Concrete drying kinetics: development of an accelerated drying protocol in fire testing

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RESUME Le comportement au feu du béton dépend fortement de la teneur en eau du matériau. A l'heure actuelle, des essais au feu sont réalisés sur des corps d'épreuve au moins après 3 mois de conservation en atmosphère ambiante. Ces essais sont coûteux, chronophage et considérés comme peu réalistes ainsi le béton à 3 mois n'aura pas la quantité d'eau qu'il aurait eu à plusieurs années de service. Ce travail propose un protocole de séchage accéléré qui permet d'atteindre un état hydrique et mécanique représentatif des conditions normales de service. Une campagne expérimentale sur quatre formulations de béton est réalisée, comprenant des essais de séchage (ambiant et accéléré) et des essais de résistance au feu à des durées et types de stockage différents. En parallèle, le protocole de séchage accéléré est proposé sur la base de la modélisation du séchage à température modérée. Selon l'étude prédictive, une teneur en eau cible peut être atteinte dans plusieurs conditions de séchage, montrant que des solutions ad hoc peuvent être proposées. La reproductibilité du protocole de séchage accéléré est essentielle pour assurer une application généralisée dans les laboratoires de recherche et industriels. L'étude étant en cours, seuls les premiers résultats seront présentés. Cet article se concentre sur l'évaluation de l'état hydrique.

Mots-clefs : séchage des bétons, teneur en eau, écaillage du béton.

I. INTRODUCTION

The fire behavior of concrete is a critical aspect of civil engineering since spalling can lead to extreme durability and safety problems. Fire characterization tests are therefore required to study and prevent spalling. The spalling of concrete is a complex phenomenon involving several mechanisms, such as water vapor pressure (liquid water can also be thermally pressurized), thermomechanical stresses, and deformation incompatibility between cement paste and aggregates (Hertz, 1984; Robert et al., 2008). Furthermore, it is strongly dependent on the hydric state and material permeability (Maier et al., 2020). The fire resistance test standard NF EN 1363-1 stipulates that at the testing time, the condition of the specimen should be representative of its normal service condition (mechanical strength and water content), considered to be obtained at equilibrium after storage in an ambient atmosphere. A conditioning period of at least three months is preconized. Therefore, drying processes play a crucial role in the characterization of concrete susceptibility to spalling and should be studied carefully. Concrete drying is a complex phenomenon involving several mechanisms, i.e., molecular diffusion, Knudsen diffusion, surface diffusion, and permeation. The hydric and hygric gradient between the material and its external environment generates the movement of liquid water, water vapor, and dry air. Several research works focus on describing the drying mechanisms (Bissonnette et al., 1999; Baroghel-Bouny, 2007) and show that concrete drying occurs in two main phases. The first phase is fast and lasts only a few hours to a few days. The second phase is much slower and evolves as a square root of time. This last phase is even slower in massive structures. So, the preconized conditioning period may underestimate the fire resistance due to higher spalling susceptibility associated with high water content. This shortcoming is also reported by (Lenglet, 2011), who observed that a longer normalized conditioning time reduces the susceptibility of concrete to spalling. This study aims at optimizing the fire test protocol by reducing the conditioning period via the definition of a hydric state representative of a structure with a few years of exploitation. The approach adopted is based on an experimental and numerical study of drying in concrete. The experimental campaign allows defining the material parameters (porosity, density, etc.), characterizing the drying kinetics (desorption isotherm test, mass loss monitoring, water content measurement, etc.), and validating the numerical predictions of the water profiles in concrete structures.

II. MATERIAL AND METHODS

A. Materials

Four mix designs covering two principal families of concrete were selected for this study. The table below shows the main characteristics of each mix design. The average compressive strength f_{cm} is measured at 28 days on 12 specimens per concrete. The porosity is measured at 28 days, also according to the AFPC-AFREM recommendations. The binder in the 4 concretes is CEM I 52,5 N. PERFDUB38 mix contains 8,4 % silica fume, and PERFDUB41 mix includes 25 % metakaolin. No additives are used in ordinary concretes: VERCORS and CETU.

