



ORIGINAL ARTICLE

# Ultrasound monitoring of setting behavior of concrete mixtures

*Monitoramento do período de pega de misturas de concreto com ultrassom*

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**Abstract:** The setting behavior of concrete mixtures can be indirectly monitored by the penetration resistance test given by ASTM C 403. Arbitrary values of penetration resistance are associated to the initial and final set times. This test is carried out on the previously sieved mortar portion of the concrete mixture, which makes its practical use rare. Alternatively, ultrasound testing can be used to characterize this transition period, since the propagation of ultrasonic waves is greatly affected by the formation of cement hydration products and the extent of microstructure development. In this research, the propagation of ultrasonic waves in fresh concrete during the setting period was studied. In addition to the ultrasonic pulse velocity (UPV), other ultrasonic parameters such as group velocity, parameters related to ultrasonic energy and frequency were monitored during the setting period of various concrete mixes. It was observed that as setting progressed, there was a greater ease of ultrasonic transmission with a continuous increase of wave amplitudes. The results indicated that a combined analysis of the time domain parameters of UPV, group velocity and the time that 10% of energy has propagated ( $t_{10}$ ) could be used to improve the characterization of the microstructure development regarding the setting behavior of concrete mixtures.

**Keywords:** fresh concrete, setting time, ultrasonic wave propagation, ultrasonic waveform parameters, equivalent ages.

**Resumo:** O período de pega do concreto pode ser monitorado indiretamente pelo teste de resistência à penetração fornecido pela ASTM C 403. Valores arbitrários de resistência à penetração são associados aos tempos de início e fim de pega. Este ensaio é realizado na porção de argamassa previamente peneirada da mistura de concreto, o que torna rara sua utilização prática. Alternativamente, o teste de ultrassom pode ser empregado para caracterizar esse período de transição, uma vez que a propagação das ondas ultrassônicas é grandemente afetada pela formação dos produtos de hidratação do cimento e pela extensão do desenvolvimento da microestrutura. Nesta pesquisa, estudou-se a propagação de ondas ultrassônicas no concreto fresco durante o período de pega. Além da velocidade do pulso ultrassônico (UPV), outros parâmetros ultrassônicos, como velocidade de grupo, parâmetros relacionados à energia ultrassônica e frequência, foram monitorados durante o período de pega de várias misturas de concreto. Observou-se uma maior facilidade de transmissão ultrassônica com aumento contínuo das amplitudes das ondas durante o período de pega. Os resultados indicaram que uma análise combinada dos parâmetros no domínio de tempo como UPV, velocidade de grupo e tempo de propagação de 10% da energia ( $t_{10}$ ) poderia ser utilizada para melhorar a caracterização do desenvolvimento da microestrutura em relação ao comportamento de pega de misturas de concreto.

**Palavras-chave:** concreto fresco, tempo de pega, propagação de ondas ultrassônicas, parâmetros do formato de ondas ultrassônicas, idade equivalente.

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Corresponding author: Roberto Caldas de Andrade Pinto. E-mail: [r.pinto@ufsc.br](mailto:r.pinto@ufsc.br)**Financial support:** This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior – Brasil (CAPES) – Finance Code 001.**Conflict of interest:** Nothing to declare.**Data Availability:** the data that support the findings of this study are openly available in Repositório Institucional - UFSC at <https://repositorio.ufsc.br/handle/123456789/211574>.

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## 1 INTRODUCTION

Concrete, like other cement-based products, is a material that undergoes a gradual and progressive transformation over time. Cementitious compositions in their initial stages are plastic and workable while at later stages they become rigid structures. Concrete's transition period from a liquid to a brittle solid is commonly called as the setting period. This stiffening process depends on the development of the concrete microstructure, which will be the main agent responsible for the desirable mechanical properties required in civil engineering projects.

The setting phase begins with the total loss of plasticity and workability of the material; setting ends with the beginning of the  $C_3S$  hydration, one of the principal chemical compounds of cement. The method to determine initial and final setting times of a concrete mixture is based on the penetration resistance of its mortar portion. ASTM C403 [1] indicates the initial setting time of concrete as the time required for the sieved mortar to reach a penetration resistance of 3.5 MPa, while final setting time is established when the penetration resistance reaches 27.6 MPa. However, the initial and final setting times obtained using this method are considered purely reference points, since it is not possible to correlate the results of penetration resistance measurements with the development of the material's microstructure. These times do not represent a specific change in the physical-chemical characteristics of the cement paste.

