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1	Combination of the Wenner resistivimeter and Torrent Permeameter methods for
2	assessing carbonation depth and saturation level of concrete
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15	Abstract

Assessing carbonation depth is of great interest for the diagnosis of reinforced concrete 16 structures because carbonation is one of the origins of steel corrosion. The assessment of 17 carbonation depth is usually performed by a simple and reliable semi-destructive test 18 consisting in spraying a colored indicator on a sample extracted from the structure. When the 19 structure is large, this test must be reproduced many times if an assessment of the variability 20 of carbonation depth is required. In this case, the extraction of multiple samples can be 21 22 prohibitive from the technical and economic points of view. So, in this case, a non-destructive testing (NDT) method could be relevant. However, even when NDT methods can be used, 23 there is a need to improve the interpretation of their results. In this study, the use of two usual 24 25 NDT methods is proposed: resistivity measurement by a Wenner probe and surface permeability assessment by a Torrent permeameter. Both techniques are implemented on 26 27 carbonated slabs having different carbonated depths and at different saturation degrees. The results show that the two techniques are sensitive to moisture and carbonation. For a given 28 saturation level, resistivity increases when the carbonated depth increases and resistivity 29 decreases when the saturation level increases. Torrent permeability decreases when the 30 saturation degree and carbonated depth increase. Good repeatability is observed for resistivity 31 measurements while a larger scatter is obtained for Torrent permeability. Empirical laws are 32

built for the relationships between resistivity or permeability and saturation degree and 33 carbonated depth. For resistivity, either a linear model between resistivity and saturation 34 degree is used or Archie's law is adapted to take the effect of carbonation into account. 35 Following the same idea, a linear law between Torrent permeability and saturation degree is 36 adapted to the effect of carbonation. Because resistivity can be measured only if the saturation 37 degree is rather high, i.e. if there is a continuity of the interstitial solution, and because 38 permeability assessment is impossible if the concrete is fully saturated, these laws are limited 39 to saturation degrees in the 40 to 83% range. 40

Then, these laws are used to predict carbonation depth and saturation degree on a wall designed with the same concrete but stored in different conditions. The results show that resistivity and Torrent permeability can be used for the combined assessment of carbonation depth and saturation degree in laboratory conditions.

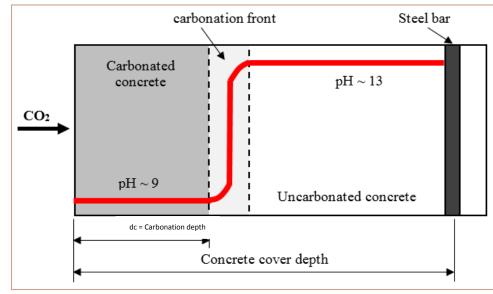
Keywords: Concrete, carbonation depth, saturation level, Torrent permeability, Wennerresistivity

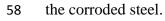
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1 Introduction

The corrosion of reinforcing steel within reinforced concrete structures produces damage such 49 50 as spalling of the concrete cover. Steel parts then remain uncovered and cause a sharp decrease in the load-bearing capacity of the structure. This failure condition is usually due to 51 the presence of chloride or to a carbonation process. The present study was conducted to 52 examine, more specifically, the carbonation process. Carbonation is a physical-chemical 53 process, which may cause a decrease in concrete pH value from 13 to 9. Carbon dioxide from 54 the air (CO_2) reacts with the portlandite of concrete $(Ca(OH)_2)$ to form calcium carbonate 55 $(CaCO_3 + H_2O)$ (Figure 1). If the whole of the concrete cover is carbonated, steel frame de-56

57 passivation is initiated and results in the delamination of the concrete cover due to swelling of





59



Figure 1: pH decrease in concrete cover due to carbonation [Ta 2016]

According to a literature review conducted by Ta et al. [Ta 2016 and 2018], a considerable amount of effort has been devoted to the study of the propagation of CO₂ and to the examination of the factors affecting the carbonation rate, such as mix proportions in concrete (e.g., cement content and water to cement ratio) [Chang 2004], [Papadakis 1989], mineral admixtures [Hui-Sheng 2009] [Papadakis, 1999 and 2000] [Younsi 2011], exposure conditions (e.g., temperature (T) [Saetta 1993] and external relative humidity (RH) [Houst 1983] [Papadakis, 1989]).

Because carbonation takes place throughout the life cycle of concrete, accurate assessment of its progress is essential to predict the initiation of steel corrosion. To achieve this, the condition of the concrete cover of structures must be surveyed at regular intervals in order to plan maintenance. The most common technique used for measuring carbonation depth is the semi-destructive coring method [RILEM CPC-18]. Phenolphthalein is sprayed on the fresh cores obtained [Chang 2006, Villain 2006]. Phenolphthalein is an organic compound used as a pH indicator: it is colorless at low pH values (around pH 9) but has a characteristic purple or
magenta color at pH values exceeding 10.5.

77

Testing is carried out at structure level with a sufficiently representative number of

measurement points, requiring a large variety of core samples [Breysse, 2017]. For improved 78 conservation of structures, non-destructive survey techniques must be favored as they can be 79 used to detect and characterize defects or damage without the need for coring [Bungey 2006]. 80 The objective of this research is to examine the sensitivity to the presence of carbonation in 81 concrete structures of two currently available non-destructive methods: the Torrent 82 permeameter and the Wenner resistivimeter. Carbonation affects the microstructure of 83 concrete by reducing porosity [Nagla 1997, Auroy 2015] because the volume of hydrates 84 increases by an average of 11.7% [Song and Kwon 2007]. As the carbonation front 85 progresses, an increasing number of pores become clogged and reduce porosity in the depth of 86 carbonated concrete. Porosity is one factor among others (e.g. water content and rebars) 87 affecting resistivity measurements in cementitious materials. Sbartaï et al. [Sbartaï 2007] have 88 demonstrated that resistivity increases when the W/C ratio of the studied concrete decreases, 89 leading to decreased porosity. A similar trend was observed by Lübeck et al. [Lübeck 2012] 90 after measuring concrete specimens with different water to binder ratios. As regards 91 permeability, some studies addressing the problem of permeability measurements using the 92 Cembureau method have shown that permeability decreases with the porosity of the studied 93 concrete [Hui-Sheng 2009] [Djerbi 2008 and 2013]. This trend is also reflected by 94 95 permeability measurements performed using the Torrent permeameter: Romer has measured greater permeability values in mortars with a W/C ratio of 0.6 than in mortars with a W/C 96 ratio of 0.35 [Romer 2005] and the same tendency was also found by Neves et al. [Neves 97 2012]. The results presented by Neves et al. show that the permeability values obtained using 98 the Torrent method decrease as compressive strength rises and that porosity therefore 99

decreases [Neves 2015]. Sena da Fonseca et al. found a relation between Torrent air-100 permeability and open porosity measured on stones [Sena da Fonseca 2015]. So it can be 101 assumed that the permeability of carbonated concrete is bound to decrease and that, 102 103 conversely, its resistivity may increase. We therefore selected two already available nondestructive methods: the Wenner resistivimeter [Polder 2001] used to measure concrete 104 105 resistivity and the Torrent permeameter [Torrent, 1992] used to measure surface permeability. In situ measurements were carried out on structures that were never completely dry for 106 107 auscultation. Depending on the weather conditions, the saturation degree of concrete may differ. Because resistivity and permeability are both sensitive to concrete moisture content, 108 109 the sensitivity must be taken into account in the measurement analysis of both devices. The influence of the resistivity as a function of the saturation level has been investigated by 110 Sbartaï et al. [Sbartaï 2007] and the results demonstrate that concrete electrical resistivity 111 112 decreases when saturation level increases. Resistivity varies between 54 and 960 Ω .m but cannot be measured when the saturation level is less than 60%. Similar results are found in 113 other studies. In [Saleem 1996], resistivity ranges from 80 to 780 Ω .m and, the lower the 114 water content, the higher the resistivity. At a mass water content of about 1.5%, the resistivity 115 cannot be measured because the concrete slab is too dry to allow the current to flow within the 116 sample. The work of Lopez and Gonzalez [Lopez and Gonzalez, 1993] underlines the effect 117 of the pore saturation level in relation to resistivity. They carried out tests on mortars with a 118 W/C ratio of 0.5. The resistivity values changed within a narrow range when the pore 119 saturation level varied between 60 and 100%. Then, between 60 and 30%, the resistivity 120 increased sharply, up to a limit corresponding to a saturation level at which measurement was 121 impossible. Recent studies have improved the resistivity techniques to monitor water and 122 chloride ingress in concrete by using a multi electrode resistivity system [Du Ploy 2015, 123 Lecieux 2015, Fares 2018]. 124

