Experimental determination of freeze-thaw resistance in self-compacting concretes

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Abstract. The paper describes the determination of freeze-thaw resistance in self-compacting concretes and compares several test methods used for this purpose. The basic principle of testing freeze-thaw resistance is the observation of changes, in this case the loss of certain properties, in concrete attacked by freeze-thaw cycles. This paper documents measurements of tensile strength, tensile splitting strength, and dynamic Young's modulus measured by the ultrasonic pulse velocity test and resonance method. The outcome of the experiment is an evaluation of how suitable each test is for determining the freeze-thaw resistance of self-compacting concrete.

1. Introduction
Self-compacting concrete (SCC) possesses properties thanks to which it quickly received popularity in civil engineering. The first SCC was manufactured in Japan approximately 30 years ago [1] and has entered widespread usage since then. Unlike ordinary concretes, SCC is more sensitive to the quality of feedstock (mainly aggregate and admixtures), composition (variations in particle size distribution, too many fine particles), and the actual placement and curing [2,3].

Environmental matters of building design and construction have recently come into focus as well, whether they pertain to service life or sustainability [4]. Resistance to various adverse effects is therefore important for self-compacting concretes as well [5,6,7]. One of the main factors that may substantially reduce the durability of a concrete structure is the combination of freezing temperatures and high moisture. When concrete is subjected to repeated freezing and thawing, it begins to degrade if water is present. Dry concrete suffers practically no damage from this type of attack [8]. When water comes in contact with concrete, it enters its pore structure and when it freezes (at roughly -0.5 °C in the capillary system), problems may occur. The volume of ice is approximately 9 % greater than the volume of water, which is what causes internal stress in the pore structure. The ensuing damage to the structure may bring about an irreversible reduction in the basic material properties of the concrete [9,10].

It is therefore the objective of this paper to assess the possibilities of determining the freeze-thaw resistance of self-compacting concretes with an emphasis on non-destructive tests (NDT).
2. Experiment
The experiment was performed with specimens made of four self-compacting concretes, which were subjected to freeze-thaw (F-T) cycles. Besides that, all the concretes were tested for basic material properties using both destructive and non-destructive tests.

2.1. Material
For better navigation, the four SCC formulas were identified by the letters A through D. All SCC were made with the same cement CEM I 42.5 R, the same 0-4 mm sand, and the same coarse aggregate of the fraction 4-8 mm and 8-16 mm. They only differed in the amount of plasticiser and the addition of micro-milled limestone and fly ash at varying ratios. Because these concretes were being developed for the commercial market, the paper will not disclose their composition in any more detail.

2.2. Test specimens
Each SCC was made into 9 prisms with the nominal dimensions of 100×100×400 mm. Once cast in steel moulds, the specimens were left to set for approximately 1 hour at standard laboratory conditions and afterwards covered by a PE sheet. At the age of 24 hours the specimens were removed from the moulds and stored at a temperature of (20±2) °C and relative humidity over 95%. When they reached the age of 25 days they were placed under water at (20±2) °C, where they remained for three days until beginning of the tests.

2.3. Test methods
At the age of 28 days, all the specimens were tested for dynamic Young’s modulus. Given the rather low result variability (there were only minor differences between the specimens), see table 1, the specimens of all the concretes were divided into three sets as follows – prisms no. 1 to 3 were reference, 4 to 6 were tested for F-T resistance after 75 cycles and prisms no. 7 to 9 were tested for F-T resistance after 150 cycles.

