Ultrasound monitoring of the influence of pozzolanic additions and accelerating admixtures on setting and hardening behaviour of concrete and mortar

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Abstract

The sensitivity of transmitted ultrasound signals to accelerator type (alkaline aluminate or alkali-free) and dosage were evaluated. A stepwise increase of the accelerator dosage resulted in increasing values for the ultrasound pulse velocity at early ages. In the accelerated mortar no dormant period could be noticed before the pulse velocity started to increase rapidly, indicating a quick change in solid phase connectivity. The alkaline accelerator had a larger effect than the alkali-free accelerator, especially at ages below 90 minutes. The increase of ultrasound energy could be related to the setting phenomenon and the maximum energy was reached when the end of workability was approached. Only the alkaline accelerator caused a significant reduction in compressive strength and this for all the dosages tested.
Furthermore, the properties in the fresh and hardening state of concrete with 0%, 10%, 40%, or 60% replacement of cement by fly ash were determined. The ultrasonic pulse velocity changed in all mixes from 200-500 m/s to 4000-5000 m/s, but at a different rate. An increase in fly ash content resulted in a longer dormant period, decreasing values for the pulse velocity at early ages and an increase of the time necessary to asymptotically reach the maximum velocity (from 20 h for the reference to more than 47 h for the 60% fly ash mix). The final setting time, defined by an ultrasound velocity of 1500 m/s also increased significantly with increasing fly ash content. This point seemed to correspond to the point of inflection of the ultrasound velocity curve.

Introduction

Non-destructive techniques, such as ultrasonic pulse velocity measurements, allow to monitor microstructural changes of concrete at early age in a continuous way, in contrast to conventional methods such as the Vicat test (EN 196-3) or the penetration resistance method (ASTM C 403). The possible use of ultrasound measurements for monitoring setting and hardening of concrete containing different accelerating admixtures or with different cement by fly ash replacement levels was investigated.

Materials and methods

Mortar with accelerating admixtures

Ultrasound measurements were used to investigate the performance of different accelerating admixtures for shotcrete. The aim was to determine the sensitivity of ultrasound measurements to changes in accelerator type and dosage.

To allow accurate measurements, starting rapidly after the mix preparation, the experiments were performed on mortar. Also the more traditional methods for monitoring concrete setting are mainly performed on cement paste or mortar.

The reference mortar consisted of 1350 g standard sand according to EN 196-1 [1], 450 g Portland-limestone cement CEM II/A-LL 42.5 R including 6-20 % limestone according to EN 197-1 [2] and 225 g water. Portland-limestone cement is sometimes suggested as an alternative to Portland cement for shotcrete applications in Germany. The tested accelerators included an alkaline aluminate based solution (AlA) and an alkali-free solution based on aluminium sulphate (AlS). Disadvantages of the traditional alkali-rich accelerators are a significant reduction of the ultimate strength (typical values for strength reduction at 28 days range between 20-50%) [3] and the health hazards associated with handling alkalis. A new generation of alkali-free accelerators was therefore introduced for the improvement of the mechanical parameters, working conditions, safety, lower environmental impact and easier maintenance of existing tunnel facilities [4]. The tested accelerator dosages were 0.5, 0.75 or 1 time the maximum allowable dosage of 50 ml accelerator per kg cement. The different mortar mixes are coded as follows:

- II (CEM II/A-LL 42.5 R)
- AlA (alkaline aluminate based solution) or AlS (alkali-free solution based on aluminium sulphate)
- number representing the accelerator dosage relative to the maximum allowable dosage of 50 ml accelerator per kg cement (0, 0.5, 0.75 or 1)

The mortar was prepared in accordance with EN 480-1 [5]. Then the accelerator was added and the mix procedure was concluded with 5 s mixing at low speed and 5 s at high speed. The FreshCon container for mortar and three moulds for mortar prisms with dimensions 40 x 40 x 160 mm were filled and the mortar was compacted for about 15 to 60 s on a vibrating table. The vibration time was limited in order not to hamper the setting process. The ultrasound measurements were started within 2 minutes after addition of the accelerator to the mix. The mortar prisms were stored for 24 h in sealed moulds at 20°C. After demoulding they were stored under water at 20°C for 28 days.