Gas permeability is a transport property characterizing gas flow under a pressure gradient and 125 is used to determine the speed with which a gas (potentially an aggressive gas like carbon 126 dioxide) may penetrate into concrete. Air permeability is affected by the concrete moisture 127 content and the concrete saturation level has been shown to have an effect on transport 128 properties. For instance, the air permeability of a concrete specimen, measured using the 129 Cembureau method, increases when the saturation level decreases [Villain 2001, Picandet 130 2001]. Kameche et al. [Kameche 2014] confirmed this influence and the present study was 131 conducted to examine the intrinsic permeability variations of CEM II concrete materials 132 according to the degree of saturation, from a saturated state to a completely dry state. Because 133 gas intrinsic permeability decreases when the saturation level increases, Cembureau 134 measurements are not possible with saturation rates above 80%. Romer conducted an 135 experimental study to assess the Torrent and Cembureau methods in CEM I mortar samples 136 137 prepared with different cement contents [Romer, 2005]. The tests were carried out by varying the relative humidity of the samples via processing at different ambient conditions. The 138 results thus obtained confirm that the permeability values measured using both Cembureau 139 140 and Torrent methods increase as the samples become drier. However, the study does not determine the sample saturation level. 141

142 It thus appears from the studies listed above that both resistivity and permeability decrease 143 when the saturation level increases. Moreover, resistivity measurements are possible with 144 saturation rates above 30% (water content higher than 1.5%) whereas Cembureau 145 permeability measurements are only possible with rates below 80%.

Permeability is a transport property depending on the porosity of the material studied. Because carbonation decreases porosity, the surface permeability of carbonated concrete measured using the Torrent permeameter can be expected to change. As regards water permeability, the water flow is weaker in carbonated than in non-carbonated concrete [Song and Kwon, 2007]. Auroy et al. underline a decrease in intrinsic permeability after carbonation
for CEM I Portland cement [Auroy, 2015].

Similarly, because resistivity is affected by the material microstructure, variations in 152 resistivity can be expected according to the carbonation depth. However, the literature 153 reviewed for this paper contains no research on the resistivity and permeability of carbonated 154 concrete specimens using Wenner's and Torrent's methods. The present study, therefore, 155 addresses carbonation and moisture effects on resistivity and permeability measurements in 156 157 terms of both quality and quantity in laboratory conditions. It is also important to note that rebar close to surface affects the resistivity. We chose to avoid this effect as it is difficult to 158 evaluate, except maybe by using a numerical model for the determination of apparent 159 resistivity [Nguyen 2017]. 160

Using the Wenner resistivimeter and the Torrent permeameter, and taking account of the 161 162 sensitivity of the measurement equipment, resistivity and permeability results measured in concrete slabs made of the same mixture, with varying carbonation depths and saturation 163 levels ranging from a dry state to a saturated state, were determined. The experimental results 164 are analyzed below and translated into laws considering the dispersion of the measurement 165 values. The objective of this analysis is the simultaneous use of both non-destructive methods 166 167 (NDT) to assess saturation level and carbonation depth in real structures. Finally, the laws of correlation established were validated using a concrete wall made of the same concrete mix as 168 the experimental slabs. 169

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175 2 <u>Materials and experimental program</u>

176 2.1 <u>Concrete specimens</u>

The concrete mix was composed of CPA-CEM I 52.5 Portland cement with a water/cement 177 ratio of 0.8 to accelerate the carbonation process (Table 1). Eleven slabs (rectangles 50 x 25 178 and 12 cm thick) were prepared from a single batch of concrete in a laboratory in Toulouse 179 (France): nine were cast for non-destructive testing, one was used for determining the 180 physical and mechanical properties and the last one was sealed on five faces and put into the 181 carbonation chamber to monitor carbonation depth. The concrete mixtures were cast in plastic 182 molds and compacted under vibration. After casting, the slabs were stored in a room at 20 °C 183 184 with about 95% relative humidity (RH) for 24 h until demolding, after which they were cured 185 in water at 20 °C for 28 days.

After 28 days, two slabs were used to assess the mechanical and physical properties of the 186 concrete. Cylindrical cores were extracted from these slabs to drive these tests. The results are 187 summarized in Tables 2 and 3. Open porosity was measured using the water saturation 188 method on three specimens. Three specimens were tested under axial compressive loading 189 conditions with measurement of lateral and axial displacements, and three other discs were 190 sealed with two layers of adhesive aluminum tape to ensure a one-dimensional gas flow 191 192 through the discs. These specimens were oven-dried at 105 °C to constant weight before permeability testing was conducted using the Cembureau device. 193

194

Constituents (kg/m3)	OPC C1
Cement CEM I 52.5 N CE CP2 NF	240
Siliceous Sand 0/2 or 0/4	941
Siliceous aggregates 4-14 or 10-14	1019
Water	193
Superplasticizer	0.96
W/C	0.8
Slump (cm)	14

Properties	OPC C1
Dry apparent density (kg/m ³)	2 222
Open porosity measured by water	18.3
saturation method (%)	
Cembureau apparent permeability	
with an injection pressure of 0.5	7.71
bars measured in dried specimen	/./1
$(10^{-16} \text{ m}^2) (= \text{K}_{\text{ref}},\text{C})$	

Table 2: Physical and transport properties (average of three measurements)

197

Mechanical properties (28 days)	OPC C1
compressive strength (MPa)	21.5
Modulus of elasticity (MPa)	24927
Poisson ratio	0.208

199

Table 3: Mechanical properties at 28 days (average of three measurements)

200

201 2.2 <u>Carbonation procedure</u>

The nine slabs intended for non-destructive testing were referenced as N or T (Table 4). After exposure to carbonation, the slabs referenced N were stored in Nantes (France) and those referenced T, in Toulouse.

After a 28-day residence time in water, the slabs were oven-dried at 80 °C until constant 205 weight was reached. The dry mass, W_d, was used to calculate the water content at time t 206 (Equation 1), which, in turn, was used to calculate the degree of saturation at time t (Equation 207 2). At the end of the drying phase, the slabs were stored in laboratory conditions. The four 208 209 slabs referenced as 1 (C1-1-7N, C1-1-8N, C1-1-1T, C1-1-2T) were completely sealed with 210 two layers of adhesive aluminum tape to prevent carbonation. The slabs referenced C1-2N, C1-3N, C1 4N, C1-2T, and C1-3T were sealed on five faces only, to ensure a one-211 dimensional carbonation processing from one side of the slab only (50 cm x 25 cm). These 212 slabs were placed in a chamber at 20 °C, with a relative humidity of 65% and a CO₂ content 213 of 50% to accelerate the carbonation process. The core samples were extracted from the 214

196

specific sampling slab at the end of the curing phase and stored with the slabs in the 215 laboratory. They were also placed in the carbonation chamber to monitor carbonation 216 progress, just like the slabs. However, during this phase, the different core samples were 217 regularly broken and immediately sprayed with the phenolphthalein color indicator so as to 218 assess the carbonation depth at a given point of time. When the different desired carbonation 219 depths had been reached on the control core samples, the slabs were removed from the 220 chamber and immersed in water to stop carbonation and to ensure complete saturation of the 221 222 slabs.

