Ultrasonic monitoring of setting and hardening behaviour of concrete and mortar with blast-furnace slag cement

N. Robeyst, E. Gruyaert, N. De Belie
Magnel Laboratory for Concrete Research, Ghent University, Belgium

Abstract: Blast-furnace slag cement is a blend of Portland clinker and blast-furnace slag, a by-product of the steel production. Since the slag has latent-hydraulic properties, the early-age behaviour of blast-furnace slag cement is different from Portland cement.
The setting of fresh mortar and concrete samples, made with Portland cement and four types of blast-furnace slag cement, was continuously monitored with the ultrasonic wave transmission method. A revised measurement setup with new sensors and a preamplifier improved the quality of the acquired signal at very early age. The evolution of the velocity and frequency spectrum of the ultrasonic wave was investigated and compared with the results of traditional methods, such as penetrometer tests.
The results lead to the conclusion that characteristic points in the graphs of the penetration resistance and the ultrasonic velocity curves are correlated.

1 Introduction

Blast-furnace slag, a by-product of the steel production, can partially replace Portland clinker in cement and thereby contribute to a sustainable cement industry. However, blast-furnace slag changes some of the properties of the fresh and hardening concrete or mortar leading for instance to a slower initial strength development, a decrease of the hydration heat [1], a longer setting and hardening time [2] and a great sensitivity to curing [3]. The influence of blast-furnace slag cement on the setting and hardening behaviour of concrete is investigated in this research project with the penetrometer and the ultrasonic wave transmission method. The latter allows continuously monitoring the setting and hardening behaviour of fresh concrete and is suitable to examine the effect of chemical admixtures and supplementary cementing materials on the evolution of the material stiffness [4, 5, 6].

2 Materials and methods

2.1 Mortar and concrete mixtures

To investigate the influence of blast-furnace slag cement on the setting and hardening behaviour of mortar and concrete, mixtures made of Portland cement (CEM I) were compared to mixtures with different types of blast-furnace slag cement (CEM III). According to EN 197-1, CEM III/A contains 36-65% blast-furnace slag by mass, CEM III/B 66-80% and CEM III/C 81-95%. The properties of the five cement types used in this study are summarized in Table 1. The mortar samples consisted of 1350 g standard sand, 450 g cement and 225 g water and were mixed according to EN 196-1. The concrete mixes contained 350 kg cement per m³ of concrete and the water/cement ratio amounted to 0.5 for all mixes.

Table 1: Properties of the cement types used in this study

	CEM I 42.5 N HSR*	CEM III/A 42.5 N	CEM III/B 42.5 N HSR*	CEM III/A 32.5 N	CEM III/C 32.5 N HSR*
Initial set (h :min)	4 :25	4 :40	6 :15	6 :00	4 :30
Final set (h :min)	5 :35	6 :00	8 :05	7 :25	5 :15
Strength 1d (N/mm²)	12	13	3	5	2
Strength 7d (N/mm²)	34	40	38	25	30
Strength 28 d (N/mm²)	54	52	51	44	40
Specific Surface (m²/kg)	315	485	450	345	317
Cl^- (%)	0.01	0.04	0.04	0.04	0.22
SO_3 (%)	1.8	3.1	3.2	3.1	2.2

Tests according to EN 196-3 (setting times - Vicat), EN 196-1 (strength) and EN 196-6 (specific surface - Blaine). The Cl^- and SO_3 content were specified by the producer.
(*) HSR = High Sulphate Resistance

2.2 Determination of initial and final setting times by traditional methods

To determine the initial and final setting times, current standard methods were used:

- test with the apparatus of Vicat (EN 196-3) on cement paste with a standard consistency
- test with the penetrometer (ASTM C403) on the mortar fraction of the concrete mixtures

For the latter, the wet concrete mixture has to be sieved with a sieve of 4.75mm, which is not practical for concrete compositions with a water/cement ratio of 0.5 and without plasticizers. Instead, the test was performed on an equivalent mortar mixture, calculated with the MBE method (Mortier de béton équivalent) [7]. In the MBE mixture, the gravel fraction is replaced by an amount of sand that has the same specific surface and the water amount is adjusted according to the difference in absorption between gravel and sand. The penetration tests were

performed twice for every cement type and the mean results are discussed in the following sections.
The results of the tests with the apparatus of Vicat are summarized in Table 1. They will not be discussed infra since it is difficult to compare tests on cement pastes with experiments on fresh mortar or concrete mixtures.

