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Identifying the mechanisms of concrete drying: an experimental-numerical approach

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Abstract

The loss of non-chemically linked water from concrete has a significant effect on its structural behaviour through a modification of its strength, Young modulus, fracture properties, creep and volume stability. The knowledge of the hydric state of concrete is therefore of utmost importance regarding the mechanical behaviour and durability potential of concrete structures. We use a non-linear diffusion equation for predicting the drying kinetics, including processes of the diffusion of water vapour and the permeation of liquid water. The multiple parameters of this model are then identified based on an in-house optimization algorithm and on a thorough experimental setup including measurements of mass loss and located relative humidity evolutions on various sample geometries. This coupled experimental and numerical approach allows identifying robust and meaningful material parameters of concrete used for drying predictions. This approach allows also identifying the mechanisms in concrete drying phenomena and processes by inverse analysis

Keywords : Concrete drying, relative humidity, transport properties

1. Introduction

Concrete subjected to drying quickly develops strong inner gradients of water content. The loss of non-chemically linked (or free) water from a concrete has a significant effect on its structural behaviour through a modification of its strength [POP86], Young modulus [BAO14], fracture properties and creep [BAZ94]. The water loss also induces drying shrinkage [KIM98] and drying creep [GAM78], which induce deformation of higher magnitude than the autogenous deformation [ZHA11]. This contributes to deflection and cracking (due to drying gradients with shrinkage induced cracking, but also strains incompatibilities between cement paste and aggregates [BIS02]). It also may induce prestress loss. In addition, the concrete diffusivity and

38 permeability are highly affected by its water content [MIL61, MUA76, VAN80, HUA15,
39 ZHA16], thus affecting its durability potential. The knowledge of the hydric state of concrete is
40 therefore of utmost importance regarding the mechanical behaviour and durability potential of
41 concrete structures.

42 The widest used measurement to characterize the hydric state of the concrete is the weight loss in
43 drying conditions. However, this macroscopic measurement does not allow obtaining the
44 distribution of the degree of saturation inside the specimen. Transport properties (porosity, liquid
45 and gas permeability, diffusivity) can also be measured, but they generally suffer from
46 hypothesis and shortcomings (cracking for instance if the material is completely dried to
47 determine intrinsic gas permeability) that prevent these values to be used as is in a complete
48 multi-parameter drying model. Moreover, measurements of water permeability by imposing a
49 gradient of pressure in saturated conditions are very sensitive to the preparation of the samples
50 (pre-dried specimens show greater “intrinsic permeability” due to drying cracks, [HAL00]).
51 Rather, they are very powerful in order to compare different concrete compositions. This is why
52 indirect methods are generally used to determine the moisture (or degree of saturation) gradient
53 present in the material. These methods include electromagnetic methods [SBA07], ultrasonic
54 methods [POP05, SBA12], X-ray tomography [JER15], or relative humidity measurement
55 [GRA14, ZHA15, HUA15]. All these possibilities are based on physical indicators that are
56 dependent on the water content. In order to get the actual saturation degree of the material from
57 these measurements, calibration curves are necessary. This calibration is specific to each method,
58 and the protocol required for its determination is not straightforward. In the case of the
59 measurement of relative humidity, obtaining the degree of saturation relies on the knowledge of
60 desorption isotherms of the material. Even if the measurement of relative humidity (RH) is
61 generally local and does not allow a full mapping of the water content of concrete, the advantage
62 of this method is that concrete desorption isotherm has been widely studied in the last decades
63 [XI94, BAR07, ZHA14, ZHA16]. Therefore, the determination of the saturation degree from
64 local RH measurements is straightforward during desaturation of the material.

65 In any case, the experimental determination of the hydric state of concrete during drying is
66 complex, thus justifying the many modelling approaches for the water transport in unsaturated
67 porous media, and in particular in concrete [KIM98, MUA76, VAN80, HUA15, ZHA15,
68 ZHA14].

69 In this paper, the drying of various concrete samples is cautiously studied through refined
70 measurement of the mass loss (global measurement) and internal relative humidity distribution
71 (local measurement). Various sample sizes and shapes are tested. From the results of these tests,
72 the values of the model input parameters are assessed through the use of an in-house
73 identification tool. The variation of these parameters as well as the relevance of performing
74 combined measurements of the global and local measurements of the hydric state of concrete is
75 discussed.

76 **2. Drying model**

77 In order to be accurate, our model includes predictive transfer equations as well as the non-linear
78 coefficients that control the transfer, completed by realistic applied boundary conditions.

79 **2.1. General expression**

80 In the concrete porosity, two main phases coexist. The solid phase and the liquid phase (liquid
 81 water and vapour water). The amount of each phase depends on parameters such as porosity,
 82 tortuosity, connectivity, ambient conditions and material saturation. These phases are not pure
 83 (presence of chloride ions for instance). Within the material, different exchanges are set up in the
 84 form of mechanisms such as permeation of liquid water as well as diffusion of the water vapour.
 85 The drying model used in this study is based on the one developed by [MAI01]. It can be written
 86 in the form of a non-linear diffusion equation (equation **Erreur ! Source du renvoi**
 87 **introuvable.**) [BAR07b]. The apparent coefficient of water diffusion D_w is a function of the
 88 degree of saturation S , and is considered to combine two main mechanisms, which are the
 89 diffusion of water vapour and the permeation of liquid water. The water diffusion coefficient is
 90 computed according to equation **Erreur ! Source du renvoi introuvable.**, where K_l and D_v are
 91 non-linear coefficient relative to the transport of liquid water and water vapour respectively.
 92 While it is known that permeation is clearly predominant for high saturation and that diffusion is
 93 predominant for low saturation, there is currently no clear consensus as to which is the
 94 predominant mechanism in intermediate saturation degrees, especially when hydraulic gradients
 95 are considered

$$\frac{\partial S}{\partial t} = \text{div}(D_w \overrightarrow{\text{grad}}(S)) \quad (1)$$

$$D_w = K_l + D_v \quad (2)$$

96
 97 All these exchanges are considered as isotherm (latent heat of vaporization of water is not taking
 98 into account). The material is assumed to be mature and dimensionally stable (no interaction
 99 between drying and drying shrinkage). The contribution of dry air to the mass transport is
 100 neglected [ZHA16]. If the concrete porous network is saturated, the permeation mechanism is
 101 the predominant one, whereas for a low degree of saturation, the movement of water in the form
 102 of vapour diffusion cannot be neglected.

103 The liquid water flow can be expressed through Darcy's law, which relates the fluid mass flow
 104 through a medium to the pressure gradient of this fluid, as expressed in equation (3). In which
 105 k_{la} is the concrete apparent permeability to liquid water (m^2), μ_l is the dynamic viscosity of
 106 water ($Pa \cdot s$), ρ_l is the water density ($kg \cdot m^{-3}$), P_l is the liquid water pressure (Pa), and J_l
 107 is the liquid water flux ($kg \cdot m^{-2} \cdot s^{-1}$).

$$\vec{J}_l = -\rho_l \frac{k_{la}}{\mu_l} \overrightarrow{\text{grad}}(P_l) \quad (3)$$

109
 110 The water vapour flow can be expressed with Fick's law, representing the flow of water in form
 111 of vapour due to a gradient in relative humidity in concrete. It can be expressed in the form of
 112 equation (4), where J_v is the water vapour flux ($kg \cdot m^{-2} \cdot s^{-1}$), D_{va} is the apparent diffusion
 113 coefficient of water vapour in air ($m^2 \cdot s^{-1}$), P_v is the partial vapour pressure (Pa), M_w is the
 114 molecular mass of water ($kg \cdot mol^{-1}$), R is the gas constant ($J \cdot K^{-1} \cdot mol^{-1}$) and T is the
 115 temperature (K).

$$\vec{J}_v = -D_{va} \frac{M_w}{RT} \overrightarrow{\text{grad}}(P_v) \quad (4)$$

117

118 As shown in equation (5), the difference between the gas pressure P_g (Pa) and liquid water
 119 pressure is the capillary pressure P_c (Pa). According to Kelvin's equation, the relationship
 120 between the capillary forces and the relative humidity in concrete can be expressed as shown in
 121 equation (6), where h is the relative humidity, since P_g is considered constant and equal to
 122 atmospheric pressure.
 123

$$P_c = P_g - P_l \quad (5)$$

$$P_c = \frac{-\rho_l R T \ln(h)}{M_w} \quad (6)$$

124
 125 **2.2. Non-saturated concrete behaviour**

126 In this model, the main unknown is chosen to be the relative humidity. This is why all further
 127 equations are expressed as function of that parameter. The desorption isotherm links the
 128 saturation degree to the relative humidity. The most common equation used to model this
 129 relationship is the van Genuchten equation [VAN80] (equation **Erreur ! Source du renvoi**
 130 **introuvable.**), where a_{vg} , b_{vg} and c_{vg} are three parameters that can be obtained from experimental
 131 results of the desorption isotherm for the tested material. A commonly used restriction is applied
 132 on the value of c_{vg} , as shown in equation **Erreur ! Source du renvoi introuvable.**

$$S = \left(1 + \left(-a_{vg} \ln(h) \right)^{b_{vg}} \right)^{-c_{vg}} \quad (7)$$

$$c_{vg} = 1 - \frac{1}{b_{vg}} \quad (8)$$

133
 134 From combining equations (3) to (6) with mass balance principles, the equation that has to be
 135 solved for the moisture transport can be expressed as equation **Erreur ! Source du renvoi**
 136 **introuvable.**, where Φ is the concrete water porosity and P_v^{sat} is the saturation vapor pressure
 137 (Pa).
 138

$$\Phi \frac{\partial S}{\partial h} \frac{\partial h}{\partial t} = \frac{1}{\rho_l} \text{div}(\vec{J}_l + \vec{J}_v) = \text{div} \left[\left(-\frac{k_{la} \rho_l R T}{\mu_l M_w h} - \frac{D_{va} M_w P_v^{sat}}{\rho_l R T} \right) \overrightarrow{\text{grad}}(h) \right] \quad (9)$$

