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Ultrasound Study of Setting and Hardening Behaviour of Mortar Using Portland Composite Cements CPJ45 and CPJ35 and Different Dosages of An Alkali-Free Setting Accelerator for Shotcrete

Nabil Khatib, El Houssaine Ouacha, Bouazza Faiz, Mohamed Ezzaidi and Hicham Banouni
Laboratory of Metrology and Information Processing, Faculty of Science,
Ibn Zohr University, 8106 B.P., Agadir, Morocco

Abstract: Ultrasound pulse echo measurements have been successfully used to evaluate the setting and hardening behaviour of cementitious materials. In the first part of this study, the different hydration stages of Portland composite cements CPJ45 and CPJ35 have been identified by following the evolution in time (mortar age) of the frequency dependent attenuation coefficient and the modulus of the reflection coefficient as well as the setting time of both cement types has been identified. A second part of this study was to investigate the effects of an alkali-free setting accelerator for shotcrete and its dosage on setting and hardening behaviour of cementitious mixtures. Ultrasound measurements were done using P-waves, generated by a 1 MHz central frequency immersion transducer.

Key words: Ultrasound, pulse echo method, attenuation coefficient, reflection coefficient, cement-mortar, alkali-free setting accelerator

INTRODUCTION

Nowadays, concrete is the most used material for construction in the world. Its behaviour is strongly time-dependent, since, the mixture of concrete components, i.e., cement, sand grains, coarse aggregates and air bubbles with water is transformed from a liquid suspension into a porous solid material with considerable load-bearing capacity, through the hydration reactions.

Various techniques have been applied in order to evaluate the setting and hardening behaviour of cement based materials in a nondestructive way. Among these techniques, ultrasound P-wave measurements technique using backscattered signals has been greatly developed during this last two decades in order to assess the structural performance of concrete, shotcrete and cement-mortar (Bita *et al*., 2017; Lotfi *et al*., 2013). Among the measured ultrasound parameters, ultrasonic wave attenuation and the modulus of the reflection coefficient can help to predict the material's performance on the macro scale. This performance is hardly affected by the porosity volume ratio of the material. Since, the porosity volume ratio as a parameter is difficult to control in the production process, ultrasound wave attenuation and the modulus of reflection coefficient can be used to evaluate the air void content in cement based materials. In this study, cement mortar mixes were evaluated by following the evolution of the attenuation coefficient and the modulus of reflection coefficient during the setting and the hardening period, using a 1 MHz central frequency immersing transducer.

MATERIALS AND METHODS

Measured ultrasound parameters: In the paraxial approximation, the 1-D P-wave attenuates as it propagates in the cementitious material (Treiber *et al*., 2010; Aggelis *et al*., 2005).

Because of the complexity of the attenuation sources, the attenuation losses can be characterized in a simple, ad hoc fashion (Schmerr Jr., 2016; