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Comparison between two ultrasonic methods in their ability to monitor the setting process of cement pastes

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A B S T R A C T

This paper presents the comparison between ultrasonic wave transmission (USWT) method and ultrasonic wave reflection (USWR) method in their ability to monitor the setting process of cement pastes. The velocity of ultrasonic longitudinal waves and shear wave reflection coefficient were measured simultaneously on cement pastes with different hydration kinetics. Even though both methods are able to reliably monitor the hydration process and formation of structure of an arbitrary cement paste, they monitor the setting process in different ways. The relationship between the velocity of longitudinal waves and shear wave reflection coefficient can be simplified into three characteristic phases and the end of the first phase can be used to define the beginning of the setting process of cement paste.

1. Introduction

Acoustic waves are being used successfully to monitor the setting and hardening process of different cement-based materials. Two types of waves are generally used, namely shear waves (s-waves) and longitudinal waves (p-waves). Numerous attempts have been done in order to investigate the relationship between the ultrasonic measurements and some properties of different cement-based materials.

The ultrasonic shear wave reflection method was first applied to the area of cementitious materials by Stepišnik et al. [1] and has been advanced further by Valič [2]. This method can be used to monitor the strength development of cementitious materials at early age [3–5]. By using shear waves, the measurements can be correlated to the shear modulus and viscosity, which are parameters of interest for most engineers and researchers [6]. A linear relationship was found between degree of hydration and reflection loss [7].

Reinhardt and Grosse [8,9] evaluated changes in the material properties associated with concrete aging by measuring the velocity of longitudinal waves, signal shapes of transmitted waves, and their frequency spectra and very recently, some studies have been done in order to correlate the initial setting time of different cement-based materials and the velocity of ultrasonic longitudinal waves [10–12]. Krauss and Hariri [13] presented the procedure to determine the end of the dormant phase and initial degree of hydration by the ultrasonic wave transmission method.

However, little research has been done in the analysis of the correlation between the velocity of ultrasonic longitudinal and shear waves and the comparison between ultrasonic wave transmission and ultrasonic wave reflection method to monitor the hydration process and formation of structure of cement-based materials. Voigt et al. [14] presented a comparison of ultrasonic wave transmission and reflection measurements with longitudinal and shear waves on early age mortar and concrete. They indicated that these two ultrasonic methods monitor the setting process of mortar and concrete in significantly different ways.

The objective of this paper was to study the correlation between the ultrasonic wave transmission method (USWT) and ultrasonic wave reflection method (USWR) in their ability to monitor the setting process of cement pastes. Therefore, a comprehensive experimental work has been performed. The evolution of the velocity of ultrasonic longitudinal waves, v_p, and shear wave reflection coefficient, r, with time, t, were measured simultaneously on different cement pastes in order to get the most appropriate correlation between v_p and r. The influence of water/cement ratio (w/c), environmental temperature (T), cement type (CT), cement fineness (BS), and amount of C_3A (C3A) on the $v_p - r$ relationship was studied. In addition, a possibility of using this combined ultrasonic method to determine the beginning of the setting process of cement pastes is also proposed.

2. Experimental methods

2.1. Wave transmission method

Within this study, the wave transmission method was used, which has been already described in Ref. [11]. The v_p measurements started

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Table 1
Characteristics of cement types used in the study.

Cement type	Label	AC [%]	BS [cm^2/g]	C_3S [%]	C_2S [%]	C_3A [%]	C_4AF [%]
CEM II/A-S, 42.5R	C1	>80	4260	32.8	46.3	10.4	10.5
CEM I, 42.5 N	C2	>95	2640	60.2	13.6	7.2	9.3
CEM I, 42.5 N SR	C3	>95	3130	55.9	21.9	2.3	15.0
CEM I, 52.5 R	C4	>95	4310	57.7	13.0	6.9	8.9

Table 2
Influential parameters of cement paste mixtures used in the study.

