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ULTRASONIC CHARACTERIZATION OF CEMENT COMPOSITES DURING HYDRATION

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Preliminary notes

Ultrasonic measurements used to monitor the hydration of cement based materials are becoming an important tool in the quality assurance in production. Also, using these methods can be cost benefit for the construction companies because they can provide information about evolution of material properties in real time. In this paper ultrasonic testing is used to characterize a hydration process of cement paste. A technique is based on the system for measuring the ultrasonic pulse velocity (UPV). To obtain more information about wave propagation characteristics the technique of acoustic emission (AE) is also used. Entire waveform of the signal is recorded. AE parameters are extracted from the signal and used to characterize different processes in cement paste during hydration. An analysis is performed on cement pastes made with different water-cement ratios. Ultrasonic wave parameters are compared to the initial and final setting time using the Vicat needle test and compressive strength development.

Keywords: *cement composites, hydration, ultrasonic testing*

Ultrazvučna karakterizacija cementnih kompozita tijekom hidratacije

Prethodno priopćenje

Korištenje ultrazvučnih mjerenja za praćenje hidratacije materijala na bazi cementa postaje važan alat osiguranja kvalitete u proizvodnji. Također, korištenje tih metoda može biti ekonomski isplativo za izvođačke tvrtke jer omogućavaju trenutno dobivanje informacija o razvoju svojstava materijala. U ovom radu ispitivanje ultrazvukom je primijenjeno za karakterizaciju procesa hidratacije cementne paste. Metoda mjerenja temeljena je na sustavu za mjerenje brzine ultrazvučnog impulsa (UPV). Kako bi se dobilo više informacija o svojstvima širenja valova korištena je i metoda akustične emisije (AE). Cijeli valni zapis je sniman te su iz signala izračunati parametri akustične emisije koji su korišteni za karakterizaciju različitih procesa u cementnoj pasti tijekom hidratacije. Analiza je provedena na cementnim pastama izrađenim s različitim vodo-cementnim omjerima. Parametri ultrazvučnih valova uspoređeni su s početkom i krajem vezanja dobivenim Vicatovom iglom i razvojem tlačne čvrstoće.

Ključne riječi: *cementni kompoziti, hidratacija, ispitivanje ultrazvukom*

1 Introduction
Uvod

Concrete setting and hardening processes are the most critical phases during construction works, influencing properties of a concrete structure. The transformation from liquid to solid state is characterised with two points in hydration time: initial and final setting. The initial set is important because it provides an estimate when concrete has reached the point of stiffening to such an extent that it can no longer form a monolithic element with concrete poured on top of it. The point at which final set occurs is important since it provides an estimate when the development of concrete strength and stiffness starts. Hardening process of concrete makes it possible for a concrete element to carry loads. Knowing the strength of the concrete during hardening is the tool to determine the moment of formwork removal, moment of prestressing or calculation of the load bearing capacity.

Non-destructive testing methods able to follow the setting and hardening processes of concrete are being developed. The mostly used method is the maturity method based on the temperature measurements. To improve understanding of the hydration process and setting times new methods are investigated. One group of methods consists of ultrasonic measuring techniques. Different techniques have been presented by many authors. Different types of ultrasonic waves are used, for example shear waves [1, 2] or longitudinal waves [3, 4, 5, 6]. Using these methods a finer representation of the hydration process is obtained, but there are still problems left unsolved.

2 Experimental works
Eksperimentalni rad

In order to characterize different stages of hydration in cement paste and physical changes that follow, an experimental work is performed under laboratory conditions to identify the setting and hardening of cement paste using ultrasonic waves during early ages. Changes in the ultrasonic wave parameters passing through the fresh cement paste and setting time determined using the Vicat test method are analyzed. Also, the compressive strength development of cement pastes in the first 7 days is tested.

2.1 Materials and mixing of methods
Materijali i metoda miješanja

Cement used in the analysis is CEM I 42,5R. Properties of cement are shown in Tab. 1. A cement paste is prepared with a water to cement (w/c) ratios 0.4, 0.5 and 0.6 by weight. The paste is mixed according to a procedure given by the standard HRN EN 196-3. Initial and final setting times of the cement paste are determined using the Vicat needle method according to the same standard. Initial setting is the age of the cement paste when the needle penetrating into paste stops in the range 6 ± 3 mm from the bottom of the mould. Final setting is the age at which needle penetrates less than 0,5 mm into the cement paste. Changes in the penetration depth during the testing of setting times are shown on Fig. 1. Time "0" on Fig. 1 is the time of mixing of cement and water.