Concrete with fly ash

Four different concrete mixes, containing 400 kg cementitious material (CM) per m³ of concrete, were made for the ultrasonic pulse velocity measurements. In the reference mix (ref) the binder consisted solely of an ordinary Portland cement CEM I 42.5 R with characteristics according to the ENV 197-1 [6]. In the three other mixes 10%, 40% or 60% of the cement was replaced with the same amount of fly ash (by weight). The fly ash used was a residue from the combustion of pulverised coal in a thermal power plant furnace in Ghent. It is characterised by the activity index, i.e. the ratio of the compressive strength of standard mortar bars prepared with 75% reference cement plus 25% fly ash by mass, to the compressive strength of standard mortar bars prepared with reference cement alone. The fly ash had an activity index of 79% at the age of 28 days and 98% at 90 days, indicating a high pozzolanic activity. The water/cementitious material ratio (W/CM) amounted for all mixes to 0.3. A superplasticizer of the new polymer family with high molecular weight was used (sulphated naphthalene methanale condensate). Although the maximum amount of superplasticizer recommended by the producer was applied, the mixes showed a low workability (flow class F0-F1 according to NBN 15-233 [7]). An overview of the mix proportions is presented in Table 1. The ultrasound measurements could be started about half an hour after addition of the mixing water.

Table 1: Concrete composition

Mix	Mix proportions (kg/m³)							W/CM (-)	W/C (-)
	Gravel 4/8	Natural sand 0/2	Natural sand 2/4	CEM I 42.5 R	Fly ash	Water	Super plasticizer (l/m³)		
ref	1150	600	150	400	0	120	6	0.3	0.30
FA10	1150	600	150	360	40	120	7	0.3	0.33
FA40	1150	600	150	240	160	120	8.5	0.3	0.50
FA60	1150	600	150	160	240	120	10	0.3	0.75

Ultrasound wave transmission measurement

The ultrasound device used was the FreshCon developed at the University of Stuttgart [8-10]. The sample container consists of two polymethacrylate (PMMA) walls which are tied together with four screws with spacers. The mould is a U-shaped rubber foam element with high damping properties, suppressing waves from travelling through the mould and thus around the mortar. Separate containers are available for concrete and mortar, with a sample volume of approximately 35 cm³ and 450 cm³, respectively.
At one side of the mould a pulse was generated using a broadband frequency generator (Hameg), an amplifier (Develogic) and an ultrasound transmitter (Vallen VS 30). After travelling through the mortar sample in the mould, the signal was recorded at the other side by an ultrasound receiver (Vallen VS 30). To be able to capture the rapid changes in the mortar with accelators, a small recording interval of 0.5 min was used during the first half hour, followed by a recording interval of 2 to 5 minutes. For the fly ash concrete, ultrasound measurements were taken every 10 minutes.
Before the experiments, the FreshCon device was calibrated to determine the time the ultrasound 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 ultrasound velocity in the concrete or mortar sample. Furthermore, the ultrasound energy determined by numerical integration of the squared amplitude values following the onset time, is divided by the reference energy and presented as a dimensionless value. This reference energy was also measured during the calibration.
All the tests were conducted at a room temperature of 20°C. The FreshCon container was sealed with plastic tape to allow cement hydration to proceed normally, and to avoid as much as possible shrinkage of the mortar resulting in decoupling of mortar and container walls. During the experiments, the FreshCon software showed the received ultrasound signals and their frequency spectrum (using an FFT-algorithm) online during the experiment. Also the change in ultrasound 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. The repeatability error of the measured velocity curves was determined to be approximately 1% [8].

Results and discussion

Mortar with accelerating admixtures

The results for ultrasound velocity and energy are shown in Fig. 1.