No.	Mixture label		CT	w/c	BS [cm^2/g]	C_3A [%]	T [°C]
1	MC1035		C1	0.35	4260	10.4	21
2	MC1040		C1	0.4	4260	10.4	21
	MC1050	MT1	C1	0.5	4260	10.4	21
3		MT2	C1	0.5	4260	10.4	26
		MT3	C1	0.5	4260	10.4	32
4	MC2, MF1, MC3A1		C2	0.5	2640	7.2	21
5	MC3, MC3A2		C3	0.5	3130	2.3	21
6	MC4, MF2		C4	0.5	4310	2.6	21
7	MF3		C2, C4	0.5	3490	7.1	21
8	MC3A3		C2, C3	0.5	2800	4.8	21

immediately after casting and continued for 30 h. The results were recording at 1 min intervals.

2.2. Wave reflection method

A model of an apparatus using the pulse USWR method has been already described by Valič [2]. In the exploration studies of the method and in particular of the apparatus several ideas for improvements came out. The corresponding modifications were implemented in a new apparatus, USWR-4 Hardening meter. Its basic components are: main frame box with transmitter/receiver electronics, A/D converter board and power supply, measuring heads, and PC with suitable software. Measuring head is of rugged construction and consists of a cylindrical aluminium body ($\Phi=30$, $l=40$ mm) in which a very pure fused quartz rod of rectangular cross-section ($a=10$, $b=16$ mm) and length $l=50$ mm is rigidly fastened. The two end surfaces of the quartz rod are flat, very parallel and highly polished. On one end (bottom) a PZE ultrasound transducer, acting as a transmitter and receiver, is hard bonded. On the other end (top), with a measuring surface of 2 cm^2, the sample to be tested is smeared.

Hydration/hardening process quite often lasts very long and the multitude/complexity from the influential parameters is high. For this reason, USWR-4 Hardening meter is constructed in a multi-head version with four measuring heads operating simultaneously. For temperature hydration/hardening dependence studies the measuring head as a whole could be inserted in a variable temperature oven. Detailed description of the principles of operation and performance of the apparatus can be found in Ref. [2].

Within the present study all reflection coefficient measurements started immediately after casting and continued for at least 30 h. The results were recorded at 1 min intervals.

3. Experimental program

3.1. Materials

Ten cement pastes were prepared in order to achieve the objective of this study. Four types of Portland cements, produced by Salonit Anhovo, were used (Table 1). In the table AC stands for the clinker content in each cement type.

In Table 2 the relevant characteristics of all (8 in total) mixtures used in this study are given. In the last column the sample temperature

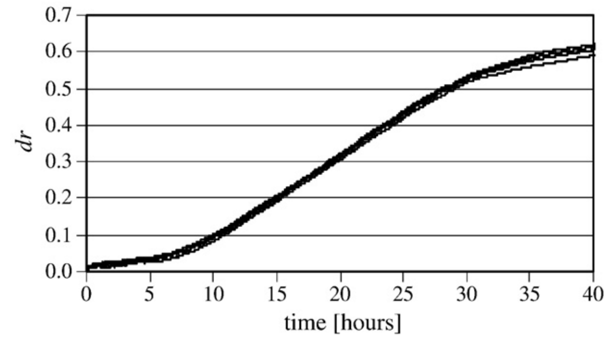


Fig. 1. Reproducibility of the USWR method used in the study.

during measurements are added. In order to get three different values of cement fineness, cement types C2 and C4 were used separately and one mixture (namely MF3) was prepared by mixing these two cements in the ratio of 50/50. This can be done because of very similar chemical composition of these three cements. A similar procedure was used in order to obtain three different values of C_3A content. Cement types C2 and C3 were used for this purpose.

