

## ***Supplementary material***

### **Durability performance assessment of non-standard cementitious materials for buildings: a general method applied to the French context**

#### **Fact sheet 4 - Risk of concrete damage by internal freezing**

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#### **1 Introduction**

This type of degradation affects hardened concretes that are subjected to "severe" or "moderate" freezing conditions. For France, the geographic zones concerned are defined in the booklet FD P 18-326-2004 [1]. Generally speaking, the definition used comes from the 2003 LCPC Recommendations guide [2]: the areas of severe freezing reach temperatures below -10°C more than ten days out of the year and the areas of moderate freezing reach a temperature below -5°C more than two days per year. This fact sheet only addresses degradation linked to simple freezing, called "internal freezing", i.e. freezing in the absence of de-icing salts (see the dedicated sheet on concrete scaling during freeze/thaw cycles).

#### **2 Consequences**

- Swelling of the concrete.
- Internal micro-cracking.
- Decrease of mechanical performance (bulk modulus, compressive strength).

- Aggregate are dislodged due to loss of cohesion with the hydrated cement paste leading to the destruction of pieces of concrete in extreme cases.

#### **3 Physiochemical mechanisms**

When a cementitious material, partially saturated with interstitial solution, is subjected to freezing and thawing temperature cycles in the presence of humidity, internal damage by freezing may occur. The risk of degradation is related to fluid pressure after some of the water contained in the porous network freezes. There are several theories that may describe the phenomenon, but none gives a perfectly satisfying explanation for all experimental evidence. The most commonly used is explained below.

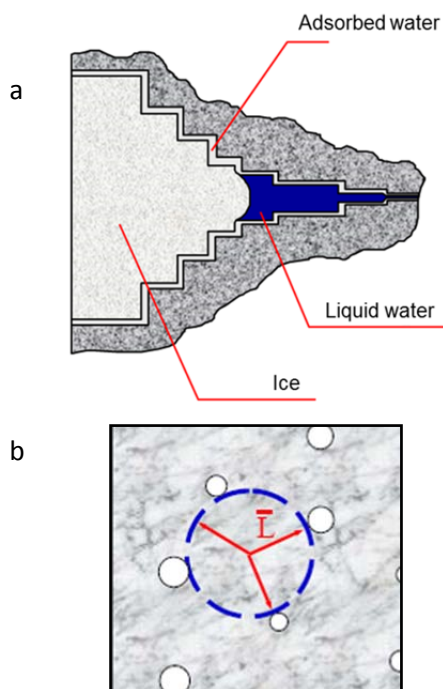
Powers' theory of hydraulic pressure [3]:

When the temperature falls, the water contained in the porous network freezes progressively based on capillary diameter (first in the largest pores, then in the smaller ones). As it freezes, the volume of this water increases, creating hydraulic pressure in the water that remains in a liquid state (see Figure on the left below). This increase in liquid pressure creates stresses in the cement paste, which may lead to its

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cracking if the stresses generated exceed the material tensile strength.

It has been observed in poured concrete that the presence of dispersed air voids in the paste improved resistance to freezing/thawing cycles. Air content seems to help reduce hydraulic pressure by facilitating the flow of unfrozen solution. By applying Darcy's law to the flowing fluid, Powers [3] was able to determine the ideal distribution of air voids in order to limit the generated stresses. That is why concrete is often formulated with an air-trapping agent that stabilises the air that is incorporated during mixing. The distribution of air voids is characterised by a spacing factor ( $\bar{L}$ ), which is the average half distance between voids in the paste (see Figure 1b) and the testing method in section **Error! Reference source not found.**). Taking a "critical" threshold into account helps to ensure proper resistance to freezing/thawing cycles. It should be noted that concrete can still be quite resistant without this network of voids in certain cases (for example, UHPC, HPC with a W/C ratio below 0.32). On the other hand, staying below this threshold does not, in and of itself, guarantee that the concrete will behave well when frozen if it has an unconventional composition, e.g. an aggregate that is sensitive to freezing, or a paste with insufficient tensile strength, etc.



**Figure 1.** Ice formation in a pore as a function of radius (a) [13] and spacing factor ( $\bar{L}$ ) (b).

#### 4 Main models

There is no agreed-upon predictive model (as of 2015). Q. Zeng's thesis [4] offers a bibliographical synthesis on the topic.

