

# 1 Testing concrete E-modulus at very early ages through several techniques: an inter- 2 laboratory comparison

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## 28 29 ABSTRACT

30 The design of concrete structures is based on calculation rules, which often do not take into account the very early  
31 age behaviour of the material. However, during this period, structural concrete is subjected to strains due to the  
32 hydration process of cement. If these strains are restrained by concrete itself or surrounding boundaries, stresses  
33 start to build up that can lead to the formation of cracks. Among the parameters involved in the stress build up, the  
34 stiffness evolution is of major importance. This paper reports the use of eight different techniques aimed at stiffness  
35 evolution assessment, applied on the same concrete mix, in a round robin experimental test within three  
36 laboratories. The observations are compared after having expressed the results at the same equivalent age. Both  
37 the loading stress rate and amplitude are observed to have an effect of limited importance on the determination of  
38 the quasi-static elastic modulus, which might be explained by very short term creep. Ultrasonic measurements  
39 provide values of E-modulus that are higher than the values provided by the quasi static tests at the time of the  
40 concrete setting. Similar mechanisms associated to very short term creep could explain the difference between the  
41 quasi-static and high-frequency elastic modulus.

## 42 43 KEYWORDS

44 Early age behaviour of concrete; Young's modulus; Round robin test; High-frequency; Ultrasonic measurements.

## 45 46 1 INTRODUCTION

47 Concrete is a composite material that undergoes major mechanical properties changes during its curing process.  
48 Thus for a good structural design, it becomes extremely important to know the concrete structural behaviour from  
49 the beginning of the hardening process. However, design rules are not yet well adapted to the real behaviour of  
50 concrete for the period between the setting time, or  $t_0$ , and approximately one day. Before  $t_0$ , good practices for  
51 making, casting and curing of concrete allow avoiding cracking of fresh concrete [1, 2]. After  $t_0$ , physico-chemical  
52 strains can develop. Upon restraint (i.e. by rebars, formworks, already cast concrete), these volume changes can  
53 be the cause for surface or traversing cracks. Obviously, permeability, service life and aesthetic of structures are  
54 affected by these cracks. A lack of attention to this early age period at the stages of design, experimental assessment  
55 or in practice can lead to major defects. For example, Aïtcin *et al.* [3] describes a value of “forgotten” strain  
56 between  $t_0$  and one day in the case of an autogenous shrinkage measurement on a high performance concrete.  
57 Indeed, they report that the autogenous shrinkage between  $t_0$  and 1 day is equal to  $250 \times 10^{-6}$  m/m whereas its  
58 development between 1 and 3 days solely amounts to  $50 \times 10^{-6}$  m/m. For a concrete, this difference can explain the  
appearance of cracks or not. Another important issue is the experimental determination of the setting time of

59 concrete. This point was already been addressed in the work of Weiss [4] and Sant *et al.* [5]. In these articles, the  
60 opportunity to use a parameter or another to monitor the concrete setting (thermal, chemical, electrical, mechanical  
61 parameters) is discussed.

62 In this paper, similar or complementary methods are compared within three laboratories working on the same  
63 concrete. As stresses are induced by restrained volume changes, the E-modulus evolution of concrete since the  
64 earliest age is of major importance, not only for the determination of the setting time but also for modelling and  
65 numerical computation purposes. That is why three laboratories involved in the work reported herein, researches  
66 have been focused on experimental methods leading to a better assessment of mechanical properties. This is a  
67 timely and opportune research work, as no recent inter-technique and inter-laboratory comparisons have been  
68 found in the literature except the preliminary work done by the authors in [6], in concern to concrete E-modulus  
69 at early ages. Eight different techniques have been implemented in the three laboratories. They can, a priori, be  
70 sorted in three classes, according to their respective ranges of loading rates. There are four techniques of quasi-  
71 static loadings (**QS<sub>0</sub>**: classical extensometry (all three labs); **QS<sub>1</sub>**: automated loading with BTJASPE (IFSTTAR),  
72 **QS<sub>2</sub>**: automated loading using QS<sub>1</sub> protocol (IFSTTAR), **QS<sub>3</sub>**: automated loading with a TSTM (ULB)); one  
73 technique of resonant frequency loadings (**RF**: EMM-ARM testing technique (UMinho)) and three techniques of  
74 high frequency loadings (**HF<sub>1</sub>**: classical UPV measurements with FreshCon (ULB), **HF<sub>2</sub>**: classical UPV  
75 measurements with BTPULS (IFSTTAR), **HF<sub>3</sub>**: smart aggregates (ULB)). These different protocols were applied  
76 without necessarily controlling the temperature history of the specimens in the same way, often due to practical  
77 limitations associated to the test setups and the size of specimens themselves. That is why observations are  
78 compared after having expressed results at the same concrete maturity using the equivalent time method [7]. These  
79 techniques scan a wide variety of ways for monitoring the E-modulus of concrete since the earliest age. They are  
80 suitable for both industrial and research applications. In the following lines, the specimen preparation and the  
81 concrete characterization will be presented first. Then, each of the eight testing methods (**QS<sub>0</sub>**, **QS<sub>1</sub>**, **QS<sub>2</sub>**, **QS<sub>3</sub>**,  
82 **RF**, **HF<sub>1</sub>**, **HF<sub>2</sub>**, **HF<sub>3</sub>**) will be described into details. Finally, it will be followed by the analysis of the results  
83 organized in three parts: firstly, a comparison between the results obtained with the low frequency testing methods  
84 (**QS<sub>1</sub>**, **QS<sub>2</sub>**, **QS<sub>3</sub>**, **RF**) with a discussion on the effect of the stress amplitude and the stress-rate on the results  
85 obtained with the low frequency testing methods; secondly, a discussion on the results obtained with the high  
86 frequency testing methods only (**HF<sub>1</sub>**, **HF<sub>2</sub>**, **HF<sub>3</sub>**) and thirdly, a discussion on the relation between the results  
87 obtained with the low frequency testing methods and the high frequency testing methods.  
88

## 89 2 SPECIMEN PREPARATION AND MATERIAL CHARACTERIZATION

90 The same material is used in the three laboratories. The mixture proportions are given in Table 1. Fresh concrete  
91 was mixed mechanically or manually depending on the volume of the batch (from 2 litres to approx. 40 litres).  
92 Even though these differences can lead to scattered results, no significant effect was observed in the performed  
93 experiments on this concrete as presented in section 4.1.4.

94 The strength evolution was useful for quantifying the limits of the automatic loadings (tests presented in sections  
95 2.1.3 and 2.1.4) applied at early age in order to avoid any damage of the samples. The samples were cylinders (Ø  
96 110 mm x 220 mm) or cubes (100 mm). They were cured inside adhesive aluminium tape at 20 °C, and capped  
97 with sulphur mortar when necessary. Results are reported on Figure 1.

98 For the sake of these calculations, the compressive strength is modelled by the following model:  
99

$$f_{cm} = a \cdot e^{-\frac{c}{t}} - b \quad (1)$$

With:  $f_{cm}(t)$  expressed in MPa

$a = 38.04$  MPa,  $b = 1.67$ MPa,  $c = 24$  hours

$t$ : equivalent time since mixing (in hours)

100 The activation energy was determined through calorimetric testing. This parameter, associated to temperature  
101 recordings, allowed calculations, based on Arrhenius laws, of an equivalent age ( $t_{eq}$ ) [8, 9,10] for each of our  
102 results. These calculations were performed at a fixed reference temperature equal to 20 °C.

103 The setting period corresponds to a period between the time when a needle penetrates completely a sample of  
104 mortar or cement grout and the time when it does not penetrate anymore the sample [5, 11, 12]. In field  
105 applications, the final setting time has also been identified, as mentioned by Sant *et al.* [5], like the time when the  
106 bleeding water is reabsorbed which indicates the creation of vapour spaces in the matrix. As penetration tests do  
107 not apply specifically for concrete, the work of Robeyst *et al.* [13] compared ultrasonic testing to penetration tests  
108 performed on sieved concrete by removing the coarse aggregates. They used ultrasonic P-waves in transmission  
109 mode. The evolution of the ultrasonic pulse velocity shows an experimental S shape and the corresponding  
110 derivative function is calculated. The final setting was considered to occur when the value of this derivative

111 function decreases down to 80 % of the peak value. This last technique was applied in the present research work.  
112 It is worth underlining that the final setting corresponds to a time when a sample can be manipulated, but gently  
113 to avoid any rupture. Before this time, any manipulation leads to a rupture. It is also the time when the stiffness of  
114 the material begins to increase significantly. This final setting is, here, called  $t_0$  [14]. An estimation of this time is  
115 required for starting tests just after concrete setting.

116  
117 Measurements of the modulus of elasticity were performed in the 3 laboratories and stress rates were ranging from  
118 0.20 to 0.55 MPa/s for the three laboratories (see Table 2 and Table 3). Table 4 shows the results of the mean  
119 values of the quasi-static loading E modulus obtained by the three laboratories according to the reference test set  
120 up (QS<sub>0</sub>) presented in section 3.1.1.

121  
122 As loading protocols are very similar, these results are mixed in order to obtain a single description of the evolution  
123 of the E-modulus by these classical means as it is shown in Figure 2.

124 For the sake of the analysis of our results, the following mathematical model is proposed (Figure 2):  
125

$$E(t) = d - e \left( \frac{\tau}{t} \right)^f \quad (2)$$

Where  $E(t)$  is expressed in GPa,

$t$ : equivalent time since mixing (in hours),

$d = 41.57$  GPa,  $e = 191.04$  GPa,  $\tau = 1$  h and  $f = 0.72$ .

126 This mathematical model has been calibrated to fit the quasi-static loading E-modulus results obtained with the  
127 reference set up (QS<sub>0</sub>) and shown in Table 4. This model is used in Figures 2, 13, 14, 16, 17, 25 and 27 as a  
128 reference curve for comparison.

129 This equation was already used in [15] to model the growth of bacteria populations but it has been chosen only for  
130 its mathematical properties for the determination of the early age properties of cement based material.

131  
132 This function is accepted as the reference for the comparisons between the different methods and will thus be used  
133 recurrently in the following sections.

134  
135 This kind of test does not provide an accurate estimation of  $t_0$ ; as the first measurement could only be made 1h and  
136 40 minutes after a  $t_0$  determined by other means (UPV, QS<sub>1</sub> or RF) presented in section 3.1; 3.2 and 3.3.  
137  
138

### 139 3 DESCRIPTION OF THE METHODS

#### 140 3.1 Quasi-static loadings

##### 141 3.1.1 Reference tests (QS<sub>0</sub>)

142 In the three laboratories, the reference tests (QS<sub>0</sub>) were performed, after the concrete setting ( $t_0$ ), on cylinders at  
143 different ages. For each test, strains were measured by extensometers. For the three laboratories, the extensometers  
144 are almost similar: i.e. three transducers (LVDTs) measuring the relative displacement between two rings fixed to  
145 the specimen, as presented on Figure 3. Prior to the test, specimens were kept in a curing chamber at 20°C.  
146 However, the protocol of loading, size of the specimen and extensometers used for the E-modulus determination  
147 presented some differences among the three involved laboratories, as synthesized in Table 2 which follows the  
148 nomenclature of Figure 3. The selected testing ages associated to the results are given further in Table 4.  
149

150 At IFSTTAR, the testing specimens were cylinders with 110 mm in diameter and 220 mm in length. The first tests  
151 were performed as early as 7 hours after casting. Strains were measured by extensometers, with results comparable  
152 to those of strain gauges [16]. The protocol of loading consisted in applying four cycles between 5 and 30 % of  
153 the strength measured on other cylinders (with the same geometry) just before the test [17]. The quasi-static E-  
154 modulus is evaluated during the loading cycle. The loading rate was set to 0.50 MPa/s. The testing equipment  
155 included a hydraulic actuator with 500 kN of maximum load.

