Characterization of DEF affected concretes: detection and modification of properties

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Many engineering structures (dam, bridge, nuclear structure) are susceptible to develop Internal Sulfate Attack (ISA) by Delayed Ettringite Formation (DEF). This reaction requires the presence of three main factors: high sulfate and aluminate contents, water, and a rise in temperature of concrete. This reaction leads a swelling of concrete materials generating microcrack, cracks, loss of material performances and structure damage.

The reaction mechanisms of DEF are complex. Brunetaud has proposed a global mechanism, described in four phases, grouping many theories to explain swelling (crystallisation pressure, osmotic pressure, homogeneous swelling, swelling at the Interfacial Transition Zone, electric double layer). The swelling can be represented by a sigmoidal curve of expansion of the time in various regime. This first phase corresponds to the dissolution of the primary ettringite. Secondly and under certain conditions, the latent period can begin (low expansion) with the precipitation of delayed Ettringite in the microstructure (porosity, Interface Transition Zone (ITZ), Hadley grain). When the tensile strength of concrete is reached due to internal pressure, cracks were generated (end of latent period). Thirdly, after an inflection point, the precipitation of delayed Ettringite in cracks, leads to cracks propagation. It is the acceleration of expansion. Finally, the deceleration of swelling occurs when one reactant is consumed and/or when the cracks opening is sufficiently significant to accommodate new products without generation of supplementary expansion.

Several parameters have an influence on the development of DEF, and can be divided into two groups: those related to the formulation of concrete (binder nature, water / cement ratio) and those related to the environment (temperature curing, relative humidity).

Main consequences of DEF are expansion and cracks. They affect the physicochemical and mechanical properties of the concrete. Cracks lead to an increase of transfer properties and a decrease of mechanical properties and may lead to a loss of tightness, which is harmful for nuclear power plants. Few data can be found in the literature on the evolution of these properties as a function of the degree of advancement of DEF.

The present study had two objectives: first, to find a sensitive test allowing DEF to be detected before cracking can be observed visually and, second, to evaluate the evolution of the properties that impact the tightness and also the durability of concrete. A test protocol to accelerate DEF development was carried out and provided a good representation of the reality on site. A large number of samples were used to characterize the impact of DEF on concrete properties at different levels of expansion. Chemical tests were performed to observe the initiation and the presence of DEF, while physical and mechanical tests evaluated the impact of DEF on mechanical and transfer properties.

In this study, two cements and two types of aggregates were used. The choice of cements focused on a CEMI 52.5N and a CEMII/A-LL 42.5. For aggregates, siliceous and limestone ones were used. The proportion of aggregates was determined with respect to Dreux method in order to reproduce the same aggregate skeleton. The choice of siliceous and limestone aggregates respectively with CEMII and CEMI was conducted to reproduce mix design representative of nuclear structures. They have also been used for concrete blocks, as part of a new platform ODOBA test developed by the Radioprotection and Nuclear Safety Institute (IRSN), to study the impact of DEF degradation at scale structure. The concrete casting procedure and moulding (∅11x22cm^3) used in this study followed the protocol of French standards.

French-recommended performance test for DEF-reactivity MLPC #66 was adopted in order to allow a rapid development of DEF. This test comprises four stages: concrete mixing, heat treatment, dry/wet cycles, and final immersion of samples in water at 20°C. Once cast, specimens were placed in a climate chamber at controlled temperature and relative humidity. In container, the heat treatment was applied for 7 days and comprised four stages: a hold at 20°C, 95% RH for 2h, followed by a rise in temperature from 20°C to 80°C, 95% RH at a rate of 2.5°C/h, stabilization at 80°C, 95% RH for 72h and a temperature decrease from 80°C to 20°C, 95% RH at a rate of 1°C/h (Figure 3a). The effective thermal energy generated by the heat treatment was 1395°C.h.

The mass and expansion monitoring were performed on three cylindrical test pieces (11 x 22) cm3). Microscopic observations were performed to ensure the presence of DEF in the concrete. When the swelling was initiated (expansion of 0.04%), measurement of the physical properties (electrical resistivity and gas permeability) revealed the impact of the micro-cracking generated on the transfer properties. Finally, measuring the mechanical properties (compressive strength and elasticity modulus) gave some indications on the level of damage of the concrete subjected to these pathologies. 6 measurements of all the properties were made at different levels of expansion: latent, acceleration and stabilization phases.

One of the first aims of this experimental program was to find a sensitive test allowing DEF detection. DEF can be detected if the evolution of one of the properties measured in this work is significant (sufficiently higher than the scatter on results) and only due to DEF expansion (not to concrete hydration). In this aim, a DEF detection criterion for physical and mechanical properties was developed, taking the effect of hydration (ageing) and experimental scatter into account. These threshold values are used for the detection of DEF, presented in the charts for each pathological concrete. The evolution part of properties due to hydration was considered by the measurement of reference concretes at 35, 90 and 180 days. This evolution might be different between pathological and reference concrete due to the thermal treatment in case of pathological concretes. However, it provides an estimation of the evolution of the properties if no pathology was developed. The evolution of threshold values is considered as constant between two property measurements (35, 90 and 180 days).

An experimental investigation has been carried out to characterize DEF impact on measurable physical and mechanical properties of concrete. The second aim was to evaluate the evolution of the properties that have an impact on the tightness of concrete. Several physicochemical and mechanical properties were measured by the use of a large quantity of samples. These tests were performed on two concretes showing a high swelling potential. Original results have been obtained:

- Electrical resistivity and gas permeability are sensitive to the expansion generated by DEF. However, the electrical resistivity could be difficult to use for damaged structures.
- For a same expansion, the apparent permeability increase is greater for concrete containing siliceous aggregate than for concrete with limestone aggregate.
- The measurement of the elastic modulus and electrical resistivity seems to allow the detection of DEF during the latent period.
- Three durability indicators seem to be relevant for the detection and the monitoring of pathology: gas permeability, electrical resistivity, and elastic modulus.
- For the investigated concretes, the loss of tightness related to the presence of DEF occurs for less than 20% of damage.

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