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APPLICABILITY OF FREEZE-THAW RESISTANCE TESTING METHODS FOR HIGH STRENGTH CONCRETE AT EXTREME -52.5°C AND STANDARD -18°C TESTING CONDITIONS

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Abstract

In the present paper an attempt was made to evaluate reliability and efficiency of two freeze-thaw testing methods by testing high strength concrete (HSC) with two different supplementary cementitious materials as a partial substitute to cement in binary blend. Silica fume (SF) or metakaolin containing by-product (MKW) was used replacing with them 5, 10 or 15 wt% of cement. The freeze-thaw resistance of HSC samples saturated with 5% NaCl solution was tested at standard -18°C and extreme -52.5°C testing conditions. HSC series with SF exhibited higher initial strength, while poor resistance against freeze-thaw cycles was observed. Strength loss from 8 to 25% was observed after 12 freeze-thaw cycles at -52.5°C, while 15 cycles reduced the strength by 30 to 53%, which was similar to 110 or 150 freeze-thaw cycles at -18°C. Hence, it was concluded that extreme low temperature testing can significantly reduce the time, which is necessary for evaluating freeze-thaw durability of HSC. HSC without air entraining additives with W/C ranging from 0.38 to 0.45 proved to be vulnerable to freeze-thaw exposure as its water absorption gradually increased. Ultrasonic pulse velocity measurements during freeze-thaw tests allowed to determine indirectly the strength loss and good correlation between the two was observed.

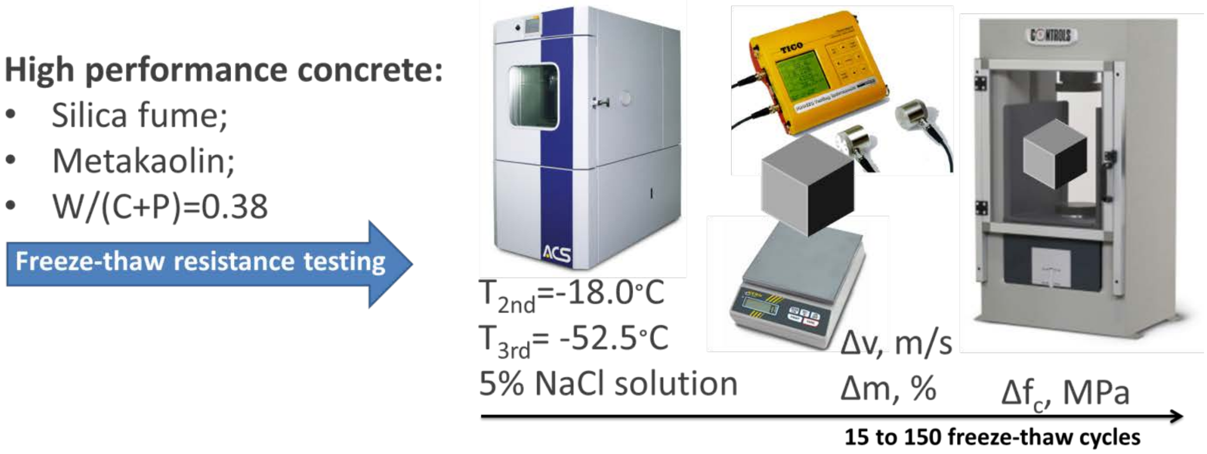
Keywords: freeze-thaw resistance, high strength concrete, ultrasonic pulse velocity

Highlights

- Freeze-thaw resistance of high strength concrete (HSC) with supplementary cementitious materials (5-15wt% of cement) was determined;
- HSC samples saturated with 5% NaCl solution were compared under standard -18°C and extreme -52.5°C testing conditions;
- Similar results were obtained by reducing the testing time from 150 to 15 freeze-thaw cycles;
- Ultrasonic pulse velocity and weight change proved to be promising methods for indicating structural and strength changes;
- Silica fume reduced freeze-thaw resistance of HSC samples compared to both reference and metakaolin containing HSC compositions.

Declarations of interest: none

Graphical abstract



Introduction

The traditional, normal strength concrete (NSC), which is widely used in the civil engineering, belongs to the strength class from C8/10 to C50/60 and precise testing methods are developed for this material. The high strength concrete (HSC), which has shown superior properties over NSC, does not have sufficient amount of approved quick methods for assessing its durability (freeze-thaw resistance, resistance to chloride migration, alkali-silica corrosion, etc.). According to the recent research, HSC could be considered as a special material group due to incorporation of pozzolanic additives and chemical admixtures in a composition so that the compressive strength exceeds 70 MPa and more, which requires different methods for designing and testing and especially guidelines for data interpretation [1], [2], [3]. The importance of the above mentioned problem is demonstrated by the activities of the RILEM, which are related to the research and efforts to create new standards and methods for durability assessment of the materials, such as Technical Committee 246-TDC “Test methods to determine durability of concrete under combined environmental actions and mechanical load”, TC 176-IDC: "Internal Damage of Concrete Due to Freeze-Thaw Attack", etc.

It is important to raise awareness of the stakeholders, such as producers, users and legislators with regard to the properties of HSC including its durability. For example, in the standards of several countries (USA, Germany, Canada, Latvia etc.) it is defined that the freeze-thaw resistance of the ordinary NSC is linked to certain amount of air voids; this is used as an indicator for predicting the concrete freeze-thaw resistance [4], [5], [6]. Namely, determination of air volume in the concrete can be considered as an indirect method for predicting its freeze-thaw resistance [7]. In contrast, the studies performed during the last years have demonstrated that water saturated HSC can reach 500 freeze-thaw cycles class without use of the air-entraining admixtures, if the composition is proper and optimal conditions are ensured during the initial hardening period [8], [9], [10]. The freeze-thaw resistance class for HSC is considerably higher than for average concrete. It shows that it is necessary to update the existing national standards revising the requirements for the mandatory use of air-entraining admixtures in order to reach certain freeze-thaw resistance class as well as considering air volume as an indirect method for predicting the concrete freeze-thaw resistance [8], [11]. Second most important aspect is to choose the most appropriate method for determining the HSC freeze-thaw resistance; it should be rapid and unambiguous, when interpreted. The existing standards often do not fulfil these requirements. Wang et al has reported that freeze and deicing solution of 5% NaCl has the most severe damage due to fact that specimens absorb higher water content that would form an ice at the temperature of -28°C [12]. The principle of extremely low temperature freezing is based on fact, that temperature range from 0 to -20°C is associated with water freezing in larger pores only causing little contractions due to excess water escape to partially filled pores or cavities; while freezing below -20°C and down to -60°C forms high stresses and cracks in smaller pores [13]. This is an important aspect to consider of testing HSC, because according to the test results, HSC typically has lower permeability properties and higher corrosion resistance compared to the NSC. This is due to the lower porosity of the HSC, where pores with relatively smaller size dominate, the capillary pores are not interconnected, which results in lower water and steam permeability, as well as lower rate of penetration of aggressive substances [14], [15], [16].

