



Alternative salt frost scaling test procedure for exposure class XF2

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Abstract Concrete structures in cold and moderate climates are often exposed to a combined salt frost attack, which can cause scaling damage. Consequently, numerous test procedures were developed to determine the resistance of concrete compositions against this kind of attack. These tests mimic a severe attack with high levels of saturation, which is typical for horizontal concrete elements or structures, such as concrete pavements. Very few approaches exist for testing the salt frost scaling resistance of concretes for vertical concrete elements like bridge columns, which are subjected to only moderate levels of saturation during salt frost attack (exposure class XF2). As such elements usually didn't show notable scaling damage in practice, this hasn't posed a problem until recently. However, the increasing use of low carbon cements with high clinker substitution rates might increase the vulnerability against salt frost exposure for such concrete elements to some extent. To ensure adequate durability it is desirable to determine the performance of the concrete in an actual test procedure instead of relying on descriptive criteria alone. Thus, a less

severe test method was developed, which is based on the CDF test. It was shown that the modified test procedure can distinguish correctly between different concrete qualities. Furthermore, the applicability of an acceptance criterion for the method was investigated.

Keywords Salt frost attack · Concrete · Test method · CDF · XF2 · Low temperature differential scanning calorimetry · Scaling · Adapted CDF test · Vickers hardness

1 Introduction

In regions with cold or moderate climates concrete structures can be subjected to the combined action of moisture, frost and salt [1]. Apart from marine structures [2] mostly traffic structures are affected by this attack, as de-icing salts are applied in cold weather to remove ice and snow from pavements [3]. Predominant appearance of salt frost damage is surface scaling—the removal of small flakes of hardened cement paste or mortar from the concrete surface [4].

For concretes with high clinker contents a good salt frost scaling resistance can easily be achieved by applying descriptive criteria regarding concrete composition and curing procedures [5]. The descriptive concept assumes that concretes with comparable composition also perform in a similar way with regard

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to mechanical properties and durability. However, that concept cannot be applied to modern concretes with increasingly lower clinker contents [6]. Compliance with descriptive rules alone does no longer guarantee a good salt frost scaling resistance [1, 7]. Consequently, many efforts were and are being made to implement performance concepts, in which the properties of concrete are assessed with durability tests in the laboratory.

For frost and salt frost attack the European concrete standard DIN EN 206-1 [8] contains four different exposure classes, which define different levels of severity for frost exposure (c.f. Table 1).

Performance test methods were mostly developed for the severe exposure classes XF3 and XF4. For salt frost scaling resistance (XF4) the Slab test and the CDF test (Capillary suction of de-icing salt solution and freeze thaw test) according to DIN/CEN TS 12390-9 [9] are predominantly used in Europe.

The intensity of these test methods is so strong, that extremely high levels of damage occur, when typical XF2 concrete compositions are tested. This is due to the fact, that (in Germany) XF2 concretes are usually produced without air-entrainment. They consequently possess a much weaker salt frost scaling resistance than air-entrained XF4 concretes. Hence, less severe procedures should be developed for XF2 concretes [10].

An exception is the Swiss test method SIA 262/1 [11] for salt frost scaling resistance, which is applied for XF4 and XF2 concretes alike. For XF4 a stricter acceptance criterion is used than for XF2. However, this method can only be used for air-entrained concretes, as very strong scaling and extreme test scatter occur in concretes without air voids.

A key difference of XF2 conditions to XF4 is the lower level of saturation. In deeper layers (e.g. 3 cm below surface) of XF2 concrete elements a high degree of saturation is never achieved [12]. Consequently, no internal damage should occur in such concrete elements. A test method for XF2 should reflect that.

To our knowledge only one test procedure was developed explicitly for testing XF2 concretes. This procedure was published by the German Federal Highway Research Institute (BAST) in 2007 [10]. It is based on the CDF test, which was reduced in intensity by increasing the minimum temperature from $-20\text{ }^{\circ}\text{C}$ to $-10\text{ }^{\circ}\text{C}$ and by reducing the number of freeze–thaw cycles from 28 to 14.

Though the application of this method is now required by some tendering authorities, it is not yet generally accepted or standardized. Comparative studies have shown that this adjusted CDF test method has a relatively high test scatter and can sometimes lead to incorrect evaluations, especially if clinker reduced cements are used [13]. The increase in the minimum temperature to $-10\text{ }^{\circ}\text{C}$ must be critically questioned, since the minimum temperature of $-20\text{ }^{\circ}\text{C}$ was considered an essential prerequisite for low test scatter in the original CDF method [14].

This results in the problem that, on the one hand, descriptive regulations are increasingly no longer sufficient to guarantee salt frost scaling resistance in exposure class XF2. On the other hand, there is no suitable test method available to evaluate the performance of such concretes.

Consequently, it was the aim of a research project to develop an alternative salt frost scaling test procedure

Table 1 Exposure classes for frost and salt frost attack according to DIN EN 206-1 [8]

Exposure class	Description	Typical elements
XF1	Moderate level of saturation, no de-icing salts	Exterior concrete elements
XF2	Moderate level of saturation, with de-icing salts	Concrete elements in the spray mist of traffic surfaces treated with de-icing agents or in the spray mist of seawater
XF3	High level of saturation, no de-icing salts	Open water tanks Concrete elements in the tidal zone of fresh water
XF4	High level of saturation, with de-icing salts	Traffic surfaces treated with de-icing agents Concrete elements in the tidal zone of sea water



for XF2 concretes based on the CDF method. Relevant findings are reported in the present paper.

2 Materials and methods

2.1 Materials

2.1.1 Concrete

Two different concrete qualities (A and B), which differ in water/cement-ratio, were used for the investigations. An important performance criterion for the new test procedure was its ability to correctly distinguish between those two qualities. Quality A was intended to fulfil all requirements for non-air entrained concrete for XF2 according to DIN EN 206-1 [8] and DIN 1045-2 [15]:

- Maximum water/cement-ratio: 0.50
- Minimum compressive strength class: C35/45
- Minimum cement content: 320 kg/m³

C35/45 signifies a characteristic compressive strength of 45 MPa determined on concrete cubes with the edge length of 15 cm. 6 MPa was defined as allowance of tolerance, resulting in a target minimum strength for quality A of 51 MPa. No supplementary cementitious materials (apart from those already present in the cements) were included. Ten cements with different clinker replacement levels were used in total. Table 2 gives an overview on the cements

including their standard 28 day (28 d) compressive strength according to DIN EN 196-1 [16] and their approximate composition according to DIN EN 197-1 [17]. “C-R” was used as reference cement.

Depending on the standard 28 d compressive strength of the cements the w/c-ratio had to be reduced for some concretes to achieve the target strength class C35/45 for quality A.

Concrete quality B was defined by increasing the w/c-ratio of quality A by 0.10. A cement content of 320 kg/m³, natural sand 0–2 mm and gravel 2–8 mm, 8–16 mm were used for both qualities. Furthermore, the particle size distribution of the natural sand was optimized with small amounts of quartz powder and sand 1–2 mm. The target workability was a flow spread of 420–480 mm (Workability class F3, measured according to DIN EN 12350-5 [18]). A PCE based superplasticizer was used to achieve this workability in quality A. In quality B superplasticizer was not necessary in most cases, as the target workability was met (or slightly exceeded) due to the increased w/c-ratio. Table 3 shows the composition for the two concrete qualities based on the reference cement. The compositions for the other concretes were derived from these by considering the different w/c-ratios, cement densities and superplasticizer dosages. The respective w/c-ratios are given in Table 4.

The concrete was produced in a compulsory mixer. Ten minutes after the end of mixing, workability and temperature of the concretes were tested and the test

Table 2 Cement types with compressive strength and composition

No	Cement type	28 d compressive strength (MPa)	Main constituents* (wt.%)
C-R	CEM I 42.5 R	59.3	OPC clinker (95–100)
C1	CEM II/A-S 42.5 R	55.1	OPC clinker (80–94), GGBFS (6–20)
C2	CEM II/B-S 42.5 N	53.0	OPC clinker (65–79), GGBFS (21–35)
C3	CEM II/A-LL 42.5 R	53.9	OPC clinker (80–94), LL (6–20)
C4	CEM II/A-LL 32.5 R	46.2	OPC clinker (80–94), LL (6–20)
C5	CEM II/B-LL 32.5 R	40.9	OPC clinker (65–79), LL (21–35)
C6	CEM III/A 42.5 N-LH	48.2	OPC clinker (35–64), GGBFS (36–65)
C7	CEM II/B-M (S-LL) 42.5 N	43.2	OPC clinker (65–79), GGBFS + LL (21–35)
C8	CEM I 52.5 R	66.0	OPC clinker (95–100)
C9	CEM II/A-M (V-LL) 42.5 N	51.0	OPC clinker (80–88), V + LL (12–20)

*Ordinary Portland cement (OPC), ground granulated blast furnace slag (GGBFS), limestone powder (LL); siliceous fly ash (V)

specimens for the hardened concrete tests were produced. Compaction was carried out on a vibrating table according to DIN EN 12390-2 [19]. The temperature in the laboratory, of the equipment, of the starting materials and of the fresh concrete was 20 ± 2 °C. The specimens were stored at 20 °C and a relative humidity of more than 95% until demoulding after 24 h. Subsequently, the specimens were stored under water at 20 °C until they were 7 days old, then in a standard climate of 20 °C and 65% relative humidity. Compressive strength was determined according to DIN EN 12390-3 [20] on cubes ($15 \times 15 \times 15$ cm³). Table 4 shows the water/cement-ratios and the resulting compressive strength for all concretes at an age of 28 d.

Table 3 Reference concrete compositions

Material	Quality A Content (kg/m ³)	Quality B
CEM I 42.5 R (C-R)	320	320
Water	160	192
Natural sand 0–2 mm	530	506
Gravel 2–8 mm	482	461
Gravel 8–16 mm	748	715
Sand 1–2 mm	74	71
Quartz powder	28	27
Superplasticizer (PCE)	1.12	–

In two cases the targeted minimum strength of 51 MPa for quality A was not met. As the difference to target was only small, the compositions were left unchanged. Concrete C-R was used as reference concrete to test modifications of the CDF procedure.

2.1.2 Hardened cement paste

Additional experiments were carried out on hardened cement paste (HCP) samples. The w/c-ratios were identical to the respective concrete compositions (c.f. Table 4). Fresh cement pastes were produced by mixing cement and water for 2 min with an electrical hand mixer at high intensity. The fresh paste was filled in a 50 ml test tube ($\varnothing 30$ mm), which was sealed airtight. The tube was slowly rotated for four hours to avoid segregation of the solids. Afterwards the tube was stored in water saturated atmosphere at 20 °C. At an age of 7 days, discs of hardened cement paste with a thickness of ~ 1.5 mm were cut from the tube and subsequently stored in standard climate (20 °C; 65% relative humidity) until the age of 28 days.

From 28 to 35 days the discs were stored in 3.0 wt.% NaCl solution for one week. The pre-storage conditions of these samples were meant to mimic the conditions for the concrete surface layer before a salt frost scaling test according to the CDF method. The specimens were then used to characterise the freezing behaviour of the solution in the pore structure of the HCP. Further details regarding sample preparation are

Table 4 Water/cement-ratios and 28 day compressive strength (average of 3 specimens) of concretes

No	Quality A		Quality B	
	w/c-ratio	28 d compressive strength (MPa)	w/c-ratio	28 d compressive strength (MPa)
C-R	0.50	51.5	0.60	36.3
C1	0.50	58.2	0.60	45.8
C2	0.50	54.0	0.60	44.3
C3	0.50	49.0	0.60	36.0
C4	0.45	52.6	0.55	40.4
C5	0.40	61.4	0.50	44.5
C6	0.45	58.3	0.55	33.7
C7	0.50	49.7	0.60	34.7
C8	0.45	62.5	0.55	53.0
C9	0.45	58.3	0.55	43.5



given in the description of the test method (c.f. Section 2.2).

2.2 Methods

2.2.1 Experimental program

The experimental program was divided into two main parts (c.f. Fig. 1). Aim of Part 1 was to modify parameters of the CDF method, that allow a reduction of its severity. The standard CDF test was starting point and main reference for the experiments.

Concrete qualities A and B based on CEM I 42.5 R were used. Quality A fulfilled descriptive criteria for XF2. In quality B the water/cement-ratio was increased by 0.10, to provoke a reduction in salt frost scaling resistance to quality A.

The following criteria were defined to assess the suitability of the CDF test modifications:

- clear differentiation between concrete qualities A and B with regard to scaling
- appropriate scaling intensity below 1.500 kg/m² for quality A
- only minor internal damage (remaining E-modulus above 75%)
- practical feasibility
- reproducibility

The “CDF-method XF2 (BAsT)” [10] was used here as a second reference. As a result of Part 1, a promising, less severe modification of the CDF method was identified.

This modified CDF test was then applied in the second part of the experimental program to investigate the salt frost scaling resistance of different concretes. Again, concrete qualities A and B were used for the investigations, but this time in combination with a wide range of different cements.

The concrete tests in part 2 were accompanied by experiments on HCP samples, to provide a basis for the assessment of the salt frost scaling tests. In a concrete without air voids, salt frost scaling resistance is strongly influenced by its capillary porosity [21]. Since scaling is a surface phenomenon, the microstructure at the concrete surface zone is of particular importance. HCP was chosen as a model material to characterize the microstructural properties, as these are mainly influenced by the cement (composition, properties), by the w/c-ratio and by the pre-storage conditions (age, curing, carbonation).

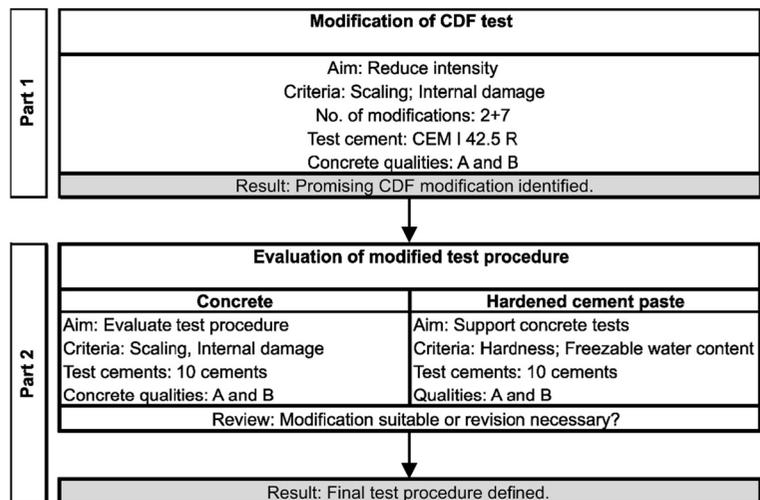
Following the same criteria as in Part 1, the suitability of the modification was evaluated. Additionally, the concrete results were also assessed in relation to the microstructural properties of the HCP and to the concrete compressive strength, which represents a descriptive criterion for XF2 concretes.

Based on the final evaluation the modifications to the CDF test were revised and a test procedure was defined.

2.2.2 Standard CDF procedure

Basis for the experiments was the CDF test, which is standardized in DIN CEN/TS 12390-9 [9]. Five

Fig. 1 Schematic overview on experimental program



concrete slabs per test served as specimens, which were produced by dividing a 150 mm cube mould with a PTFE plate placed in the centre. The surface facing the PTFE plate was used as test surface. At the end of pre-storage, the specimen lateral surfaces were sealed with aluminium-laminated butyl tape (c.f. Fig. 2a, b).

At 28 days, the 7-day pre-saturation period began, in which the specimens take up 3.0 wt.% NaCl solution by capillary suction at +20 °C. Thereafter, the specimens were subjected up to 28 freeze–thaw cycles (ftc), also with 3.0 wt.% NaCl solution. It is a specific characteristic of the CDF procedure that the test specimens are placed upside down on the test solution in a stainless-steel container. The containers are positioned in a so-called CDF-chest that is filled with a cooling liquid for temperature control during the freeze–thaw cycles (c.f. Fig. 2a).

The temperature profile for a single freeze–thaw cycle in a standard CDF test (among others) is shown in Fig. 3. The scaled material was collected after 4, 6, 14 and 28 freeze–thaw cycles. A scaling intensity of 1.500 kg/m² after 28 freeze–thaw cycles is typically applied as standard acceptance criterion [22].

In addition, the internal damage was determined as change in relative dynamic modulus of elasticity by measuring the ultrasonic transit time according to CEN/TR 15177 [23]. Deviating from the standard the ultrasonic probes were coupled directly to the specimens instead of measuring in a water bath (c.f. Fig. 2b). Thus, a leaching of de-icer solution from the concrete was avoided. When the relative modulus of

elasticity remains above 75%, the concrete is classified as internally undamaged [22].

2.2.3 CDF method XF2 (BAsT)

Furthermore, the “CDF method XF2 (BAsT)” [10, 24] was used as a second reference procedure. It is based on the CDF test (2.2.2), which was reduced in intensity to be applicable for XF2. This was done by:

- increasing the minimum temperature from -20 to -10 °C
- reducing the number of freeze–thaw cycles from 28 to 14.

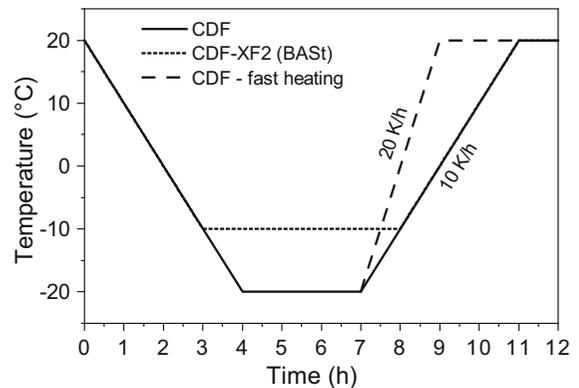
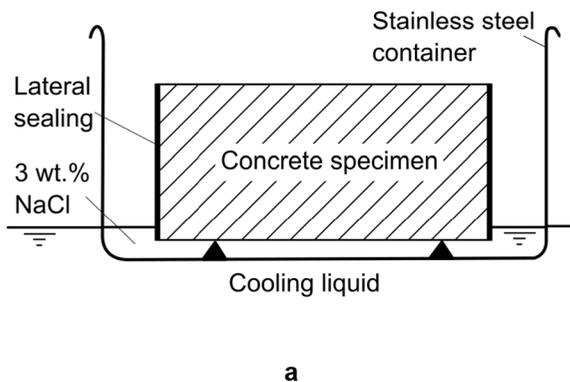
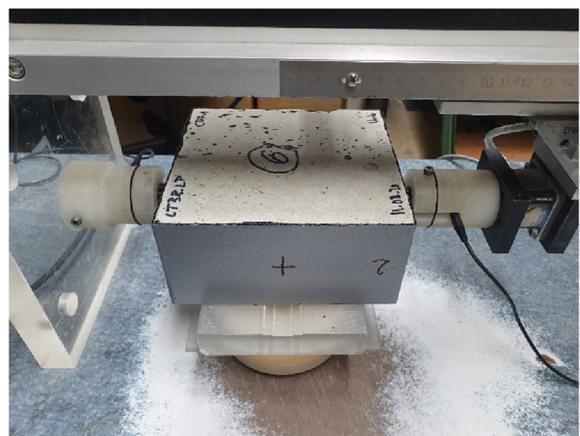


Fig. 3 Temperature profiles for a single freeze–thaw cycle in the CDF test, the modified CDF-test XF2 (BAsT) and of the modification “fast heating”



a



b

Fig. 2 CDF test setup: **a** Concrete specimen standing in test solution and **b** CDF-test specimen during ultrasonic transit time measurements with sealed lateral surfaces (right)

The temperature profile of that method is also shown in Fig. 3. A value for scaling of max. 1.000 kg/m² was later proposed as an acceptance criterion [24]. No criterion for internal damage exists for that method.