2.3 Ultrasonic wave transmission measurements

The ultrasonic wave transmission measurements on the hardening samples were performed with the new version of the FreshCon system developed at the University of Stuttgart (Fig. 1) [8].

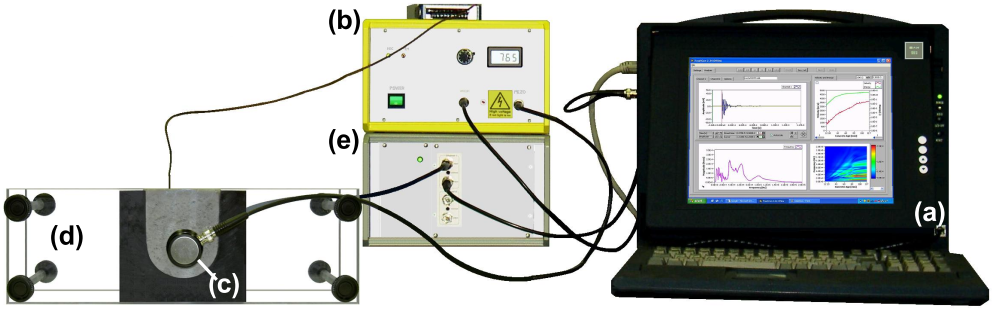

Fig. 1: Ultrasonic measurement setup (FreshCon): (a) computer with DAQ card, (b) amplifier, (c) piezoelectric sensor, (d) container, (e) preamplifier

A pulse signal is generated by the DAQ card of the FreshCon computer, of which the amplitude is increased by the amplifier (up to 800V). The ultrasonic wave is then sent through the fresh concrete or mortar sample with the aid of a piezoelectric broadband transmitter. After travelling through the hardening sample, the signal is received by the ultrasonic receiver and sent back to the DAQ card through a preamplifier. The first hours after mixing, the concrete is still a suspension through which ultrasonic waves cannot easily propagate. This results in a poorer quality of the received signal during this period and a less accurate determination of the signal onset time and thus wave velocity. However, in comparison with the previous system, the quality of the received signal at very early age was improved by the use of a reliable power amplifier on the transmitting side and a preamplifier on the receiving side. In Fig. 2 the signal/noise ratio versus concrete age for the reference concrete used in this study and for a concrete made of CEM I 42.5 considered in [5], are compared. The revised measurement set-up was also provided with new sensors, having a good linearity concerning the frequency response in the desired frequency range [8].
Every 5 minutes an ultrasonic wave was sent through the hardening sample. Before the experiments, the FreshCon device was calibrated to determine the time the ultrasonic signal needs to travel through the hardware, the sensors and the container walls. This time delay has to be

subtracted from the measured time to calculate the ultrasonic velocity in the concrete or mortar sample.

All the tests were conducted at a room temperature of 20°C. The FreshCon container was sealed with plastic film to allow cement hydration to proceed normally, and to avoid shrinkage of the mortar or concrete resulting in decoupling of the sample and the container walls. The mortar mixtures were tested twice for all cement types. No replications were carried out on the concrete mixtures.

