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Comparison Between Different Experimental Techniques for Stiffness Monitoring of Cement Pastes

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Abstract

In 2009, a new methodology for the continuous monitoring of E-modulus of cement-based materials since casting was proposed, under the designation EMM-ARM (*E-modulus Measurement through Ambient Response Method*). This methodology is a variant to classic resonant frequency methods that allows continuous stiffness monitoring from the instant of casting. After the encouraging results obtained in the first applications of EMM-ARM to cement pastes, the present paper gives continuity to previous developments, through validations with additional experimental methodologies and extension to thermal activation testing. At first, a comparison is performed between the results of EMM-ARM and those obtained through: pulse velocity methods (both ultrasonic contact probes and bender-extender elements), penetration resistance (Vicat needle) and cyclic compression on cylindrical specimens. Afterwards, the possibility of studying the activation energy of the stiffness evolution on tests conducted at 20ºC and 40ºC is explored.

1. Introduction

It is nowadays known that the long-term structural performance and durability of cement-based structural elements is strongly influenced by curing conditions and early age events (thermal cracking, scaffolding removal, loadings). Hence, the capacity of characterizing the evolution of mechanical properties, such as E-modulus, since casting assumes crucial importance, especially taking into account the increasing demands that society poses on construction, with tight schedules and technically challenging projects. In order to fulfil these needs, several destructive and non-destructive methods have been proposed over the years for characterization of mechanical properties of cementitious materials since early ages (Azenha *et al.* 2010; Boulay *et al.* 2010; Reinhardt and Grosse 2004; Staquet *et al.* 2012; Zhu *et al.* 2011b; Jau and Yang 2010; Naik 2003).

This paper is focused on non-destructive approaches for characterization of the cement paste hardening, particularly on setting and stiffness monitoring. To evaluate these properties, several non-destructive methods can be found in the literature, namely: mechanical methods (Chamrová 2010; Maia *et al.* 2012b; Boulay *et al.* 2010; Staquet *et al.* 2012; Boulay *et al.* 2013b); wave propagation methods (Voigt 2005; Reinhardt and Grosse 2004; Song *et al.* 2008; Zhu and Kee 2010; Zhu *et al.* 2011a; Zhu *et al.* 2011b; Voigt *et al.* 2005; Kim *et al.* 2009); resonance methods (Kim *et al.* 2009, Malhotra and Sivasundaram 2003; Wang *et al.* 2010; Shin *et al.* 2008; Azenha *et al.* 2012); and methods based on dielectric properties (Beek 2000, Beek *et al.* 1999; Beek and Hilhorst 1999; Princigallo *et al.* 2003).

The Cyclic Compression (CC) method is the oldest and the most accepted method for characterizing the elastic modulus of concrete. It consists of subjecting a concrete specimen to centred compressive cyclic loads and evaluate the corresponding strains in the direction of load application (ISO, 2010). Even though there is not yet a standard or recommendation for E-modulus testing in cement pastes, the research works developed by Chamrová (2010) and Maia *et al.* (2012b) have demonstrated that such downscaling can be done, simply by using test specimens with adapted dimensions. Regardless of the tested material, classic approaches to CC testing have limitations in regard to the earliest possible testing age, as they require demoulding of the specimen prior to testing. To overcome this limitation, Boulay *et al.* (2010) developed a new method called BTJASPE, based on a specially devised mould and testing rig, which allows the application of compression cycles without demoulding the specimen. Another method, which also allows the evaluation of elasticity modulus through cyclic mechanical testing, is the *Temperature Stress Testing Machine* (TSTM) (Staquet *et al.* 2012; Boulay *et al.* 2013b). Even though this method was de-

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veloped for monitoring creep of concrete in tension under controlled temperature, it also allows the application of loading cycles along the curing period of the material, without removing the specimen from the mould. Although these recent methodologies have eliminated some of the disadvantages of the classic CC method, their relative complexity is higher both in terms of execution of the tests and in terms of the data processing that is required to obtain the stiffness of the tested material.

The methods based on wave propagation, often called acoustic methods, can be divided into two main groups: wave transmission methods, which include the ultrasound wave transmission methods (Dumoulin *et al.* 2012) and the *bender-extender elements* (Lings and Greening 2001; Zhu and Kee 2010; Silva *et al.* 2013); and wave reflection methods (Voigt 2005; Akkaya *et al*. 2003). These testing techniques are based on the principle that the velocity (V) of a wave in a given material varies according to its elastic properties and density. Within all the ultrasonic wave transmission methods, the one that is most commonly used in the context of cementitious materials is based on contact probes generating and receiving compression (P) waves (Zhu and Kee 2010). However, acoustic methods have relevant limitations for concrete testing as these are quite sensitive to local effects caused by aggregates (Neville 1995). Moreover, it has been recognized that the air content in cementitious materials can have drastic effects on the reduction of longitudinal ultrasonic wave velocities since it causes a severe attenuation due to wave reflection (Zhu *et al.* 2011a). Alternatively, Zhu and Kee (2010) have applied a methodology commonly used in soil characterisation to cement paste: *bender-extender elements* (BE) (Thomann and Hryciw 1990). Even though the BE methodology does not eliminate problems associated with local effects, this method is not influenced by the air bubbles since the measurements are performed with shear waves instead of compressional waves (Zhu *et al.* 2011b).

Another important method to mention is the one based on classic resonant frequency identification that allows the continuous monitoring of the mechanical properties of cementitious materials through the measurement of resonance of a small specimen (Malhotra and Srivasundaram 2003, ASTM 2002). In contrast with other non-destructive methods, this one allows the measurement of the global properties of a sample. However, in order to allow the specimen to vibrate freely, the sample needs to be demoulded prior to the test. Due to this fact, and also due to the need for applying an impact on the specimen, this methodology does not allow the continuous measurement since the fresh state.

Given the limitations of the existing methods for evaluating the modulus of elasticity of cementitious materials, Azenha *et al.* (2010) have recently proposed a variant to the above-mentioned resonant frequency method, named *E-Modulus Measurement through Ambient Response Method* (with the acronym: EMM-ARM), and allows the automatic and continuous evaluation of the elastic immediately after casting. The method has two fundamental differences compared to conventional resonant frequency methods: the specimen being tested is not demoulded, and the resonant frequency identification is made based on the vibration that naturally occurs in the surrounding environment (usually termed 'ambient vibrations'), which avoids the need for imposing a forced vibration to the sample (Azenha *et al.* 2010; Azenha 2009; Azenha *et al.* 2009; Azenha *et al.* 2012). Despite the successful results obtained through the studies involving EMM-ARM (Maia *et al.* 2012b; Maia *et al.* 2012c; Maia *et al.* 2012a; Azenha *et al.* 2012), there were in fact additional aspects pointed out for further studies. This methodology, in its variant for cement pastes, was compared with only some other methods such as cyclic compression and calorimetry tests (Azenha *et al.* 2012; Maia *et al.* 2011; Maia *et al.* 2012a; Maia *et al.* 2012b; Maia *et al.* 2012c). These studies have demonstrated that EMM-ARM is able to measure E-modulus values are similar to those obtained through cyclic compression tests at the same ages and same curing conditions. However, there is still the need for conducting a more profound and systematic study to analyse the performance of the EMM-ARM for characterising cement pastes at early ages, focusing on its relative behaviour to other methods. In fact, there are already some studies regarding the comprehensive comparison of different experimental methods to access the behaviour of cement-based materials at early ages (Bullard *et al*. 2006; Sant *et al*. 2009). However these works focus mainly on the detection of the setting time, rather than the evolution of stiffness itself. This paper aims to fulfil the identified gap through an extensive comparison between several experimental methodologies capable of quantifying the stiffness of cement pastes, such as: EMM-ARM, ultrasonic wave transmission, bender-extender elements and penetration resistance. An additional goal of this article is to assess the feasibility of extending EMM-ARM to the study of maturity and activation energy by conducting simultaneous testing under different temperature conditions (e.g. 20ºC and 40ºC).