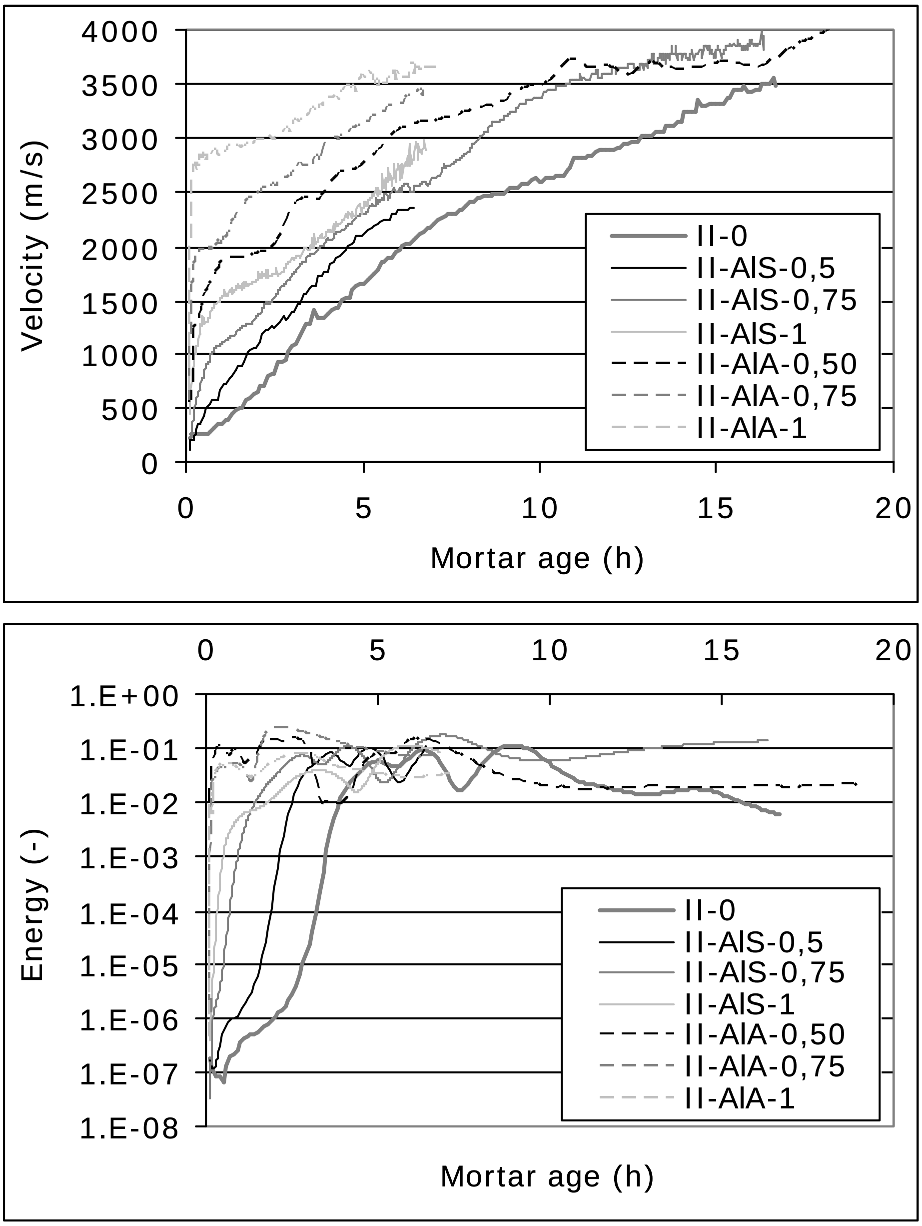

Fig. 1. Ultrasound velocity (top) and energy (bottom) vs. mortar age in function of accelerator type and dosage

In all tested mixes the ultrasound velocity evolves from 100 to 700 m/s at a mortar age of 6 min (this is 6 minutes after adding the water to the mix, thus 2 minutes after accelerator addition) to about 4000 m/s at later ages. The ultrasound measurements are clearly sensitive to the effect of accelerator type and dosage on the binding and hardening behaviour of the mortar. A stepwise increase of the accelerator dosage resulted in increasing values for the pulse velocity at early ages. While non-accelerated mortar showed a dormant period of about 30 min before the pulse velocity started to increase rapidly, no such threshold could be noticed in the accelerated mortar. For Portland cement concrete, the point where the pulse velocity starts to increase suddenly, can be indicated as a threshold of solid percolation [11].

