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Nordic Concrete Research workshop: “Accelerated freeze-thaw testing of concrete”, Lyngby, 20th April 2022

Presenters at the Nordic Concrete Research workshop (and authors of the present paper). From left to right: Matthias Müller, Katja Frid, Frank Spörel, Elisabeth Helsing, Jukka Lahdensivu, Sara Al Haj Sleiman (screen), Marianne Tange Hasholt (front), Abdul Faheem and Stefan Jacobsen.

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ABSTRACT

A one-day Nordic Concrete Research workshop on “Accelerated freeze-thaw testing of concrete” attracted approx. 30 participants. The workshop included presentations on various aspects, such as observed frost damage in the field and the importance of the temperature curve during testing as well as other interactions with the surroundings of the concrete. The workshop also included examples of recent research, which can improve our knowledge about the frost damage mechanism and therefore provide input to improving the standardised test methods. The present paper is a summary of the nine presentations and the discussion arising from the presentations.

Key words: Frost deterioration, accelerated testing, field studies, temperature curve, frost damage mechanism.

1. INTRODUCTION

Pioneering work on accelerated freeze-thaw testing was made in the early 1950es [1-2]. For example, the first tentative ASTM methods were published in 1952 and 1953. As mentioned by T. C. Powers [2], for concrete in areas where freezing temperatures are common, "effects of freezing may be anything from negligible to catastrophic", and for this reason there is a need for test methods to quantify the concrete frost resistance.

T. C. Powers made some basic considerations about requirements for test methods [1]:

- What happens during the test somehow has to relate to what happens in the field. For example, how long time should the concrete be soaked in water prior to exposure to freezing and thawing in the test, to represent the saturation condition of the concrete in the field?
- If the laboratory test is not a one-to-one replicate of what happens in the field (if the test method e.g. prescribes a quicker temperature cycle or a lower minimum temperature to accelerate the development of damage), it should be possible to state a limit value for rejection/acceptance of the frost resistance. Most test methods make it possible to compare different concrete mixes. If specimens from one concrete mix show a certain degree of damage after 150 freeze-thaw cycles, and specimens from another concrete mix show the same degree of damage after 300 freeze-thaw cycles, it is the expectation that the latter of the two concrete mix provides better frost resistance. However, when used for a concrete structure, both types of concrete may be sufficiently frost resistant to provide a long service life (or none of them may be good enough).
- For a standardised test method, it is also important that both the repeatability and the reproducibility are good, so all laboratories performing the test will reach the same result. Which range of deviation on e.g. cooling rate is acceptable, without compromising the reproducibility?

Even though we today know much more about possible damage mechanisms, impact of mix design variables, etc., we basically still discuss the same questions that Powers considered in the 1950es. There is a general trend, where concrete specifications move from being prescriptive to being performance-based, among other things to ease the introduction of more environmentally friendly binders. This push for performance-based concrete design at the expense of deemed to satisfy approaches also pushes for more performance testing, making the questions more relevant than ever. For example, CEN/TS 12390-9 [3] contains three different test methods that differ in curing regime prior to freezing and thawing, amount of freezing medium, and temperature cycles – and which method is then the optimal choice?

To discuss this, a Nordic Concrete Research workshop "Accelerated freeze-thaw testing of concrete" was held at DTU on April 20, 2022. The workshop had approx. 30 participants. The following is a summary of the presentations made at the workshop (following the workshop program) as well as a summary of the final discussion of the workshop.

2. OBSERVATIONS FROM THE FIELD

2.1 Frank Spörel: Freeze-thaw attack and concrete resistance in the CIF-test and under field conditions

Performance tests to evaluate the freeze-thaw resistance of concrete require a balanced adaption and acceleration of the exposure variety under field conditions. As concrete properties change with time, at the test date representative concrete properties should prevail for a reliable evaluation of the concrete resistance. Additionally, a balanced adaption of damage scenarios occurring under field conditions is required.

For concrete that is not going to be exposed to de-icing chemicals (exposure classes XF1 and XF3), the CIF test is often used for testing (CIF: Capillary suction, Internal damage and Freeze-thaw test). A survey on non-air entrained concrete showed that for an existing structure, frost damage is seen in the form of surface damage (scaling). When the same type of concrete is tested in the laboratory with the CIF test on lab specimen with the standardized curing regime, hardly any surface deterioration is seen, but internal damage can be observed by measuring the dynamic modulus of elasticity at various time intervals.

One challenge when comparing field results and laboratory results is that the concrete mixes have changed over the years. Hydraulic structures built before the introduction of the CIF test were typically made with blast furnace slag cement, possibly combined with trass or fly ash, and without air-entrainment (though it became more and more common to use air-entraining agents in the period 1960-1980). After introduction of the CIF test in 2004, hydraulic structures are still built with blast furnace slag cement (still with a slag content of 50-70%), fly ash has also been used in about half of the structures but the use of trass is no longer common. The concrete is typically air-entrained, and the w/b (including an activity factor for fly ash) is typically 0.05-0.10 lower than it was before ($\leq$ 0.55 for air entrained concrete).

Another reason may be that the CIF test (or other freeze-thaw tests) does not take carbonation into account, but surface carbonation is almost unavoidable in real structures. For concrete with slag, carbonation typically results in pore coarsening, see e.g. Figure 1, where non-carbonated concrete has more pore volume in the range 1-10 nm and less pore volume in the range 100-1000 nm, compared to carbonated concrete. The change in pore structure also influences the frost resistance. This may be the reason why frost damage for structures often is in the form of surface damage. The pore structure is also influenced by the curing regime. It was observed that the water contact during the curing of the specimen has a stronger influence on pore structure and performance in the CIF-Test than the testing age. This needs to be taken into account in comparison with the curing of real structures.

