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Green and early age compressive strength of extruded cement mortar monitored with compression tests and ultrasonic techniques

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Abstract

Knowledge about the early age compressive strength development of cementitious materials is an important factor for the progress and safety of many construction projects. This paper uses cylindrical mortar specimens produced with a ram extruder to investigate the transition of the mortar from plastic and deformable to hardened state. In addition, wave transmission and reflection measurements with P- and S-waves were conducted to obtain further information about the microstructural changes during the setting and hardening process. The experiments have shown that uniaxial compression tests conducted on extruded mortar cylinders are a useful tool to evaluate the green strength as well as the initiation and further development of the compressive strength of the tested material. The propagation of P-waves was found to be indicative of the internal structure of the tested mortars as influenced, for example, by the addition of fine clay particles. S-waves used in transmission and reflection mode proved to be sensitive to the inter-particle bonding caused by the cement hydration and expressed by an increase in compressive strength.

Keywords: Hydration; Compressive strength; Mortar; Extrusion; Ultrasonic testing

1. Introduction

The parameter compressive strength is very often used to quantify the quality of cementitious materials. In this context, the compressive strength measured at 28 days after casting usually provides the basis for the design and the acceptance of concrete structures. However, many concrete structures are and must be subjected to severe loading during construction, when the concrete is still of early age. If the special properties of early age concrete are not fully understood and correctly addressed, this scenario can lead to the failure of concrete elements, such as excessive floor sagging, spalling or increased long term deflections.

These circumstances emphasize the importance of the detailed knowledge of early age concrete and its performance in structures under construction. This necessity goes along with the requirement to incorporate the specifics of early age concrete into design codes, which in turn calls for research on this subject. The objective of the research dealing with early age concrete should be to understand the microstructural changes occurring during the early hydration period. Only this allows developing relationships between the early-age microstructure and later age properties of the concrete (Carino [1]).

This paper has the objective to investigate the development of the compressive strength of cementitious materials on the example of two specifically designed mortar mixtures. In the presented investigations, emphasis is placed on the evolution of the compressive strength as freshly mixed mortar gradually changes from plastic and deformable to hardened state. The compressive strength was measured with uniaxial compression tests.

In general, compression tests on regular cast specimens cannot be performed until the initial set has occurred. Therefore, the compressive strength of regular cast cementitious materials at any time before initial setting is practically a not defined parameter. Even after initial setting it is very

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difficult to remove specimens from their mold without damage until the final set has occurred. To overcome this problem mortar specimens were produced with an extrusion technique. By means of this technique cylindrical specimens were produced that have excellent shape retention, even if the mortar is in fresh state.

The compressive strength obtained from such mortar cylinders in fresh state is commonly called green strength of the tested material. The possibility to measure the compressive (or green) strength of the extruded cylinders at early ages allows monitoring the initiation of the compressive strength from the very beginning.

In addition, the mortars were subjected to wave transmission and reflection measurements to obtain further information about the early age development of the mortars. The wave transmission tests were conducted with both, longitudinal (P-) and shear (S-) waves of a frequency of 150 and 180 kHz, respectively and used to determine the velocity and attenuation of the waves in the hydrating mortars. The wave reflection method was used to determine the reflection loss of S-waves at an interface between a steel plate and the investigated mortars. Based on the results of the ultrasonic measurements it was possible to investigate the relationships between changes in the cementitious microstructure and the observed increase of the green or compressive strength. Another important outcome of the conducted experiments is information about the sensitivity of the applied ultrasonic methods to the early age compressive strength development of cementitious materials.

Investigations on mortars with low water contents as presented in this paper are also especially useful for understanding the development of early age properties of materials used for slip-form casting of concrete pavements. The concrete mixtures used for this technology have a dry consistency similar to that of the used mortars to ensure a good shape retention immediately after the casting process.

2. Experimental program

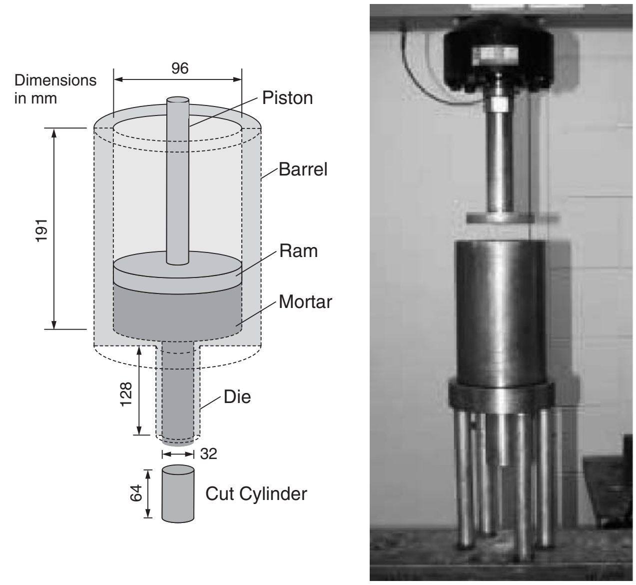
2.1. Materials

Two different mortar mixtures were used for the investigations presented in this paper. To make the mortars suitable for the extrusion technique special binders had to be added to

Table 1
Mix proportions of the investigated mortar mixtures (by weight)

	Mortar A	Mortar B
Cement[a]	1	1
Water	0.35	0.39
Fine aggregates[b]	3.46	3.41
Clay[c]	–	0.034[d]
HPMC	0.016[e]	0.014[e]
Superplasticizer	0.010	0.010

[a] Type III.
[b] River sand.
[c] Rurified magnesium aluminosilicate.
[d] 1% of fine aggregates.
[e] 0.4% of fine aggregates.