156 At ULB, similar cylinders and the same extensometer were used. 20% of the concrete strength at the time of the  
157 test was applied within 10 seconds, on four different samples. Corresponding stress rates were ranging from 0.20  
158 to 0.55 MPa/s, depending on the age of the sample. The quasi-static E-modulus is evaluated during the loading  
159 cycle. The testing equipment included a hydraulic actuator with 1000 kN of maximum load.

160 At the University of Minho, cylinder specimens (150 mm in diameter and 300 mm in length), have been tested  
161 under cyclic compression according to LNEC E397 [18]. The protocol of loading consisted in applying five cycles

162 between 0.80 MPa and 33 % of the ultimate strength at the age of testing. The quasi-static E-modulus is evaluated  
163 during the loading cycle. The loading rate was set to 0.30 MPa/s. The testing equipment included a hydraulic  
164 actuator with 2000 kN of maximum load.

### 165 3.1.2 Automated and quasi-static loading with BTJASPE (Béton Très Jeune Age Suivi de la Prise et du 166 module d'Elasticité) or $QS_1$

167 BTJASPE ( $QS_1$ ) is a recent device, developed at IFSTTAR [19, 20, 21,22]. It allows the automatic monitoring of  
168 the stiffness of a concrete cylinder remaining in its mould. Measurements start just after the concrete casting and  
169 continue up to a few days. The device is placed between the plates of an automatic testing machine. The  
170 temperature of the sample is kept at a constant value thanks to a circulation of water inside a double walled mould.  
171 The sample (Figure 4 #1) is 100 mm in diameter and 200 mm in length. The mould wall (#2), made of stainless  
172 steel, has a thickness of 1 mm. Its depth is 254 mm. Once the concrete has been placed, the loading is applied via  
173 a steel piston (#3) guided inside the upper part of the mould. Fixed to this piston, three arms (#4) support three  
174 LVDTs, placed at 120° around the mould. These transducers measure the mean longitudinal displacement. This  
175 measurement includes the length change of the sample and the length changes of the steel piston and of the base  
176 of the rig (#5).

177 An upper conical loading plate (#6) includes a system, composed of a moving tip whose position is measured by  
178 a LVDT sensor with a resolution of 0.65  $\mu\text{m}$ . This measurement indicates to the testing machine, during the  
179 approach, the relative position between the upper platen and the piston (#3). When the contact is detected, a  
180 loading, controlled thanks to the measurement of the mean longitudinal displacement, is triggered. This protocol  
181 is effective even if the concrete is still in its fresh state. Then, the compressive loading of the sample is ensured at  
182 a constant strain rate (5 $\mu\text{strain/s}$ ). When a relative strain equal to 100  $\mu\text{strains}$  is reached, the ramp is reversed in  
183 order to unload the sample. This strain value is chosen to avoid any damage of the sample in compression. This  
184 protocol allows starting the tests shortly after the concrete casting. A new loading cycle is triggered after a delay  
185 of about 15 to 30 minutes. The device is placed on a lower plate (#7). It is surrounded by a cylindrical housing  
186 where a circulation of water allows removing the heat coming from the piston of the testing machine.

187 The quasi-static E-modulus is evaluated during the loading cycle. The main measurements (load vs mean  
188 longitudinal displacement) allow the calculation of an experimental resulting stiffness of the sample in series with  
189 the bearings of the device (K). The separation of these two parts is based on a relationship (2<sup>nd</sup> degree polynomial)  
190 giving the E-modulus of the concrete in function of the experimental stiffness, K:

$$191 E = g.K^2 + h.K \text{ with } E \text{ in GPa, } K \text{ in kN/mm, } g=1.03 \cdot 10^{-5} \text{ kN}^{-1} \text{ and } h= 0.0227 \text{ mm}^{-1} \quad (3)$$

192  
193 The coefficients of this polynomial have been fitted on the results obtained by a finite element calculation  
194 performed during the design of the device [19]. During this calculation increasing values of the numerical E-  
195 modulus of concrete were used to obtain the corresponding stiffness. It is assumed that, in the experimentation,  
196 the concrete sample remains in its elastic zone so that the experimental stiffness and the numerical stiffness are  
197 equal.

198 It can be underlined that the effect of the reinforcement due to the presence of the stainless steel mould has been  
199 stated, experimentally, as negligible. Indeed, prior to the casting, a thin layer of grease is applied on the inner wall  
200 of the mould. The Poisson's effect does not lead to a radial displacement exceeding the thickness of this layer of  
201 grease.  
202

### 203 3.1.3 Automated quasi-static loading using $QS_1$ protocol ( $QS_2$ )

204 In order to check the accuracy of BTJASPE device and the degree of confinement in the stainless steel mould of  
205 the sample, tests using the same loading protocol were performed on concrete cylinders removed from their  
206 cardboard mould just after the concrete setting detected by UPV measurements. The sample, capped with a sulphur  
207 mortar, is equipped with three extensometers and placed between the upper and the lower platens used for  $QS_1$   
208 (Figure 5). A ball joint is placed between the upper face of the sample and the upper loading platen. In that case,  
209 the sample is not confined thus the results are not affected by any lateral effect excepted near the ends of the  
210 sample. These tests were used to validate the results obtained with  $QS_1$  [19]. This test used the same type of  
211 extensometer as those of  $QS_0$  and  $QS_2$ .

### 212 3.1.4 Automated quasi static loadings with a TSTM ( $QS_3$ )

213 Since 2006, a Temperature Stress Testing Machine (TSTM) has been under development in ULB-BATir for testing  
214 concrete since  $t_0$ , under free and restraint conditions [20, 23, 24]. The testing machine is a Walter+Bay LFMZ 400  
215 kN electromechanical testing setup. The machine is composed by a fixed steel head, a central unidimensional part  
216 and a moving end (Figure 6a). In the central part, where the measurements of the displacements are taken, the  
217 stress field is assumed to be homogenous (Figure 6b) and the section is equal to 100x 100 mm<sup>2</sup>. The displacements

218 are recorded by Foucault current's sensors (contact free sensors) having a resolution of 0.014  $\mu\text{m}$ . Before casting,  
219 a plastic sheet is placed in the mould to ensure sealed conditions. Moreover, the plastic sheet helps also to reduce,  
220 with the presence of Teflon, the friction between the sample and the mould. The machine is equipped with a double  
221 walled mould allowing a thermal regulation and, in particular, ensuring isothermal curing conditions. Temperature  
222 measurements took place in the central part of the specimen and in each head. The experiment was conducted in  
223 a climatic chamber with temperature of  $20\pm 1^\circ\text{C}$ .  
224

225 A new methodology was developed for the monitoring of the E-modulus. The test is controlled at a constant  
226 loading rate, thanks to the software DION<sup>®</sup> (Walter & Bai). The test begins shortly after  $t_0$ . For each cycle of  
227 loading, the moving end of the testing machine is controlled by the force sensor, up to 20% of the compressive  
228 strength at the age of testing. The sample is then unloaded until it reaches a null force. Recordings (displacements,  
229 force and temperature) are taken during the cycles and more specifically during each loading and unloading. These  
230 displacement measurements can then be directly used to compute the E-modulus. The quasi-static E-modulus is  
231 evaluated during the loading cycle. The duration between each loading was approximately 60 minutes. The relation  
232 between the stresses and the strains is quasi linear during the loading. In order to keep the strictly linear zone of  
233 the stress/strain curve, the E-modulus is calculated between 30 and 80% of the maximum load reached in each  
234 cycle.  
235

### 236 3.2 Resonant frequency method (RF)

237 The EMM-ARM testing technique (Elasticity Modulus Measurement through Ambient Response Method) has  
238 been initially proposed by Azenha *et al.* [25] for the continuous measurement of concrete stiffness since the instant  
239 of casting. It is called here RF (Resonant frequency). Since its initial proposal for concrete testing, it has been  
240 extended towards application to cement paste [26], cement stabilized soil [27] and structural adhesives [28]. A  
241 more recent work has demonstrated the in situ applicability of the method and introduced changes that allow re-  
242 usability of parts [29]. The application of the RF method, in the context of this research, was fully made at the  
243 UMinho laboratory.

244 The general principle of RF consists on continuously monitoring the first flexural resonant frequency of a  
245 composite beam with known geometry and support conditions, which contains the material to be tested since the  
246 fresh state. The evolution of resonant frequency can be directly and quantitatively correlated to the evolution of E-  
247 modulus of the tested material, without any kind of ambiguity of user dependency. The test procedure is fully  
248 automated and provides results in real time.

249 For the experiments envisaged in this research, three testing geometries have been adopted, both based on simply  
250 supported conditions for the test beam: (i) the cylinder shaped specimen inside an acrylic mould (RF<sub>A</sub>) (Figure 7);  
251 (ii) a similar cylindrical shape beam similar to the one presented in Figure 7 but with internal/external diameters  
252 of 84/90 mm and 900 mm of span, and made of PVC (RF<sub>P</sub>); (iii) the prismatic shaped specimen inside a reusable  
253 metallic mould (RF<sub>M</sub>) (Figure 8). Even though the experimental setups, and interpretation techniques have been  
254 thoroughly explained and discussed in previous publications [25, 27], some brief remarks are given here for the  
255 sake of self-sufficiency of this paper.

256 The acrylic mould (RF<sub>A</sub>) is 2000 mm long, with internal/external diameter of 92/100 mm, density  
257  $\rho = 1286.89 \text{ kg/m}^3$  and E-Modulus of  $E_{\text{acrylic}} = 3.30 \text{ GPa}$ . The PVC mould (RF<sub>P</sub>) is 1000 mm long, with  
258 internal/external diameter of 84/90mm, density of  $1463.49 \text{ kg/m}^3$  and Young's Modulus of 4.50 GPa. Both moulds  
259 are crossed by horizontal rods at their supporting points (longitudinally spaced by 1800 mm and 900 mm for the  
260 beams RF<sub>A</sub> and RF<sub>P</sub>, respectively) and vertical rods that act as connectors of mould/specimen. Lids are utilized at  
261 the extremities: one fixed lid, placed before casting and a removable lid, aimed to be placed after casting. Due to  
262 the nature of the moulds, they require inclined/vertical position for casting. As soon as the removable lid is placed  
263 in position and excess air is purged, the composite beams are placed under simply supported conditions on the  
264 rods which are in turn placed on concrete blocks. At this stage, an accelerometer is attached to the mid span section  
265 of each of the beams (see Figure 7), and the test starts.  
266

267 The metallic beam was devised for reusability and increased robustness for in situ application [29]. It is made of  
268 a 'U-shaped' metallic alloy plate ( $\rho_{\text{plate}} = 7800 \text{ kg/m}^3$  and  $E_{\text{plate}} = 170 \text{ GPa}$ ), that has a total length of 2600 mm.  
269 The space for concrete placement spans through the central 2400 mm of the mould, with a cross section of  $150 \times$   
270  $150 \text{ mm}^2$ . The cross section shape of the mould was devised to assure that the centre of gravity of the metallic  
271 beam coincides with the centre of gravity of the tested concrete. Lids are placed near the extremity of the beam,  
272 right over the place where steel supports are placed to assure a free span of 2.4 m. Two aluminium stiffeners exist  
273 at 800 mm from the supports, in order to avoid significant transverse deformations of the metallic plates due to the  
274 weight of concrete. Connectors are also placed at 200 and 700 mm from the supports, according to the cross  
275 sectional scheme shown in Figure 8, in order to assure full bond conditions. Three accelerometers are placed on  
276 the bottom surface of the beam according to the positions shown in Figure 8. This variant geometry of EMM-

277 ARM results in easier casting operations, as the mould is kept in its final testing position since the instant of  
 278 casting, with the accelerometers placed in position even before casting, which was not possible with the  
 279 acrylic/PVC moulds. Also, the reusability of the mould provides a more sustainable testing protocol, from both  
 280 the economic and environmental points of view. The feasibility of the metallic mould for EMM-ARM has been  
 281 tested and reported in a previous work [29] with satisfactory results. All mould beams are tested in the scope of  
 282 this work to extend their mutual validation and make a deeper analysis of the relative adequacy of these methods  
 283 to test very early stiffness developments of concrete.