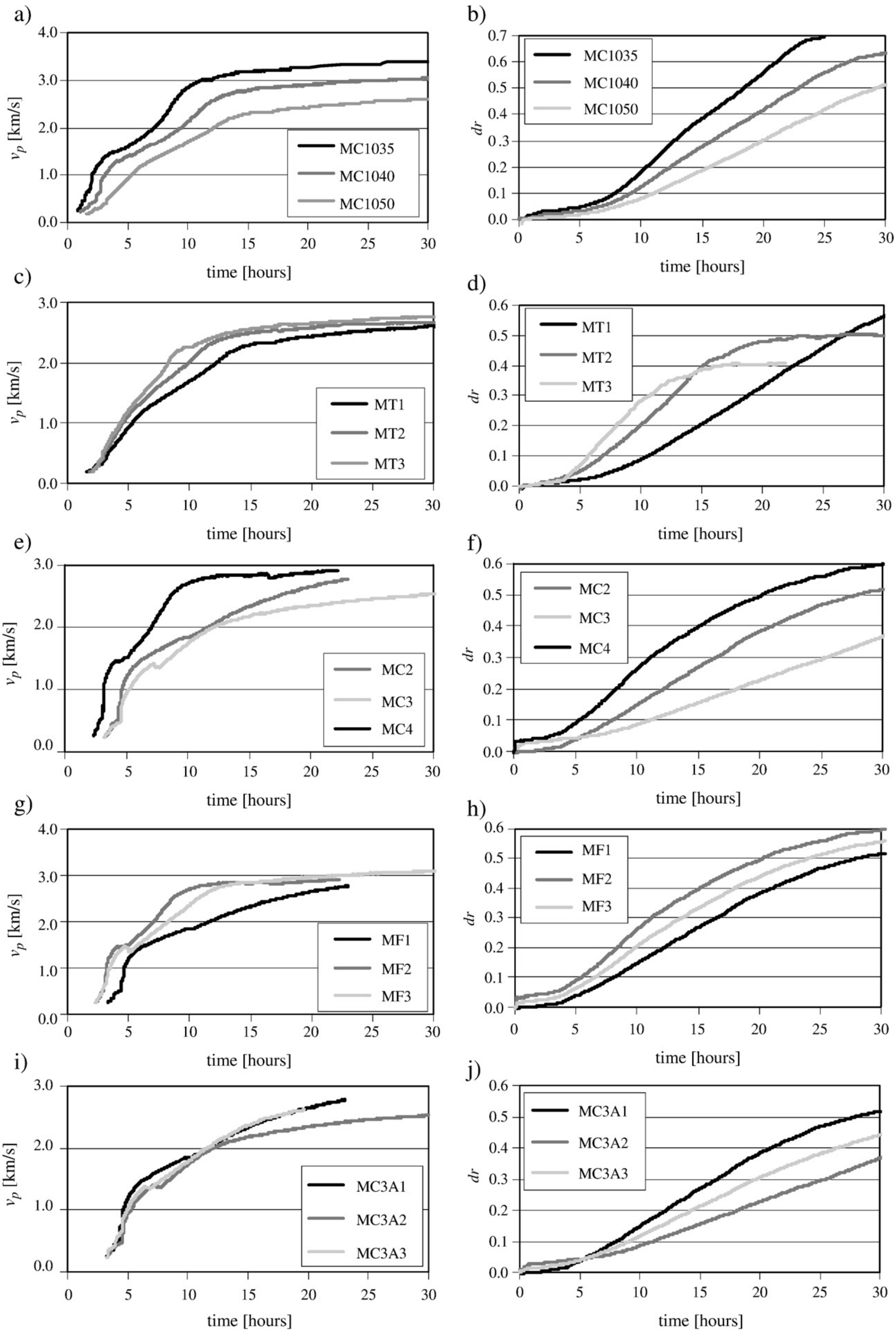
3.2. Reproducibility of the measurements

The reproducibility of the USWT method used in this study can be found in Ref. [11], while the results measured with the USWR method are presented in Fig. 1. It can be seen from this figure that the inherent scattering in this test is rather small and the equipment's reproducibility very high. Note: instead of the time dependence of the reflection coefficient $r(t)$, its complement, the change $dr(t)$ $[=1-r(t)]$ is plotted in all USWR figures in the study. In this way the USWR curves resemble the hydration and hardening growth with time. Based on the definition of the reflection coefficient, both r and dr values are expressed without units [2].