(a) Schematic (b) Photograph

Fig. 1. Ram extruder used to produce extruded mortar cylinders.

provide good material cohesion, low adhesion to die surfaces, and low elasticity of the mortars (Srinivasan [2]). The use of such binders is also very common for the extrusion of ceramics (Sarkar and Greminger [3]; Schuetz [4]). The mix proportions of the two different mortar mixtures are given in Table 1. For both mortars hydroxypropyl methylcellulose (HPMC) was used as a water soluble, organic binder.

In addition, clay on the basis of a purified magnesium aluminosilicate was added as an inorganic binder to Mortar B. First, cement, sand and HPMC were premixed in a planetary mixer for 2 min. Then water and superplasticizer were added and mixed for another 3 min. For the mixing of Mortar B, the clay was added to the HPMC, sand and cement and then premixed for 2 min before the water was added. Additional adsorption water was added for sand (2.3% of mass) and clay (200% of mass).

2.2. Extrusion technique

The extrusion technique was used to produce mortar cylinders that allowed testing the green or compressive strength of these specimens at very early age. A ram extruder as shown in Fig. 1 was used for the extrusion process. The piston of the extruder was connected to a compression test machine with a maximum load capacity of 100 kN. The mortar was extruded at a velocity of 1.0 mm/s. After being extruded, the specimens were cut into samples with a diameter of 32 mm and a length of 64 mm (see Fig. 1a). To prevent the surface of the freshly extruded cylinders from drying, the specimens were wrapped in plastic foil. During the time period between extrusion and testing, the sealed cylinder specimens were stored in horizontal position at a room temperature of ca. 22 °C.

It has to be stated that the investigated mortars could only be extruded because of the addition of HPMC. It was reported by Schuetz [4] that, when used in ceramics, this polymer very effectively reduces the surface tension of water and thereby causes an improved wetting of the particles present in the mixture. It also significantly increases the ability of mixtures to retain water and reduces the interparticle friction. It could be observed that HPMC has similar effects when used in cementitious materials. In fact, the use of methylcellulose or HPMC for the extrusion of cement-based is reported by many researchers (e.g., [2,5]).

2.3. Compressive strength tests

The green and later the compressive strength of the extruded cement mortar cylinders were tested with the uniaxial compression tests. Because of the excellent shape retention of the extruded specimens the green strength of the mortar could be determined immediately after the extrusion process. The compression tests at this stage were performed with a load cell of a capacity of 444 N and two stiff plates. The tests were performed in displacement control at a rate of 0.1 mm/s. At later ages, when the mortar was hardened, the capacity of the load cell and the loading rate was increased and a movable and a stiff plate were used for the tests. An extruded cylinder during a compression test to determine the green strength is shown in Fig. 2.

2.4. Wave reflection measurements

The wave reflection method as used for the experiments described in this paper was introduced by Öztürk et al. [6] and Rapoport et al. [7]. The technique monitors the reflection loss of S-waves at an interface between a steel plate and a cementitious material over time. The amount of the lost wave amplitude depends on the reflection coefficient, which in turn is a function of the acoustical properties of the materials that form the interface.

A schematic of this experimental technique is shown in Fig. 3. A steel plate is brought in contact with fresh mortar and a transducer with a frequency of 2.25 MHz transmits an S-wave pulse into the steel plate. When the mortar is in liquid state the pulse is entirely reflected at the steel–mortar interface, since S-waves do not propagate in liquid materials (Fig. 3a). Thus,

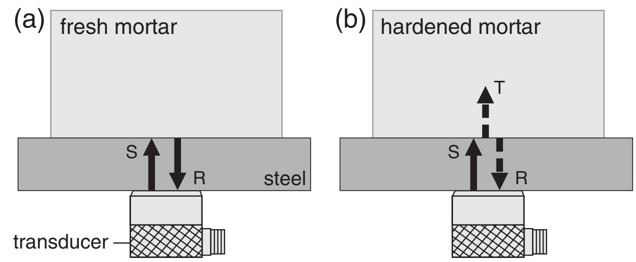


Fig. 3. Principle of wave reflection method: (a) complete wave reflection for fresh mortar; (b) partial wave reflection and transmission for hardened mortar.

the reflection coefficient is unity. With proceeding hydration the cement grains percolate and build up a skeleton allowing the shear waves to propagate. This allows the shear waves to pass the interface resulting in reflection losses during the reflection process (Fig. 3b). Consequently, the reflection coefficient starts to decrease. With proceeding hydration the ability of the cement mortar to transmit shear waves gains higher levels. More and more wave energy is transmitted into the mortar and the reflection coefficient decreases further. After a certain time this process decelerates and the reflection coefficient approaches a final value. At this time changes in the microstructure of the cement mortar due to hydration are too small to alter the shear wave propagation properties significantly.

The investigations conducted with this method so far have shown that the wave reflection method can reliably detect the differences in the hydration rate caused by factors such as retarding or accelerating admixtures [6,7]. The experiments have also proven that the WR-method is able to follow the development of parameters such as compressive strength, elastic moduli and degree of hydration [8–10].

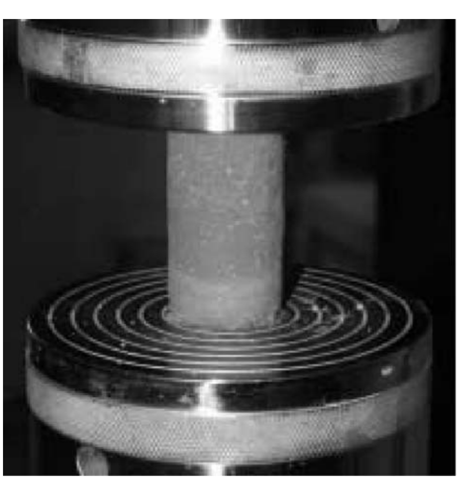


Fig. 2. Extruded cylinder during compression test.