2. Measurement methods in this study

2.1 EMM-ARM

The version of EMM-ARM devised for studying cement pastes was introduced by Azenha *et al.* (2012). By continuously monitoring the ambient-induced accelerations at the free end of a cantilevered composite tube containing the tested material, the evolution of the first resonant frequency is obtained, thus allowing the determination of the E-modulus of the cement paste. The fundamental component of this method is a hollow tubular beam made of acrylic, with outside/inside diameters of 20/16 mm. The composite beam consists of a 550 mm long

acrylic tube filled with fresh cement paste, with extremity caps made of polypropylene. The composite beam is then fixed in the horizontal position, operating as a cantilevered structural system with a span of 450 mm – see **Fig. 1**. The specimen is fixed using a metal clamping device with inner diameter matching the outer diameter of the beam. The metal clamping device is then rigidly connected to a rigid base to ensure complete fixation. A lightweight accelerometer (mass and sensitivity equal to 23.25 g and 1 V/g, respectively) is afterwards attached to the free end of the cantilevered beam.

The cantilevered beam is afterwards excited by ambient noise (e.g., wind, people walking nearby, vibrations originated by mechanical equipment, etc.) which can

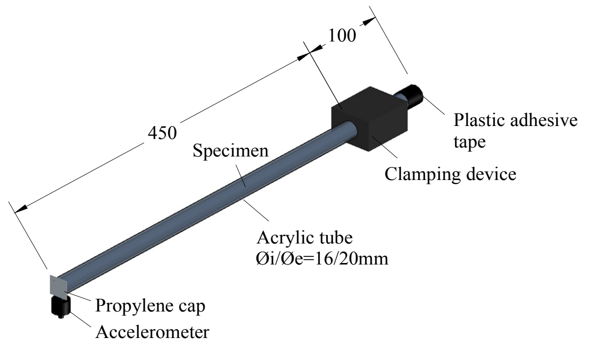


Fig. 1 Experimental setup for EMM-ARM testing of cement paste. Units [mm].

conceptually be assumed to have an average behaviour of white noise, i.e. a stochastic process with constant spectral intensity at all frequencies. It should be noted that, due to the slenderness of the beam, the vibrations that are merely caused by the surrounding environment are high enough to be registered by the accelerometer, enabling modal identification without using any external or forced excitation. However, in order to intensify the ambient vibration associated to air movement, and thus facilitate the process of automatic modal identification, fans were placed in the vicinity of EMM-ARM specimens. The experiment starts as soon as all components are correctly placed.

In regard to data processing, **Fig. 2** presents a scheme with a brief overall description of the operations involved for obtaining the evolution of the elastic modulus. The measured accelerations are acquired using a 24-bit data logger at a frequency of 200 Hz, and divided into groups of 120 seconds (**Fig. 2a**). From the recorded accelerograms, and using the Welch procedure (Welch 1967), the collected data is transformed into the frequency domain, thus resulting in the frequency spectra (**Fig. 2b**) – see further details in Azenha *et al.* (2012). Typically, for a better understanding of the vibration evolution over time, the various spectra are condensed into a single graph (a 3D surface, as shown in **Fig. 2c**). Then, the resonance frequencies of the first vibration mode are identified through the highest peak in each

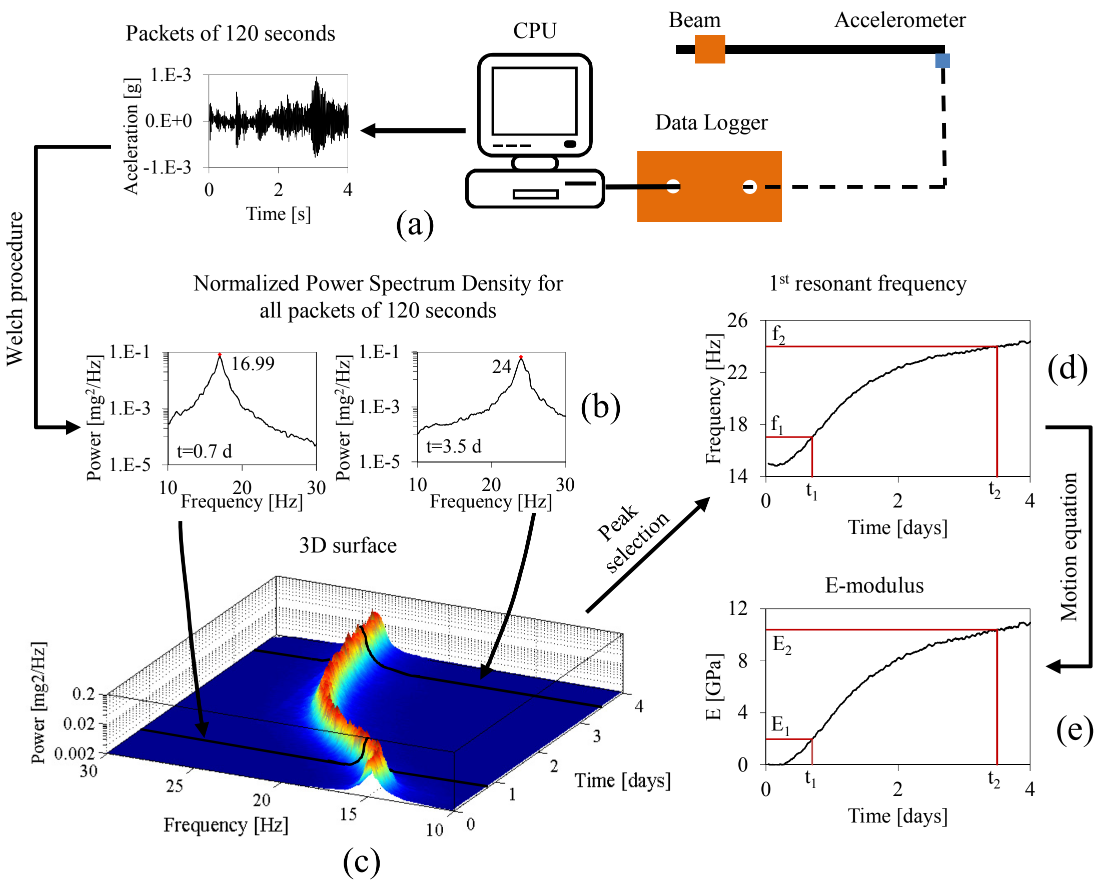


Fig. 2 Data processing scheme.

amplitude spectrum (**Fig. 2d**).

After determining the first flexural resonant frequency of the composite beam, it is possible to infer the stiffness of the tested material, using the vibration equation of a cantilevered structural system (**Fig. 2e**). The full derivation of the free vibration equation of a cantilevered beam is shown in Azenha *et al.* (2012). The final solution is the differential equation shown below:

$$a^3\left[\cosh(aL)\cos(aL)+1\right]+\frac{w^2 m_p}{EI}\left[\cos(aL)\sinh(aL)-\cosh(aL)\sin(aL)\right]=0$$
$$a=\sqrt[4]{\frac{w^2\cdot\bar{m}}{EI}} \qquad (1)$$

where E is the elasticity modulus (Pa), I is the homogenized second area moment of the composite cross-section (m^4), $\bar{m}$ is the uniformly distributed mass along the cantilever (kg/m), m_p is the concentrated mass located at the extremity of the cantilever that represents the masses of the accelerometer and lid (kg), L is the span of the cantilever (m), f is the first flexural resonant frequency (Hz), and the corresponding angular frequency is denoted by $w=2\pi f$. With the results of modal identification, all variables in Equation 1 are known except for *EI*, allowing to obtain this unknown mathematically. Bearing in mind the composite tubular section of known internal/external diameters (ϕ_i, ϕ_e), and the known E-modulus of the acrylic mould E_a, it is possible to compute the E-modulus of the tested cement paste E_c through equation 2:

$$EI=E_a\frac{\pi(\phi_e^4-\phi_i^4)}{64}+E_c\frac{\pi\phi_i^4}{64} \qquad (2)$$

By performing the above procedure for all ages of testing, the E-modulus *versus* time evolution curve can be obtained.

2.2 Pulse velocity methods

The idea of using the measurement of the propagation velocity of a wave to determine the initial and final setting time of cementitious materials was first described in the 1940's (Jones 1949). The basic principle of the wave transmission methods relies on the velocity of a wave propagated through a medium, which depends on its density and elastic properties. According to Chotard *et al.* (2001) and Smith *et al.* (2002), the velocity of the transmitted wave is sensitive to the formation of solid hydrates and therefore varies over time with the cement hydration and hardening process. In this method, a pulse is generated on one side of the sample, transmitted through the material and received on the opposite side of the sample. Both the generated and received signals are recorded in order to measure the time delay between the signals and thus obtain the wave travel time through the material. These methods can be applied using compressional (P) or shear (S) waves (Van Den Abeele *et al* 2009), generated by means of: an ultrasonic wave transmitter (Reinhardt and Grosse 2004), a smart-aggregate (embedded piezoelectric element) (Dumoulin *et al.* 2012), a bender or bender-extender element (Zhu *et al.* 2011b), or an external impact (Lin *et al.* 2010).