2.2.4 Modifications to the CDF method to reduce its severity

Various parameters of the standard CDF test procedure were changed to reduce its severity. The two concretes (A and B) based on CEM I 42.5 R were used to evaluate the effect of the modified parameters. After a promising modification was identified, its suitability was assessed with the complete range of concrete compositions. Table 5 gives an overview on the investigated modifications.

2.2.4.1 Ice layer thickness (ILT) According to the Glue Spall theory, a reduction of the ice layer thickness in a salt frost scaling test should reduce the scaling intensity [25–28]. In the CDF test the standard ice layer thickness is 5 mm [9]. By using rod shaped spacers with a diameter of 1 mm underneath the concrete specimen, the ice layer thickness was reduced accordingly to 1 mm.

2.2.4.2 Intermittent drying phases Under practical conditions intermittent dry periods occur, which can lead to changes in moisture and salt distribution in concrete [29]. Here, intermittent drying was primarily used to reduce the degree of saturation in the concrete specimens during the freeze–thaw exposure in the

CDF test. The following variations were tested (for more details see [30]):

- Drying for 1 h in a heating cabinet at 40 °C after every two freeze–thaw cycles, except on weekends.
- Drying for 2 h in a heating cabinet at 40 °C after every two freeze–thaw cycles, except on weekends.
- Drying for 24 h in standard climate after every two freeze–thaw cycles. This variation was only carried out until the 14th freeze–thaw cycle, to keep the overall test duration under 2 months.

2.2.4.3 Fast heating According to the micro ice lens theory [31] the moisture uptake during a freeze thaw cycle occurs during the thawing phase. Consequently, it was assumed, that a shorter heating phase might reduce the moisture uptake and thus the damage intensity. To our knowledge no systematic study on the effect of the thawing rate was conducted so far. The shorter heating phase was realized by doubling the heating rate (20 K/h instead of 10 K/h, c.f. Fig. 3). As a side effect the faster heating gradient also leads to a prolongation of the duration above 0 °C in the freeze–thaw cycle.

The faster heating was also combined with reduced ice layer thickness (1 mm), which should ensure a more effective heat transfer to the concrete. It was furthermore combined with a test solution with 6.0 wt.% NaCl, as NaCl concentrations above 3.0 wt.% NaCl are known to reduce scaling intensity [32].

Table 5 Investigated modifications of the CDF test

No	Description/modification
R1	Standard CDF test
R2	CDF test XF2 (BAST) [10, 24]
M1	Ice layer thickness (ILT) reduced to 1 mm
M2	Intermittent drying phase; 1 h at 40 °C, every two freeze–thaw cycles except on weekends
M3	Intermittent drying phase; 2 h at 40 °C, every two freeze–thaw cycles except on weekends
M4	Intermittent drying phase; 24 h in standard climate after every two freeze–thaw cycles
M5	Fast heating (20 K/h)
M6	Fast heating (20 K/h) combined with ice layer thickness of 1 mm
M7	Fast heating (20 K/h) combined with 6 wt.% NaCl as test solution

2.2.5 Characterisation of hardened cement paste

The HCP was characterized by the amount of freezable water in the range to $-20\text{ }^{\circ}\text{C}$. According to Sant et al. [33], the pore solution in interconnected capillaries freezes completely in the temperature range above $-23\text{ }^{\circ}\text{C}$. The pre-storage of the HCP in 3 wt.% NaCl solution should have caused an additional reduction of the freezing point by approximately 1.8 K. However, as the minimum temperature in the CDF test is $-20\text{ }^{\circ}\text{C}$, that temperature was applied as minimum temperature for determining the relevant amount of freezable water.

The experiments were conducted at a specimen age of 35 days with a Netzsch Differential Scanning Calorimeter (DSC 214 Polyma) in combination with an intercooler, which can realize a minimum temperature of $-70\text{ }^{\circ}\text{C}$. A small sample of approximately $3 \times 3\text{ mm}^2$ was obtained from each HCP disc. The HCP sample was surface dried with a paper towel and inserted into an aluminium crucible, which was then sealed. The measurements were conducted in the temperature range from $+20$ to $-20\text{ }^{\circ}\text{C}$. The necessary heat of fusion for melting the frozen pore solution during the heating phase was used to determine the ice content in the samples. More details regarding the measurement and calculation of the ice content can be found in [34].

Vickers hardness was used as a second characteristic for the microstructure of the HCP. General principles of Vickers hardness testing can be found in [35]. The measurements within the scope of this project were carried out on the hardened cement paste at the age of 28 days with a test load of 10 kg or 98.1 N at a load duration of 15 s. The diagonals of the pyramid indentations were then measured by light microscopy and converted into Vickers hardness. More details can be found in the extensive report on the project [30].

3 Results and discussion

3.1 Part 1: modification of the CDF test

3.1.1 Damage intensity after 28 freeze–thaw cycles

As described in Sect. 2.2.1, starting point for all modifications was the CDF procedure with 28 freeze–

thaw cycles. Consequently, all modified CDF tests were carried out with 28 freeze–thaw cycles as well. However, it was found that irrespective of the particular changes in the parameters of the CDF procedure, the extent of damage (scaling and internal damage) after 28 freeze–thaw cycles exceeded the desired damage level significantly (c.f. Table 6). Accordingly, it was decided to use the results after 14 freeze–thaw cycles (ftc) for the further evaluation and to limit the maximum number of freeze–thaw cycles for further tests to 14.

3.1.2 Damage intensity after 14 freeze–thaw cycles

Figure 4a shows the accumulated scaling after 14 freeze–thaw cycles for the different modifications of the CDF test.