4. Experimental results

4.1. Sensitivity of USWT and USWR methods to monitor the setting process

In Fig. 2 the results on the sensitivity of both USWT and USWR methods on the hydration process and formation of internal rigid structures in different cement pastes are presented. It can be seen from Fig. 2a and b that mixtures with a higher w/c ratio show lower values of v_p and dr. In these experiments the temperature for the three samples was kept the same (T1). Further, from Fig. 2c and d it is seen that higher environmental temperatures result in a more rapid increase of v_p and dr values. In these experiments the sample composition is the same, namely MC1050. Interestingly, the USWR method is more sensitive to the change of the curing temperature than the USWT counterpart. A well known cross-over effect can be clearly seen from Fig. 2d which was also observed by some other researchers in their studies of the influence of the curing temperature on the development of concrete compressive strength and adiabatic hydration curves [15,16]. Fig. 2e and f presents the influence of the cement type on the evolution of v_p-t and $dr-t$ curves. As expected, cement type has a significant influence on the evolution of both v_p and dr values. The sensitivity of the USWR method is more pronounced. The influence of the cement fineness on the evolution of v_p and dr values with time can be seen in Fig. 2g and h. It is well known that higher fineness leads to a faster hydration process and consequently to the more rapid evolution of v_p and dr. The sensitivity of both techniques is comparable in this case. Fig. 2i and j demonstrates the influence of the amount of tri-calcium aluminate (C_3A) together with the influence of cement fineness (see Table 2). Even though the cement type C4 is of


a)
MC1035
MC1040
MC1050
v_p [km/s]
time [hours]
b)
MC1035
MC1040
MC1050
dr
time [hours]
c)
MT1
MT2
MT3
v_p [km/s]
time [hours]
d)
MT1
MT2
MT3
dr
time [hours]
e)
MC2
MC3
MC4
v_p [km/s]
time [hours]
f)
MC2
MC3
MC4
dr
time [hours]
g)
MF1
MF2
MF3
v_p [km/s]
time [hours]
h)
MF1
MF2
MF3
dr
time [hours]
i)
MC3A1
MC3A2
MC3A3
v_p [km/s]
time [hours]
j)
MC3A1
MC3A2
MC3A3
dr
time [hours]

higher fineness than cement type C3 it has a lower amount of C_3A, which finally results in the slower evolution of both v_p and dr values.

The conclusions of experimental results from Fig. 2 are: 1) Both USWT and USWR methods are able to reliably monitor the hydration process and formation of rigid structures in all cement pastes, used in this study. 2) The effects of cement paste initial parameters on the hydration process of cement-based materials are in good accordance with the well known rules of mix proportioning and other initial characteristics of fresh cement paste mixture, observed by some other researchers [15–17], and 3) The measurements with both methods conducted on cement pastes with different composition and hydration kinetics, evaluated on a qualitative basis, yield similar results.

4.2. Correlation between v_p and dr values for different cement pastes

In order to achieve the objective of this study, comparisons of the general evolution of v_p–t and dr–t curves have to be analyzed in more details first. In Fig. 3a and b the v_p–t and dr–t curves together with their numerical derivatives (v_p'–t and dr'–t) for the same cement paste MC1035 (Table 2) are presented.

It is seen from Fig. 3 that both USWT and USWR curves can be divided into five stages. Considering first the results in Fig. 3a, the sound speed v_p in the first stage, as pointed out already, could not be measured with the USWT experimental set-up used [11]. From that point on relatively low values of sound speed v_p are observed in the second stage of which upper bound is the first inflection point IP1 on the v_p–t curve. Observations of low speed values initially have been also reported and discussed by other researchers [12,18–20]. The time from the initial start of hydration to the first inflection point is designated as t_{IP1}. The third stage extends up to the second inflection point IP2 at which the hydration process, causing the fast increase in speed of sound, begins to slow down. The IP2 point defines the beginning of the stage 4 in this study which is extending until a plateau is reached [21]. From there on a long duration stage 5 follows in which speed of sound v_p continuously and very slowly increases with time. Inflection points become clearly distinctive in the derivative curves included in all diagrams of Fig. 3.