139
 140 The dependency of k_{la} to the degree of saturation can be taken into account with equation (10),
 141 where k_l (m²) is the intrinsic water permeability of concrete in saturated conditions, and k_{rl} is the
 142 relative permeability. The relative permeability can be determined through the Mualem
 143 conceptual relationship [MUA76] shown in equation (11), where a_{mu} and b_{mu} are material
 144 parameters. Generally, the parameter a_{mu} is considered equal to parameter c_{vg} . While the used
 145 model considers these parameters equal, it leaves the opportunity to uncouple them during their
 146 identification process.. For cement-based materials, the recommended value for b_{mu} is within the
 147 large range of -4 to 5.5 [MON04, LEE08, POY11, ZHO14].

$$k_{la}(S) = k_{rl} k_l \quad (10)$$

$$k_{rl}(S) = S^{b_{mu}} \left(1 - \left(1 - S^{a_{mu}} \right)^2 \right) \quad (11)$$

$$D_{va}(S) = d_{rl} D_0 \quad (12)$$

$$d_{rl}(S) = \Phi^{a_{mq}} (1 - S)^{b_{mq}} \quad (13)$$

149
 150 In the same way, the diffusion of water vapour is highly dependent on the saturation degree. The
 151 tortuosity of the concrete porosity also affects this parameter. The empirical model shown in
 152 equation (13) and (13) was developed in order to take both parameters into account [MIL61].
 153 The apparent diffusion is related to the relative diffusion function d_{rl} and to the diffusion
 154 coefficient of water vapour in air D_0 . The parameters a_{mq} and b_{mq} can yield variable values
 155 between 1.3 and 2.74 for one and 3.3 and 4.2 for the other, respectively [THI07]. However, since
 156 these parameters are relative to the tortuosity of the material, it is to be expected that their value
 157 is depending on the concrete pore structure.

158 Based on the above equations, the respective contributions of liquid water permeability and
 159 water vapor diffusion to the apparent water diffusion coefficient can be expressed with equations
 160 (14) and (15).

$$K_l(S) = - \frac{k_{la} \rho_l R T}{\mu_l M_w H R \Phi} \frac{dh}{dS} \quad (14)$$

$$D_v(S) = - \frac{D_{va} M_w P_v^{sat}}{\rho_l R T \Phi} \frac{dh}{dS} \quad (15)$$

161
 162 **2.3. Boundary conditions**

163 Various types of boundary conditions are generally considered for modelling concrete drying.
 164 First, it is possible to impose the outdoor relative humidity directly at the air-concrete interface
 165 [HIL14, OXF16]. However, this does not take into account the presence of a so-called boundary
 166 layer of air at the air-concrete interface whose moisture content depends on the distance to the
 167 concrete surface. The thickness of this boundary layer strongly depends on the ambient
 168 conditions (wind speed, temperature) and the material surface condition (surface porosity,
 169 surface roughness, and surface moisture) [ZHA15]. Generally, this boundary condition can be
 170 taken into account in the form of equation (16), where δ is the size of the boundary layer (in m),
 171 or more generally by equation (17) [SAK83, HUA15, ZHA15, OZB16]. The area factor h_{CL}
 172 reaches values varying by several orders of magnitude depending on the study. A study suggests
 173 the value of 1.5×10^{-5} [HUA15]. According to [OZB16, AKI97], its value ranges from 8×10^{-7} to
 174 2×10^{-6} for w/c ranging from 0.43 to 0.55 respectively. According to other authors, it can reach
 175 much higher values in the range of 10^{-3} to 10^{-2} [SAK83, ZHA15].

$$J_{CL} = -D_0 \frac{M_w P_{vs}}{RT} \frac{h_s - h_e}{\delta} \quad (16)$$

$$J_{CL} = -h_{CL} \cdot (h_s - h_e) \quad (17)$$

176
 177 **3. Materials and methods**

178 The tested concrete composition is referred to as B11 concrete. Three sample geometries for
 179 different drying patterns are placed in a storage bin and mass loss and relative humidity are

180 recorded regularly. With the experimental data obtained in this study, a finite element simulation
 181 is performed and an identification procedure is developed on Matlab to find out the optimised
 182 parameters.

183 3.1. Concrete composition

184 The composition of B11 concrete and major properties are shown in Table 1. It has been widely
 185 studied in previous studies [BRI11, HIL14] and its mechanical properties are therefore mostly
 186 known, including its density and water porosity. The mixing procedure is as follows : the dry
 187 materials (aggregates, sand and cement) are mixed for 60 seconds. Then, water and 1/3 of the
 188 superplasticizer are added with further mixing during 150 seconds. Then, the rest of the
 189 superplasticizer is included, followed by 90 additional seconds of mixing. 30L of concrete were
 190 mixed in one batch.

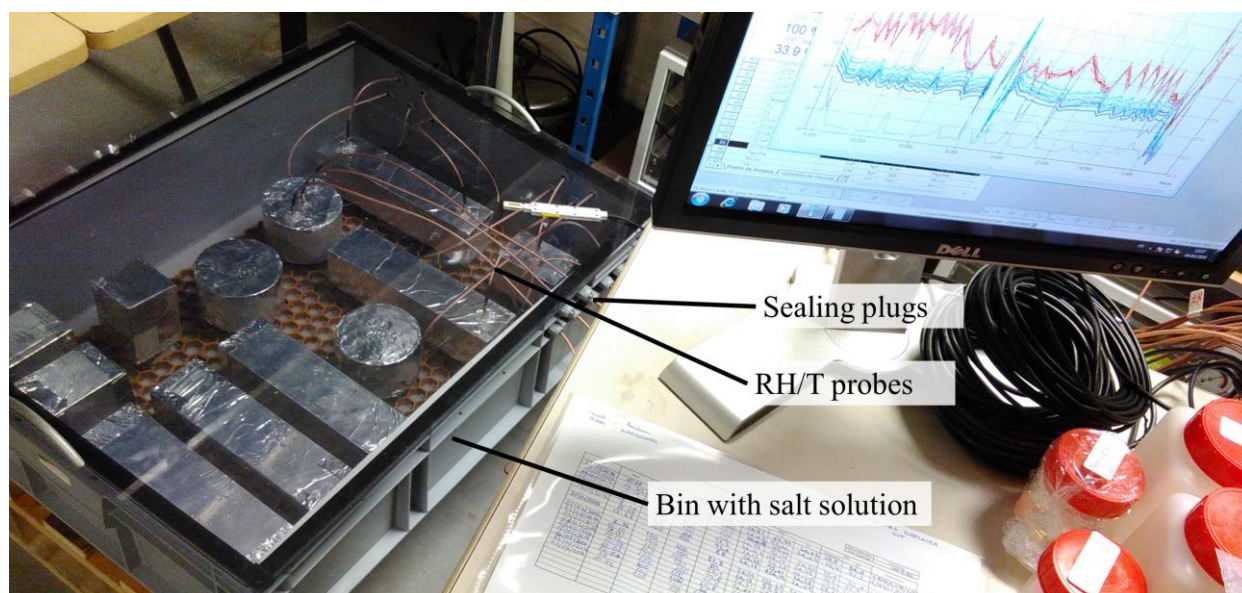
191 Table 1. B11 mix design and main properties [HIL14]

	B11 [kg/m ³]	w/c	[-]	0.573
Cement (CEM II Airvault Calcia)	336	ρ	[kg/m ³]	2324
Sand (0/4)	740	Slump	[cm]	8-11
Aggregates (4/12.5)	303	Porosity	[-]	13.2-13.8
Aggregates (10/20)	752	f_c (28 days)	[MPa]	46.5
Water	193	f_t (28 days)	[MPa]	3.29
Superplasticizer (Plastiment HP)	1.174	E (28 days)	[GPa]	31.34

192

193 3.2. Test setup

194 In order to ensure the same controlled curing conditions to all concrete specimens (temperature T
 195 and relative humidity RH), a large storage bin is developed. The use of a climatic chamber is
 196 rejected due to the difficulty of limiting the forced convection on the faces subjected to drying,
 197 and of ensuring the uniformity of this phenomenon on all sides and on all samples. The storage
 198 bin is impermeable to air and moisture.



199

200 Figure 1. Experimental setup linked to the continuous measurement of RH and T sensors

201 In order to enable placing several temperature and relative humidity sensors inside the storage
202 bin, holes are performed, and sealing plugs are installed. The concrete samples are placed on a
203 plastic honeycomb duckboard allowing maximized interaction between the salt solution and the
204 ambient air. This setup is shown in Figure 1. The saturated salt solution is prepared using
205 potassium carbonate in order to reach an ambient RH of 45%. The room temperature is
206 controlled at $25 \pm 1^\circ\text{C}$ (this has been checked using the probes).