The real carbonation depths measured after completion of the whole experimental procedureusing NDT techniques, are presented in Table 4.

225

Slab reference	Expected carbonation depth (mm)	Measured carbonation depth d _c (mm) (cf. Figure 1)			
C1-1-7N	0	0			
C1-1-8N	0	0			
C1-2N	10	9.1			
C1-3N	20	18.2			
C1-4N	30	27.3			
C1-1-1T	0	0			
C1-1-2T	0	0			
C1-2T	10	10.5			
C1-3T	20	17			

226

Table 4: *Expected* and measured carbonation depths

227

228 2.3 <u>Experimental procedure</u>

The operating method consisted in varying the slab water content in order to obtain permeability and resistivity correlation curves according to the saturation level. After saturation, different phases of drying were undertaken in order to achieve measurements with

targeted water contents corresponding to identical saturation levels for all the slabs. For that 232 purpose, the slabs were placed in a drying oven at 80 °C and dried while their weight loss was 233 monitored. It was assumed that the concrete was not modified at 80 °C, since ettringite is 234 considered to dehydrate at a temperature higher than 80 °C [Zhou and Glasser 2001] and 235 therefore would have remained almost stable at 80 °C. When the targeted mass was obtained 236 and the drying phase completed, the slabs were hermetically sealed in airtight bags for forty 237 days to balance the humidity level of the slabs and prevent natural carbonation. Their mass 238 was measured regularly to leak-check the bags. Once the slabs were taken to be homogenized, 239 the slab mass, W(t), was measured again. Then, resistivity and permeability measurements 240 241 were carried out. The water content was determined from Equation 1:

242
$$w(t) = (W(t)-W_d)/W_d$$
 (1)

where w(t) is the water content at time t, W(t) is the mass of the slab at time t (kg) and W_d is the mass of the dried slab (kg).

245 The degree of saturation at time t is obtained from Equation 2 as:

246
$$S(t) = w(t) / w_{sat}$$
 (2)

247 where w_{sat} is the water content of the saturated slabs.

248

249 For each measurement method (Wenner resistivimeter and Torrent permeameter), a total of ten measurements per test point and saturation level were performed. Measurements were 250 carried out in the center of all the slabs (Point 1 in Figure 2). However, in order to check slab 251 homogeneity and measurement sensitivity to side effects, measurements were also carried out 252 on either side of Slab C1-1-8-N center (Points 2 and 2' in Figure 2). The measurements were 253 made first at point 1, then at point 2 and finally at point 2' before returning to spot 1. So there 254 was enough time between consecutive measurements in the same spot to allow vacuum 255 dissipation. 256

The Wenner resistivity measurements were first carried out to determine the resistivity values, which were afterwards used for the Torrent surface permeability measurements [Torrent 1992].

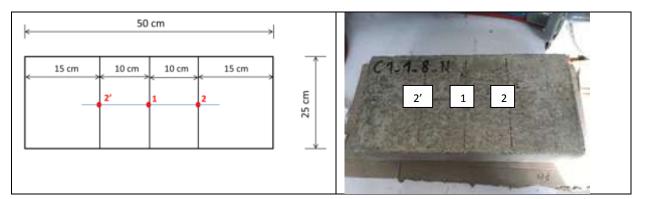


Figure 2: Location of measurement points on the slabs

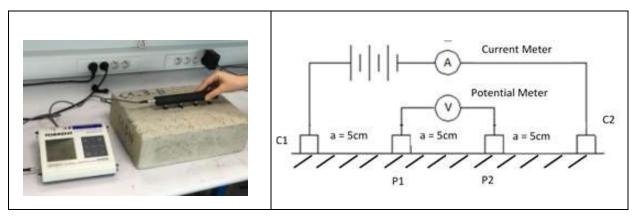
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261

The Wenner resistivimeter is a measurement device using Wenner protocol to determine the 262 apparent resistivity of concrete facings. Measurements are assumed to be carried out on a 263 semi-infinite homogeneous material. The measurement device is called a Wenner 264 resistivimeter because of the electrode arrangement, in which the distance interval between 265 the electrodes, called a, is the same, with $a = \overline{C_1 P_1} = \overline{P_1 P_2} = \overline{P_2 C_2}$ (Figure 3). The device used 266 in this work had a distance interval of 5 cm. First, an electric current, I, is injected between 267 electrodes C1 and C2. Then, the electrical potential is measured between electrodes P1 and 268 P2. The apparent resistivity is calculated by the control acquisition unit, based on the 269 following formula: 270

$$\rho = 2\pi a \frac{v}{I}$$
(3)

where ρ is the resistivity in Ω .m, a is the distance interval between the electrodes in m, V is the voltage in volts and I is the current intensity injected into the sample in amperes.



276

Figure 3: Wenner resistivity measurements

After the resistivity measurement was finished, the Torrent permeability was measured, if possible, depending on the slab saturation level. The test principle is to create a vacuum inside the test chamber using a vacuum pump and then disconnect the pump when the desired pressure is reached (Figure 4).

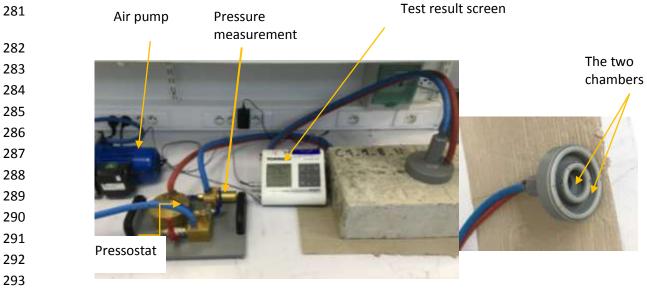


Figure 4: Torrent permeameter



294

296 The rate of increase of pressure inside the vacuum cell can be measured and the air 297 permeability can be calculated according to the following formula:

298
$$K = \left(\frac{V_c}{A}\right)^2 \cdot \frac{\mu}{2 \varepsilon P_a} \left[\frac{\ln\left(\frac{P_a + \Delta P}{P_a - \Delta P}\right)}{\sqrt{t} - \sqrt{t_0}}\right]^2$$
(4)