2.5. Wave transmission tests

Wave transmission tests according to ASTM C597 were conducted to determine the P- and S-wave velocity in the tested mortar mixtures. To conduct the wave transmission tests the extruded cylinders were placed between two transducers and slight pressure was applied to establish contact between transducers and specimens. To improve the acoustic coupling viscous coupling agent was applied to the face of the transducers. When the measurements were conducted with P-waves a frequency of 150 kHz was used. The experiments with S-waves were performed with a frequency of 180 kHz.

During the hydration of the tested mortars the transition time of the ultrasonic waves was measured by a pulse velocity meter in regular time intervals. By knowing the length of the wave path l and the transition time t the wave velocity v of the test material can be calculated with $v = l/t$. The measured velocities itself can be used as an indicator of the progress of the setting and hardening process of the test materials.

The wave transmission tests were also used to measure the attenuation of the ultrasonic waves when propagating through the hydrating mortar. To determine the attenuation at a given time the amplitude of the wave transmitted through the test material is compared to the wave amplitude measured when the transmitting and receiving transducers are brought in direct contact. The amplitudes were measured in mV using an oscilloscope. Based on these parameters the attenuation A can be calculated with Eq. (1), where V_t is the voltage of the wave transmitted through the test material and V_c the voltage of the wave signal when the transducer contact each other.

$$A = 20\log\frac{V_t}{V_c} \quad (1)$$

3. Results and discussion

3.1. General procedure

The setting and hardening of cementitious materials can be measured by means of many different parameters. Within the scope of this paper, the green and compressive strength are the main parameters that will be used to describe this process. In this context, special emphasis will be placed on the transition of the mortars from plastic material behavior (green strength) to hardened state (compressive strength). In addition, the results of the ultrasonic measurements will be analyzed to determine how the different types of ultrasonic waves can be used to indicate the development of the green and compressive strength of the mortars during the various stages.

The investigations were conducted on materials that differ from cement-based materials for common applications by the use of the organic binder HPMC. This binder needs to be added to make the mortar extrudable. If the binder is not added to the mixture, the material lacks the plasticity and water retention that is necessary for the extrusion process. Investigations conducted to evaluate the general influence of methylcellulose on the physical properties of cementitious materials have found that its addition increases the tensile strength of cement paste and slightly decreases the compressive strength (Fu and Chung [11]). No significant effects on the setting time or hydration kinetics have been reported. This is supported by the measured compressive strength development of the mortars that follows the trends that are generally observed for cement-based materials.

3.2. Green and compressive strength development

The development of the compressive strength of the two mortar mixtures in the first 36 h is shown in Fig. 4. For both mixtures, the same general trends can be observed. Initially, the green strength of both mixtures develops on the same, very low level. After a certain time, the strength values gradually start to increase and begin to develop according to a power law. The period between the first significant increase and the beginning of the power law trend can be interpreted as the transition from the green strength to an actual compressive strength. At a later age the compressive strength follows a hyperbolic trend function as given in Eq. (2).

The evolution of the green and compressive strength of the mortar containing HPMC (Mortar A) in the first 20 h is given in Fig. 5. The inset in the figure shows that the green strength develops at ages below 6 h at a very low level with only minimal increase. This can also be seen on the crack or failure patterns of the cylinders tested at 2.9 and 6.5 h. The patterns are identical and typical for the plastic and deformable behavior of the extruded mortar during this stage.

Between approximately 6 and 8 h the measured strength values increase by about 100%. At an age of 7.6 h the cylinder shows a shear crack pattern. This pattern indicates that the cement hydration has already created a rigid structure enabling the cylinder to sustain a significant compression force. However, the rigidity of the specimen does not allow the

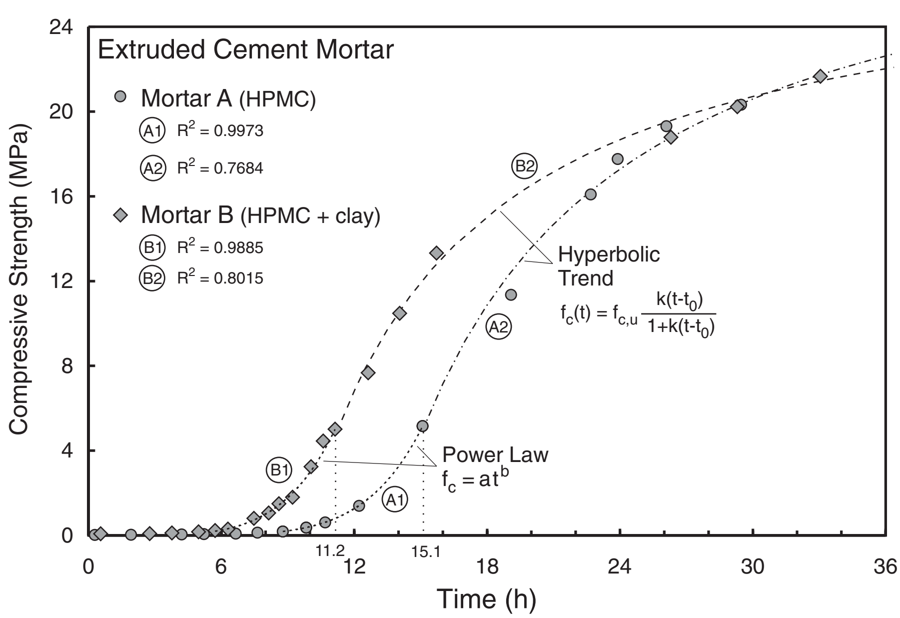


Fig. 4. Development of compressive strength of extruded cement mortars.