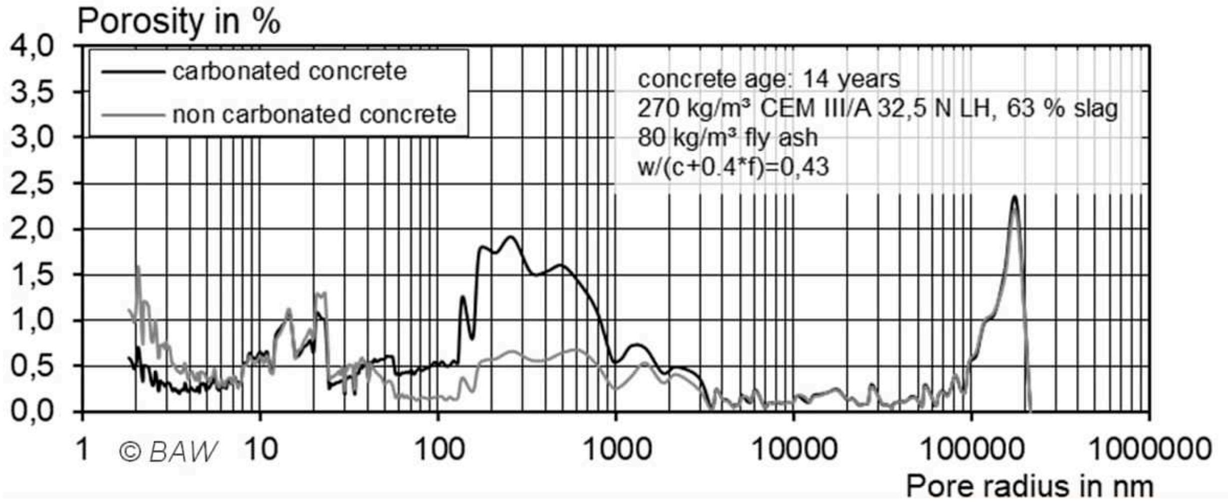

Figure 1 – Pore size distributions

Yet another reason for differences between field and laboratory conditions may be the differences in exposure (moisture state and temperature). A major study has been performed on a German lock structure, where the moisture state was monitored over several years by resistivity measurements. Some of the results were presented at the workshop. More results can be found in reference [4]. Despite less intense exposure at structures damage can occur.

2.2 Jukka Lahdensivu: Freeze-thaw damage in existing Finnish concrete facades and balconies

In Finland, a database is available, which contains information about material properties and observed deterioration of façade panels and balconies from 947 buildings erected during the period 1960-1996. The data were collected as part of condition surveys of the buildings.

In Finland, air-entrainment has been used since the 1980s to ensure frost resistance. In the beginning, the use of air-entrainment failed in many cases, but since the beginning of the 1990s, it can be stated that air-entrainment has been used relatively successfully. The results of ongoing M.Sc. study shows that air-entrainment has failed in approximately 10% of the 1990es concrete facades and balconies. Like this, many of the façade panels and balconies in the database have insufficient air void structures to make them frost resistant, and many of them also show visible signs of frost damage. Despite of this, most of the facades and balconies are still in use, also those that are not fully frost resistant.

From the database, it can e.g. be seen that

- 42.7% of the facades show signs of damage (local damage: 35.4%; widespread damage: 7.3%)
- 27.4% of the balconies show signs of damage (local damage: 21.8%; widespread damage: 5.6%)

In a recent Ph.D. project by Toni Pakkala [5], it was investigated if the frost damage could be correlated with data from the weather observations since 1961 by the Finnish Meteorological Institute (FMI). At three locations, corresponding to inland, southern Finland, and the coastal area in the very south, the annual numbers of freeze-thaw cycles were investigated, depending on the minimum temperature in the cycle (< -2°C, < -5°C, and < -10°C, respectively). It was also investigated how often the concrete structure was exposed to wind-driven rain up to 3 days before freezing temperatures occurred, as it is expected that if the concrete is wet, when it is exposed to frost, damage will be more severe, compared to if the concrete had been relatively drier.

When plotting the observed damage depending on a single variable, e.g. the number of freeze-thaw cycles with at certain minimum temperature, the scatter is large. This is because there are many other contributing variables, e.g. concrete quality and true stress level of the concrete. Incipient freeze-thaw damage has been detected in thin sections. The numbers of freeze-thaw cycles that led to various levels of damage reveal that the same level of damage normally requires more freeze-thaw cycles inland than in the southern coastal area.

3. INTERACTIONS WITH THE SURROUNDINGS (OTHER THAN TEMPERATURE)

3.1 Elisabeth Helsing and Peter Utgenannt: Influence of carbonation on the salt-frost resistance and possibilities to incorporate this factor in freeze-thaw testing

In Sweden, a large study has been conducted to investigate the combined effect of carbonation and frost action (many of the results are also reported in [6]). The study comprised 13 concrete mixes:

- All mixes had water/binder ratio 0.45.
- The mixes varied in binder composition (two different cement types, use of SCMs (up to 35% fly ash (FA) or up to 65% ground, granulated blastfurnace slag (GGBS))
- Ten mixes were air-entrained, whereas three mixes were produced without air-entrainment.

For each concrete mix, six different curing regimes were tested (see also Figure 2):

- Curing regime corresponding to the reference method of CEN/TS 12390-9 (with the additional requirement that the CO_2 content of air in the climate chamber with 65% RH should be 0.04%)
- Five curing regimes, where either the carbonation was more severe than in the reference method or the concrete was more mature, when the frost exposure started, or both.

After curing, the specimens were exposed to capillary suction, followed by 56 freeze-thaw cycles according to the reference method of CEN/TS 12390-9. Some specimens were also exposed in the field, where their condition was followed for 4½ years.

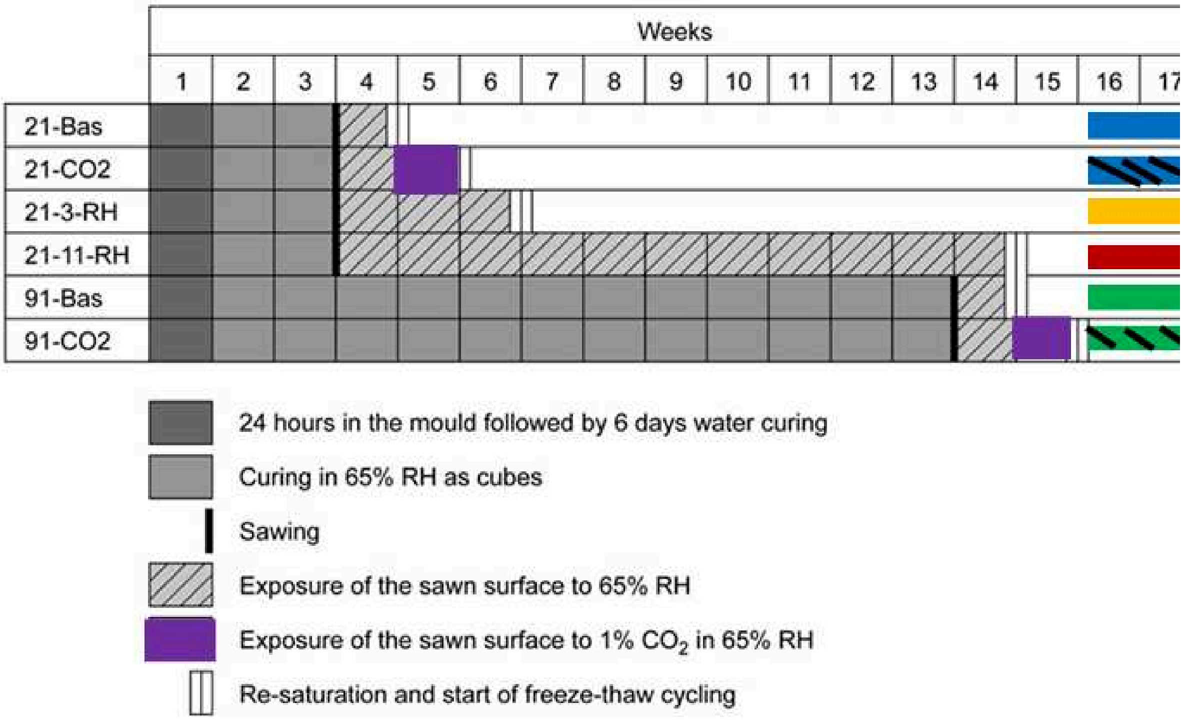

Figure 2 – Overview of curing regimes.