207 The concrete samples are prevented from drying until the test starts, which occurred two months
208 after mixing. Due to the high w/c ratio of the B11 concrete, we consider that the hydration
209 reactions are almost completed and that the auto-desiccation phenomenon can be neglected
210 [BRI11]. The initial state of the material is therefore not fully saturated, but with a very high
211 saturation degree due to the high w/c ratio of the B11 concrete.

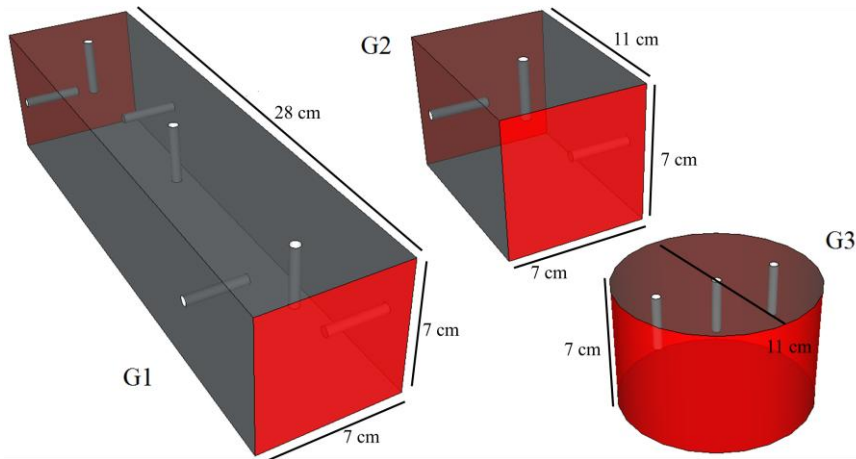
212 The experimental measurements consist in mass loss measurements at regular intervals and of
213 the internal relative humidity in concrete samples at various distances from the drying interface.
214 Three sample geometries are considered (see Figure 2):

- 215 - G1: 70x70x280mm prism, drying on the two opposite lateral faces (5 samples),
- 216 - G2: 70x70x110mm prism, drying on the two opposite lateral faces (3 samples),
- 217 - G3: 70x110mm cylinder, drying in the radial direction (3 samples),

218 For each of these geometries, the average drying radius (r_d , in m) is computed as the ratio
219 between the drying surface and the sample volume. This results in r_d of 0.14, 0.055 and 0.0275
220 and for geometries G1, G2 and G3 respectively.

221 The mass loss measurements are performed on 3 samples for G1 and on 2 samples for G2 and
222 G3. This study focuses on the first few weeks after drying is initiated. The relative humidity is
223 measured at various distances from the drying interface on geometries G1, G2 and G3 according
224 to the distribution shown in Figure 2. The used sensors are capacitive probes Ahlborn FHA 646
225 R. These probes are chosen due to their extremely small dimensions (5 mm diameter) as well as
226 their high accuracy and wide operating RH and T range. The RH sensors are placed inside
227 concrete through holes drilled on hardened concrete. This method is considered more appropriate
228 than reservations in concrete from the casting since the latter results in a layer of mortar around
229 the hole that is not representative of the tested material. In order to avoid damaging the sample, 6
230 mm diameter holes are obtained by drilling with progressively increased diameter from 3 mm to
231 6 mm.

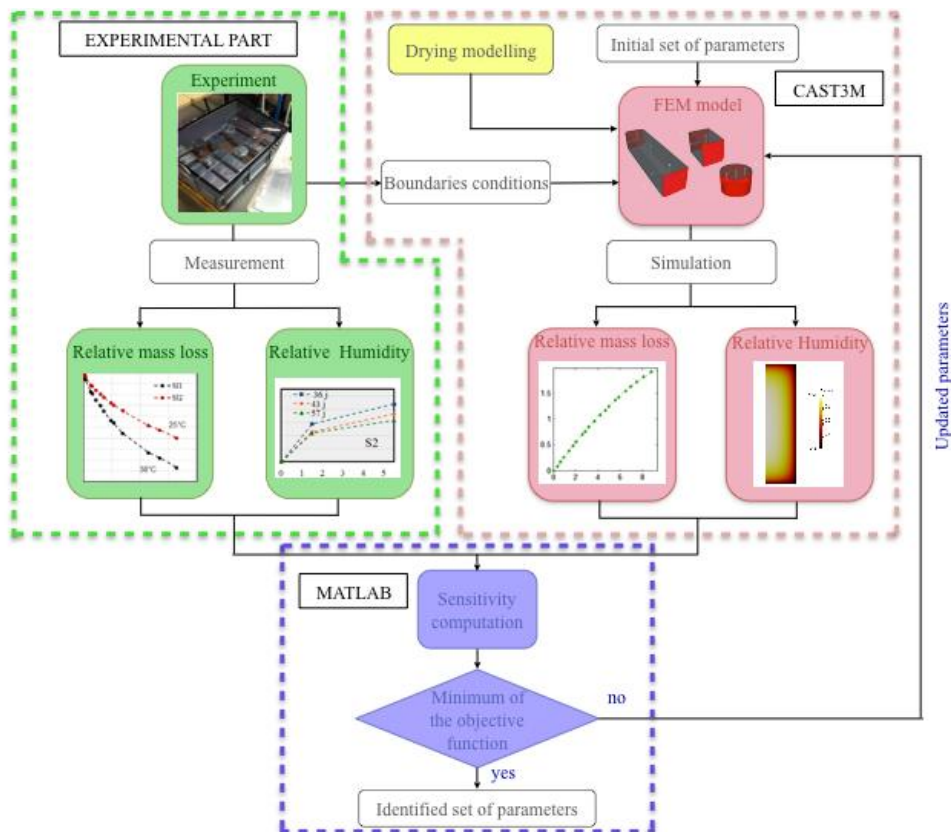
232 All sensors are placed in 30mm deep reservations obtained by perforation of the hardened
233 concrete. Sensors are placed in the centre of each sample. In geometry G1, additional sensors are
234 placed at 15, 30, and 60 mm from each drying interface. In geometries G2 and G3, two
235 additional sensors are placed at 15 mm from the drying interface.



236
237 Figure 2. Layout plan of RH sensors (sides submitted to drying displayed in red : two opposite
238 lateral faces for G1 and G2, drying in the radial direction for G3)

239 **3.3. Identification procedure**

240 In this study, an identification procedure based on Finite Element Modelling Updating (FEMU)
241 [NEG15] was developed using Cast3m [CAS07] and Matlab. This method (FEMU) is based on
242 the minimisation of an error function built with the difference between data gathered from
243 experimental work and numerical results. The identification flow chart is presented in Figure 3.
244 It can be divided into three parts. The first is related to the experimental data obtained in this
245 study. Then, a second part is related to the FE simulation. The last part is associated with the
246 minimization algorithm, developed on Matlab.



247
248 Figure 3. Identification algorithm flow chart

249 This inverse modelling relies on a priori knowledge of the drying model and its ability to
 250 accurately predict the drying process. In this case, the used numerical model was presented in
 251 section 2. Thus, the experimental process is simulated with relative humidity and temperature as
 252 boundary conditions of the modelling. It is then possible to build an error function, shown in Eq.
 253 (18). This error function \vec{f} includes experimental data (indicated by the e exponent), simulated
 254 data (indicated by the s exponent), as well as results from mass loss measurements (indicated by
 255 the ML index) and from relative humidity measurements (indicated by the RH index). This
 256 function ensures that:

- 257 - More relative importance is assigned to higher mass loss or lower relative humidity,
- 258 - The same relative importance is assigned to mass loss resulting from one sample and to
 259 relative humidity measurements resulting from one sensor,
- 260 - Several samples or sensors for the same measurement can easily be included
 261 independently from one another in the identification process.

262

$$\vec{f} = \sum_{\text{samples}} \left[\frac{\vec{C}_{ML}^e}{\max(\vec{C}_{ML}^e)} - \frac{\vec{C}_{ML}^s(\vec{p})}{\max(\vec{C}_{ML}^s)} \right] + \sum_{\text{sensors}} \left[\frac{\vec{C}_{HR}^e}{\max(100 - \vec{C}_{HR}^e)} - \frac{\vec{C}_{HR}^s(\vec{p})}{\max(100 - \vec{C}_{HR}^s)} \right] \quad (18)$$

263 where C^e and C^s are the vectors of experimental and modelling results for respectively the
 264 relative mass variation (ML) and the relative humidity values (RH). \vec{p} is a vector composed of
 265 model's input parameters. It is possible to add weighting factors in this error function.

266 Finally, the minimisation is calculated, based on Levenberg-Marquart's optimisation method
 267 implemented in Matlab. The framework of the algorithm is presented in Figure 4.