The cement hydrates then form a complete path of connected particles for the ultrasonic pulse wave, as simulated with the HYMOSTRUC microstructural model [12]. We found this dormant period to be around 30 min long in non-accelerated mortar, and non-existing (or shorter than the 2 min interval between accelerator addition and start of measurements) in accelerated mortar. The following quick increase of pulse velocity is caused by the quick change in connectivity of the solid phase. When all particles are connected, the slow increase in pulse velocity follows the evolution of the total solid fraction of the paste.
The alkaline accelerator *AlA* had an even more pronounced influence on the microstructure development than the alkali-free accelerator *AlS*, especially at ages below 90 min. Mortar accelerated with *AlA* was characterised by a very steep increase in pulse velocity during the first 15-30 min after which the velocity curve levelled off. For mortar with alkali-free accelerator *AlS* the velocity curve evolved more smoothly. [4] mentions that the slightly longer setting times of the alkali-free admixture, compared to the alkali-rich admixture, appear to be favourable for the shotcreting efficiency. Due to the higher plasticity of the cementitious mass a better adhesion onto the tunnel wall is achieved, whereas the very fast setting attained by the alkali-rich admixture promotes a fast hardening of the cementitious mass, which in contact with the tunnel wall is easily rebounded.
According to prEN 934-5 [13] a requirement for sprayed concrete set accelerating admixtures is that the final setting time determined on reference mortar should be less than or equal to 60 min. Practical experience showed that the final setting time could be defined by an ultrasound velocity of 1500 m/s [8]. The ultrasound velocity at 60 min is for the different experimental mixes presented in Fig 2. If a velocity of 1500 m/s is taken to indicate the final setting time, the maximum dosage of alkali-free accelerator in combination with CEM II would just fulfil the requirements, while lower dosages would not be sufficient. For the alkaline accelerator even a dosage of half the maximum amount would suffice.

