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Five year drying of high performance concretes: Effect of temperature and cement-type on shrinkage

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ABSTRACT

This experimental study imposes limited relative humidity (*RH*) gradients to small mature concrete samples, at a constant temperature $T = 20$, 50 or 80°C. Mass loss and shrinkage are recorded until stabilization at each *RH* and *T*, for up to 1991 days. Firstly, our mass loss data are consistent with those presented in former research (on different samples of the same batch). After presenting and analyzing shrinkage kinetics, experimental data are fitted with usual models for shrinkage prediction, at each temperature of 20, 50 and 80°C. An adequate match is obtained by combining capillarity (i.e. Vlahinic's model coupling poro-elastic constants and water saturation level) and desorption (Bangham's equation).

Subsequently, relative mass variation (*RMV*) is plotted against shrinkage ε_{sh}^{dry} data. Three distinct phases are obtained at 20 or 50°C and down to 30%*RH*; up to four distinct phases are observed at $T = 80$°C and down to 12%*RH*. The latter are confirmed by experiments on (60°C; 7%*RH*) dried concrete. The four phases in the $(RMV, \varepsilon_{sh}^{dry})$ diagram are interpreted against shrinkage data on mature cement paste dried at 60°C; 7%*RH* and against the literature.

1. Introduction

1.1. Industrial and scientific context

The French National Agency for Nuclear Waste Management (Andra) is in charge of the design and safety of a deep geological disposal for High Level and Intermediate Level-Long Lived radioactive Wastes (HLW and IL-LLW).

In particular, during the time from the tunnel drilling phase, the building of the retaining structure, the operating phase and until the progressive closure of the repository, partial de-saturation is bound to occur within the geological medium, and in concrete components of the engineered structure, mainly due to ventilation and human activities [1,2]. Moreover, HLW and some IL-LLW wastes may generate heat: in their vicinity, temperature may reach up to 50 °C, and in specific conditions, temperature may even display peaks up to 70 to 80 °C [1,2].

In concrete components, a major consequence of the coupled thermal and hydrous loadings is drying shrinkage. The latter generally induces micro-cracking, even in unconstrained conditions [3–5]. At the structure scale, temperature-activated shrinkage may induce the opening of interfaces between concrete components, or even a de-cohesion with the host rock. In such context, we anticipate the creation of preferential pathways for fluid release (gas and aqueous species). Therefore, identifying drying shrinkage quantitatively, at specific drying and thermal conditions relevant to the *in situ* situation, is a key point. This will allow to take adequate preventive measures, e.g. in the design of dilation joints.

1.2. Characterization of shrinkage under temperature

For concrete, shrinkage under temperature refers mainly to maturation, drying and thermal deformations, named respectively ε_{sh}^{matur}, ε_{sh}^{dry} and ε_{sh}^{th} [6]. Maturation shrinkage ε_{sh}^{matur} occurs during cement paste hydration, whereas thermal shrinkage ε_{sh}^{th} is due to temperature decrease, e.g. after the hydration heat peak is reached. ε_{sh}^{th} is generally expressed with concrete linear thermal expansion coefficient, which is on the order of $10–20 \times 10^{-6}$ °C^{-1}, and which may depend on moisture conditions [6]. For mature concrete, ε_{sh}^{dry} is the consequence of moisture changes, i.e. of variations in the relative humidity (*RH*)

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surrounding concrete, at constant imposed temperature T.

For a given concrete structure, Bažant [7] has shown that drying is dimension-dependent, with greater shrinkage close to the surface than in the core, due to the moisture gradient between the two. Drying shrinkage may also extend over many years, depending on the structure scale. Wittmann [8] advises to consider an infinitesimal shrinkage over a limited concrete volume, which represents a constant length change at given *RH* and *T*, characteristic of the sole material, and not of the structure too. Due to this, in the following, drying is performed in small decreasing hydric steps i.e. by imposing limited *RH* gradients, and concrete sample dimensions are limited to about three times the size of the biggest aggregate.

Although maturation and drying shrinkages do not correspond to the same lifetime periods of concrete, it is widely accepted that several similar mechanisms are involved [6,8-17], depending mainly on *RH* (Appendix A.1).

1.3. Aims and scopes

In this study, we focus solely on maturation and drying shrinkage: no thermal shrinkage, due to temperature changes, is considered. ε_{sh}^{dry} at 50 and 80 °C is assessed after cooling down to 20 °C in airtight conditions (residual measurements). The experiments are performed on two high performance concretes. Their formulation is based on pure Portland CEMI and blended CEMV/A-type cements (European standard denominations). Samples of a few centimeters size are prepared from mature concrete, kept for more than 6 months curing at 20 °C under lime-saturated water.