Algorithm 1: Levenberg-Marquardt resolution

```

1 Input : Experimental points  $C_i^{exp}$ ,  $\delta_{max}$ ;
2 Arbitrary initialization  $\mathbf{p}_0$ 
3 begin
4    $k \leftarrow 0$ ;
5    $\theta \leftarrow 10^{-3}$ ;
6    $\mathbf{J} \leftarrow \frac{dC}{d\mathbf{p}_0}$ ;
7    $\mathbf{p} \leftarrow \mathbf{p}_0$ ;
8   while STOP-CRIT and  $(k < k_{max})$  do
9      $k \leftarrow k+1$ ;
10    if  $0 \equiv \sqrt{k} \pmod{2}$  then
11      Actualisation :  $\mathbf{J} \leftarrow \frac{dC}{d\mathbf{p}_k}$ ;
12      Get  $C_i^{sim}(\mathbf{p})$  from simulation;
13       $\mathbf{f} \leftarrow C_i^{exp} - C_i^{sim}(\mathbf{p})$ ;
14       $\lambda \leftarrow \theta \|\mathbf{f}\| + (1 - \theta) \|\mathbf{J}^T \mathbf{f}\|$ 
15      Find  $\delta$  such that  $(\mathbf{J}^T \mathbf{J} - \lambda \mathbf{1}) \delta = \mathbf{J}^T \mathbf{f}$ ;
16      if  $abs(\delta) \leq \delta_{max}$  then
17         $\mathbf{p} \leftarrow \mathbf{p} + \delta$ 
18      else
19         $\mathbf{p} \leftarrow \mathbf{p} \pm \delta_{max}$ 

```

268

269

Figure 4. Minimisation algorithm

270 The minimized parameters resulting from this process are entered in CAST3M as input
271 parameters. The identification runs until it reaches a criterion based on the average relative
272 increment of the identified parameters increments. If this average variation remains below the
273 limit of 0.1% of the current parameters' values, the identification procedure is stopped.

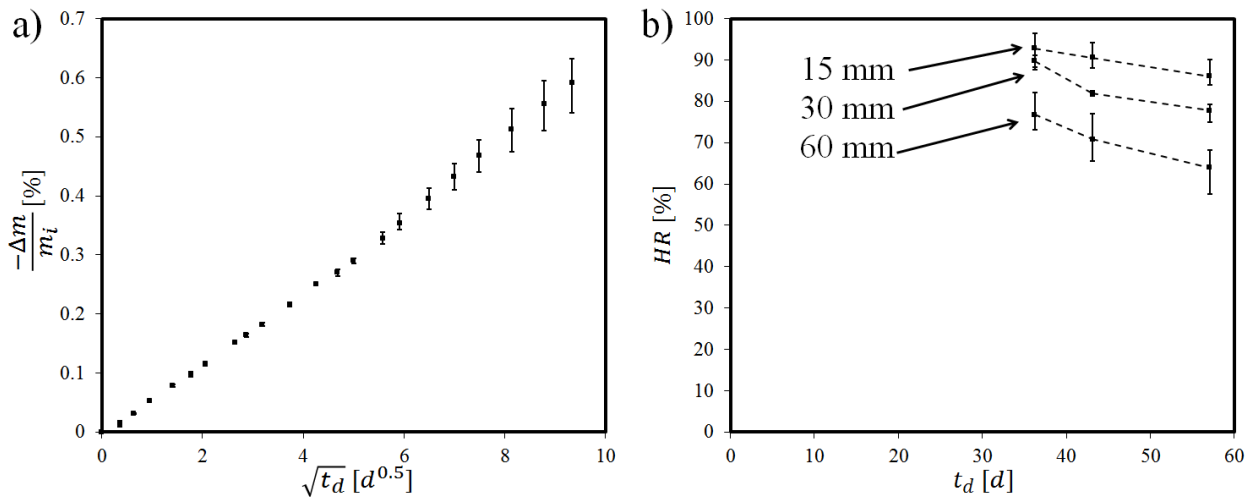
274 Lastly, as our experimental work involves different shapes and dimensions, it is possible to
275 diversify the identification process. Indeed, the process allows for identification of one or more
276 shapes, and allows taking into account relative mass loss and relative humidity at the same time
277 or separately.

278 4. Results

279 In this section, the measurement results of mass loss and relative humidity, as well as
280 identification of model parameters is analyzed.

281 4.1. Mass loss and relative humidity measurements

282 The mass loss (ML) due to water drying and the relative humidity (RH) evolution resulting from
283 water transport are presented hereafter. First, it was verified that the RH sensors provide
284 reproducible results, and that their presence does not affect the overall ML of drying samples. In
285 order to do so, three G1 samples without drilled holes were tested in parallel with two G1
286 samples with drilled holes and RH sensors.



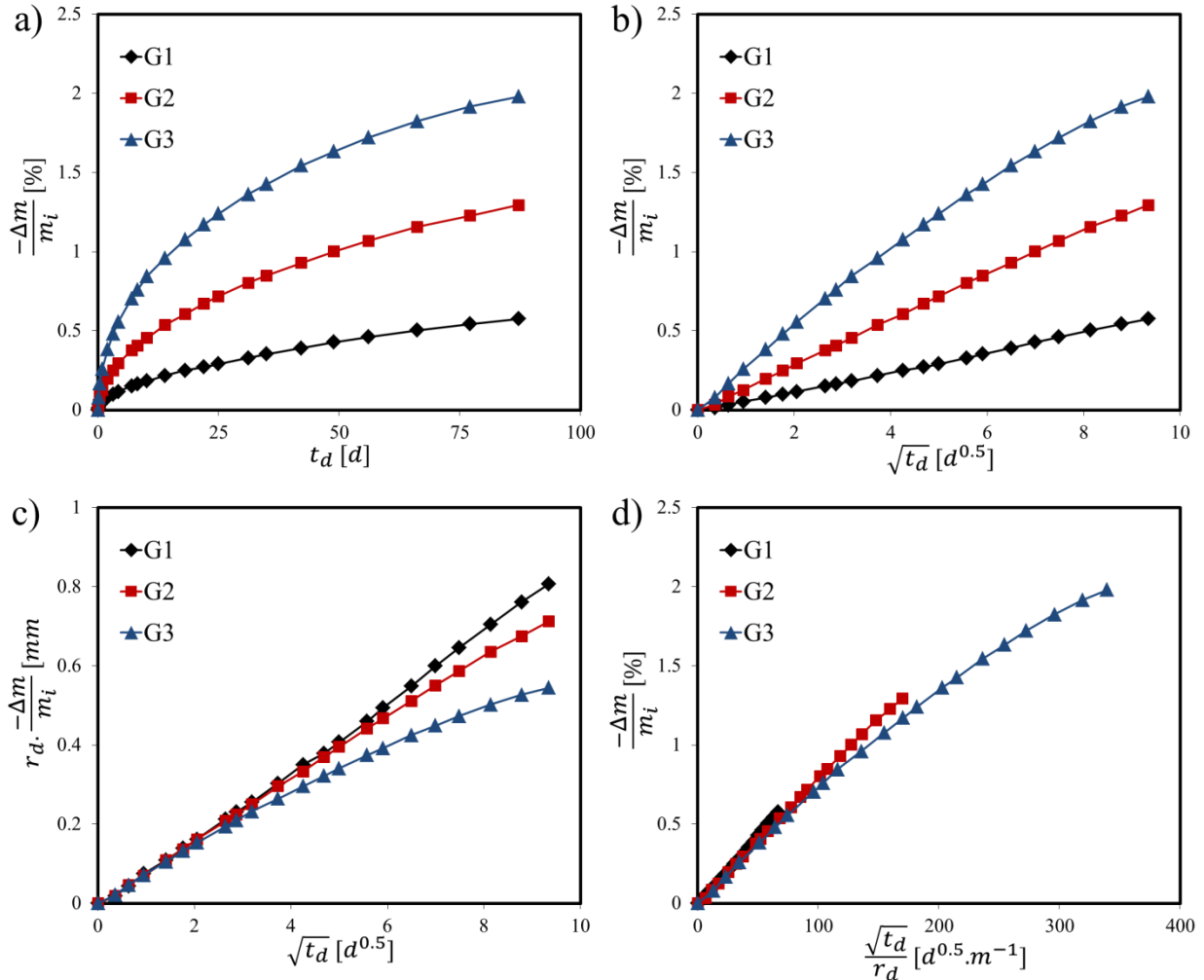
287

288 Figure 5. (a) Maximum, minimum and average values of relative mass loss for 5 G1 samples,
289 with and without RH sensors (b) Maximum, minimum and average value of the RH
290 measurement at three different distances from the drying interface from 3 G1 samples

291 The results shown in Figure 5(a) indicate that the variability on the ML measurement is low, and
292 that the presence of the sensors do not affect the ML measurements. Mass loss being defined as a
293 decrease of the mass through time, it is displayed as a positive value when drying occurs. On the
294 other hand, the RH measurements are more variable, as displayed in Figure 5(b). This is most
295 likely due to the heterogenous aspect of the concrete, for which the drilled holes (6 mm diameter,
296 30 mm depth) might be in contact with a variable amount of aggregate/mortar ratio. In addition,
297 the presence of a large pore filled with water close to the drilled hole might locally affect the RH.
298 Finally, local temperature gradients or discrepancies in the paricule size distribution can also

299 contribute to these variations. However, the general trend shown by all sensors is consistent, and
 300 the reproducibility of the measurement is satisfactory for the purpose of this study.