Instead, as shown by Abel et al. [2], the setting times given by the penetration resistance method are related to the time when the concrete mixture becomes unworkable (achieves zero-slump), and to the time at which carefully handled concrete specimens can be broken in compression. Reaching final set, as indicated by the ASTM method, does not indicate that concrete has become a solid material, but simply that concrete can no longer be evaluated by this test. Moreover, in order to determine the setting times by means of this method, it is necessary to sieve the concrete in order to remove the mortar portion, which makes this method unusual in practice.

On the other hand, changes in the concrete microstructure during setting of the concrete also alter other properties of the material, in addition to its penetration resistance. The use of a non-destructive test method, such as the ultrasound directly on the concrete mixture, and not on its mortar portion, allows for real-time monitoring of the development of the microstructure.

Changes in the ultrasonic waveforms are associated with physical changes in the material's microstructure [3]. Although the main parameter obtained in the ultrasonic test is the Ultrasonic Pulse Velocity (UPV), other parameters associated with the waveform can be determined by considering the entire behavior of the ultrasonic pulse. These parameters may be more sensitive and efficient in the characterization of the tested material.

The stiffening of the mixture not only allows stress waves to propagate more quickly, but also that less wave attenuation occurs. A typically ultrasonic waveform is shown in Figure 1. The UPV is obtained by measuring the elapsed propagation time between two transducers placed at a known distance. Alternatively, one could calculate the group velocity as the velocity with which the major part of the energy propagates. It can be calculated from the waveform signal by recording the time at maximum amplitude [4].

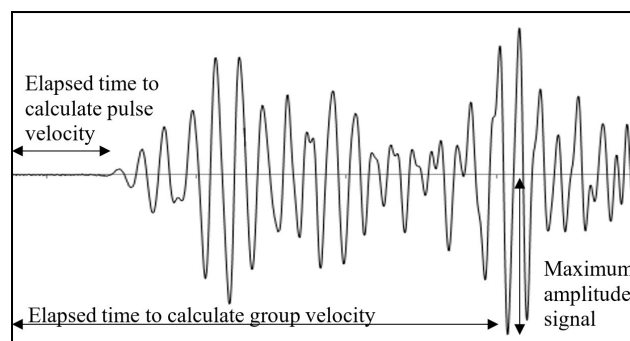


Figure 1. Typical ultrasonic waveform

The quantification of the energy of the ultrasonic signal can be adopted as the area under the rectified signal of the wave envelope according to Equation 1, with  $t_f$  being the end of the time window defined in the experiment and  $A(t)$  the amplitudes of the signal as a function of time [5].

$$E = \int_0^{t_f} |A(t)| dt \tag{1}$$

Shiotani and Aggelis [4] using Equation 1, showed that the accumulated energy curve underwent a decrease in its initial inclination with an increase of small plastic inclusions in mortar specimens. Contents of 1%, 5% and 10% in total mortar volume were used to simulate materials with different levels of internal damage. Even though this finding was related to damaged materials, this approach can also be applied to different levels of homogeneity achieved during setting.

This potential delay of energy arrivals can be described by the time that a certain small percentage of the accumulated energy arrived. In this study, the time at which 10% and 25% of total energy has propagated ( $t_{10}$  and  $t_{25}$ , respectively) was explored, as given by Equation 2 and Equation 3.

$$10\% = \frac{\int_0^{t_{10}} |A(t)| dt}{\int_0^{t_f} |A(t)| dt} \tag{2}$$

$$25\% = \frac{\int_0^{t_{25}} |A(t)| dt}{\int_0^{t_f} |A(t)| dt} \tag{3}$$

In addition, the ultrasonic signal can also be analyzed in the frequency domain, through a Fast Fourier Transform (FFT) yielding frequency-related parameters, such as the peak frequency and center frequency. The former is defined as the frequency value corresponding to the largest amplitude in the frequency spectrum, while the latter is defined as the centroid of the frequency spectrum, according to Equation 4.

$$C = \frac{\int_0^y f M(f) df}{\int_0^y M(f) df} \tag{4}$$

where  $C$  = center frequency (kHz);  $f$  = frequency (kHz);  $M(f)$  = frequency magnitude;  $y$  = frequency limit.

This research explores other stress wave parameters besides UPV, such as group velocity, energy and frequency associated parameters to characterize setting of concrete mixtures. Concrete mixtures with varying water-cement ratios were produced in laboratory. Their initial and final setting times were determined by ASTM C403 [1]. The evolution of ultrasonic parameters during these early ages was monitored. The results indicate a clear sensibility between wave parameters and the development of setting of concrete mixtures.