The general trend is that the amount of scaling increases, the higher the dosage of FA or GGBS. Further conclusions depend on if the concrete is air-entrained or not.

- *Air-entrained concrete*
  If the frost resistance in the reference method is acceptable, i.e. scaling after 56 freeze-thaw cycles < 1 kg/m^2, then increased carbonation of the test surface either in the form of 11 weeks at 65% RH, CO_2 content > 0.04% or in the form of 1 week in air with CO_2 content 1% increases the amount of scaling. If the frost resistance is not acceptable when tested according to the reference method, carbonation does not make much of a difference (this is the case for the mix with 35% FA).
- *Non-air-entrained concrete*
  For concrete without air-entrainment, the general trend is not clear (maybe because there are only three mixes in the test program), but there are indications that carbonation in some cases may reduce scaling (especially for concrete without SCM).

The knowledge gained from the project has been implemented in the Swedish application document to EN 206, SS 137003. For concrete, where Portland cement clinker makes up more than 80% of the binder, there is no change in rules. For concrete, where the content of Portland cement clinker in the binder is less than 80%, freeze-thaw testing now has to be conducted both with a curing regime including moderate carbonation (1 week at 65% RH, CO_2 content > 0.04% as in the reference method of CEN/TS 12390-9) and with a curing regime that leads to increased carbonation. The rules were introduced in 2021, so it is going to be interesting to gain more experience with this procedure over the coming years.

3.2 Matthias Müller, Maik Seidel, Horst-Michael Ludwig, and Christoph Müller: Testing salt frost scaling resistance of XF2 concretes

In Germany, salt frost scaling tests are typically only applied for concretes in the exposure class XF4. The salt frost scaling tests such as the slab test (reference method of CEN/TS 12390-9) and the CDF test (one of the alternative methods of CEN/TS 12390-9) also mimic the conditions of XF4, i.e. the degree of water saturation is high, and a de-icing agent such as NaCl is present.

In exposure class XF2, the degree of water saturation is only moderate, but a de-icing agent is still present (example: vertical wall next to a road, where de-icing agents are used during wintertime). In 2007, a test method for concretes in exposure class XF2 was proposed by the German Federal Highway Agency (BASt), but it is not yet generally accepted. This prompted considerations to develop alternative approaches for testing XF2 concretes based on the CDF method and the slab test method. The alternative methods have to be reduced in severity to achieve an applicability to XF2 concretes.

In the first part of the present study, two types of concrete, A1 and B1, were tested in alternative versions of the CDF test. Both concrete A1 and concrete B1 were produced with CEM I 42.5 R cement and no air-entraining agent was used.

A1 w/c = 0.50 (expected compressive strength at least C35/45)
B1 w/c = 0.60

Concrete A1 is a type of concrete that is known to perform satisfactory in XF2. Therefore, there were two criteria for success of the proposed test method: First, the level of scaling for concrete A1 should be less than 1.5 kg/m^2 (often used acceptance criterion for the CDF test), preferably without internal damage. Second, the test should be able to show a difference between A1 and B1. Some of the adaptions tested were: reduced ice layer thickness (1 mm instead of 5 mm), increased salt concentration, increased heating rate (20 K/h instead of 10 K/h), and reduced number of freeze-thaw cycles (14 cycles instead of 28 cycles) and increased minimum temperature (-10°C instead of -20°C).

Based on the tested adaptions and combinations of adoptions, it was decided to continue the work with a method, where the heating rate was 20 K/h and where the concrete was only exposed to 14 cycles. This procedure was used to test 18 concrete mixtures with 9 different types of cement. The cements had different declared strength classes, and most of them were CEM II and CEM III cements with slag, fly ash, limestone filler or a combination of these. For each type of cement, two concrete mixes were produced:

A2 w/c so that compressive strength fulfils the requirements of C35/45 (53 MPa, w/c ≤ 0.50)
B2 w/c = w/c of concrete A2 + 0.10.

It was found that the number of freeze-thaw cycles should be further reduced to six cycles, as the scaling intensity is more appropriate (in combinations with an acceptance criterion for scaling on 1.0 kg/m^2). Some of the results are shown in Figure 3. More results are available in [7].

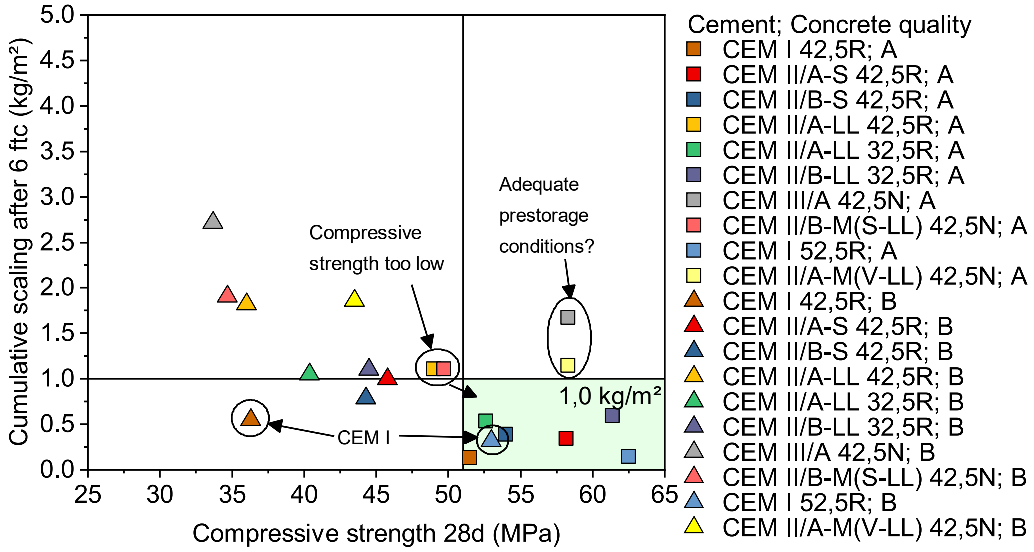

Figure 3 – Results from modified CDF test (heating rate 20 K/h) vs. compressive strength.

It is concluded that:

- The change of heating rate from 10 to 20 K/h works well to reduce the severity of the test, though the reason for the influence of the heating rate cannot be fully explained.
- For all types of cement, there is a clear difference in scaling between A2 and B2 concretes.
- Concretes that fulfil the descriptive requirements for XF2 (quality A2) mostly also fulfil the acceptance criterion of 1.0 kg/m², though the large binder specific differences illustrate that descriptive criteria alone are not sufficient to guarantee a good performance.
- Modification of pre-storage and capillary saturation should be considered for concrete with binder types, where the part of the cement clinker is substituted with supplementary cementitious materials, as is the case for CEM II and CEM III cements.