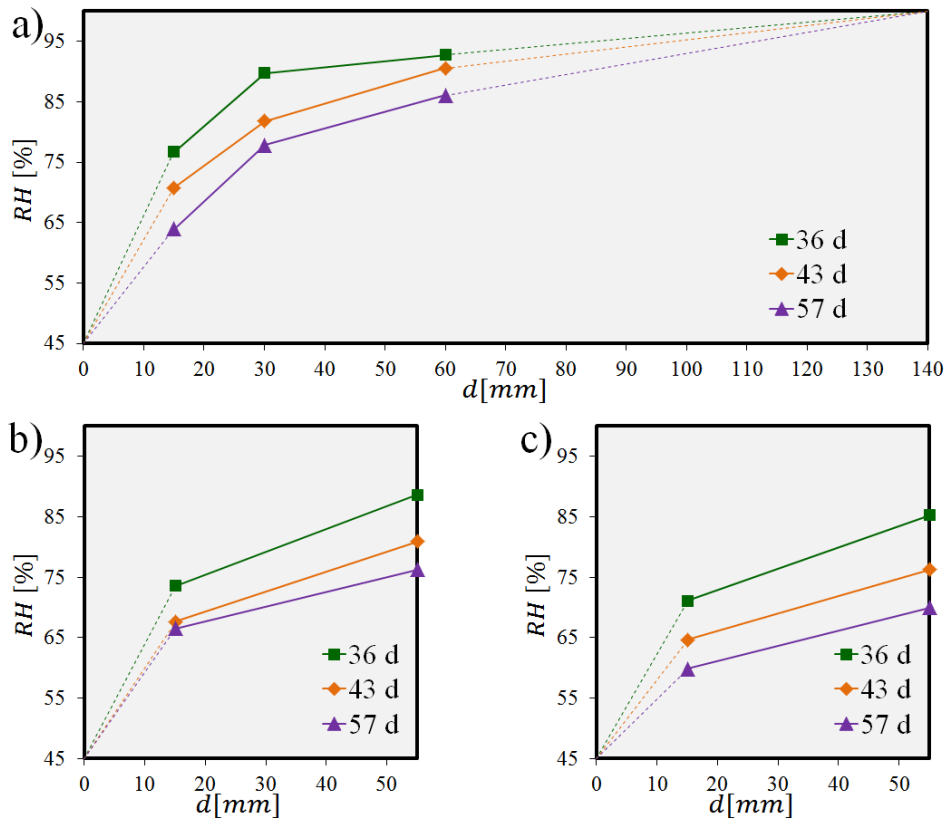
301 The mass loss observed in Figure 6(a) shows a very high dependency on the geometry. As the
 302 drying radius increases (meaning that the average distance between the free water and the drying
 303 interface increases), ML increases in a more slow and progressive way. Figure 6(b) represents
 304 ML as a function of the square root of time. In the first days after samples are submitted to
 305 drying, ML increases as a linear function of $t_d^{0.5}$, which is representative of processes that can be
 306 described by a diffusion-like differential equation.



307
 308 Figure 6. Mass loss of G1, G2 and G3 geometries expressed by (a) ML as a function of time (b)
 309 ML as a function of square root of time (c) ML multiplied by drying radius as a function of
 310 square root of time and (d) ML as a function of square root of time divided by drying radius

311 As drying occurs, the concrete saturation degree decreases. This leads to a variation of the
 312 transport properties of the material, and thus to the non-linear behaviour observed after a few
 313 weeks in G2, and after a few days in G3. This process is actually more visible in Figure 6(c),
 314 where ML is multiplied by the sample drying radius. This confirms that the initial slope of the
 315 $ML-t_d^{0.5}$ curve is independent on the geometry, and only depends on the material transport
 316 properties. This slope is therefore affected both by the concrete mix design and by its initial
 317 saturation degree. Then, as the drying radius decreases, the drying occurs faster, thus leading to a

318 decrease of the transport properties, and therefore to a decrease of the rate of ML. This
 319 observation can also be observed in Figure 6(d), in which ML is represented as a function of the
 320 square root of time divided by the drying radius. This figure actually shows that for a given mass
 321 loss, the rate of drying depends mostly on the time and drying radius. However, the G1, G2 and
 322 G3 curves are not superimposed, suggesting that the gradient of saturation degree inside the
 323 material also affects the drying behaviour. This observation highlights once again the importance
 324 of being able to measure and predict the water distribution inside the material in addition to
 325 global information such as mass loss measurements.



326
 327 Figure 7. RH measurement inside the material at 36, 43 and 57 days as a function of the distance
 328 from the drying interface for geometry (a) G1 (b) G2 and (c) G3

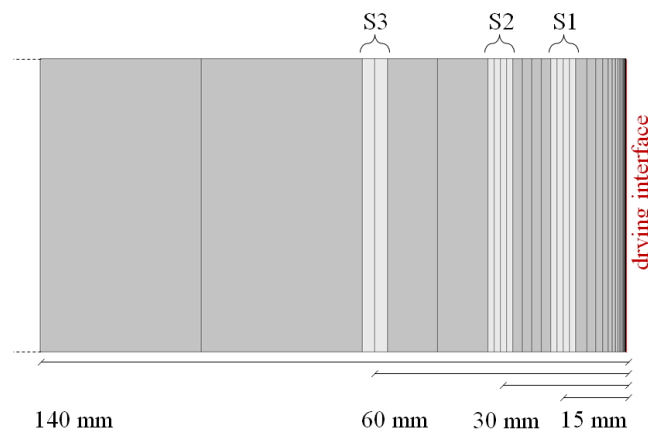
329 Local RH measurements are performed in geometries G1, G2 and G3 at various distances from
 330 the interface. The results of these measurements are shown in Figure 7(a), (b) and (c)
 331 respectively. Only measurements when all sensors provide RH values under 95% are presented.
 332 This is due to the risk of condensation on the sensors for higher RH values, and to the limited
 333 accuracy of the sensors in such high RH environment. However, based on these measurements, it
 334 is considered that the RH in the centre of G1 is saturated through the whole experiment (3
 335 months). This is represented by the dashed lines in Figure 7(a) between a distance of 60mm and
 336 140 mm. In the same way, for representation purposes, it is considered in this section that the RH
 337 at the surface of the samples is equal to the RH of the ambient air, which is fixed at 45% through
 338 the whole test.

339 The results of Figure 7 show as expected that the RH profile decreases in proximity with the
 340 drying interface, due to the initially high humidity gradient between the concrete and the ambient
 341 air. Similarly, the RH profile decreases as the drying time increases. This phenomenon occurs in

342 every point of all geometries. Finally, as expected, at a given distance from the drying interface,
 343 at a given drying time, the local RH value is lower as the drying radius decreases. This is
 344 explained by the lower water supply of the core of the sample as the drying radius decreases,
 345 thus resulting in a lower RH for a similar amount of evaporated water. For instance, at 36 days,
 346 G2 shows an overall lower RH at 15 mm from the drying interface than G1. In the same way, G3
 347 has a lower RH than G2 at 15 mm from the drying interface. The core RH of G3 is lower than
 348 the core RH of G2 even though both sensors are situated at the same distance to the surface due
 349 to the fact that G2 is a prismatic sample drying from the two opposite surfaces, whereas G3 is a
 350 cylinder drying from its whole circumference. This confirms the important effect of the drying
 351 radius.

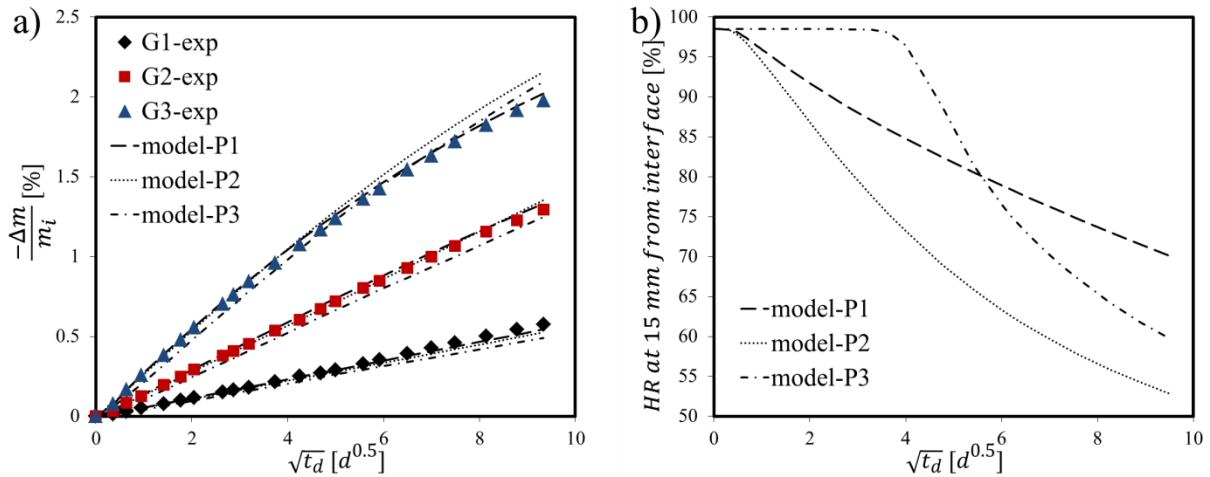
352 4.2. Identification of model parameters

353 The model described in section 2 is implemented in the CAST3M finite element software. The
 354 geometries are meshed with discretisation along the x-axis. More elements are placed in direct
 355 proximity with the drying interface due to the higher humidity gradient in that area. The meshing
 356 of G1 is given as an example in Figure 8, where only half of the prism is meshed since the test is
 357 symmetrical. The RH value is computed as the average value of the elements related to the
 358 position of the corresponding sensor S1, S3 or S3.