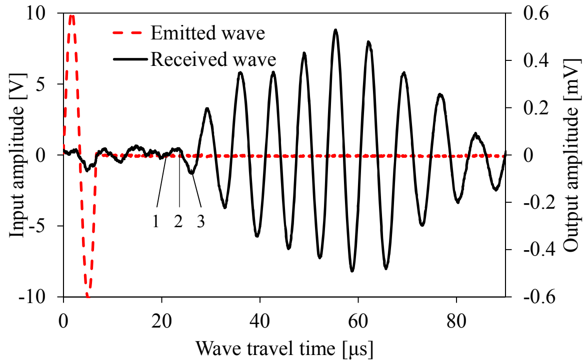


Fig. 3 Determination of the ultrasonic wave propagation time.

The exact determination of the wave propagation time is of crucial importance for the accuracy of this method. However, such determination is frequently difficult, time consuming and often inaccurate (Reinhardt and Grosse 2004). When the determination of the propagation time is manually performed, there is an uncertainty in regard to the exact arrival point of the ultrasonic wave, as depicted in **Fig. 3**, where both an emitted and a received P-wave are presented (Granja 2011). The referred figure clearly illustrates a particular case where it is possible to consider three different estimates of the arrival time of the wave in the received signal (points 1, 2 and 3). The propagation time may therefore vary depending on the experience and knowledge of the operator since the exact arrival point of the propagated wave is of subjective identification (Greening and Nash 2004; Viana da Fonseca *et al.* 2009). Similar difficulties are observed when automatic algorithms were used for the identification of the arrival point of the propagated wave, as described in the work of Kurz *et al.* (2005).

After obtaining the velocity of the wave, a correlation can be made with the elastic properties of the medium, through the following equations of wave propagation theory, which are applicable for homogeneous and isotropic media (e.g. Meyers and Chawla 2008):

$$V_P=\sqrt{\frac{(1-\vartheta_{dyn})\cdot E_{dyn}}{(1+\vartheta_{dyn})(1-2\vartheta_{dyn})\cdot\rho}} \qquad V_S=\sqrt{\frac{E_{dyn}}{(1+\vartheta_{dyn})\cdot 2\cdot\rho}} \qquad (3)$$

with V_P being the compressional (P) wave velocity (m/s), V_S he shear (S) wave velocity (m/s), ϑ_{dyn} the dynamic Poisson's ratio, E_{dyn} the dynamic elastic modulus (Pa) and ρ the density (kg/m^3).

2.2.1 Ultrasound Pulse Velocity (UPV)

In the present work, the method for monitoring the velocity of ultrasonic waves in cement pastes is based on

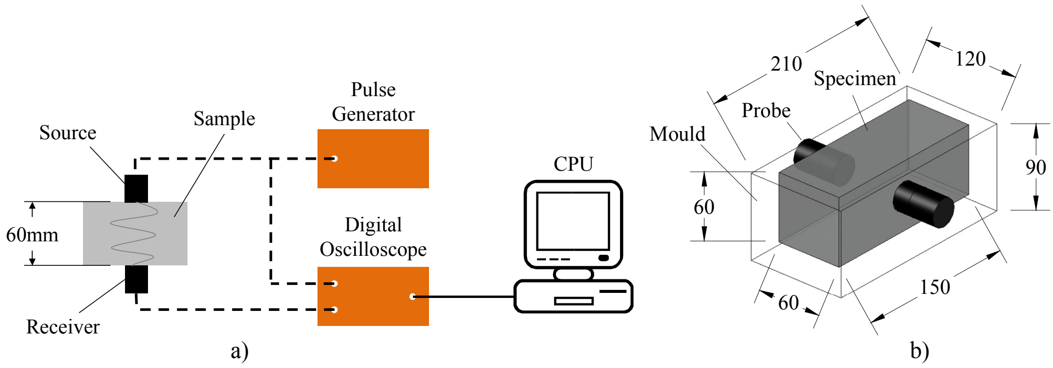


Fig. 4UPV method: a) experimental set-up; b) container. Units [mm].

the generation and transmission of a single-period sine wave through an ultrasonic probe transmitter, according to the test configuration shown in **Fig. 4a**. The setup consists of an ultrasonic transmitter placed on one side of the sample, as depicted in **Fig. 4b**, and a receiver positioned on the opposite side. The wave, defined in a function generator (model TTI - TG1010A) with 0.1 mHz resolution, an accuracy of <10 ppm and a range of 0.1 mHz to 10 MHz, is transmitted to the sample through a contact probe (P-wave probe with operating frequency of 150 kHz and diameter of 25mm), and received in the opposite side of the sample by another contact probe (identical to the source probe). The size of the probes is considered to be adequate for application to cement pastes since its diameter is greater than the largest expected heterogeneity. An oscilloscope with 16-bit resolution and a sensitivity of 10 mV/div to 20V/div (PicoScope 4424) performs the analog-to-digital conversion of both emitted and received waves and transmits the digitized waves to the computer for signal processing.

In order to avoid complications related to signal contamination with "cross-talk" parasite waves (Santamarina *et al.* 2001), the spacing between the probes must be longer than two periods (two wavelengths). Thus, considering that the expected wave velocity in a cement paste is around 3500 m/s (Boumiz *et al.* 1996), and that the probes have operating frequency of 150 kHz, the distance between the probes should be at least 47 mm. Another important requirement to take into account for the design of the mould was the fact that the tested material has very low stiffness in the early ages of testing, making it necessary to prevent wave transmission directly through the structure of the mould itself. By ensuring that the rigidity of the mould is very low, and warranting that the path between the probes is much longer through the wall than through the specimen, it is guaranteed that the received wave has propagated through the specimen. Based on these principles, a prismatic mould, made of extruded polystyrene, with dimensions 60×60×150 mm^3 was devised, as seen in **Fig. 4b**. To allow measurements immediately after mixing, the UPV probes were positioned in advance in the opposite faces of the cross-section of the mould. Preliminary tests allowed to confirm that the adopted solution performed well, without any noticeable negative effects on the quality of the received wave.

2.2.2 Bender-extender elements (BE)

Bender elements (BE) were first used to measure the velocity of shear waves (S-waves) in marine sediments (Shirley and Hampton 1978). A BE is a double layer transducer, composed of two thin piezoceramic plates rigidly attached to a metal core sheet with electrodes on its outer surface. The BE model proposed by Dyvik and Madshus (1985) (see **Fig. 5**) is still currently used, though variations have been introduced, namely to its size and to specific arrangements of pairs of transducers in a single probe (usually T-shaped). The controlled excitation of the two piezoelectric elements causes bending of the BE at a particular frequency, which in turn generates a shear wave in the material into which it is embedded.

Bender-extender elements were accidentally discovered when the typical BE connection scheme was incorrectly performed, as detailed by Lings and Greening (2001). The most interesting feature of this transducer is its reversibility of functions: simply by altering its electrical connections, the transducer can be used to propagate P and S waves.

In the present work, the adopted test configuration for this methodology is quite similar to the one described in the previous section for UPV, except for the use of BE probes instead of ultrasonic probes (**Fig. 6a**). In this study, T-shaped bender-extender elements made in the University of Western Australia (Brignoli *et al.* 1996), and are schematically depicted in **Fig. 6b**. These BE enable the measurement of P and S waves and operate in a wide frequency range, common for this type of probes.

In order to avoid errors in the identification of the wave propagation time, special attention must be given

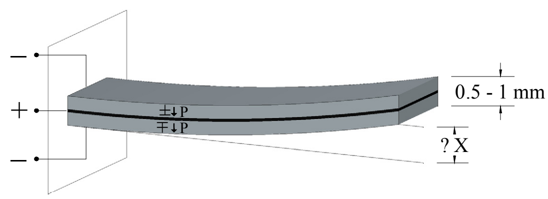


Fig. 5 Scheme of a BE sensor. Adapted from Ferreira (2009).