Considering the USWR results in Fig. 3b in a similar manner, the dr–t curve can also be divided into five stages. In stage 1 the finest cement grains hydrate in a very short period and a small increase of dr value is observed. This stage is not detected with USWT method [11]. In stage 2 (induction stage) a short plateau is reached after which, in stage 3, dr values start to increase slowly at first and faster later to a hardly distinctive inflection point DR. After the inflection point the rate of dr increase slows down somewhat, but continues to increase in stage 4 until a new plateau is reached, from which stage 5 follows. From the preceding results it follows, that both USWT and USWR methods are able to monitor the hydration processes of cement pastes. However, the results reveal that, originating from the different propagation properties of the p- and s-waves in a medium, the two ultrasonic methods monitor the setting processes of cement pastes in different ways. In Fig. 4 the relationships between v_p and dr values for all cement paste mixtures studied are presented. These diagrams were obtained from Fig. 2 by plotting dr vs. v_p values at equal times t. Noticeably and not surprisingly the dr–v_p curves in all diagrams of Fig. 4 are not equal for all cement pastes. There are larger and smaller discrepancies or the curves take similar course during certain stage. Far the largest deviations are produced by the amount of water added (Fig. 4a). Also, the slopes of dr–v_p curves change significantly meaning that USWT and USWR methods applied are not sensitive to the effects of cement paste initial parameters and of internal structures developing during hydration to the same degree. Included on each dr–v_p curve are

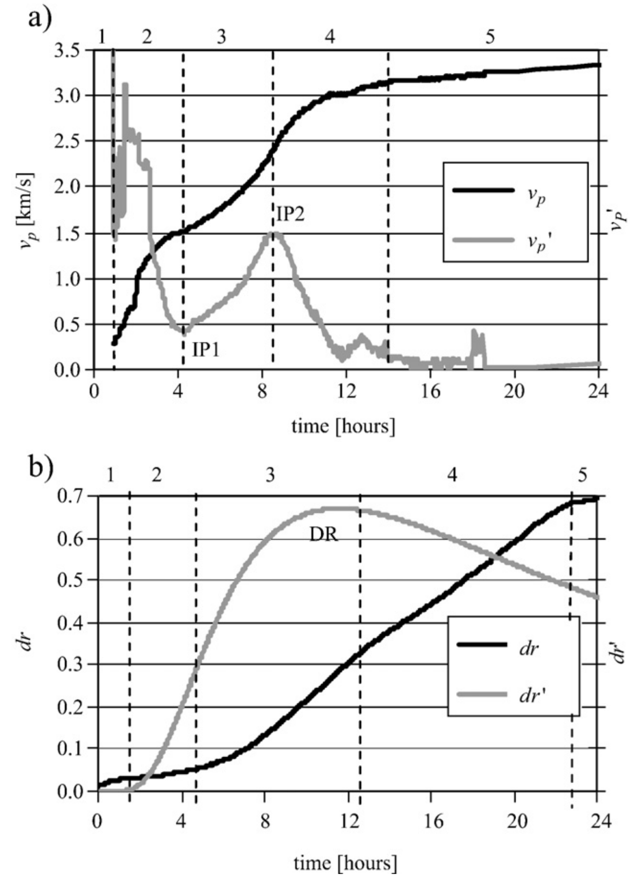


Fig. 3. Time evolution of hydration curves (mixtures MC1035); a) v_p–t and v_p'–t curves, b) dr–t and dr'–t curves.

characteristic inflection points (IP1, IP2, DR) discussed earlier and in some more details in the next chapter.

4.3. Characteristic points on the dr–v_p curves

On each of the experimental results in Fig. 2 some characteristic inflection points can be defined by way of example shown in Fig. 3: two (IP1, IP2) and one (DR) on each v_p–t and dr–t curve, respectively. The inflection points so found are entered also in each of dr–v_p curves of Fig. 4. All of these dr–v_p curves can be simplified into 3 characteristic phases as shown in Fig. 5. In phase 1 large increases of the ultrasonic pulse velocity of p-waves can be observed. On the other hand, the initial values of shear wave reflection coefficient do not change appreciably during this phase. This observation is indicated by an almost horizontal line in each dr–v_p curve in phase 1. This further indicates that v_p is very sensitive to the internal structures in cement pastes even before the initial setting time [11]. It is well known that v_p speed is strongly affected by the formation of ettringite crystals [21], which develop during the early age of the hydration process [15,22]. On the other hand formation of new internal structures in cement pastes do not seem to influence the shear wave reflection coefficient appreciably, which is in good agreement with the results presented by Voigt et al. [4]. This originates from the fact that s-waves cannot propagate through the suspension state of cement paste mixtures at the very beginning of the hydration process.