TABLE 1. The main characteristics of the studied concrete formulations.

Concrete formula	Source	f_{cm} [MPa]	Porosity [%]
PERFDUB38	French national project PERFDUB ("Projet National PERFDUB," 2015)	86 ± 2,3	12,3 ± 0,2
PERFDUB41	French national project PERFDUB ("Projet National PERFDUB," 2015)	93 ± 3,3	11,7 ± 0,2
VERCORS	EDF (Charpin et al., 2021)	45 ± 2,0	15,4 ± 0,3
CETU (Containing polypropylene fibers)	CETU (Centre d'Etudes des Tunnels)	44 ± 3,3	21,1 ± 0,9

B. Concrete drying simulation

1. Drying at an ambient temperature

We briefly recall the nonlinear diffusion equation describing concrete drying. The formulation is based on the assumption of mass conservation and allows simulating the movement of water in its liquid and vapor forms in cementitious materials using the Darcy and Fick laws (Mainguy, 1999). The hydration and the wall effects are not considered in this model. The liquid phase is assumed to be incompressible, so the density is constant. The gaseous phase decomposes into dry air and water vapor. An isothermal temperature at 20 °C is considered (Mainguy et al., 2001; Thiery et al., 2007). The Van Genuchten model is used to simulate the desorption isotherm (van Genuchten, 1980). The balance equation used in the simulation reads:

$$\rho_l \left[\Phi \left(1 - \frac{\rho_v}{\rho_l}\right)\right] \frac{\partial S_l}{\partial t} = \mathrm{div}\left[\frac{\partial p_c}{\partial S_l}(K_{eq}) grad(S_l)\right] \quad (1)$$

where ρ_l liquid density, ρ_v vapor density, Φ porosity, p_c capillary pressure, S_l liquid water saturation degree, μ_l dynamic liquid viscosity, K_{eq} equivalent apparent permeability, T the temperature and t time.

The transfer properties are determined by calibrating numerical and experimental mass loss curves. The parameters of the Van Genuchten model are defined using a parametric study to identify the optimal set of parameters by the least-squares method applied to the experimental and numerical desorption isotherm curves. Boundary conditions are Dirichlet-type ones. (Zhang and Angst, 2021) show that this condition is sufficient to simulate drying in cementitious materials.

2. Drying at a moderate temperature

The choice of temperature is limited to moderate temperatures (up to 60 °C) to assume that no significant microstructural changes occur. For this study, the temperature is taken equal to 40 °C ± 2°C. The equivalent transfer coefficient is used in the drying simulation, as shown in equation (1). The temperature effect on the material parameters (porosity, density, intrinsic permeability, etc.) is negligible (CEN, 2004). We explicitly account for the temperature dependence of the water viscosity, the saturation vapor pressure (Bary et al., 2012), and the desorption isotherm (Chhun, 2017). The same method as at 20°C is used to identify the transfer and desorption isotherm parameters by calibrating numerical and experimental mass loss and isotherm curves at 40°C.

C. Experimental program

The reference geometry corresponds to the specimens of the fire test, i.e., slabs of dimensions 1.7 x 1 x 0.3 m^3. For technical, operational reasons and to simplify, the numerical study and the characterization of water profile evolution are carried out on cylinders Ø 15x30 cm which dry unidirectionally in height. This drying mode represents an element located in the middle of the slab.

1. Drying kinetics characterization

The experimental characterization of the drying process consisted firstly of measurements, at both 23°C and 40°C, of mass loss, porosity, and desorption isotherms for the numerical identification of the concrete transfer parameters. Secondly, water content measures were carried out at different conditioning durations to validate the numerical prediction. The water content profile is measured on slices of 2,5 cm thickness cut from a cylinder Ø 15x30 cm after drying unidirectionally.
All the tests were launched in the two conditioning modes:

- 23 °C, 50 % RH, which corresponds to the normalized conditioning (no specific cure before drying), and

- 40 °C, 65 % RH, which corresponds to the accelerated drying (defined numerically for each concrete) after 7-day conservation in sealed condition.

