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Supplementary Materials.

Fact sheet 7
Risk of degradation by an Internal Sulphate Attack (ISA)

1. Introduction

The risk of Delayed Ettringite Formation (DEF) affects concretes that have been heated over 65°C for several hours or several days after installation and that are made up of a cement matrix whose physio-chemical characteristics favour DEF. Only DEF, a specific form of Internal Sulphate attack (ISA), is addressed here. Damage may occur if these concretes are then exposed to a very humid environment.

This scenario may affect concrete structures that generate great heat during binder hydration that is only partially evacuated to the exterior (large structures, compositions with high hydration heat cements, use of insulating formwork), or those that are subjected to high temperatures at a young age (concrete poured during hot weather, excessive thermal treatment). The degradation is linked to late formation (over several years) in humid environment of large deposits of very expansive secondary ettringite that can ruin the structure.

N.B.: For Portland cement-based concrete, the basic parameters are water, the maximum temperature reached, how long this temperature is maintained, the sulphate and aluminate content of the cement and the alkali content of the concrete. It has been shown at the laboratory scale that if temperatures exceed 65°C and if the other basic parameters are present, DEF may develop. Even for materials that do not experience ettringite formation, excessive heating at a young age should be avoided in order to prevent thermal stress due to self-restraint and the appearance of thermal cracks when the concrete cools down.

2. Consequences

- Large and deep cracks in the core of concrete.
- Very significant loss of mechanical properties.
- Increased risks of reinforcement corrosion and failure due to cracking of the concrete cover.

3. Physiochemical mechanisms

The conditions that lead to harmful swelling due to an ISA are not yet fully understood. This phenomenon was only recently identified as opposed other known concrete pathologies (in the 1980s) and the link between laboratory tests and behaviour in the field is tenuous. All mechanisms are not yet fully understood, but the current research work suggests that the degradation is the result of a combination of the following mechanisms:

- if the temperature of the concrete is over 65°C during the days after its hydration begins, primary ettringite, which forms normally, does not form or is unstable and disappears. Sulphate and aluminate ions that are not used to make primary ettringite form calcium monosulfoaluminate microcrystals that are dispersed throughout the C-S-H; sulphates also adsorb onto the C-S-H surface whereas aluminium is integrated into the C-S-H structure [5].
- A pessimum effect linked to temperature/heating duration appeared after some laboratory tests [6] [7]. This means that there is no one threshold temperature, but rather a specific threshold temperature for each case study. This is for example the reason why short heat treatments used in the precast concrete industry are permitted by the ISA recommendations [3], despite the use of temperatures above 65°C.
- if, over the months and years following placing, the material is in frequent contact with free water (stagnant water, immersion, etc.), the calcium monosulfoaluminate microcrystals will react with the sulphate and aluminate ions freed by the C-S-H to form massive and expansive secondary ettringite. In this instance, the cement paste is subject to generalised expansion that leads to cracking of concrete. This reaction is generally quite slow and the first signs of structural degradation often do not appear for at least 10 years. Wetting/drying cycles seem to accelerate the formation of secondary ettringite.

4. Main models

There are existing models to predict the temperature rise in concrete structures, allowing for preventive measures to be taken. The most complete model is the CESAR-LCPC model [1] developed by IFSTTAR; it returns 2D or 3D temperature maps of concrete pieces over time). The RGIB module [2] of this model allows resulting expansion due to an internal reaction (alkali-aggregate reaction and internal sulphate attack) to be estimated.

As a first approach, appendix IV of the LCPC Recommendations [3] may be used, which gives a formula for estimating the maximum temperature reached within a concrete element.

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5. Influential parameters

"Material" parameters that contribute to temperature rise within concrete:

- dimensions of the concrete element,
- binder type and content within the concrete:
  - hydration heat of the cement,
  - the presence of one or several reactive additives (slag, fly ash, natural pozzolans, metakaolin, and silica fumes) in specific proportions can reduce the increase in temperature when the additives are used as clinker replacement,
- type of aggregate.

"Material" parameters that facilitate ISR in the case of high temperatures at a young age:
For materials that meet current concrete standards, the current parameters are given below:

- binders that are rich in aluminates, sulphates, and alkalis are the most susceptible to this phenomenon (nevertheless, using a CEM I SR cement is not enough to guarantee that no ISA will occur),
- the presence of one or several additives may, depending on the type used, promote the binding of sulphates in non-expansive forms (increase of the critical threshold temperature [3]).

"Environment" parameters that contribute to rising temperatures within the concrete:

- temperature of the concrete's constituents when they are mixed and the temperature of the concrete when it is placed,
- external temperature when the concrete is placed and in the days that follow,
- the presence of an insulating formwork,
- thermal treatments: maximum temperatures that are too high and periods of high temperature that are too long should be avoided [3].

"Environment" parameters that facilitate ISA in the case of high temperatures at a young age:

- frequent wetting/drying cycles or constant contact with free water.

6. Testing method stages

The link between laboratory tests and the reality in the field requires further study because this type of degradation was only identified recently and remains relatively rare. The laboratory test developed and published by IFSTTAR in 2007 is one example [4]. It involves proving whether the ISR phenomenon can develop for a given concrete composition and for a given thermal heating:

- Samples of concrete (with measuring inserts to track dimensional variation) are made, then quickly exposed to a heat treatment that is characterized by the speed of heating, the duration of the period of sustained heat, the maximum temperature, and the speed of cooling. There is no standard treatment suggested by the test. This treatment would need to reflect a real exposure scenario, whether known or estimated, as indicated in section 4.
- The samples are then subjected to wetting/drying cycles. This cycles help to accelerate the degradation mechanisms, shortening them by several weeks.
- The test samples are then stored in water. Weight and dimensional variation measurements are taken regularly for at least one year. Based on the observed trends, the total duration of the test may be between 12.5 and 15.5 months, including the initial treatment. A SEM (Scanning Electron Microscope) image analysis should be done at the end of the test to make sure that the degradation is in fact the result of expansive secondary ettringite formation.

The quantities of interest are:

- weight and dimensional variations of the test samples.
- the massive formation of ettringite in the material after testing, observed, for example, with the SEM.

7. Standard testing methods

For France, the test was developed by IFSTTAR and published in 2007.

Name	Initial treatment	Pre-conditioning	Measurements	Total test duration	Threshold	Comment
LCPC-ME66: 2007 [4]	Heat treatment representing the heat history of the concrete structure until it reaches ambient temperature.	2 cycles (of 7 days each) of drying (RH < 30% [38 ± 2]°C)/ wetting (RH = 100% [20 ± 2]°C) Then storage in water at [20 ± 2]°C.	Weight and dimensional variations every 7 days for 2 months, then every 15 days for 4 months, then every month	Between 12.5 and 15.5 months including the initial treatment	Criteria according to [4]	This qualitative test is generally seen to be conservative. The standard deviation is quite high.

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8. Performance assessment

Assessment method:

- "absolute" assessment: the quantity of interest should remain below the thresholds recommended in the LCPC ISR guide [3] and the LPC No. 66 [4] testing method.

9. References

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