## 2 MATERIALS AND METHODS

Six concrete mixtures were produced in the laboratory. Concrete mixture proportions are presented in Table 1. The paste content and the mortar content were kept constant at 32% and at 55% for all mixtures, respectively. The fine aggregates consisted of a mixture of 70% of granite rock crushing sand with a fineness modulus of 2.84, and 30% natural sand with fineness modulus of 0.58. A granite coarse aggregate with maximum aggregate size of 19 mm, and a Brazilian early-strength cement CPV-ARI, similar to ASTM Type III cement, were used. The addition of a polycarboxylate-based superplasticizer was necessary in different amounts to achieve a slump of  $150 \pm 10$  mm for all concrete mixtures.

**Table 1.** Concrete mixtures proportions (kg/m<sup>3</sup>)

Mixture	w/c	Cement	Manufactured sand	Natural sand	Coarse aggregate	Water	Admixture
C40	0.40	445	575	243	980	178	1.65
C45	0.45	416	583	246	969	187	1.28
C50	0.50	391	590	249	959	195	0.98
C55	0.55	368	596	251	951	203	0.61
C60	0.60	348	601	254	943	209	0.21
C65	0.65	330	606	256	936	215	-

For each concrete mixture, three mortar cylinder samples of 150 mm x 150 mm and one concrete cube sample of 200 mm x 200 mm x 200 mm were cast. The mortar samples were used to evaluate the penetration resistance according to ASTM C403 [1], while the concrete sample was employed for ultrasound testing.

Thus, freshly prepared concrete was sieved with a 4.75 mm opening sieve and the resulting mortar placed in the cylindrical molds. Penetration resistance readings were performed using a Proctor equipment, with standard needles of different cross section areas. The penetration resistance was determined by the pressure required to penetrate 25 mm over 10 seconds. The initial and final setting times were determined as the times when the penetration resistance reached 3.5 MPa and 27.6 MPa, respectively. Mortar temperatures were monitored by thermocouples immersed in the samples.

The ultrasound measurements were performed with a commercially available equipment using 54 kHz frequency transducers at an acquisition frequency of 2 MHz. A layer of an acoustic coupler was applied between the transducers and the concrete sample. For each mixture, ultrasonic monitoring started immediately after the concrete cubic sample has been cast and ended two hours after final set given by the penetration resistance test. Mortar penetration resistance and concrete ultrasonic measurements were performed simultaneously.

The apparatus used for ultrasonic testing in fresh concrete was developed by Irrigaray [6]. It consists of an external structure in plywood with circular openings, centered on opposite faces that serve as a guide for placing the transducers. The apparatus was filled with fresh concrete in two layers of equal height (approximately 100 mm). Each layer was manually consolidated by applying 20 strokes with a metallic rod. Any internal void was eliminated with light strokes on the external face of the apparatus. Thermocouples at mid-height were embedded in the sample in order to monitor concrete temperature during ultrasonic monitoring.

Ultrasonic waveforms were acquired at a five-minute interval. Initially, ultrasonic signal de-noising was performed by a filtering algorithm developed in the Matlab® software. A low-pass filter was applied to the frequency-domain data. Then, from the filtered ultrasonic signals, UPV, group velocity,  $t_{10}$  and  $t_{25}$  were determined, as well as the peak frequency and the central frequency, for each acquired waveform.

The concrete mixtures were prepared on different days, at different environmental temperature conditions. Although they were placed in a controlled temperature environment, the specimens underwent different temperature evolutions. Therefore, the maturity method was used to take into account the influence of the different concrete and mortar temperatures.

The Freiesleben-Hansen and Pedersen Maturity Function (FHP) was used, as indicated by ASTM C1074 [7]. Regarding the apparent activation energy value, a value of 30 kJ/mol was applied. This value lies between the values presented by Pinto and Schindler [8] and Carette and Staquet [9] for the activation energy during the setting period. Equation 5 presents the FHP function for calculating the equivalent age at a reference temperature of 20°C.

$$t_e = \sum_0^t \exp \left[ \frac{-E_a}{R} \left( \frac{1}{T+273} - \frac{1}{293} \right) \right] \Delta t \tag{5}$$

where  $t_e$  – equivalent age at a 20°C;  $E_a$  – apparent activation energy (30000 J/mol);  $R$  – universal gas constant (8.314 J/mol/K);  $T$  – concrete or mortar temperature (°C);  $\Delta t$  – time interval.

Thus, the initial and final setting times obtained by the penetration resistance test, as well as the elapsed times of each waveform acquired in the concrete cube, were transformed into equivalent ages according to Equation 5, using the temperature values given by the thermocouples.

### 3 RESULTS AND DISCUSSION

#### 3.1 Penetrations Resistance Test

Table 2 shows the initial and final setting times for each concrete mixture. The results represent the average of the values obtained at each cylinder specimen. Figure 2 shows the temperature history, as well as the initial and final setting times for each mixture.