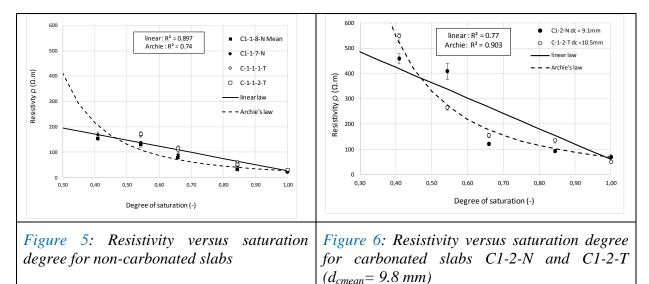
299

300	with: K: Torrent gas permeability coefficient (m ²),
301	μ: Air dynamic viscosity at 20 °C (2.0.10-5 N.s/m ²),
302	Vc: Capacity of the inner chamber and of the elements, through which air flows during
303	the filling phase (Vc = 222.10^{-6} m^3),
304	ε: Concrete porosity introduced in the data (18.3 %, cf. Table 2).
305	A: concrete cross-section where air flows in the vacuum cell (19.6.10 ⁻⁴ m ²),
306	ΔP : Pressure difference in the inner chamber in N/m ² ,
307	Pa: Atmospheric pressure in N/m ² ,
308	t0: Test start time (60 s),
309	t: Test duration (< 720 s).
310	
311	3 <u>Results</u>
312	3.1 <u>Resistivity results</u>
313	Resistivity measurements at the three points on a given slab showed little scatter in the results,
314	indicating that the concrete was homogeneous over the whole slab volume. It was concluded
315	that the saturation level of the slab was spatially homogeneous on the surface investigated.
316	Moreover, resistivity values were not influenced by side effects. Consequently, only the
317	average of the different measuring points appears in Figures 5 to 8. The standard deviations
318	were calculated from thirty measurements (ten measurements for Point 1, ten measurements
319	for Point 2 and ten measurements for Point 2').
320	For saturation levels below 41%, measurements proved impossible for the most carbonated
321	slab because it was too dry to allow electricity to flow freely between the electrodes.
322	Whatever the carbonation depth reached, Figure 5, 6, 7 and 8 show a lower resistivity when
323	the sample saturation level increases. The trend observed in Figures 6 and 7 for non-
324	carbonated samples has been highlighted by many authors [Sbartaï 2007, Saleem 1996, Lopez

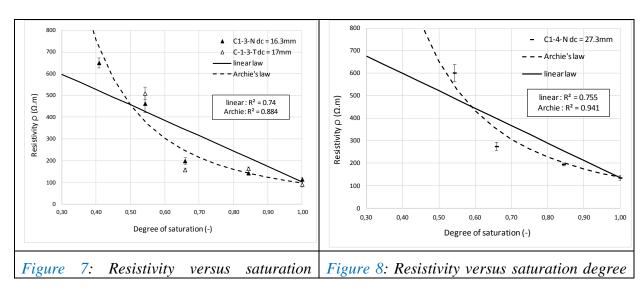
325 1993]. When the saturation level decreases, the concrete liquid phase gradually changes to a 326 discontinuous pattern that makes ion conduction more difficult. Resistivity values then 327 increase sharply. In non-carbonated slabs, resistivity increases by a factor of six between 328 saturated samples and samples with a 41% saturation level. This explains the non-linear 329 behavior of the electrical resistivity in relation to the variations of concrete saturation levels.

Carbonation depth also affects resistivity. For the same saturation level, resistivity increases
because of concrete structural changes due to carbonation reactions. The decrease in porosity
induced by carbonation increases the material compactness, resulting in a less prevalent liquid
phase. The current then flows less freely and resistivity increases.









degree for carbonated slabs C1-3-N and C1- $3-T (d_{cmean} = 16.65 \text{ mm})$ for carbonated slab C1-4-N with $d_c = 27.3 \text{ mm}$

336 Some mathematical laws are now examined for best fitting of the experimental points. We 337 first select a linear law because the results achieved for the non-carbonated slabs, within the 338 saturation level range for which measurements are possible, follow a linear trend. Then the 339 linear equation to determine the mean resistivity of saturated slabs when the degree of 340 saturation is 100% is taken as:

341
$$\rho = a * (1 - S) + b$$
 (5)

342

In the case of non-carbonated and fully saturated slabs, b is the reference value ρ_{ref} , called the reference resistivity. This value corresponds to the mean resistivity obtained on noncarbonated slabs in saturated conditions (C1-1-8-N, C1-1-7N, C1-1-1-T, C1-1-2-T). Here, the value of b is 27.627 Ω .m.

In the case of carbonated slabs, b is the mean resistivity calculated from the two saturated slabs (S=100%). For a carbonation depth of 27.3 mm, the value of b is obtained using Slab C1-4-N only. The values of a obtained by fitting the experimental data using Equation 5 are reported in Table 6.

351

The empirical Archie's law [Archie 1942] is also used to describe the general trend identified in these figures. This law describes the dependency of resistivity on the degree of saturation (S), porosity (ϕ) and resistivity of the composition pore solution (ρ_{sol}) as:

$$\rho = \rho_{\rm sol} * S^{-n} \phi^{-m} \tag{6}$$

356 Parameters m and n are usually regressed over the experimental data.

357

To use this law, some parameters must be set. The pore solution resistivity is assumed to be constant throughout the carbonation process. However, it is reasonable to believe that the 360 change in the cement matrix due to the dissolution of the portlandite modifies the pore361 solution and therefore its conductivity.

362 The conductivity of the pore solution (σ sol=1/ ρ _{sol}) can be expressed as:

363
$$\sigma_{sol} = \sigma_{wat} + \Sigma(C_i * \lambda_i)$$
(7)

364 where $\sigma_{wat} = 10^{-5}$ S/m, corresponding to the water conductivity as found in [Shi 2003]

365 C_i ionic content (mol/m3)

366
$$\lambda_i$$
 ionic molar conductivity (S.m²/mol)

367

The conductivity of the pore solution is assessed by combining data by Andersson et al. 368 369 [Andersson 1989] and Nguyen [2006] (Table 5). Two solution conductivities are calculated: σ sol = 8.92 S/m with the values by Nguyen and σ sol = 6.41 S/m with the values by 370 Andersson. Both these authors used very similar Portland cement but the mixes were 371 different. The mean value ($\sigma_{sol} = 7.66$ S/m) is used to calculate the resistivity of the pore 372 solution of the sample tested. This value is of the same order of magnitude as the values found 373 in the literature, although the literature values obviously depend on cement type and water to 374 cement ratios [Sanish 2013, Sant 2011, Neithalath 2006]. 375

376

Ion	Ionic molar	Ionic content	Ionic content
	conductivity	(mol/m^3)	(mol/m^3)
	(mS.m²/mol)	[Nguyen	[Andersson 1989]
		2006]	
Na ⁺	5,01	100	42.3
\mathbf{K}^+	7.35	272	161.1
Ca ²⁺	11.9	2.6	2.2
Cl	7.63	0.5	Not evaluated
OH	19.86	321.4	251.2
			(calculated using
			measured pH)

Table 5: Values used to calculate the conductivity of the pore solution

378

377

In order to determine the factor n, which is assumed to be constant for both carbonated and 379 380 non-carbonated slabs, the data obtained on non-carbonated slabs are used. In this case, the factor m is calculated using Archie's law for S=100% ($\rho_{ref} = \rho_{sol} * \phi^{-m_{ref}}$). We then have 381 m_{ref} = 3.153 with ρ_{ref} =27.627 Ω .m. The factor n is therefore determined by fitting the 382 experimental data of the non-carbonated slabs using Archie's law as: n = 2.24 (Figure 5). For 383 the carbonated slabs (Figures 6, 7 and 8), n is kept constant and only m is adjusted to achieve 384 the best possible fitting with the experimental data because it is assumed to depend on the 385 microstructure, which changes during carbonation. 386

Table 6 displays the different law parameters and determination coefficients obtained. Both laws can be used to represent the trend obtained because the determination coefficients are correct in both cases. The determination coefficients given by the linear law, however, are lower, except on non-carbonated slabs, as the linear law does not represent points obtained for low saturation levels very accurately. It seems likely that, for a dry material, resistivity tends toward a very high value. This trend is thus best represented by Archie's law.