With regard to scaling two criteria are important:

- 1) Quality A and B must clearly be distinguished by the test.
- 2) The scaling intensity for quality A should not exceed established damage levels for salt frost scaling resistant concrete (e.g. 1.500 kg/m^2).

It was found that with one exception (R2) all variations could clearly distinguish between concrete quality A and B. The level of scaling for quality A was below 1.500 kg/m^2 for all tests. A notable reduction in the severity of the test was achieved by modifying the CDF test. The modifications (M5–M7) with increased heating rate were most effective in reducing the level of damage. A particularly favourable aspect of this form of adaptation is that the testing procedure does not increase in complexity compared to the standard CDF procedure. The introduction of intermittent drying phases (M2–M4) would be associated with a considerably higher testing effort.

Precision is of particular importance for the selection of a suitable modified test procedure. The coefficient of variation for the accumulated scaling after 14 ftc for each test modification (series of 5 specimens) can be used as a first indication of this (c.f. Fig. 4b).

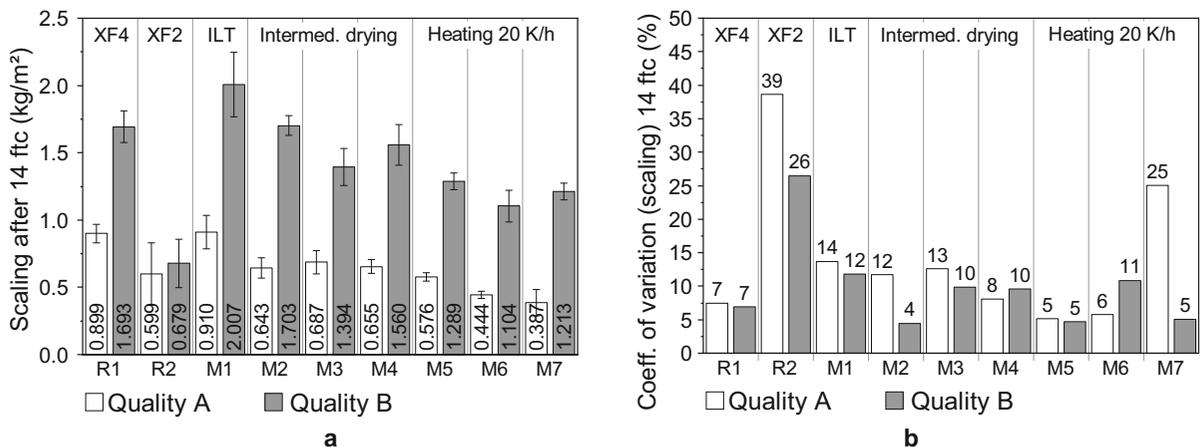
With the standard CDF method (R1) a very high precision with regard to scaling intensity was achieved, which is in line with experiences with this test procedure [36]. The “CDF test XF2” (R2) demonstrated a comparably high scatter with coefficients of variation of 39% and 26% for quality A and



Table 6 Results of the modified CDF tests after 28 freeze–thaw cycles

No	Accumulated scaling after 28 ftc (kg/m^2)		Remaining E-Modulus after 28 ftc (%)	
	Quality A	Quality B	Quality A	Quality B
R1	3.336	4.508	40	24
R2	4.547	2.920	44	34
M1	3.798	5.990	39	19
M2	3.219	4.625	50	25
M3	2.699	3.780	44	33
M4	0.655*	1.560*	86*	62*
M5	2.907	2.620	61	70
M6	1.692	2.635	60	53
M7	3.391	4.953	63	44
Criterion	≤ 1.500		≥ 75	

*14 ftc

**Fig. 4** **a** Accumulated scaling and **b** coefficient of variation for scaling after 14 freeze–thaw cycles for different modifications of the CDF test

B. With only one exception significantly lower scatter was observed for the modifications M1–M7, in the order of magnitude below 15%.

A second important aim for reducing the severity of the CDF procedure was to avoid internal damage in the concrete, which is determined as remaining dynamic elastic modulus (c.f. Fig. 5a). The lower the remaining elastic modulus, the higher is the internal damage. A value below 75% signifies that the concrete is internally damaged.

For concrete quality A the highest internal damage occurred for the reference CDF tests (R1, R2) with a remaining dynamic elastic modulus of 66% and 75%. The damage intensity was reduced by the changes in the CDF procedure, resulting in values $\geq 75\%$ for

quality A (M1–M7). The internal damage intensity after 14 ftc was thus in the intended range.

As expected, the damage level for quality B was higher than for quality A. As a general trend, though with one exception, the internal damage in quality B was also reduced by the modifications of the CDF test. The highest efficiency in suppressing internal damage was achieved with two variations with increased heating rate (M5, M6).

In order to understand the effects of the modifications on the damage it is also important to consider their impact on the liquid uptake during the freeze–thaw test. Figure 5b shows the respective results.

Interestingly the liquid uptake does not fully reflect the results for scaling and internal damage. Though a