**Table 2.** Initial setting time, final setting time and setting duration per mixture

Mixture	Initial setting (min.)	Final setting (min.)	Setting duration (min.)
C40	286	378	92
C45	270	355	85
C50	326	420	94
C55	294	378	84
C60	288	363	75
C65	295	380	85

Although there were observed differences in setting times, it can be noticed, from Figure 2, that the initial set generally occurred when the generation of hydration heat began, and the mortar temperature started to increase. The temperatures continued to increase after final set for all mixtures.

The temperature of the concrete cubes was always higher than that of the mortar cylinders. Thus, the evolution of the concrete cube microstructure could not be directly related to the setting times obtained in the mortar cylinders. Temperature influences the rate of cement hydration; a higher rate of cement hydration is expected at high temperatures. Therefore, the initial and final setting times obtained by the mortar penetration resistance test did not correspond directly to the setting times of the concrete sample. Setting in the concrete sample should have occurred earlier than setting given by the penetration resistance test, due to higher concrete temperatures.

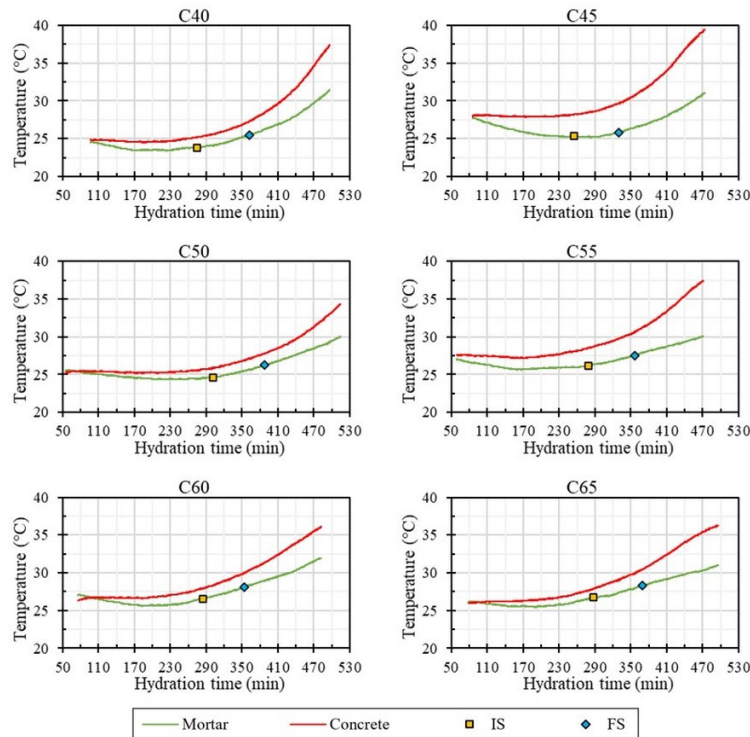


Figure 2. Temperature evolution of all mixtures

In order to account for such differences in temperatures, the maturity method was used to correlate the behavior of the concrete ultrasonic parameters during setting with the times given by the mortar penetration resistance method. Initially, Equation 5 was applied together with the temperature history of the mortar specimens (Figure 2) to transform actual initial and final setting times given in Table 2 in equivalent ages. The resultant equivalent ages at initial and final setting times are presented in Table 3. With the equivalent ages calculated, Equation 5 was again applied considering the concrete temperature history. The actual times of the concrete samples corresponding to the equivalent ages in Table 3 were obtained. Such values are presented in Table 4.

Table 3. Initial and Final setting times in equivalent ages

Mixture	Initial setting (min. at 20°C)	Final setting (min. at 20°C)	Setting duration (min. at 20°C)
C40	334	446	112
C45	354	461	107
C50	399	519	120
C55	380	493	113
C60	374	476	102
C65	376	493	117

**Table 4.** Actual times of concrete samples when setting in the mortar samples occurred.

Mixture	Initial setting (min.)	Final setting (min.)
C40	274	362
C45	255	330
C50	302	387
C55	279	356
C60	284	354
C65	288	369

