BENDER-EXTENDER ELEMENTS FOR CHARACTERIZATION OF CEMENT PASTE AT EARLY AGES

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Abstract

The embedment of bender-extender elements in cement-based materials for assessment of the early development of mechanical properties is a relatively unexplored field. This technique provides the opportunity of embedding piezoelectric elements (emitter and receiver) into the tested material at the fresh state, generate waves and assess the velocity of propagation. It has the interesting feature of allowing distinct frequencies of wave to be explored at the several stages of stiffening of the testing material, thus maximising signal intensity and facilitating the identification of velocities. This paper presents an exploratory application of bender-extender elements to cement paste specimens, in parallel with other established experimental techniques, such as the Vicat needle, ultrasound pulse velocity measurements (with external probes), measurement of E-modulus through cyclic compressive testing and continuous assessment of the E-modulus of the cement paste through EMM-ARM. The results are evaluated and discussed in an integrated manner and conclusions are drawn in regard to the potential of using bender-extender elements in cement-based materials.

1   Introduction

The experimental characterisation of the behaviour of cement-based materials since early ages has been tackled by many researchers through a wide range of techniques. The use of non-destructive techniques based on wave propagation has been subject of wide discussion and developments, both in view of the type of propagated waves (e.g. P or S wave) [1], but also on the methods to identify the velocity of the waves (e.g. time or frequency domain methods) [2]. A relatively recent contribution by Zhu J., et al. [3] has shown the feasibility of adopting bender extender elements for wave propagation assessment in cement based materials. Bender extender elements are composed of piezoelectric plates that are normally coated by an epoxy resin, which are able to generate and measure waves in the tested material. Even though this technique has been widely applied in the study of cohesive soils, the application to cement-

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based materials is still at its infancy. The present work aimed to contribute in such field of research, particularly by implementing and testing the application of bender-extender elements in the evaluation of hardening cement pastes since very early ages.

This paper reports a proposed test setup and its pilot experiment applied for two distinct cement pastes. In parallel to this, complementary experimental techniques have been used for comparative purposes: (i) EMM-ARM, (ii) cyclic compression tests for E-modulus assessment, (iii) ultrasound pulse velocity measurement, and (iv) Vicat needle testing. The discussion of obtained results allows obtaining insights about the possibilities and challenges brought about by applying bender-extender elements to the characterisation of cement-based materials.

2 Experimental Program

2.1 Materials

The experiments were conducted on two cement pastes containing Type I and II Portland cement, respectively, while having the same water-to-cement ratio. The mixture proportions of the cement pastes as well as the corresponding nomenclatures are presented in Table 1.

Table 1: Cement pastes adopted in this research work.

Reference	Cement type	w/c ratio	Density (kg/m^3)
c32.5	CEM II/B-L 32.5 N	0.50	1787.4±4.0
c42.5	CEM I 45.5 R	0.50	1840.3±4.1

2.2 Experimental program and procedures

The experimental program involved the assessment of the E-modulus of both cement pastes through 4 distinct methods: bender-extender elements (BE), ultrasonic pulse velocity (UPV), EMM-ARM, and classic cyclic compression (CC). Penetration resistance was also measured through Vicat needle testing, according to EN 196-3:2005. The details about the procedures used in each method are presented in the next section. The list of all the specimens used during this experimental program, comprising the two cement pastes under study is shown in Table 2. BE, UPV and EMM-ARM tests were performed continuously since casting, whereas the cyclic compression tests were conducted at the ages of 2, 3, 7, 14 and 28 days.

Table 2: Specimens used in the study.

Specimen	Cement paste	Monitoring method
32.5-Vic	c32.5	Vicat
32.5-CC	c32.5	CC
32.5-BE	c32.5	BE
32.5-UPV	c32.5	UPV
32.5-EMM	c32.5	EMM-ARM
42.5-Vic	c42.5	Vicat
42.5-CC	c42.5	CC
42.5-BE	c42.5	BE
42.5-UPV	c42.5	UPV
42.5-EMM	c42.5	EMM-ARM

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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 196-1:2005: (i) introduce the cement in the mixer and immediately after add water (instant defined as "t=0"); (ii) start mixing at 500 rpm for 90 seconds; (iii) stop mixing during the 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 (BE, UPV and EMM-ARM). All these tests on c32.5 and c42.5 were performed under moist sealed conditions at 20°C 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%.