Table 1 *Properties of the cement CEM I 42,5 R*
Tablica 1. *Svojstva cementa CEM I 42,5 R*

Density, kg/m³	Fineness passing 45 µm, %	Blaine, m²/kg	Mineral composition, %			
			C_3S	C_2S	C_3A	C_4AF
3120	92,2	353	59,86	11,49	8,36	8,82

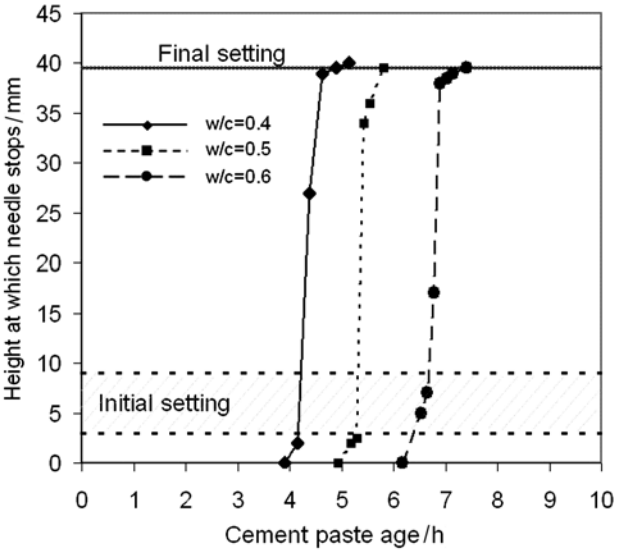

Figure 1 *Changes in the penetration depth of standardized needle into the cement paste*
Slika 1. *Promjene dubine penetracije standardizirane igle u cementnu pastu*

The compressive strength of pure cement paste is tested on the samples with dimensions 40×40×160 mm. Samples are cured at the ambient temperature of 20 ± 1 °C and covered with a plastic sheet to prevent evaporation. When samples had strengthen enough they were removed from the moulds and stored into climatic chamber where they were air cured at the 20 ± 1 °C and relative humidity > 95 %. The compressive strength is tested at different time intervals (in the first 24 hours, 24, 48, 72 and 168 hours) after mixing. At each time interval 3 samples were broken and the result is an average value of the compressive strength. Results of the compressive strength tests are shown on Fig. 2.

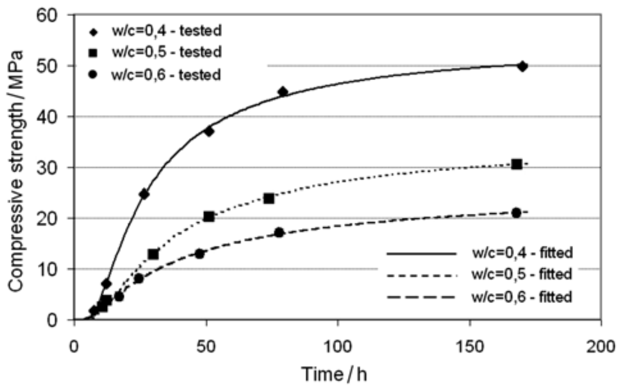

Figure 2 *Compressive strength development of pure cement pastes*
Slika 2. *Razvoj tlačne čvrstoće čiste cementne paste*

2.2 Ultrasonic setup
Ultrazvučni sustav

The measuring system consists of the Portable Ultrasonic Non-Destructive Digital Indicating Tester or shorter Pundit produced by C.N.S. Electronics. It generates low frequency (54 kHz) ultrasonic pulses and measures the time taken for them to pass from one transducer to the other through the material interposed between them. A modification of the original system was made in order to be able to record information about the time of flight continuously to a PC. Software was also developed for recording data in ASCII format and controlling the frequency of data recording. A wave signal from the receiving transducer is also collected by an acquisition unit for the acoustic emission (AE) measurement. The acquisition system used is the Physical Acoustics Corporation acoustic emission 8-channel system PCI-8 controlled through the AEwin software. From the recorded ultrasonic waves different wave parameter, typically used in the acoustic emission measurements, are then extracted. A configuration of the ultrasonic setup is shown in Fig. 3. Transducers are set in through transmission so that speed of the ultrasonic wave can be calculated by knowing the distance between the transducers and the time take for the pulse to travel between them. Distance between the transducers i.e., length of the specimen is 7 cm. Mould used for testing is made of extruded polystyrene. That way the pulse is transmitted directly through the specimen and not through the sides of the mould.