284  
 285 Accelerations of the monitored points have been measured for 28 days with sampling frequency of 200 Hz and  
 286 PCB accelerometers with 10 V/g sensitivity, 0.15 to 1000 Hz frequency range and 225 grams of mass. Packets of  
 287 60 seconds of data have been taken every 15 minutes with a 24 bit data logger (NI 9234). Data processing to obtain  
 288 the resonant frequency of the beams was made through the Welch procedure [30], involving windowing, averaging  
 289 and Fourier transforms, according to a process that has been described in detail in [25]. Based on the resonant  
 290 frequency of the composite beams, and the detailed knowledge of their geometries, mould stiffness and global  
 291 masses, the stiffness of the tested concrete was obtained through analytical derivations based on the dynamic  
 292 equations of motion of the corresponding composite beam (detailed explanations in [25]). This procedure was  
 293 implemented in LabVIEW [31], thus allowing continuous automatic information about the calculated E- modulus  
 294 in all tested beams. Temperature measurements took place in the central point of the cross section of each concrete  
 295 specimen. The experiment was conducted in a climatic chamber with temperature of  $19.2 \pm 0.5$  °C.

296 A final remark is given in regard to the applicability of the analytical derivation of the equations of motion used  
 297 for E modulus estimation: before the structural setting instant, such equations that rely on several assumptions  
 298 related to concrete as a solid material may not hold, and therefore the EMM-ARM E-modulus estimations before  
 299 setting time shown in Figure 16 are plotted as dashed lines. In fact, even though the reported evolutions seem quite  
 300 feasible, they may not be rigorous at this period.

### 302 3.3 Ultrasonic Pulse Velocity (UPV) measurements

#### 303 3.3.1 Classical UPV measurements: $HF_1$ and $HF_2$

304  
 305 On this concrete, two classical techniques of ultrasonic measurements were performed [32, 33, 34]. First, the  
 306 FreshCon ( $HF_1$ ) device (see Figure 9) developed at the University of Stuttgart was used due to its ability to detect  
 307 very early age signals. Ultrasonic pulses of 5  $\mu$ s at 800 V are sent through ultrasonic transducers (0.5 MHz resonant  
 308 frequency). The ultrasonic pulse velocity (UPV) can therefore be computed before setting. In addition, the UPV  
 309 was measured with a BTPULS (Béton Transmission Par Ultra Sons,  $HF_2$ ) device developed at IFSTTAR [20, 35].  
 310 This additional test, performed after the setting, was performed for comparative purposes between these two  
 311 techniques.

312  
 313 For  $HF_1$ , the sample thickness is 47 mm, which is more than twice the size of the largest aggregate (44 mm). Two  
 314 samples are cast inside two similar containers: one for P-waves measurements and one for S-waves measurements.  
 315 Samples are placed in a thermally regulated chamber and their temperatures are measured continuously during the  
 316 test. Detailed information about this method can be found in [34].

317 The P-waves ( $V_P$ ) and S-waves ( $V_S$ ) velocities can be used to compute the high frequency Poisson's ratio ( $\nu_{HF}$ ) and  
 318 E modulus ( $E_{HF}$ ) through equations 4 and 5, where  $\rho$  is the concrete density:

$$319 \nu_{HF} = \frac{1 - 2 \cdot \left( \frac{V_S^2}{V_P^2} \right)}{2 - 2 \cdot \left( \frac{V_S^2}{V_P^2} \right)} \quad (4)$$

$$320 E_{HF} = V_P^2 \cdot \rho \cdot \frac{(1 + \nu_{HF}) \cdot (1 - 2 \cdot \nu_{HF})}{(1 - \nu_{HF})} \quad (5)$$

321 These equations apply for a homogeneous and isotropic solid. When they are used during the hardening process  
 322 of a concrete, it is assumed that these conditions are fulfilled as soon as the age is higher than  $t_0$ . Before this time,  
 323 the UPV is highly affected by the presence of entrapped air inside the concrete [36]. Thus, even though very early  
 324 age recordings are feasible, the relevance of the results remains controversial.

325 For  $HF_2$  [37], the P-waves are emitted at the bottom and received at the top of a 110 mm diameter and 220 mm  
 326 height cylinder in its cardboard mould (Figure 10). Up to three samples can be tested simultaneously.

### 3.3.2 Smart aggregates ( $HF_3$ )

The main drawback of the  $HF_1$  and  $HF_2$  systems is the use of a fixed sized mould which strongly limits the possible testing conditions of the sample. In particular, it does not allow applying specific hygral and/or mechanical boundary conditions on the concrete sample. An alternative is to perform ultrasonic testing directly inside a concrete specimen of an arbitrary shape and size using embedded piezoelectric transducers. The use of embedded transducers, also called “Smart Aggregates” (SMAGs) has been initially proposed by Gu *et al.* [38] to monitor the strength of concrete using harmonic excitation, and later to monitor the state of damage using chirp excitation [39, 40]. Embedded transducers have also been used to monitor the elastic mechanical properties of concrete in [41]. At ULB-BATir, transducers based on a similar design were recently developed. Each  $HF_3$  consists in a flat piezoelectric patch which is wrapped in a waterproof coating and embedded in a small cube or cylinder made of mortar (Figure 11). One of the advantages of this technique is that no coupling agent is needed between the sensor and the concrete. The  $HF_3$ s made at ULB-BATir have been used for the monitoring of the P-wave velocity in early age concrete [42], and to monitor the crack growth in concrete and reinforced concrete in [43, 44]. The results for early age testing are used in this paper and compared to the results obtained with the other techniques presented. We shortly recall the testing procedure which is detailed in [42].

A prismatic mould, containing a pair of  $HF_3$ s with a distance  $d = 56$  mm is used (Figure 12). The  $HF_1$  system is used to excite the emitter with a pulse of 800 V and 2.5  $\mu$ s and record the wave at the receiver side. The testing procedure is therefore identical to the one described in Section 3.3.1, except for the fact that piezoelectric transducers of the mould are replaced by  $HF_3$ s directly embedded inside the concrete specimen.

## 4 RESULTS AND DISCUSSIONS

According to the results, this wide set of methods (quasi-static, resonant frequency and ultrasonic) can, finally, be divided in two groups: low frequency testing ( $QS_0$ ,  $QS_1$ ,  $QS_2$ ,  $QS_3$  and RF) and high frequency testing ( $HF_1$ ,  $HF_2$  and  $HF_3$ ). The strain rates or loading rates, covered by these methods, are gathered in Table 3. It is worth underlining that stress amplitudes or strain amplitudes are also different from one technique to the other. Comparisons of the results obtained with these techniques are largely influenced by these parameters [45, 46].

### 4.1 Low frequency testing

#### 4.1.1 Automated quasi static loading results ( $QS_1$ and $QS_2$ )

Eight tests with cyclic loadings were performed: four with BTJASPE ( $QS_1$ ) and four validation tests on samples removed from the mould after the setting called  $QS_2$ . All these results are plotted in Figure 13a. For each cycle, the behaviour was sufficiently linear so that the modulus of elasticity could be calculated with a simple linear regression [20].

It can be observed (Figure 13a) that these data are fairly in good agreement, both between themselves and also when compared to classical measurements (whose stress rate was 0.50 MPa/s).

Just after  $t_0$ , from the time when the E-modulus starts to become different from a null value (6 hours) to the time when its evolution reaches a quasi-straight line (8 hours), the 4  $QS_1$  recordings are in a range of variation of  $\pm 1$  GPa (Figure 13b). They become more scattered after 8 hours. Considering the validation tests ( $QS_2$ ), as soon as the data are available, this range presents the same type of scattering. Despite this scattering, from one batch to the other, the global response of such a protocol is quite satisfying. Indeed, the series of eight tests led to a coefficient of variation of E-modulus not greater than 13 % over a period ranging from the setting of the concrete to 48 hours. If a single concrete setting time has to be defined, from  $QS_1$  results, a value between 6 and 8 hours can be adopted depending on a chosen definition. In our case, this definition could be the time when the data start to increase at 6 hours. In real time this point is ambiguous. Indeed, one cannot decide if this value is included in the noise of the measurements. The beginning of the quasi linear part of the recordings, at 8 hours, is totally unambiguous but, may be, a little late. A definition of a point situated between the two times could suit our problem. Indeed, such a point is clearly outside the noise and it is very close to the intersection of the extrapolated linear part with the x axis. This extrapolation could be used even if no data are available before this point.

#### 4.1.2 TSTM results ( $QS_3$ )

Three samples have been cyclically loaded according to the protocol described in Section 3.1.4. In this protocol, 20 % of the compressive strength is reached in 10 seconds. This means that a constant stress rate is applied at each cycle but, as the strength is evolving continuously, this stress rate is increasing with the age of the sample. The range covered goes from 0.002 to 0.50 MPa/s over the first four days. On this period, and based on the results reported in Figure 14, a very good agreement, between the E-modulus evolutions for one sample in the  $QS_3$  device

382 and the model of the classical tests, can be observed, despite the fact that stress rates are not strictly similar. As a  
383 consequence, it can be inferred that the influence of the stress rate on the evolution of the E-modulus is low as it  
384 was observed for  $QS_1$  results.  
385

#### 386 4.1.3 EMM-ARM results (RF)

387 The resonant frequency evolutions for the three composite beams (concrete-filled moulds), resulting from  
388 processing the recordings of the accelerometers are shown in Figure 15a. All the data of EMM-ARM (RF) is  
389 plotted according to the equivalent age, normalized to 20 °C. By observation of Figure 15a, it is clear that all  
390 composite beams endured a significant frequency shift ranging between 27.49 Hz in the beam  $RF_M$  and 109.12 Hz  
391 in the beam  $RF_P$ . However, the differences in the resonant frequency shift in the beams reveal that the three  
392 experimental setups have quite different sensitivities. To better illustrate such added sensitivity, parametric  
393 analyses with the equations of motion of both composite beams were made, as to infer the changes in resonant  
394 frequency ( $dF$ ) that occur per each GPa of increase in E-modulus of the tested concrete ( $dE$ ), along the process of  
395 hardening of concrete. The results of such parametric analyses for all test setups is shown in Figure 15b, where  
396 three main conclusions can be drawn: (i) the sensitivity of all experimental setups decreases with the increase in  
397 concrete E-modulus; (ii) the frequency sensitivity of the PVC mould ( $RF_P$ ) setup to changes in concrete E-modulus  
398 is markedly during the all hardening process; (iii) the metallic beam ( $RF_M$ ) is the one that has the lower sensitivity  
399 to changes in the E-modulus of the material inside the mould despite being almost constant. This point indicates  
400 that the results should be more accurate in the beam  $RF_P$  when compared to the other beams.

401 The overall results of E-modulus measured by the three EMM-ARM composite beams and the reference curve of  
402 classical tests are shown in Figure 16. It is evident that the coherence of the four methods is very good at most  
403 ages of testing, once more confirming the feasibility and robustness of RF. It is however remarked that the E-  
404 modulus obtained from the metallic mould test is always slightly higher than the E-modulus obtained from the  
405 other beams (acrylic and PVC). This may be partially explained by some uncertainties in the geometry of the  
406 metallic beam due to its high local deformability (1 mm thick plates) that resulted in some local warping due to  
407 previous uses of the mould. A slight under-estimation of the final E-modulus value of RF methods in regard to the  
408 model can also be observed in Figure 16.