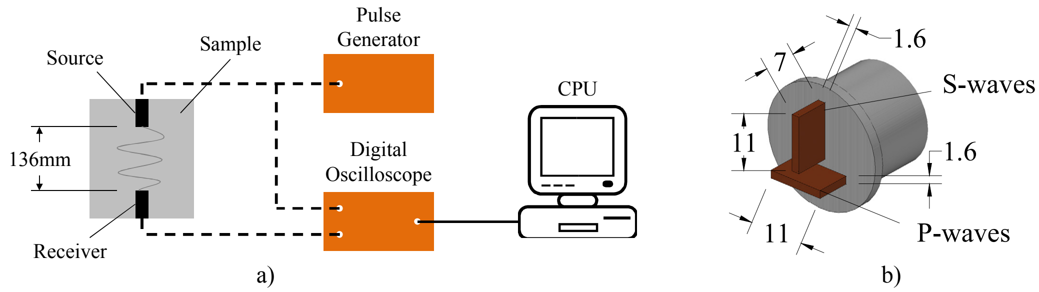


Fig. 6 BE method a) experimental set-up; b) BE probe. Units [mm].

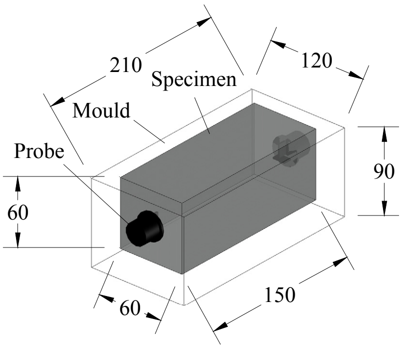


Fig. 7 Container for the use of *bender-extender* elements. Units [mm].

to the mould used for the BE methodology, due to the possible existence of "cross talk" parasite waves. However, as the usual operation of BE elements involves a wide frequency range that can start as low as 1 kHz, the distance between the probes must hence be higher, in comparison with the distance adopted for the UPV method. Preliminary tests in the scope of this research work have shown that the adopted geometry for the UPV (60×60×150mm, as shown in **Fig. 7**) made in extruded polystyrene is also suitable for BE experiments on cement pastes. However, in this case the probes were placed in the opposite faces of the mould that are separated by 136mm. Similarly to the UPV, the BE probes were fixed to the mould in order to allow measurements immediately after mixing.

The choice of the best frequency for wave velocity assessment at each instant of testing was made by manually sweeping the frequency to obtain the highest output signal amplitude. At such frequency, the identification of the wave arrival time is facilitated, since the noise-to-signal ratio is at its lowest. It is reasonable to assume that within the range of frequencies used in this method, the travel time is independent of frequency, that is, the travel time remains the same regardless of the applied input frequency (Viana da Fonseca *et al.* 2009).

2.3 Cyclic compression tests

Cyclic Compression (CC) tests with on-sample strain measurements were also performed to quantify the E-modulus of cylindrical specimens. The cement paste cylindrical specimens utilized in this study had a diameter/height of 50mm/100mm. The testing apparatus includes a hydraulic actuator with 50kN capacity and 3 displacement transducers (LVDTs), supported by 2 steel rings attached to the specimens, as presented in **Fig. 8**. The test protocol adopted in this work was based on the experiments reported by Chamrová (2010) and Maia *et al.* (2012b). Each test involved 3 loading/unloading cycles, with a loading rate of 200 kPa/s, and the E-modulus was computed in the loading branch of the last load/unload cycle. The maximum cyclic load reached 33% of the compressive strength of the cement paste at the age of testing, obtained through destructive compressive tests in 5x5x5 cm^3 cubes.

3. Experimental program

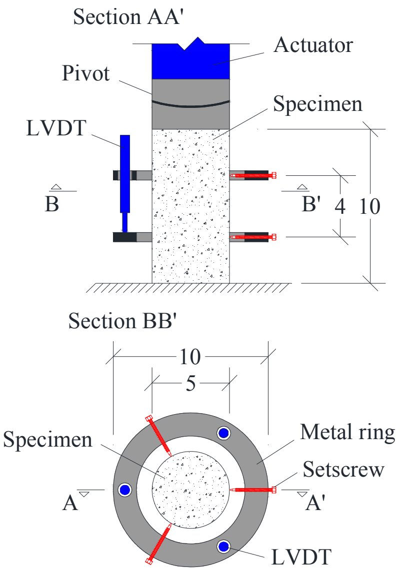
3.1 Materials

The experiments were conducted on cement pastes containing Type I and Type II Portland cement. **Table 1** presents the compositions and properties of the two cements, based on information provided by the cement producer (average analysis of the month in which the

Table 1 Tested cements: chemical and Bogue composition together with other cement characteristics (percentages with respect to mass).

Cement	*CEM II/B-L 32.5N*	*CEM I 42.5R*
Loss of ignition [%]	13.50	2.69
Insoluble residue [%]	3.02	1.34
Silicon Oxide [%]	15.91	19.58
Aluminate Oxide [%]	4.21	4.72
Iron Oxide [%]	2.56	3.24
Calcium Oxide [%]	58.02	63.42
Magnesium oxide [%]	1.38	2.12
Sulphates [%]	2.71	3.52
Potassium oxide [%]	-	-
Sodium oxide [%]	-	-
Chlorides [%]	0.04	0.05
Free lime [%]	-	1.05
N/D (no dosed) [%]	-	-
C_3S [%]	81.38	62.99
C_2S [%]	15.78	8.61
C_3A [%]	-	7.03
C_4AF [%]	ss*=13.20	9.86
C_2F [%]		-
Limestone filler [%]	-	-
Gypsum [%]	-	-
Blaine [cm^2/g]	4899	3891
Specific gravity [g/cm^3]	2.99	3.13

*ss - calcium aluminoferrite solid solution(C_4AF+ C_2F)



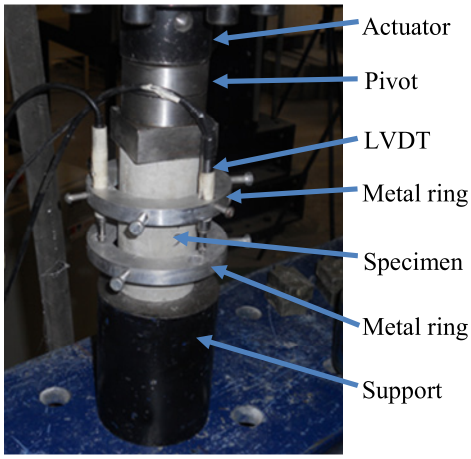


Fig. 8 Cyclic compression method: a) test setup; b) photo of a specimen instrumented with LVDTs fixed by steel rings. Units [mm].

Table 2 Cement pastes adopted in this research work.

Reference	*Cement type*	*w/c ratio*	*Density (kg/m^3)*
c32.5wc0.5	CEM II/B-L 32.5 N	0.50	1787.44±3.97
c42.5wc0.5	CEM I 45.5 R	0.50	1840.33±4.08
c42.5wc0.45	CEM I 45.5 R	0.45	1926.81

Table 3 Specimens used in the study.

Specimen	*Cement paste*	*Monitoring method*	*Curing temperature [°C]*
32.5-Vic	c32.5wc0.5	Vicat	20
32.5-CC	c32.5wc0.5	CC	20
32.5-UPV	c32.5wc0.5	UPV	20
32.5-BE	c32.5wc0.5	BE	20
32.5-EMM1	c32.5wc0.5	EMM-ARM	20
32.5-EMM2	c32.5wc0.5	EMM-ARM	20
42.5-Vic	c42.5wc0.5	Vicat	20
42.5-CC	c42.5wc0.5	CC	20
42.5-UPV	c42.5wc0.5	UPV	20
42.5-BE	c42.5wc0.5	BE	20
42.5-EMM1	c42.5wc0.5	EMM-ARM	20
42.5-EMM2	c42.5wc0.5	EMM-ARM	20
42.5A-EMM	c42.5wc0.45	EMM-ARM	20
32.5-EMM40	c32.5wc0.5	EMM-ARM	40
42.5-EMM40	c42.5wc0.5	EMM-ARM	40

cement was produced). The Bogue composition was calculated according to ASTM C150 (2004).

In the scope of this experimental program, two cement paste compositions with two different water/cement ratios (w/c=0.45 and w/c=0.50) were adopted. The mixture proportions of the cement pastes as well as the corresponding nomenclatures are presented in **Table 2**. For both cement pastes with w/c=0.5, more than one batch was tested. Therefore, the density value indicated in **Table 2** corresponds to the average value and the observed variation (less than 0.23%). This care in the determination of density is particularly important due to its role in the evaluation of E-modulus of the cement paste through the EMM-ARM method, as shown in equations 1 and 2.