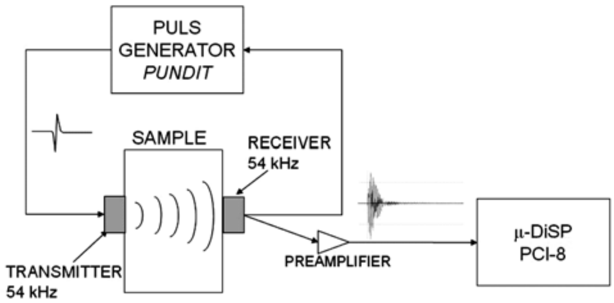

Figure 3 *Scheme of the ultrasonic setup used during testing*
Slika 3. *Shema ultrazvučnog sustava korištenog tijekom ispitivanja*

3 Analysis of ultrasonic wave characteristics
Analiza karakteristika ultrazvučnih valova

During the analysis it was observed that there are different wave parameters that are sensitive to the changes in the structure of cement paste. It is noticed that some parameters change rapidly during the transformation of paste from a liquid to hardened state and after the transformation rate of changes drops significantly. Some other parameters show a different trend which is closer to the trend of hardening, i.e. strength development. For this reason ultrasonic wave parameters were divided into two categories, the first representing the period of setting and the second representing hardening of the cement paste.

3.1
Setting of cement paste
Vezanje cementne paste

Results obtained from the ultrasonic measurements showed three parameters that change significantly during transformation of cement paste from the liquid to solid state. Those parameters are:

- Amplitude - defined as the maximum (positive or negative) of the signal in one wave.
- Duration - defined as the time from the first threshold (detection of the signal) crossing to the end of the last threshold crossing of the signal in one wave.
- Risetime - defined as the time between the first threshold crossing and the peak amplitude of the signal in one wave.

These parameters are shown in Fig. 4.

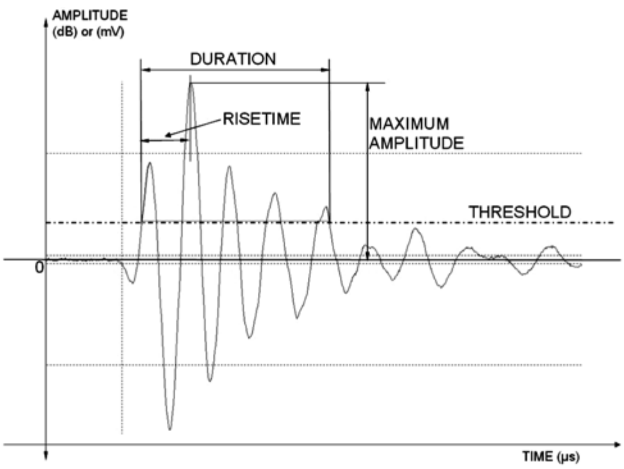

Figure 4 *AE parameters extracted from the waveform*
Slika 4. *AE parametri izračunati iz zapisa vala*

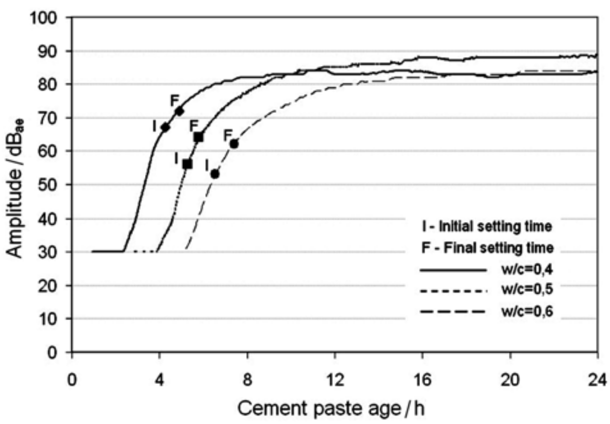

Figure 5 *Changes in the amplitude during setting and hardening*
Slika 5. *Promijene amplitude tijekom vezanja i očvršćivanja*

Changes in the Amplitude, Duration and Risetime are shown on Fig.-s 5, 6 and 7 respectively. On the figures initial and final setting times are also indicated. All ultrasonic wave parameters start to change before any change in the Vicat penetration test can be observed. W/c ratio has an influence on all three AE parameters. Higher the w/c ratio the changes of parameter start later. Linear correlation is found between the start of the increase of Amplitude, Duration and Risetime and w/c ratio of the mixes.

