



HAL
open science

Freeze–thaw field exposure and testing the reliability of performance test temperature cycle for concrete scaling in presence of de-icing salts

Sara Al Haj Sleiman, Laurent Izoret, Syed Yasir Alam, Frédéric Grondin,
Ahmed Loukili

► To cite this version:

Sara Al Haj Sleiman, Laurent Izoret, Syed Yasir Alam, Frédéric Grondin, Ahmed Loukili. Freeze–thaw field exposure and testing the reliability of performance test temperature cycle for concrete scaling in presence of de-icing salts. *Materials and Structures*, 55 (1), pp.2, 2021, 10.1617/s11527-021-01831-6 . hal-04752491

HAL Id: hal-04752491

<https://hal.science/hal-04752491v1>

Submitted on 24 Oct 2024

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



Distributed under a Creative Commons Attribution - NonCommercial 4.0 International License

Freeze-thaw field exposure and testing the reliability of performance test temperature cycle for concrete scaling in presence of de-icing salts

Sara AL HAJ SLEIMAN^{1,2}, Laurent IZORET², Syed Yasir ALAM¹, Frédéric GRONDIN^{*,1},
Ahmed LOUKILI¹

¹ *Institut de Recherche en Génie Civil et Mécanique (GeM), UMR 6183, Centrale Nantes-Université de Nantes-CNRS, 1 Rue de la Noë, BP 92101, 44321 Nantes cedex 3, France*

² *Syndicat Français de l'Industrie Cimentière, 7, place de la Défense 92974 Paris-la-Défense Cedex, France*

**frederic.grondin@ec-nantes.fr; tel: +33240371668 (Corresponding author)*

ABSTRACT

Testing methods for freeze-thaw scaling of concrete in presence of de-icing salts are developed in the form of normative documents to assess the frost resistance of concrete, aiming to unify the testing standards. These tests consist of exposing cured concrete specimens to several freeze-thaw cycles inside a climatic chamber. The amplitude of each cycle may reach 40°C with considerable freezing and thawing rates that may reach 10°C/h, depending on the temporal cycle length (24h versus 12h). When comparing different testing methods, it appears that the imposed experimental conditions are significantly different and may lead to conflicting results on a same concrete mix. Moreover, there is a lack of agreement between the test methods and the real environmental conditions. Yet, the assessment of the representativeness of a testing method for freeze-thaw durability should start with the measurement of the concrete response to its natural environment and comparison to its response under laboratory conditions. Therefore, a first experimental campaign was held in which temperature measurements and scaling monitoring of concrete blocks were carried out at Mont Aigoual in France. This paper is reporting the conditions of measurements, the meteorological analysis as well as the scaling results of concrete specimens exposed in real freeze-thaw conditions with a comparison to other specimens, with same concrete mixture, tested in laboratory conditions. All scaling concrete specimens were prepared and cured together; they had the same age before the beginning of the exposure to freeze-thaw cycles. We show that the applied thermal cycle of [+20/-20°C] does not correspond to real conditions, even in regions with a harsh winter environment. Furthermore, a considerable dispersion was found in laboratory thermal conditions when applying

the normative cycle. Therefore, a reflection about the relevance of the applied temperature cycle and its representativeness of field conditions is proposed.

Keywords: concrete, freeze-thaw, de-icing salt, frost scaling, standard tests.

1 Introduction

Under highly aggressive environments, exposed concrete suffers from many types of pathologies that lead to degradation of the structure such as leaching, shrinkage, carbonation, sulfates attack, chlorides attack, alkali-aggregates reaction and damage due to freeze-thaw cycles. Among various durability and sustainable construction concerns, resistance of concrete structures to freeze-thaw actions, with and without presence of deicing salts, is one of the most preoccupant subjects in civil engineering research, in countries where cold winter conditions occur. The deterioration of concrete under the freeze-thaw action, especially in the presence of de-icing salts, is often accompanied by other equally severe types of deterioration such as the diffusion of aggressive agents (chloride and sulfates for example) through the degraded surface and frost-induced cracks. This undoubtedly penalizes the durability of structure. Concrete exposed to freeze-thaw cycles may undergo two types of deterioration: internal damage and surface scaling. Scaling is a mode of deterioration that consists of a progressive detachment of concrete masses from the exposed surface. As for internal damage, a severe deterioration is induced under the effects of hydraulic [1], osmotic [2] and crystallization [3], [4] pressures accompanying water phase change and ice formation inside the porosity. The pressures' theories and models explain how this above combination leads to the internal damage mechanisms of concrete [5]. The role of the entrained air voids as expansion valves is also understood in these theories. However, theories and models to explain concrete surface scaling damage are limited. A review of the main existing theories is presented in [6] with the limitation of each one. The effect of the entrained air in protecting concrete from scaling is not clearly understood. One of the most known models so far is the glue-spalling model of Valenza and Scherer [7]. The salt scaling of concrete in presence of de-icing salts is related to the cracking of the salt solution placed on top of the concrete specimen during the test. The stresses developed in the ice brine solution are transferred to

the concrete surface. This latter is assumed to be deteriorated under the effect of the induced stresses [7]. The maximum amount of scaling damage was observed when a solution with a moderate amount of salt is applied at the top of concrete exposed surface (in the case of solutions with around 3% of salts by mass), this is known as “pessimum effect”. It was first observed in the work of Verbeck and Klieger in [8]. This observation was then explained by Valenza and Scherer in [9] and confirmed in other studies such as [10], [11], [12]. The glue-spalling and the pessimum salt concentration effect were lately numerically modeled by [13]. This fact leads to a common use of a 3% salt solution in the testing protocols for concrete resistance to scaling such as the slab test and the CDF test of the CEN/TS 12390-9 [14], the French norm XP P18-420 [15] and ASTM C672 [16]. These experimental testing methods and many others had been developed in a normative context in order to ensure the longevity of the material, to increase its resistance to severe conditions and to protect constructions and final user. They had been developed by standard authorities such as CEN, SNV, DIN and AFNOR mainly used in European countries, in addition to ASTM and BNQ used worldwide. In a general way, the provided test methods consist of the following steps:

- Concrete specimens preparation (casting and demolding after 24 hours).
- Curing specimens according to each test method and pre-conditioning before exposure.
- Exposing concrete specimens to temperature cycles inside a climatic chamber over 12 or 24 hours with a common range of +20/-20°C (40°C full amplitude) with variable cooling and heating rates, specific to each test method, which can reach up to 10°C/h [17].
- Taking measurements of different criteria in order to quantify the intensity of degradation e.g. mass of scaled materials, relative dynamic modulus (RDM)...