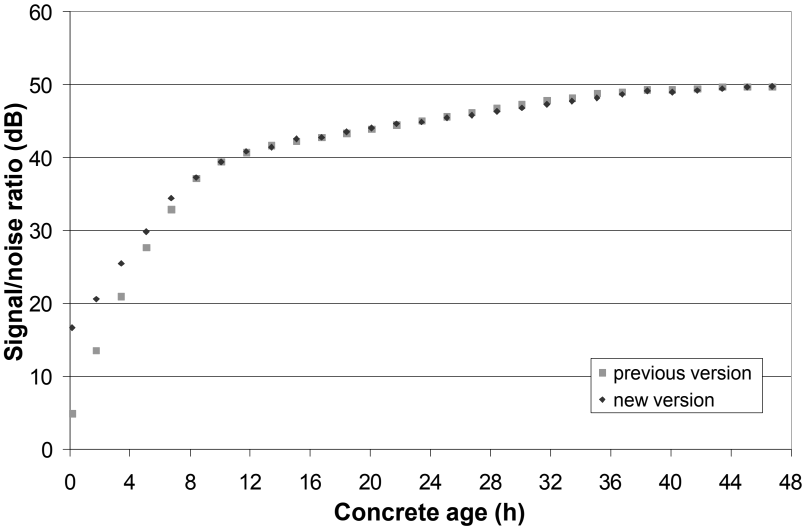

Fig. 2: Evolution of the signal/noise ratio in time for a concrete mixture with CEM I 42.5, measured with the previous [5] and the current version of the FreshCon.

During the experiments, the FreshCon software showed the received ultrasonic signals and their frequency spectrum (using an FFT-algorithm) online. Also the change in ultrasonic velocity and energy and the frequency content versus concrete age were represented. An offline version of the software allowed re-evaluating the data after the test, using different algorithms for picking the onset times of the signals.

2.4 Modelling of the velocity curves

The evolution of the velocity of an ultrasonic p-wave (compression wave) in fresh concrete is already discussed in several papers [5, 6]. By modelling the measured velocity curves mathematically, smooth curves can be presented in the following paragraphs which make the comparison between the different curves clearer. Moreover, characteristic points (e.g. point of inflection) can be determined accurately. Several disciplines such as biology or economics use logistic functions to describe quantities that grow exponentially at the outset after which the growth is gradually decelerated by feedback mechanisms, producing an S-shaped curve. As proposed by Grosse et al. [8], this growth model can also be used on the velocity curves (Eq. 1):

$$v(t)=\frac{v_1-v_2}{1+e^{(t-t_0)/dt}}+v_2 \qquad (1)$$

v_1 is the velocity at t = -∞, v_2 the velocity at t = ∞, t_0 the time corresponding with the center point between v_1 and v_2 and dt is the gradient at t_0. More details about these parameters can be found in [8]. An example of this model applied to the measurements on the concrete mixture with CEM III/A 42.5 is shown in Fig. 3a.
Grosse et al. [8] also proposed that a better fit can be obtained by replacing the exponential function by a power function (Eq. 2):

$$v(t)=\frac{v_1-v_2}{1+(t/t_0)^p}+v_2 \qquad (2)$$

In this formula the power p replaces the gradient dt and v_1 is the initial velocity at t = 0 instead of t = -∞. This model fits the measured velocity better, but the shape of the two curves still do not fully correspond (Fig. 3b).

Many growth processes consist of several subprocesses. These processes can be modelled using bi-logistic or multi-logistic functions [9] (Eq. 3):

$$v(t)=\sum_i \frac{k_i}{1+e^{(t-t_i)/dt_i}}+c \qquad (3)$$

This formula is a generalization of Eq. (1) and superposes two or more logistic functions with different gradients, points of inflection and end values. Since the setting of concrete is the result of several physical and chemical reactions, the multi-logistic model was also tried out on the velocity curves. It is able to fit the measured velocity curves very accurately (Fig. 3c and d) and will be used in the following part of the paper. The disadvantage of this model is the use of more parameters of which the physical meaning is less clear as with Eq. (2). The latter seems therefore better suited for a classification of the measurements given a few simple parameters.