#### 5 Influential parameters

*Material parameters:*

- Concrete composition:
  - type and frost-sensitivity of the aggregate; the concrete must be formulated with aggregate or other inclusions that are not susceptible to frost),
  - presence or absence of a network of air voids, and, if they are present, the characteristics of this network,
  - structure of the porous network of the cement paste described by its pore size distribution.
- Age of the product – Strong impact on concretes that experience freezing before 28 days curing.
  - Concrete may be susceptible to frost starting with the first few exposure cycles. The age of the material can change its susceptibility to frost: significant damage may be observed in case of early exposure, for example.
- Methods of concrete placing: poor compacity if there is insufficient vibration, bad air void network because of pumping conditions, drop height, excessive vibration,...
- Mechanical tensile strength (generally indirectly assessed by measuring compressive strength).
- Water saturation of the product: the higher it is, the more susceptible to frost is the material.

*Environment parameters:*

- Cycle type (thermal and hydrological conditions).
  - Based on the type of cycle (amplitude, frequency) and thermal diffusion, the product may be frozen to its core.
- Degree of inclination of the structure (horizontal, drained surface, surfaces).

#### 6 Testing method stages

- Compaction methods, which strongly influence the structure of the porous network, especially when the concrete contains air voids.
- Age of the concrete.
- Curing duration and conditions (temperature, generally stored in water): a 28-day cure is common for cement materials. The methods can be adapted based on the specificities of the material and its application, especially the amount of time it takes for the properties to stabilise. In all cases, this duration must be compatible with the age of the concrete when it is first exposed to freezing temperatures.
- Creation of measurement plots to track deformation (if applicable).
  - according to the reference testing methods.
- When measurements are taken:

- sample thawing conditions before measurement.
- measurements of the resonance frequency and the relative expansion.

The quantities of interest may be:

- Spacing factor (if necessary).
- Expansion (non-destructive measurement), changes in the resonance frequency or in the speed of sound (also non-destructive measurements).
- Loss of mass (non-destructive measurement).
- Residual mechanical properties (destructive measurement).

## 7 Standard testing methods

Apart from the air void network characterisation tests, the other performance tests generally involve exposing concrete samples to freezing/thawing cycles, then detecting any degradation after a certain number of cycles. There are other specific standards that can be applied to particular concrete industrial products (for example, standard NF P 98-052 2002 [5] for monoblock window supports).

It should be noted that there is a specific test for freeze/thaw cycle resistance for aggregate, which may be applied to a new product of this kind.

**Table 1.** Tests listed in current provisions valid in the place of use for conventional concrete:

Name	Cure	Thermal cycle	Duration	Comment
ASTM C 457M :2012 [6]	7 days under water.	-	total time: about 1 week, including 2h of counting.	Measurement: spacing factor.
NF EN 480-11 :2006 [7]	7 days under water.	-	total time: about 1 week	Measurement: spacing factor by image analysis. Used to characterise additive effectiveness.
NF P 18 424 [8]	28 days under water. T=20°C±2	300 cycles [-18°C ; + 9 °C] 4 cycles / 24 hours. Freezing in water and thawing in water	3.5 months	Concrete performance test
NF P 18 425 [9]	28 days under water. T=20°C±2	300 cycles [-18°C ; + 9 °C] 4 cycles / 24 hours. Freezing in air and thawing in water	3.5 months	Concrete performance test
PD CEN/TR 15177 :2006 [10]	In a closed system for 6 days then under water for 21 days T=20°C±2	56 cycles [-20°C ; + 20°C] 2 cycles / 24 hours. Freezing in air and thawing in water	56 days	Concrete performance test
NF EN 1367-1 :2007 [11]	1) Washing, 2) drying at 110°C±5.3) imbibition under water at 20°C±5 for 24±1 hours	10 cycles [-17.5°C ; + 20°C] 1 cycle/24 hours. Under water	≈ 13 days	Aggregate test

## 8 Performance assessment

Assessment method:

- "absolute" binary assessment: if the measured quantity of interest of frost-resistant material meets the corresponding critical threshold values, the product is considered as performing and conform.

For the standards NF P 18-424 [8], NF P 18-425 [9], and ASTM C 457M [6], mentioned in the table above, the conformity interpretation thresholds are defined in the 2003 LCPC Recommendations guide [2].

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