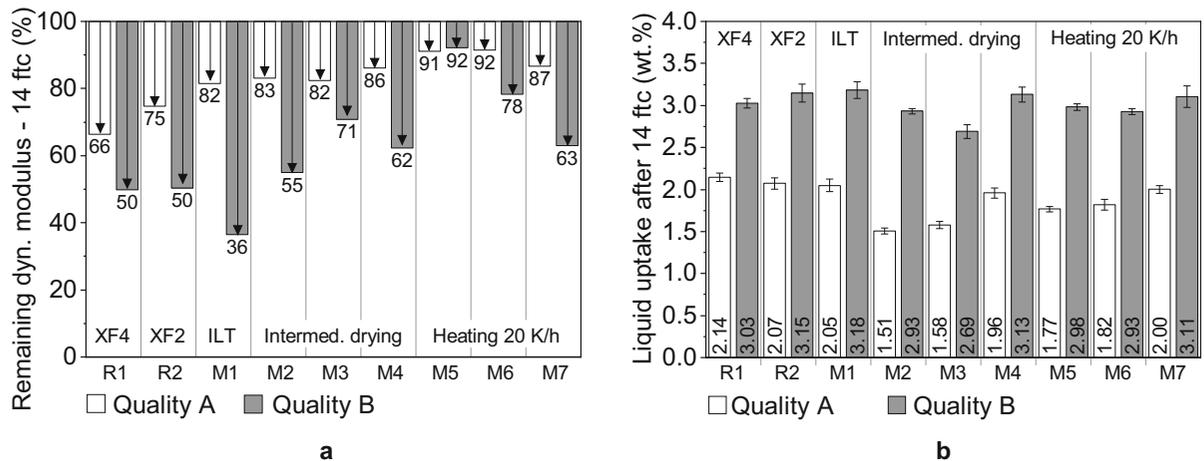


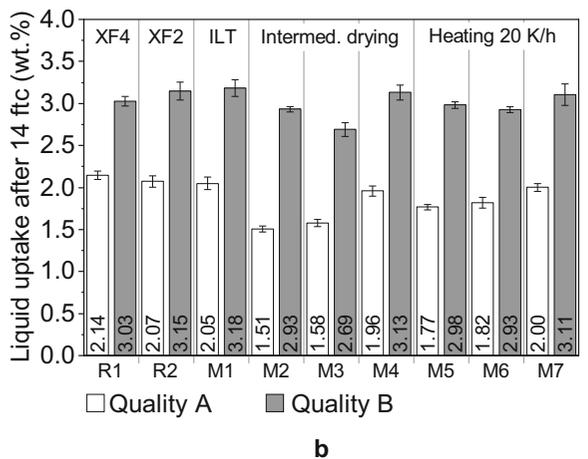
Fig. 5 a Internal damage and b liquid uptake after 14 freeze–thaw cycles for different modifications of the CDF test

strong reduction of the liquid uptake was achieved in two modifications with intermediate drying phases (M2, M3), the general impact of the modifications was rather low. Still, a small reduction of liquid uptake was achieved in most cases.

Previous studies have shown, that typically no correlation should be expected between liquid uptake and salt frost scaling damage, supposedly because the latter is a surface phenomenon [37]. However, a correlation between liquid uptake and internal damage has been observed [38]. Typically, only the properties of the concretes were varied in such studies, but not the parameters of the test methods.

With regard to the investigated modifications of the CDF method it can be assumed, that the reduction of the damage intensity is not related to the bulk liquid uptake, except for the modifications with intermediate drying phases.

The damage mitigating effect of the faster heating rate can possibly be attributed to the longer residence time at temperatures above the freezing point during each freeze–thaw cycle (c.f. Fig. 3). Consequently, there is a longer time period in which diffusion processes can lead to a more uniform distribution of moisture inside the concrete. Thus, critical degrees of saturation at the specimen surface as well as deeper inside the specimens were possibly avoided more effectively. However, as it was not the main aim of the study to investigate the underlying damage mechanisms, no further experiments were carried out regarding the moisture distribution in the specimens.



Ultimately, M5 was identified as the most suitable modification with appropriate damage intensity and a very low coefficient of variation of 5% for both concrete qualities. In order to confirm the results with the M5, three repetitions with this test procedure were carried out. In the repetitions M5-1 to -3 an adequate precision was achieved with coefficients of variation in the range between 3 and 10% for each test. When considering the whole series of four tests with M5 a coefficient of variation of 10% (Quality A) and 8% (Quality B) was obtained (for detailed results see [30]). This variation is acceptable when compared to the precision data for the standard CDF as given in DIN CEN/TS 12390-9 [9]. There, the following coefficients of variation are given for a scaling intensity of 1.5 kg/m²—repeatability v_r of 14% and reproducibility $v_R = 29\%$.

3.2 Evaluation of the modified CDF method (M5)

3.2.1 Performance of concretes with different cements

The tests were carried out with the modified CDF method (M5). 10 cements (9 + 1 reference cement) with different clinker contents, main constituents and strength classes were used. Again, two concrete compositions (quality A and quality B) were tested per cement, which differed in their w/c-ratio by 0.10. Figure 6a gives an overview of the accumulated scaling after 14 ftc for the different concretes.

For each cement, the different concrete qualities are accurately differentiated with the modified CDF method. On average the scaling intensity for quality B was about 1.5 times higher than for quality A. However, the differences between the various cements are very high and clearly exceed the influence of the w/c-ratio (quality A vs. B).

The use of the clinker-efficient cements led to an increase in the scaling level after 14 ftc compared to Portland cement. This is not an unusual behaviour. Comparable experiences are available from the testing of air-entrained concretes with the “regular” CDF method (e.g. in [1]).

The overall scaling level with maximum values up to 5.0 kg/m² for quality B were regarded as too high for the test method. It was therefore assessed, whether with a limitation to only 6 freeze–thaw cycles the necessary reduction in damage intensity could be achieved, while retaining a good differentiation of the two concrete qualities. Figure 6b shows that the limitation to 6 ftc was effective in achieving this goal.

After 6 ftc the measured difference between the concrete qualities was even more distinct than after 14 ftc, with a scaling intensity of quality B about 1.9 times higher than for quality A. The variation of the cements still caused notable differences in the scaling intensity, though these were not so pronounced as after 14 ftc. Another positive effect of the reduced number of freeze–thaw cycles was the total prevention of internal damage. After 6 ftc the remaining dynamic modulus of elasticity was about 100% in most cases,

with a minimum value of 95% in one case (not displayed here, for detailed results see [30]).

With regard to the applicability of only 6 ftc the precision must also be considered. Thus, the scatter was calculated for each series of 5 specimens. In Fig. 7 the respective coefficient of variation is plotted over the average amount of scaling for the individual series after 6 and after 14 ftc.

A reduction in the coefficient of variation occurs with increasing scaling level. Considering, that the coefficient of variation is calculated by dividing the standard deviation by the average value, this trend was to be expected. Accordingly, lower coefficients of variation tend to be obtained for the results after 14 ftc. For scaling levels above 2.0 kg/m², the coefficient of variation is always less than 10%. Accordingly, in terms of precision, testing with 14 ftc might seem to be preferable.

For a test with only 6 ftc it is advisable to compensate for the increased scatter by a higher number of specimens or by multiple testing. Due to the short test duration, this would only cause a small additional effort compared to the test with 14 ftc.