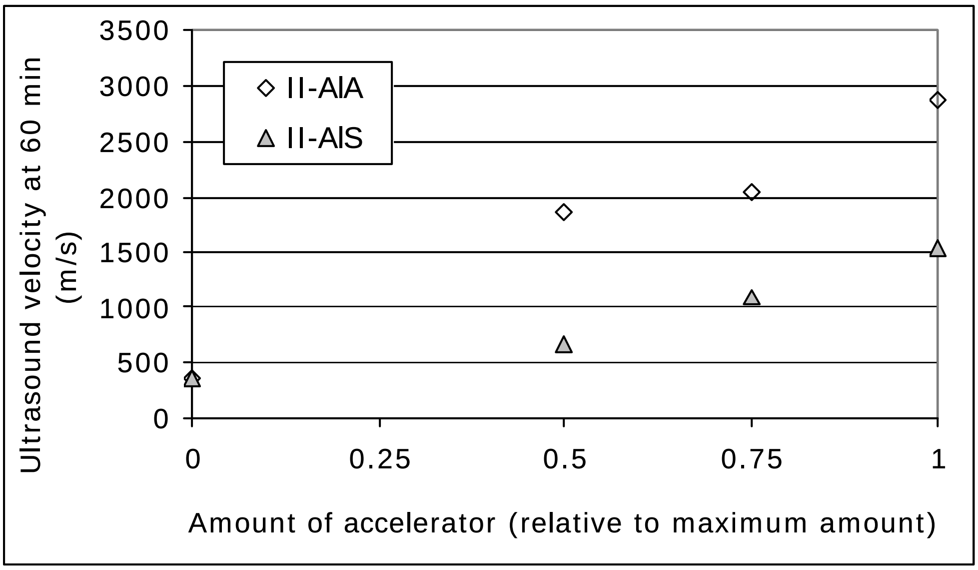

Fig. 2. Ultrasound velocity at 60 min for the different experimental mixes

The energy curves all showed the same pattern: an increase from 10^{-6}-10^{-7} to 10^{-2}-10^{-1}. The following 3 local maxima are caused by the characteristics of the sensors, and do not contain relevant information. The rate of energy increase depended on accelerator type and dosage, and based on the energy curves the same classification of mixes could be made as based on the velocity. The energy change of the transmitted ultrasound wave is a parameter that has been less discussed in literature, mostly because it was difficult before to reproduce the energy of the ultrasound emitter. The first local maximum in the energy curve is reached on average 20 min after the age at which a velocity of 1500 mm/s is reached. Therefore we could hypothesise that the energy is related to the setting phenomenon and that the maximum is reached when the end of workability is approached.

Fig. 3 shows the effect of accelerator type and dosage on the compressive strength of the mortar prisms at 28 days of age. For the dosages tested only the alkaline accelerator *AlA* caused a significant reduction in the compressive strength in comparison with the reference without accelerator (t-test with level of significance p = 0.01). The strength reduction was as high as 55% when the maximum amount of accelerator was applied. For the mix with the alkali-free accelerator the decrease in compressive strength in comparison with a non-accelerated reference mortar was not statistically significant. Similar conclusions were made by [3] and [14]. [3,15,16] mention a decrease in 28 day compressive strength of 20-25% when an alkali aluminate based accelerating admixture is applied. In our study the strength reduction was even larger. This could be due to an increase in void volume in our specimens (the mortar density decreased significantly e.g. with about 3% for the II-*AlA*-1 mix), which was not experienced by [3]. A decrease in density of shotcrete with aluminate based admixtures was also described by [17], who suggested that the flash-setting of shotcrete indicated a certain loss in self-compaction ability. We experienced indeed for some of the mixes with high accelerator dosage, that by the time that the moulds of the mortar prisms were filled, further compaction became very difficult, since the setting process had already started. However, this does not necessarily imply that the same density decrease will occur in the field, since in practical applications the shotcrete projected with high velocity on the substrate.

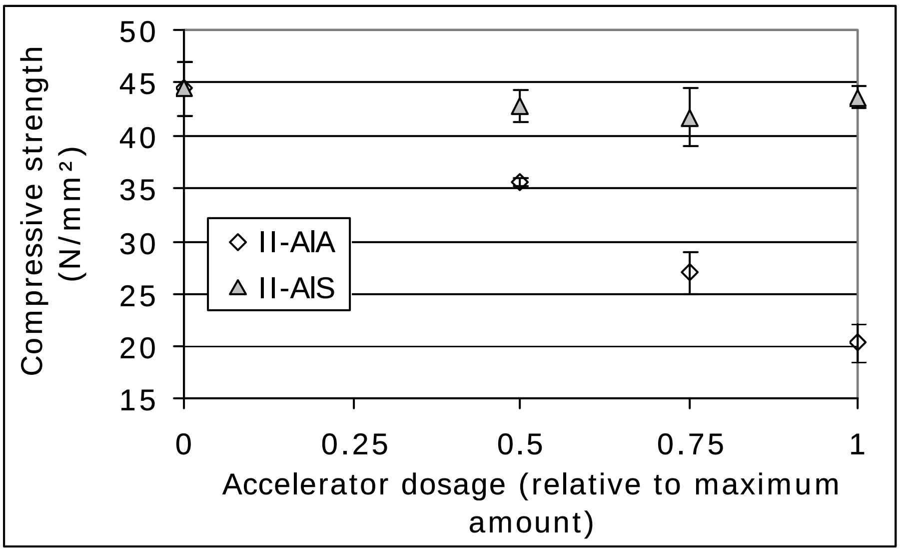

Fig. 3. Effect of accelerator type and dosage on the compressive strength of mortar prisms at 28 days (n = 6)

Further results on the effect of accelerators on mortar with different cement types, can be found in [18].