In present research HSC with compressive strength >70 MPa has been prepared by using two microfillers (microsilica, metakaolin), to replace with them 5, 10 and 15wt% of cement in the concrete compositions. The freeze-thaw resistance was tested at the freezing temperature -18°C and extreme -52.5°C to enhance frost damage and 5% NaCl deicing solution was used. The durability to freeze-thaw resistance of the HSC without air entraining agents was evaluated.

2. Materials and Mixtures

2.1. Cement

CEM I 42.5 N type cement made by Cemex Ltd (Latvia) was used in the research. Specific gravity and fineness (Blaine) of the cement are 3.15 g/ cm^3 and 3787 cm^2/g, respectively. The chemical composition of the cement is given in Table 1. The mineral composition of cement clinker is C_3S – 57.7%, C_2S – 18.2%, C_3A – 6.4%, C_4AF – 9.8%, the free lime 2.0%, Na_2O_{ekv} – 0.9%. The particle size was determined with Laser Particle Sizer Analysette 22 NanoTec (FRITSCH GmbH) and calculation was performed according to Fraunhofer calculation and Automatic Modell Detection. Measurement interval was from 0.01 to 200 µm. The results is given Figure 1.

Component	Compound, wt%								
	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	SO_3	Na_2O	K_2O	L.O.I.
Cement	63.2	18.8	3.9	3.0	3.2	3.3	0.2	1.1	2.1
SF	0.2	98.4	0.2	-	-	0.1	0.15	0.2	0.5
MKW	0.1	51.8	34.2	0.5	0.1	-	0.6	-	11.9

Table 1. Chemical composition of the cement and supplementary cementitious materials

2.2. Supplementary cementitious materials (SCM)

Undensified silica fume (SF) Elkem Microsilica Grade 971-U (Norway) was used in preparation of the HSC, whose chemical composition given in Table 1. Coarse particles >45µm was 0.2%, bulk density - 300 kg/m^3. Specific gravity of the SF was 2.15 g/ cm^3.

Metakaolin containing by-product (MKW) coming from the foam glass granule production plant JSC Stikloporas Ltd. (Lithuania) was used. Kaolin clay was used as a substance for anti-agglutination at the final stage of expanded glass granule production. During the production the kaolin has been calcined at 850°C for about 40-50 minutes. The MKW with fraction <0.355mm was used. Chemical composition of the MKW is given in Table 1 and particle size distribution is given in Figure 1.

2.2. Aggregates

Natural washed gravel with fraction size 4/12mm was used as coarse aggregate and natural washed sand <4mm and fine sand <0.3mm were used as fine aggregates in HSC. The particle size distribution of aggregates, selected SCM and cement is given in Figure 1.

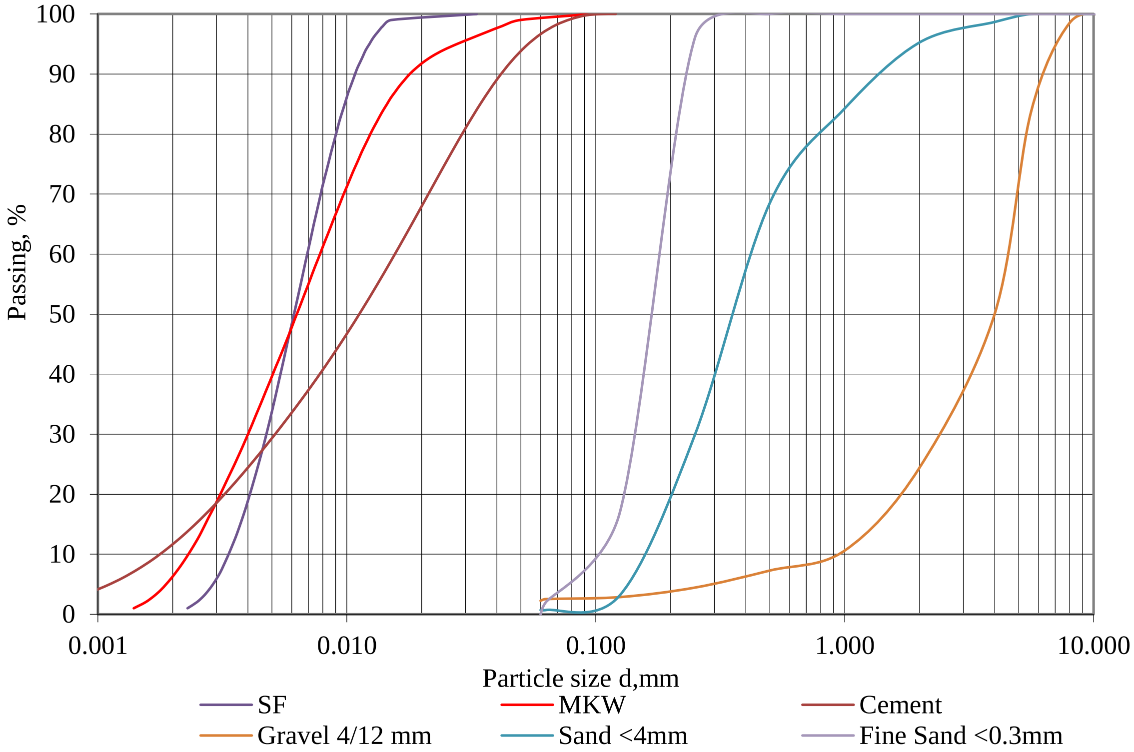


Figure 1. Particle size distribution of aggregates, SCM and cement

2.3. High range water reducer (superplasticizer)

Carboxylate-based high range water reducer superplasticizer Vinplast CL10 (Vincents Polyline Ltd., Latvia) was used. The chemical composition and physical properties of the superplasticizer correspond to the requirements of the national Standard LVS EN 934-2 as indicated in the manufacturer's data sheets. Max chloride content is 0.008 wt%.

2.4. *Mixtures*

In this research seven HSC mixture compositions were prepared. In all mixtures, water/(cement+SCM) ratios were kept constant at 0.38. The HSC workability was controlled by changing the amount of superplasticizer to maintain the cone flow of the HSC >600 mm. The cement dosage was 500 kg/m^3 for reference mixture (REF). Two series of HSC were prepared with different amount of selected SCM. 5 wt%, 10 wt% and 15 wt% of cement was replaced by SF or MKW in each series. Codes and mixture composition of the HSC are presented in Table 2.