Many factors are not considered in freeze-thaw test conditions such as concrete ageing, drying/wetting cycles and possible carbonation that concrete may undergo in real exposure conditions. These factors may have a non-negligible effect on concrete resistance to scaling due to changes in microstructure that they induce. Moreover, during the scaling test according to the cited standardized methods, a layer of salt solution is always present and renewed at the top of concrete tested specimens. It was observed that no considerable scaling

is produced when concrete had no free solution on it [8], [7], [10]. This position is agreed during test procedures. We observed that dried specimens (after evaporation or leakage of brine solution) were not exhibiting considerable scaling. However, concrete surface in real situations is not constantly covered by a salt solution. It is exposed to drying/wetting cycles that can modify its saturation degree as well as its microstructure. In addition to the cited factors, the applied temperature range [+20/-20°C] does not seem to correspond to real environmental conditions, even in regions with a harsh winter environment as shown in [18]. In this work, we focused on testing the reliability of the thermal cycle of [+20/-20°C] imposed in the standardized testing methods for frost scaling of concrete, in presence of de-icing salts. Multiple field evidence show that degradation due to freeze-thaw scaling occurs under much milder local climate compared to conventional laboratory conditions. However, when questioning the test method representativeness, the methodological approach is to correlate laboratory test results with the deterioration of concrete specimens, with the same concrete mix design, placed in a natural environment. Therefore the following experimental campaign was carried out: temperature measurements and scaling monitoring were carried out respectively on large blocks and concrete specimens placed on a test platform at Mont Aigoual meteorological station in France. Concrete samples made with the same mixture have been subjected to laboratory freeze/thaw cycles according to the slab test of the CEN/TS 12390-9. Temperatures in the brine solutions and the scaled masses have been measured and compared to what is obtained in the case of field exposed specimens. Since curing and ageing are important parameters that have a large effect on frost damage, all tested concrete specimens used for scaling measurements had the same age at the beginning of exposure to freeze-thaw. They were all cured together following the same procedure that is described in the following section.

2 Experimental campaign

2.1 Presentation of Mont Aigoual site

The Mont Aigoual (geographical coordinates: 44.12°N | 3.58°E), a French mountainous region in the southern part of Cevennes mountains, was chosen for the experimental program

for two main reasons:

- It is an area classified as a "severe frost zone" (according to the exposure classes of the NF EN 206-1 [19]) at 1567 m of altitude (Figure 1, left). A severe frost zone is characterized by a temperature that stays more than 10 days per year under -10°C [19].
- There is a full meteorological station in Mont Aigoual (Figure 1, right) which is an official test center in France, so meteorological parameters data are accessible such as air mass temperature, relative humidity, wind speed, visibility, and global solar radiance.

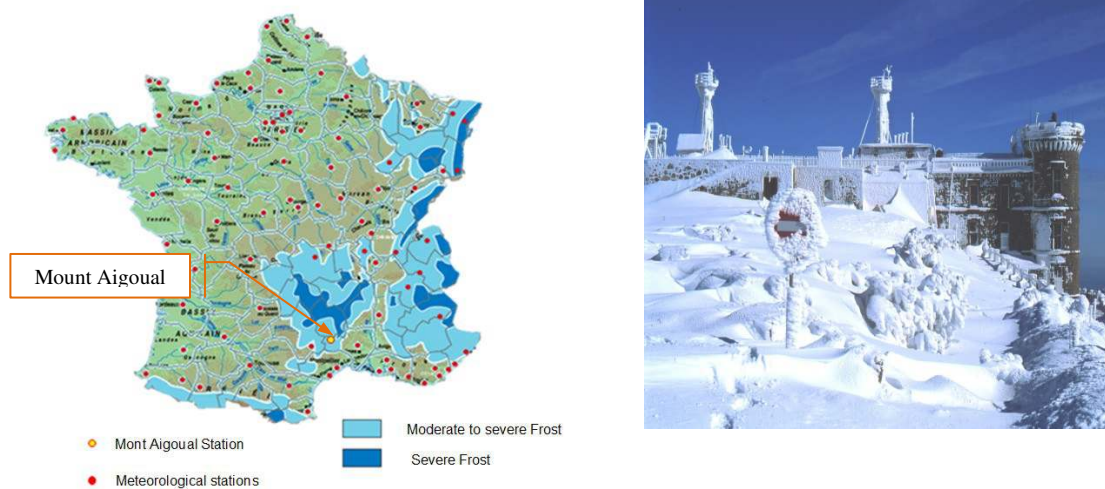


Fig.1 Geographical location of Mont Aigoual in a moderate to severe frost zone (left), Mont Aigoual meteorological station (right).

Air mass temperature (known as sheltered temperature) is measured using a Pt100 probe located in a double sheltered cabin protected from the effects of wind and direct sunshine. A pyranometer CMP6 from Kipp&Zonen gives the global radiance (W/m^2) as the sum of direct and diffusive solar radiance. More details about the chosen site can be found in [18].

2.2 Mix design of concrete specimens

The tested concrete mixture and its properties are reported in Table 1.

Table 1 Concrete mixture (for $1 \text{ kg}/\text{m}^3$) and properties

Components	Composition	Properties	
Cement CEM II/A 42,5 LL (kg.m ⁻³)	320	Air entrained	5%
0/4 sand aggregates (kg.m ⁻³)	537	Slump	150 mm
0/1 sand aggregates (kg.m ⁻³)	249	Apparent density	2090 kg/m ³
4/10 gravels (kg.m ⁻³)	310	Tensile strength	3 MPa
10/20 gravels (kg.m ⁻³)	641	Compressive strength	32,6 MPa
Water reducer super plasticizer		Water porosity	14,1%
Water/cement ratio	0.5	Chloride ions diffusion coefficient	2,3 x10 ⁻¹² m ² /s
Air entrained	5%	Spacing factors	281 ¹ , 270 ² μm

¹Corresponds to the spacing factor at 1 cm from the mould bottom face, ²section in the inner part of the concrete specimen (representing the spacing factor at the sawn face).

Air entrained as well as the slump tests were done at fresh state of concrete. According to [20], a minimal content of entrained air void content is 4% to meet frost durability in the cases of moderate frost and frequent salting. As for compressive strength, a minimum value of 35 MPa is required in the case of frost medium in presence of de-icing salts. A minimum tensile strength of 2.5 MPa is recommended. In addition, aggregates must be freeze-thaw resisting in order to reduce the effect of pure aggregate damage on overall scaling results. Both, compressive and indirect tensile strengths were determined using three cylindrical specimens Ø15×30 cm² at the age of 28 days. In order to determine the total porosity accessible to water and chloride ions diffusion coefficient (both are well known durability indicators), the tests were conducted according to the standards XP P18-462 [22] and XP P18-459 [23] respectively. The calculated porosity is the ratio of the pore volume of the tested specimen to its apparent volume. This latter is considered as the sum of the volumes of solid material and open and closed pores of the specimen and calculated as the difference between the mass of the specimen in air and its mass in water. Apparent density is the ratio of the dry mass of the body to its apparent volume. The depth of the chloride ion penetration

front is 2.8 cm. Values of diffusion coefficient, porosity accessible to water and apparent density are given in Table 1. The objective of determining the durability indicators of this concrete mix is to show that the concrete used in this study corresponds well to the concrete of common use with a sufficient durability. The considered concrete mix design is neither too weak nor too strong; therefore it allows having exploitable results in a reasonable time. Regarding spacing factor, it is approximately the half-distance between two air bubbles. An effective air entrained system is often considered to have a spacing factor less than 250 μm [20], so that the expelled unfrozen water can easily reach an air bubble that has the role of an expansion valve. The role of entrained air bubbles was first put forward in the study of Powers [1]. The modified point-count method (procedure B) of the ASTM C457 is the most used method for determining the value of the spacing factor. It is a very sensitive technique that consists on counting the number of air bubbles intercepted with the eye on a section of concrete using the microscope.