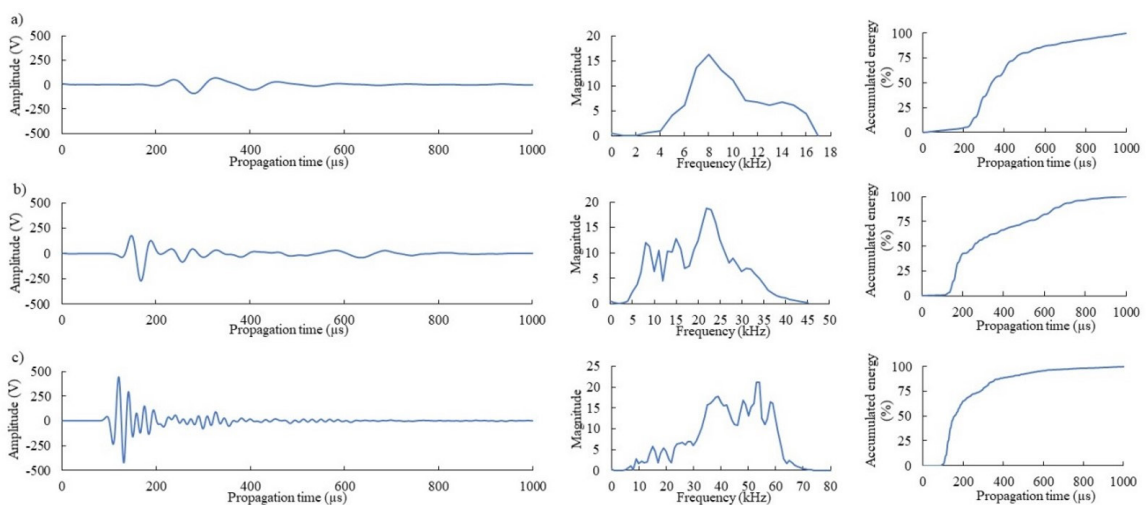
Table 3 does not show any trend regarding setting times and the w/c ratio of the mixtures studied here. One would expect that, as the w/c ratio decreased, the setting times for the concrete mixtures would also decrease. However, Table 3 shows a small variation in the final and initial setting times, and similar duration of the setting period. Different amounts of superplasticizer were used in each mix, as can be seen in Table 1, mixtures with lower w/c ratio required greater amount of superplasticizer to maintain similar workability. Therefore, any possible acceleration effect expected for low w/c mixes may have been overcome by the greater superplasticizer amount employed in such mixtures.

Since the temperature of the concrete samples were always higher than that of the mortar samples, when final setting occurred, as given by the mortar penetration resistance, the corresponding concrete sample has already set. The observed difference between final setting in mortar and in the concrete specimens, as given in Tables 2 and 4 varied from 9 to 33 minutes depending on the mixture and temperature histories. For instance, the equivalent age at final set for mixture C45 was 461 minutes at 20°C, which was achieved in the mortar specimen at 355 minutes and in the concrete sample at 330 minutes. Thus, final set in the concrete sample occurred 25 minutes before final set in the mortar sample for that particular mixture.

### 3.2 ULTRASONIC WAVEFORMS

Several ultrasonic waveforms were acquired for each mixture. For each waveform, UPV, group velocity, as well as the frequency spectrum and the normalized accumulated energy curve were calculated. The frequency spectrum would yield the peak frequency and the center frequency while the normalized accumulated energy would give the  $t_{10}$  and  $t_{25}$  energy parameters.

As an example, Figure 3 shows the acquired waveforms, and corresponding frequency spectra and normalized accumulated energy curves for mixture C40 at three different times. One close to initial set (275 min), another during the setting period (315 minutes), and the last at a time close to final set (360 minutes).



**Figure 3.** Ultrasonic waveforms, frequency spectrum and accumulated energy curves for C40: a) close to initial setting; (b) between initial and final setting (c) close to the final setting.

During monitoring, it was necessary to modify the amplitude gain of the ultrasonic signal in order to better visualize the waveform. Amplitude gains of 1000, 200 and 10 were used to acquire the waveforms presented in Figure 3. This variation in the amplitude gain did not alter the UPV, the group velocity, and any frequency-based parameters obtained from the waveform. Moreover, the energy related parameters used in this study were based on normalized amplitude values, as depicted in Equation 2 and Equation 3, and thus were also not affected by differences in amplitude gain of the ultrasonic signal.

Figure 3 shows that as setting progressed, a much more significant wave transmission occurred through the C40 specimen since a continuous increase of the stress wave amplitudes, as given by the ordinate magnitudes of Figure 3a-3c, could be observed. There was an observed shift of transmitted energy at earlier times. Also, the frequency spectra show that the dominant frequency component also changed as setting progressed. The dominant frequency was close to the center frequency of the transducers (54 kHz) at final setting.

Since waveforms were acquired at a five-minute interval, it was possible to obtain the development with time of the UPV, group velocity, peak and center frequencies, and the energy related parameters of  $t_{10}$  and  $t_{25}$  for each mixture. Figure 4 presents the development of these ultrasonic parameters at early ages for all mixtures. The time of initial and final setting, as given in Table 4 are also depicted in Figure 4.