With continuing hydration the amount of solid products and the amount of connected solid phase increases rapidly [21]. Consequently,

Fig. 2. Comparison of sensitivity of USWT and USWR methods to the influence of different parameters on hydration process of cement pastes; w/c ratio (a,b), curing temperature (c,d), cement type (e,f), cement fineness (g,h), and cement composition (i,j).

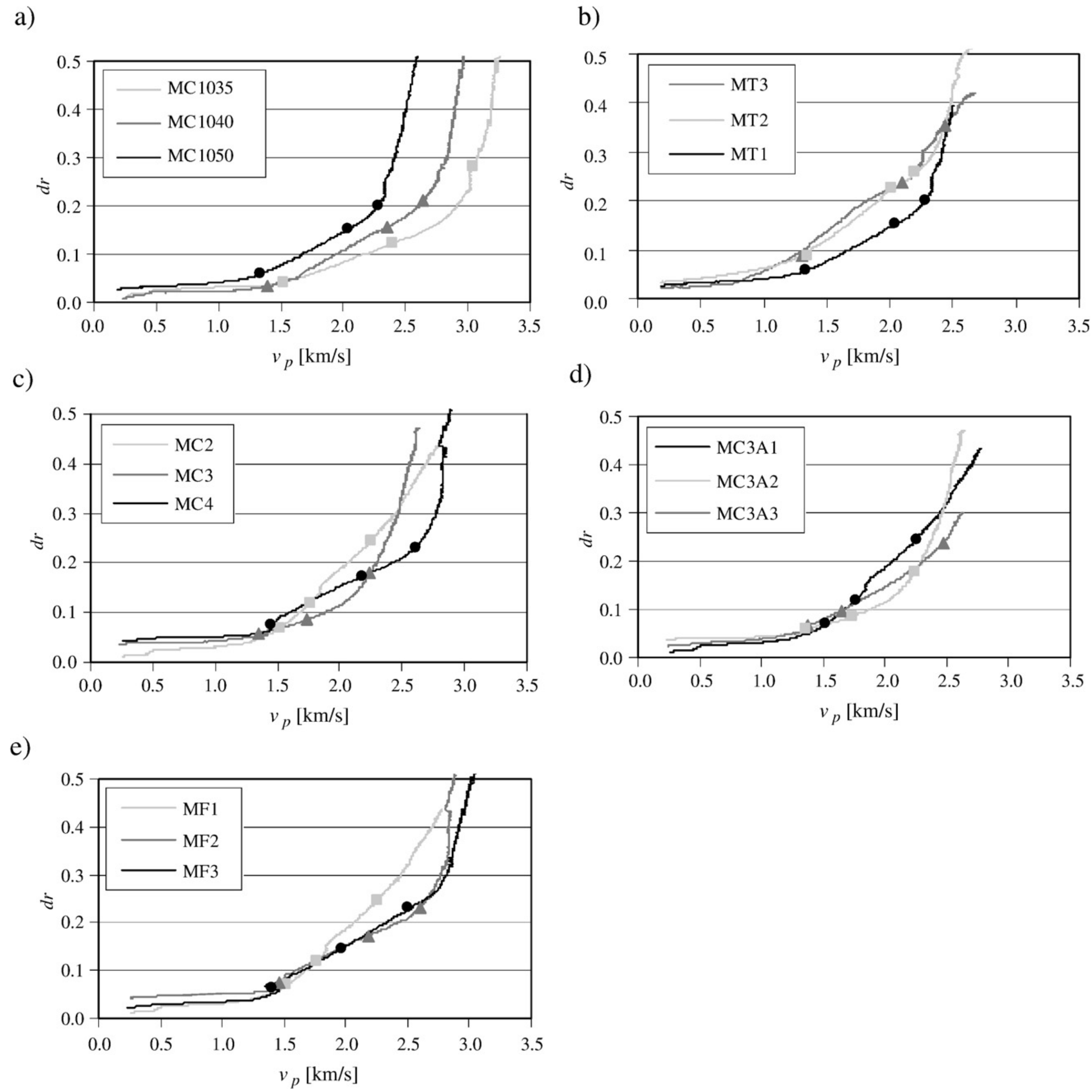


Fig. 4. dr–v_p relationships for all cement pastes, used in this study influenced by: a) w/c ratio, b) curing temperature, c) cement type, d) cement composition, and e) cement fineness.