Component	Mixture design, kg/m^3						
	REF	M-5	M-10	M-15	S-5	S-10	S-15
Cement Cemex CEM I 42.5N	500	475	450	425	475	450	425
Sand 0.3/4mm	700	700	700	700	700	700	700
Fine Sand <0.3mm	118	118	118	118	118	118	118
Gravel 4/12 mm	908	908	908	908	908	908	908

Water	190	190	190	190	190	190	190
Carboxylate-based superplasticizer	6.5	7.1	8.4	9.5	6.7	7.5	8.0
Metakaolin containing by-product	-	25	50	75	-	-	-
Silica fume	-	-	-	-	25	50	75
W/C	0.38	0.40	0.42	0.45	0.40	0.42	0.45
W/(C+SCM)	0.38	0.38	0.38	0.38	0.38	0.38	0.38

Table 2. Mixture composition of concrete used in the test series

The mixing procedure was carried out in a planetary drum mixer and included the following stages: all dry components were mixed together for 120 s to obtain homogenous mixture of dry components. Then half of the calculated amount of water was added and mixing was continued for another 120 s. The remaining water with superplasticizer was added and mixing was continued for additional 120 s. Afterwards the consistency of HSC was determined and fresh concrete density was measured. The fresh concrete was compacted in cubical 100mm steel molds and allowed to harden in the laboratory under plastic sheets. In total 22 l batch was mixed for each composition. After 24 h, the steel molds were removed and the cubes were placed in a water at T = 20 ± 2°C. When the specimens reached the age of 7, 28 and 180 days, 3 of them were removed from water and after 2h ± 0.5h compression test was performed.

3. Methods

3.1. Physical and mechanical properties

Density of fresh concrete was measured according to Standard LVS EN 12350-6 and workability of HSC was tested according to LVS EN 12350-8. The compressive strength was determined according to LVS EN 12390-3. Three specimens were tested at the age of 7, 28 and 180 days and average value with deviation was calculated. Concrete compressive strength after freeze-thaw tests was determined for 6 specimens.

Volumetric water absorption test for cubic specimens was carried out as described below: a) 1/3 of sample was immersed in water for 24h; b) the following 2/3 of sample was immersed for the next 24h, and c) then fully immersed samples were kept for additional 48h. Then the saturated sample mass was determined and water absorption by volume (1) and mass (2) was calculated:

$$W_V = \frac{m_{sat} - m_{dry}}{V_s * \rho_w} * 100\% \qquad (1);$$

$$W_{wt} = \frac{m_{sat} - m_{dry}}{m_{dry}} * 100\% \qquad (2);$$

where

W_V– water absorption by volume, vol% (content of permeable pores, vol%)

W_{wt}– water absorption by weight, wt%

m_{sat} – apparent mass of water saturated sample, g

m_{dry}- mass of oven-dried sample in air, g

ρ_w - density of water, 1 g/cm^3

V_s – volume of the sample, cm^3

Water absorption test was performed both for hardened concrete samples before freeze-thaw test and after freeze-thaw test at -52.5°C.

3.2. Freeze-thaw cycle exposure

When the concrete specimens had reached an age of 180 days, they were exposed to a predefined number of freeze-thaw cycles in an appropriate chamber (ACS Sunrise climate chamber with temperature range from -80 to 190°C). These tests were organised according to the National annex of Latvian standard to European standard EN 206-1 - Part 1: Requirements for classification and attestation of conformity LVS 156-1:2009 [17]. In this case accelerated 2nd and 3rd freeze-thaw testing methods were selected (Figure 2):

1) 2nd method - one cycle of freezing and thawing lasts about 24 hours. The samples were saturated with 5% NaCl solution before the test for 72h and then placed in freezing chamber at -18± 2°C. After 8h of freezing samples were placed at 5% NaCl solution at 18± 2°C for deicing and further saturation maintenance before the next freezing cycle; testing of samples was done in up to 150 freeze-thaw cycles;
2) 3rd method - duration of one freeze-thaw cycle remains the same – 24 hours. Concrete samples were saturated with 5% NaCl solution for 72h and then placed in stainless steel containers with dimension 120x120x150mm and 5% NaCl solution was filled so that it covered sample at least 20mm; then containers with samples were stored at cooling chamber and temperature gradually (~1°C/min) decreased to -52.5± 2.5°C and samples were kept for 3h±0.5h at the minimal temperature. Afterwards temperature was increased to -10°C (1.5h±0.5h) and then the containers with samples were removed from the climate chamber and placed in 5% NaCl solution at 18± 2°C for deicing. Before the freezing cycle ultrasonic pulse velocity and mass change of saturated concrete samples were measured. Up to 15 freeze-thaw cycles were completed.

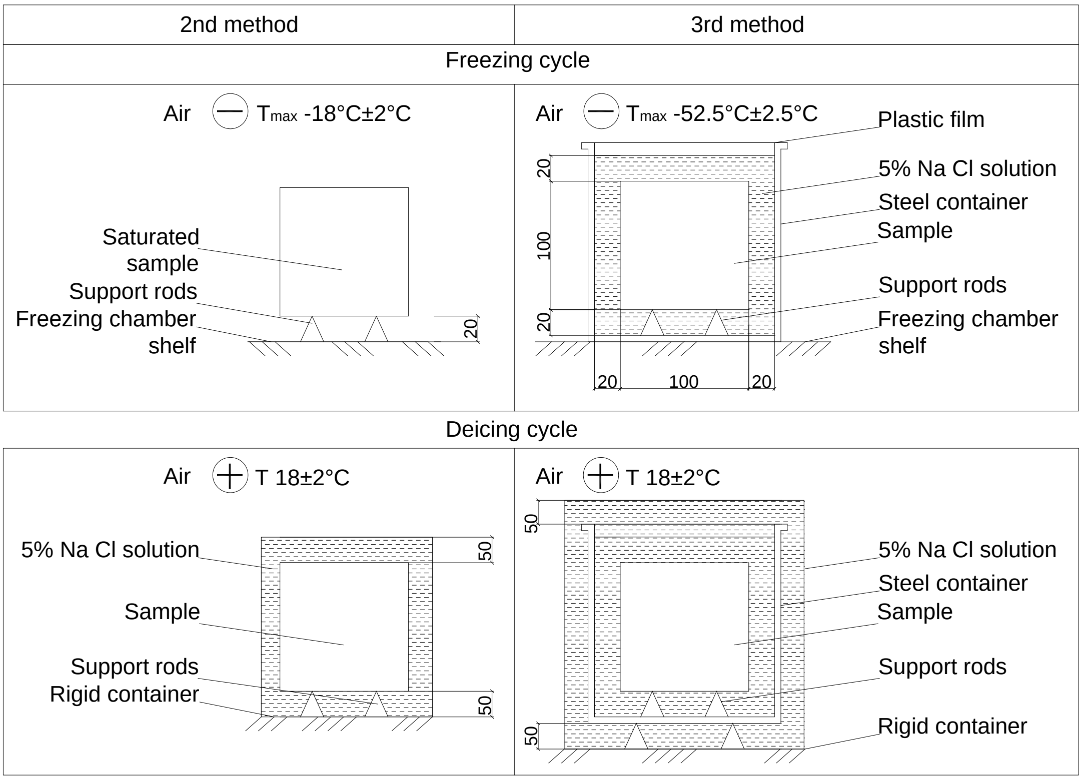


Figure 2. Principle of freeze-thaw durability testing according to the selected testing methods