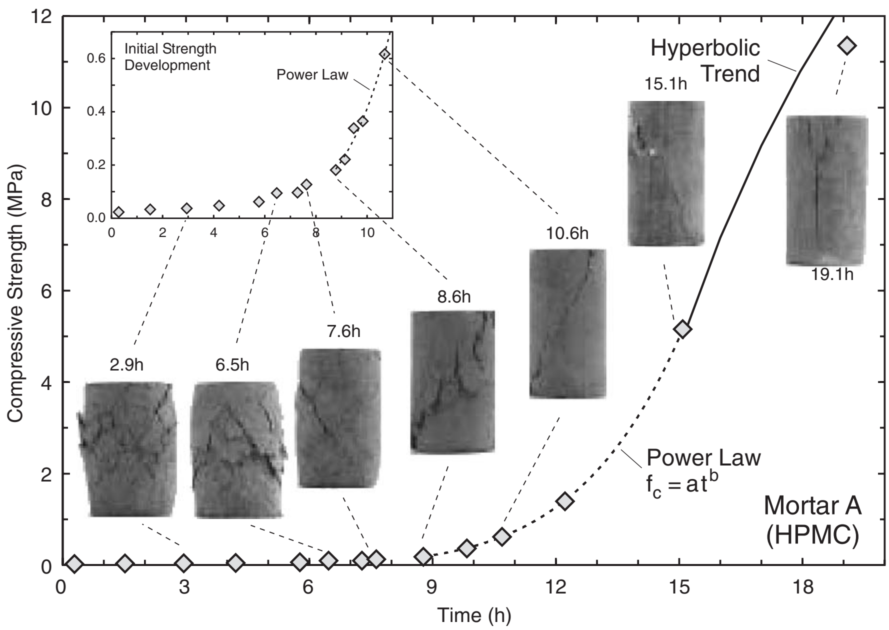


Fig. 5. Development of compressive strength of extruded cement mortar (Mix A) and appropriate cracking patterns of tested cylinders.

development of tensile stresses, acting lateral to the compression force, which are high enough to cause the cylinder to fail. Instead, the inter-particle cohesion forces are exceeded, which leads to the development of a shear plane as it can be observed in the testing of cohesive soils (Delenne et al. [12]; Ang et al. [13]). The cylinder tested at 8.6 h also fails by showing a shear crack with very undefined edges. The inset in Fig. 5 shows that the two cylinders that first exhibit the shear crack pattern (7.6 and 8.6 h) also mark the time of the first significant increase in strength. It can be assumed that the occurrence of the shear crack pattern indicates the beginning of the setting process, that is the transition from green strength to compressive strength. This assumption is validated by the fact that the strength data measured after 8.6 h follow a power law-type of trend, which after Popovics [14] is characteristic for the setting process of cement-based materials.

The strength measured at 10.6 h and thereafter can safely be considered as an actual compressive strength. The shear crack pattern can still be observed between 10.6 and 15.1 h, but the crack edges are very well defined and no signs of plasticity in the material behavior could be observed. After 15.1 h, the trend of the compressive strength development changes to a hyperbolic trend (see Eq. (2)). The cylinder tested at 19.1 h exhibits a clearly vertical splitting crack pattern, which is one of the typical crack patterns for hardened mortar or concrete cylinders (ASTM C39 [15]). The general form of the hyperbolic trend that is followed by the compressive strength data is given by

$$f_c(t) = f_{c,u}\frac{k(t-t_0)}{1+k(t-t_0)} \quad (2)$$

where f_c is the compressive strength, $f_{c,u}$ the ultimate compressive strength at infinite time, k the rate at that f_c increases, and t_0 the fictitious time at that f_c starts to increase.

The strength development process of the extruded mortar can also be monitored by observing the stress-displacement (σ-d) curves of the tested material as the immediate result of the conducted uniaxial compression tests. This curve provides information about the material behavior under compression forces. The σ-d curves measured on the cylinders of Mortar A at very early ages are given in Fig. 6. The different curves represent the behavior of specimens tested between 2.9 and 10.6 h. The crack patterns of the same specimens are shown in Fig. 5.

The presented curves illustrate the transition of the material behavior from deformable and plastic state to that of a stiff material. The σ-d curve measured at 2.6 h shows a small stress increase followed by a long flow plateau, where the stress remains on a constant level while the displacement increases. This behavior is typical for a plastic material, such as freshly extruded cement mortar. At an age of 6.5 h essentially the same behavior can be observed. The only difference is the higher stress level of the flow plateau. This similarity in the material behavior is also expressed by the failure patterns of the cylinders tested at these times (see Fig. 5).

The σ-d curve measured at 7.6 h exhibits a different characteristic. The curve now increases until a distinctive maximum value is reached. Although the stiffness of the mortar is still very low, the cylinder fails at a certain stress level with a subsequent decrease in the measured stress. This change in the failure mechanism is also reflected by the shear crack pattern of the cylinder tested at this time. The cylinder tested at 8.6 h shows an even more distinct maximum stress value accompanied with a further increase of the stiffness. The mortar tested at this time clearly starts to develop properties of a solid material. The shear crack pattern of the failed cylinder is more pronounced and the appropriate strength value marks the beginning of the power law trend of the compressive strength values (see Fig. 5).