3   Experimental techniques and protocols

3.1 Bender-extender elements (BE)

In the present work, the evolution of the velocity of ultrasonic waves was measured along hydration through bender-extender elements (BE), according to the test configuration shown in Figure 1a. In this study, T-shaped bender-extender elements made in the University of Western Australia were used, and they are schematically depicted in Figure 1b. These BE enable the measurement of P and S waves and operate in a wide frequency range, common for this type of probes. The setup consists of two BE probes, one transmitter placed on one side of the sample, as depicted in Figure 1b, and a receiver positioned on the opposite side. A single period 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 and received in the opposite side of the sample by another BE probe. An oscilloscope with 16-bit resolution and a sensitivity of 10 mV/div to 20 V/div (PicoScope 4424) performs the analog-to-digital conversion of both emitted and received waves and transmits the digitised waves to the computer for signal processing.

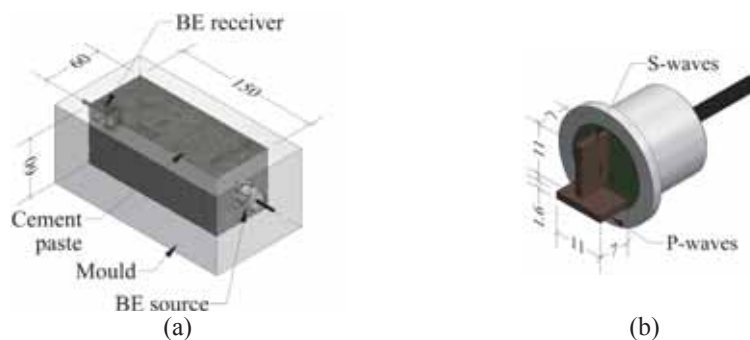(a)   (b)

Figure 1: BE method (a) experimental set-up; (b) BE probe [units: mm].

In order to avoid errors in the identification of the wave propagation time, special attention had to be given to the mould used for the BE methodology, due to the possible existence of

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"cross talk" parasite waves [4]. 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 a specimen sized $60 \times 60 \times 150$ mm^3, moulded inside extruded polystyrene walls is suitable for BE experiments on cement pastes. The probes were placed in the opposite faces of the mould that are separated by 150 mm, as shown in Figure 1a. In order to have a good coupling between the BE probes and the material they were fixed to the mould prior to casting and 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 several frequencies in the range 1 to 50 kHz 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 considered 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 [5].

3.2 Ultrasound Pulse Velocity (UPV)

The test configuration adopted for this methodology is quite similar to the one described in the previous section for BE, except for the use of ultrassonic contact probes instead of BE probes. The contact probes were P-wave probes with operating frequency of 150 kHz and diameter of 25 mm. 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. The same prismatic mould mentioned for BE testing was used: $60 \times 60 \times 150$ mm3. To allow measurements immediately after mixing, the UPV probes were positioned in advance in the opposite faces of the cross-section of the mould, at a distance of 60mm, as shown in Figure 1a.

3.3 EMM-ARM

The cement pastes were tested with the corresponding version of EMM-ARM, as suggested by Granja J. [6]., with the test apparatus shown in Figure 2. The accelerations at the free end of the composite beam were measured with an accelerometer 352C04 (sensitivity: 1 V/g; range: ±5 g) connected to a dynamic acquisition system NI 9234 with 24-bit resolution. The accelerations were recorded at an acquisition frequency of 200 Hz in packages of 120 seconds each 10 minutes.
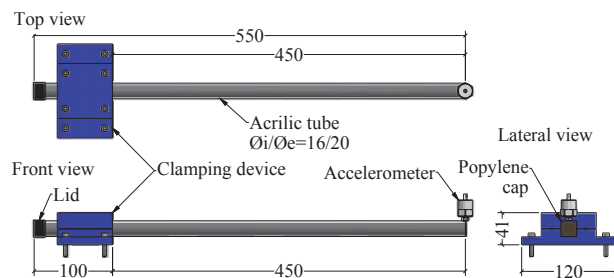

Figure 2: Experimental setup for EMM-ARM testing [units: mm].

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3.4 Classical cyclic compression tests (CC)

Classical cyclic compression tests (CC) with on-sample strain measurements were performed to quantify the E-modulus of cylindrical specimens. The cement paste cylindrical specimens utilised in this study had a diameter/height of 50/100 mm. The testing apparatus includes a hydraulic actuator with 50 kN capacity and 3 displacement transducers (LVDTs), supported by 2 aluminium rings attached to the specimens (spaced 40 mm). The test protocol adopted in this work was based on the experiments reported by Maia L., et al. [7]. 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 $50\times50\times50$ mm^3 cubes.