#### 409 4.1.4 Comparison between low frequency testing method

410 All low frequency testing results are shown in Figure 17. For these testing methods, the standard deviations are  
411 plotted in dashed lines. The beginning of the increase of the E-modulus seems to well correspond to the final  
412 setting as explained in section 4.1.2 whatever the testing method. Between the setting and 48 hours, the kinetic of  
413 the E-modulus looks very similar whatever the testing method whereas a limited scattering is observable between  
414 the results, especially at very early age. It appears that the difference of protocol of loading (strain rate, stress  
415 amplitude) and the type of testing method did not induce any strong effect on the kinetic and the amplitude of the  
416 E-modulus. All low frequency testing method have a very good correspondence with classical extensometry  
417 results.  
418

#### 419 4.1.5 Effect of the stress amplitude and stress-rate on low frequency testing method

420 The variation of E-modulus as measured by these different methods is attributed to their variable strain rates and  
421 amplitude of loading. In order to quantify these effects the study of these two parameters, two types of additional  
422 tests are performed with the TSTM device at ULB. First, the effect of loading amplitude is studied. A cyclic test  
423 started a few hours after the setting. Then, every hour, a load up to 5, 10, 20 or 40 % of the compressive strength  
424 is applied to the sample within 10 s. Secondly, a similar test was performed with constant load amplitude up to 20  
425 % of the compressive strength. In this second test, the loads were applied within 5, 10, 30 or 300 s.

426 The E-modulus values corresponding to the variable stress/strength ratio are displayed in figure 18 a. It is observed  
427 that with the increase of the applied stress, the estimated E-modulus decreases, and this effect progressively  
428 diminishes as the concrete hardens. On Figure 18 b, this amplitude effect is quantified. Each point is obtained by  
429 linear interpolation of the experimental results. For any given loading age, a non-linear relationship is observed  
430 between the loading amplitude and the E-modulus. This confirms that the stress-strain relationship is in fact non-  
431 linear, and can be described by a quadratic equation [46] such as equation 6, where  $m$  is the non-linear parameter.  
432 This expression is not based on any mechanical concept but it is the result of a mathematical fitting on basis of the  
433 experimental results. As it was shown in [46], the non-linear parameter  $m$  in the stress-strain relationship is  
434 dependent on the concrete mix design and the degree of hydration. As concrete hardens and the E-modulus  
435 increases, the slope of this stress-strain relationship decreases.  
436



$$\sigma = E \cdot \varepsilon - m \cdot \varepsilon^2 \quad (6)$$

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The dependency of E to the stress/strength ratio is described in equation 7, where  $\sigma$  is the applied stress,  $f_{cm}$  is the compressive strength,  $E_{ref}$  is the elastic modulus corresponding to a reference stress/strength ratio of 20 % applied in 10 s, and A is a time-dependent amplitude parameter describing the effect of a variation of stress on the apparent measurement of E.

$$E\left(t, \frac{\sigma}{f_{cm}}\right) = E_{ref}(t) + A(t) \cdot \left(\frac{\sigma(t)}{f_{cm}(t)} - 0.2\right) \quad (7)$$

444

Where  $E(t, \sigma/f_{cm})$ ,  $E_{ref}(t)$  and  $A(t)$  are expressed in GPa,  
 $t$ : equivalent time since mixing (in hours),  
 $\sigma(t), f_{cm}(t)$  are expressed in MPa.

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The evolution of parameters  $E_{ref}$  and A are shown in Figure 20 a. The evolution of A confirms that the decrease of E due to an increase of the loading amplitude is especially strong at very early age, and drops to low values during hardening. The expression of A is provided in equation 8.

$$A(t) = A_1 \cdot \left(\frac{t}{\tau}\right)^{A_2} \quad (8)$$

Where  $A_1 = -128$  GPa,  $A_2 = -1.015$  and  $\tau = 1$  h.

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A second test was performed at various loading durations ( $t_{loading}$ ) of 5, 10, 30 and 300 seconds for a constant load corresponding to 20 % of the compressive strength. The results presented in Figure 19 suggest that for longer loading duration, the E-modulus decreases. For any loading age, it is observed that the E-modulus increases as a power law of  $t_{loading}^{-1}$ . The best-fit parameters indicate that as the E-modulus increases, the relative impact of the loading duration decreases. In other words, as hydration advances, the E-modulus is less affected by variations in the loading stress rate. This observation is due to the visco-elastic behaviour of concrete, which viscous properties decrease with hydration.

This loading stress rate effect is taken into account in equation 9, where  $E_{ref}$  represents the E-modulus corresponding to a loading duration of 10 seconds up to a stress/strength ratio of 20 %,  $t_{loading}$  is the loading duration, and V is a time-dependent velocity parameter describing the effect of a variation of the loading stress rate on the apparent measurement of E.

$$E(t, t_{loading}) = E_{ref}(t) \cdot \left(\frac{10 \cdot \tau}{3600 \cdot t_{loading}}\right)^{V(t)} \quad (9)$$

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The evolution of parameters  $E_{ref}$  and V are shown in Figure 20 b. The evolution of V confirms that the decrease of E due to an increase of loading stress rate is especially strong at very early age, and drops to low values during hardening. The expression of V is provided in equation 10.

$$V(t) = \frac{V_1}{\ln\left(\frac{t}{\tau}\right) + V_2} \quad (10)$$

Where  $E(t, t_{loading})$  and  $E_{ref}(t)$  are expressed in GPa,  
 $V_1 = 0.03$ ,  $V_2 = -1.179$  and  $\tau = 1$  h.

$t$  is expressed in equivalent time since mixing (in hours) and  $t_{loading}$  is expressed in hours

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Ultimately, the combined effect of loading stress rate and amplitude can be taken into account by equation 11.

$$E\left(t, \frac{\sigma}{f_{cm}}, t_{loading}\right) = \left( E_{ref}(t) + A(t) \cdot \left( \frac{\sigma(t)}{f_{cm}(t)} - 0.2 \right) \right) \cdot \left( \frac{10 \cdot \tau}{t_{loading}} \right)^{V(t)} \quad (11)$$

474  
475 Quasi-static testing for the determination of E-modulus is generally performed for loading durations between 1  
476 and 300 seconds and that the loading amplitude is generally between 5 and 40 % of  $f_{cm}$ . Based on these parameters,  
477 two envelope curves can be computed, showing the upper and lower boundaries of the E-modulus based on the  
478 effect of loading stress rate and amplitude. The envelope curves are shown in Figure 21 and confronted to the  
479 experimental E-modulus results obtained by extensometry at ULB, IFSTTAR and U Minho (Figure 2). These  
480 experimental points, obtained for similar testing parameters, indicate that the error relative to the reproducibility  
481 of the test is of the same order of magnitude than the effect of loading stress rate or amplitude. As explained later  
482 on, by changing by several orders of magnitude the loading stress rate and/or amplitude (such as in the case of  
483 ultrasonic testing) these effects are much more visible.

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## 486 4.2 High frequency testing (HF<sub>1</sub>, HF<sub>2</sub>, HF<sub>3</sub>)

487 The results obtained on three batches with the FreshCon (HF<sub>1</sub>) for the transmitted P-waves and S-waves velocity  
488 are shown in Figure 22. The P-waves velocity increases rapidly during setting and stabilizes after the final setting  
489 time to a value slightly superior to 4000 m/s. However, the S-waves hardly propagate through fluids. It is only at  
490 the beginning of the hardening that the S-waves velocity increases. Then, it slowly reaches a value close to 2500  
491 m/s. These observations are in good agreement with the few results found in the literature [47, 48]. The results  
492 obtained on three batches with the BTPULS (HF<sub>2</sub>) for the P-waves velocity transmission are shown in Figure 23  
493 a.

494 The P-wave velocity results obtained, for the same concrete, with the SMAGs (HF<sub>3</sub>) and the BTPULS (HF<sub>2</sub>)  
495 systems are compared to the results obtained with the FreshCon (HF<sub>1</sub>). Results are plotted on Figure 23 b. In this  
496 figure, the average of three samples is shown for the HF<sub>1</sub> and the HF<sub>2</sub>.

497 HF<sub>1</sub> seems to give slightly different results, in the first part of the curve. However, this difference mainly occurs  
498 before  $t_0$ . More explanations about this observation are given in [49]. From 12 hours onward, the overall tendency  
499 is the same for all techniques, with similar values of P-waves velocity. Discrepancies before  $t_0$  can be due to  
500 different air content in the different moulds. Indeed, different techniques are used for placing the concrete in the  
501 moulds. HF<sub>3</sub>S are placed in a large steel mould. Its high weight (concrete + steel) makes the vibrating table less  
502 effective. On the other hand, the HF<sub>1</sub> samples are efficiently vibrated until no more air bubbles are trapped between  
503 the concrete and the sensors. Vibrating needles are used for HF<sub>2</sub>. Indeed, it is known that a strong dependency of  
504 the early age P-waves velocity to the air content of the mix exists. In aired mixes, the initial velocity is around 250  
505 m/s, whereas for de-aired mixes, initial values close to 1500 m/s can be observed [13, 50]. Ultimately, the  
506 coherence between the three experimental techniques and the good reproducibility of HF<sub>1</sub>, the distance of 47 mm  
507 between sensors is enough in order to provide representative measurements.

508

509 With the HF<sub>1</sub> system, P-waves and S-waves were simultaneously measured on the same concrete with dedicated  
510 HF<sub>1</sub> containers, which allows an accurate computation of the high frequency E-modulus ( $E_{dyn}$ ) and Poisson's ratio  
511 as shown on Figure 24.

512 In the case where no S-waves measurements is possible, taking a constant value of  $v_{dyn}$  in the equation for the  
513 computation of  $E_{dyn}$  (equation 5) leads to substantial errors, especially at early age. Indeed, the  $E_{dyn}$  value is  
514 governed by the S-waves velocity evolution. The approximation made by considering a constant  $v_{dyn}$  results in an  
515 overestimation of the early age high frequency modulus. However, in order to evaluate the relevance of HF<sub>3</sub> and  
516 HF<sub>2</sub> methods compared to usual UPV measurements, a constant  $v_{dyn}$  of 0.3 has been considered as no S-wave  
517 measurements have yet been performed with these techniques. In figure 25, the values for the HF<sub>1</sub> mean curve  
518 with a constant  $v_{dyn}$  equal to 0.3 and the HF<sub>3</sub> mean curve seem to stabilise after 30 hours, whereas the values of  
519 the HF<sub>1</sub> mean curve with consideration of the evolution of  $v_{dyn}$  follows the trend of the static E-modulus given by  
520 the model with increasing values after 30 hours.

521 Before the setting time, a scattering is observable between all the high frequency testing methods (see Figure 25).  
522 However, the results computed from the P-wave and S-wave velocity (HF<sub>1</sub>) exhibit the lowest amplitude because,  
523 before  $t_0$ , the values of  $v_{dyn}$  are much higher than 0.3. After  $t_0$ , the kinetic looks similar whatever the high frequency  
524 testing method is. On the contrary for this case, the results computed from the P-wave and S-wave velocity (HF<sub>1</sub>)  
525 exhibit the highest amplitude because, after  $t_0$ , the values of  $v_{dyn}$  tend to lower values than 0.3 (see Figure 24).  
526 Therefore, as observed in [47], the computation of  $E_{dyn}$  only from P-wave velocity should be considered as a  
527 qualitative indicator of the E-modulus development.

### 528 4.3 Relation between quasi-static or low-frequency and high frequency measurements

529 Since the 1970s, high frequency determination of the elastic modulus began to be performed on concrete due to  
530 obvious advantages of non-destructive and continuous aspects of this method [51, 52, 53]. Some authors found  
531 relationships linking the quasi-static or low-frequency elastic modulus to the high frequency elastic modulus  
532 (Table 5) of hardened concretes.