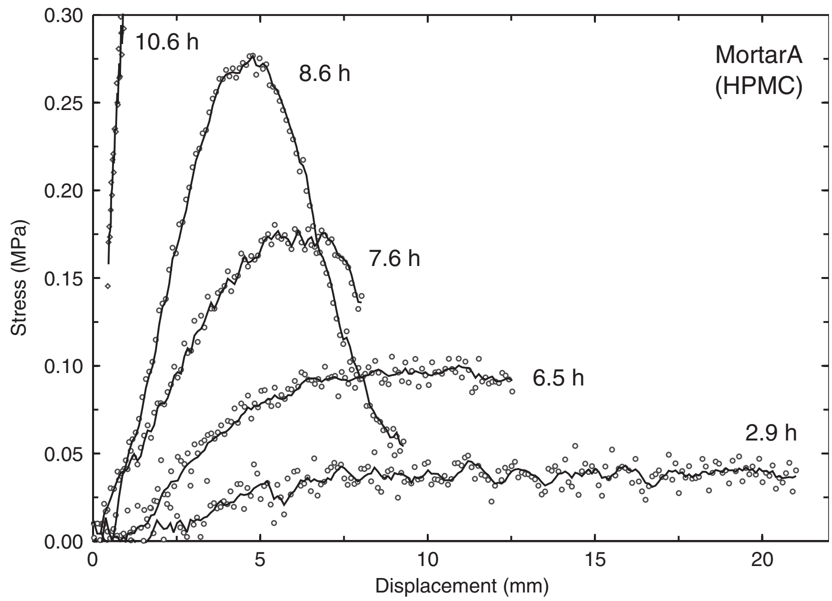


Fig. 6. Stress-displacement curve measured on extruded mortar cylinders at early ages (Mortar A).

3.3. Ultrasonic measurements

The results of the ultrasonic wave transmission and reflection measurements are presented in Fig. 7. The comparison of the P- and S-wave velocity especially at early ages allows important conclusions about the sensitivity of these two wave types. In Fig. 7a it can be observed that at the very beginning, the P-wave velocity measured on Mortar B, which contains clay, is significantly higher than that of Mortar A at the same age. This indicates that the P-wave velocity is very sensitive to the internal structure of the tested materials even before the so-called initial set. The fine clay particles added to Mortar B cause the already very dense microstructure of the extruded material to become even more dense and compact. This improves the mechanical coupling between the constituents of the mortar (cement, clay, and sand particles) and thereby improves the P-wave propagation properties of the fresh mortar material.

However, the differences in the internal structure of the mortars do not influence the initial values of the S-wave velocity. In Fig. 7b can be seen that both mortar mixtures show similar velocity values at the time of onset of the S-wave propagation. This originates from the fact that S-wave propagation is not influenced by the inter-particle distance inside a material. S-waves can only propagate if the individual particles are part of a shear-rigid microstructure and this shear rigidity can only be created when the particles become connected due to the cement hydration process. For this reason, the S-wave velocity measured on Mortars A and B starts to increase at different times since both mortars exhibit different hydration dynamics.

The observed differences in the sensitivity of P- and S-waves are in good agreements with the results obtained by other researchers. Morin et al. [16] found that the initial value of the reflection coefficient measured with P-waves on different cement mortars is influenced by the density of the tested

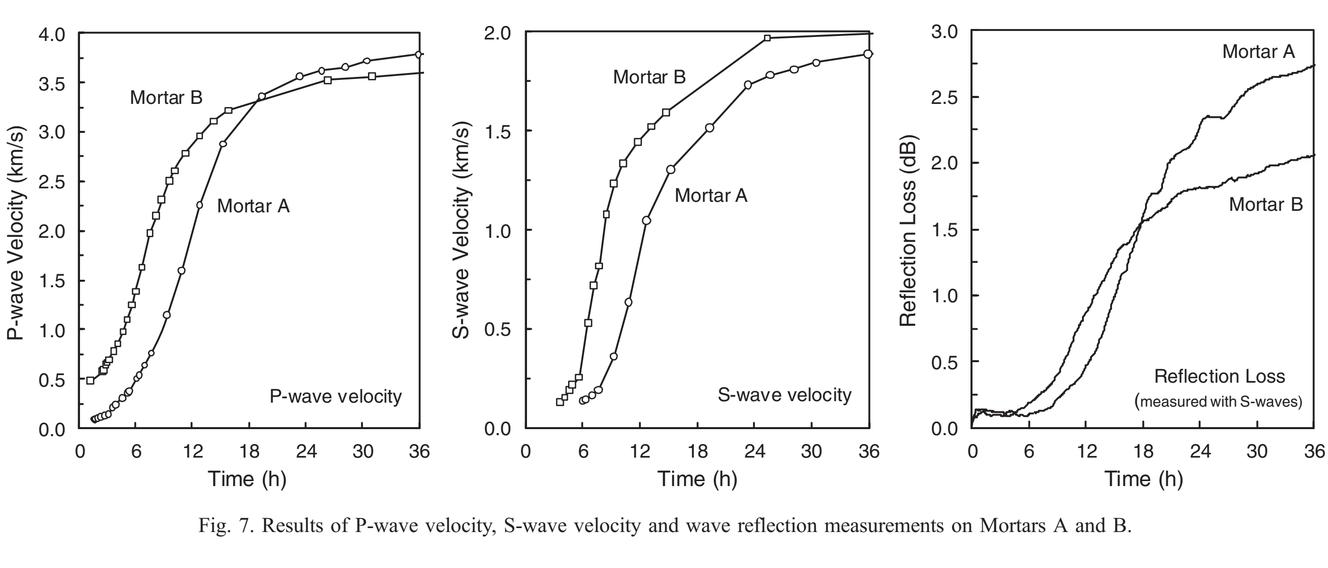


Fig. 7. Results of P-wave velocity, S-wave velocity and wave reflection measurements on Mortars A and B.

material. Measurements using S-waves did not show this kind of sensitivity. The density differences of the tested mortars was caused by different amounts of entrapped air. The sensitivity of P-waves to entrapped air in cement paste in form of microbubbles was also found by Keating et al. [17] and Sayers and Dahlin [18].