2.3 Experimental procedure

For scaling measurements, eight concrete specimens were casted in 15 cm^3 cubic polyvinyl chloride molds in laboratory, four of them were tested in laboratory conditions, inside a climatic chamber Binder MKF-240 in GeM laboratory at Centrale Nantes, and four others were exposed on site, at Mont Aigoual. Figure 2 presents the followed curing steps. After demolding, all specimens were placed in lime-saturated water until the age of 7 days. Then, they were stored inside a climatic chamber at 65 % RH and 20°C during 6 days. After this curing period they were cut in two parts of dimensions 15 \times 15 \times 7 cm^3 (length \times height \times thickness) to obtain eight specimens with a sawn surface and eight specimens with a bottom cast surface. These two different types of surface textures were exposed to freeze-thaw in order to compare their behavior. Even though sawing at early age may increase scaling of the sawn surface as seen in [24], this step was done at the age of 10 days as shown in Figure 2 . Therefore studied faces, both sawn and bottom cast, may be carbonated and dried at the same level. The curing of all specimens (laboratory and field specimens) was carried out inside a climatic chamber until the age of 28 days. At this age, configuration according to the standard

was done and a 72-hours pre-saturation phase began with a 3 mm layer of demineralized water poured on the top of each specimen. At the end of this phase, field specimens were placed at Mont Aigoual to be exposed to real climatic conditions, while laboratory specimens were subjected to freeze-thaw cycles inside a climatic chamber, according to the slab test. Along the test, field and laboratory specimens were covered with a 3 mm layer of 3% NaCl saline solution that was renewed every 7 days.

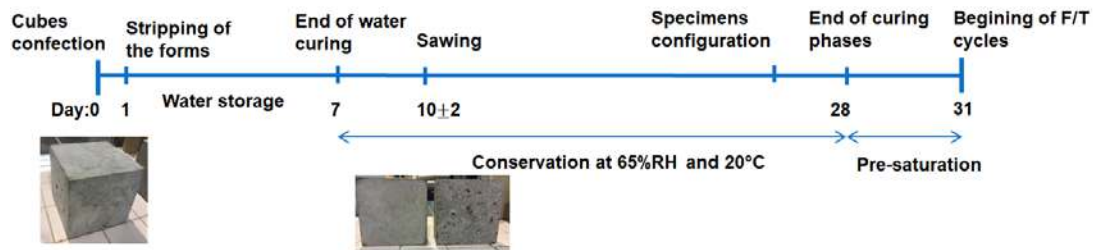


Fig. 2 Followed steps in the curing period of the test.

In European scaling tests such as the slab test of the CEN/TS 12390-9 [14], the sawn surface is considered as the testing surface. The origin of this choice is that the first millimeters of concrete skin will be detached after few years of exposition; therefore, the durability of the inner part represented by the sawn surface should be tested. Whereas, the French norm XP P18-420 [15] specifies to run the test on the bottom cast surface since in reality, the cast surface is the first to be exposed to weather conditions. However, the cast surface tested in the Swiss standard [25] may be more representative of the exposed surface of a concrete structure than the bottom cast surface considered in the XP P18-420. This point is actually a subject of discussion at the European scale, since the choice of the type of testing face influences the concrete response due to differences in composition and microstructure of each face, as well as differences in paste content.

2.4 On-site conditions

Meteorological measurements were done along a large period of time, from 2013 to 2016, during many months over these years. A second experimental campaign was re-conducted for the winter season 2019-2020. Two large concrete blocks of dimensions $60 \times 46 \times 17 \text{ cm}^3$ (length \times height \times thickness) were installed outdoor, starting from the beginning of the

winter season of 2013, for two winters and then in 2019 for one more winter season. Each block was instrumented with five thermocouples: three of them were inserted during casting at each vertical side of the block at 2 cm from the surface, one thermocouple was placed at 0,2 cm from the surface and one in the middle of the block. The thermocouple at 0.2 cm was positioned by mean of a saw cut, large enough to be filled by the thermocouple and sealed with a fine grain mortar (0/2mm) derived from the initial mix-design. Figure 3 shows the concrete blocks instrumented with thermocouples. These blocks were used to monitor concrete surface temperature only; scaling was measured on concrete specimens placed at the Mont Aigoual platform starting form 2019.

Concrete blocks and scaling specimens were de-iced regularly and scaled masses were collected every 7 days during the winter period 2019-2020. A 3 mm thick layer of a 3% saline solution was applied on the top of each specimen/block by a technician who was present at Mont Aigoual station. The upper limit of the joint was 3 mm higher than concrete surface in order to ensure the correct volume of saline solution. Specimens were checked every 7 days during the winter season, in case they were found:

- without frost, the brine volume was completed to a 3 mm thick layer on the top of each test specimen,
- with frost, a 3 mm thick layer of brine was added on top of each specimen.
- in case of presence of snow, snow was removed from the surface and then the solution was added,

Therefore, saturation conditions for field specimens were similar to those of laboratory specimens that were tested according to the slab test.



Fig. 3 Concrete blocks instrumented with thermocouples (left), (right) same concrete blocks as well as scaling monitoring specimens, all configured according to the CEN/TS 12390-9 standard.

2.5 Laboratory conditions and scaling measurements

For laboratory testing, the thermal cycle of the slab test standard was applied. The temperature evolutions in the brine solution of the specimens placed at different positions inside the climatic chamber are shown in Figure 4, as well as the temperature tolerance zone given by the standard. According to the slab test specifications, the surface temperature of tested concrete specimens should be within this tolerance zone. Figure 4 shows the temperature scattering of the samples in the climatic chamber and we can observe that all the permitted range of fluctuations (the temperature tolerance zone) is swept. This was also noticed in the measurements of a Belgian laboratory highly qualified in freeze-thaw testing as it was reported during the 8th meeting of the European comity of freeze-thaw testing normalization in June 2015 at Mount Aigoual Station (unpublished data).

Scaling was measured every 7 cycles using a 63 μ m sieve and a brush composed of synthetic fibers (Polyamide). The specifications of the brush were conformed to the guidelines for the brush selection in annex B in the FprCEN/TS 12390-9 [14]. The brine solution was renewed after collecting scaled masses and specimens were returned to the climatic chamber to pursue the freeze/thaw cycles. The collected masses were dried in an oven at 105°C until constant masses.