2. Fire testing

TABLE 2 summarizes all the fire tests performed so far and provides the different parameters of the tests for each family of concrete. Fire tests were carried out at three months of standardized conditioning correspond to the usual tests on concrete structures. And an examination under the ISO 834 fire curve is conducted on PERFDUB concrete proof bodies subjected to the representative accelerated drying protocol. This accelerated drying is representative of a 3-month drying period under standard conditions according to the first approach of the numerical study. The accelerated drying protocol was tested only on PERFDUB concretes. Indeed, the study's main objective is to set up an accelerated drying protocol representative of drying for a few years in ambient conditions. As fire tests are costly, as for a first evaluation they were carried out on PERFDUB concretes only, logically the most sensitive to fire due to their high compactness. The main reason behind that test is to have an initial assessment of the representativeness of accelerated drying in the short term and to be able to optimize it before carrying out a test in the long term. Long-term tests are planned for July and September 2022 for the four concretes.

TABLE 2. Assessment of the fire tests carried out to date.

Concrete	Fire curves	Loading [MPa]	Specimens	Conditioning period	Conditioning mode
PERFDUB38 and PERFDUB41	ISO 834	0 and 4	Two slabs (1,7 x 1 x 0,3 m^3)	90 days	Normalized conditioning
	ISO 834	0	Two slabs (1,7 x 1 x 0,3 m^3)	15 days	Representative accelerated drying
Vercors and CETU	HCM (HydroCarbure Majoré)	0 and 4	Two slabs (1,7 x 1 x 0,3 m^3)	90 days	Normalized conditioning

III. RESULTS AND DISCUSSION

A. Prediction of concrete drying under normalized conditioning

The following figure illustrates the numerical results after identifying material parameters for VERCORS concrete compared to the experimental results. FIGURE 1 shows the theoretical/experimental results of the average water content per 2.5 cm in VERCORS concrete. With only the first section, the theoretical and empirical results are very close considering the standard deviation on the experimental measurement. For the first slice, which corresponds to the skin area, the drying is underestimated, which may suggest the impact of wall effects (i.e., the effect of the formwork, microcracking, hydration evolution, etc.).

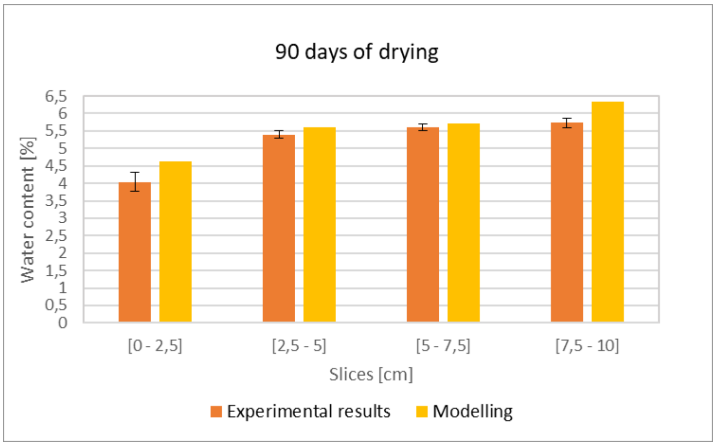


FIGURE 1. **Water content per cylinder Ø 15x30 cm slice of uni-directionally drying VERCORS concrete.**

B. Development of an accelerated drying protocol

1. Numerical prediction and validation

According to the numerical results, to reach a similar water profile as a 23 °C, 50 % RH curing after 90 days, it is sufficient to cure for 15 days at 40 °C, 65 % RH after 7-day conservation in sealed condition. This period is necessary to allow the hydration to progress. The allowed deviation is equal to ± 0,4 % in absolute value for the water content. It is set to be similar to the maximum uncertainty value in the experimental measurements after a literature review. Indeed, it is impossible to put a permissible deviation more restrictive than uncertainty. The accelerated drying protocol is tested in the short term on PERFDUB38 concrete. Water. The experimental results show good consistency between the moisture content measured in the two drying modes. The deviation is minimal and remains below the permissible deviation. This first test shows that it is possible to achieve a given moisture profile target by modifying the drying conditions and duration. This first short-term test of the accelerated protocol provides more confidence for the study continuation (cf. FIGURE 2).