As stated earlier, the first property to be determined was dynamic Young’s modulus; it was measured by the ultrasonic pulse velocity test and resonance method. Young’s modulus measured by the ultrasonic pulse velocity test bears the designation $E_{cu}$ and its value was calculated in compliance with ČSN 73 1371 [11]. The pulse velocity was measured using a Pundit PL-200 with 150 kHz transducers. Dynamic Young’s modulus measured by the resonance method, in keeping with ČSN 73 1372 [12], is identified as $E_{crL}$ in the case of longitudinal vibration and $E_{crf}$ in flexural vibration. The natural vibration frequencies were determined using a Handyscope HS4 oscilloscope and a software capable of the fast Fourier transform. The reference specimens were then tested for flexural strength $f_{cf}$ according to EN 12390-5 [13] and tensile splitting strength $f_{ct}$ according EN 12390-6 [14] (this test used prism fragments left after the $f_{cf}$ test).

Specimens no. 4 to 9 of all four concretes were then placed in a KD-20 test chamber that allows cyclic freezing and thawing. One F-T cycle consisted of 4 hours of freezing at an air temperature of -18 °C and 2 hours of thawing in water at 20 °C in compliance with ČSN 73 1322 [15].

3. Results and discussion
After 25, 50, and 75 F-T cycles the specimens were tested for dynamic Young’s modulus to see how this property changes depending on the number of cycles. The outcome is the relative dynamic modulus of elasticity (RDM), which is relative to the reference value measured prior to the commencement of the tests (as well as the relative strength development – “f-ratio” in the graphs). The relative development of Young’s modulus calculated from the data obtained by the ultrasonic pulse velocity test is identified as RDM (U), resonance method with longitudinal vibration RDM (FL), and resonance method with flexural vibration RDM (FF). Figure 1 shows plots of these results.
Table 1. Average values of the properties before testing including sample standard deviations (s.s.d.).

| Concrete | $E_{cu}$ [GPa] | $E_{crL}$ [GPa] | $E_{crf}$ [GPa] | $f_{cf}$ [MPa] | $f_{ct}$ [MPa] |
|----------|----------------|-----------------|-----------------|----------------|----------------|
| A: average | 43.8 | 41.3 | 42.0 | 8.6 | 5.2 |
| A: s.s.d. | 0.45 | 0.34 | 0.41 | 0.35 | 0.24 |
| B: average | 41.1 | 38.7 | 39.4 | 8.0 | 4.6 |
| B: s.s.d. | 0.32 | 0.21 | 0.12 | 0.22 | 0.20 |
| C: average | 40.2 | 38.1 | 38.6 | 8.5 | 5.2 |
| C: s.s.d. | 0.42 | 0.45 | 0.49 | 0.17 | 0.34 |
| D: average | 45.1 | 42.4 | 43.3 | 7.5 | 5.2 |
| D: s.s.d. | 0.50 | 0.44 | 0.48 | 0.54 | 0.28 |

Figure 1. Relative development of the properties of all 4 concretes depending on the number of F-T cycles.

Prisms no. 4 through 6 were also tested for flexural strength and tensile splitting strength. The development of the parameters (see figure 1) and visual examination of the prisms (figure 2) clearly show that the prisms failed to resist the planned 150 F-T cycles. At 50 F-T cycles there is already
a visible drop in RDM, especially in concrete B (down to almost 80 % of the original value) and after 75 F-T cycles it is clear that all the concretes suffered severe degradation. The high degree of damage to the internal structure was also manifested in the unorthodox failure that occurred during the flexural strength test (figure 3) in several prisms. The test was therefore ended after 75 F-T cycles and the strength values were tested in all the specimens. Table 2 shows the results of dynamic Young's modulus and strength properties after 75 F-T cycles.