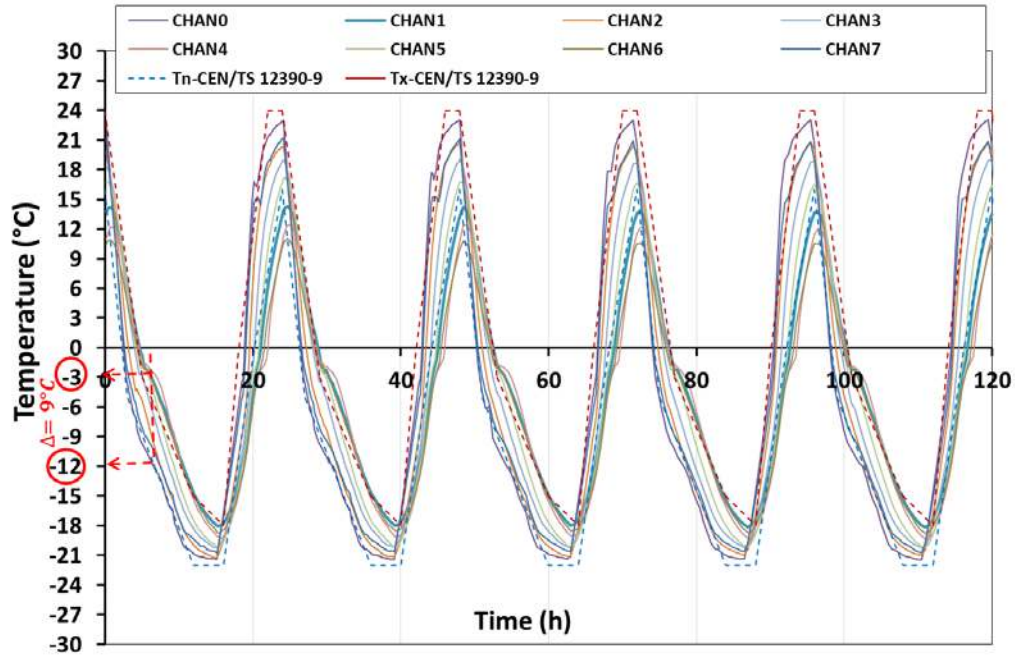


Fig. 4 Temperature evolution in the brine solution of the specimens placed at different positions inside the climatic chamber and the temperature tolerance area given by the standard (in dotted lines).

3 Results and discussion

3.1 Real freeze-thaw thermal profiles

Time series at Mont Aigoual during winter 2019-2020 from 1st February to 1st March 2020 are presented in Figure 5, giving fluctuations of relevant parameters such as air mass temperature, solar radiation (W/m^2) and concrete surface temperature. During this winter period, 34 freeze/thaw cycles ranging from $+5$ to $-5^\circ C$ were registered, such that each freeze-thaw cycle is considered as a temperature fluctuation around $0^\circ C$, fixing a minimum daily temperature equal to or lower than $-1^\circ C$ and a maximum temperature equal to or higher than $0^\circ C$. No correlation was found between real cycles that concrete undergoes at Mont Aigoual and $[+20/-20^\circ C]$ temperature cycle applied in laboratory normative testing methods; temperature cycles ranging between $+20^\circ C$ and $-20^\circ C$ never occurred in field. This has been found true along the whole period under investigation (beginning from 2013, more data are provided in authors' previous publication [18]). When real freeze-thaw cycles appear on

concrete surface, they are ranging between $+5^{\circ}\text{C}$ and -5°C . This observation is in accordance with Kaufmann studies [26]. Moreover, from Figure 5, concrete surface temperature profile is strongly asymmetric in contradiction with the thermal cycle applied in the slab test of the CEN/TS 12390-9. Concrete temperature signal is characterized by an expansion towards positive values compared to air mass temperature during day times and more consistency with air mass temperature during diurnal times [18]. In fact, the thermal behavior of exposed concrete is the result of combination of different involved phenomena. Fluctuations of concrete temperatures are strongly correlated to those of the solar radiance, air mass temperature and the day/night alternations are governed by the solar radiance effect. In authors' previous research [18], this behavior was modelled and involved mechanisms were presented. The concrete surface is subjected to solar radiation that heats the concrete at diurnal phase. At night-time, this absorbed energy can be transmitted to the surrounding area as part of thermal exchange; therefore concrete surface can be found colder than the surrounding air in many cases when some meteorological conditions are coupled, such as a high visibility and a low wind velocity, this was modelled in [18]. This phenomenon is known as nocturnal radiation [27] [28]. Beside the great differences in the thermal amplitudes that are noticed between laboratory and real conditions, several phenomena are involved in the thermal response of exposed concrete.

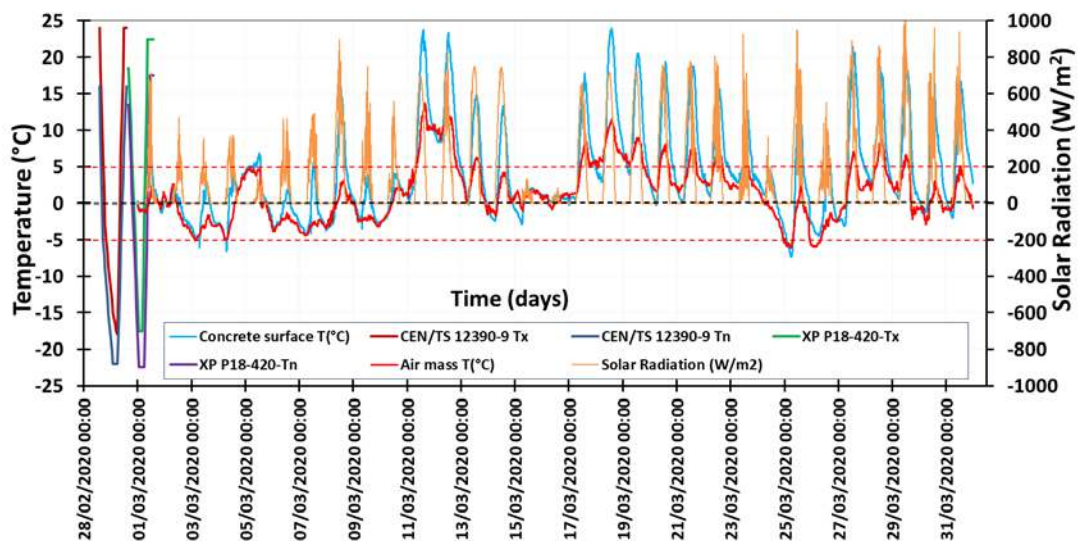


Fig. 5 Time series of concrete surface temperature, air mass temperature (Y axis) and solar radiation (Y' axis) with reference to CEN/TS 12390-9 and XP P18-420 thermal cycles

3.2 Scaling results analysis

3.2.1 Studying the effect of the type of the testing face

Scaling results for laboratory specimens are given in Figure 6 (left), those of the Mont Aigoual specimens after one winter season 2019-2020 are given in Figure 6 (right). The averages (calculated on 4 specimens) of the cumulative losses are plotted as a function of the freeze-thaw cycles for specimens with bottom cast face and sawn surface. In the case of laboratory specimens (Figure 6, left), after 56 cycles, the average of scaled cumulated masses from specimens with bottom cast surface and sawn surface are 1739 and 1004 g/m² respectively. No clear scaling threshold level was defined in the CEN/TS 12390-9 as an acceptance criterion for concrete scaling resistance after 56 freeze-thaw cycles. However, in the French norm XP P18-420, a scaling level between 600 and 750 g/m² is already defined as an acceptance criterion to assess concrete resistance to scaling after 56 freeze-thaw cycles of [+20/-20°C], in presence of de-icing salts. This was shown in dotted black lines on the left graph of Figure 6. The effect of the type of the testing face can be noticed from this test. Bottom cast face shows lower scaling resistance than sawn surface, this is true in case of field laboratory. Furthermore, in case of laboratory specimens, after 28 cycles the pattern of the cumulative loss evolution curves for the sawn surface continues to increase along a linear line of low speed compared to the curve of the bottom cast face. The higher amount of scaled masses obtained in the case of bottom cast face specimens can be explained by the fact that the bottom cast face is covered by a higher proportion of cement paste than the sawn surface. Marchand et al [29] indirectly verified the importance of surface strength by comparing results obtained on finished and sawn surfaces. The authors [29] showed that the amount of scaling depends on the proportion of the surface covered by the cementitious binder, indicating that the stronger aggregate inhibits scaling. Moreover, segregation and skin formation (external boundary layer) are responsible of the weak resistance to scaling of the mold bottom face. In fact, segregation will lead to high local porosity at the level of the mold bottom face. Consequently, there is a strong deviation in the results of the same concrete tested by each method depending on the type of the tested face (such as the case of slab test

and French standard). Therefore, a unification of the choice of the testing face appears necessary.