The acrylic tubes used for the EMM-ARM had an average elastic modulus of 4.72 GPa at 20°C (with a variation of ±0.02 GPa in all tested moulds) and an average density of 1172 kg/m^3. The values of E-modulus and density of the acrylic were verified in the laboratory through modal identification of the empty moulds, which were weighed before the start of each test.

3.2 Experimental program and procedure

The experimental program involved the application of the EMM-ARM together with all the other methods and specimen sizes described in section 2: ultrasonic pulse velocity (UPV), *bender-extender elements* (BE) and cyclic compression (CC) tests. Penetration resistance was also measured through Vicat needle testing, according to EN 196-3 (2005b). All EMM-ARM tests involved simultaneous testing of two specimens for repeatability checking. The list of all the specimens used during this experimental program, comprising the three cement pastes previously mentioned, is presented in **Table 3**. EMM-ARM, BE and UPV tests were performed continuously since casting, whereas the cyclic compression tests were conducted at the ages of 2, 3, 7, 14 and 28 days.

In specific regard to the preparation of the cement pastes, the mixing operations were performed in an automatic mixer, according to the following procedure that conforms the recommendations of EN 196-1 (2005a): (i) introduce the cement and immediately after add water (instant defined as “t=0”); (ii) start mixing at 500 rpm for 90 seconds; (iii) stop mixing during the

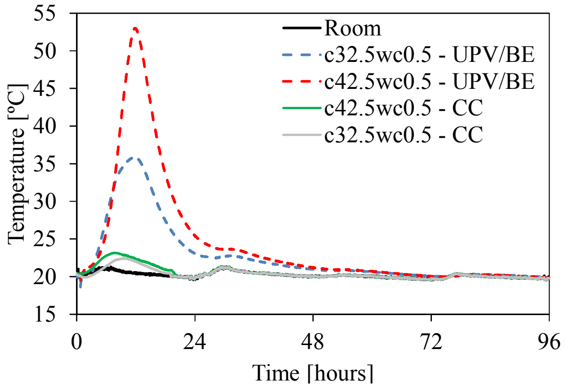


Fig. 9 Specimens temperature history.

following 90 seconds; (iv) resume mixing operation at 500 rpm for another 30 seconds. After the mixing process, the resulting cement pastes were poured into the moulds, which were simultaneously slightly vibrated for removal of air bubbles during the casting process. The time elapsed between mixing and the beginning of monitoring did not exceed 20 minutes for any of the continuous methods (EMM-ARM, UPV and BE).

All these tests on c32.5wc0.5, c42.5wc0.5 and c42.5wc0.45 were performed under moist sealed conditions at 20°C (exception for two cases documented later, which were cured at 40ºC) and carried out for at least 7 days. The only exception to the mentioned situation corresponded to the CC specimens that were demoulded right before the first test (t=2days) and were placed, unsealed, in a controlled environment with T=20ºC and RH=60%. The temperature inside the distinct samples was assessed through embedded K-type thermocouples. In the EMM-ARM samples, the maximum temperature increase with respect to the registered room temperature was lower than 0.5°C, thus rendering temperature variation effects negligible. In the remaining specimens, maturity corrections were necessary to take into account the temperature effects during the cement hydration process (D'Aloia 2003; Malhotra 1956; Rubinsky 1954) shown in **Fig. 9**.

The comparative results to be presented in the next section refer to their equivalent age (t_{eq}), computed according to Waller *et al.* (2004):

$$t_{eq} = \int_0^t e^{\frac{-E_a}{R}\left[\frac{1}{T(\tau)}-\frac{1}{T_r}\right]} d\tau \qquad (4)$$

where t is the instant at which the equivalent age is being computed, E_a is the apparent activation energy, R is the universal gas constant (8.314 J/mol.K), $T(\tau)$ is the temperature at instant τ and T_r is the reference temperature (adopted as 293.15K=20°C). In order to assess the apparent activation energy, two additional tests were performed with the EMM-ARM. These additional experiments were conducted on cement pastes c32.5wc0.5 and c42.5wc0.5, in a temperature-controlled room that guaranteed an average temperature of 40.2±0.2ºC. The obtained E_a values that were used for maturity corrections in the scope of this study are discussed in section 4.2.

4. Results and discussion

4.1 Comparison between monitoring methods

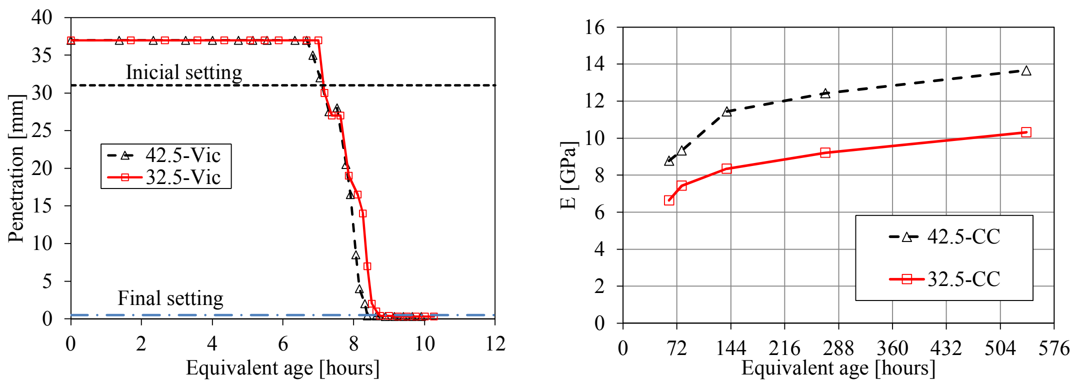
The results are cumulatively presented below, beginning with the results of classic methods (Vicat test and cyclic compression), followed by the EMM-ARM results. The comparison proceeds with the results obtained through the wave propagation methods (UPV and BE). Finally, a comparison between the data collected through all analysed testing methodologies is presented.

4.1.1 Penetration tests and cyclic compression tests

The Vicat and cyclic compression tests conducted on the cement pastes c32.5wc0.5 and c42.5wc0.5 yielded the results presented in **Fig. 10a** and **10b**. The results of penetration testing (**Fig. 10a**) show that the structural setting time occurs earlier for the cement paste prepared with the higher cement class (c42.5wc0.5) – see **Table 4**. Cyclic compression testing of c42.5wc0.5 also yielded

Table 4 Structural setting time of the cement pastes.

Cement paste	Initial setting (hours)	Final setting (hours)
c42.5wc0.5	7.05	8.39
c32.5wc0.5	7.17	8.75



a) b)

Fig. 10 Results obtained through the classic methods a) penetration resistance (Vicat needle) b) Cyclic compression.

higher elastic modulus than of c32.5wc0.5 during the whole period of study as shown in **Fig. 10b**.

4.1.2 EMM-ARM

The resonant frequencies identified by the EMM-ARM method for the three studied cement paste mixtures are shown in **Fig. 11a**. It is worth mentioning that a wide range of frequencies was covered throughout the curing process of the cement pastes, ranging from ~14.6 Hz to 26.0 Hz within the testing period. Moreover, all frequency evolution curves appear to be plausible, showing an initial dormant period (where the frequency remains almost constant within ±0.3 Hz). After this threshold, the frequencies evolved significantly for all tested specimens until approximately 48 hours of curing period, after which a dramatic reduction in the slope of frequency evolution occurs.

The elasticity modulus of the tested cement pastes was estimated by applying equations 1 and 2 to the resonant frequencies presented in **Fig. 11a**. The resulting E-modulus evolution is shown in **Fig. 11b**.

Firstly, it is possible to verify that the E-modulus evolution curves of the same cement pastes have very good coherence with each other, demonstrating adequate repeatability of EMM-ARM. Furthermore, when comparing the results obtained with the two pastes containing the same w/c ratio (c42.5wc0.5 and c32.5wc0.5), it can be seen that the cement paste containing the CEM I 42.5R cement has a higher stiffness, with a difference of ~2GPa at the age of 7 days (168 hours) - see **Fig. 11b**. However, even though the cement paste c42.5wc0.5 has reached a higher stiffness after the first day of curing, the E-modulus evolution at very early ages is fairly similar to that of paste c32.5wc0.5. In fact, this would not be expected by strictly considering the chemical composition of the utilized cements, namely due to the higher C_3S content of cement CEM I 42.5R. Nonetheless, another important characteristic might justify this behaviour at very early ages: the specific surface or Blaine index. In fact, the Blaine index of CEM I 42.5 R is lower than that of CEM II/B-L 32.5N (3891 against 4899 cm^2/g according to **Table 1**). The similarity of the E-modulus evolution at very early ages between the two pastes can thus be considered reasonable, taking into account these two aspects that justify the apparent inverse trends: clinker composition and specific surface.