Number of cycles was selected according to the Table 3 given in Standard [17]. 150 freeze-thaw cycles according to the 2nd testing method correspond to 500 standard freeze-thaw cycles for samples saturated with deionized water at -18°C, while according to 3rd testing method 15 freeze-thaw cycles correspond to 500 standard freeze-thaw cycles. The test duration according to 3rd testing method could be reduced significantly (up to 10 times comparing to 2nd method and up to 33 times comparing to 1st method in deionized water) which could remarkably reduce testing time especially for HSC with increased resistance to freeze-thaw cycles.

Six cubical specimens of prepared compositions were tested with each method. Applying the 3rd method the ultrasonic pulse velocity with Proceq TICO Ultrasonic Testing Instrument and mass change were measured for each sample. The strength reduction was determined after certain amount of freeze-thaw cycles and compared to reference samples which were tested at the age of 180 days (just before freeze-thaw test).

Concrete	Freeze-thaw cycles necessary to obtain final result													
	Freeze-thaw method	Number of freeze-thaw cycles for specified concrete freeze-thaw class												
		F25	F35	F50	F75	F100	F150	F200	F300	F400*	F500*	F600	F800	F1000
All kind of concrete except for pavement and roads, runways	1st method	25	35	50	75	100	150	200	300	400	500	600	800	1000
All kind of concrete except for pavement and roads, runways, lightweight concrete with ρ<1500 kg/m^3	2nd method*	-	-	8	13	20	30	45	75	110	150	200	300	450
	3rd method*	-	-	-	2	3	4	5	8	12	15	19	27	35
Pavement and roads, runways	2nd method	-	-	50	75	100	150	200	300	400	500	600	800	1000
	3rd method	-	-	-	-	5	10	20	37	55	80	105	155	205

*Selected testing conditions and number of freeze-thaw cycles performed

Table 3. Evaluation of freeze-thaw test results according to testing conditions applied

4. Results and discussion

4.1. Fresh concrete properties

The reference mixture (REF) containing 500 kg/m^3 of cement and 1.3 wt% of superplasticizer from the weight of the cement had workability described by cone flow t_{500} value of 2.5 s and t_{max} 16.3 s, when maximal cone flow diameter 640x650mm was reached (Table 4). Increasing the use of MKW instead of cement from 5 to 15 wt% also increased the required quantity of superplasticizer from 1.4 wt% to 1.8wt% due to the fine nature of MKW particles in order to maintain the cone flow value t_{500} from 1.9 to 2.6s and the maximal cone flow from 750 to 780mm at t_{max} 26.6s to 27.5s. Similar tendency was observed for HSC series with SF incorporation in the mixture composition. While 5wt% of SF increased the amount of superplasticizer slightly, using 10 and 15wt% of SF instead of cement required the increased amount of superplasticizer - 1.5wt% and 1.6wt% respectively. The cone flow time t_{500} was 1.3 to 2.6s and maximal cone flow diameter was from 610 to 700mm with t_{max} 14.4 to 25.9s.

The fresh concrete density slightly decreased with the increased amount of SCM in HSC. With MKW incorporation in HSC composition fresh concrete density decreased from 0.8 to 3.0 wt% and with SF – from 1.7 to 2.0 wt%.

Composition	Cone flow time, t_{500} [s]	Cone flow time, t_{max} [s]	Cone flow max diameter, mm	Fresh concrete density, kg/m^3
REF	2.5	16.3	640x650	2456
M-5	2.5	27.3	750x760	2437
M-10	2.6	26.6	760x760	2412
M-15	1.9	27.5	770x780	2383
S-5	2.6	21.8	680x700	2415
S-10	1.3	25.9	660x680	2413
S-15	2.6	14.4	610x650	2407

Table 4. Properties of fresh HSC

4.2. Mechanical properties of hardened HSC

The compressive strength at the age of 7, 28 and 180d is given in Figure 3. The results indicate that at the age of 7d reference mixture samples (REF) had strength of 56 MPa and it increased to 68 MPa at the age of 28d and to 83 MPa at the age of 180d. The use of MKW to replace 5, 10 and 15wt% of cement lead to similar strength results as for REF. At early age (7d) the compressive strength of compositions M-5 and M-10 was 56 and 57 MPa, while M-15 showed slightly lower result – 53MPa. At the age of 28d M-5 and M-10 showed slightly lower compressive strength comparing to REF – 63 and 66 MPa respectively, while M-15 - 70 MPa. Long-term curing increased the compressive strength of HSC with MKW in all cases comparing to REF (from 86 to 88MPa). Similar strength results with REF were associated with well know pozzolanic reactions involving reactive metakaolin. At early ages higher substitution level (M-15) provided lower strength due to reduced amount of cement and slow rate of pozzolanic reactions, while long-term curing proved to be beneficial and even increased strength was detected.

Significant strength increase of HSC was detected for compositions with SF. At early age (7d) compressive strength rapidly increased to 75-79 MP,a while the amount of SF in the composition slightly affected the early strength gain. At the age of 28d the strength difference of HSC was more expressive – for mixture composition S-5 compressive strength was 78 MPa, while for S-10 it was 88MPa and S-15 – 90 MPa respectively. In case of SF long-term curing increased compressive strength to 89MPa for S-5 and 97MPa for both S-10 and S-15. The strength gain during prolonged curing was less intensive comparing to REF and samples with MKW – strength gain between 28d and 180d was 22% for REF, 26 to 36.5% for HSC series with MKW and 8 to 14% for HSC series with SF. High reactivity of SF ensured rapid pozzolanic reactions and strength gain of HSC even at early age, while long-term curing lead to maximal strength value which could be obtained with the defined cement and W/C ratio – close to 100MPa (97 MPa for S-10 and S-15).