On the other hand, a large variability is noticed in the scaling values of laboratory specimens (Figure 6, left), the dotted vertical lines show the dispersion around the mean value of the maximum and minimum values obtained for the four specimens of the same series (sawn and bottom cast faces series). After 56 cycles, the coefficients of variation of the cumulative losses of the four specimens with sawn face and bottom cast surface tested in laboratory conditions reach 77% and 41% respectively.

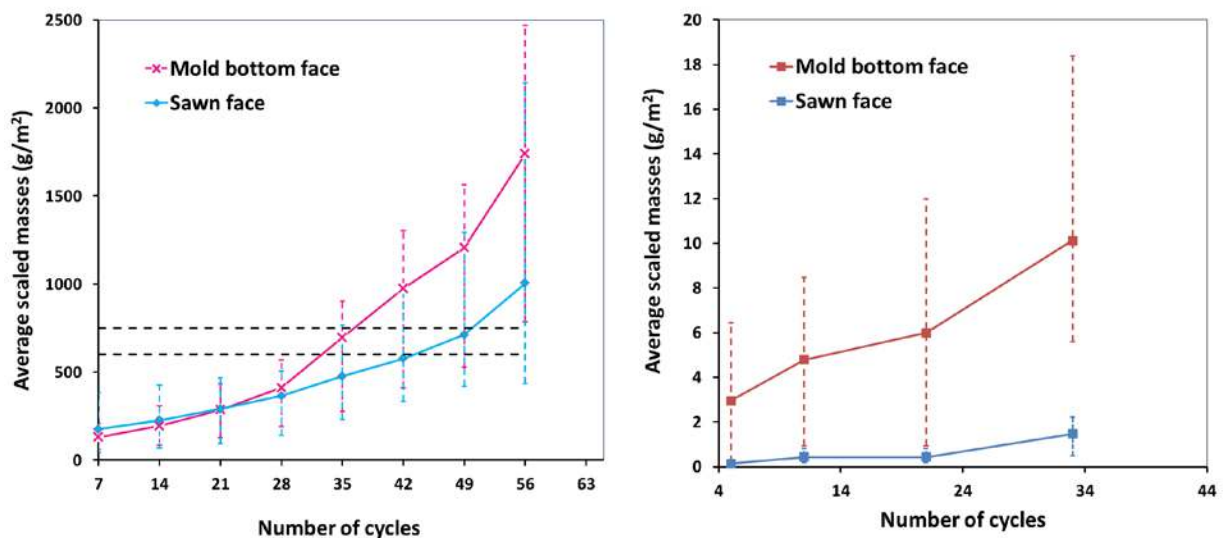


Fig. 6 Laboratory (left) and Mont Aigoual (right) specimens average scaled masses (mold bottom and sawn surface specimens).

3.2.2 Factors affecting concrete resistance to scaling

Measurements of temperature inside the climatic chamber showed that at a time t during the test, temperatures in the brine solutions of two specimens placed at two different positions could differ by almost 10°C , as shown in Figure 4. The minimum temperatures reached in the brine solutions differ by almost 5°C from one specimen to another, depending on its position in the climatic chamber. The temperature tolerance zone defined by the slab test seems to be too tolerant and this may be one of the reasons behind of the dispersion found in

the scaling results. Therefore, the reliability of the existing methods may be questioned in view of these high discrepancies in temperature and scaling results.

On the other hand, the scaling levels of specimens with sawn or bottom cast surfaces, after one winter season in “a severe frost zone” (Figure 6, right), are much lower than those of specimens subjected to the laboratory freeze-thaw cycles (Figure 6, left). In fact, this may be mainly attributed to the minimum temperature effect to which concrete is exposed. The cycles imposed in the laboratories fluctuate between +20 and -20°C, however at Mont Aigoual, the cycles fluctuate between +5 and -5°C. In addition to the thermal stresses developed due to the thermal mismatch between the ice layer and concrete surface described by the glue spalling [9], more the minimum temperature is decreased, finer pores (in the order of one nanometer) near the surface will be frozen too. According to the Gibbs-Thomson equation used in [5], which links the freezing temperature to the threshold radius of frozen sites, at -5°C the pores that freeze are of the order of 14 nm in radius. As the temperature decreases, ice crystals grow in finer pores: at -20°C, the pore radius is about 4 nm, then about 4 times finer than the pores that freeze at -5°C. Consequently, the freezing of the finest pores near the exposed surface favors the scaling degradation because of the increased impermeability (more pores of the system are clogged with ice) and because of the developed pressures in the porous medium leading to the pop-out of concrete fragments from the surface. The role of entrained air is to protect concrete from scaling by providing space where water can freeze without exerting pressures. Although this explanation does not correspond to the existing models for scaling, including glue-spalling, it is in agreement with Fabbri et al. [30] who showed that scaling can occur due to the localization of pore overpressure near the skin surface caused by the low permeability of cementitious material, this was demonstrated by a poromechanical developed model. According to Fabbri, scaling is a "localized internal damage" type of concrete degradation [30]. Scaling appeared in his study in case of parafilm sealed saturated specimens, even without the presence of saline solution at their top. In addition to the effect of the minimum temperature, the cooling and thawing rates that specimens undergo inside the climatic chamber are two to three times the real values. An in-depth study on the effect of the temperature cycle parameters (such as minimum temperature

and freezing rate) is presented in a recent research work [31] reporting results of [32] and in [33]. The minimum temperature was seen to have a significant effect on the scaling. In the glue spalling model [9], the effect of minimum temperature can be seen in the increased stresses developed in the brine solution on the top of concrete specimen because of increased thermal mismatch. It was reported in [10] that the -20°C temperature applied as the minimum temperature in the slab test produce more than twice as much scaling as the -10°C . This shows clearly the severity of the slab test method, especially in what it concerns imposed thermal conditions. In this same study, it was shown that increasing cooling rate to a fixed minimum temperature did not lead to significant change in the scaling. However, the effect of cooling rate found in [33] was different. The test was performed according to the CDF test configuration and for non-entrained air concrete specimens scaling increased by reducing the freezing rate (still with a minimum temperature of -18°C) and by extending the period at the minimum temperature. This was explained by the fact that the time spent freezing the sample should be significant since an osmotic mechanism involves mass transport which requires time. Hence, above a temperature of -9°C , the degradations become non-existent [6], [31]. Concerning the effect of the duration of the phase at the minimum temperature h_{\min} , it was seen that scaling exists even if $h_{\min} = 0$, but it grows with h_{\min} since a sufficient time is necessary to build up osmotic pressure mechanisms [10]. This effect of h_{\min} was also cited in [34]. However, as far as we know, no study is available on the influence of the thawing rate on the deterioration of a cementitious material.