From Figure 4, one could state that as concrete aged, cement hydration increased, since the values of the UPV and group velocity also increased with age. This behavior would continue to occur, albeit in a less accentuated way, even after the end of the setting period, as also seen by Chávez-García et al. [10] and Pellegrino et al. [11].

Table 5 presents the values of the ultrasonic parameters here investigated of the concrete samples obtained from the waveforms acquired when initial and final setting of the mortar samples occurred (Table 4).

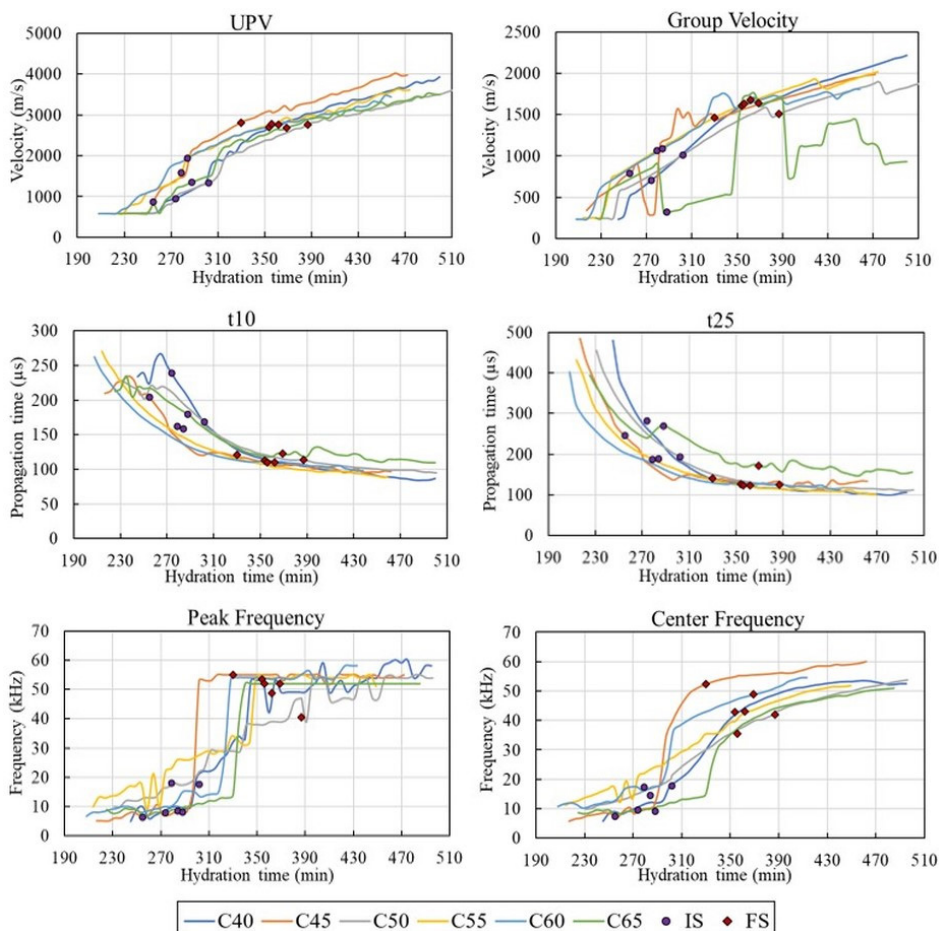


Figure 4. Development of ultrasonic parameters at early ages.

In order to better show the behavior of the ultrasound parameters during the setting period, and also to be able to correlate them with the results of the penetration resistance, the abscissa axis of Figure 4 was transformed to reflect the development of UPV during the setting period given by the penetration resistance method, as shown in Figure 5. The axis was divided in regular intervals depending on the percentage of the setting duration, with 0% corresponding to initial setting time and 100% corresponding to the final setting time.

**Table 5.** UPV, group velocity,  $t_{10}$ ,  $t_{25}$ , peak frequency and center frequency values at initial and final setting times

Mixture	UPV (m/s)	Group Velocity (m/s)	$t_{10}$ ( $\mu$ s)	$t_{25}$ ( $\mu$ s)	Peak Frequency (kHz)	Central Frequency (kHz)
Initial set						
C40	946	704	238	281	7.8	9.6
C45	866	790	204	245	6.4	7.5
C50	1338	1011	168	192	17.6	17.7
C55	1586	1066	162	187	18.0	17.4
C60	1937	1090	158	188	8.5	14.5
C65	1343	1022	179	269	8.0	9.15
Final set						
C40	2762	1680	110	123	48.6	43
C45	2816	1435	120	140	55.0	52.3
C50	2763	1509	113	125	40.4	40.3
C55	2781	1630	109	122	32.8	35.4
C60	2698	1610	112	126	53.5	42.9
C65	2681	1642	122	172	52.0	36.5