3.3 Marianne Tange Hasholt: Interaction between concrete and freezing medium during accelerated freeze-thaw testing

The presentation was based on a student project by Master student Sofie Wolter [8], carried out at DTU. The project comprised two parts, where concrete and cement paste, respectively, were examined.

Concrete

Four different concrete mixes were used to produce specimens (Ø150 mm cylinders) for freeze-thaw testing according to the reference method of CEN/TS12390-9. Two mixes were produced with ordinary Portland cement (OPC) as the only binder (water/binder ratio: 0.48) and another two mixes were produced with 50% OPC and 50% fly ash (water/binder ratio: 0.31). The water/binder ratios were chosen so that the expected 28 days compressive strength were similar for the two binder combinations. For each binder combination, two mixes were produced: one with and one without entrained air.

The scaling results were as expected, based on literature (more scaling for concrete without air-entrainment than for concrete with air-entrainment; more scaling for concrete with fly ash than for concrete without fly ash). The news value of the project was that during the freeze-thaw test, liquid samples of the freezing medium were collected every time scaling was collected and one day after re-newing the freezing medium. The liquid samples were analysed by using ion chromatography, IC (Cl^-), and inductively coupled plasma spectroscopy, ICP (Na^+, Ca^{2+} and a number of other cations). Examples of results are shown in Figure 4.

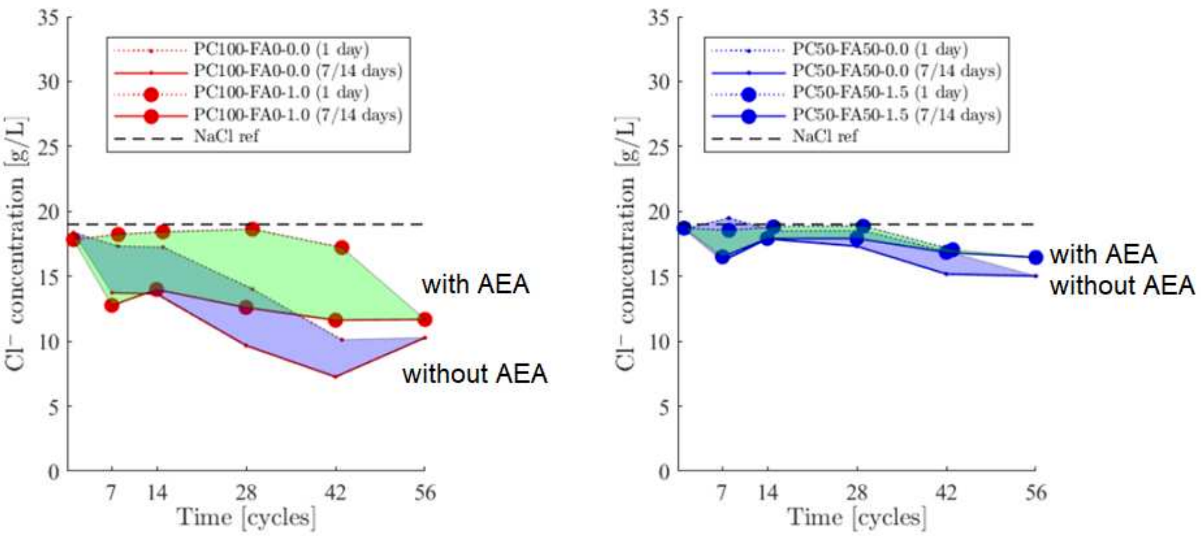

Figure 4 – Chloride concentration of freezing medium, after it has been in contact with concrete. Left: Concrete with 100% OPC as binder. Right: Concrete with 50% OPC+50% FA.

The results show that chlorides gradually diffuse into the concrete and disappear from the freezing medium. The process is quicker for concrete without air entraining agent (AEA) than for concrete with AEA. This is probably because frost damage leads to cracking of the concrete without AEA. From the beginning, only sodium and chloride are present in the freezing medium. During the freeze-thaw test other ions leach out from the concrete to the external liquid reservoir. For most of the investigated ions, the transport is quicker for concrete without AEA than for air-entrained concrete, again probably because of development of frost damage in the concrete in the form of internal cracks, where transport is relatively fast. Another important observation is that the change in ion concentration over time is very different for concrete with 100% OPC and concrete with 50% OPC + 50% FA. This means that the freezing points of the freezing media will differ. This is problematic, if both types of concrete are placed in the same freezing cabinet during testing, and the cabinet is controlled by only one thermocouple, since the freezing media will not start to transform to ice at the same temperature.

Cement paste

Cement paste corresponding to the concrete mixes described above were prepared, i.e.

- 100% OPC, water/binder ratio 0.48
- 50% OPC + 50% FA, water/binder ratio 0.31

The paste samples were cast in Ø22 mm cylindrical moulds. The paste specimens were used to investigate the composition of the paste before and after chloride ingress by thermo-gravimetric analysis (TGA). To accelerate chloride ingress, a migration set-up similar to the set-up described in NT BUILD 492 was made, however, the set-up was designed for the much smaller Ø22 mm specimens.

TGA results are shown in Figure 5.