More studies were done where a lack of correlation was found between laboratory and field results for multiple reasons. A study was conducted in Sweden [35]–[37] aiming to compare results of field and laboratory performance tests for a range of concrete mixes prepared with different types of binders, after 10 years of exposure. Specimens were placed along the highway between Gothenburg and Boras, where the salt exposure is considered severe (the application of de-icing salts follows the procedures of the Swedish road authorities). In the laboratory test, the SS 13 72 44 test method, which corresponds to the reference slab test method of CEN/TS 12390-9, was applied. After ten winters of field exposure, it was found that all the tested mixes had no or very little visible surface damage (classification "1" on a

scale of 0-5), regardless of the results of the laboratory evaluation. Moreover, no cracks were reported. One of the reasons behind these results is given by the records of air mass temperature, clearly showing that the most frequent ranges of freeze-thaw cycles are [+5/-10°C] or [+5 and -5°C] [37]. However, some laboratory specimens showed significant levels of scaling depending on the type of cement used, the w/c ratio and the percentage of entrained air in the mix design. From this study, it appears that no good correlation was detected between laboratory results and field performance even after 10 years of exposure for concretes with good or intermediate quality levels; however the slab test method was found able to correctly identify concrete qualities with poor and high salt-frost resistance. Therefore, we can see that there is an unstable correlation between laboratory and field results depending on many factors; hence, many concretes that fail in the laboratory can have an extremely good service life. The reason behind this lack of correlation was attributed in this study to the excessive degree of saturation of the laboratory specimens compared to the field conditions. In real situations, concrete surfaces such as bridges, road surfaces and hydraulic structures walls are subjected to drying/wetting cycles due to simultaneous effects of solar radiation and rain. Therefore, imposing a saturation state that will never be reached in real conditions produces unrealistic damage of the tested material [38]. Moreover, discrepancies in on-site and laboratory performances were identified in this study for concrete specimens designed with slowly reacting binders such as fly ash. Hence, early age testing (such as in the case of laboratory testing) of these concretes becomes unfair compared to field service conditions. In [39], two concrete qualities suffered from considerable damage in field after only 3 winter seasons exposure, however, they were evaluated as resistant when tested in laboratories as reported in [39]. This result was attributed to the negative effect of carbonation often detected on concrete containing large amount of slag in their binders. Laboratory specimens had been subjected to air for only few days (during drying periods), whereas concrete in the field had been continuously carbonated. In addition to the cited parameters that appear to have considerable effect on concrete resistance to scaling, carbonation of concrete surface exposed to real conditions may affect its resistance to scaling and the effect is depending on binder type. As mentioned previously, carbonation may have a

negative effect on concrete resistance to scaling when it contains slag. However, in the case of Portland cement, carbonation allows to fill the porosity of the surface by the formation of calcite whose molar volume is higher than the one of portlandite. This should tend to improve the strength of the surface. However, for laboratory tests, carbonation can only take place during a relatively short period (storage period of 21 days at 65% RH and 20°C). No surface carbonation is possible during the freeze-thaw test since the specimens are topped with the saline solution on the surface which does not constitute conditions that allow CO₂ diffusion. This puts the point on the necessity of proposing a method that is able to identify correctly all concrete qualities.

Moreover, it was discussed in [36] on the effect of ageing of concrete especially when it is not air entrained. In fact, the amount of freezable water in the capillary pores is important at the age of one month, which corresponds to the age of concrete at the beginning of the test. In absence of entrained air, concrete disintegrates because of freezing. Therefore, the period of ageing is important because during this phase, hydration can proceed and the volumes of capillary pores and freezable water decrease which can protect concrete from damage that may be developed under freeze/thaw exposure. The beneficial effect of ageing was seen especially in case of concrete with low w/c ratios (0.3-0.4) as it was shown in [36].

Concrete scaling results differs when it is exposed in real conditions from laboratory results due to the several cited factors above. It was agreed in [40] that the application of the test methods in TS 12390-9:Testing hardened concrete - Part 9: Freeze-thaw resistance-scaling is very severe and in some CEN member states, the application of these tests will cause concretes with a long history of satisfactory use to fail. A reason behind the lack of correlation between laboratory performance tests and real freeze/thaw conditions can be attributed to the imposed thermal cycle itself. Climatic conditions were reported during the study of [35] and show that the minimum temperature registered in the winter season of 1997-1998 did not reach -20°C, moreover the cycle of [-20/+20 °C] was never detected. In our study, since all concrete scaling specimens were prepared and cured together, a special interest was given to characterize the different thermal conditions and to test the representativeness of the [+20/-20°C].

In view of the detected dispersion of thermal conditions imposed during standardized methods (Figure 4) and considering the lack of representativeness of the applied [+20/-20°C] of real freeze-thaw conditions, even in regions with severe climates as shown in [18], a proposal of an alternative freeze-thaw cycle may need to be considered in such a way that the new cycle has to be based on real freeze-thaw conditions in countries with moderate to severe climate. Moreover, and most importantly, the parameters of the alternative cycle (minimum/maximum temperatures, total amplitude, freezing and thaw rates) have to ensure homogeneous thermal distributions inside the climatic chamber. According to [41], the cooling rate should probably not significantly exceed 2C°/hour, as that would be much faster than expected field cooling rates and may not reproduce the same damage mechanisms as in the field. The thawing rate could probably be about double the freezing rate.

3.2.3 A chemical mechanism inducing scaling

A thermogravimetric analysis of the scaled masses from laboratory specimens shows, as expected, that the main component is calcium carbonate (CaCO_3) (Figure 7). Same results were found for scaled masses of field specimens. Therefore, the exposed carbonated layer is the first which detaches from the concrete surface under the action of freeze-thaw cycles. As the aggregates are granitic in nature, limestone is not the main constituent that could be present in the scaled masses. However, theories and models proposing explanation of concrete surface scaling damage are limited because it appears that scaling deterioration may be caused by coupling of multiple mechanical mechanisms such as stresses that accompanies water transformation to ice on the top of the tested surface and inside the porosity located near the exposed surface, in addition to other chemical mechanism. According to the phase change diagram of aqueous NaCl, the nucleation temperature in a solution with 3% NaCl is close to -2°C; obviously ice starts to form in the brine below this temperature. Therefore, the observed scaling in the case of field specimens that were exposed to mild minimum temperature (freeze-thaw cycles in Mont Aigoual were ranging from +5°C to -5°C) may be related to other mechanisms. A chemical explanation of the scaling phenomenon is put forward in the thesis work of Girodet [42]. The portlandite or calcium hydroxide ($\text{Ca}(\text{OH})_2$) that remains on (and close to) the surface of the air-cured specimens dissolves owing its high

solubility at low temperature. The portlandite, which crystallized before freezing rather in the form of plates, is presented after freezing in weak hexagonal form [42]. This change of morphology characterizes the phenomenon of dissolution-recrystallization of $\text{Ca}(\text{OH})_2$. In presence of de-icing salts, such as NaCl , dissolved portlandite is transformed to CaCl_2 . The reaction between CaCl_2 and calcium aluminates (C_3A) contained in cement lead to the formation of Friedel's salt (and ettringite in presence of sulfates). The crystallization pressure exerted by formed Friedel's could weaken the surface so it is more susceptible to scaling [7] [9] [43]. Moreover, the relation between scaling damage and precipitation of Friedel's salts is suggested by the observation that cements containing higher C_3A contents are more susceptible to scaling, as cited in [7] after [44] and in [45] and [43]. Another study on the effect C_3A content in cement on freeze-thaw resistance is given in [46].