A further comparative interpretation can be made by observing the behaviour of the two pastes containing the same type of cement: c42.5wc0.5 and c42.5wc0.45. The corresponding results are shown in **Fig. 11b**, where the expected trend was confirmed: the reduction of the w/c ratio increases early hydration velocities, and leads to higher values of E-modulus (after 6 days of curing, there is a difference of approximately 1.3 GPa).

The comparison between the elastic modulus results obtained by EMM-ARM and by classic methods (CC and Vicat) for the cement pastes c32.5wc0.5 and c42.5wc0.5 is shown in **Fig. 12**. It can be seen that the values obtained through the EMM-ARM are similar to those collected in CC tests in terms of magnitude and evolution kinetics. However, the results for c32.5wc0.5 (**Fig. 12a**) show a non-negligible difference of 1.4 GPa at t_{eq}=22.4 days (538 hours). This deviation may possibly be explained by differences in the curing conditions of the samples. In fact, the EMM-ARM samples remained in perfectly sealed conditions during the whole test, while the samples used for the CC tests were exposed to drying during the testing period. This small variation in the curing conditions may have influenced the hydration process at the surface of the CC specimens (Parrott 1990), which are significant in view of the small size of the specimen, thus resulting in lower stiffness. As the porosity of c32.5wc0.5 is higher than that of c42.5wc0.5, it is plausible that this deficient curing of CC specimens may have affected c32.5wc0.5 more significantly, as opposed to c42.5wc0.5.

The results presented in **Fig. 12** also show good agreement between EMM-ARM and the data collected by the Vicat needle, in the sense that the end of setting determined by Vicat testing coincides with the end of the dormant period observed in EMM-ARM, followed by a strong acceleration of the hydration kinetics.

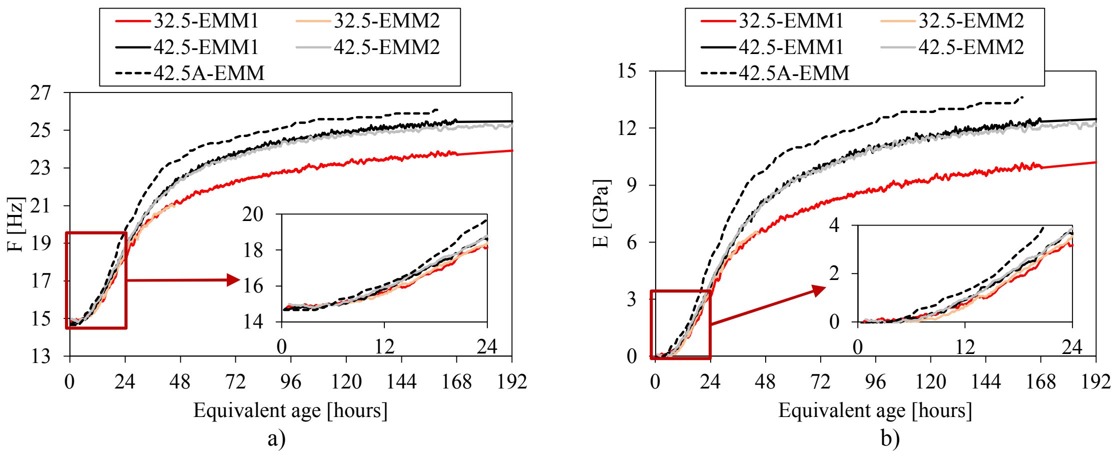


Fig. 11 EMM-ARM results: a) frequency evolution; b) E-modulus evolution.

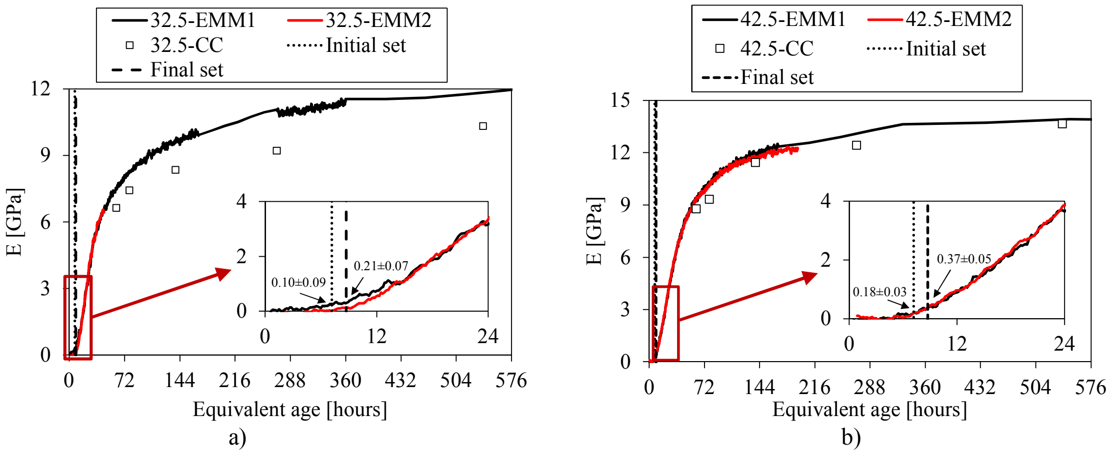


Fig. 12 Comparison of the results of EMM-ARM, Vicat, and cyclic compression for cement pastes: a) c32.5wc0.5 b) c42.5wc0.5.

4.1.3 Ultrasonic Pulse Velocity (UPV)

The evolution of the P-wave velocity for c42.5wc0.5 and c32.5wc0.5 is shown in **Fig. 13**, together with information collected by Vicat testing.

Firstly, it should be noted that UPV was unable to provide measurements of P-wave velocity in the cement pastes at very early ages, including the setting period. The earliest measurement was only possible at t_{eq}=12.4 h, when EMM-ARM already exhibited E-moduli above 0.8 GPa for both pastes (see **Fig. 12**). The reason for this problem can be attributed to the presence of air bubbles in the samples, which have been reported to attenuate and delay the wave propagation, as well as due to the high impedance mismatch between the transducers and the fresh cement paste (Zhu *et al.* 2011a). In fact, in order to monitor the evolution of the P-wave velocity in cement pastes, some authors (Boumiz *et al.* 1996; Zhu *et al.* 2011a) used de-aired samples (previously placed in vacuum) with successful results. However, since the cement pastes always contain some air bubbles the de-airing of the samples may end up producing unrealistic results. In order to avoid this drawback in UPV, some authors (Reinhardt and Grosse 2004) successfully performed measurements during the setting by using smaller distances between the probes and more energetic excitation signals (with higher voltage) even with the wave attenuation. Such alternative was not available in this research work. Another solution to this problem, currently under research, involves adjusting the input frequencies to lower values (below the reference frequency of the ultrasonic transducers) at very early ages. Despite the reduction in the performance of the transducer, this procedure would enable to comply with the initially low stiffness of the tested material. However, despite the absence of UPV measurements in this initial period (~12 hours), the wave velocity measurements of **Fig. 13** exhibit an evolution which can be considered plausible. In fact, the various stages usually observed in the cement hydration kinetics after the dormant period can be identified: (i) an initial stage where a substantial increase in wave velocity occurs; (ii) a subsequent stage in which the velocity evolution becomes less significant. Lastly, it can be noted that the c42.5wc0.5 paste shows a greater increase in wave velocity, which is consistent with the results of the EMM-ARM that were already reported.

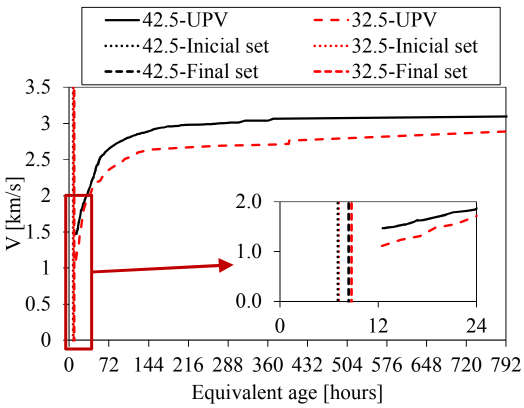


Fig. 13 P-wave velocity evolution for cement pastes c42.5wc0.5 and c32.5wc0.5.