Concrete with fly ash

In all tested mixes the ultrasound velocity evolved from 200-500 m/s in the fresh mix to about 4000-5000 m/s at later ages (Fig. 4). It is clear that the ultrasound measurements are sensitive to the effect of fly ash replacement on the binding and hardening behaviour of the concrete. An increase in fly ash content resulted in a longer dormant period, decreasing values for the pulse velocity at early ages and an increase of the time necessary to asymptotically reach the maximum velocity. The percolation threshold increased from about 1-2 h for the reference mix, to 2-3, 3-4 and 7-8 h for the mixes with 10%, 40% and 60% fly ash respectively. After the dormant period, a quick increase of pulse velocity follows, due to the quick change in connectivity of the solid phase. Afterwards, the slow increase in pulse velocity follows the evolution of the total solid fraction of the paste. The maximum pulse velocity was reached after about 20, 22, 40 and more than 47 hours for the mixes with increasing fly ash content.

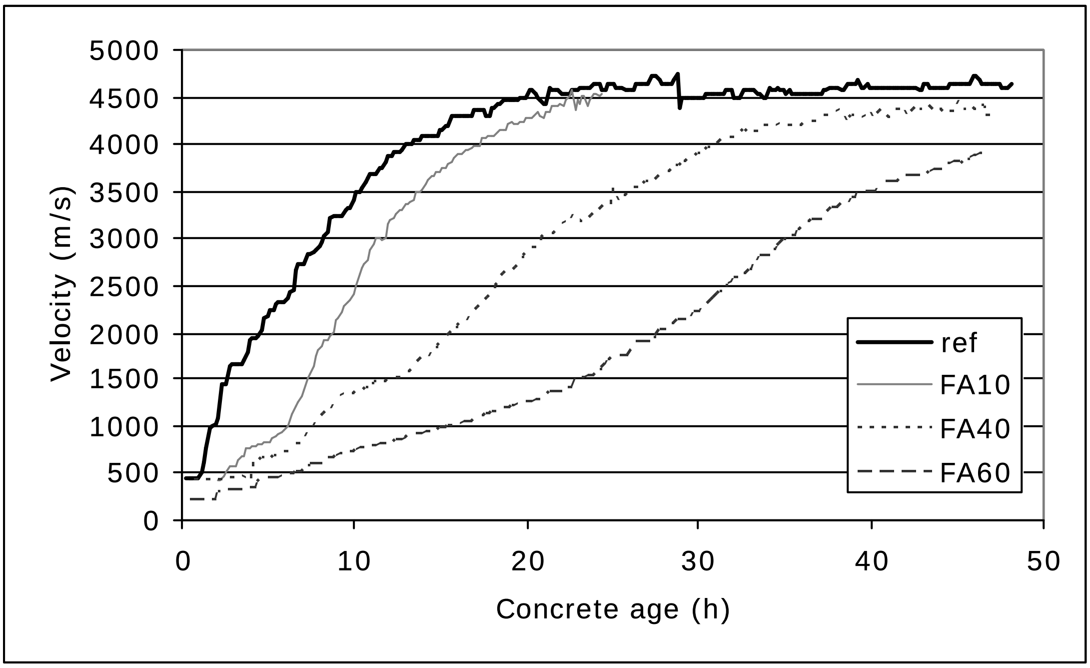

Fig. 4. Ultrasound pulse velocity for concrete types with different fly ash content

For concrete with additions the relation between ultrasound velocity and final setting time has not yet been clearly defined. For the concrete types ref, FA10, FA40 and FA60, an ultrasound velocity of 1500 m/s (defined as the final setting time in Portland cement concrete) was reached after 3, 7, 12 and 23 h, respectively. For the different mixes, this point is in the neighbourhood of the point of inflection of the ultrasound velocity curve (although exact localisation of this point is difficult), which could lead to the hypothesis of a fundamental relation between this point and the final setting time.