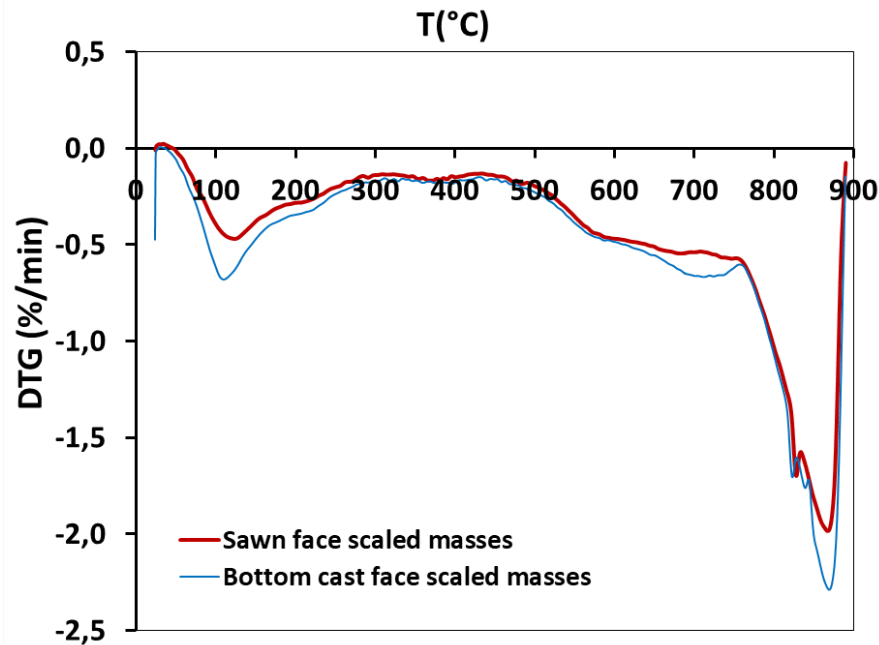


Fig. 7 Thermogravimetric analysis of the scaled particles from the sawn face and bottom cast surface laboratory specimens (left).

4 Conclusion

In this paper, a lack of representativeness of existing normative methods to characterize concrete resistance to scaling in presence of de-icing salts is found. This could be attributed to various factors already mentioned, such as the excessive degree of saturation that is imposed during the test which generates an unrealistic degradation as well as wetting/drying cycles that happens in reality under the simultaneous effect of rain and solar radiation and may change concrete microstructure, although these are not taken into account in the laboratory tests. Moreover, ageing of the exposed surface and its carbonation are factors that should be considered when comparing field to laboratory conditions. One more essential factor that may have a major effect is the thermal cycle imposed during the normative test. The thermal amplitude imposed during the laboratory test is found not representative of the real freeze-thaw conditions to which concrete might be exposed in countries with moderate to severe climates. Moreover, in addition to its lack of representativeness of real freeze-thaw conditions, the [+20/-20°C] normative cycle does not ensure a homogeneous temperature distribution inside the climatic chamber. This is attributed to its large total thermal amplitude and associated freezing and thawing rates. Although these tests are accelerated performance tests, the performance of the climatic chamber may be highly influenced by such large applied thermal amplitude. As a consequence, a difference of 10°C was detected at a time t during the test between the temperatures of the brine solutions of two specimens placed in two neighboring positions in the chamber during scaling test. A difference in minimum temperature reached in brine solutions of the different tested specimens was also found. Knowing the importance of the thermal conditions in concrete salt-frost resistance testing, the reliability of the test may be questioned especially that in a standardized performance method many specimens are tested at the same time, therefore, the result may depend on the position of each one inside the climatic chamber. This dispersion found in applied thermal conditions in the chamber may be also a reason behind the lack of repeatability of scaling results in case of a laboratory testing (high discrepancies were detected in scaling results of laboratory specimens).

On the other hand, the slab test method was found to be incapable to qualify correctly all types of concrete, only concrete with poor or very good scaling resistance may be qualified correctly.

In view of all the cited factors, new studies have to be made aiming to propose a method that may be able to provide a stable correlation between laboratory and field results. A reflection about the relevance of the applied normative freeze-thaw cycle and its representativeness of field conditions is proposed. A new freeze-thaw cycle needs to be found, based on meteorological observations made for different countries with moderate to severe cold climates. The proposed freeze-thaw cycle has to ensure homogeneous thermal conditions inside the climatic chamber during scaling test. Finally, the repeatability as well as reproducibility of the test with the proposed cycle must be assessed and compared to results obtained with the current standardized testing methods. This is the subject of a research work in progress.

5 Declaration of conflicting interests

The authors declared no conflicts of interest with respect to the research, authorship, and/or publication of this work.

6 Acknowledgments

The financial support of this work by ATILH (Technical Association of French Cement Industry) now absorbed by SFIC (Professional Syndicate of French Cement Industry) and the grant by ANRT by mean of CIFRE convention n°2017/891 for Sara AL HAJ SLEIMAN are great fully acknowledged.

The authors address sincere thanks to SigmaBéton laboratory of VICAT for providing technical support in producing concrete blocks and the technical maintenance of the recording system. The authors thank as well Meteorological-France team in Mont Aigoual (Chantal Vimpere and Christian Pialot) for their technical assistance and on-site maintenance.