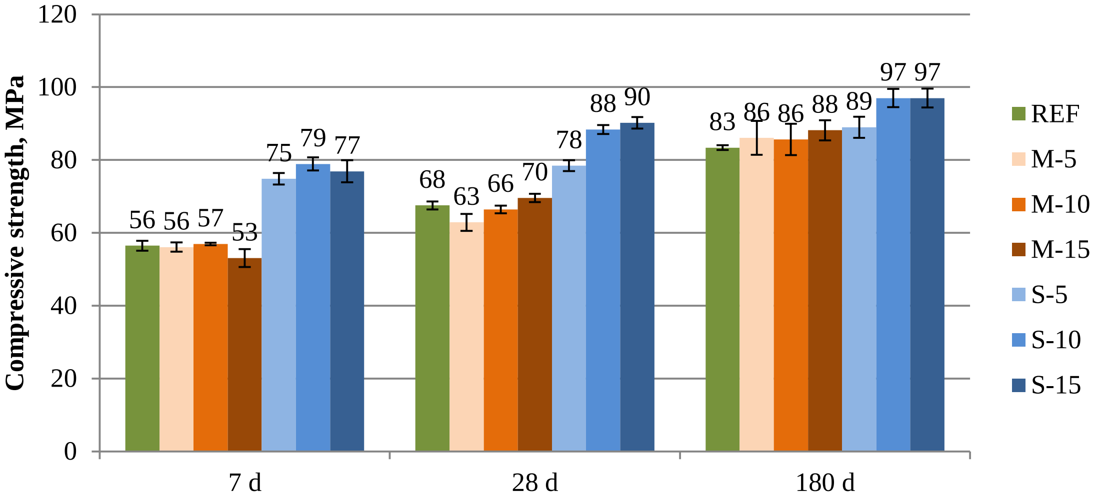


Figure 3. Compressive strength results of HSC with MKW (series M-5; M-10; M-15) and SF (series S-5, S-10 and S-15) after standard curing conditions

4.2. Freeze-thaw test results

Freeze-thaw test results can be evaluated depending from testing method (-18 or -52.5°C freezing conditions) and depending from the amount and type of SCM used in preparation of HSC. More common accelerated freeze-thaw testing method is associated with testing conditions at -18°C and samples saturated with 3 or 5% NaCl solution. Results indicate that after 150 freeze-thaw cycles the 5wt% and 10wt% incorporation of MKW (mixtures M-5 and M-15) reduced the frost resistance comparing to REF and strength reduction was higher, while for the mixture M-10 strength reduction was more even and similar to REF (Figure 4). The strength loss after 150 freeze-thaw cycles was from 83 MPa to 78±4 MPa for REF, while for M-5 it reduced from 86 MPa to 70 ±10 MPa and for M-15 - from 88 to 61±14 MPa with high deviation respectively. The best freeze-thaw performance for HSC series with MKW was for the composition M-10 with strength reduction from 86 to 77±2 MPa. According to the standard 150 freeze-thaw cycles in 5% NaCl solution correspond to 500 standard freeze-thaw cycles in water [17]. The strength reduction was satisfactory for mixtures REF (7%) and M-10 (10.5%), while for M-5 and M-15 it was 18% and 31% respectively. However, strength reduction limits vary according to different opinions published in the scientific literature including the results, where 20% strength reductions is still classified as satisfactory [18].

The 3rd testing method at extreme freezing temperature at -52.5°C was repeated for the same series with other six samples saturated with 5% NaCl solution. Their strength was determined after 12 and 15 freeze-thaw cycles. After 12 freeze-thaw cycles strength reduction was from 72 to 79 MPa which is 8 to 17% reduction from the initial strength. The next 3 freeze-thaw cycles were critical for strength reduction as significant strength loss was observed. After 15 freeze-thaw cycles the compressive strength reduced from 46 to 58 Mpa, which is 32 to 48% reduction from the initial compressive strength. For all mixture series the strength reduction exceeded 30%; therefore HSC failed to withstand 15 freeze-thaw cycles in extreme freezing temperature, which corresponds to about 500 standard freeze-thaw cycles. In the same time 12 freeze-thaw cycles correspond to 400 standard freeze-thaw cycles. In this case satisfactory results were obtained for all mixture compositions with MKW.

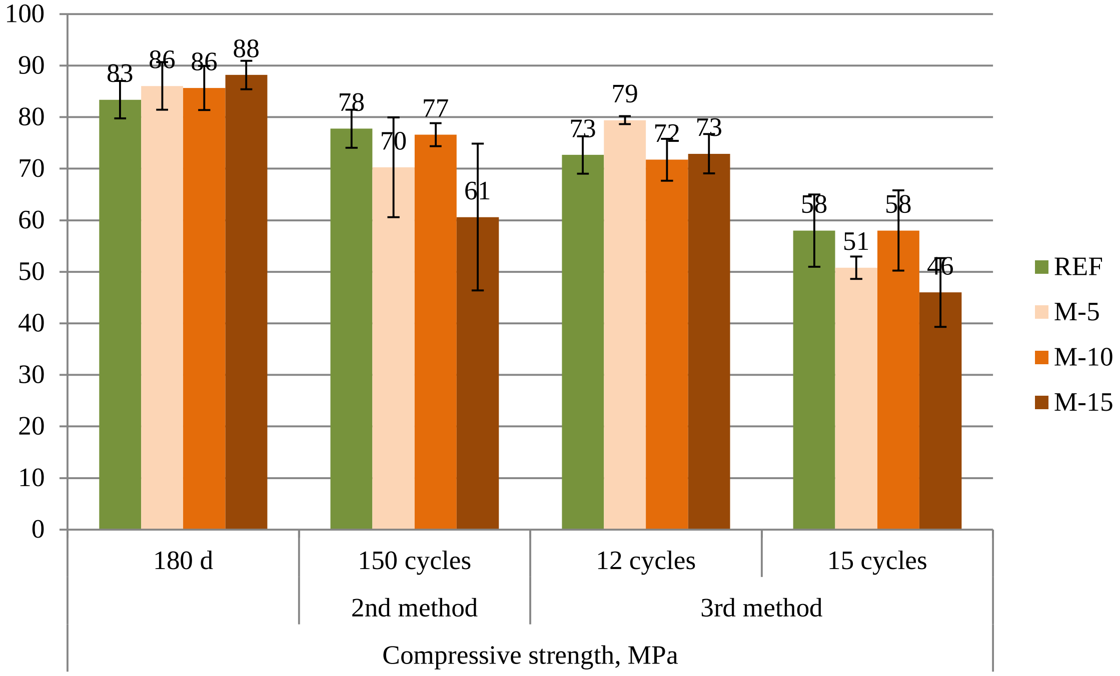


Figure 4. Compressive strength of HSC series with MKW after frost resistance test and reference strength before the test

The ultrasonic pulse velocity for saturated HSC samples was controlled between freeze-thaw cycles just before freezing cycle (Figure 5). The beginning ultrasonic speed was 4700 m/s for mixture composition M-15, 4770 for M-5 and from 4810 to 4840 m/s for M-10 and REF respectively. Gradual ultrasonic pulse velocity reduction during the freeze-thaw cycles was detected. Highest overall velocity decrease was observed after 11th freeze-thaw cycle. Up to 12th freeze-thaw cycle velocity loss was from 4.0 to 4.9%, while during the next 3 freeze-thaw cycles ultrasonic pulse velocity decreased to 5.8% for M-10, 7.6% for REF, 8.4% for M-15 and 8.8%

for M-5, which could indicate rapid damage of concrete internal structure and is a sign of strength reduction of HSC.