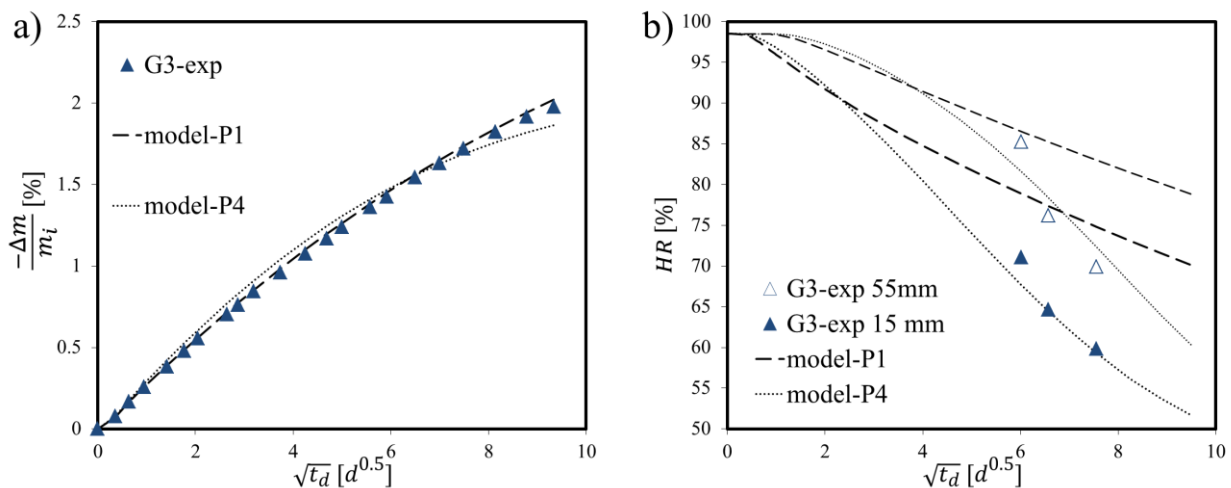
359
 360 Figure 8. Meshing of G1 in the CAST3M software

361 In a first stage, inputs parameters for the model are extracted from the recommendations of the
 362 thesis A. Hilaire [HIL14], who worked on the same concrete. In addition, an exchange
 363 coefficient h_{CL} is chosen as equal to 10^{-5} . This value is extracted from previous research based on
 364 the coupled measurement and modelling of the surface RH of concrete during drying [CAR16].
 365 Finally, the intrinsic permeability of concrete is manually modified in order to ensure a
 366 superimposition of the model mass loss in comparison with experimental values. A value of k_f of
 367 $1.13 \times 10^{-21} \text{ m}^2$ is thus determined. This set of parameter (P1) is shown in Table 2, and results in
 368 the model-P1 curves in Figure 9(a) and (b).



369
 370 Figure 9. (a) experimental ML values and the corresponding simulation values for three sets of
 371 parameters P1, P2 and P3 (b) RH evolution at 15 mm from the drying interface in G3 resulting
 372 from sets of parameters P1, P2 and P3

373 Due to the large number of parameters in the drying model, several solutions can be found, all of
 374 them satisfying the mass loss curves of G1, G2 and G3. This is the case of sets of parameters P1,
 375 P2 and P3 (Table 2), all of which provide good superimposition of the experimental curves.
 376 However, the mass loss being a global measurement, it is shown in Figure 9(b) that these three
 377 sets of parameters do not provide the same hydraulic profiles inside the material. From a strictly
 378 modelling perspective, this is understandable, since a given saturation degree history (equivalent
 379 to the local distribution of the mass loss in the material) can result in variable relative humidity
 380 profile only by modifying the desorption isotherm values a_{vg} , b_{vg} or c_{vg} . This observation justifies
 381 the need to include additional experimental measurements in the parameter identification
 382 process. Indeed, based on the coupled measurement of the mass loss and RH at 15mm and 55mm
 383 from the drying interface, a new set of parameters can be determined (P4). This set of parameter
 384 provides an overall better representation of both measurements (Figure 10(a) and (b)).



385
 386 Figure 10. Experimental values and corresponding simulation of G3 for two sets of parameters
 387 P1 and P4 (a) mass loss and (b) relative humidity at 15 and 55mm from the drying interface

388 The developed identification algorithm is used in order to evaluate the effect of various
 389 experimental or simulation parameters on the identified parameters. The evaluated parameters

390 are the specimen geometry, the input parameters of the identification, the chosen model for the
 391 relative permeability function, and the temperature. This evaluation is made through comparison
 392 of the identified apparent coefficient of water diffusion D_w . For each case, this property is
 393 described as a function of the relative humidity, as well as its two contributing factors K_l and D_v .

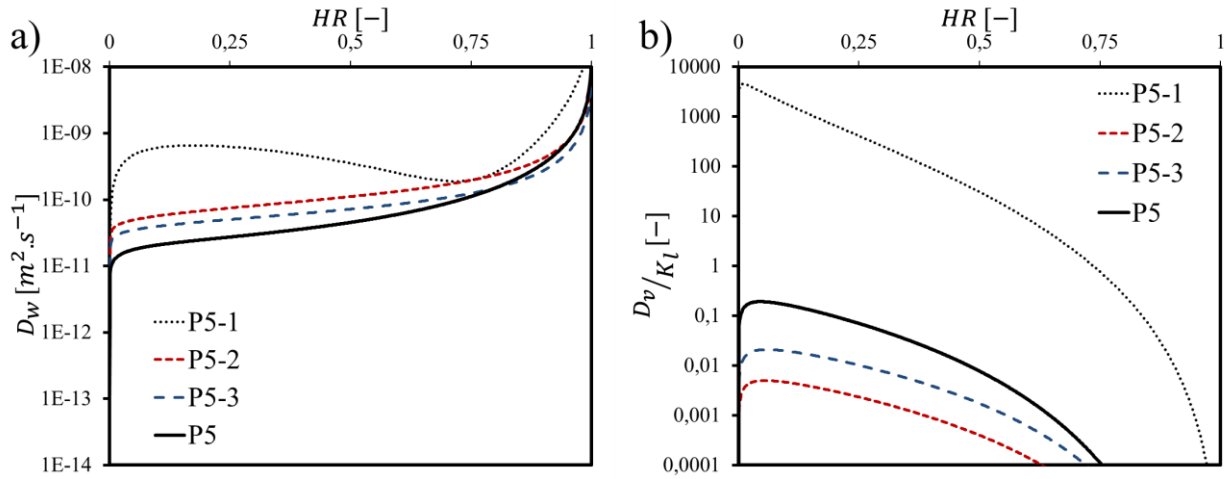
394 4.2.1. Effect of specimen geometry

395 From a macroscopic phenomenological perspective, it is observed experimentally that the drying
 396 process is independent of the specimen geometry. For a given concrete, as the average drying
 397 radius decreases, the global mass loss decreases faster. The difference between G1, G2 and G3
 398 observed in Figure 6 mostly come from the fact that a faster drying induces change in the
 399 transport properties due to the faster decrease of the water saturation. However, the geometry
 400 does not affect the model parameters themselves. Therefore, the developed algorithm should
 401 yield the same identified parameters, whatever the considered geometry. In order to verify this,
 402 identification is performed successively on the experimental results of mass loss and relative
 403 humidity of G1 (P5-1), G2 (P5-2), G3 (P5-3) as well as on all results simultaneously (P5). Since
 404 the latter considers all experimental results, P5 is most likely more representative of the concrete
 405 drying behaviour. The input set of parameters for these identifications is P1. Tables 3 lists the
 406 different parameters identification strategies.

407 Table 2. Drying model parameters

	P1	P2	P3	P4	P5-1	P5-2	P5-3	P5
k_l	1.13E-21	1.25E-22	9.01E-22	1.54E-20	1.00E-19	9.57E-21	3.81E-21	4.68E-22
a_{vg}	4.00	1.96	100.00	10.00	6.76	6.51	5.87	2.42
b_{vg}	1.67	2.04	1.22	1.21	1.14	1.19	1.28	1.65
c_{vg}	$= 1-1/b_{vg}$	$= 1-1/b_{vg}$	$= 1-1/b_{vg}$	$= 1-1/b_{vg}$	$= 1-1/b_{vg}$	$= 1-1/b_{vg}$	$= 1-1/b_{vg}$	$= 1-1/b_{vg}$
a_{mu}	$= c_{vg}$	$= c_{vg}$	$= c_{vg}$	$= c_{vg}$	$= c_{vg}$	$= c_{vg}$	$= c_{vg}$	$= c_{vg}$
b_{mu}	0.50	-3.00	-3.00	-2.84	14.31	-3.57	-2.94	-1.57
a_{mq}	2.72	2.72	1.80	2.72	0.14	3.00	3.00	3.00
b_{mq}	4.20	2.72	1.70	4.20	2.21	5.00	5.00	3.72
h_{CL}	1.0E-05	3.3E-05	9.0E-06	1.0E-05	2.8E-06	5.4E-06	5.3E-06	1.4E-05

408
 409 It is observed in Figure 11(a) that a lower drying radius results in a better identification of the
 410 drying model parameters. This is expected, since the same experiment duration was applied to all
 411 geometries. As a result, low relative humidity has already been reached in G3 at a time when G1
 412 has higher water saturation. P5-1 exhibits a significant difference with the other parameter sets.
 413 We explain this by the fact that G1 exhibits the least advanced drying state, which makes it less
 414 reliable for determining the model parameters. For the test duration of this study (3 months), G3
 415 is obviously more representative of the overall drying process than G1, due to its more advanced
 416 drying state.