393

		a/n	b/m	Determination
				coefficient R ²
Non-carbonated	Linear	238.16	27.627	0.897
slabs	Archie	2.24	3.153	0.74
$d_{\rm cmean} = 9.8 \rm mm$	Linear	725.955	59.9	0.77
$u_{c mean} - 9.0 \text{ IIIIII}$	Archie	2.24	3.70	0.903
$d_{\rm c\ mean} = 16.65$	Linear	864.852	102.63	0.74
mm	Archie	2.24	3,89	0.884
$d_c = 27.3 \text{ mm}$	Linear	913.16	135	0.755
$u_c = 27.3$ mm	Archie	2.24	4.10	0.941

Table 6: a and b (linear law), n and m (Archie's law) and corresponding determination
 coefficient R²

397 The relations of coefficients a and b with mean carbonation depth are described as a 398 logarithmic function (Equation 8) with $R^2 = 0.984$ and a linear function (Equation 9) with R^2 399 = 0.982, respectively. As regards Archie's law, the plot of coefficient m versus mean 400 carbonation depth is described by a linear function with $R^2 = 0.91$ (Equation 10).

401
$$a = 91.645 * \ln(d_c) + 442.03$$
 (8)

402
$$b = 4.0198 * d_c + 27.627$$
 (9)

403
$$m = 0.0389 * d_c + 3.153$$
 (10)

By combining relationships 5 and 6 with Equations 8, 9 and 10, we obtain the laws describing
electrical resistivity versus carbonation depth and saturation degree, respectively (Equations
11 and 12):

407
$$\rho = (91.645 * \ln(d_c) + 442.03) * (1 - S) + (4.0198 * d_c + 27.627)$$
(11)

408
$$\rho = \rho_{sol} * S^{-2.24} \phi^{-(0.0389*d_c+3.153)}$$
 (12)

- 409 where d_c is the carbonation depth in mm and ρ the Wenner resistivity (Ω .m)
- 410

The coefficients of determination obtained for the linear law parameters are higher than that 411 obtained for parameter m of Archie's law. These laws may be used for the calculation of the 412 saturation level from a resistivity measurement with knowledge of the carbonation depth. This 413 depth would have been determined, for instance, by spraying phenolphthalein on a freshly 414 drilled core sampled from the structure. Carbonation depth determination is faster than 415 saturation level determination because it can be read immediately after sampling without 416 waiting for the complete drying needed for saturation level measurements. However, the laws 417 established here apply only to concrete with similar porosity (about 18%) and mixed with 418 CEM I cement. Solution resistivity and carbonation process both depend on the type of 419 cement and any addition modifies the chemical reactions. These laws should also consider the 420 measurement error aspects. In order to seriously take the standard deviations displayed in 421 422 Figures 5, 6, 7 and 8 into account, Figure 9 presents the Coefficient of Variation (CoV),

corresponding to the standard deviation divided by the average of the values measured, for all 423 the saturation levels. CoV values are very low (lower than 10% when the value obtained from 424 the saturation measurements carried out on the non-carbonated slabs is eliminated). Moreover, 425 this maximum value of 20% is of the same order of magnitude as the CoV calculated from 426 laboratory measurements (approximately 18%) [Aït-Mokhtar, 2013]. It may be recalled that 427 measurements appear reliable here, because the lower the CoV value, the higher the 428 measurement accuracy. The results obtained on the non-carbonated slabs correspond to the 429 lowest resistivity values and, hence, to higher CoV values. This analysis suggests that the 430 Wenner measurements carried out using this device can be reproduced. 431

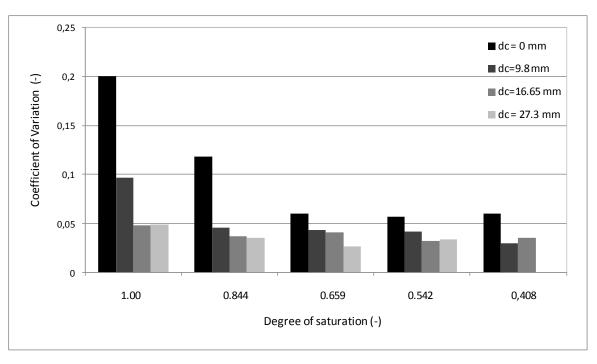


Figure 9: Coefficient of variation of the resistivity measurements as a function of saturation
degree and carbonation depth

435

432

436 3.2 <u>Air permeability results</u>

437 Permeability measurements at the three points on a given slab showed scatter in the results.
438 The differences observed cannot be attributed to the heterogeneity of the concrete material as
439 all the resistivity measurements performed on the same slab for a given saturation level are

440 almost identical whatever the measurement point. Because side effects have not been 441 demonstrated, the three point averages are presented with, on either side, the range defined by 442 the standard deviation calculated from all the values (30 measurements). Moreover, on 443 different concrete compositions, Neves et al. showed that, if the average of a set is used as the 444 representative value, it is possible to distinguish between different concrete mixes and 445 different curing (for the same mix) [Neves 2012].

446 Figures 10 through 13 show that, for a given carbonation depth, gas permeability decreases when the saturation level increases and also decreases when carbonation depth increases. 447 Permeability also varies greatly in the slabs, which are supposed to be of the same concrete 448 449 and in the same saturation state. Figure 10 shows that, when Sr = 15%, permeability ranges from 17 to 25.10-16 m², i.e. an increase of almost 50%. Although measurements are not 450 possible on the fully saturated samples, they can, nevertheless, be performed up to 84%. 451 452 Above this saturation rate, gas can no longer flow freely because of water molecule continuity in the concrete porous network. Concrete relative humidity reduces pore interconnectivity 453 454 and, therefore, affects its permeability. The permeability vs. saturation level curve has already been assessed in the literature for non-carbonated materials using Cembureau measurements 455 carried out on different concrete types [Kameche 2014, Abbas 1999]. These authors have 456 established a logarithmic law to describe, as far as possible, the experimental results regarding 457 intrinsic permeability as a function of saturation level for different sample sizes. 458 Measurements are possible up to approximately 80% of the saturation level. As regards the 459 work of Abbas et al. [Abbas 1999], a linear law best represents the results obtained for the 460 apparent permeability measurements at different pressures as a function of the saturation 461 level, with a saturation threshold of 90%. It should, however, be noted that the measurement 462 variation ranges of both studies are larger than that obtained in the present paper using the 463 Torrent permeameter, which results in a logarithmic display of their results. The present 464

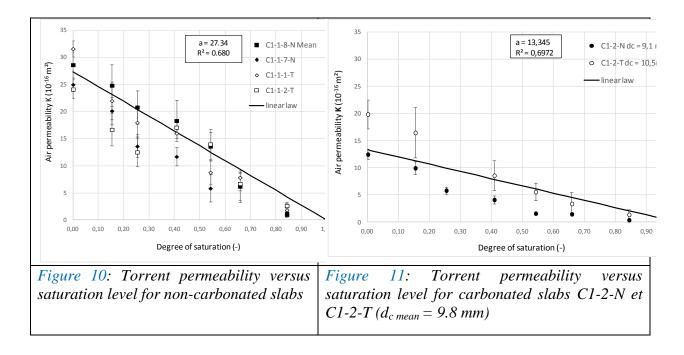
results, on the other hand, are displayed on a linear scale for viewing comfort. They are fitted with a linear law for all the carbonation depths reached by imposing zero permeability as a condition for the saturated samples (S = 100%). This law is described by Equation 13. As regards dry, non-carbonated slabs, the reference value, $K_{ref,T}$ is imposed as the value for a. This value corresponds to the average obtained from the Torrent permeability measurements carried out on the dry non-carbonated Slabs C1-1-8-N, C1-1-7-N, C1-1-1-T and C1-1-2-T, is 27.34 10-16 m².