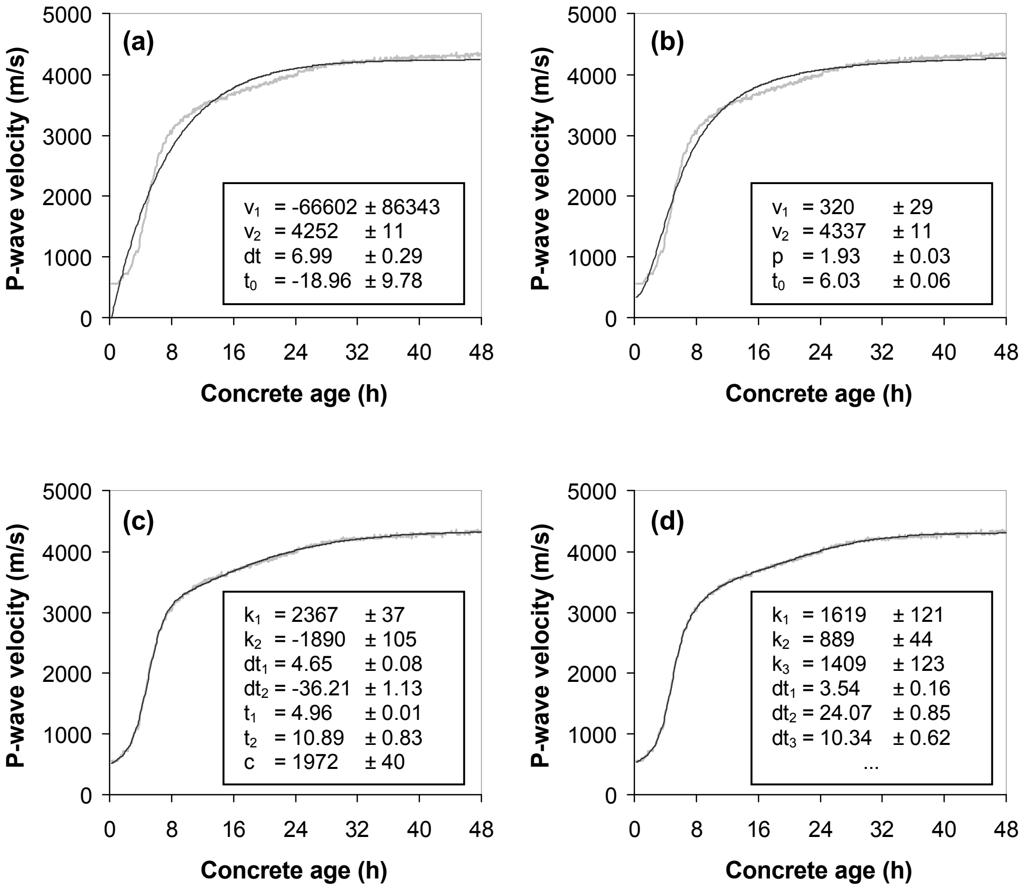

Fig. 3: Modelling of the velocity curves with (a) logistic, (b) power logistic, (c) bi-logistic and (d) multi-logistic functions (concrete with CEM III/A 42.5)

3 Results and discussion

3.1 Ultrasonic velocity

3.1.1 Discussion of the velocity curves

The ultrasonic velocities for the mortar mixes made of Portland and blast-furnace slag cement are presented in Fig. 4. In all the velocity curves more or less the same parts can be distinguished. The first part is the dormant period, characterised by a constant velocity value. During this period the determination of the signal onset time and thus wave velocity is still less accurate and therefore no significant difference in the dormant period can be observed between the tested mortar compositions, except for CEM III/C.

After the dormant period that lasts approximately 3 hours, the velocity increases very rapidly at first (second stage) and then more gradually (third stage). For cements of the strength class 42.5, the stiffness of the mortar sample seems to develop slower with increasing blast-furnace slag content. However, for the strength class 32.5, during the first 12 hours the velocity increases faster in the mortar made with CEM III/C in comparison to the mortar with CEM III/A. This might be due to the higher chloride content of CEM III/C in comparison to CEM III/A (Table 1). According to Moranville-Regourd [1], chlorides act as accelerating agent. CEM III/A also contains a higher amount of SO_3 (Table 1), possibly leading to a slower hydration in the beginning.