3.2.2 Comparison to microstructural properties of HCP

A further opportunity to evaluate the modified CDF method is to relate the measured concrete scaling intensity to the microstructural properties of the HCP. Two indicators are applied here for the microstructure

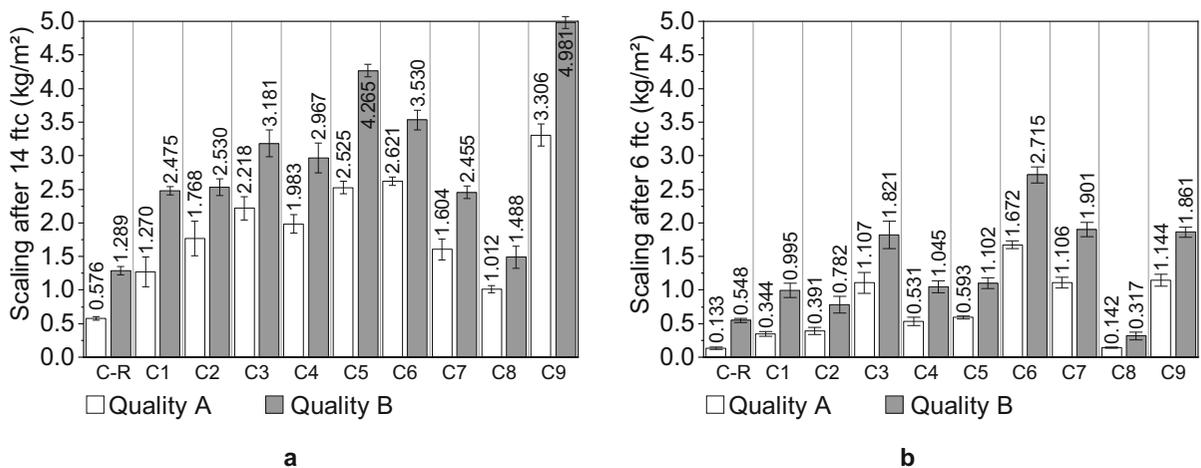


Fig. 6 **a** Accumulated scaling after 14 freeze–thaw cycles and **b** after 6 freeze–thaw cycles (right) with the modified CDF test on different concretes

of the HCP in comparison to the scaling intensity in the modified CDF test after 6 and 14 ftc (c.f. Fig. 8):

- Capillary porosity (expressed as freezable water at $-20\text{ }^{\circ}\text{C}$)
- Vickers hardness

For the compositions with cement C9 no results are shown, as this cement was included in the study at a very late stage, after the measurements on the HCP were already finished.

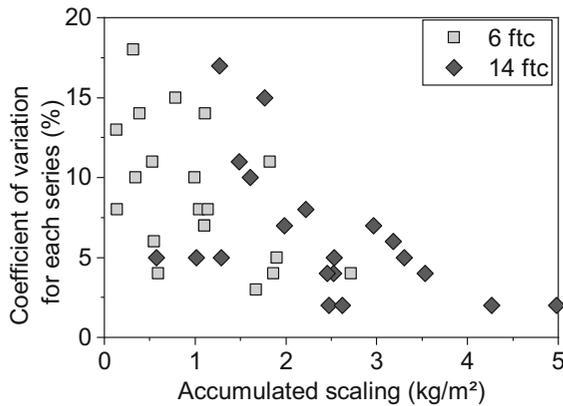
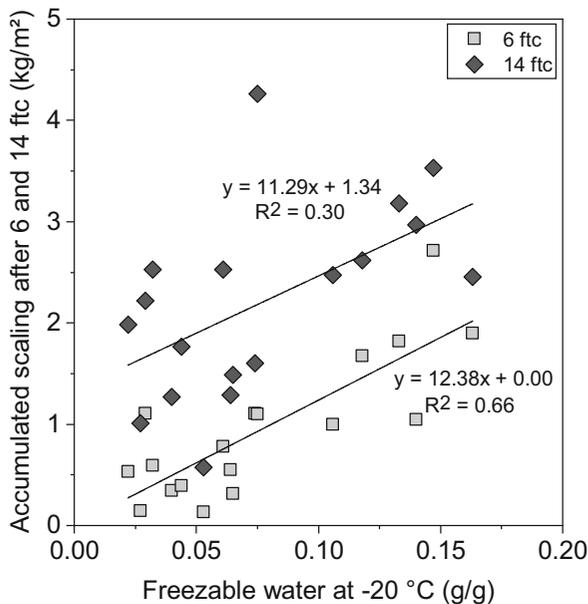


Fig. 7 Coefficient of variation for each series plotted over the average accumulated scaling after 6 and 14 ftc



As expected, the increase in the amount of freezable water (c.f. Fig. 8a) correlates to an increased scaling intensity. A notably better correlation can be observed for the scaling after 6 ftc ($R^2 = 0.66$). This indicates, that the reduced number of freeze–thaw cycles is better suited to represent the damage processes and the material properties, which govern the resistance against salt frost scaling of concretes without air-entrainment.

An increase in Vickers hardness signifies a denser microstructure with decreased capillary porosity. The scaling intensity should thus decrease with increasing hardness. This general trend can be observed both after 6 and 14 ftc (c.f. Fig. 8b). Similar to Fig. 8a, the scaling intensity after 6 ftc ($R^2 = 0.54$) shows a much better correlation.

It can be summarized, that the reduced number of only 6 freeze–thaw cycles is more suitable to assess the salt frost scaling resistance of concretes for the exposure class XF2. This can probably be attributed to the fact, that an excessive liquid uptake and thus internal damage of the concrete specimen can be avoided. Consequently, the moderate saturation with de-icing salt solution, which is typical for exposure class XF2, should be better reflected.

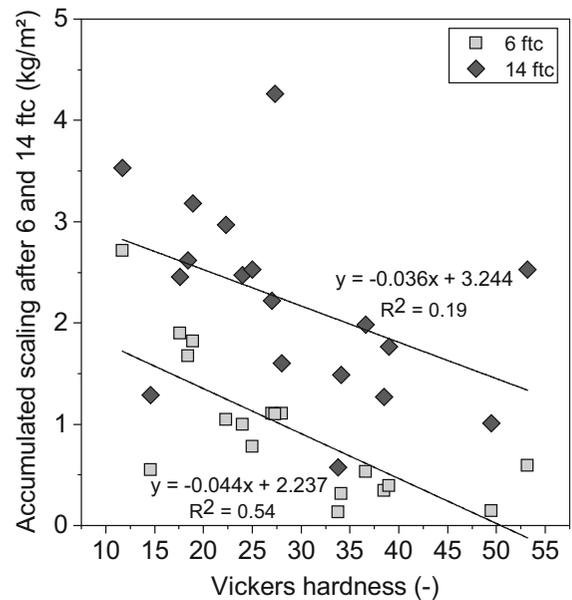


Fig. 8 Accumulated scaling after 6 and 14 ftc plotted a over the amount of freezable water at $-20\text{ }^{\circ}\text{C}$ and b over Vickers hardness, without C9



3.3 Comparison to descriptive requirements

For a new test method with partly novel building materials it is difficult to introduce a meaningful evaluation criterion without an extensive background of experience from practice. To ensure compatibility with expected practical performance to some extent, the results of the modified CDF test have been compared with relevant microstructural properties of the concrete (or HCP as a model substance) in the previous section.