From this initial water-saturated state, drying is imposed at a constant temperature *T* of 20, 50 or 80 °C under progressively decreasing relative humidity *RH* = [98%, 90%, 80%, 70%, 65% (only at 50 °C), 60%, 50%, 30%, 12%]. This allows comparison between both concrete types, in terms of drying (relative mass loss) and shrinkage. Each *RH* step is changed only after sample mass stabilization, so that these experiments have lasted more than five years (1991 days) for the first desorption alone. The reference dry state is measured on independent samples, oven-dried at 105 °C until mass stabilization. Our experimental data are fitted with usual models for shrinkage prediction, at each temperature of 20, 50 and 80 °C. We also provide relative mass loss vs. shrinkage data, which are reputed independent of sorption/desorption cycles [18].

Finally, quicker, independent and complementary drying experiments are performed at *T* = 60 °C and 7%*RH*, on concrete samples of the same batch, and on cement pastes of the same formulation as the concretes. These confirm the specific observations associated with drying at 80 °C and 12%*RH*.

2. Experimental methods

This experimental campaign is undertaken in parallel with that of first desorption isotherms under temperature [2], by using the same concrete batches. This means that water retention and shrinkage properties may be closely related between [2] and this study. A graphical summary of the experimental campaign is given in Fig. 1.

2.1. Materials and sample preparation

2.1.1. Concrete samples

Details on concrete manufacturing are given in [2]. Table 1 presents the full formulation of both concretes, where 'CEMI' refers to the concrete made with pure Portland cement, while the second concrete is labelled 'CEMV'.

Eighteen prismatic (4*4*16) cm^3 samples are made per concrete. Owing to a maximum aggregate size of 1.2 cm, this sample size is considered sufficiently representative of the material macroscopic structure (it is three to 13 times the biggest aggregate size). Prior to concrete casting, standard end studs are placed inside the moulds in order to measure sample shrinkage.

Cylindrical samples of 37.5 mm diameter are also prepared. For each concrete, six samples of 50 mm height are used for length change measurements. All other cylindrical samples are used solely for dry mass assessment (Sub-section 2.2.2).

All materials are cured in lime-saturated water at 20 °C for six months. During this period of storage under water, maturation shrinkage is measured (Sub-section 2.2.3).

2.1.2. Cement paste samples

Cement pastes of equivalent formulation to CEMI and CEMV concretes are manufactured, by mixing only the cement and the tap water in the proportions indicated in Table 1, and cast into steel moulds of 36 mm diameter and 150 mm height. After un-moulding, samples are cured under lime-saturated water for six months, and cut to 63–84 mm length.

2.2. Experimental methodology

2.2.1. Progressive drying methods

After six months maturation, for each concrete and target temperature *T* = 20, 50 and 80 °C, three prismatic samples are dried progressively in climatic chambers, with the following *RH*-values: *RH* = [98%, 90%, 80%, 70%, 65% (only at 50 °C), 60%, 50%, 30%, 12%]. Three supplementary samples are sealed in airtight aluminum sheets to prevent moisture changes, and placed in the climatic chambers together with the unsealed samples, in order to measure autogenous shrinkage. Each *RH* value is changed as soon as mass stabilization of the unsealed samples is reached, i.e. when mass varies by less than ± 0.1% for three successive measurements. The mass measurement is performed twice a week for the first five weeks, and then once a week. Moreover, from *RH* = 80% and below, nitrogen gas is added in the climatic chamber after each measurement to prevent concrete carbonation, which is reputed maximal between 65 and 70 % *RH* [19]. With such a method, the experiment spans over five years to reach 12% *RH* at 80 °C and 30%*RH* at 50 and 20 °C.

For cylindrical concrete and cement paste samples, oven-drying is performed at 60 °C and *RH* = 7% ± 1. This brings intermediate mass and length change values when compared to *T* = 50 or 80 °C. Also, at such temperature, ettringite decomposition is assumed not to contribute to the observed changes, as its degradation starts at 70 °C [20,21].

Nota. For length measurement, hot samples (at *T* = 50, 60 or 80 °C) are brought out of their climatic chamber, wrapped in plastic sheets and placed in vacuumed bags. This hinders any exchange of moisture with their environment (hermetic sealing), because the mass change at the start and at the end of the cooling phase is negligible (less than 1%). Samples are stored at ambient temperature (T = 20 °C ± 1 °C and *RH* = 40% ± 5%) until complete cooling: this requires about four hours (± 30 min). After cooling, the samples are brought out of vacuumed bags successively, for mass and length measurement, and then placed back in their climatic chamber at fixed *T* and *RH*. This protocol is closely analyzed in Section 4, because *RH* changes upon cooling from 50–80 to 20 °C potentially generate significant shrinkage strains.

2.2.2. Relative mass variation RMV

The experimental protocol is described in more detail in Appendix A.2.1.

For each concrete sample placed at given temperature T = 20, 50, 60 or 80 °C, relative mass variation *RMV* is defined at mass stabilization (i.e. without moisture gradient inside the sample) as:

$$RMV = \frac{100\times\frac{m_{sat}^{sample} - m(RH)}{m_{sat}^{sample}}}{\%Mass} \tag{1}$$