As indicated by the compressive strength development given in Fig. 4, Mortar B starts to gain strength faster than Mortar A. The same effect can be observed for the S-wave velocity in Fig. 7b. This can be taken as an indication for the fact that the S-wave velocity is governed by the same material property as the compressive strength, that is how well the individual particles of the cementitious microstructure are bonded together as a result of cement hydration.

The development of the reflection loss measured with S-waves is given in Fig. 7c. With respect to the early age the reflection loss resembles the development of the S-wave velocity. The curve for Mortar B starts to increase first followed by that of Mortar A. The time of increase of reflection loss and S-wave velocity for the two mortar mixtures is very similar.

The sensitivity of S-waves to the connectivity of hydrating cement particles, which is proposed earlier in this section, was investigated and confirmed in a study conducted by Voigt et al. [19]. In this investigation the specific contact area between hydrating cement particles was determined with the numerical model HYMOSTRUC and compared to the reflection loss measured with S-waves on mortars with different w/c-ratios. It was found that reflection loss and specific contact area have a unique relationship, independent of the w/c-ratio of the tested mortars.

A very close relationship between S-wave propagation and connectivity of the particles within the cementitious microstructure was also found by Boumiz et al. [20]. In this study, the onset of S-wave propagation in hydrating cement pastes could only be detected shortly after the percolation threshold. Similarly to the present study, propagation of P-waves was detected well before the occurrence of the percolation threshold. Along this line, examinations of ultrasonic wave propagation in early age cement pastes conducted by D'Angelo et al. [21] found that S-waves are more sensitive to the connectivity of solid particles than P-waves. These experiments also showed a correlation between the initial setting time and the onset of S-wave propagation.

It should be noted, that the reflection loss for both mortars increases immediately to a relatively high value (ca. 0.1 dB) and remains on this level for a longer period of time. This is in contrast to earlier studies, where the reflection loss was found to develop at values close to zero during the initial phase (Voigt et al. [9]; Voigt and Shah [10]). It is believed that the early increase observed here is attributed to the nature of the extruded cement mortar. Cement paste and sand are exposed to a high triaxial pressure condition during the extrusion process, which creates a material with a very dense internal structure. Due to this pressure and the low w/c-ratio, the cement and sand particles are in much closer contact as they are in a regular cast mortar mixture. This obviously allows the shear waves to propagate across the steel–mortar interface for a small extend and thereby causes the reflection loss to increase.

3.4. Comparison of compressive strength with results of ultrasonic measurements

The objective of this section is to further investigate the relationships between green or compressive strength of the tested mortars and the parameters obtained from the ultrasonic measurements. Special emphasis will be placed on the period when the green strength of the mortar transforms to an actual compressive strength. In Fig. 8a the compressive strength development of Mortar A within the first 15 h after mixing is given. The reflection loss, velocity and attenuation measured on the same mortar are given in Fig. 8b–d, respectively. The initiation of the compressive strength shown in Fig. 8a has already been discussed in the previous section (see Fig. 5).

After approximately six hours the reflection loss, given in Fig. 8b, starts slightly to increase from its initial constant value. The time of this increase matches the time when also the first significant increase of the compressive strength can be observed (Point ①). This suggests that the structural changes caused by the hydration in this time step affects both quantities in a similar way. The percolating cement particles start to develop a solid microstructure that lends compressive strength to the cylinder and allows the shear waves to propagate in the mortar, which causes the reflection loss to increase. The initiation of the hydration process also results in the onset of the shear wave propagation in through transmission mode (Fig. 8c).

Since transmitted shear wave signals could not be detected before this time, it can be concluded that the hydration products now provide a continuous path allowing the shear waves to pass the test specimen from one side to the other. Thus, the time marked by Point ①, at which the reflection loss starts to increase, can be considered as the time when the cement particles are percolated (percolation threshold). Investigations that support this theory were presented by Voigt et al. [19], where the time of increase of the reflection loss was found to be in close correlation with the percolation threshold of the cement paste determined with numerical simulation.

It should be noted that the attenuation of the transmitted shear wave signals shown in Fig. 8d remains on a constant level although the shear wave velocity is increasing. This indicates that the microstructural changes in the mortar are still rather small, which is also reflected in the small rates of increase of compressive strength, reflection loss and shear wave velocity between Point ① and Point ② (at ca. eight hours). At this time, all three of the latter parameters start to develop after a power law and the attenuation is decreasing. This observation indicates that Point 2 marks the time of initiation of the actual compressive strength. From the observations described above can be concluded that the measurements conducted with S-waves in reflection and transmission mode are in very close