Thereafter, the velocity curve of the mixture with CEM III/C levels off, while the curve of the CEM III/A continues to increase. However, after approximately 26 hours, the ultrasonic velocity of the mortar containing CEM III/C shows a second steep increase that might be caused by the reaction of the slag accounting for the major part of the cement composition. This hypothesis will be investigated more thoroughly in the future.

Fig. 4: Ultrasonic velocity vs. mortar age for Portland cement and blast-furnace slag cement mortar (result of two replications)

In the last stage, the p-wave velocity reaches an asymptotic value. This stage was only observed for the mortar made of CEM I 42.5 and CEM III/A 42.5 within the measuring period of 42 hours. For the other mortar mixes, the velocity still increases significantly.

The results of the ultrasonic velocity measurements on the concrete samples lead to the same conclusions (Fig. 5) and the second steep increase in the mixture with CEM III/C is also noticed (Fig. 5e).

3.1.2 Comparison between ultrasonic velocity and penetration resistance

Fig. 5 gives the comparison between the ultrasonic measurements performed on concrete and the penetration resistance tests performed on the equivalent mortar mixture calculated with the MBE method. The initial and final setting times according to ASTM C403 are indicated and are determined by a penetration resistance of respectively 3.5 N/mm² and 27.6 N/mm². The latter corresponds quite well with the beginning of the third stage of the velocity curves, namely the slow increase in velocity (CEM I 42.5: 8.83h, CEM III/A 42.5: 8.83h, CEM III/B 42.5: 13.27h, CEM III/A 32.5: 12.82h, CEM III/C 32.5: 11.93h).