4 Results and discussion

4.1 Bender-extender elements (BE)

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), that poses relevant hurdles for 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 3 shows four S-wave signal readings performed for the paste c42.5 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 Figure 3a). In the four measurements presented in Figure 3b, 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 C. M. [8] and Viana da Fonseca A., et al. [5] regarding the performance of BE in stiff materials.
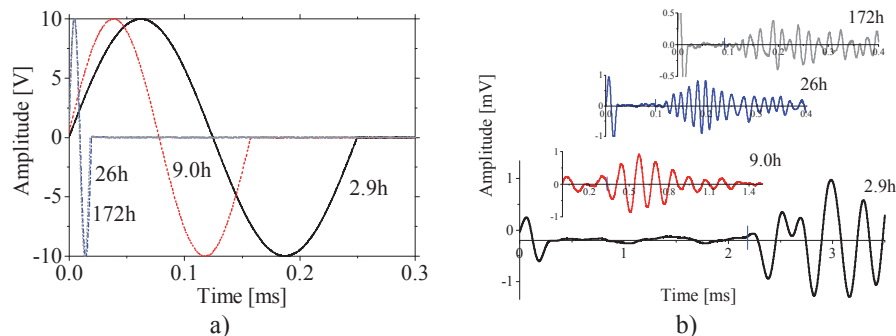

Figure 3: BE Signal readings during tests of c42.5: a) Input; b) Output.

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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 likely to rely on the characteristics of the BE probes, as mentioned by Lee J.-S. and Santamarina, J. C. [9], and schematically shown in Figure 4a: 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. Additionally, at very early ages, i.e. in fresh pastes, the propagation of P-waves is difficult, due to the presence of entrapped air and to the high stiffness impedance between the transducer and the material. 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.
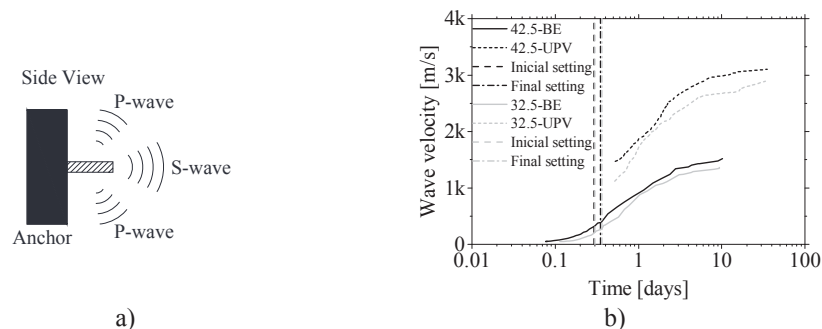

Figure 4: a) Waves in a BE probe. Adapted from [9]; b) Results for c32.5 and c42.5 (waves).

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 Figure 4b. 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 c42.5 paste throughout the entire curing period. It should also be noted that the difference in the wave velocity increases along the curing process. Therefore, the application feasibility of this methodology to cement

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pastes was confirmed in coherence with the conclusions of the research work conducted by Zhu J., Tsai, Y.-T. and Kee, S.-H. [3].

4.2 Ultrasonic Pulse Velocity (UPV) and Vicat

The evolution of the P-wave velocity for c42.5 and c32.5 is shown in Figure 4b, 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 = 12.4 hours for both pastes (see Figure 5). 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, the low power used to excite the transducer (10Vpp as opposite to >100Vpp used by other authors), as well as due to the high impedance mismatch between the transducers and the fresh cement paste [3]. In fact, in order to monitor the evolution of the P-wave velocity in cement pastes, some authors [10] used de-aired samples (previously placed in vacuum) with successful results. However, since the 'real' 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 [2, 3] 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. However, despite the absence of UPV measurements in this initial period (~12 hours), the wave velocity measurements of Figure 4b 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 c42.5 paste shows a greater increase in wave velocity, which is consistent with the results of the EMM-ARM that were already reported.

4.3 EMM-ARM and CC

The E-modulus evolution identified by the EMM-ARM method for two cement paste mixtures under study are shown in Figure 5. When comparing the results obtained with the two pastes containing the same w/c ratio (c42.5 and c32.5), it can be seen that the cement paste containing the CEM I 42.5R cement has a higher stiffness, with a difference of ~2 GPa at the age of 7 days (168 hours) - see Figure 5. However, even though the cement paste c42.5 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 c32.5. In fact, this would not be expected by strictly considering the chemical composition of the utilized cements, namely due to the higher C3S 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). 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.