4.1.4 Bender-extender elements (BE)

The application of BE for studying the evolution of the stiffness characteristics of cement pastes is still taking its first steps, with very few published works so far (Zhu and Kee 2010; Zhu *et al.* 2011b). However, the success already achieved in the application of BE to cement-stabilized soils at the University of Minho (Azenha *et al.* 2011; Silva *et al.* 2013) has justified the interest on this testing methodology.

As opposed to the UPV method, the use of BE easily allows to perform high quality measurements immediately after casting. This is mainly due to the high efficiency over a wide frequency range that the BE probes possess, which enables an adjustment of the input frequency to provide better results at each instant of measurement. However, despite this benefit, the use of BE is often accompanied by difficulties associated with a high sensitivity to external disturbances (such as the existence of electrical noise in the testing room), which ob-

scure and compromise the interpretation of test results. This high sensitivity can be partly explained by the relatively low power of the signal generator adopted in this research that solely allowed a maximum excitation amplitude of 20 V. On the other hand, the use of power amplifiers (to boost the input signal) is limited by the transducer itself, which depolarises approximately above 60 V.

Figure 14 shows four S-wave signal readings performed for the paste c42.5wc0.5 at ages of 2.9, 9.0, 26 and 172 hours. These readings were conducted at optimum increasing frequencies between 1 kHz at 2.9 hours and 50 kHz at 172 hours (see **Fig. 14a**). In the four measurements presented in **Fig. 14b**, one can clearly observe the difficulty the identification of the first arrival of the wave. This problem was already mentioned in the works of Ferreira (2009) and Viana da Fonseca *et al.* (2009) regarding the performance of BE in stiff materials.

The BE used in these tests have the capability of measuring compressional (P) and shear (S) waves, hence initially all tests included the recording of both wave types. However, after some measurements, it was found that both sensors measured exactly the same type of wave: S-waves (noted by the same wave shape and the order of magnitude of the recorded velocities). The justification for this phenomenon is threefold: (i) at very early ages, i.e. in fresh pastes, the propagation of P-waves is difficult, as already mentioned in regard to UPV tests, due to the presence of entrapped air and to the high stiffness impedance between the transducer and the material; (ii) as the paste hardens, the compressional wave velocity increases rapidly and the frequency required to measure such high velocity causes the BE to vibrate in complex mode shapes, which in turn strongly reduces the amplitude of the effectively propagated wave and makes it difficult to detect the P-wave in the received signal; (iii) due to the characteristics of the BE probes, as mentioned by Lee and Santamarina (2005), and schematically shown in **Fig. 15**: the probe generates two P-wave side lobes normal to their plane and a S-wave frontal lobe. Therefore, since the generation of response signal in the receiver demands that the sensor itself bends, when the BE transmitter and BE receiver are perfectly aligned (which is the case in our work), the P-waves are parallel to the longitudinal axis of the receiving transducer, thus being unable to flex it. On the other hand, the S waves disturb the transducer in the direction perpendicular to its longitudinal axis, thus causing a larger bending motion and as a result a larger signal output. This feature causes them to lose the ability to adequately receive P-waves, while having a significant resolution in the measurement of S-waves.

Consequently, the attempt to measure P-waves with BE in the scope of this research work was abandoned. Moreover, these results demonstrate that the use of S-waves is more suited to monitor these complex evolving processes than P-waves, since shear waves only propagate through the solid skeleton of the specimens, providing a higher sensitivity towards the structural changes that occur during setting.

After obtaining the propagation time of the S-waves, the evolution of the wave velocity was computed, as shown in **Fig. 16**. It can be observed that there are no significant differences in the recorded wave velocities for the two types of pastes. However, the S-wave velocity is higher in the c42.5wc0.5 paste throughout the entire curing period. It should also be noted that the difference in the wave velocity increases along the curing process, which is in agreement with the results of the previous methods. Therefore, the application feasibility of this methodology to cement pastes was confirmed in coherence with the conclusions of the research work conducted by Zhu *et al.* (2011b). It should also be noted

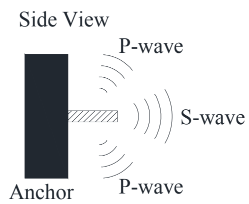


Fig. 15 Waves generated by a BE probe. Adapted from Lee and Santamarina (2005).

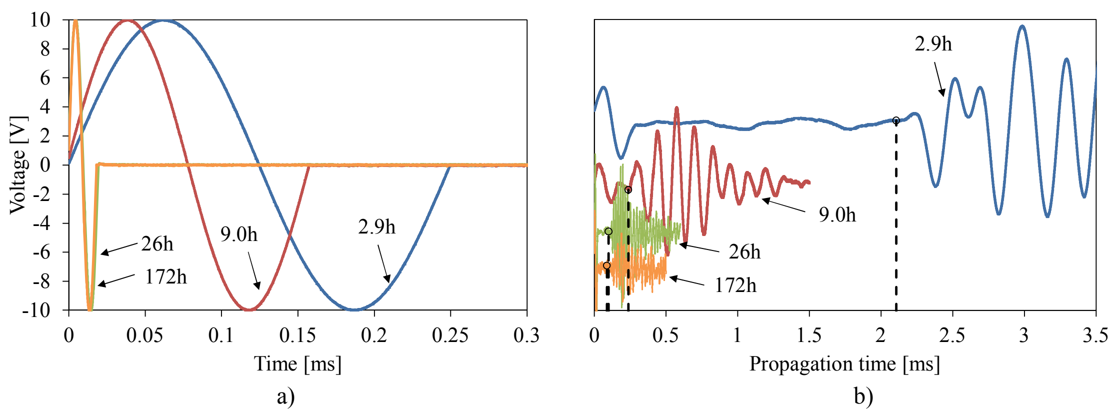


Fig. 14 Signal readings during the use of the BE method in the paste c42.5wc0.5 at 2.9, 9, 26 and 172 hours of curing: a) Input signal at the transmitter BE; b) Output signal registered by the receiver BE.

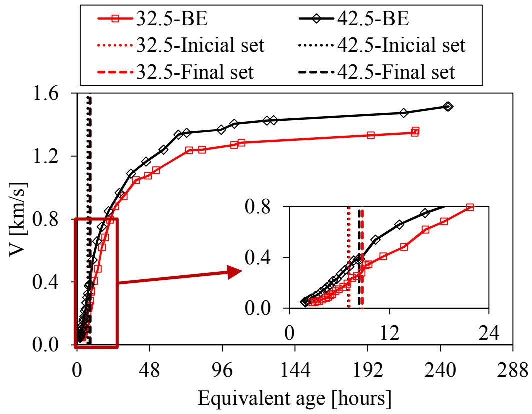


Fig. 16 S-waves velocity evolution in the c32.5wc0.5 and c42.5wc0.5 cement pastes.

that, in the very early ages of curing (before setting), this method is more sensitive than the EMM-ARM. In fact, the measurements with BE method begin ~2hours after mixing, which is significantly sooner than the initial setting time that only occurred at 7 hours age. This is a clear indication that a robust solid skeleton for S-wave transmission exists much sooner than the conventional initial setting time (Sant *et al.* 2009), thus highlighting the potential of S-waves for measuring pre-setting behaviour. In fact, if we analyse the viscoelastic properties (for example through a rheometric test) it can be observed that significant changes occur during this period. However the EMM-ARM results remains unchanged, while there is already evolution of the velocity of the propagated wave measured with the BE.

4.1.5 Overall comparison

Taking into account that the methodologies based on wave propagation measure dynamic parameters, for the purpose of comparison of all methodologies under study, the results were normalized by dividing all results of each specimen/methodology by their corresponding values at t_{eq}=7 days. Moreover, in order to simplify the analysis and to compare both methods based on wave propagation (BE and UPV) with the results of quasi-static methods, the velocity values were squared (V^2) prior to normalization, as V^2 is proportional to the elasticity modulus (see equation 3).

The results of all experimental methods are given in **Fig. 17**, which demonstrates a quite reasonable reciprocal agreement, thus mutually validating the studied methodologies. The good performance of EMM-ARM in the scope of this comparative study, together with its ability to provide precise, continuous and quantitative estimates of E-modulus confirms the versatility and applicability of this methodology.