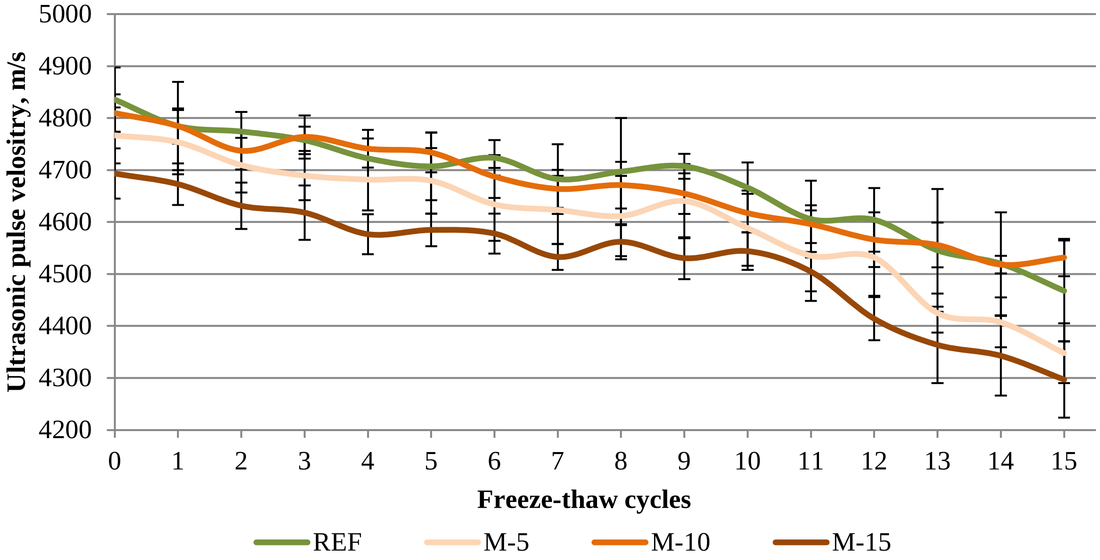


Figure 5. The ultrasonic pulse velocity results of HSC series with MKW during freeze-thaw test at extreme temperature (-52.5°C)

Mass changes of saturated cubical specimens before each freezing were measured during the freeze-thaw cycles according to 3rd method (Figure 6). Results indicate continuous mass increase up to 12th cycle for all mixture series due to increase of testing solution absorption. The highest mass increase was for HSC composition M-15 (0.56%), while for rest of samples it was about 0.45%. Before the 15th freeze-thaw cycle the mass change decreased due to surface scaling of the tested specimens. The mass change was still with positive mark, while the value of mass increase reduced to 0.44% for M-15, to 0.38% for M-10 and to 0.34% to M-5 and REF. The scaling was collected and weighted after 15th freeze-thaw cycle and calculations were performed to display the real mass change of the tested HSC series at the end of the freeze-thaw test (dotted lines in Figure 6). The true mass increase was up to 0.7% for REF, 0.8% for M-5, 0.7% for M-10 and 0.9% for M-15 indicating rapid damage of the samples at extreme freezing temperature.

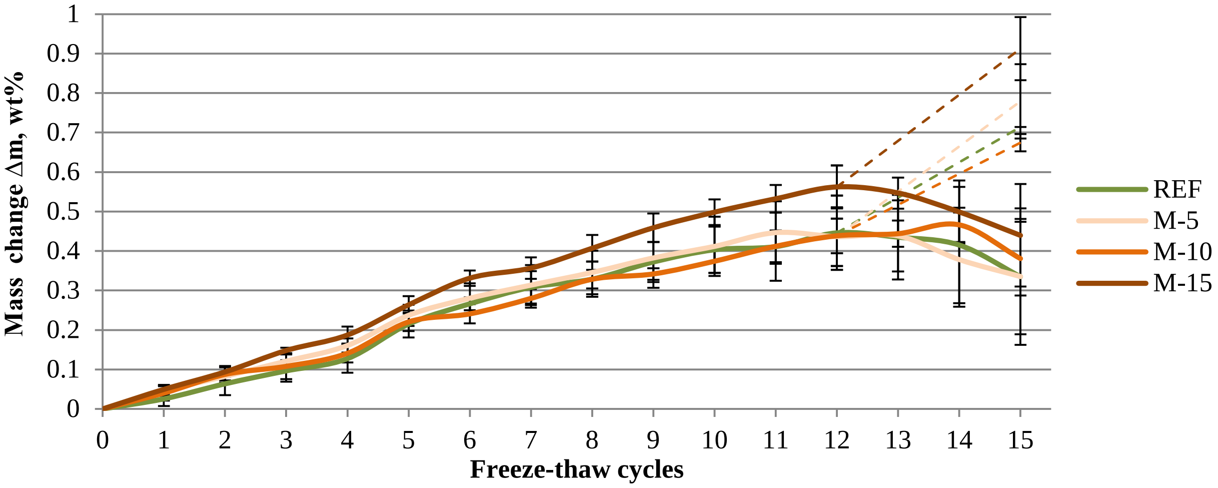


Figure 6. Measured weight change of the HSC mixture series with MKW and true weight change (dotted lines) after calculating measured weight of scaling after the freeze-thaw test

Due to the frost attack the pore structure of HSC was damaged and extra testing solution could absorb in the structure of the material. This was indicated by the water absorption measurements before and after freeze-thaw test (Table 5). The initial water absorption was from 4.6 to 5.2 wt% for HSC series with MKW. After frost resistance test it increased from 5.5 to 6.2 wt%. The open porosity also increased. Initially it was from 10.5 to 11.9 vol%, while after the test it increased to 12.3 – 13.8 vol%. These results correlate well with the strength reduction, which could be associated with the increase of porosity and structural damage of the HSC.