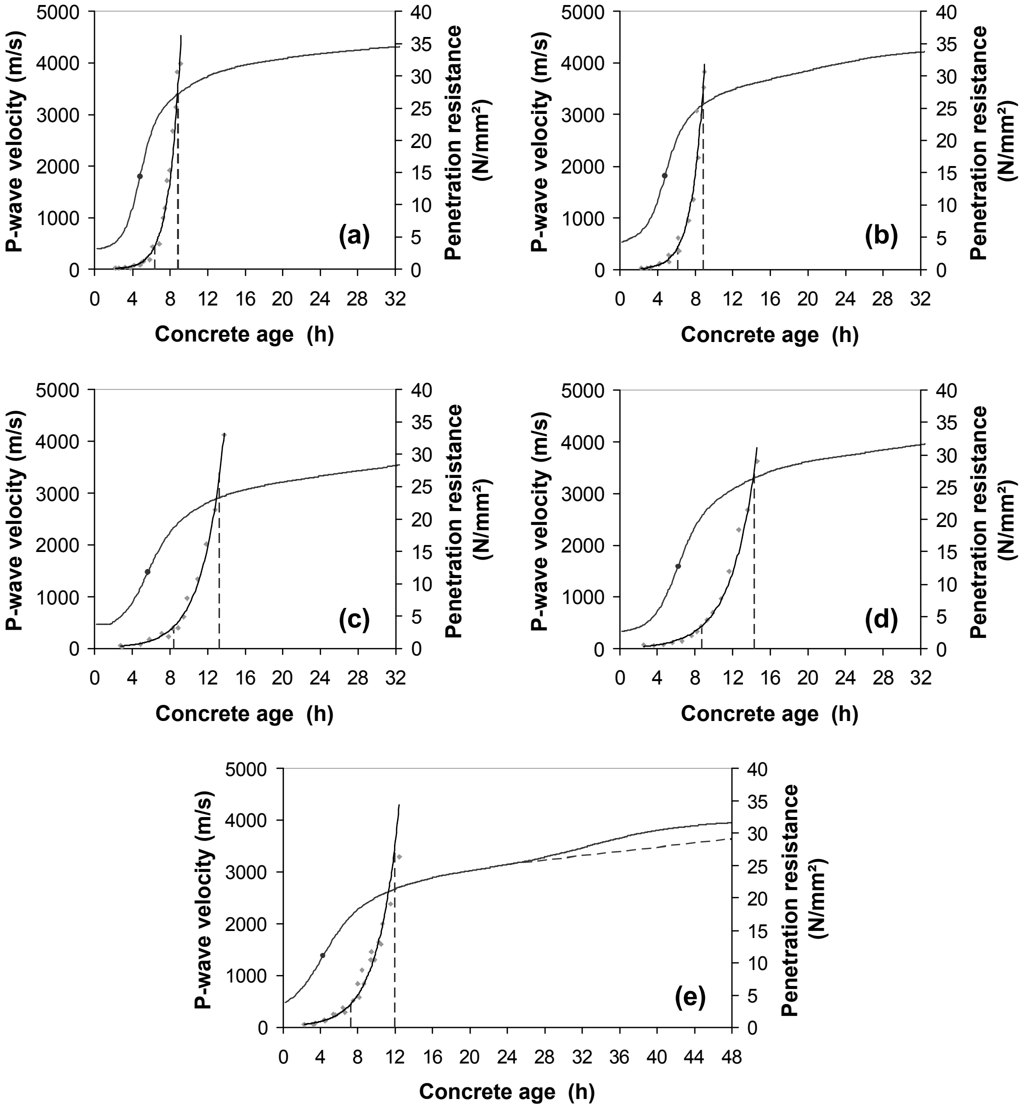

Fig. 5: Comparison between p-wave velocity and penetration resistance of the concrete mixtures with (a) CEM I 42.5, (b) CEM III/A 42.5, (c) CEM III/B 42.5, (d) CEM III/A 32.5 and (e) CEM III/C 32.5. The inflection point (·) and the initial and final setting times according to ASTM C403 (- - -) are indicated.

For all the mixtures, the ultrasonic velocity starts to increase earlier than the penetration resistance. According to Voigt et al. [10] this can be attributed to two reasons. Firstly, ultrasonic waves propagate not only through solid material but also travel through gases and liquids. An increase in velocity without increase in penetration resistance corresponds with the formation of hydration products which have no or little influence on the stiffening process and thus on the penetration resistance. They fill pore space but create no connected particles. Only connections between cement grains caused by hydration products influence the penetration resistance. A second reason can be internal settling processes in the concrete, mainly caused by gravity. The particles have a better mechanical coupling without a real bond (no increase in penetration resistance) and the ultrasonic velocity increases.

The time at which the penetration resistance increases (CEM I 42.5: 4.84h, CEM III/A 42.5: 4.57h, CEM III/B 42.5: 5.56h, CEM III/A 32.5: 5.52h, CEM III/C 32.5: 4.28h), corresponds well with another characteristic point in the velocity versus concrete age plot, namely with the point of inflection (CEM I 42.5: 4.82h, CEM III/A 42.5: 4.81h, CEM III/B 42.5: 5.69h, CEM III/A 32.5: 6.22h, CEM III/C 32.5: 4.29h). This conclusion was also reported by Voigt et al. [10].

In previous work [5], it was suggested that the point of inflection in the curve corresponded to a velocity of approximately 1500m/s. According to the tests performed on these concrete mixtures, the point of inflection is located at 1600 m/s (standard deviation = 190 m/s).

3.2 Frequency spectrum

Besides the ultrasonic velocity, the FreshCon software allows monitoring the evolution of the frequency content in time. The frequency spectra were calculated with an FFT-algorithm and the results for concretes made of Portland and blast-furnace slag cement are presented in Fig. 6.

In the beginning of the measurement, when the concrete is still a fluid-like suspension, only weak low frequencies can be detected. High frequencies are completely damped in a liquid and gradually appear when the concrete stiffens. The first 3-4 hours no significant changes are noticed in the spectra, which corresponds with the dormant period mentioned above. The frequency spectrum evolves faster to a broader spectrum for the Portland cement concrete and evolves slower with increasing blast-furnace slag content for concretes of strength class 42.5. This evolution of the frequency bandwidth in time can be compared with the evolution of the velocity in time.

After a few hours (7 to 12 hours according to the cement type), a clear peak frequency of 11 kHz becomes visible, which is probably the resonance frequency of the system. The other frequencies with a high magnitude shift in time and follow an S-shaped curve. The graph of the frequencies for the mixture with CEM III/C looks different from the other mixtures, not showing a clear S-shape in the evolution.