533 These empirical equations illustrate the fact that high frequency values are always higher than low-frequency  
534 values of elastic modulus, which is generally true for all viscoelastic materials. This observation was confirmed  
535 experimentally at early age by several authors as well as in this study [54, 55, 56]. However, in this research, these  
536 three relations do not seem to apply, especially at very early age. Two main drawbacks to these equations are  
537 advanced here. First, the lack of physical meaning to  $p$  and  $q$  in the general equation:  $E_{LF} = p \cdot E_{HF} - q$  which does  
538 not allow to take into account different concrete compositions (w/c, paste volume, cement type). Secondly, as  
539 shown in this study, the “low-frequency” or “high frequency” value can be dependent on the measuring method.  
540 Depending on the strain rate or loading amplitude for the low-frequency and high frequency measurements,  
541 different values of  $E_{HF}$  and  $E_{LF}$  can be obtained [46]. Therefore, the relationship between both values is ambiguous  
542 and certainly not intrinsic for a given concrete. In summary, the  $p$  and  $q$  parameters are dependent on the concrete  
543 composition, on the measurement method of low-frequency and high frequency modulus and, certainly, on the  
544 hydration degree. However, based on the hypothesis that the main parameters affecting the difference between  $E_{HF}$   
545 and  $E_{LF}$  are the loading stress rate and amplitude, such a linear relationship between both properties is consistent  
546 with the modelling approach of equation 11.

547 In the case of a given experimental protocol to determine  $E_{HF}$  and  $E_{LF}$  on a given concrete, an equation of the type  
548  $E_{LF} = p \cdot (E_{HF} - q)$  (with  $p > 1$  and  $q > 0$ ) seems appropriate to describe the behaviour of concrete throughout the  
549 hardening process. Indeed, at the initial setting time, the low-frequency modulus is close to 0. However, due to the  
550 initial increase of the ultrasonic pulse velocity before that time, the high frequency modulus has already reached a  
551 significant value. The  $q$  parameter represents this initial gap. Then, as concrete hardens,  $E_{LF}$  tends to increase  
552 whereas  $E_{HF}$  stabilizes more rapidly. Therefore, the gap between low-frequency and high frequency values tends  
553 to decrease throughout hydration, thus explaining the  $p$  parameter, which is representative of the different kinetics  
554 of the low-frequency and high frequency modulus evolution.

555 Figure 26 shows this relationship in this case by comparison of the HF<sub>1</sub> results and the classical extensometry  
556 results between the setting time and 66 hours. No linearity between  $E_{HF}$  and  $E_{LF}$  is directly observable here. But if  
557 only results after 18 hours ( $E_{LF} = 16$  GPa) are considered, a linear relationship is observable. Then, it appears that  
558 the relationship between  $E_{HF}$  and  $E_{LF}$  cannot be assumed as fully linear. Here, this assumption is only available  
559 between 18 and 66 hours. Further research should be carried out in order to be able to determine with precision  
560 the loading stress rate and amplitude of the high frequency measurements. Such measurement could improve the  
561 model presented in equation 11, and confirm the hypothesis that through the mechanism of very short term creep,  
562 the loading stress rate and amplitude are indeed the main parameters affecting the apparent value of the E-modulus.  
563 Finally Figure 27 presents the synthesis of all results. Only mean values of each testing method are shown here.  
564 As expected, clear differences appear between low-frequency and high frequency results. High frequency modulus  
565 values with consideration of the evolution of  $v_{HF}$  are always higher than low-frequency results. A faster evolution  
566 of high frequency results is also observable.

567

## 568 5 CONCLUSIONS

569 Different automatic techniques aimed at measuring changes in the stiffness of a concrete at early age were used in  
570 three different laboratories. They were grouped in low-frequency and high frequency methods.

571 The low-frequency methods gave responses similar to classical measurements. These classical measurements  
572 consist in performing the test after having removed the samples from their mould just after the setting time. Despite  
573 the fact that the samples are very brittle, the concrete begins to harden at the end of the working day thus, automatic  
574 methods are almost obligatory to study this initial phase of concrete behaviour. Two of the low-frequency methods  
575 are based on the use of laboratory testing machine (TSTM or QS<sub>3</sub>, BTJASPE or QS<sub>1</sub>) whereas the third method  
576 (EMM-ARM or RF) is well adapted for laboratory testing and in field. Their mutual performances are in good  
577 agreement. For such inter-laboratories tests, the protocols of mixing and loading should be improved, though.

578 Both the loading stress rate and amplitude are observed to have an effect on the determination of the low-frequency  
579 elastic modulus. This observation, which might be explained by very short term creep, is of limited importance  
580 when quasi-static measurements are performed, regarding the reproducibility of such tests. However, similar  
581 mechanisms could explain the difference between the low-frequency and high frequency elastic modulus, since  
582 both are measured at very different loading rate and amplitudes.

583 The ultrasonic measurements are also automatic methods and they are good candidates for the monitoring of the  
584 stiffness of the concrete at very early age. Two classical techniques (FreshCon or HF<sub>1</sub>, BTPULS or HF<sub>2</sub>) are  
585 compared to a newly developed technique (SMAGs or HF<sub>3</sub>). Ultrasonic measurements provide values of E-

586 modulus that are higher than the values provided by the quasi static or low frequency tests at the time of the  
587 concrete setting. This difference decreases as the concrete hardens. Their results show a clear effect of the loading  
588 rate on the E-modulus calculation compared to values obtained with quasi static tests. A correlation between the  
589 results of high frequency techniques and low frequency ones is not yet clearly accessible and models should be  
590 found to promote the use of ultrasonic techniques applied to the monitoring of the concrete stiffness at very early  
591 age.  
592 Further experimentations are needed to quantify accurately the effect of the strain or stress rates on the evolution  
593 of the elastic modulus in the low frequency and high frequency ranges.

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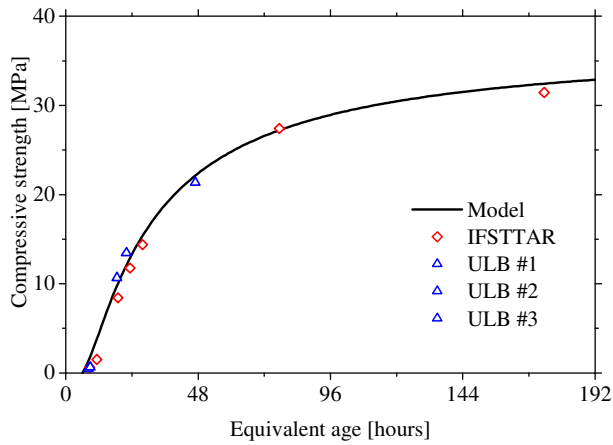
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603

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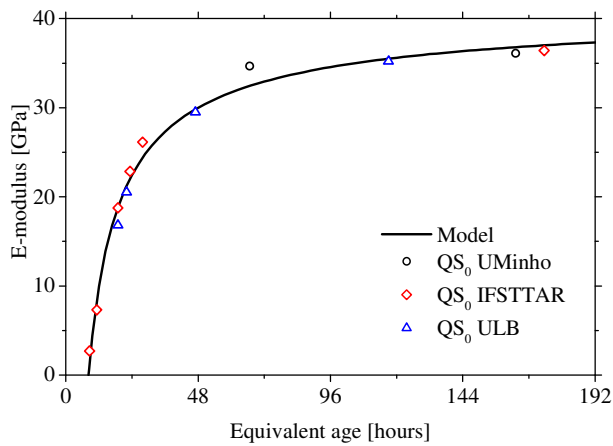
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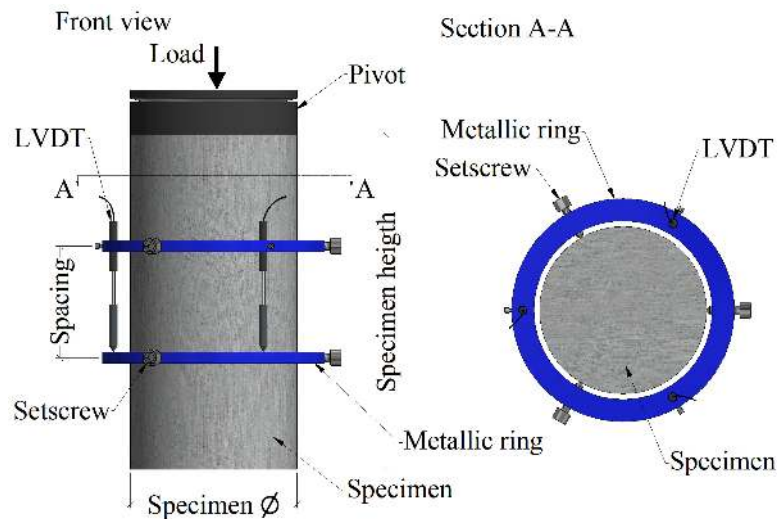
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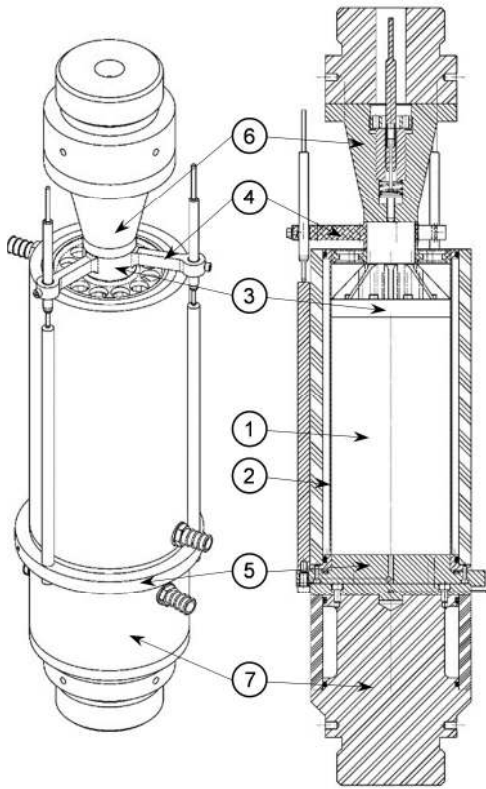
1  
2 **Figure 1: Compressive strength results obtained by the teams of IFSTTAR and ULB.**  
3



4  
5 **Figure 2: Evolution of the quasi-static E-modulus obtained with classical methods (QS<sub>0</sub>). A model for**  
6 **quasi-static E-modulus is calibrated on these data.**  
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8



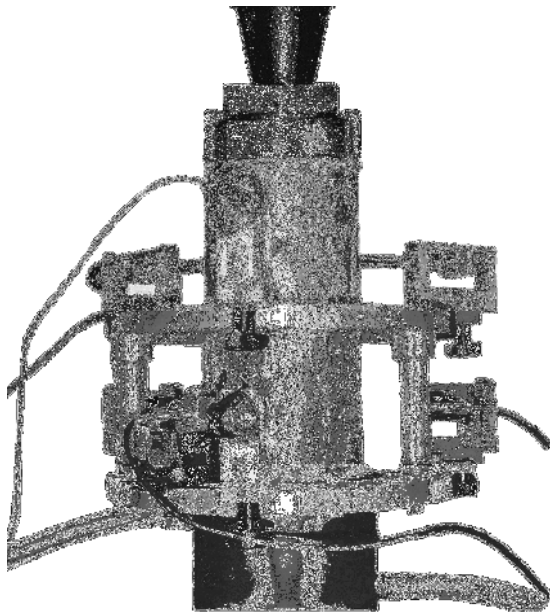
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10 **Figure 3: Classical test setup (QS<sub>0</sub>) with extensometers for quasi static loadings on concrete cylinder for**  
11 **quasi-static E-modulus monitoring.**  
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13

14 **Figure 4: Test setup (QS<sub>1</sub>) for cyclic loadings (BTJASPE) for quasi-static E-modulus monitoring.**