Mixture composition	Water absorption			
	Initial		After 15 f/t cycles at -52.5°C	
	Wwt, wt%	Wv, vol%	Wwt, wt%	Wv, vol%
REF	4.9	11.3	5.5	12.3
M-5	5.2	11.9	5.8	13.2
M-10	4.6	10.5	5.7	13.0
M-15	4.7	10.7	6.2	13.8
S-5	4.5	10.4	5.3	12.1
S-10	4.3	9.9	4.9	11.3
S-15	4.1	9.4	4.9	11.2

Table 5. Water absorption test results for HSC mixture series with MKW and SF before and after freeze-thaw resistance test

The results for HSC series with SF in composition are given in Figure 7. The compressive strength before the freeze-thaw test was 97 MPa for both compositions S-10 and S-15, while for S-5 it was 89 MPa. It is slightly higher comparing to 83 MPa for REF. The second freeze-thaw test method at -18°C was continued to 110 cycles due to severe visible micro-cracking which occurred in samples with SF. Compressive strength test was performed and it indicated that the compressive strength after 110 freeze-thaw cycles increased for REF to 93 MPa (11% strength increase due to hydration in 5% salt solution during 110 freeze-thaw cycles which took around 180 extra days during the test). For HSC series with SF compressive strength decreased by increasing the amount of SF in mixture composition – to 79 MPa for S-5, 71 MPa for S-10 and 65 MPa for S-15 (strength reduction 11 to 32%) and high deviation in results was observed causing unsatisfactory performance.

After 12 freeze-thaw cycles at extreme frost conditions compressive strength reduced to 73MPa for REF (strength reduction 13%), to 79 MPa for S-5 (11%), to 72 MPa for S-10 (26%) and to 73 MPa for S-15 (25%). Further testing to 15 freeze thaw cycles reduced the compressive strength significantly – from 30 to 53% (46 to 58 MPa), while sample ultrasonic pulse velocity and mass change were less impressive comparing to HSC sample series with MKW SCM (Figure 8 and Figure 9).

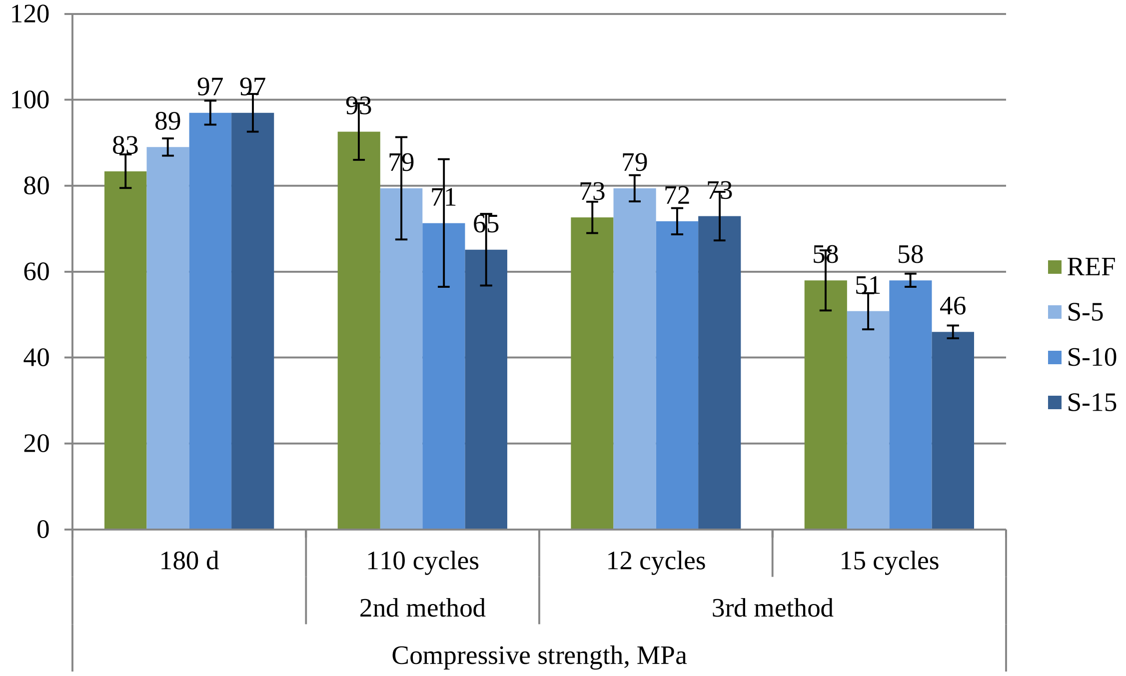


Figure 7. Reference compressive strength of HSC series with SF and strength reduction after freeze-thaw resistance

The ultrasonic pulse velocity during the test reduced only by 1 to 3% for HSC series with SF after both 12 and 15 freeze-thaw cycles, while for REF ultrasonic pulse velocity reduction was 5% after 12 freeze-thaw cycles and 8% after 15 freeze-thaw cycles (Figure 8). The structural integrity of HSC with SF somehow remained dense, therefore neglibly affected ultrasonic pulse velocity, while high strength reduction still occurred.

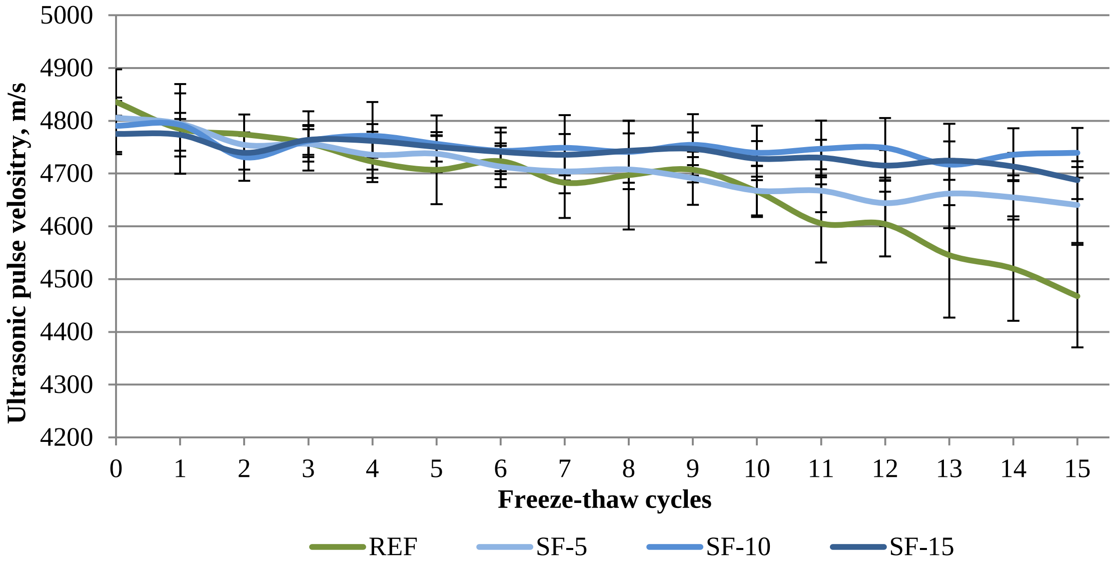


Figure 8. The ultrasonic pulse velocity results of HSC series with SF during freeze-thaw test at extreme temperature (-52.5°C)

The mass change for HSC series with SF was less intense comparing to HSC mixtures with MKW (Figure 9). Only for mixture composition S-15 the weight increase was over 0.2 wt%, while for S-5 there was a weight loss -0.1 wt% according to measurements. By weighting scaling and calculating true mass change the weight of the samples increased for all samples. Comparing

HSC mixture series with SF to REF the weight increase was from 0.2 to 0.3 wt% comparing to 0.7 wt% for reference (REF).