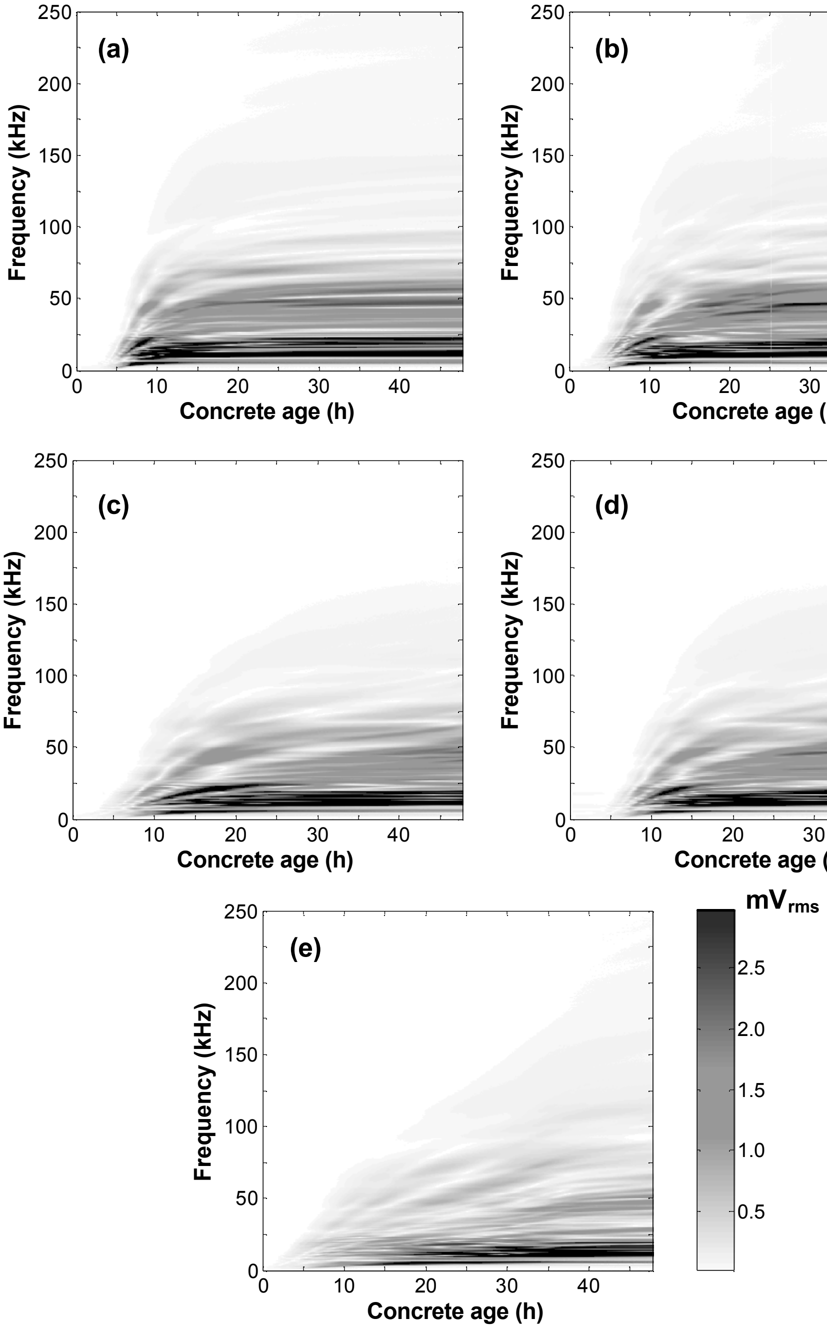

Fig. 6: Frequency content versus frequency and concrete age of the transmitted ultrasonic signal for concrete mixtures with (a) CEM I 42.5, (b) CEM III/A 42.5, (c) CEM III/B 42.5, (d) CEM III/A 32.5 and (e) CEM III/C 32.5

4 Conclusions

Ultrasonic measurements and penetrometer tests were used to monitor the setting and hardening behaviour of concrete and mortar with blast-furnace slag and Portland cement.