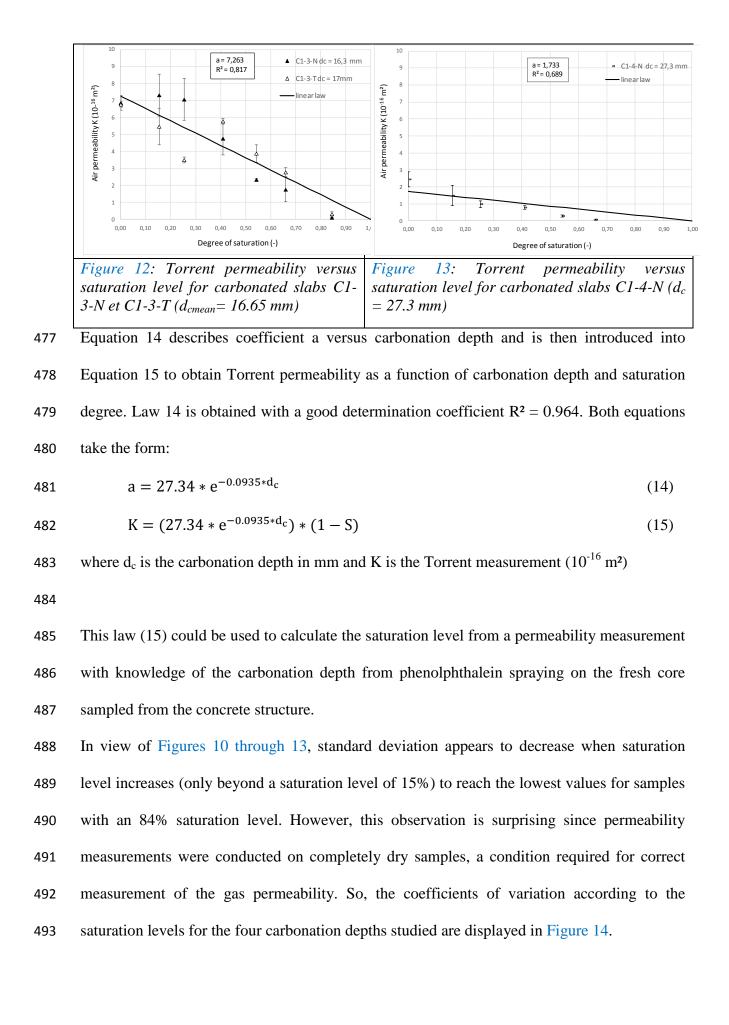
$$K = a * (1 - S)$$
 (13)

473

472

The coefficients of determination are correct in view of the large standard deviations, which make it possible to confirm that the linear laws express Torrent permeability evolution satisfactorily as a function of the saturation level.





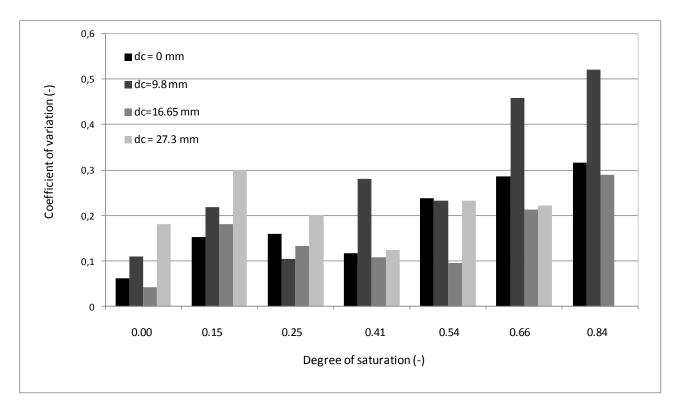


Figure 14: Coefficient of variation of Torrent permeability measurements as a function of
 saturation degree and carbonation depth.

497

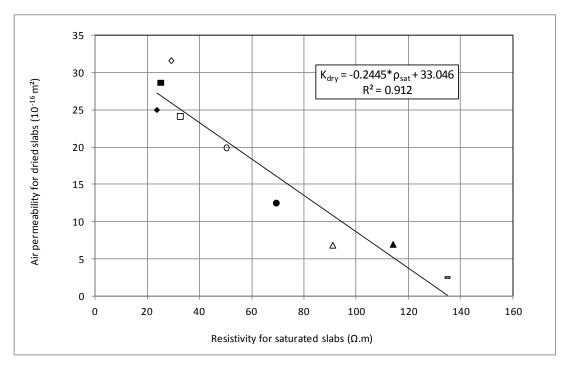
498 The results presented in Figure 14 are closer to expectations. Carbonation depth has no effect on the measurements because trends observed in the carbonated slabs differ depending on the 499 500 saturation level. CoV, on the other hand, effectively increases with the degree of saturation. It should be noted, however, that CoV could not be measured for $d_c = 27.3$ mm at an 84.1% 501 saturation level, which was at the device operating limit. Moreover, with this saturation level, 502 the coefficient of variation ranges between 30 and 50%. This high range raises questions as to 503 504 measurement reliability. With these saturation levels, the device operating limits are exceeded, all the more so when the concrete is compact. A key idea of this analysis is that 505 saturation level S and carbonation depth d_c can be determined from the measured K and ρ by 506 combining Equation 15 with Equation 11 or 12. However, these established relationships 507 apply only if the concrete porosity is equivalent (about 18%) and the formulation uses CEM I 508 cement, as in this study. Resistivity and permeability measurements depend on the material 509 composition, which in turn affects the porosity and conductivity of the pore solution. 510

511	Moreover, the combination of the two laws is possible only if both measurements can be
512	performed simultaneously, i.e., when the concrete saturation level falls within the range 40-83
513	%, which is quite often the case where in situ concrete is concerned.

515 4 <u>Result analysis</u>

516 4.1 <u>Correlation between resistivity and Torrent gas permeability</u>

517 Figure 15 shows the correlation between Torrent gas permeability measurements for dried slabs (S = 0%) and resistivity measurements carried out on saturated slabs (S = 100%). This 518 comparison is possible since the saturation level is set aside as the impact of pore saturation is 519 520 canceled out. The symbols used are the same as in Figures 5 through 8 and 10 through 13. A linear correlation is observed between the two parameters. The slabs with the highest 521 permeability present the lowest resistivity. Both properties share some crucial pore structure 522 characteristics, such as total porosity, pore tortuosity and connectivity, of the cover concrete 523 subjected to carbonation. They are sensitive to the different phases occurring within pores: 524 525 gas permeability is linked with the characteristics of the gas phase and resistivity depends on the liquid phase through its electrochemical composition. Nevertheless, a good linear 526 correlation is observed for the material studied here. The correlation established by Gui et al. 527 528 [Gui 2016] for a study conducted to examine gas permeability using the Cembureau device and electrical conductivity using the high frequency alternating current method on laboratory 529 concrete specimens with different water to binder ratios and different admixtures is not as 530 531 good as the correlation proposed in this paper.