The energy curves all showed the same pattern: increase from about 10^{-2} to 10^{+4} (Fig. 5). The rate of energy increase depended on the level of fly ash replacement, and based on the energy curves the same classification of mixes could be made as based on the velocity. The maximum energy was reached between the final setting time (defined by a pulse velocity of 1500 m/s) and the final velocity. Further research is necessary to find out the physical meaning of the time at which the energy maximum is reached and to establish the relation with concrete microstructure and workability.

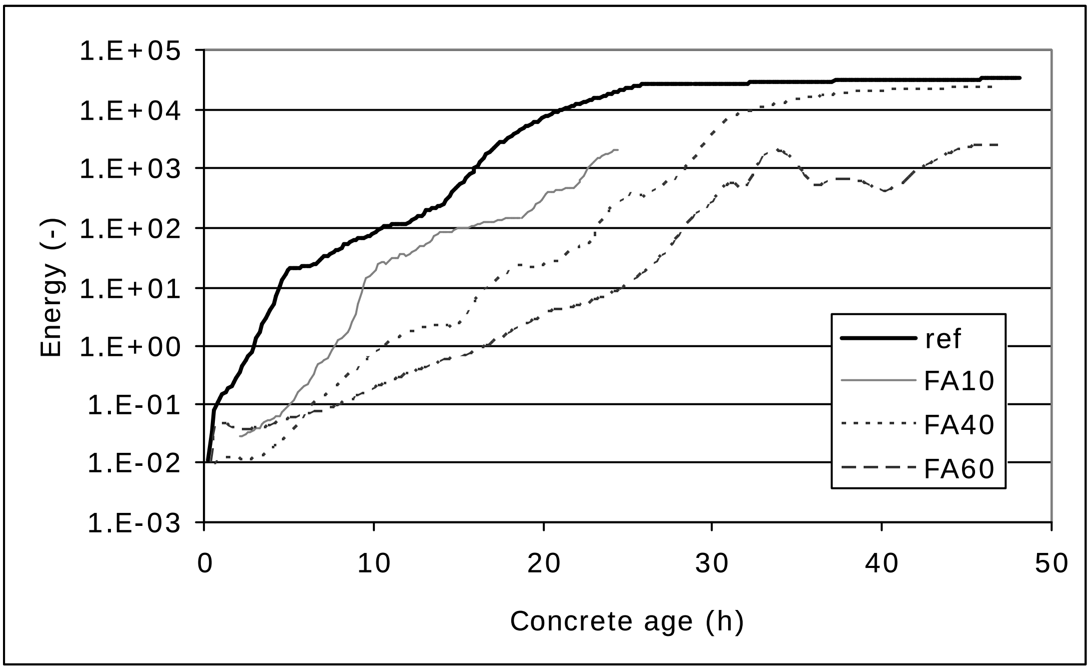

Fig. 5. Relative energy transmission for concrete types with different fly ash content

The hardened concrete was removed from the FreshCon after the measurements and stored at room temperature and non-controlled relative humidity. To illustrate the quality of the samples on which ultrasound measurements were performed, the compressive strength at an age of 6 weeks was determined on cores of 50 mm diameter and 47 mm height. At the considered age, only a replacement of 60% of the Portland cement by fly ash, resulted in a consistently lower compressive strength (45 N/mm² compared to 70 N/mm² for the other 3 mixes).

Conclusions

Ultrasound transmission measurements can be used to assess the effect of accelerators and additions on the setting and hardening behaviour of mortar and concrete.

A stepwise increase of accelerator dosage resulted in increasing values for the pulse velocity at early ages. While non-accelerated mortar showed a dormant period of about 30 minutes before the pulse velocity started to increase rapidly (related to the quick change in connectivity of the solid phase), no such threshold could be noticed in the accelerated mortar. The alkaline accelerator had a larger accelerating effect on the microstructure development than the alkali-free accelerator, especially at ages below 90 minutes. The increase of ultrasound energy could also be related to the setting phenomenon and the maximum energy was reached when the end of workability was approached. For the dosages tested, only the alkaline accelerator caused a significant reduction in compressive strength in comparison with the reference without accelerator.