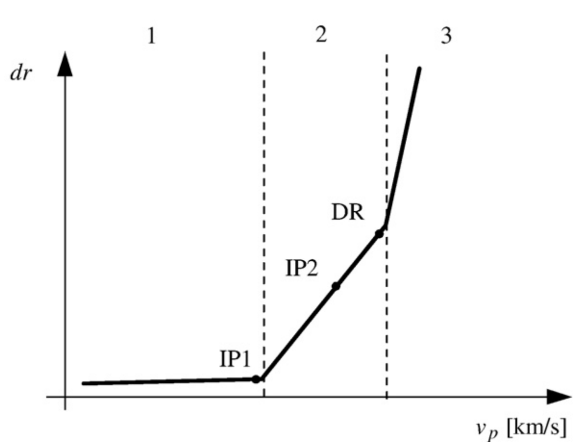


Fig. 5. Simplification of dr–v_p curves into three characteristic phases.

both v_p and dr values increase greatly during phase 2 as indicated by the steeper slope of the line during this phase on each of the dr–v_p curves. When the amount of solid phase reaches a certain value, the rate of increase of v_p values slows down while the values of dr continue to increase appreciably. This is indicated by almost vertical line during phase 3 of the dr–v_p curve in Fig. 5.

Included in Table 3 are the times t_i (i = IP1, IP2, DR) from the initial start of hydration to the individual inflection point (IP1, IP2, DR) and the corresponding v_{pi} and dr_i values of v_p and dr at all characteristic points shown on each dr–v_p curve in Fig. 4. From Fig. 4 it can also be seen that the first inflection point IP1 corresponds quite well with the end of the first phase on the dr–v_p curves. Next, it can be noticed that IP2 points occur within the phase 2 of the dr–v_p curves, while DR points appear more or less at the end of this second phase. This indicates that the maximum rate of v_p evolution, occurring at the second inflection point IP2, appears earlier than the maximum rate of dr evolution in all cement paste mixtures (Table 3). Moreover, when

Table 3
Characteristics of IP1, IP2, and DR inflection points for all cement paste mixtures, used in his study.

Mixture		Characteristic times [hours]			Values v_p [m/s]			Values dr [−]		
Label		t_{IP1}	t_{IP2}	t_{DR}	v_{pIP1}	v_{pIP2}	v_{pDR}	dr_{IP1}	dr_{IP2}	dr_{DR}
MC1035		4.3	8.5	12.3	1490	2390	3040	0.042	0.121	0.281
MC1040		5.3	11.0	12.8	1430	2360	2650	0.032	0.156	0.213
MC1050	MT1	7.2	12.5	14.6	1420	2040	2280	0.059	0.152	0.199
	MT2	5.8	10.4	13.4	1320	2090	2440	0.088	0.236	0.353
	MT3	5.1	8.3	8.5	1350	2020	2200	0.086	0.225	0.260
MC2, MF1, MC3A1		6.7	8.9	14.1	1520	1760	2250	0.070	0.119	0.245
MC3, MC3A2		7.7	10.0	16.7	1370	1740	2240	0.059	0.086	0.179
MC4, MF2		4.5	7.6	9.2	1450	2180	2610	0.076	0.171	0.232
MF3		4.9	8.1	10.2	1400	1970	2500	0.062	0.145	0.230
MC3A3		7.1	9.0	16.5	1370	1650	2480	0.067	0.097	0.238

the maximum rate of dr evolution appears, the evolution of v_p has almost reached the plateau value.

Finally, the times (t_{IP1}, t_{IP2}, t_{DR}) are shorter in the case of cement type C2, lower w/c ratio, higher curing temperature, higher cement fineness, and higher amount of C_3A.