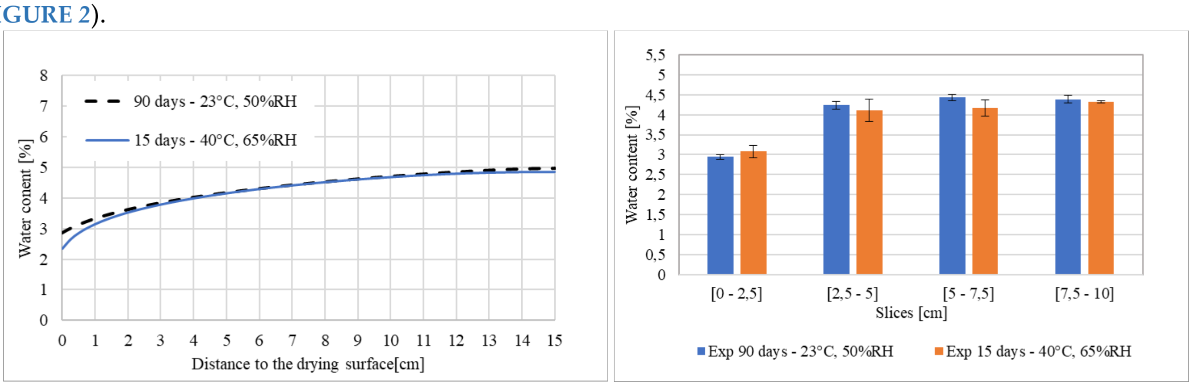


FIGURE 2. **Predicted water profile of a uni-directionally drying PERFDUB38 concrete after 90 days of normalized drying compared to the profile after 15 days of accelerated drying. On the right, the average water content per 2,5 cm from the drying surface to the core of Ø 15x30 cm cylinder.**

2. Effect of accelerated drying on microstructure

Carbonation and microcracking are the two main possible microstructural changes that may occur because of the drying acceleration at moderate temperature. (Drouet et al., 2019) have recently shown, through accelerated carbonation tests temperature in concretes formulated with CEM I at temperatures ranging from 20 °C to 80°C, that the increase in the carbonation depth is not significant. Therefore, the effect of the accelerated drying protocol (40 °C, 65 % RH) on the increase of the carbonation risk remains low. The evaluation of carbonation at 23 °C, 50 % RH, and 40 °C, 65 % RH confirms this hypothesis. The experiment consists of spraying Phenolphthalein on the Ø 11x10 cm cylinders, having dried for 13 months uni-directionally. At 23 °C, 50 % RH, the carbonate thickness observed in PERFDUB concretes from 0 to 1 mm, from 0 to 3 mm in VERCORS concrete, and from 2 to 5 mm in CETU concrete. At 40 °C, 65 % RH, the carbonate thickness observed in PERFDUB concretes varies from 0 to 2 mm. It goes the same way in VERCORS concrete and from 2 to 3 mm in CETU concrete (cf. FIGURE 3).