This can be explained by a larger distances between the cement particles in the mixture with a higher w/c ratio so

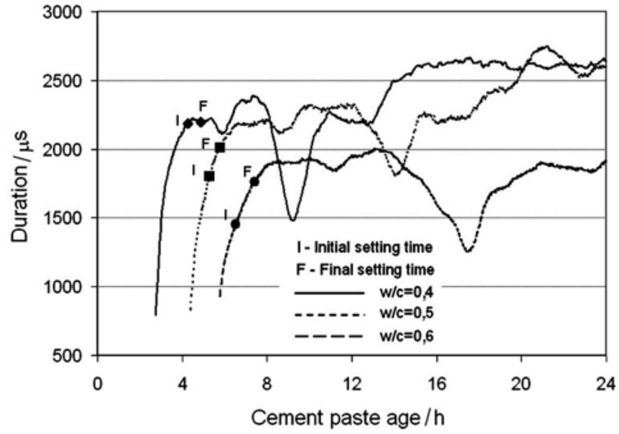

Figure 6 *Changes of the parameter Duration during setting and hardening*
Slika 6. *Promjene parametra Duration tijekom vezanja i očvršćivanja*

that particles become interconnected at a higher degree of hydration [7].

From Fig. 6 it can be noticed that a parameter Duration goes through a several maxima and minima. This could be attributed to the problematic contact between the transducer and a specimen but all mixtures seem to have similar influence on these variations. The first maximum at the graphs appears at the age of the cement paste close to the final setting time.

The Fig. 7 shows that the parameter Risetime has relatively low values for all cement pastes at the beginning of the ultrasonic signal appearance. Then a sudden increase of the Risetime appears and after that values are returned to those close to the values at the beginning. End of described initial changes appears at the age of the cement paste close to the final setting time.

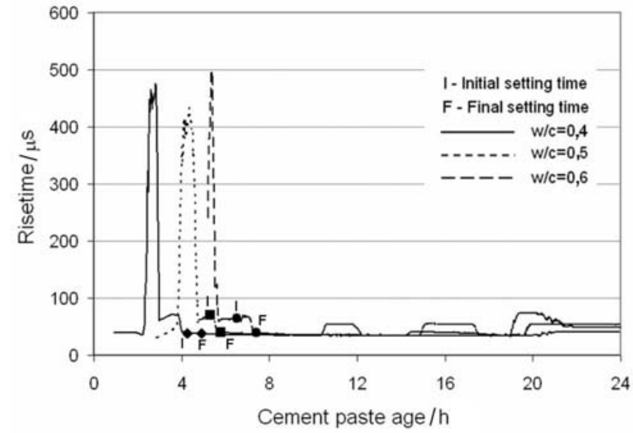

Figure 7 *Changes in the parameter Risetime during setting and hardening*
Slika 7. *Promjene parametra Risetime tijekom vezanja i očvršćivanja*

3.2
Hardening of cement paste
Očvršćivanje cementne paste

From the analysis of the ultrasonic signal two parameters were found to show a different trend of changes during the hydration of the cement paste which seemed to be related to the changes other than the transformation from liquid to solid state. Those parameters are:

- Speed of the ultrasonic wave or ultrasonic pulse velocity (UPV) which is calculated as the length of the specimen divided by the time of flight and

- Signal strength which is mathematically defined as the integral of the rectified voltage signal over the Duration of the waveform.

Changes in these parameters are shown on Fig.-s 8 and 9. On Fig. 8 changes in the UPV are shown. Speed of the ultrasound is greater in the cement paste with lower w/c ratio. The first inflection point at the graphs is around the final setting time and appears at the speed close to 1300 m/s. This is also the point after which influence of the w/c ratio on the UPV becomes evident. On the Fig. 9 changes in the relative Signal strength are shown. Relative values of Signal strength are used because absolute values are highly dependant on the quality of a contact between the transducers and the specimen considering transmission of ultrasonic wave energy [8, 9]. Higher values of the relative Signal strength are obtained for mixtures with lower w/c ratio. Changes of this parameter exhibit much lager scattering than the changes of the UPV. One reason for this could be related with the shrinkage of the cement paste which influences the amount of ultrasonic wave energy transferred into the specimen. Another reason could be that the UPV is primarily influenced by the stiffness of the cement paste (Elasticity modulus) while the changes in the relative Signal strength are a measure of attenuation of the signal which is according to Vary more closely related to the microstructure, morphology, porosity, microcracks of the material [10]. Therefore it is possible that the changes in the relative Signal strength are indeed governed by different processes than the speed of the ultrasound. Probably both influences take a part in forming its shape.