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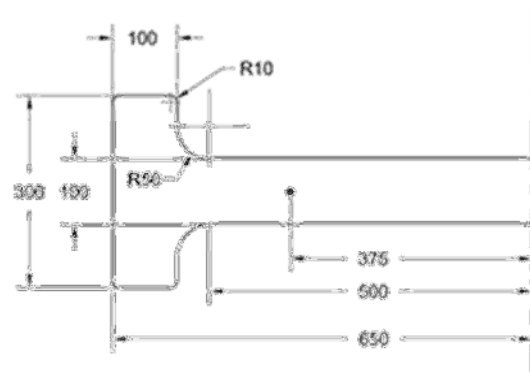
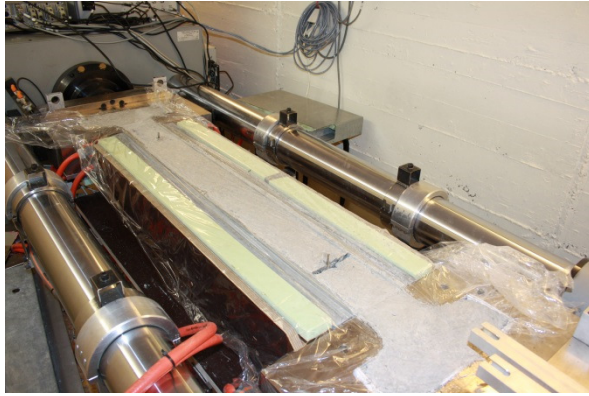
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17 **Figure 5: Validation test (QS<sub>2</sub>) for results obtained with BTJASPE (QS<sub>1</sub>) for quasi-static E-modulus**

18 **monitoring.**

19

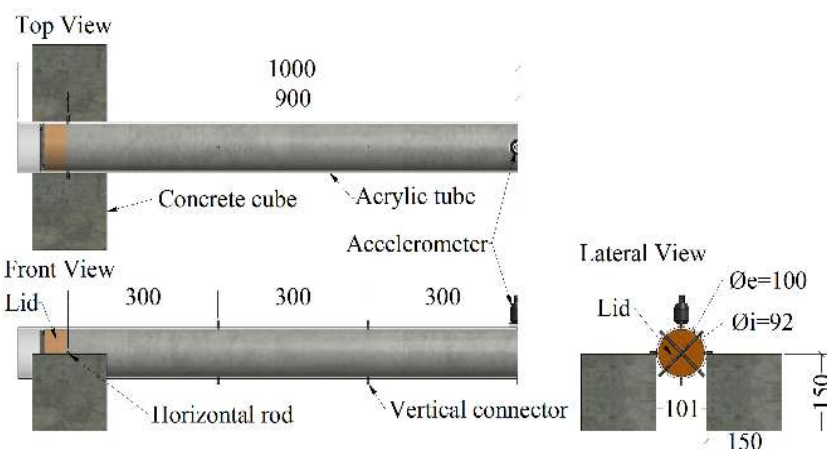




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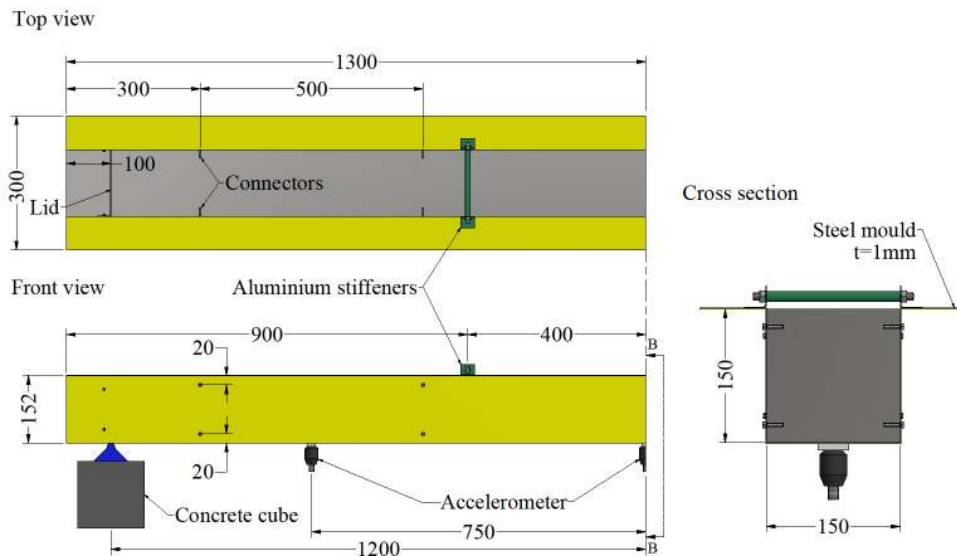
b)

20 **Figure 6: TSTM (QS<sub>3</sub>) equipment for cyclic loadings for quasi-static E-modulus monitoring: a) Photo; b)**  
 21 **Dimension of the specimen (units: mm).**  
 22



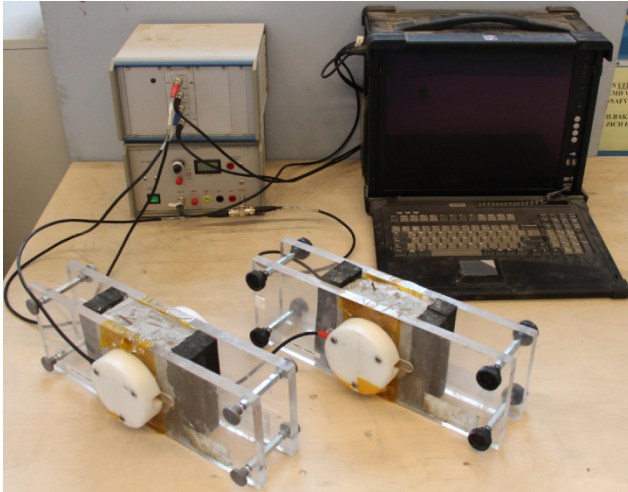
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24 **Figure 7: EMM-ARM testing with acrylic mould (RF<sub>A</sub>) for quasi-static E-modulus monitoring (units:**  
 25 **mm).**  
 26



27

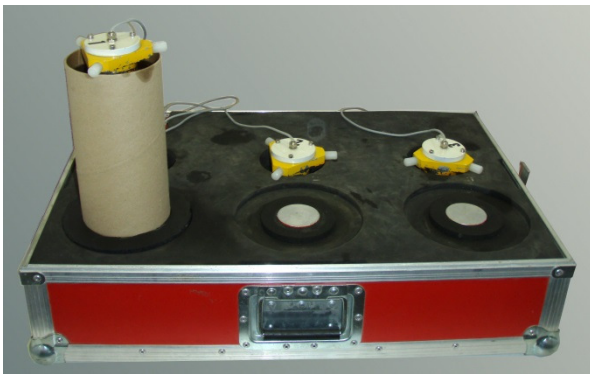
28 **Figure 8: EMM-ARM testing with metallic mould (RF<sub>M</sub>) for quasi-static E-modulus monitoring (units:**  
 29 **mm).**  
 30



31

32 **Figure 9: FreshCon System (HF<sub>1</sub>) for high frequency E-modulus monitoring.**

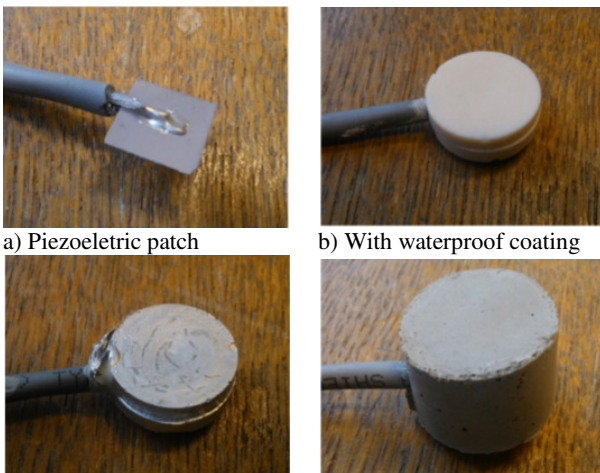
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35 **Figure 10: BTPULS system (HF<sub>2</sub>) for high frequency E-modulus monitoring.**

36



a) Piezoelectric patch

b) With waterproof coating

c) With conductive paint

d) Smart Aggregate

37 **Figure 11: SMAGs sensors (HF<sub>3</sub>) for high frequency E-modulus monitoring.**

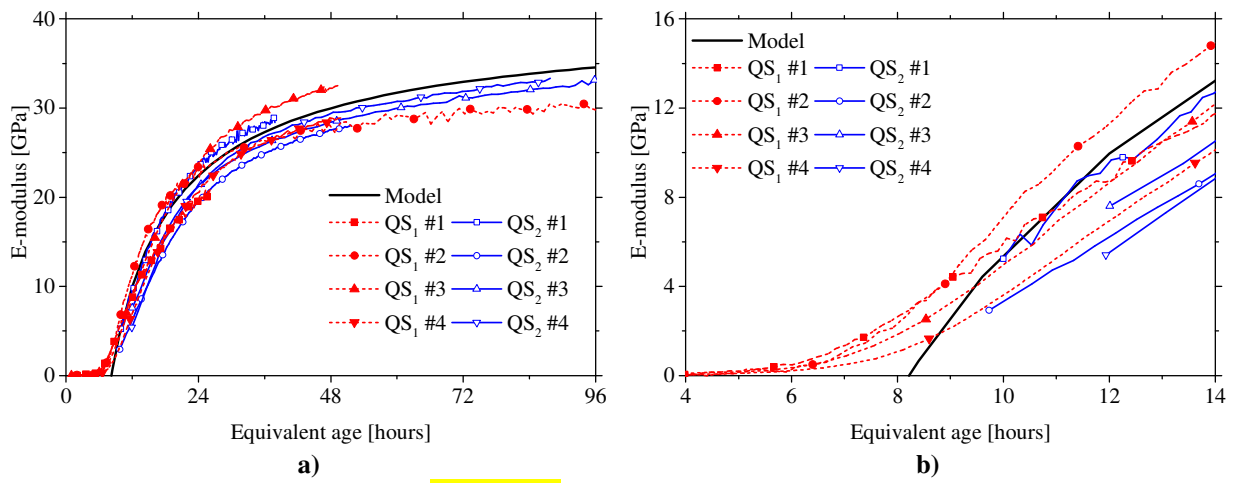
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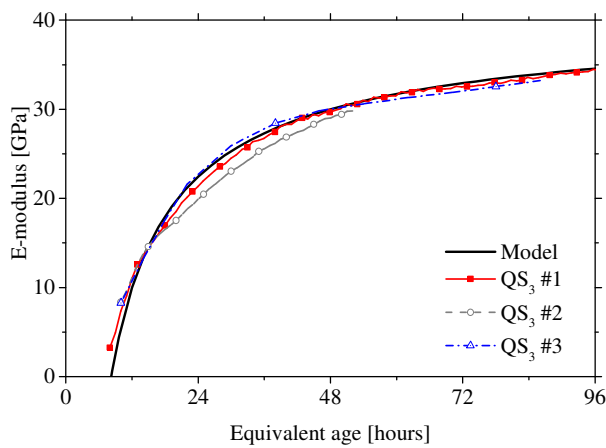
40 **Figure 12: Prismatic mould with SMAGs before casting the concrete.**

41



42 **Figure 13: a) Determination of the quasi-static E-modulus with BTJASPE ( $QS_1$ ) and cyclic loadings on**  
 43 **unconfined specimens ( $QS_2$ ) compared to calibrated model for quasi-static E-modulus; b) Detail of the**  
 44 **beginning of the recordings during the concrete setting.**