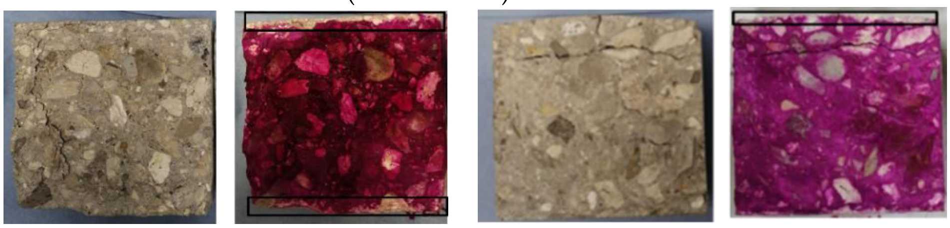


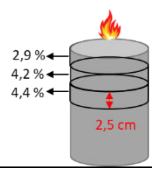
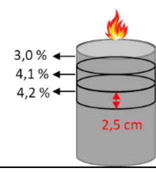
FIGURE 3. Evaluation of carbonation by colored indicator (Phenolphthalein) in specimens of CETU concrete after unidirectional drying for 13 months at 23°C, 50% RH (left), and at 40 °C, 65 % RH (right).

Microcracking is observed with a Keyence optical microscope on Ø 11x10 cm cylinders dried at 40 °C, 65 % RH, and cylinders of the same geometry and size dried at 23 °C, 50 % RH. In both conditioning modes, the drying lasted 13 months and occurred in all directions. Comparing the cracking rate observed for the two drying modes in each concrete, the micro-cracking is almost identical (insignificant) for PERFDUB concretes and more critical in the case of drying at 23 °C for both VERCORS and CETU concretes. In this case, the effect of the deformations due to the thermal gradients passing from 23 °C to 40 °C might be negligible. The impact of the water gradient will be investigated for a better understanding.

C. Spalling sensibility: effect of the concrete conditioning

The effect of the conditioning mode on spalling due to fire has been tested on both PERFDUB mixes. TABLE 3 compares spalling sensibility, water content, and compressive strength between the normalized conditioning and the accelerated drying in the PERFDUB38 mix. Results show good representativeness of the accelerated protocol compared to the normalized conditioning of 3 months. In the case of a drying time of 3 months, only the first centimeters of the specimen have dried, and the core remains almost saturated. Therefore, for a water state representative of a short duration standardized drying (≤ 3 months), the 40 °C, 65 % RH accelerated drying protocol is validated. Furthermore, this accelerated drying protocol is also validated for a representative skin water state regardless of the drying duration. The same findings are observed PERDUB41 mix.

TABLE 3. The impact of the storage conditions on the spalling susceptibility PERFDUB38 mix.

Storage	90 days under 23 °C, 50 % RH	15 days under 40 °C, 65 % RH
f_{cm} [MPa]	91	88
Water content		

Spalling	Max depth [mm]	21	25
	Mean depth [mm]	12	12
	Topograp hy of the spalling		

CONCLUSION AND PERSPECTIVES

In this work, the experimental study allowed us to identify the concrete drying parameters and validate the theoretical predictions. The drying prediction allowed us to evaluate the hydric profile evolution in the reference structure in the short and long term and define the target profile and then the optimal drying conditions to reach it in a few months only. The main conclusions are:

- The modeling of the drying phenomenon at different thermal and hydric solicitations shows that temperature plays an essential role in the acceleration of concrete drying. The relative humidity controls the water content on the surface of the test bodies. This numerical prediction has been validated experimentally on 4 different concretes
- According to the numerical results so far, drying for 15 days at 40°C, 65% RH after preservation in sealed condition for 7 days allows to reach the same water profile as drying 90 days at 23 °C, 50% RH: a drying period 6 times shorter. This result has been validated on high performance concretes.
- The first test of the accelerated drying protocol shows good consistency between theoretical predictions and experimental results of concrete water content. It also indicates that the accelerated drying did not modify the fire behavior.

Perspectives of this study include optimizing the numerical study, optimizing and refining the accelerated drying protocol. Also, we envision validating the accelerated drying protocol in the long term by assuming a target hydric state in a structure with a few years of operation. Applications of this study are broad and can go beyond fire behavior because of the strong dependence on the water profile of, for instance, delayed behavior and pathologies in concrete.

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