Another possibility is the comparison with descriptive requirements for the concrete, which are based on years of experience, but which can be transferred to novel cements and concretes only to a limited extent. Ideally, concretes that meet the requirements for exposure class XF2 should also pass the performance test. Concretes that do not meet the requirements should fail the test. In the present study, concretes of quality A met the descriptive requirements, except for two minor shortfalls in the minimum compressive strength. Quality B concretes did not meet the requirements.

In Fig. 9, scaling is plotted over the concrete compressive strength after 28 days, separately for quality A and B. A scaling amount of 1.0 kg/m^2 was used as a possible evaluation criterion. Furthermore, a vertical line was drawn at 51 MPa, which corresponds to the minimum compressive strength ($f_{c,cube}$) required for a C35/45 concrete, including an allowance of

tolerance of 6 MPa. This divides the diagram into 4 sectors—A, B, C and D.

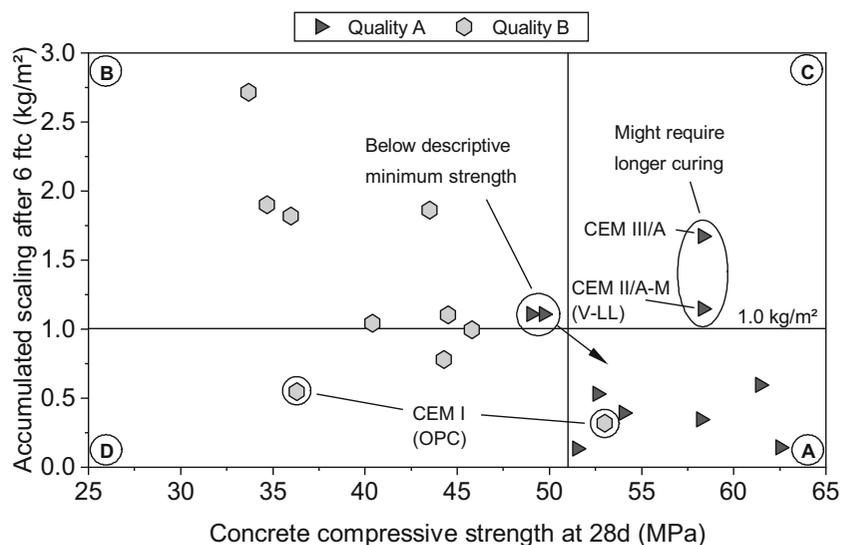
Sector A should ideally incorporate all results from quality A (all descriptive criteria fulfilled, scaling below 1.0 kg/m^2). Sector B should likewise contain all results of quality B.

The measured accumulated scaling after 6 ftc fairly well represents the categorization by descriptive criteria, though for a limited number of concretes there is a disparity between expected and determined performance.

The concretes with Portland cement exhibit very low scaling values, independent of w/c-ratio and compressive strength. The performance of these concretes is so robust, that even “inadequate” compositions show a good salt frost scaling resistance in the laboratory test procedure. Consequently, the compliance with descriptive criteria should be sufficient to guarantee the salt frost scaling resistance of such concretes under XF2 exposure.

Two concretes of quality A exceed the scaling criterion, even though they possess a sufficient compressive strength (sector C). One of these concretes contains GGBFS cement (C6). Previous experience has shown that such concretes often show excessive scaling in laboratory tests when compared to practical performance. This is mostly attributed to the fact, that pre-storage in the laboratory provides optimal conditions for an intensive carbonation of the concrete. The carbonation of concrete with GGBFS causes a coarsening of the pore structure of the surface layer, thus

Fig. 9 Comparison of accumulated scaling (6 ftc) to descriptive criteria



reducing the salt frost scaling resistance [1, 39]. Modified curing conditions, that better reflect practical conditions might be necessary here, as well as for the concrete that contains cement with fly ash (CEM II/A-M (V-LL)). Probably, these two concretes would also take profit from more intensive curing in practice.

Two concretes with quality A exceed the scaling criterion, while not quite reaching the minimum compressive strength (sector B). It can be concluded here, that these concretes might have met the scaling criterion, if they had also fulfilled the compressive strength requirements.

4 Conclusions

It was the aim of a research project to develop an alternative approach for testing the salt frost scaling resistance of concretes for exposure class XF2. The frost exposure in XF2 is characterized by the occurrence of only moderate levels of saturation in combination with de-icing salts. Typically, vertical structural elements (e.g. bridge columns) are subjected to that kind of exposure.

Starting point of the study was the CDF method, which is the commonly used in Germany to determine the salt-frost scaling resistance of concretes for very severe salt-frost exposure (XF4). Modifications were carried out on the CDF test procedure, to obtain a method which can be applied on concretes for exposure class XF2. The following modifications were found to be promising:

- reduced number of freeze–thaw cycles (ftc) from 28 to 6.
- increased heating rate of 20 K/h in the freeze–thaw cycle, which results in a longer holding phase a +20 °C.
- acceptance criterion of 1.0 kg/m² for accumulated scaling
- 10 specimen per test series instead of 5

Regarding the temperature cycle it was important to keep the minimum temperature of -20 °C to not compromise the precision of the test [13]. Though the strong reduction of the number of freeze–thaw cycles might seem drastic, the scaling intensity at 6 ftc better correlates to the material properties, which govern the resistance against salt frost attack in non-air entrained concretes. As the scatter of the scaling results can be

increased by the reduction of freeze–thaw cycles, the use of 10 specimens instead of 5 is recommended.

As a consequence of the modification to the CDF-procedure internal damage is completely avoided in the test, while achieving a scaling intensity that reflects practical experience and mostly meets expectations based on descriptive criteria. The modified CDF procedure is thus a promising approach to allow the implementation of the performance concept for XF2 concretes.

Author contributions MM: Conceptualization, methodology, investigation, formal analysis, writing—original draft, visualization. MS: Investigation, formal analysis, writing—review and editing. H-ML: Writing—review and editing, supervision, project administration, resources. CM: Writing—review and editing, supervision, project administration, resources.

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Availability of data and materials Not applicable.

Code availability Not applicable.

Declarations

Conflict of interest We wish to confirm that there are no known conflicts of interest associated with this publication.

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