- 533 534
- 554

Figure 15: Torrent permeability for dried slabs versus resistivity for saturated slabs

535 4.2 <u>Resistivity analysis</u>

Relative resistivity is considered because it could be useful to compare different concretes: it 536 has been used by Gui el al. to study electrical conductivity and gas permeability of concrete 537 formulated with CEM I and blast-furnace slag cement [Gui, 2016]. Using a relative value for 538 resistivity and permeability could also be very useful for a modeling approach [Li, 2016]. 539 Resistivity values are then processed after dividing them by the reference value ρ_{ref} . This 540 541 value can be considered as an intrinsic transport property since the impact of carbonation and pore saturation is canceled out. This value is measured directly on the slabs using the Wenner 542 543 method. However, in future, taking the value measured on laboratory samples as the reference value when selecting concrete type might be contemplated [Baroghel-Bouny, 2004; Aït-544 Mokhtar, 2013]. 545

546 We use the following relationship to fit the experimental data:

547
$$\frac{\rho}{\rho_{\text{ref}}} = A * (1 - S) + B$$
 (16)

548 with A=a/ ρ_{ref} and B = b/ ρ_{ref} , a and b being the values reported in Table 6.

549 Solutions are obtained from Equations 8 and 9 as:

550
$$A = 3.317 * \ln(d_c) + 16$$
 (17)

551
$$b = 0.1455 * d_c + 1$$
 (18)

552 Considering Archie's law, Equation 16 becomes:

553
$$\frac{\rho}{\rho_{\text{ref}}} = S^{-n} \phi^{-(m-m_{\text{ref}})}$$
(19)

where n= 2.24, m_{ref} corresponds to the value calculated in Section 3.1, m_{ref} = 3.153, and values of m are given by Equation 10. We have:

556
$$\frac{\rho}{\rho_{\text{ref}}} = (3.317 * \ln (d_c) + 16) * (1 - S) + (0.1455 * d_c + 1)$$
 (20)

557
$$\frac{\rho}{\rho_{\text{ref}}} = S^{-n} \Phi^{-(m-m_{\text{ref}})}$$
(21)

where $m = 0.0389 * d_c + 3.153$ and d_c is the carbonation depth in mm

559

If these laws are validated with concretes presenting other porosities, they may be used for different concrete types if the ρ_{ref} measurement is given. This ρ_{ref} measurement value should become a durability indicator on which the choice between concretes for engineering structures could be based [Baroghel-Bouny, 2004].

These laws present equations with two unknown variables (saturation level and carbonation depth) whereas the survey of the structure provides only the resistivity ρ . Consequently, it is essential to combine this equation (linear or exponential) with the relationship established for permeability.

568

569 4.3 <u>Permeability analysis</u>

570 In order to establish correlations that are independent of the permeability measured on dry, 571 non-carbonated concrete, a relative permeability approach is used [Djerbi Tegguer 2013, 572 Kameche 2014]. Permeability values are then processed after being dividing by the reference 573 value K_{ref,T}. This value, which is the average value of the Torrent permeability measurements 574 on dry Slabs C1-1-8-N, C1-1-7-N, C1-1-1-T and C1-1-2-T, is 27.34 10-16 m². The 575 permeability measured on a sound, dry sample should become a mandatory parameter for 576 selecting concrete mixtures. This value can be considered as an intrinsic transport property 577 since the impact of carbonation and pore saturation is canceled out. This measurement value 578 might become a durability indicator that could be provided with the formulation of concrete 579 [Baroghel-Bouny 2004, Aït-Mokhtar 2013].

The results are displayed in Figures 16 through 19 for different carbonation depths. They are fitted first with a linear law, then with a Van Genuchten law modified by Mualem [1976] and used by [Kameche 2014] to represent relative permeability as a function of saturation level.

583 When fitting with the linear law, the coefficients of determination achieved using Equation 15584 are obtained as:

585
$$\frac{K}{K_{\text{ref,T}}} = e^{-0.0935 * d_c} * (1 - S)$$
(22)

586 with d_c the carbonation depth in mm.

587 The modified Van Genuchten law (VG law) is then defined as:

$$\frac{K}{K_{\text{ref},T}} = (1 - S)^{q} * (1 - S^{2})$$
(23)

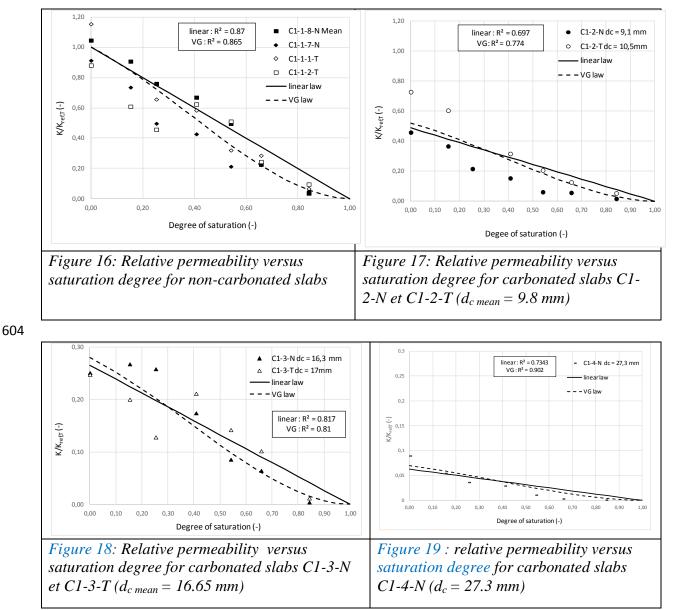
Parameter q is determined by fitting this law with the experimental results obtained on non-589 carbonated slabs. The results displayed in Figure 16 show that q = 0.89. This value is lower 590 than the values previously achieved for cementitious materials. Monlouis-Bonnaire 591 [Monlouis-Bonnaire 2004] proposes q = 5.5 for cementitious materials and [Kameche 2014], 592 q = 3.5 for relative gas permeabilites measured using the Cembureau method. Permeability 593 variations are very pronounced when they are measured using the Torrent permeameter. For a 594 40% saturation level, relative permeability is 0.6 with the Torrent method but only 0.1 with 595 596 the Cembureau method [Kameche 2014]. Parameter q, used to quantify the effect of saturation level on relative permeability, is held constant for carbonated slabs. However, a C factor is 597 introduced to consider the decrease in relative permeability with carbonation. 598

600 The VG law then becomes:

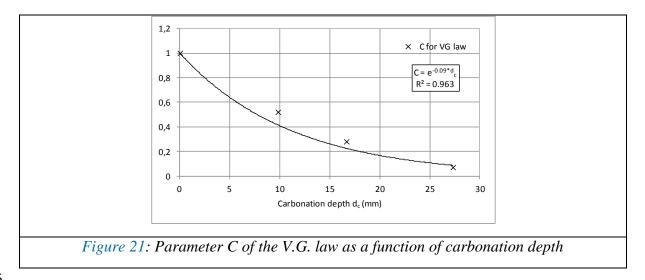
601
$$\frac{K}{K_{\text{ref},T}} = C * (1 - S)^{q} * (1 - S^{2})$$
(24)

Figure 20 presents the empirical parameter, C, determined by fitting with the experimental

603 results.



605



The coefficients of determination demonstrate that the VG law describes relative permeability evolution better. This law can therefore be used to describe Torrent permeability evolution according to carbonation depth and saturation level as:

610

611
$$\frac{K}{K_{\text{ref},T}} = e^{-0.09 * d_c} * (1 - S)^{0.89} * (1 - S^2)$$
(25)

612 with d_c the carbonation depth in mm.