4.4. Estimation of the initial setting time of cement pastes with combined ultrasonic method

Initial and final setting times are considered as two critical points during cement hydration. In this study, initial and final setting times were determined with standard Vicat method [23]. Penetration tests were performed at regular time intervals until the cement paste was completely set. The mean values of readings from three batches were used to define the initial t_{VI} and final t_{VF} setting time of each cement paste mixture. The results are summarized in Table 4 in which (v_{pVI}, v_{pVF}) and (dr_{VI}, dr_{VF}) stand for values of (v_p, dr) at times (t_{VI}, t_{VF}), respectively.

In comparing the results of Tables 3 and 4 it can be seen that the times t_{IP1} of the first inflection point on the dr–v_p curves correspond very well with the initial setting time t_{VI} for all cement paste mixtures at the same (room) temperature, investigated in this study. This is in a good agreement with the results obtained by Robeyst et al. [12]. Detailed description of this phenomenon can be found in Ref. [11]. Therefore, it follows that the beginning of the setting process of an arbitrary cement paste could be indicated as the end of the first (horizontal) phase on the corresponding dr–v_p curve. At times t_{IP1} ($\approx t_{VI}$) the value v_{pVI} reaches the ultrasonic speed of p-waves in water (1430 m/s) and the values of dr start to increase rapidly. Next, it can be seen that the v_{pVF} values are quite similar for all cement paste mixtures at the same temperature. A convenient approach would be to define the final setting time as the time when the v_p value reaches a value of about 1650 m/s, which is the average of 8 samples in Table 4. This means that the final Vicat setting time occurs within the second phase of the dr–v_p curve. A similar observation is not found with dr results.

Table 4
Initial and final setting time data for all cement paste mixtures, used in this study.

Mixture		Setting times [hours]		Values v_p [m/s]		Values dr [−]	
Label		$t_{V,I}$	$t_{V,F}$	v_{pVI}	v_{pVF}	dr_{VI}	dr_{VF}
MC1035		4.6	5.5	1510	1630	0.044	0.053
MC1040		5.4	6.8	1430	1640	0.034	0.049
MC1050	MT1	7.1	9.0	1420	1600	0.054	0.085
	MT2	5.8	6.6	1320	1480	0.088	0.113
	MT3	5.1	5.8	1350	1420	0.086	0.119
MC2, MF1, MC3A1		6.7	8.4	1520	1680	0.070	0.108
MC3, MC3A2		7.9	9.5	1400	1660	0.060	0.079
MC4, MF2		4.4	5.5	1450	1680	0.074	0.103
MF3		5.0	6.5	1420	1630	0.063	0.099
MC3A3		7.3	8.9	1410	1640	0.069	0.095

5. Conclusions

The correlation between the ultrasonic wave transmission method and ultrasonic wave reflection method in their ability to monitor the setting process of an arbitrary cement paste was analyzed. From the investigations described in this paper, the following conclusions can be drawn:

1. Both USWT and USWR methods are able to reliably monitor the hydration process and formation of structure of an arbitrary cement paste. Measurements with both USWT and USWR methods conducted on cement pastes with different composition and hydration kinetics, evaluated on a qualitative basis, yield similar results.
2. Direct relationship between v_p and dr values reveals that the two ultrasonic methods monitor the setting process of cement pastes in different ways. Two inflection points were observed on the v_p–t curves and only one on the dr–t curves.
3. The maximum rate in v_p evolution appears earlier than the maximum rate of evolution of dr.
4. The experimental dr–v_p diagrams can be simplified into three characteristic phases. Almost a horizontal line during the phase 1 indicates that p-wave speed v_p is very sensitive to the internal structure of the cement paste at the very beginning of the hydration process. On the contrary, the differences in the internal structure of the cement paste do not seem to influence the initial values of the shear wave reflection coefficient.
5. When the amount of the solid phase reaches a certain value, the rate of v_p increase slows down. However, at this point the dr values keep on increasing even more.
6. Using combined USWT–USWR ultrasonic method, the beginning of the setting process of an arbitrary cement paste can be determined by the end of the first phase on the dr–v_p curve.

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