An increase in fly ash content resulted in a longer dormant period, decreasing values for the pulse velocity at early ages and an increase of the time necessary to asymptotically reach the maximum velocity. The final setting time, defined by an ultrasound velocity of 1500 m/s also increased significantly with increasing fly ash content. This point seemed to correspond to the point of inflections of the ultrasound velocity curve. The maximum energy was reached after the final setting time, but before the maximum pulse velocity was obtained. Further research is necessary to establish the physical meaning of this moment.

A new FreshCon system with improved capabilities is available now and will be used for further experiments. The quality of the received signal is improved, in comparison with the system used for the experiments described above, by introducing a new amplifier allowing a higher input energy (up to 800V). Also the signal to noise ratio is increased by a preamplifier. The sensors used, provide a more constant transfer function, eliminating the fluctuations at the end of the energy curves. Since these sensors are sensitive for a broader frequency range and less selective, they are also better suited to monitor the changes in the frequency spectrum.

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References

[1] EN 196-1, Methods of testing cement – Part I: Determination of strength.

[2] EN 197-1, Cement – Part I: Composition, specifications and conformity criteria for common cements, 2000

[3] Prudêncio, L.R.Jr., ‘Accelerating admixtures for shotcrete’, Cement and concrete composites 20 (1998) 213-219.

[4] Paglia, C., Wombacher, F. and Böhni H., ‘The influence of alkali-free and alkaline shotcrete accelerators within cement systems. I. Characterization of the setting behaviour’, Cement and concrete research 31 (2001) 913-918.

[5] EN 480-1, ‘Admixtures for concrete, mortar and grout – test methods’ (1997).

[6] EN 197-1, ‘Cement – Part I: Composition, specifications and conformity criteria for common cements’, (2000).

[7] NBN B15-233, ‘Fresh concrete – Determination of consistency – Spreading on the vibrating table’ (1982).

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

[9] Reinhardt H.-W., Grosse C., Herb A., Weiler B., Schmidt G., Method for examining a solidifying and/or hardening material using ultrasound, receptacle and ultrasound sensor for carrying out the method. Patented under No. 09/857,536 at US Patent and Trademark Office, 2001.

[10] C. Grosse: Quality management of fresh concrete - fresh concrete analysis with ultrasound. Concrete Plant +Precast Technology/Betonwerk+Fertigteil-Technik, BFT vol. 6 (2005), pp 26-32.

[11] Ye, G., ‘Experimental study and numerical simulation of the development of the microstructure and permeability of cementitious materials’, PhD thesis, T.U. Delft, The Netherlands (2003).

[12] Van Breugel, K., ‘Simulation of Hydration and Formation of structure in Hardening Cement Based Materials’, PhD thesis, Delft University of Technology, The Netherlands (1991).

[13] prEN 934-5, Admixtures for concrete, mortar and grout – Part 5: Admixtures for sprayed concrete – Definitions, requirements and conformity, 2003.

[14] Garshol K.F., New admixtures for high performance shotcrete, In: Austin S.A. (ed.), Proc. ACI/SCA International Conference on Sprayed Concrete/Shotcrete, Chapman & Hall, London, 1996, pp. 26-38.
[15] Jolin M., Lacombe P., Le béton projeté: nouveaux développements et applications. Canedian Journal of Civil Engineering, 27 (2000) 383-388.
[16] Prudêncio L.R.Jr., Armelin H.S., Helene P., Interaction between accelerating admixtures and Portland cement for shotcrete: the influence of the admixture's chemical base and the correlation between paste tests and shotcrete performance. ACI materials journal, 93 (6) (1996) 619-628.
[17] Gebler S.H., Litvin A., McLean W.J., Schutz R., Durability of dry-mix shotcrete containing rapid-set accelerators, ACI Materials Journal, 89 (3) (1992), 259-262.
[18] De Belie N., Grosse C.U., Kurz J., Reinhardt H.-W. Ultrasound monitoring of the influence of different accelerating admixtures and cement types for shotcrete on binding and hardening behaviour. Cement and Concrete Research, 35 (2005), 2087-2094.