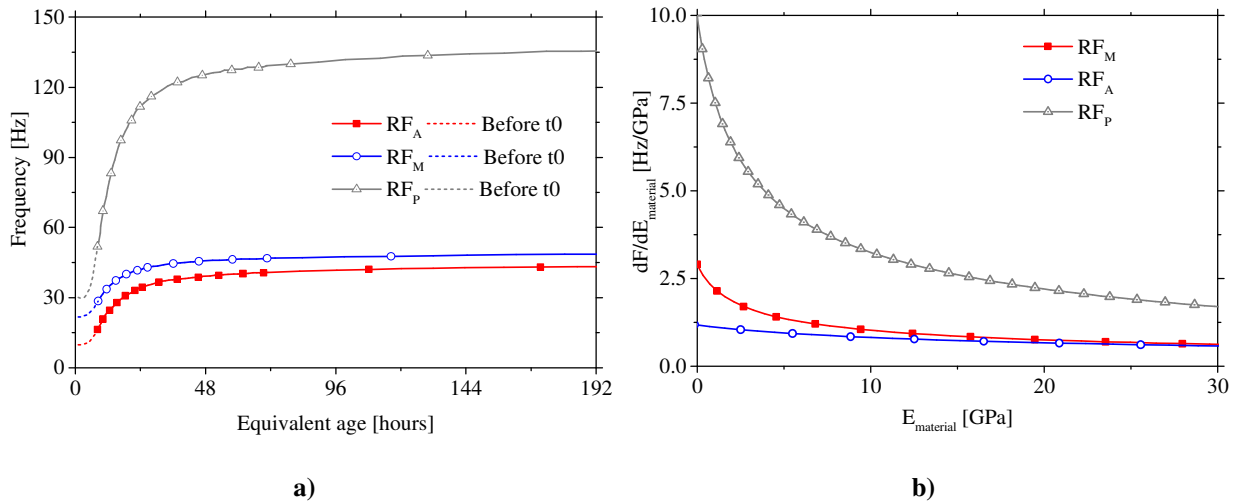
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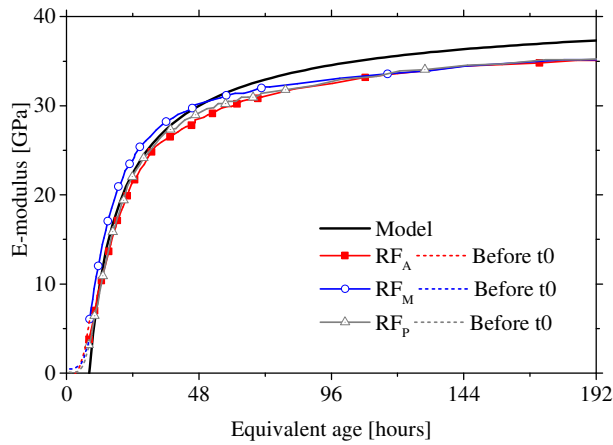
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47 **Figure 14: Quasi-static E-modulus with TSTM ( $QS_3$ ) compared to calibrated model for quasi-static E-**  
 48 **modulus.**

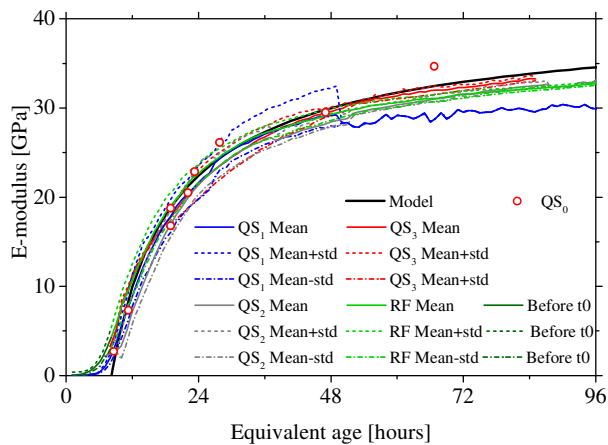
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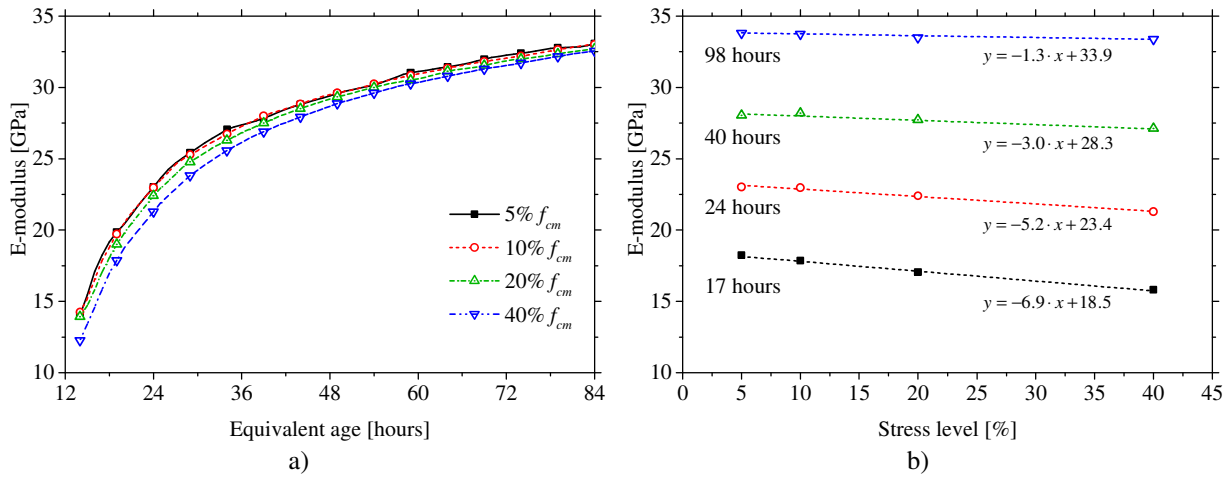
50 **Figure 15: a) Resonant frequency evolution for the acrylic, metallic and PVC EMM-ARM composite**  
 51 **beams ( $RF_A$ ,  $RF_M$ ,  $RF_P$ ); b) Frequency variation per E-modulus variation of concrete according to the**  
 52 **type of mould and the state of hardening.**  
 53



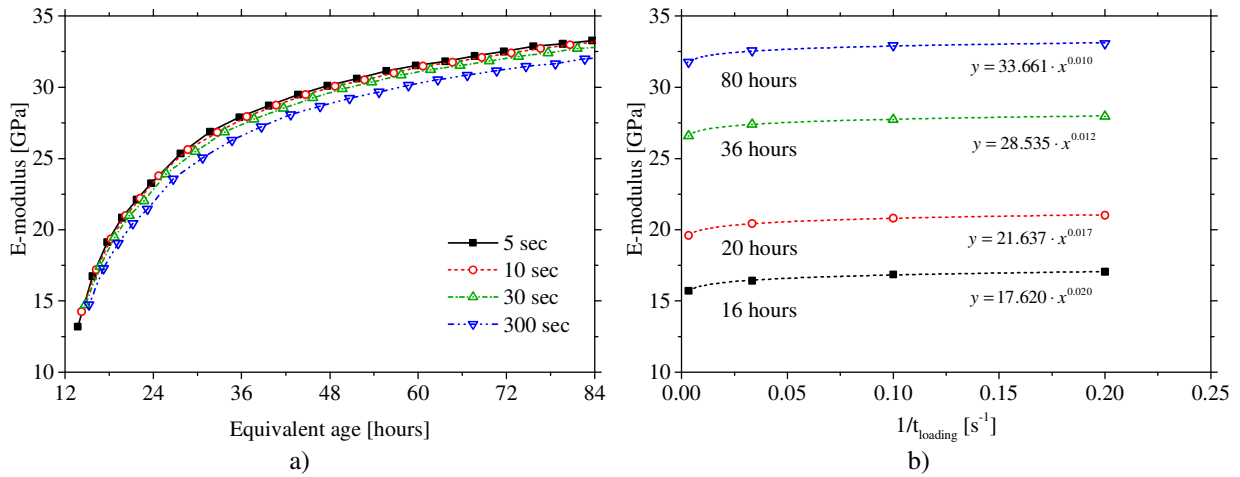
54  
 55 **Figure 16: Quasi-static E-modulus obtained through EMM-ARM (acrylic, metallic and PVC composite**  
 56 **beams  $RF_A$ ,  $RF_M$ ,  $RF_P$ ) compared to calibrated model for quasi-static E-modulus.**  
 57



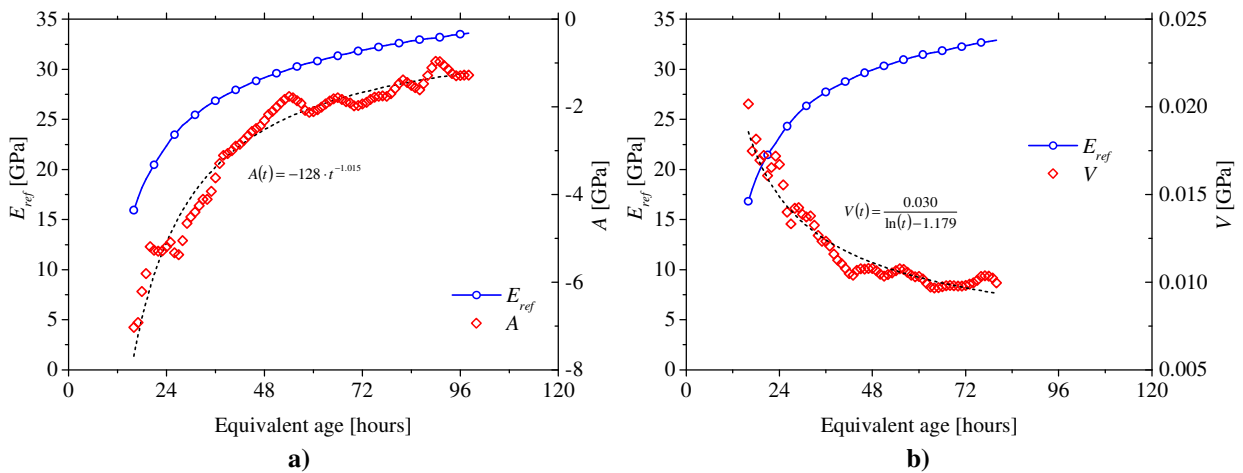
58  
 59 **Figure 17: Quasi-static E-modulus obtained with low frequency testing methods  $QS_0$ ,  $QS_1$ ,  $QS_2$ ,  $QS_3$ , RF**  
 60 **compared to calibrated model for quasi-static E-modulus.**  
 61



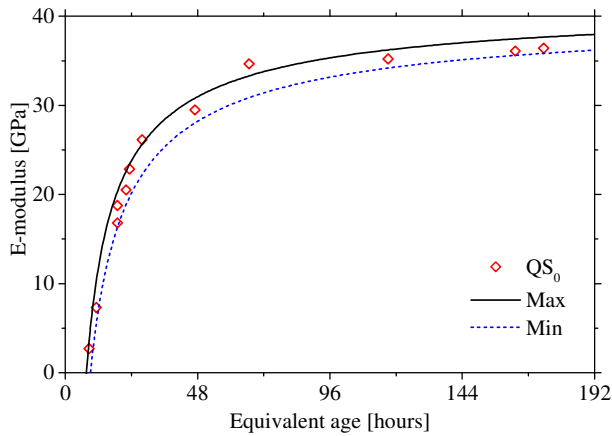
62 **Figure 18: a) Evolution of the E-Modulus for loading amplitudes of 5 – 10 – 20 – 40% of the compressive**  
 63 **strength; b) Evolution of the E-modulus according to the stress level for several ages of loading.**  
 64