417
418

419 Figure 11. identification results considering only G1, G2, G3 or all of them (a) apparent
420 coefficient of water transport as a function of relative humidity (b) diffusion to permeation ratio
421 as a function of relative humidity

422 This observation is confirmed in Figure 11(b), describing the identified D_v/K_l ratio as a function
423 of relative humidity. A D_v/K_l ratio equal to 1 indicates that water vapour diffusion and liquid
424 water permeation are present in equal proportions. In almost all cases except P5-1, the liquid
425 water transport mechanism is predominant, even for low relative humidity. As mentioned
426 previously, the reason why set of parameter P5-1 deviates from this trend is due to its lower
427 drying state.

428 Table 3. Different parameter identification strategies

P1	P2	P3	P4	P5	P6	P7
parameter set based from A. Hilaire [HIL14], complying with ML evolutions	manually modified parameters that comply with the ML evolution	manually modified parameters that comply both with ML and RH evolutions from G3 geometry		algorithm-identified parameters complying with ML and RH for all tested geometries	input : variable input parameters corresponding to different transport mechanisms / output : result of the identification	parameters resulting from various relative permeability, relative diffusivity and desorption isotherm models

429

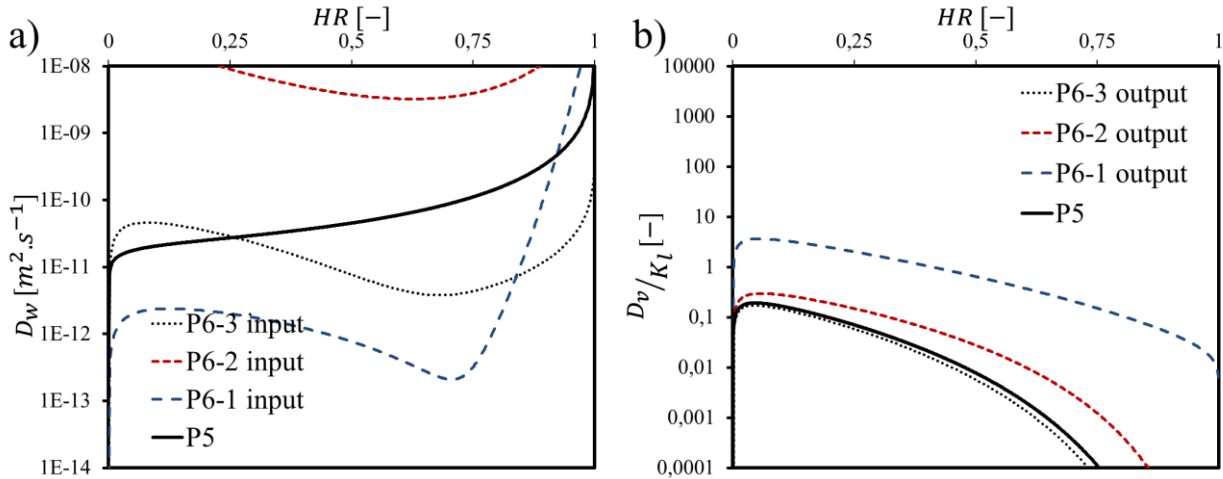
430 Interestingly, the identified set of parameters P5-2 and P5-3 show a similar D_w -RH curve,
431 indicating similar global drying physics. However, the parameters at the origin of this behaviour
432 are quite scattered. For instance, there is a one order of magnitude difference between the
433 intrinsic water permeability k_l of P5 and P5-3 (Table 2). This is fundamental, as it still shows
434 that although the physics are quite correctly captured by our identification tool, there are still
435 several possible parameter sets that can yield this physical behaviour. This is why we include all
436 3 geometries in our reference parameter set P5.

437 Finally, it should be mentioned that the identified parameters P5 are within the range of values
438 that can be found in the literature. In particular, the h_{CL} coefficient of 1.4×10^{-5} is close to the
439 value obtained from experiments [CAR16]; the a_{mq} and b_{mq} are consistent with what is expected
440 from cement-based materias [ZHA16]; b_{mu} is within the range suggested by [HIL14]. And more
441 importantly, the desorption isotherm represented by parameters a_{vg} , b_{vg} and c_{vg} is close to the

442 experimental results for this concrete obtained by Belin *et al.* [BEL09]. This indicates that the
 443 identified solution is satisfactory both from the phenomenological and theoretical perspective. In
 444 order to fully validate the reliability of this approach, the desorption isotherm as well as relative
 445 hydraulic conductivity curves could be compared with experimental data. In order to be fully
 446 relevant, this further step should be addressed with various concrete compositions in a thorough
 447 study, and will be the object of further work.

448 4.2.2. Effect of initial parameters

449 In order to verify the robustness of the identification method, the effect of the input parameters
 450 on the identification output is studied. Three different cases are confronted to the reference
 451 output parameters set P5. Each one of these cases (P6-1, P6-2 and P6-3) is chosen in order to
 452 represent different water transport mechanisms. P6-1 corresponds to a large decrease of D_w , P6-2
 453 corresponds to an increase of D_w , and P6-3 consists in lower D_w for high RH combined with
 454 higher D_w for low RH . The hydraulic conductivity resulting from these three sets of parameters
 455 are represented in Figure 12(a), showing the wide variety of transport mechanisms considered.



456
457

458 Figure 12. (a) hydraulic conductivity functions corresponding to three input sets of parameters
 459 and a reference set of parameter P5 (b) identified diffusion to permeation ratio as a function of
 460 relative humidity

461 The parameters corresponding to Figure 12 are displayed in Table 4. The output values are all
 462 very close to the reference case P5. The identified values of k_t , a_{vg} , b_{vg} and h_{CL} can be considered
 463 as independent from the input parameters. The main difference between the three identification
 464 scenarios is situated in the water vapour diffusion coefficient D_v . This explains the wide
 465 variations in Figure 12 (b), while the identified D_w is almost identical from P6-1, P6-2 and P6-3.
 466 The variability of D_v comes from the fact that its contribution is very low in the RH range
 467 corresponding to the measurements (over 45%). Therefore, the sensitivity of parameters a_{mq} and
 468 b_{mq} is very low and is therefore not reproducible in comparison with the Van Genuchten equation
 469 parameters.

470 Table 4. Selected input and resulting identified (output) drying model parameters

P5	P6-1 input	P6-1 output	P6-2 input	P6-2 output	P6-3 input	P6-3 output
----	------------	-------------	------------	-------------	------------	-------------

k_l	4.68E-22	1.13E-19	4.77E-22	6.00E-21	4.69E-22	2.00E-22	4.62E-22
a_{vg}	2.42	10.00	2.41	2.00	2.42	5.00	2.41
b_{vg}	1.65	1.67	1.65	2.00	1.65	1.43	1.65
c_{vg}	$= 1-1/bvg$	$= 1-1/bvg$	$= 1-1/bvg$	$= 1-1/bvg$	$= 1-1/bvg$	$= 1-1/bvg$	$= 1-1/bvg$
a_{mu}	$= cvg$	$= cvg$	$= cvg$	$= cvg$	$= cvg$	$= cvg$	$= cvg$
b_{mu}	-1.57	10.00	-0.54	-4.00	-1.56	0.00	-1.60
a_{mq}	3.00	4.00	2.75	3.00	3.00	0.40	3.00
b_{mq}	3.72	4.20	0.16	3.00	2.42	5.00	4.05
h_{CL}	1.4E-05	1.0E-05	1.8E-05	3.3E-05	1.4E-05	1.0E-05	1.5E-05

471

472 From these results, it can be considered that according to the used drying model, and considering
473 the mass loss and relative humidity measurements, the best fitting parameters (P5) are mostly
474 independent on the input parameters. This unicity of solution confirms the relevance of the
475 applied methodology.

476 4.2.3. *Effect of relative permeability, relative diffusivity and desorption isotherm model*

477 In order to study the relevance of the developed identification methodology, a last step consists
478 in comparing different models for the relative permeability, relative diffusivity, and desorption
479 isotherm.

480 Regarding the relative permeability model, four different approaches are considered. The first
481 option consists in applying the default model, as described previously in equation (11). This
482 model (relative permeability model 1) consists in 2 parameters to identify. The second option
483 consists in using the same equation, but with one parameter less to identify, considering the
484 approximation that a_{mu} is considered equal to parameter c_{vg} (relative permeability model 2). A
485 third model is used, based on the work of Chung and Consolazio [CHU05], wherein a single
486 parameter (a_{ch}) connects the relative permeability to the degree of saturation equation (19-20)
487 (relative permeability model 3). Finally, a fourth possibility, also suggested by Chung and
488 Consolazio, is particularly interesting because it involves no additional parameter to the model. It
489 consists in fixing the value of a_{ch} as a function of the porosity of the material according to
490 equation (21) (relative permeability model 4).