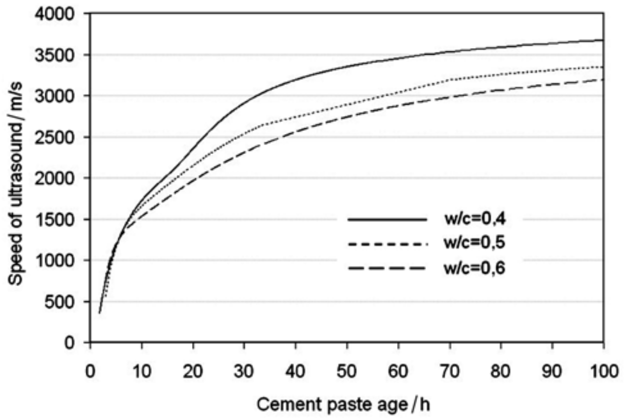

Figure 8 *Changes of the ultrasonic wave velocity*
Slika 8. *Promjene brzine ultrazvučnog vala*

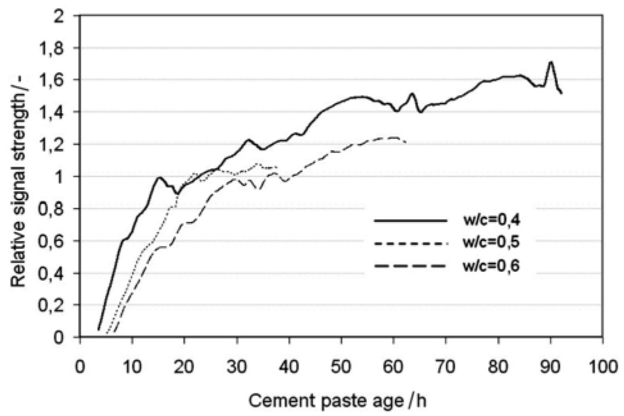

Figure 9 *Changes in the relative Signal strength of the ultrasonic signal*
Slika 9. *Promjene parametra relative Signal strength ultrazvučnog signala*

Correlation between the compressive strength and UPV is shown on Fig. 10. Although, some authors have found that for mortar and concrete the correlation between compressive strength and UPV is independent on the w/c ratio [11], from the figure it can be seen that this correlation for is dependant on w/c ration of the cement paste so a general model would have to take this into account. These differences can be attributed to the influence of the aggregate and interfacial transition zone in mortar and concrete on the compressive strength and stiffness of the cement composite [12].

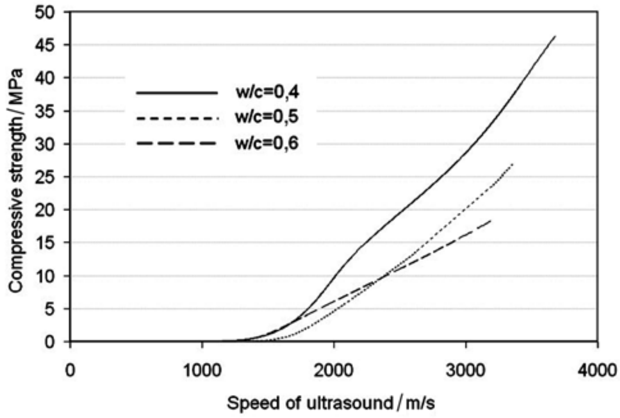

Figure 10 *Correlation between compressive strength of cement paste and UPV*
Slika 10. *Korelacija tlačne čvrstoće cementne paste i UPV*

4 Conclusion
Zaključak

Based on the obtained results it can be concluded that different ultrasonic wave parameters can be used to follow the setting and hardening of the cement paste and that not just a speed of the ultrasonic wave can be used for that purpose. Different parameters which are often used in the acoustic emission technique can help us to improve possibilities of the ultrasonic testing in this field.

It is indicated that change of cement paste from liquid to solid state could be monitored through changes in the parameters Duration and Risetime. Process of hardening is followed by changes in the velocity and changes in the attenuation of the ultrasonic signal through the parameter Signal strength. It was found that:

- the increase of the w/c ratio reduces compressive strength of the cement paste. This slows down wave velocity development and the velocity has lower values.

- the increase of the w/c ratio also slows down relative Signal strength development and the values of the relative Signal strength are lower.

While changes in the wave velocity can be explained through the changes of the stiffness of cement paste it is less clear which processes govern increase of the relative Signal strength. In order to gain more insight into the theme further work will follow.

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