As far as UPV is concerned, Figure 4 shows an increase in UPV during setting for all mixtures. This behavior has been previously observed by several researchers [3], [9], [12]-[17]. At very early-ages, ultrasonic waves propagate into a viscous suspension, at low UPV values. After the dormant period has ended, cement hydration products start to be formed, and to be interconnected. Consequently, there is a sharper increase on the rate of UPV development with time. UPV continues to increase as hydration progresses. Mixtures C50 e C65 showed UPV close to 1430 m/s at initial setting, which is close to UPV in water. By contrast, mixtures C55 e C60 showed UPV greater than 1430 m/s, (1586 m/s e 1937 m/s, respectively), while mixtures C40 e C45 showed much smaller UPV values (946 m/s e 866 m/s) at initial setting.

Very low UPV values, as in C40 and C45, can be attributed to the elongation in the wave path, given the suspension of cement grains in the fresh concrete [18] or the possibility of the low w/c ratio having generated an insufficient union between the cement and water [19].

Even though there was not a direct correspondence between initial setting time and UPV, similar final setting times were observed for the concrete samples, as shown in Table 5. For the mixtures studied here, UPV at final set varied between 2681 m/s to 2816 m/s, with a mean value of 2750 m/s and a coefficient of variation of 1.9%. This UPV value at final set lies between the ones indicated by Lee and Lee [20] (1900 to 2900 m/s at final set).

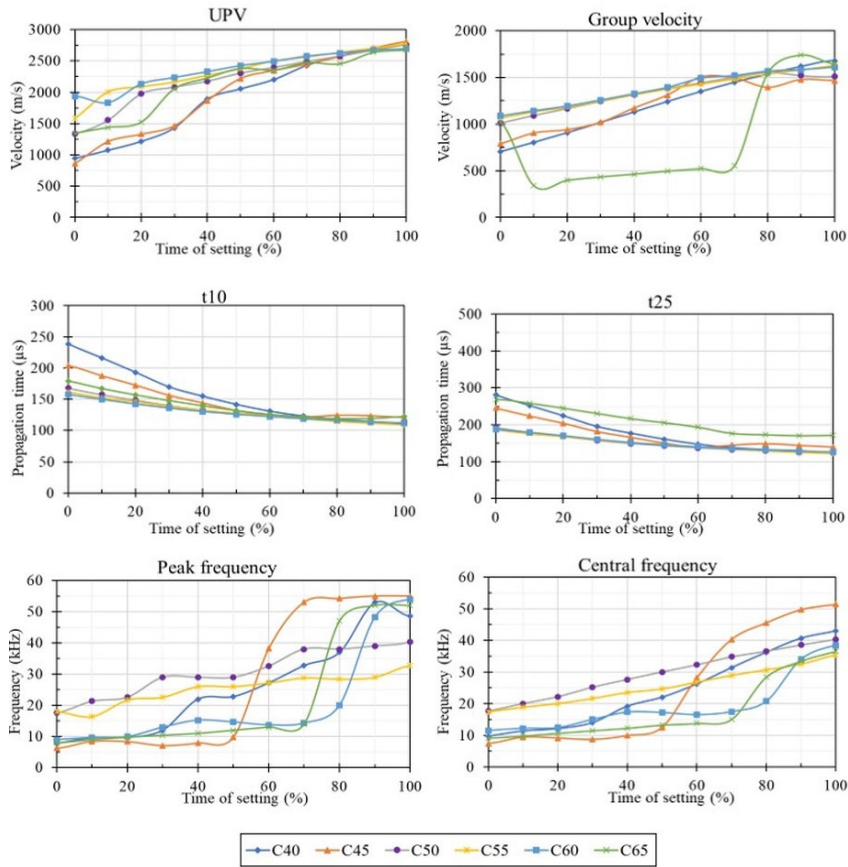
The group velocity also increased, indicating that there was an earlier shift of the most part of the energy. This behavior can also be seen in the waveforms presented in Figure 3 and in the observed decreasing values of the energy-related parameters of  $t_{10}$  and  $t_{25}$ . There was a greater ease of ultrasonic transmission as setting progressed. Even though, the group velocity development with setting time of mixture C65 showed a different behavior than the other mixtures, as shown in Figure 5, the group velocity values at final setting time were close for all mixtures, with an average value of 1585 m/s and a small coefficient of variation of 5.9%.