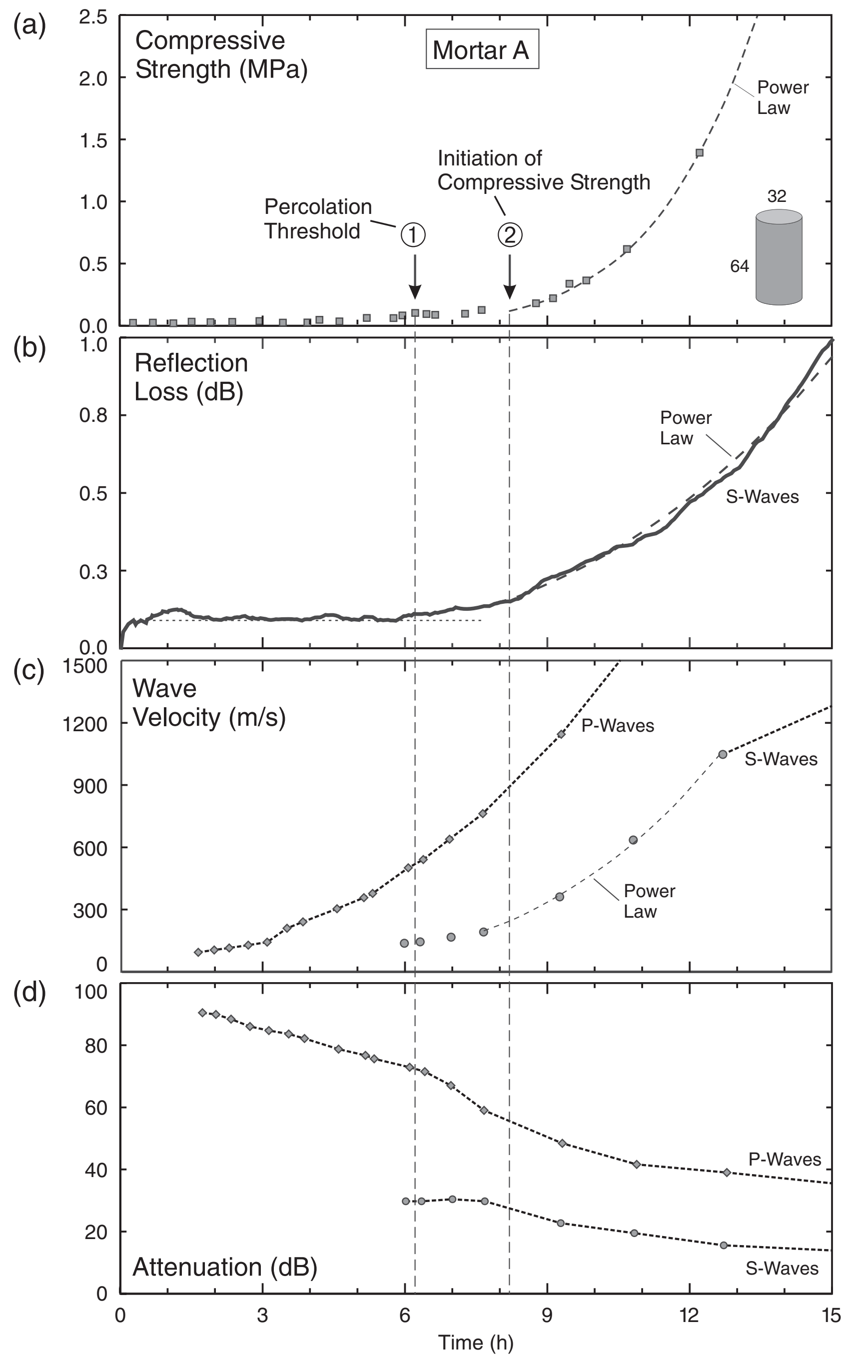


Fig. 8. Comparison of compressive strength with results of ultrasonic measurements for Mortar A.

correlation to the evolution of the compressive strength of the tested mortar at very early ages.

Fig. 8c and d also show results for wave velocity and attenuation measured with P-waves. It can be seen that the transmission of P-waves can be detected as early as 1.5 h after casting. After approximately three hours the P-wave velocity in Fig. 8c increases with a significant rate, although no increase in compressive strength can be observed. The same statement (on a qualitative basis) can be made for the attenuation of the P-waves shown in Fig. 8d.

This can be explained based on the development of the cementitious microstructure at very early ages. Scrivener [22] found that during the first three hours of cement hydration mainly ettringite is formed outside of the primarily still unhydrated cement grains in the shape of small rods. Depending on the w/c-ratio, which determines the inter-particle distance of the cement grains, and the chemical composition of the used cement, these ettringite needles have no or only a small influence on the stiffening or compressive strength of the mortar (Aïtcin and Neville [23]). In contrast to the stiffening behavior, the P-wave velocity is strongly affected by the formation of the ettringite. Although these needles do not create bonds between the cement particles they do fill pore space that was previously occupied by water with solid products, which is very beneficial for the propagation of P-waves. Since, as mentioned, no bonds are created the S-wave propagation properties remain unaffected.

3.5. Detailing the relationship between compressive strength and reflection loss

The relationship between reflection loss measured with S-waves and the compressive strength of the tested material was investigated in several previous publications [8–10,24]. It was found that both quantities are related by a bilinear function, where the first part of the bilinear function covers the early age up to approximately 15 h after casting. The second part describes the relationship between the parameters up to a range between three to five days depending on the curing conditions of the considered material and has a higher slope than the first part. The experiments reported in [8–10,24] were conducted with Portland cement type I.

The change of the slope in the compressive strength – reflection loss (f_c-R_L) relationship indicates differences in the kinetics of both parameters at early ages. Similar observations of a rapid increase of P-wave velocity accompanied with no or only small changes in compressive strength of concrete were made by Elvery and Ibrahim [25] and Byfors [26]. In these studies it was concluded that this phenomenon could be attributed to the early formation of C–S–H phases that connect cement particles and aggregates and thereby improve the P-wave propagation properties of the concrete. The compressive strength does not show significant changes until the cement hydration has progressed to a higher extent.

For the case considered here, the change in slope can be explained by a theory put forward by Boumiz et al. [20]. According to their findings the microstructural development at very early ages is dominated by the formation of hydration products (calcium hydroxide and calcium silicate hydrate) on the surface of the cement clinker grains leading to progressive connection among the particles. This increase of connectivity causes a higher change of the S-wave reflection loss (improved S-wave propagation) than of compressive strength and therefore results in a flatter slope of the f_c-R_L relationship. After a certain time (ca. 15 h) the grain connection process is accompanied by filling of capillary pore space with hydration products. During this time the compressive strength development accelerates and the slope of the f_c-R_L relationship changes.