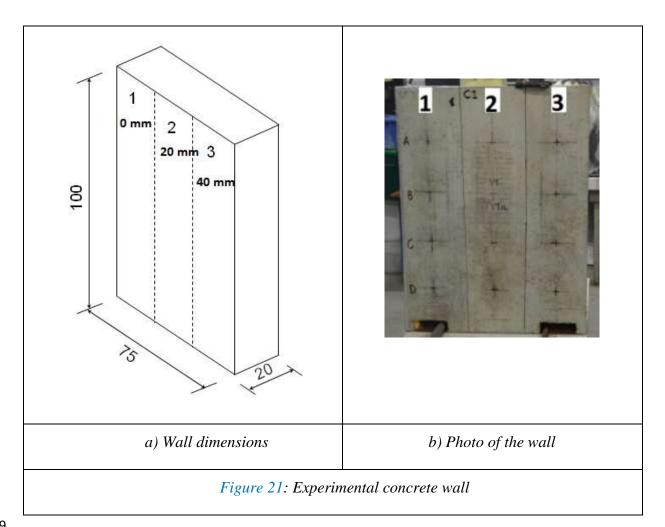
The VG law for relative permeability and Archie's law for relative resistivity can be used to obtain better coefficients of determination than with linear laws, in particular for carbonated slabs. The two laws can be combined to determine the saturation level and the carbonation depth of similar concrete structures. However, in order to use both measurement techniques simultaneously, the concrete saturation level of the structure must fall within the range 40-66%.

If validated with concrete presenting other porosities, these laws may also be used on other concrete materials if $K_{ref,T}$ measurements, carried out using the Torrent permeameter on dry non-carbonated samples, and ρ_{ref} measurements, carried out using the Wenner resistivimeter on saturated non-carbonated samples, are given. Both measurements could be performed prior to the construction in order to ensure the best choice of concrete formulations. During the survey conducted to apply these laws, the surveyed areas of the structure should be checked for cracking, as concrete damage modifies permeability [Picandet, 2001; Djerbi, 2008 and 2013] as well as resistivity measurements [Taillet, 2014; Lataste 2003]. If damage is detected, another non-destructive technique should be used and relationships between, for instance, resistivity measurement and saturation level, carbonation depth and damage should be established.

- 630
- 631

4.4 <u>Validation using a concrete wall</u>

The slabs studied in this paper were used to examine the influence of carbonation on NDT 632 measurements by studying moisture homogeneity on saturated or partially saturated concrete. 633 Equations 11, 12 and 15 were established using experimental measurements carried out on the 634 slabs. In order to validate the performance of these laws, an experimental wall, 100 cm x 75 635 cm and 20 cm thick, was prepared (Figure 21a). The concrete used for the wall was the same 636 as for the slabs. A reinforcement mesh was placed at a depth of 25 mm on one face of the 637 wall. The diameter of the bars was 10 mm and the size of square mesh was 20 cm. 638 639 Measurements were only made on the non-reinforced side of the wall in order to avoid the 640 influence of the reinforcement, especially on resistivity measurements. This face of the wall was divided into three vertical sections corresponding to different carbonation depths. After 641 28 days of storage in a humid chamber, the first section (noted 1 in Figure 21), corresponding 642 to a carbonation depth of 0 mm, was completely sealed. Then, the wall was placed in a 643 carbonation chamber at 20 °C and 65% relative humidity, with a CO2 content of 50% to 644 accelerate the carbonation process. Section 2, corresponding to a carbonation depth of 20 mm, 645 646 was also sealed after the same exposure to the carbonation process as Slabs C1-3N and C1-3T. The wall was finally removed from the carbonation chamber after the same exposure time 647 as Slab C1-4N. 648



NDT measurements were carried out at twelve points on the wall (indicated with crosses in 650 Figure 21b). Then, core samples were taken in the center of each mesh to obtain real 651 carbonation depths and saturation levels. Each core sample was cut into three pieces: one 652 from each end, with a thickness corresponding to the desired carbonated depth, and one 653 middle piece corresponding to sound concrete (non-carbonated). Because coring and sawing 654 were done under water, at the end of the operations, samples were dried with a cloth to avoid 655 the absorption of water as far as possible and so to avoid modifying the moisture content. The 656 carbonation depth was observed by spraying the core samples with the phenolphthalein color 657 indicator and the saturation level was calculated after drying the core samples as described in 658 Section 2.3. The values reported in Table 7 are the mean values obtained from 8 659

660	measurements	(four	core	samples	for	each	vertical	section	and	two	pieces	for	each	core
661	sample).													

Vertical section	Measured carbonation depth	Measured saturation level	carbonation depth level (%)			carbonation depth		
	(mm)	(%)	10^{-16} m^2	Ω.m	equations 11 + 15	equations 12 + 15	equations 11 + 15	equations 12 + 15
1	0.25	65	1.7	22	0.003	0	93.7	93.7
2	18.9	58.9	1.07	407	24.4	24.38	61.7	61.8
3	33.1	60.6	0.101	504	48.8	38.9	64.9	86

664

 Table 7: Comparison between measured and calculated values of carbonation depth and saturation level.

In Section 1, Torrent permeability measurements were carried out with a saturation level that 665 was considered high for such a measuring technique. As shown in Figure 14, CoV increased 666 with the degree of saturation. This can explain the high calculated degree of saturation 667 668 obtained in Section 1 in both cases (Equations 11+15 and 12+15). As regards the carbonated sections, the calculated values for carbonation depth were overestimated by non-destructive 669 670 testing, which provides a safety margin regarding the service life assessment of concrete 671 structures but can be expensive for owners because repair will take place earlier. The combination of Equations 12 and 15 is more consistent with the measured data especially as 672 regards carbonation depth. This combination is recommended for carbonation depth 673 674 calculations, while the combination of Equations 11 and 15 is advised for calculating the saturation level after testing concrete structures using the Wenner resistivimeter and the 675 Torrent permeameter. 676

677

678 5 <u>Conclusion</u>

679 In this study, resistivity and permeability measurements were performed on carbonated slabs 680 conditioned at different saturation degrees. The results confirm that resistivity and 681 permeability are both very sensitive to concrete moisture. They also clearly show that

carbonation affects resistivity and permeability. The higher the carbonated depth is, the higher 682 683 is the resistivity and the lower is the permeability. This can be related to the reaction of carbonation, which decreases porosity. Resistivity cannot be assessed for water saturation 684 degrees of less than 40% because, at such low moisture content, the continuity of the 685 interstitial solution vanishes and the electrical current cannot circulate. On the other hand, 686 permeability cannot be measured for saturation degrees higher than 83% due to the 687 688 obstruction of pores by the interstitial solution, which prevents gas penetration. An analysis of the test results has shown a coefficient of variation of 20% for resistivity and less than 30% 689 for permeability, which increases with the increase of saturation degree whatever the 690 carbonated depth. 691

Two empirical laws have been proposed here to model resistivity measurements versus carbonated depth and saturation degree. The first one is adapted from a linear law between resistivity and saturation degree to take the carbonated depth into account. For the second one, Archie's law is adapted to take carbonation into account. The coefficient of determination is better for the linear model, which is easier to determine. In the same way, a linear model between permeability coefficient and saturation degree is extended to take the carbonated depth into account with a good determination coefficient.

Models involving relative resistivity and relative permeability have also been proposed. For resistivity, both the linear and Archie's law are considered. For permeability modeling, a modified Van Genuchten law seems more efficient than the modified linear model. It would be interesting to check these models with concrete presenting other porosities.

Finally, the different models have been used to predict both saturation degree and carbonated depth on a wall made with the same concrete as the slabs. The results show that the models are able to distinguish different carbonated depths but the assessment precision needs to be improved. Additional research is required, in particular to take the presence of cracks into

707	consideration. These may due to shrinkage or to mechanical effects, for instance, which have
708	a significant influence on measurements. In order to improve the quality of the investigation,
709	a third technique could be proposed, such as ultrasonic surface waves, which are able to
710	investigate the cover concrete.
711	
712	
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