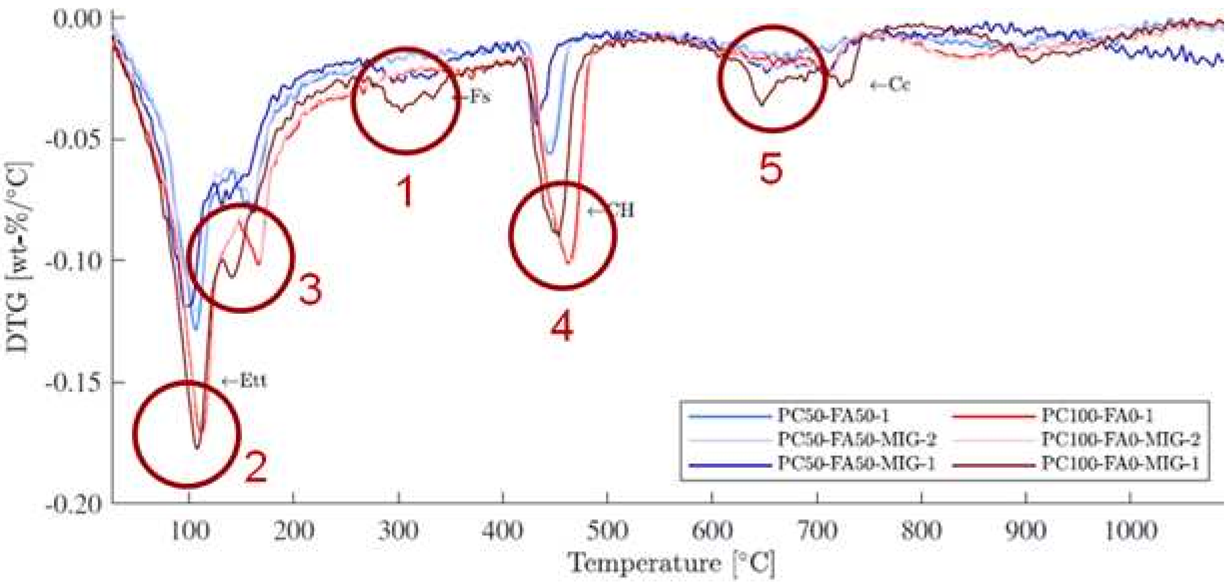

Figure 5 – TGA curves for paste specimens
(chloride exposure indicated by the last part of the ID:
"-1" paste prior to migration test;
"MIG-1" 0-5 mm from end exposed to NaCl during migration test;
"MIG-2" 0-5 mm from the opposite end).

The TGA curves for paste samples collected prior to the migration test (ID extension: "-1") and after the migration test from the end that was not exposed to NaCl during the migration test ("-MIG-2") are almost identical. They differ from the TGA curves for samples collected after the migration test from the end that was exposed to NaCl ("-MIG-1"). The TGA curves indicate that the following takes place, when chloride enters the paste:

1. Chloride becomes bound in Friedel's salt
2. The amount of ettringite is stable (independent of chloride ingress)
3. Formation of Friedel's salt seems to be at the expense of monocarbonate
4. Chloride ingress results in a reduced amount of calcium hydroxide
5. Chloride ingress results in an increased amount of calcium carbonate (calcite)

In other words: chloride takes the place of the carbonate ion in monocarbonate to form Friedel's salt and the released carbonate ions start typical carbonation reactions, i.e. they react with calcium hydroxide to form calcite. Like this, it seems that there is a secondary effect of the chloride ingress in the form of an internal carbonation attack. The reactions look similar for the two types of binders, however, because of the lower amount of OPC in the 50% OPC + 50% FA paste, there is less ettringite from the beginning, and because of the pozzolanic reaction there is also less calcium hydroxide. Since it is known that carbonation prior to freeze-thaw testing has an impact on the outcome of the freeze-thaw test, this internal carbonation attack should be investigated in more detail.

4. THE IMPORTANCE OF THE TEMPERATURE CURVE

4.1 Abdul Faheem: Influence of thermal boundary conditions and temperature distribution in concrete on frost scaling

In the reference method of CEN/TS 12390-9, and in a number of other standardised freeze-thaw test methods, the temperature of the freezing medium and the temperature distribution in the concrete are dependent variables. Typically, focus is on controlling the temperature of the freezing medium, and if the freezing medium is e.g. cooled and heated by the air in the climate cabinet, then this action will determine the thermal boundary conditions of all concrete surfaces, also the surfaces that are not exposed to the freezing medium. In a recent PhD project carried out at Technical University of Denmark, DTU, a test set-up was designed, where the temperature of the freezing medium was controlled by a separate temperature control unit (based on a Peltier element) in direct contact with the freezing medium. In this way, it was possible to make test conditions where the temperature of the freezing medium and the temperature of the air surrounding the test specimen were independent, thereby making it possible e.g. to impose different temperature gradients in the concrete specimen, see Figure 6:

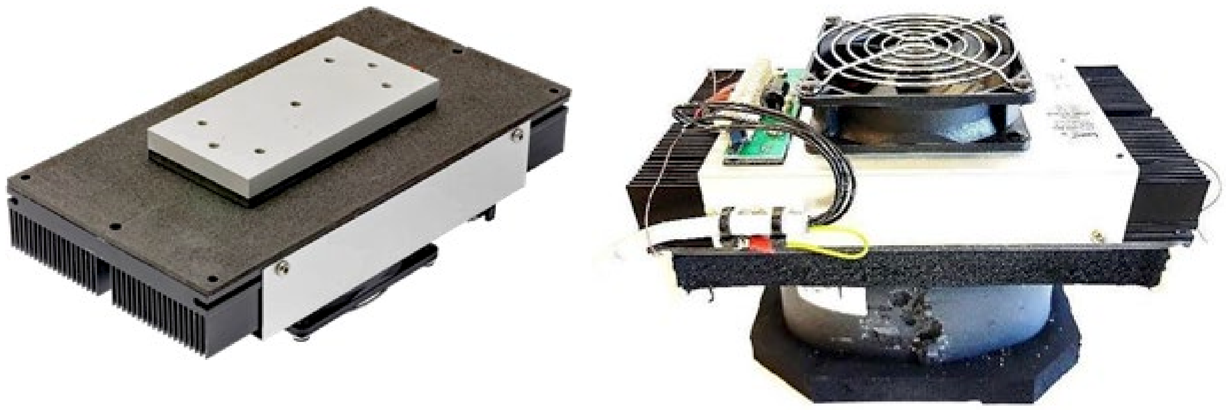

Figure 6 – Experimental set-up. Left: Peltier element mounted on cooling finns with fan (upside down). Right: Temperature control unit mounted on mortar specimen.

A factorial design of experiments was followed to assess the influence of the following thermal boundary conditions:

A. Salt concentration in the test liquid (0% or 3% NaCl solution); salt concentration was seen as a thermal boundary condition, because it changes the freezing point of the liquid, which typically results in a peak on the temperature curve.
B. Test liquid layer thickness (3 mm or 7 mm); liquid thickness was also seen as a thermal boundary condition, since more heat needs to be removed when transforming a larger liquid volume to ice.
C. Air temperature of the air surrounding the insulated concrete (constant 2°C or 20°C).

The test specimens were in all cases non-air-entrained mortar specimens, w/c = 0.45. For each experiment, seven freeze-thaw cycles were run, corresponding to the temperature cycle of the reference method of CEN/TS 12390-9. By the end of the experiment scaling was collected to quantify the extent of frost damage. Each experiment was replicated twice (with two different temperature control units).

Based on the results, it can be concluded that the salt concentration (A) is the most important variable, followed by the surrounding air temperature (C). The test liquid layer thickness did not have a significant effect. Another interesting finding is that there is an interaction between variables. For 0% NaCl, scaling increased when the air temperature increased from 2 to 20°C. The results were sensitive to small changes in the experimental set-up, i.e. different results were obtained with the two temperature control units. The results can be explained by the theory of cryogenic suction, where some temperature distributions promote and others limit cryogenic suction, either from the liquid reservoir or from unfrozen parts of the concrete.

(A few days after the workshop, the project was approved for publication, so a more thorough description of the research methodology, results, and statistical analysis can be found in [9].)