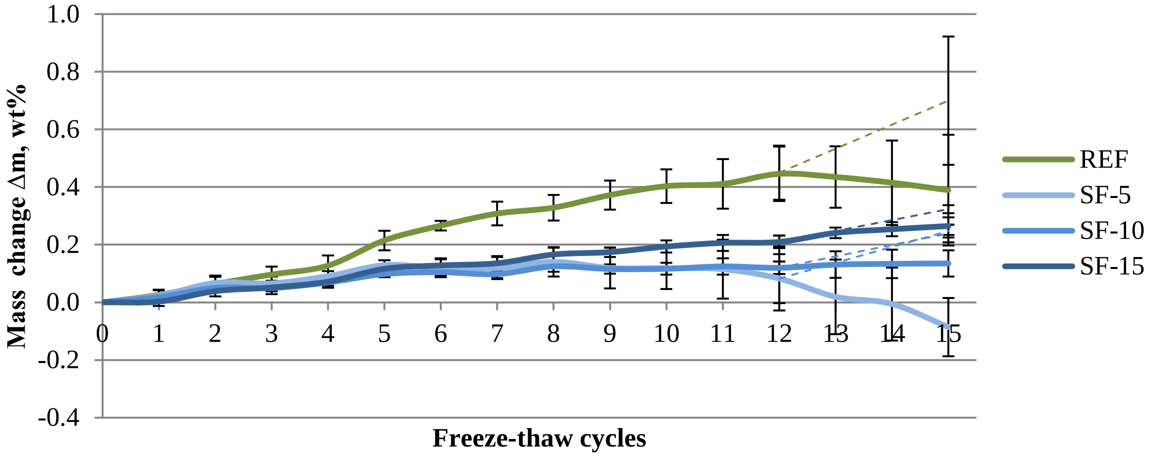


Figure 9. Measured weight change of the HSC mixture series with SF and true weight change (dotted lines) after calculating measured weight of scaling after the freeze-thaw test

Water absorption and open porosity of HSC reduced, when SF was incorporated in the mixture composition. The initial water absorption before freeze-thaw test was from 4.1 to 4.5 wt% for HSC mixture series with SF, while for REF it was 4.9 wt%. Similarly open porosity Wv was lower – 11.3 vol% for REF and it reduced with the increase of SF – for S-5 it was 10.4 vol%, for S-10 it was 9.9 vol% and for S-15 - 9.4 vol%. After 15 freeze-thaw cycles water absorption and open porosity increased but it was still lower compared to REF and HSC mixture series with MKW (Table 5).

The individual sample ultrasonic pulse velocity result and HSC compressive strength correlation before and during the freeze-thaw testing at extreme freezing conditions is given in Figure 10. It can be observed that ultrasonic pulse velocity correlates well with strength change of HSC. Higher velocity indicates higher compressive strength and reduction of pulse velocity indicates structural damage and strength of HSC. The ultrasonic pulse velocity could be a potentially progressive non-destructive method to determine strength change during freeze-thaw test avoiding long testing process and poor results in the final strength measurement. For the saturated HSC ultrasonic pulse velocity can be up to 4900 m/s, while structural changes of HSC during frost damage test could reduce ultrasonic pulse velocity to 4200 m/s, which means that approximately 60 MPa strength decrease could be evaluated with precision of pulse velocity scale of 700 m/s.

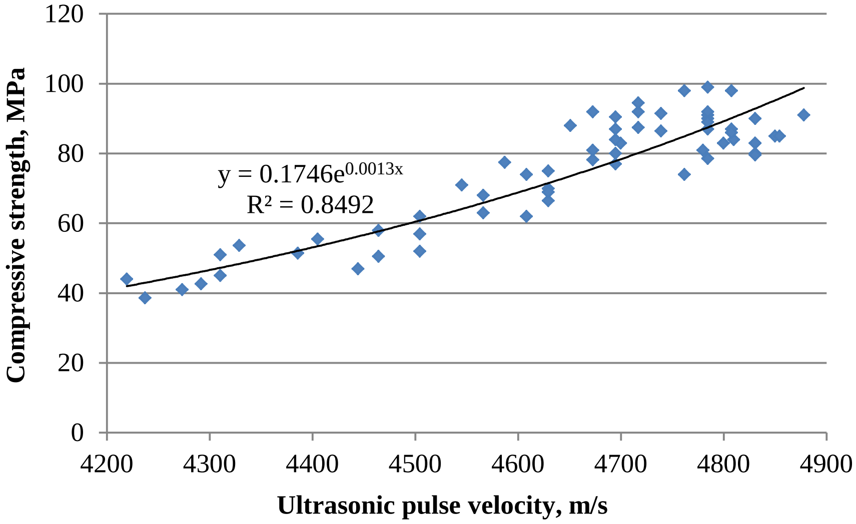


Figure 10. Compressive strength and ultrasonic pulse velocity correlation for individual samples tested during freeze-thaw resistance test

5. Conclusions

Based on the results described in this paper, it can be concluded that damage due to freeze-thaw cycles increases significantly by lowering the freezing temperature to -52.5°C. Extremely low testing conditions can seriously reduce the necessary number of testing cycles from 150 freeze-thaw cycles at standard freezing conditions (-18°C) to 15 freeze-thaw cycles, while observing similar strength loss for high strength concrete with supplementary cementitious materials, such as silica fume and metakaolin containing by-product. Ultrasonic pulse velocity is a promising method to control the strength change and concrete performance during the freeze-thaw tests before the final compressive strength detection. During the tests correlation between velocity and compressive strength of the concrete was observed. The mass change measurements can also indicate the structural changes, while these results may vary due to the surface scaling from samples during the freeze-thaw cycles and different interpretation of the results. The true weight change of samples increased proportionally to the number of freeze-thaw cycles.

In order to do realistic service life prediction for high strength concrete it is possible to establish coherence between the testing conditions of freeze-thaw cycles and indirect measurements, such as ultrasonic pulse velocity, weight changes and strength.

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