ciklų 3 % NaCl tirpale, kurie atitinka apie 800 ciklų natūraliomis sąlygomis, dalis bandinių prarado 5 % gniuždymo stiprio, po 40 paviršinio šaldymo ir atšildymo ciklų bandinių masės nuostoliai svyravo nuo 0,006 iki 0,012 kg/m². Daugelyje betonų malto stiklo panaudojimas nėra galimas, tačiau sukurtoje sudėtyje, dėl ypač tankios struktūros ir optimizuotos sudėties, maltas stiklas gali pakeisti dalį cemento ir brangius mikro užpildus, kurie leistų ženkliai atpiginti betono savikainą. Sukurta sudėtis puikiai galėtų būti realizuota gaminant tiltų, viadukų, šaligatvių elementus, dėl puikios paviršinės kokybės galėtų būti naudojamas ir išorinių fasadų elementams gaminti.

Darbo uždaviniai

1. Ištirti silicio mikrodulkių ir malto stiklo priedų įtaką cemento akmens struktūrai ir savybėms.
2. Pritaikyti Brouwers ir Amen matematinius modelius struktūros ir stiprumo savybėms prognozuoti bei sukurti ypač stipraus betono mišinio sudėties projektavimo metodiką.
3. Ištirti malto stiklo priedo įtaką portlandcemenčio hidratacijos procesui.
4. Tyrimo rezultatus patikrinti gamybos sąlygomis ir parengti rekomendacijas ypač stipraus betono gamybai

Mokslinis naujumas

Nustatyta, kad maltas stiklo priedas cemento hidratacijos metu dalyvauja pucolaninėje reakcijoje, kurios metu susiformuoja antriniai mažo bazingumo kalcio hidrosilikatai. Šių junginių susidarymas kietėjančioje sistemoje didina cemento akmens gniuždymo stiprį. Taip pat malto stiklo priedas spartina klinkerio mineralų hidrataciją.

Tyrimo metodika

Cemento mineralų hidratacijos parametrai tirti pusiau adiabatinium kalorimetru, cemento akmens struktūros parametrai tirti rentgeno, termografinės analizės ir ^{29}Si branduolių magnetinio rezonanso metodais. Šviežio betono konsistencija nustatyta matuojant mišinio pasklidimą ir dinaminio mišinio klampą. Sukietėjusio betono struktūra tirta gyvsidabrio porometrijos, fluorescenciniu, dinaminio tamprumo modulio ir ultragarsinio tyrimo metodais. Betono susitraukimo deformacijos nustatytos laboratoriniu dilatometru. Betono ilgalaikiškumas patikrintas taikant pagreintą šarminės korozijos, tūrinį ir paviršinį šaldymo ir atšildymo 3 % NaCl tirpale metodus.

Išvados

1. Taikant kokybinę ir kiekybinę rentgenografinę, termografinę, ^{29}Si magnetinio branduolių rezonanso analizę nustatyta, kad:
 - 1.1. silicio mikrodulkės gali sumažinti iki 23 % portlandcemenčio hidratacijos metu išsiskyrusio portlandito kiekį ir padidinti gniuždymo stiprį iki 20 %;
 - 1.2. naudojant silicio mikrodulkes lieka daugiau kaip 50 % nesureagavusio klinkerio. Vietoj silicio mikrodulkių panaudojus maltą stiklą, nesureagavusio cemento kiekis gali būti sumažintas iki 25 %, o gniuždymo stipris padidintas iki 27 %;
 - 1.3. 20 °C temperatūroje silicio mikrodulkių pucolaninis aktyvumas yra didesnis (iki trijų kartų) nei malto stiklo, tačiau taikant terminį apdorojimą (3+18+5) 80 °C temperatūroje malto stiklo pucolaninis efektyvumas padidėja, be to, maltas stiklas pradeda veikti kaip cemento hidratacijos greitiklis.
2. Sujungiant Brouwers ir Amen matematinius modelius, sudaryta skaičiavimo metodika, pagal kurią, įvertinant V/R santykį ir rišiklio hidratacijos laipsnį, galima prognozuoti cemento akmens struktūros parametrus (susidariusių hidratacijos produktų, nesureagavusio klinkerio, nesureagavusio vandens, susitraukimo dėl cheminių veiksnių ir kalcio hidrosilikatų tūrinės koncentracijas) bei sukietėjusio betono gniuždymo stiprį:

- 2.1. eksperimentiniais tyrimais gautas iki 7 % didesnis rišiklio hidratacijos laipsnis ir iki 5 % mažesnis nesureagavusio rišiklio kiekis lyginant su matematiniais skaičiavimais;
- 2.2. eksperimentiniais tyrimais gautas gniuždymo stipris yra iki 8 % mažesnis, lyginant su matematiniais skaičiavimais;
- 2.3. pateikta betono mišinio sudėties projektavimo metodika yra tinkama skaičiuoti 130–230 MPa gniuždymo stiprio betonams.
3. Nustatyta, kad maltas stiklas yra tinkamas priedas naudoti ypač stipriam betonui, nes efektyviau pagerina mišinio technologines savybes (mažina vandens poreikį), nesumažina sukietėjusio betono tankio ($\geq 2400 \text{ kg/m}^3$), padidina betono gniuždymo ir lenkimo stiprį (atitinkamai iki 230 MPa ir 25 MPa be plieninio armuojančio plaušo), efektyviau sumažina suminį betono poringumą (sumažėja nuo 6,0 % iki 3,8 %), padidina atsparumą šalčio poveikiui iki 7 kartų (po 40 šaldymo ir atšildymo ciklų 3 % NaCl tirpale: be malto stiklo – $0,0249 \text{ kg/m}^2$ ir su maltu stiklu – $0,0034 \text{ kg/m}^2$), nekeičia arba nedaug keičia galutines betono susitraukimo deformacijas.
4. Suprojektuotas ypač stipraus betono mišinys yra tinkamas naudoti gamybinėmis sąlygomis, tačiau bendra maišymo trukmė turėtų būti ilgesnė nei nustatyta laboratorijoje. Gamybinėmis sąlygomis paruošto betono gniuždymo stipris yra iki 12 % mažesnis, lyginant su matematiniais skaičiavimais.

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