4.2 Sara Al Haj Sleiman: Alternative temperature cycle during accelerated freeze-thaw testing

The measuring principles of the reference method of CEN/TS 12390-9 and the French test standard XP P 18-420 are very much alike, i.e. both methods are scaling tests where liquid reservoirs are established on top of the test specimens and the temperature requirements are stated for the liquid of the reservoir. However, the temperature curves are different, though in both cases the maximum temperature is +20°C, the minimum temperature is -20°C, and the cycle duration is 24 h. Moreover, in 2018 CEN/TC51/WG12/TG4 initiated a round robin test, where four European laboratories tested two concrete mixes, and this round robin test revealed that the variation in results obtained at different laboratories was unacceptably high: After 56 freeze-thaw cycles, the laboratory registering the highest amount of scaling measured approx. five time as much scaling as the laboratory registering the smallest amount of scaling. It was suspected that it was the specification of the temperature cycle that made the result sensitive to small variations. For this reason, a project was initiated to improve the reliability of the test conditions applied during the scaling tests.

A temperature cycle with both freezing and non-freezing temperatures and with a difference between maximum and minimum temperatures of 40°C is never seen in real climatic conditions. A climatic variability analysis for different sites with moderate to severe climates revealed that the temperature span between maximum and minimum temperature is typically not more than 20°C, e.g. +5 to -10°C or +5 to -15°C [10]. This has been shown for places with very different climates, such as Borås in Sweden, Névache in France, Ulaan Baator in Mongolia, and Nizhny Novgorod in Russia.

A test based on the reference method in CEN/TS 12390-9 was conducted with an alternative temperature cycle from +5°C to -15°C and a cycle duration of 24 h. The temperature curves measured for freezing medium on individual specimens are shown in Figure 7 (right). This alternative temperature cycle resulted in much reduced deviation when comparing scaling from specimens that were part of the same test. It also reduced inter-laboratory variation. This is probably not so much because of the temperature cycle itself. It is probably more linked to the fact that the smaller gap between maximum and minimum temperature made the conditions for individual specimens more uniform. As shown in Figure 7 (left), when applying the standard cycle, not all temperature curves for individual specimens actually fulfilled the requirements to the temperature cycle. If comparing temperatures for individual specimens at a certain point in time, the difference could be up to 9°C [11]. For the alternative cycle, the difference was much smaller (up to 2°C).

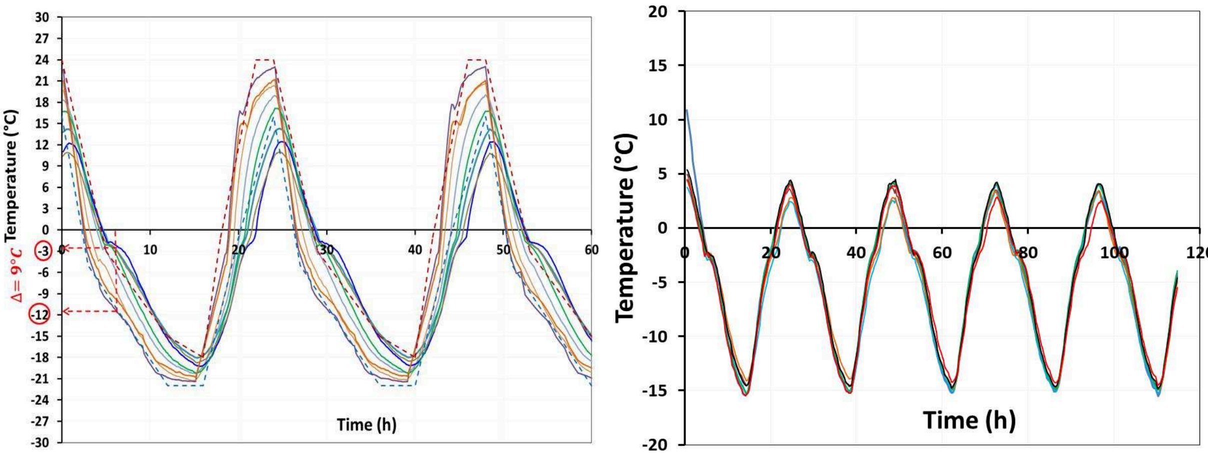

Figure 7 – Temperature curves measured in freezing media for different specimens during a freeze-thaw test. Left: Temperature cycle according to the reference method in CEN/TS 12390-9 (+20°C to -20°C). Right: Alternative temperature cycle (+5°C to -15°C).

5. THE FROST DAMAGE MECHANISM

5.1 Katja Frid: How can neutron imaging and 3D-tomography increase our knowledge about frost deterioration?

Frost deterioration of concrete is only an example of a damage mechanism that involves water, as almost all concrete deterioration mechanisms include water one way or another (leaching, corrosion, etc.). It is the expectation that when the European Spallation Source (ESS) in Lund is up and running, it will be possible to study how water moves inside the concrete during different exposure situations, as this is the key to validate many of the existing models for damage development.

Already now, it is possible to do experiments on a small scale at the NeXT beamline at Institute Laue-Langevin (ILL) in Grenoble. Therefore, a multi-disciplinary research group has formed with scientists from Malmö University, Lund University, and ILL, to explore the opportunities. The group has so far worked with a combination of CT-scanning and neutron radiography. The CT-scans are used to identify aggregates, paste and air voids in concrete. Neutron radiography is used to document the presence of water, so it is possible to see if the water is in the matrix or in the voids.

In a first test series, mortar specimens (Ø10 mm x 10 mm) made of air-entrained mortar with w/c = 0.40 were tested. The mix design was chosen to simulate the concrete quality often used for bridges in Sweden. First measurements were made for specimens, where the specimens represented different moisture states (dried, seal-cured, capillary saturated, and vacuum saturated mortar, respectively). For CT-scanning, the voxel size was 14 µm and the scanning time was 2 h. for neutron radiography, the voxel size was 7 µm and the scanning time was 3 hours. The results showed that it was clearly possible to identify the air voids and to see if the voids were empty, partly water-filled or completely water-filled.

In a second test, a mortar specimen (same size and mortar composition as in the first series) was tested in a freeze-thaw test, see Figure 8.