491 Three relative diffusivity models are considered. The first one is the default model (relative
492 diffusivity model 1). The second one is based on the same principle (equation (13)), except that
493 only one parameter is used instead of two, with the assumption that $b_{mq} = a_{mq} + 2$, as
494 recommended by the authors of that model (relative diffusivity model 2) [MIL61]. Finally, the
495 third model, shown in equation (19), is used whenever combined with relative permeability
496 models 3 or 4, as recommended by Chung and Consolazio (relative diffusivity model 3). This
497 last model has the advantage of not adding parameters to the identification.

$$d_{rl}(S) = 10^{S \cdot a_{ch}} - S \cdot 10^{a_{ch}} \quad (199)$$

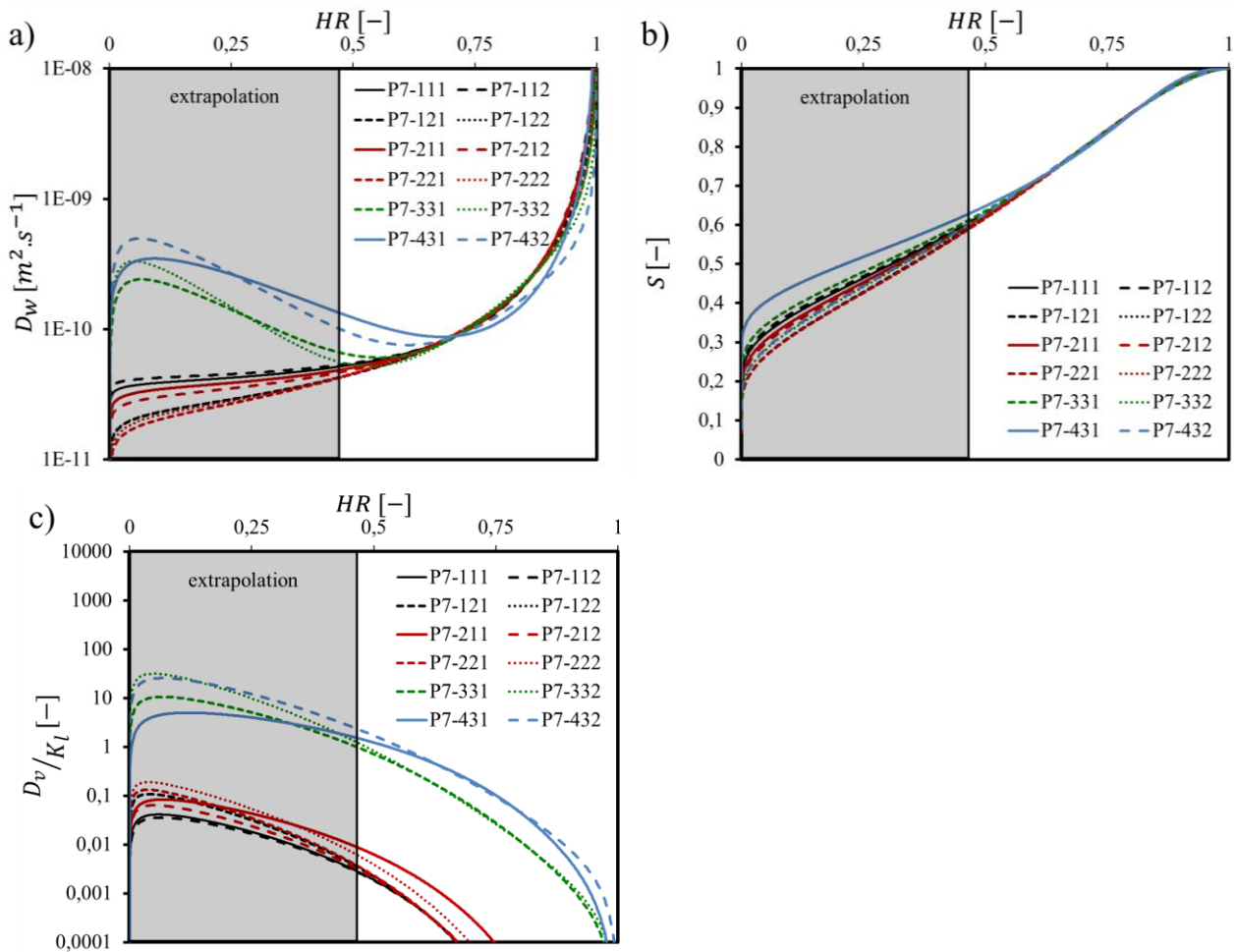
$$k_{rl}(S) = d_{rl}(1 - S) \quad (20)$$

$$a_{ch} = 0.05 - 22.5\Phi \quad (21)$$

498 Ultimately, the desorption isotherm model is either used by default (desorption isotherm model
499 1) or by applying the assumption specified in equation (8). This simplification decreases the
500 amount of parameters to identify (desorption isotherm 2).

501 The result of this multi-model identification is presented in Figure 13. Due to the large amount of
 502 combination of models, only the resulting hydraulic conductivity and saturation degree functions
 503 are presented, without the corresponding identified parameters. The nomenclature is as follows:
 504 $P7 - ijk$ corresponds to the relative permeability model i , combined with relative diffusivity
 505 model j , and desorption isotherm model k .

506 As previously specified, the measured values of relative humidity do not go under 45% RH,
 507 which corresponds to the ambient relative humidity. Therefore, the identification of parameters
 508 is only relevant for RH values between 45% and 100%. This is why in Figure 13, lower RH
 509 values are shaded in gray, indicating that these values are actually not validated by our
 510 experiments.



511

512

513 Figure 13. (a) hydraulic conductivity functions corresponding to the identified parameters from
 514 various model combinations (b) saturation degree prediction for various model combinations (c)
 515 identified diffusion to permeation ratio as a function of relative humidity

516 Figure 13 shows that whatever the model used among the various ones presented, the identified
 517 parameters correspond to a unique $D_w(RH)$ and $S(HR)$ relationship. At least, this is verified
 518 within the RH range that was measured experimentally. First, this indicates that the various
 519 models are versatile enough to represent the same physical reality. However, the relative
 520 contributions of vapour and liquid water transport to the hydraulic conductivity shown in Fig 13
 521 (c) are largely affected by the relative permeability model. Indeed, models 3 and 4 (Chung and

522 Consolazio [CHU05]) induce a large increase in the contribution of the vapour diffusion to the
523 hydraulic conductivity. On the other hand, the relative permeability and relative diffusivity
524 models do not affect these respective contributions. This observation is of major importance as it
525 shows that our current approach still requires more experimental data in order to be fully robust,
526 especially in terms of the specific underlying mechanisms of drying in concrete.

527 There is therefore no preferential model among the ones who were tested, nor an optimal
528 combination of these models. Without further validation, no model can be preferred on the basis
529 of its predictive ability. Therefore, it seems that the model containing the least amount of
530 parameters to identify could be preferred. Secondly, this further supports that the identification
531 strategy that was developed in this study is robust, and yields satisfactory solutions
532 corresponding to a global minimum of the weighed error function. The developed methodology
533 therefore allows identifying robust and meaningful material parameters of concrete used for
534 drying predictions.

535

536 **5. Conclusion**

537 An identification procedure for material parameters of a concrete drying model is proposed. It
538 consists in performing a simultaneous measurement of mass loss due to the drying process as
539 well as localised successive measurements of the relative humidity. This experimental setup is
540 coupled with a developed identification tool able to handle several identification parameters with
541 a specific weighed error function, combining both type of experiments. The numerical model is
542 based on a non-linear diffusion-type model including two phenomena: liquid water permeation
543 and water vapour diffusion. This coupled experimental and numerical approach allows
544 identifying robust and meaningful material parameters of concrete used for drying predictions by
545 inverse analysis.

546 The following conclusions are drawn:

- 547 - As expected, the mass loss measurement is not sufficient to identify the drying model
548 parameters, since there is no unicity of solutions in regard of the amount of parameters to
549 identify. This highly supports the necessity to include relative humidity measurements in
550 the identification process.
- 551 - If both experimental indicators are used in the identification, the resulting material
552 parameters are similar, whatever the experimental specimen geometry is used. Therefore,
553 when aiming at quantifying the drying process of a new type of cement-based material,
554 the experimental specimen geometry (and environment) should be chosen in order to
555 accelerate as much as possible the drying process.
- 556 - If both experimental indicators are used in the identification, the resulting material
557 parameters are independent on the input parameters. In regard of the large amount of
558 parameters used in the identification, this shows that the solution is robust, and does not
559 correspond to a local optimum.
- 560 - Several models are used and combined for the prediction of relative permeability and
561 diffusivity, as well as for desorption isotherm curves. Whatever the tested combination, it
562 is shown that the shape of the identified hydraulic conductivity and desorption isotherm
563 is equivalent. There is therefore no preferential model among the ones which were tested.

564 - The relative contribution of vapour and liquid water transport is strongly dependent on
565 the chosen relative permeability model.

566 Further works are required to confirm the relevance of this model for longer term
567 experiments, as well as additional results regarding the relative humidity gradient inside the
568 samples. This will allow a complete description of these phenomena, also an improved
569 strategy for identifying the parameters of the model. Besides, effects of modelling choice and
570 different relative humidity profiles, leading to the same weight loss prediction, on drying
571 shrinkage cracking and apparent gas permeability are currently studied. This type of study
572 could be also undertaken on the prediction of apparent diffusivity and swelling reaction
573 (ASR, DEF for instance), which are very dependent on water content.

574 **Acknowledgments**

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