**Figure 2.** Fracture (across the label) in concrete B after exposure to 75 F-T cycles.

**Figure 3.** Unusual failure in prism no. 9 of concrete B during the flexural strength test.

**Table 2.** Average values of the properties including sample standard deviations (s.s.d.) after 75 F-T cycles.

| Concrete | $E_{cu}$ [GPa] | $E_{crL}$ [GPa] | $E_{crf}$ [GPa] | $f_{cf}$ [MPa] | $f_{ct}$ [MPa] |
|----------|----------------|-----------------|-----------------|---------------|---------------|
| A: average | **29.9** | 28.3 | **30.2** | **4.3** | **3.1** |
| A: s.s.d. | 5.02 | 6.19 | 4.72 | 1.14 | 0.72 |
| B: average | **19.1** | **18.1** | **18.1** | **2.8** | **1.7** |
| B: s.s.d. | 6.37 | 6.53 | 6.37 | 0.80 | 0.35 |
| C: average | **24.2** | **25.0** | **25.4** | **3.2** | **3.2** |
| C: s.s.d. | 4.82 | 3.51 | 3.85 | 1.19 | 0.73 |
| D: average | **33.6** | **29.9** | **29.8** | **3.5** | **2.7** |
| D: s.s.d. | 2.09 | 1.98 | 2.47 | 0.75 | 0.37 |
The massive increase in internal cracking, most of which occurred between the 50th and 75th F-T cycle (in concrete B already between the 25th and 50th cycle), is well illustrated by the value and especially amplitude shape of the natural frequencies of longitudinal vibration (figure 4).

![Figure 4](image-url)

**Figure 4.** Natural frequencies of longitudinal vibration in a specimen (prism no. 7 of all the concretes was chosen for this demonstration) in dependence on the number of F-T cycles; the designation of the concrete is indicated in the graph legend.

The results show that the flexural strength test is the best method for assessing the degree of damage in self-compacting concretes after a freeze-thaw resistance test. After 75 F-T cycles, the flexural strength of all four concretes dropped below 50 % of its original value (i.e. the flexural strength of the reference specimens which were not subjected to F-T cycles). The second most sensitive method for determining the freeze-thaw resistance appears to be the tensile splitting strength test; with the exception of concrete C it showed the second highest decrease (in the case of concrete B it was nearly identical to the relative decrease in flexural strength). A critical factor at play during this test is the moment when it is ended. The photos in figure 5 show that the maximum force applied need not correspond to the force at which the SCC specimen damaged by F-T cycles suffered a failure. The first image “a” shows the beginning of microcracks, mainly near the loading strips. The second image, “b”, shows microcracks that span almost the entire height of the specimen – the concrete is
compressed and the microcracks close. Image “c” shows a vertical crack that indicates damage by tensile splitting stress. Given the fact that the self-compacting concrete was badly damaged by internal microcracks, it had a low modulus of elasticity (it lacked “stiffness”) and as the test continued and the loading stress increased, the crack became worse (images “d” through “f” at the bottom of figure 5). The test was ended once the loading stress dropped and the specimen suffered a complete failure, see image “f”. The calculation was therefore made with the force being applied when the vertical crack first appeared, because if maximum loading stress were used, the tensile splitting strength of the prisms would be only slightly lower than that of the reference prisms. This is a potential problem with using this method and should be therefore taken into account.

Figure 5. Progress of the tensile splitting strength test performed with SCC after 75 F-T cycles demonstrated on the failure of a test specimen no. 8 from concrete C.

4. Conclusion
The measurements show that all the test procedures assessed herein were quite able to reflect the decrease in the quality of the self-compacting concretes after exposure to cyclic freezing and thawing.
Differences between the methods are only in their sensitivity to internal structure damage. The greatest decrease of all the observed properties was in all cases found in flexural strength. Testing tensile splitting strength is also rather accurate, however, attention should be paid to the specific loading stress at which the specimen suffers a fracture. The relative dynamic modulus of elasticity determined by the two non-destructive tests showed almost identical progress. The decrease in dynamic Young’s modulus may have been the lowest of all the parameters being observed, but it still reflects the damage rather reliably, and the same goes for the resonance method, as documented by the data in figure 4. In case of RDM it was only necessary to determine a stricter criterion of its maximum decrease if the concrete in question could be dubbed frost-resistant. A great advantage of the NDT methods is using the same specimens during every stage of the test.

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