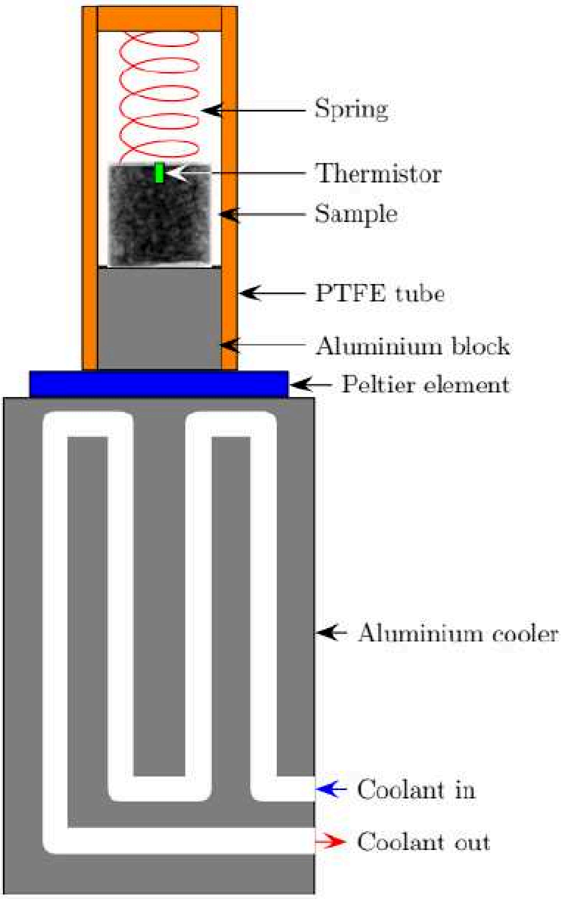

Figure 8 – In-situ freezer that makes it possible to run a freeze-thaw experiment inside the combined CT- and neutron-scanner.

During the test, the top surface of the specimen was exposed to water. With the in-situ freezer, the specimen was exposed to three freeze-thaw cycles. Each cycle went from +20°C to -13°C and back again. The temperature was kept constant during scanning (3 h).

For each identified air void, it is possible to follow the relative water volume.

The experience so far is that the in-situ freezer works very well. The possibility to study the change in water content in the large air voids is promising, and likewise is the possibility to follow the water content in the matrix surrounding the air voids.

Beamline time is not readily available. Therefore, it is important that experiments can be carried out relatively fast. A third test series has been carried out lately. Here w/c = 0.60 was used to make water transport quicker, and the experiment was run with more cycles. However, since data have just been obtained, data processing is “work in progress”, and the results are not ready for presentation yet.

5.2 Stefan Jacobsen: Shape and size of particles scaled from concrete surfaces during salt frost testing and rapid freezing and thawing in water

A number of theories explaining damage development during freeze-thaw action also hint to the thickness of the flakes that scale off. For example, the glue spall theory indicates that the maximum thickness of the flakes will be approx. ¾ of the ice layer thickness [12], i.e. it depends on the test circumstances. Fagerlund [13] predicted, based on theoretical considerations where ice formation causes a hydraulic pressure, that the thickness of the flakes would be approx. three times the critical spacing factor, i.e. the thickness will depend on the concrete. Despite of that the geometry of the flakes can be linked to the damage mechanism, this has caught very little attention before a M.Sc. project carried out at NTNU (described in more detail in [14]). In this project, scaled material from standardised freeze-thaw testing was collected and sieved. A methodology was developed to measure thickness, length, and width of the flakes, see Figure 9.

Figure 9 –Top: Scaled material after sieving. Bottom, left: Flakes mounted on a hollow steel bar with quadratic cross section by double-sided tape. Bottom, right: Digital photos taken perpendicular and parallel to the surface of the steel bar made it possible to determine thickness (T), length (L) and width (W) of the individual flakes accurately. (Illustrations from [14]).

The study included scaled material collected from accelerated freeze-thaw tests conducted with

- different types of concrete
  - two different water/binder ratios
  - concrete with Portland cement as only binder or a combination of Portland cement, fly ash, and condensed silica fume
  - concrete with and without air-entrainment
- different test methods
  - Borås method (test solution: 3 mm 3% NaCl solution; test surface: saw-cut surface)
  - ASTM C666 procedure A (test solution: 3 mm pure water; test surface; cast surface).

Based on the results, the following was concluded:

- Review and experiments show that certain concretes scale a lot during the ASTM C666 rapid freeze-thaw test in water, and in those cases air voids do not protect against scaling.
- The thickness of the flaked particles from both salt scaling and rapid freeze-thaw in water depends on the particle size. There is a tendency that T/(L+W) is larger for smaller particles.
- Before aggregate particles start to peel off, the size dependent thickness is similar for all types of concrete and both test methods.
- The particle size distribution (measured by sieve analysis) peaks in the size interval 1-2 mm for the Borås test with 3 % salt, whereas the particle size distribution is more or less uniform in the interval 0-4 mm for the ASTM C666 Procedure A test.

It is in general difficult to compare the measured flake thicknesses with the thicknesses predicted by models in the literature [12, 13].

6. DISCUSSION AND CONCLUSION

6.1 The temperature curve used for accelerated freeze-thaw testing

The temperature curve was probably the most heavily discussed topic during the workshop. Based on the study presented by Sara Al Haj Sleiman (see Section 4.2), there is no basis for concluding that the temperature cycle of the reference method of CEN/TS 12390-9 results in larger inter-laboratory variation than other temperature cycles. This is because the four laboratories that participated in the round robin test did not fulfill the requirements of the reference method. In the standard, it says that the specified time-temperature curve should be met for all specimens, regardless of their position in the freezing cabinet, and this was not the case. This was probably because the conditions in the freezing cabinet was too cramped, i.e. the specimens were placed too close to each other (Sara showed a photo, where there was almost no space between the specimens). Therefore, another condition of the reference method was not fulfilled either, i.e. that there should be good air circulation in the cabinet. However, it is a weakness of the standard that the temperature variation between specimens is not documented, as the method only requires that the temperature curve is measured for one specimen.

It may make it easier to perform testing that complies with the standard, if the gap between maximum and minimum temperature is reduced. However, it is not desirable to change the specified temperature curve right away, because if a change in temperature curve changes the level of scaling, then all the research that has been done to relate field and laboratory results and to establish acceptance criteria for scaling is no longer useful. In the study presented by Sara Al Haj Sleiman, both the maximum and the minimum temperature were changed, compared to the present reference method of CEN/TS 12390-9. As pointed out by Peter Utgenannt, previous research has demonstrated that the minimum temperature has a significant effect on the outcome of the test [15]. There is no research showing the effect of the maximum temperature. If the effect of the maximum temperature is minor, then a temperature cycle +5°C to -20°C could be considered. However, the choice of maximum temperature will probably influence the part of the temperature curve where the concrete is heated, and as showed by Matthias Müller (see Section 3.2), this is likely to influence the outcome of the scaling test.

In the slab test, the thickness of the air gap between the freezing medium and the covering plastic sheet is important, because the stagnant air works as insulation. Moreover, it is a relevant question how to measure temperature in the freezing medium. Most laboratories use thermocouples, which in many ways is a simple, cheap, and reliable technique. The challenge is that the thermocouple is made up of metal wires, which are heat conducting. Therefore, the temperature that is measured is not always the temperature at the tip of the thermocouple. If the wire in front of the soldering point is exposed to a temperature that is warmer than the temperature at the soldering point, then heat is conducted along the wire, so a higher temperature is measured than intended (and vice versa, if the wire is colder than the soldering point). In the reference method of CEN/TS 12390-9, it is specified that the thermocouple should be placed as shown in Figure 10 (left). Then the 25 mm wire in front of the soldering point (measuring point) is placed both in bulk air in the cabinet, in confined air between plastic cover and freezing medium, and in freezing medium, and the measurement becomes an ill-defined average of the mentioned surrounding temperatures. The outcome of the measurement is better defined, if the thermocouple is placed in a way so a stretch of the wire in front of the soldering point is also placed in the freezing medium, so the temperature of the wire and soldering point is identical. Marianne Tange Hasholt mentioned, that in a student project at DTU, they had observed a relatively large temperature difference for two thermos couples placed like Figure 10 left and right, respectively. At the maximum temperature of the curve, both thermocouples showed approx. 20°C, but at the minimum temperature, the thermocouple placed like the figure to the left showed -20°C, while the thermocouple placed as illustrated to the right showed -13°C.