In regard to setting times, there is also a good coherence between Vicat, EMM-ARM and BE, as observable in **Fig. 17**. Thus, these results have confirmed the applicability of the wave propagation methods to monitor the stiffness of cement pastes since the fresh state and throughout the entire hardening process, as already mentioned by other authors (Boumiz *et al* 1996; Reinhardt and Grosse 2004). Despite this fact, the results obtained by these wave-propagation based methods should be regarded qualitatively, since these refer to dynamic properties, which conversion to static properties is often arguable, particularly at very early ages, due to the evolution of Poisson's ratio during curing (Popovics *et al.* 2008).

However, it should be noted that these wave velocity methods (UPV and BE) seem to exhibit a slightly more accelerated evolution kinetics than EMM-ARM, which is more evident for the c42.5wc0.5 paste, as shown in **Fig. 17b**. This fact may be related to the early evolution of Poisson's ratio. Similar findings have been reported in other research works, where the consideration of constant Poisson's ratios led to apparent earlier acceleration of stiffness when estimated through pulse velocity methods (Boulay *et al.* 2013a).

4.2 Activation energy

The two additional EMM-ARM tests that were performed at 40°C allowed obtaining the activation energy of c42.5wc0.5 and c32.5wc0.5. Given the influence of temperature variations on the stiffness of the acrylic (Schirrer and Goett 1982), an initial modal identification

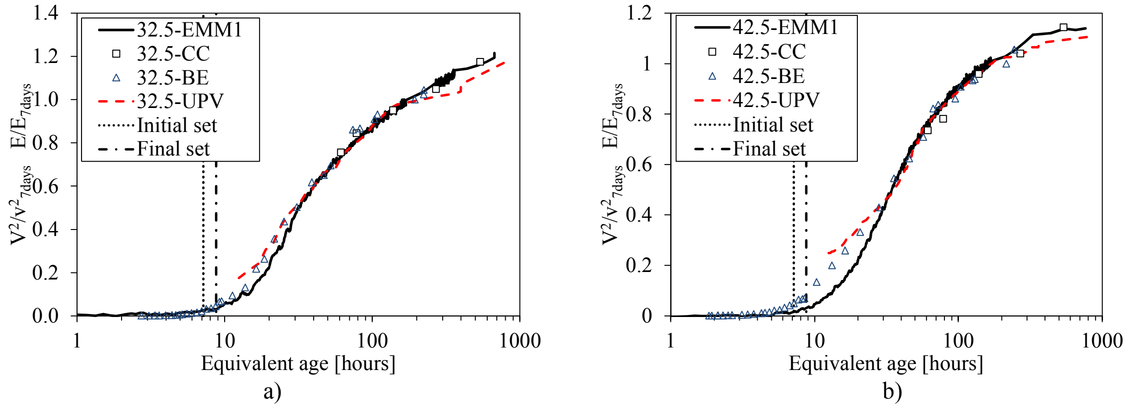


Fig. 17 Comparison of the results of all methodologies used in this study for cement pastes a) c32.5wc0.5 and b) c42.5wc0.5.

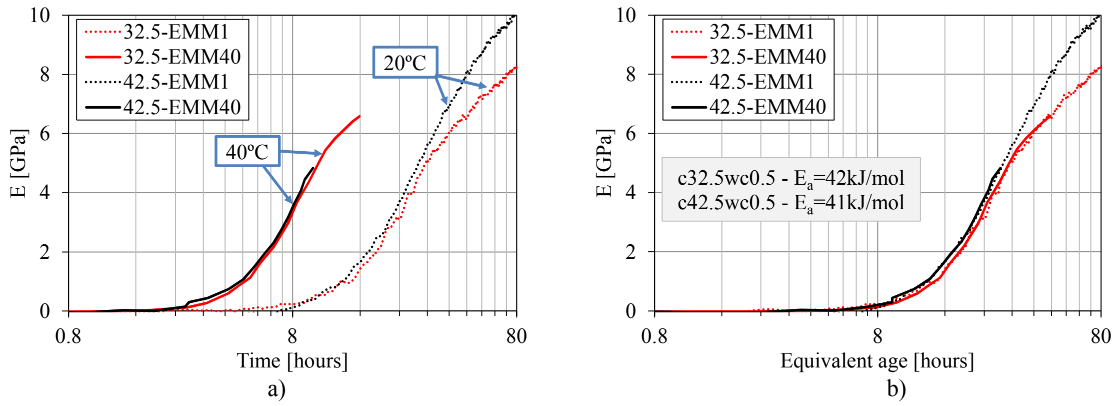


Fig. 18 E-modulus evolution of the cement pastes c32.5wc0.5 and c42.5wc0.5 cured at different temperatures (20°C and 40°C), obtained through the EMM-ARM: a) plotted in order to the curing age b) plotted in order to the equivalent age at 20°C.

test was performed on the acrylic hollow beam to obtain the E-modulus of the acrylic at 40ºC. An E-modulus of 3.6±0.2 GPa was obtained.

The results collected during the activation energy experiments are shown in **Fig. 18a**. Note that the test conducted inside the temperature-controlled chamber at ~40ºC lasted only 10 hours for the c32.5wc0.5 paste and 16 hours for the c42.5wc0.5 paste. The limited duration of these tests was due to an electrical problem in the data acquisition system. Nonetheless, despite the short duration of the tests, the repetition of the tests was not considered necessary because the extracted information was sufficient for determining the activation energy of the tested cement pastes. The results show that the initial setting in specimens exposed to 40°C occurred much earlier (~2 hours) than the initial setting observed in the tests at 20°C (~7 hours). Moreover, the reaction rate was also more significant, since it can be observed that, for example, the elastic modulus of 5 GPa is achieved at approximately 0.4 days at 40ºC, as opposed to the approximately 1.25 days required for the test conducted at 20°C.

The data collected through these particular tests enabled the possibility of determining the apparent activation energy (E_a) by the successive application of equation 4 with distinct E_a values until the E-moduli evolution at both temperatures (20°C and 40°C) matched when presented in order to their equivalent age (t_{eq}) (see **Fig. 18b**). This procedure is usually referred to as the "superposition method" (D'Aloia, 2003, D'Aloïa *et al.*, 2001). The following values of E_a were computed: 42 kJ/mol and 41 kJ/mol for pastes c32.5wc0.5 and c42.5wc0.5, respectively.

5. Conclusions

Based on the methods, experiments and results presented throughout this paper, concerning the experimental determination of early evolution of E-modulus in cement pastes, the following main conclusions can be drawn:

(1) The experiments carried out with the EMM-ARM in different periods with the same cement paste allowed to prove the repeatability of the methodology.
(2) For the first time, EMM-ARM tests were performed at 40°C to validate the feasibility of the method under temperatures distinct from 20°C. The monitoring of the E-modulus evolution at different temperatures allowed the calculation of the activation energy of the cement pastes, constituting thus another potentiality for field application of the EMM-ARM method, which had not yet been explored.
(3) The UPV method has not allowed to monitor the cement paste since the earliest ages (after mixing). It is estimated that the possible reasons may be associated with to the presence of air bubbles in the samples and to the high stiffness impedance between the transducers and the paste, particularly in the initial stage of the curing process (during setting), as was already mentioned by other authors (Zhu *et al.*, 2011a).
(4) In addition to the use of EMM-ARM, this study also presented stiffness evaluation through bender-extender elements, which had only been applied to cement paste by Zhu *et al.* (2011b). The results obtained in this research work were consistent with such previous approach, confirming that BE sensors have a high level of performance in a wide frequency range, which enables the stiffness monitoring of cementitious materials from very early ages (often not possible with UPV). This is achieved given the possibility of adjusting the input frequency to optimize the amplitude of the received signal.
(5) The results obtained with BE also demonstrate a higher suitability of the use of S-waves in detriment to P-waves to monitor the setting process, due to their sensitivity towards structure changes, since the propagation of shear waves only occurs in the solid skeleton of the specimens.
(6) The stiffness evolution results obtained by all the

methods exhibited a considerably good agreement, which points to the mutual validation of the utilized methodologies. It is also worth mentioning that EMM-ARM and BE were able to detect the setting time with very good consistency in comparison with the Vicat test.

(7) The authors consider that EMM-ARM was the method that proved to be the most advantageous by allowing continuous automatic monitoring of the E-modulus of tested cementitious materials, with collected data providing quantitative estimates of the elastic modulus.

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