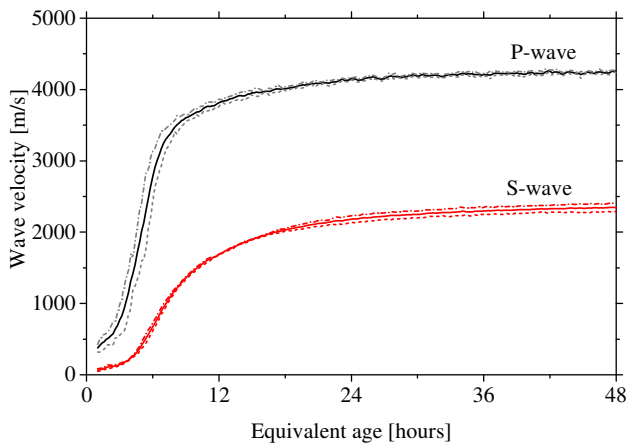
65 **Figure 19: a) Evolution of the E-Modulus for a stress applied in 5 – 10 – 30 or 300 seconds; b) Evolution of**  
 66 **the E-modulus according to the velocity of loading for several ages of loading.**  
 67



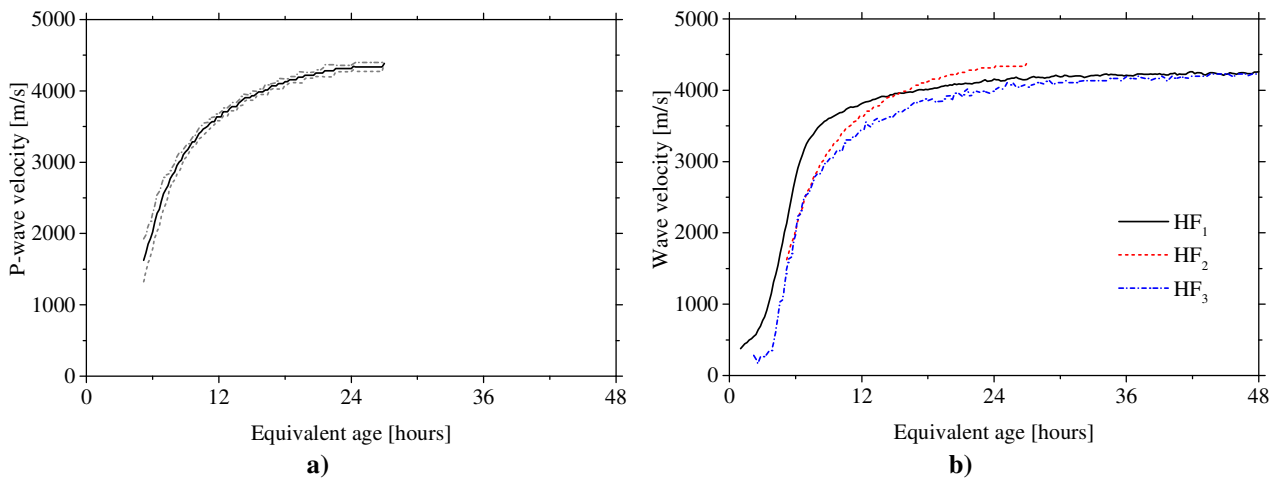
68 **Figure 20: a) Evolution of  $E_{ref}$  and amplitude parameter  $A$  determined with a single TSTM test (QS<sub>3</sub>); b)**  
 69 **evolution of  $E_{ref}$  and velocity parameter  $V$  determined with a single TSTM test (QS<sub>3</sub>).**  
 70



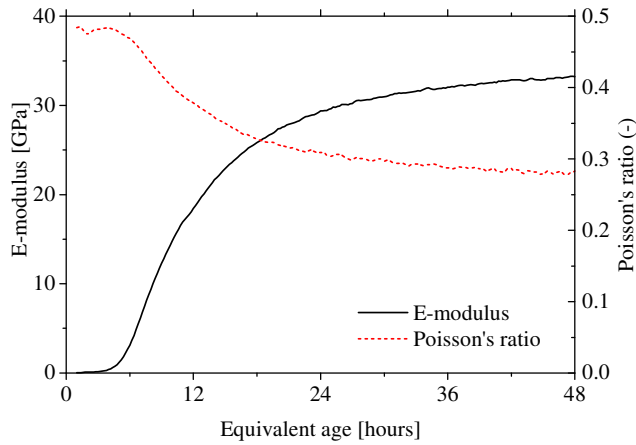
71  
 72 **Figure 21: Lower and upper boundaries for the effect of loading stress rate and amplitude on quasi-static**  
 73 **determination of E-modulus.**  
 74



75  
 76 **Figure 22: P-waves and S-waves velocity evolution through the FreshCon system (HF<sub>1</sub>).**  
 77



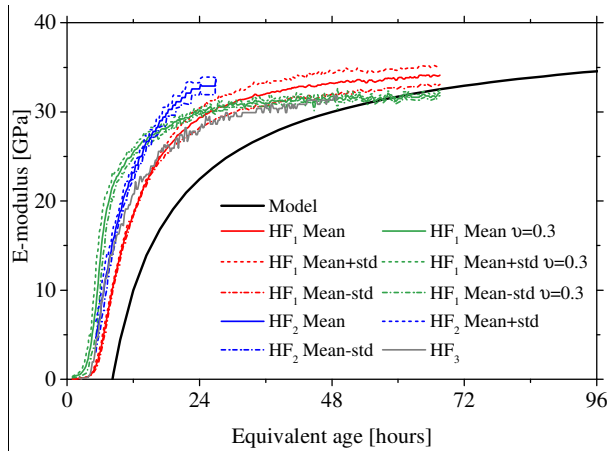
78 **Figure 23: a) Three sets of results obtained with BTPULS system (HF<sub>2</sub>); b) Comparison of the measured**  
 79 **P-wave velocities using FreshCon (HF<sub>1</sub>), BTPULS (HF<sub>2</sub>), and SMAGs (HF<sub>3</sub>).**  
 80



81

82 **Figure 24: Evolution of high frequency E modulus and Poisson ratio computed from the P-wave and S-**  
 83 **wave velocity with the FreshCon system (HF<sub>1</sub>).**

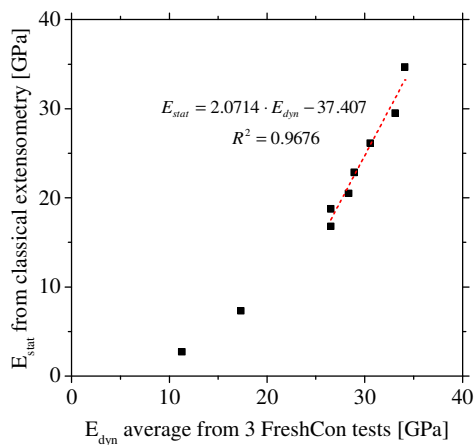
84



85

86 **Figure 25: Comparison of the high frequency E-modulus computed from the P-wave velocity only with**  
 87  **$v_{dyn} = 0.3$  (FreshCon (HF<sub>1</sub>), BTPULS (HF<sub>2</sub>), SMAG (HF<sub>3</sub>)) and from the P-wave and S-wave velocity (HF<sub>1</sub>)**  
 88 **with the quasi-static E-modulus calibrated model.**

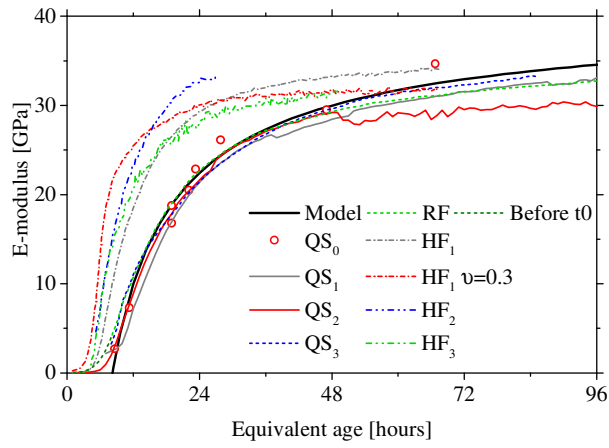
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90

91 **Figure 26: Relationship between high frequency E-modulus through HF<sub>1</sub> (from the P-wave and S-wave**  
 92 **velocity) and modelled quasi-static E-modulus through classical extensometry with the reference test set**  
 93 **up QS<sub>0</sub>.**

94



95

96 **Figure 27:** Comparison of E-modulus obtained with **quasi-static** (QS<sub>0</sub>, QS<sub>1</sub>, QS<sub>2</sub>, QS<sub>3</sub>, RF) and **high**  
 97 **frequency** (HF<sub>1</sub>, HF<sub>2</sub>, HF<sub>3</sub>) testing methods and with calibrated model for **quasi-static** E-modulus.  
 98



1 **Table 1: Mixture proportions of the concrete, w/c: 0.54.**

<i>Components / origin</i>	<i>Mass (kg /m<sup>3</sup>)</i>
CEM I 52.5 N PMES CP2 / Saint Vigor	340
Sand 0-4 / Bernières	739
Gravel 8-22 – Bernières	1072
Total water	184
Density of fresh concrete	2335

2

3 **Table 2: Classical test setup (QS<sub>0</sub>) in the three laboratories.**

	<i>Extensometer</i>	<i>Sample</i>		<i>Protocol of loading</i>	
		<i>Spacing (cm)</i>	<i>Height (cm)</i>	<i>Diameter (cm)</i>	<i>Loading (MPa)</i>
IFSTTAR	12	22	11	5% to 30% of $f_{cm}$	0.5
ULB	12	22	11	20 % of $f_{cm}$	0.2 to 0.55
U Minho	10	30	15	0.8 to 33% of $f_{cm}$	0.3

4

5

6 **Table 3: Loading rates for each device.**

<i>Device</i>	<i>Loading control</i> <i>(corresponding strain or stress rate)</i>	<i>Classification</i>
Classical tests (QS <sub>0</sub> )	0.2 - 0.55 MPa/s	Low frequency
BTJASPE (QS <sub>1</sub> )	$5 \times 10^{-6}$ /s (0.001 to 0.2 MPa/s)	
TSTM (QS <sub>3</sub> )	10 s from 0 to 0.2 $f_{cm}$ (0.002 to 0.5 MPa/s)	
EMM-ARM (RF)	9-45 Hz (0.1 to $1 \times 10^{-6}$ /s*)	
BTPULS (HF <sub>2</sub> )	10-100 kHz	High frequency
FreshCon (HF <sub>1</sub> )	10-100 kHz	
Smart aggregates (HF <sub>3</sub> )	10-100 kHz	

7 \*Strain rate computed by double integration of recorded accelerations and use of analytical derivations [56] (beam theory).

8

9

10 **Table 4: Classical extensometry tests (mean values) according to the reference set up QS<sub>0</sub>.**

<i>IFSTTAR<sup>(1)</sup></i>		<i>ULB<sup>(2)</sup></i>		<i>U Minho<sup>(3)</sup></i>	
<i>Time</i> <i>(hours)</i>	<i>E-modulus</i> <i>(GPa)</i>	<i>Time</i> <i>(hours)</i>	<i>E-modulus</i> <i>(GPa)</i>	<i>Time</i> <i>(hours)</i>	<i>E-modulus</i> <i>(GPa)</i>
8.64	2.72	18.96	16.8	66.72	34.66
11.28	7.32	22.08	20.5	163.2	36.1
18.96	18.76	47.04	29.5		
23.28	22.86	117.12	35.2		
27.84	26.14				
173.52	36.41				

<sup>(1)</sup>Mean values of 3 specimens

<sup>(2)</sup>Value of 1 specimen

<sup>(3)</sup>Mean values of 3 specimens

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12 **Table 5: Relations between high frequency modulus  $E_{HF}$  and quasi-static or low frequency modulus  $E_{LF}$ .**

<i>References</i>	<i>Model</i>
Shkolnik [46]	$E_{LF} = 0.83 \cdot E_{HF}$
Van Den Abeele, <i>et al.</i> [47]	$E_{LF} = 1.25 \cdot E_{HF} - 19$
Benmeddour, <i>et al.</i> [48]	$E_{LF} = 1.033 \cdot E_{HF} - 7.245$

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