Based on the availability of accurate early age compressive strength data of the investigated mortars the f_c-R_L relationship during the time immediately after the initiation of the strength development will be analyzed in more detail. This will help to evaluate the potential of the wave reflection method to be used as a tool to monitor the early age compressive strength development of cementitious materials.

The f_c-R_L relationships for Mortar A and B for the first 10 to 15 h are given in Fig. 9a and b. It can be seen that for both mortars the f_c-R_L relationship can be subdivided into three parts. The first part covers the time when the setting process of the mortar has not yet started. The strength of the mortar measured during this time can be considered as a green strength and no consistent trend in the data points can be identified.

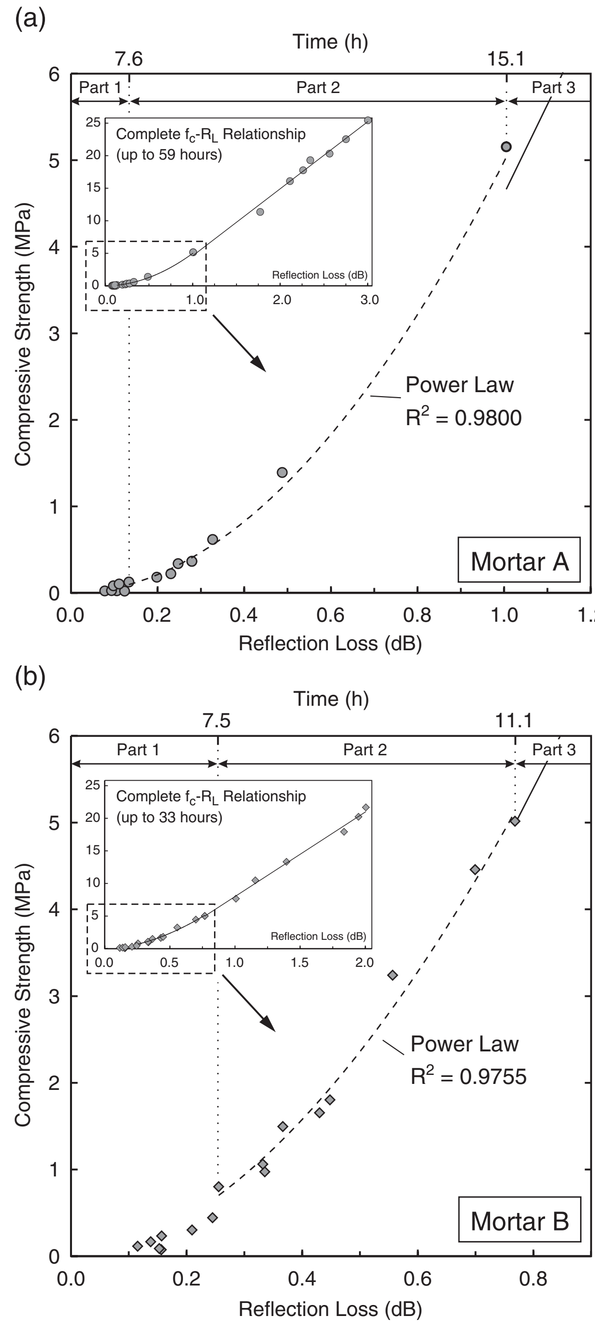


Fig. 9. Early age relationship between compressive strength and reflection loss for Mortars A and B.

In the second part of the f_c-R_L relationship, compressive strength and reflection loss are related by a consistent function, which is a power law for the cement mortars investigated here. The R^2-values of the fitted power law functions are given in the figure. For both tested mortars the time period in which the f_c-R_L relationship can be described by the power function matches that when also the compressive strength (see Fig. 4) follows a power function.

From the results presented in the previous section it can be concluded that this second part covers the setting process of the mortars. Investigations conducted on regular cast mortars containing Portland cement type I have shown that the $f_c - R_L$ relationship of these mortars follows a linear trend during setting [10]. The influence of the cement type (cement type III was used for the extruded mortars) on the second part of the $f_c - R_L$ relationship shows that the reflection loss is very sensitive to microstructural properties of the tested cementitious materials and their development.

The third part of the $f_c - R_L$ relationship, which is given in the insets of Fig. 9, follows a strong linear trend and has a steeper slope compared to part two. This third part represents the main portion of the $f_c - R_L$ relationship and exhibits the same behavior as the $f_c - R_L$ relationships of normally cast cement type I mortars and concretes.

4. Conclusions

The following conclusions can be drawn from the presented investigations:

1. The stiffening of extruded cement mortar, that is the gradual transition from a plastic, deformable to hardened state, can easily be observed based on changes in the shape of the stress-displacement curves measured in compression and the resulting failure patterns of cylindrical specimens.
2. Wave transmission experiments conducted with P-waves during the setting and hardening process show that velocity and attenuation of P-waves exhibit significant changes long before an increase of compressive strength can be detected. This confirms findings of other researchers showing that the propagation of P-waves at early ages is affected by changes in the cementitious microstructure that do not immediately contribute to the compressive strength development.
3. Measurements obtained with S-waves in transmission and reflection mode are in much better agreement with the beginning of the setting process of the mortars, indicated by the transition from green strength to compressive strength. Reflection loss and wave velocity exhibit significant changes at the time when the compressive strength starts to develop. This confirms results of other studies showing a close relationship between S-wave propagation and the percolation process of cement particles that creates a shear rigid microstructure and later results in the initiation and further increase of compressive strength.
4. The relationship between reflection loss and compressive strength for the extruded mortars at very early ages follows a power law. At later ages this relationship follows a linear trend.

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