Figure 5 shows a significant decrease in  $t_{10}$  and  $t_{25}$  with increasing time of setting, as a consequence of an increase on the initial inclination of the accumulated energy curve, as can be seen in Figure 6, obtained for mixture C65. As the microstructure is being formed, most of the ultrasonic energy is able to propagate faster, resulting in a greater accumulation of energy at the beginning of the ultrasonic signal. Similarly to UPV and group velocity, there was not a unique value at initial setting, with  $t_{10}$  values ranging from 158  $\mu$ s to 238  $\mu$ s, and  $t_{25}$  values ranging from 187  $\mu$ s to 281  $\mu$ s. However, at final setting times, an average  $t_{10}$  value of 114  $\mu$ s (coefficient of variation of 5.4%) and  $t_{25}$  values ranging from 122  $\mu$ s to 172  $\mu$ s were obtained.

The peak and central frequencies of the ultrasonic waveform increased as setting progressed, as shown in Figure 5. The increase in the magnitude values indicates more significant wave transmission through the specimen. As stated by Lee et al. [14], when concrete is changing from a viscous suspension into a porous solid, low-frequency components of

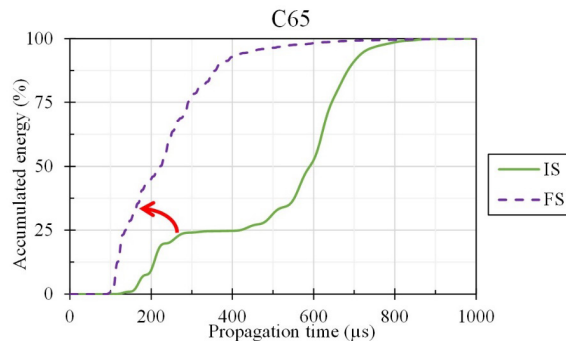


the ultrasonic waveform begin to propagate before the high-frequency components. With continuing hydration, and consequently establishment of the porous solid structure, there is an easier transmission of all frequency components.



**Figure 5.** UPV, group velocity,  $t_{10}$ ,  $t_{25}$ , peak frequency and center frequency development during setting.

Shiotani and Aggelis [4] observed decay in the central frequency for mixtures with higher amounts of plastic inclusions, indicating that frequency is related to homogeneity. This frequency-domain phenomenon only occurred during this early stage of microstructure development since as setting progressed, both the central frequency and the peak frequency tended to reach 54 kHz, the center frequency of the transducer. However, despite this continuous and similar increasing behavior, these frequency parameters seemed to be mixture dependent. It was not possible to indicate a central frequency value neither a peak frequency value at initial nor final setting for all mixtures.



**Figure 6.** Accumulated energy curves at initial setting (IS) and final setting (FS) to mixture C65.

## 4 CONCLUSIONS

Nondestructive tests have been widely used in the quality control of concrete structures. The propagation of stress waves is affected by changes in the microstructure of the material, making it possible to use ultrasound to monitor the development of the concrete microstructure. At early stages, due to high rates of cement hydration, the microstructure is in continuous development. The quality of the microstructure formed greatly influences the mechanical properties and durability of the concrete structure.

In this research, the propagation of ultrasonic waves in fresh concrete during the setting period was studied. As concrete moved from a plastic stage to a rigid solid, the ultrasonic wave format changed. Regarding the ultrasonic parameters investigated here, the following conclusions can be drawn:

- As setting progressed, there was an observed increase of wave amplitudes, as a result of a more significant wave transmission through concrete.
- None of the ultrasonic parameters investigated here was able to be correlated uniquely to initial set times as given by the penetration resistance method, since significant variations were observed in their values at initial set time.
- The UPV and group velocity values increased during setting for all mixtures, with mean values of 2750 m/s and 1585 m/s at final setting, respectively.
- An earlier shift of the accumulated energy occurred, and as a consequence, the energy-related parameters of  $t_{10}$  and  $t_{25}$  decreased as setting progressed. At final setting times, an average  $t_{10}$  value of 114  $\mu\text{s}$ , and  $t_{25}$  values ranging from 122  $\mu\text{s}$  to 172  $\mu\text{s}$  were obtained.
- The frequency parameters seemed to be mixture dependent. It was not possible to indicate a central frequency value neither a peak frequency value at initial setting for all mixtures.
- The peak and center frequencies was close to the frequency of the transducers (54 kHz) at final setting.

Although the initial and final setting times, as given by ASTM C403, are related to the concrete stiffening process, they do not represent when specific changes in the physical-chemical characteristics of the cement paste occur. Setting of concrete is a continuous phase in which the material's microstructure is constantly developing, as shown by the wide changes of the various ultrasonic parameters here investigated.

A combined analysis of the time domain parameters of UPV, group velocity and  $t_{10}$  can be used to improve the characterization of the microstructure development regarding the setting behavior of concrete mixtures.

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