The following conclusions can be drawn:

1. The velocity curves can be modelled with logistic functions. The use of a single logistic function leads to clear parameters with a physical meaning. However, multi-logistic functions can fit the measured data more accurately.
2. For cements of the same strength class, the increase of the ultrasonic velocity is generally retarded by increasing blast-furnace slag content. Other cement characteristics, such as strength class and chemical composition also showed an influence. For high replacement percentages of clinker by blast-furnace slag (CEM III/C) a second steep increase in the velocity can be detected after approximately 26 hours.
3. When the ultrasonic velocity starts to increase, the first hydration products are formed, which have no or little influence on the stiffening process. Only after a time, when the cement particles are connected, the penetration resistance starts to develop. This time corresponds with the inflection point of the velocity curve within a time window of 15min for 4 of the 5 mixtures. Another reason for the earlier increase in p-wave velocity in comparison to the penetration resistance can be internal settling processes, caused by gravity.
4. The frequency spectra broaden gradually in accordance with the stiffening process. The bandwidth of the received signal increases slower with increasing blast-furnace slag content for the strength class 42.5.

Acknowledgements

Elke Gruyaert and Nicolas Robeyst are Research Assistants of the Research Foundation – Flanders (FWO – Vlaanderen) and want to thank the foundation for their financial support.

References

[1] M. Moranville-Regourd, Cements Made From Blastfurnace Slag, in: P.C. Hewlett (Ed.), Lea's chemistry of cement and concrete (4th ed.), Arnold, London, 1998, pp. 633-674

[2] S. Kosmatka, B. Kerkhoff, W. Panarese, Design and Control of Concrete Mixtures (14th edition), Portland Cement Association, Skokie, 2002

[3] C.F. Hendriks, A.A. Nijkerk, A.E. Koppen, De Bouwcyclus (The Construction Cycle), Aeneas, Boxtel, 1999

[4] N. De Belie, C.U. Grosse, J. Kurz, H.-W. Reinhardt, Ultrasonic monitoring of the influence of different accelerating admixtures and cement types for shotcrete on setting and hardening behaviour, Cement and Concrete Research 35 (2005) 2087-2094

[5] N. De Belie, C. Grosse, N. Robeyst, E. Gruyaert, Evaluation of different ultrasonic transmission parameters to monitor microstructure development in fresh concrete or mortar, in: J. Marchand, B. Bissonnette, R. Gagné, M. Jolin, F. Paradis (Eds.), Proc. Second International Symposium on Advances in Concrete through Science and Engineering (abstract book, full paper on CD-ROM), Quebec, 2006, pp. 313 – 314

[6] H.W. Reinhardt, C.U. Grosse, Continuous monitoring of setting and hardening of mortar and concrete, Construction and Building Materials 18 (2004) 145-154

[7] A. Schwartzentruber, C. Catherine, La méthode du mortier de béton équivalent (MBE) – Un nouvel outil d'aide à la formulation des bétons adjuvantés, Matériaux et Constructions 33 (2000) 475-482

[8] C.U. Grosse, H.-W. Reinhardt, M. Krüger, R. Beutel, Ultrasonic through-transmission techniques for quality control of concrete during setting and hardening, in: H.-W. Reinhardt (Ed.), Proc. Advanced testing of fresh cementitious materials, Stuttgart, 2006, pp. 83-93

[9] P.S. Meyer, J.W. Yung, J.H. Ausubel, A primer on Logistic Growth and Substitution: The Mathematics of the Loglet Lab Software, Technological Forecasting and Social Change 61 (3) (1999) 247-271

[10] T. Voigt, C.U. Grosse, Z. Sun, S.P. Shah, H.-W. Reinhardt, Comparison of ultrasonic wave transmission and reflection measurements with P- and S-waves on early age mortar and concrete, Materials and Structures 38 (2005) 729-738