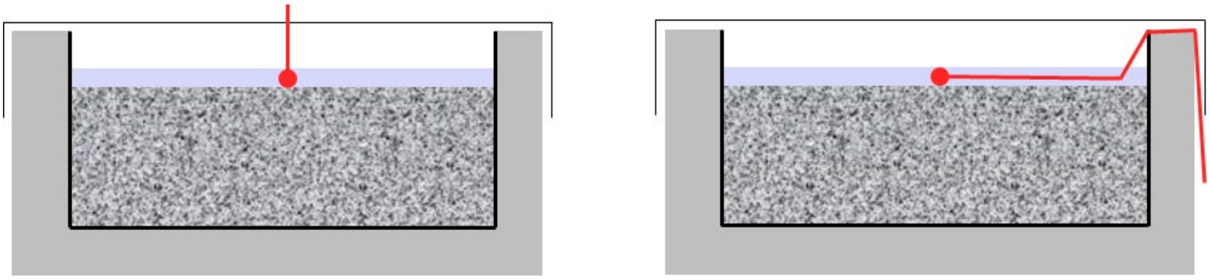

Figure 10 – Position of thermocouple. Left: position as specified in the reference method in CEN/TS 12390-9. Right: position, where a stretch of wire corresponding to at least 50 times the diameter of the thermocouple wire is placed in the freezing medium.

A quick survey among the participants of the workshop showed that approx. 50% in their laboratory place the thermocouple according to Figure 10 (left), and approx. 50% placed the thermocouple according to Figure 10 (right). Considering the effect of the minimum temperature mentioned previously, this may explain some of the inter-laboratory variability. One way of solving it is to specify the air temperature in the cabinet instead of the temperature in the freezing medium. It is suggested that both how to measure temperature in the freezing medium and how to document variations between specimens in the same freezing cabinet become focus points in a round robin test that is planned in Sweden later this year.

6.2 Measurements of internal cracking and accelerated liquid uptake

CEN/TS 12390-9 vaguely mentions that observations of internal cracking shall be reported. Furthermore, accelerated liquid uptake during the test is clearly related to internal cracking during the test [16-18], and the acceleration of surface scaling can be related to internal cracking. Therefore, the test method needs to be improved on this with descriptions of methods for measurements of internal damage as length change done on invar studs fixed in a standardized way, and for measurements of liquid uptake with a butyl-type (non-absorptive) preparation system. This will increase the credibility of the test and give more insight on how the test works.

6.3 Combined action: Carbonation and freeze-thaw

Most concrete structures exposed to freezing and thawing are outdoor structures that are also exposed to carbonation because the surface is in contact with atmospheric air. In this light, the Swedish approach is interesting, where the national annex to EN 206 now includes testing, where the concrete during the test procedure is both exposed to carbonation and freeze-thaw action. Among other things, it is interesting if the experience with the test procedure that will be gained in the years to come shows a difference in response, depending on whether or not the concrete includes supplementary cementitious materials such as fly ash or slag.

Not all laboratories have climate cabinets with increased CO_2 contents, compared to atmospheric air. The study presented in Section 3.3 indicated that chloride ingress may provoke an internal carbonation attack. If this is correct, maybe the carbonation effect can be obtained by replacing the capillary saturation by pure water with a period of capillary saturation with NaCl solution (or another solution containing chloride ions). In a study by Ahani and Nokken [19] it is shown that re-saturation (i.e. capillary suction) with NaCl solution has a similar effect as carbonation: For concrete with 100% Portland cement, 7 days of re-saturation with NaCl reduced the amount of scaling in the following freeze-thaw test, whereas the amount of scaling increased for concrete with 75% Portland cement and 25% fly ash. However, more research is needed to find out if re-saturation with NaCl solution can be used instead of exposure to CO_2. In all cases, it is interesting to come to know more about the importance of if capillary suction is performed with pure water or NaCl solution. This is one of the differences between the slab test (reference method of CEN/TS 12390-9) and the CDF test (alternative method of CEN/TS 12390-9). As mentioned in Section 3.2, we need more knowledge about the importance of conditioning and pre-saturation prior to freeze-thaw action, especially for concrete with supplementary cementitious materials.

6.4 Future research

The workshop discussion revealed that there are things that we simply do not know today, and there are – at least – the following needs:

- *Numerical modelling*: Both Abdul Faheem and Sara Al Haj Sleiman touched on modelling in their presentations (though their reflections on modelling have not been mentioned in this workshop summary). It would be convenient as well as time and resource saving, if some of the physical freeze-thaw testing could be replaced by testing in a virtual laboratory based on numerical models.
- *Frost damage mechanisms*: We need to know more about the actual damage mechanisms and relevant combined action (e.g. carbonation and freeze-thaw and freeze-thaw deicing attack), especially if we want to establish numerical models. The numerical models can only advance from the state of mathematical fitting (empirical modelling), if the damage mechanisms are known and there are experimental data of preferably different lab tests and experience from field studies for model validation.
- *Air voids*: Recent research [18] showed how air voids work protectively on all the three measurable damage parameters in CEN/TS 12390-9: accelerated liquid uptake, scaling and internal cracking. However, traditional air entrainment has become increasingly difficult with new SCMs while technology for entraining and measurement of air voids has made quantum leaps forward (SAP/"solid air", CT-scanning, etc.). Together with the question about mix

design for frost durable concrete without air voids, this warrants further research work on mix design and part material effects on performance test results.

- *Field studies*: In the studies presented in Sections 2.1 and 2.2, actual field exposure was quantified. In many countries, very little is known about the actual exposure and to which extent different concrete qualities suffer from frost damage in real structures. Compared to well-defined storage conditions in any of the lab tests according to CEN/TS 12390-09 the performance of the concrete in real structures is strongly influenced by e.g. curing-method and -duration on site. Variations of storage regimes of lab specimen already give hints to that strong influence on concrete performance. The freeze-thaw resistance of curing sensitive concrete is more likely to be negatively influenced by that in lab tests and at real structures. This is important background knowledge, if it should be possible to link performance in laboratory tests to the performance in real structures. Moreover, the present situation should be known, before it is possible to forecast how climate change will influence future performance.

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