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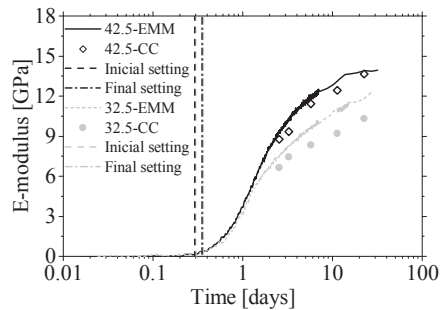22-24 August 2016, Technical University of Denmark, Lyngby, Denmark

Figure 5: Comparison of the results of EMM-ARM, Vicat, and cyclic compression for cement pastes c32.5 and c42.5.

The comparison between the elastic modulus results obtained by EMM-ARM and by classic methods (CC and Vicat) for the cement pastes c32.5 and c42.5 is shown in Figure 5. 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 c32.5 (Figure 5) show a non-negligible difference of 1.4 GPa at t = 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 [11], which may have significant effects in view of the small size of the specimen, thus resulting in lower stiffness. As the porosity of c32.5 is higher than that of c42.5, it is plausible that this deficient curing of CC specimens may have affected c32.5 more significantly, as opposed to c42.5. The results presented in Figure 5 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.

4.4 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 ('Norm' in Figure 6) by dividing all results of each specimen/methodology by their corresponding values at t = 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 [2].

The results of all experimental methods involved in this comparison is given in Figure 6, 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.

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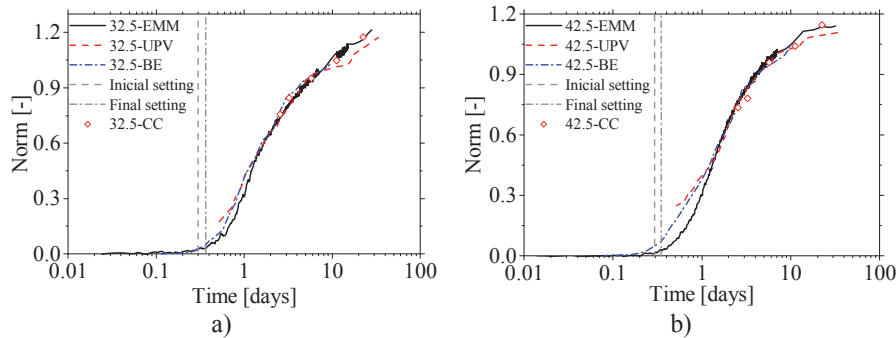22-24 August 2016, Technical University of Denmark, Lyngby, Denmark

Figure 6: Comparison of the results of all methodologies used in this study for cement pastes
a) c32.5 and b) c42.5.

In regard to setting times, there is also a good coherence between Vicat, EMM-ARM and BE, as observable in Figure 6. 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 [2, 10]. Despite this fact, the results obtained by these wave-propagation based methods should be regarded qualitatively, since these refer to dynamic properties, whose conversion to static properties is often arguable, particularly at very early ages, due to the evolution of Poisson's ratio during curing [12]. However, it should be noted that these wave velocity methods (BE and UPV) seem to exhibit a slightly more accelerated evolution kinetics than EMM-ARM, which is more evident for the c42.5 paste, as shown in Figure 6b. 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.

5 Conclusions

This paper has presented an experimental work devoted to a pilot experiment for application of Bender Extender elements for monitoring early age stiffness of cement pastes. This study involved two cement paste mixtures, which were tested through several techniques for benchmarking purposes: bender extender elements, EMM-ARM, compressive cyclic testing, ultra-sound pulse velocity and Vicat needle testing.
It was found that the application of bender-extender elements to cement paste is viable and provides relevant data at early ages, which relates well to the properties assessed through the other competing experimental techniques used in this work. Indeed, the bender extender elements were found to have special aptitude to assess the behaviour at very early ages through S-waves, with capacity to capture evolution of stiffness even before the setting period, with the best resolution among the techniques used in this research work. In spite of the capacity of bender-extender elements to be excited at variable frequencies, which offers better opportunities of increasing the amplitude of signals at all ages of testing, significant

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hurdles were identified in the assessment of wave velocity in view of the high noise-to-signal ratio.

The advantages and pitfalls observed for bender extender elements lead the authors to consider that this is a technique that is worth of further research.

Acknowledgments

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