REFERENCES

- [1] Powers, T., The air requirement of frost resistant concrete, in *Proceedings of the Twenty-Ninth Annual Meeting of the Highway Research Board*, 1949, :184–211.
- [2] Powers, T. C., and Helmuth, R. A., Theory of volume changes in hardened Portland cement paste during freezing., in *Highway Research Board Proceedings 32*, 1953, 32, :285–297.
- [3] Scherer, G. W., 2004. Stress from crystallization of salt *Cement and Concrete Research* 34:1613–1624.
- [4] Scherer, G. W., 1999. Crystallization in pores *Cement and Concrete Research* 29:1347–1358.
- [5] Rhardane, A., Al Haj Sleiman, S., Alam, S. Y., and Grondin, F., 2021. A quantitative assessment of the parameters involved in the freeze–thaw damage of cement-based materials through numerical modelling *Construction and Building Materials* 272:121838.
- [6] Valenza, J. J. and Scherer, G. W., 2007. A review of salt scaling: II. Mechanisms *Cement and Concrete Research* 37:1022–1034.
- [7] Valenza, J.J., Scherer, G. W., 2005. Mechanisms of salt scaling *Materials and Structures* 38:479–488.
- [8] Verbeck, G. . and Klieger, P. ., 1957. Studies of Salt Scaling of Concrete *Highway Research Board Bulletin*:1–17.
- [9] Valenza, J. J. and Scherer, G. W., 2007. Mechanism for salt scaling of a cementitious surface *Materials and Structures/Materiaux et Constructions* 40:259–268.
- [10] Sellevold, E. J. and Farstad, T., Frost/Salt-testing of concrete: Effect of Test Parameters and Concrete Moisture History, in *Nordic concrete research*, 1991, :121–138.
- [11] Lindmark, S., Mechanisms of Salt Frost Scaling on Portland Cement-bound Materials : Studies and Hypothesis, Lund Institute of Technology, Division of Building Materials, Lund, Sweden, 1998.
- [12] Marchand, J., Contribution a l'étude de la deterioration par ecaillage du beton en presence de sels fondants, Ecole Nationale des Ponts et Chaussées, 1993.
- [13] Çopuroğlu, O. and Schlangen, E., 2008. Modeling of frost salt scaling *Cement and Concrete Research* 38:27–39.
- [14] CEN/TC51 "Cement and building limes", FprCEN/TS 12390-9:2015-Testing hardened concrete — Part 9 : Freeze-thaw resistance - Scaling - Complementary element, 2015.
- [15] AFNOR, NF XP P18-420 AFNOR Béton : Essai d'écaillage des surfaces de béton durci exposées au gel en présence d'une solution saline.
- [16] International, A., ASTM C672 / C672M-12, Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals, West Conshohocken, 2012.
- [17] Fagerlund, G. and Janssen, D. J., 1996. CDF Test – Test method for the freeze-thaw resistance of concrete - tests with sodium chloride solution (CDF) Recommendation 29:523–528.
- [18] Izoret, L., Al Haj Sleiman, S., Matoiri-Chaibati, N., and Grondin, F., 2021. Concrete in a severe freezing environment: a meteorological characterization *Materials and Structures* 54:36.

- [19] AFNOR, NF EN 206-1/CN: Spécifications, performances, production et conformité, 2014.
- [20] (LCPC), L. C. des P. et C., *Recommandations pour la durabilité des bétons durcis soumis au gel*, IST-Diffus. France: Laboratoire central des Ponts et Chaussées, 2003.
- [21] Université de Sherbrooke, *Durabilité et réparation du béton*. Sherbrooke.
- [22] AFNOR, NF XP P18-462 AFNOR Béton : Essai accéléré de migration des ions chlorure en régime non-stationnaire. .
- [23] AFNOR, NF XP P18-459 AFNOR Béton : Essai pour béton durci Essai de porosité et de masse volumique. 2010.
- [24] Finnerup, T., Freeze-Thaw Resistance of Concrete Effect of: Curing Conditions , Moisture Exchange and Materials, The Norwegian Institute of Technology, Division of Structural Engineering, Concrete Section, 2001.
- [25] Costruzioni, F., Structures, C., and Sia, R., SIA 262/1:2019 Construction en béton – Spécifications complémentaires, 2019.
- [26] Kaufmann, J. P., 2004. Experimental identification of ice formation in small concrete pores *Cement and Concrete Research* 34:1421–1427.
- [27] Bokor, B., Kajtár, L., and Eryener, D., 2017. Nocturnal Radiation: New Opportunity in Building Cooling *Energy Procedia* 112:118–125.
- [28] Parker, D. S., Sherwin, J. R., and Hermelink, A. H., 2008. NightCool : A Nocturnal Radiation Cooling Concept Description of the NightCool Concept *ACEEE Summer Study on Energy Efficiency in Buildings*:209–222.
- [29] J. Marchand J. Boisvert, H. L. Isabelle, and O. Houdusse, M. P., Deicer Salt Scaling Resistance of Roller-Compacted Concrete Pavements Containing Fly Ash and Silica Fume *ACI Symposium Publication* 132.
- [30] Fabbri, A., Coussy, O., Fen-Chong, T., and Monteiro, P. J., 2008. Are Deicing Salts Necessary to Promote Scaling in Concrete? *Journal of Engineering Mechanics* 134:589–598.
- [31] Bouteille, S., 2013. Outils de caractérisation et analyse du comportement des matériaux cimentaires soumis à des cycles de gel-dégel en présence de sels - Université PARIS-EST:303.
- [32] Lindmark, S., *Division of Building Materials Mechanisms of Salt Frost Scaling of Portland Cement-bound Materials : Studies and Hypothesis*. 1998.
- [33] Jacobsen, S., Saether, D. H., and Sellevold, E. J., Frost durability of high strength concrete: Frost/salt scaling at different cooling rates, in *Frost Resistance of Building Materials: Proceedings of the 2nd Nordic Research Seminar*, 1996, :69–87.
- [34] Valenza, J. J. and Scherer, G. W., 2007. A review of salt scaling: I. Phenomenology *Cement and Concrete Research* 37:1007–1021.
- [35] Utgenannt, P., Influence of carbonation on the scaling resistance of OPC concrete, in *Rilem Workshop*

- on frost damage in concrete*, :104–114.
- [36] Petersson, P.-E., Scaling resistance of concrete field exposure, in *Frost Resistance of Building Materials: Proceedings of the 2nd Nordic Research Seminar*, 1996, :11–38.
 - [37] Rønning, T. F., Field performance versus Laboratory testing of freeze-thaw: various cement and concrete qualities, in *Workshop proceeding no. 8: Nordic miniseminar Hirtshals: Input to revision of EN206-1*, 2008, :153–179.
 - [38] Muller, C., Results of the Laboratory Freeze-Thaw Tests and their Transferability to Practical Conditions, in *Freeze-Thaw testing of concrete-Input to revision of CEN Test Methods. WORKSHOP PROCEEDING FROM A NORDIC MINISEMINAR*, 2010, :95.
 - [39] Utgenannt, P., Salt-frost resistance of concrete in highway environment, in *Proceedings of the 3rd Nordic Research Seminar in Lund on Frost Resistance of Building Materials*, 1999, :137–145.
 - [40] Harrison, T., Equivalent durability concept, in *Input to revision of EN 206-1 Workshop Proceeding from a Nordic miniseminar*, :1–13.
 - [41] Janssen, D. J., Requirements for a test of frost resistance of concrete, in *International RILEM Workshop on Frost Resistance of Concrete*, 2002, :277–286.
 - [42] Girodet, C., Endommagement des mortiers sous sollicitations thermo-mécaniques: Influence des caractéristiques des matériaux sur la résistance au gel interne, Institut National des sciences appliquées de Lyon, 1996.
 - [43] Ghazy, A., Concrete pavements: deterioration due to de-icing salts and repair, University of Manitoba, 2017.
 - [44] Marchand, J., Pleau, R., and Gagné, R., *Deterioration of concrete due to freezing and thawing*, Am. Ceram. Westerville, OH, 1995.
 - [45] Marchand, J., Pigeon, M., Résistance du béton à l'écaillage dû au gel en présence de sels fondants - Une revue des récents développements dans le domaine, in *Conférence RILEM: Béton - Du matériau à la structure*, 1996, :179–2010.
 - [46] Müller, A., Fuhr, C., and Knöfel, D., 1995. Frost resistance of cement mortars with different lime contents *Cement and Concrete Research* 25:809–818.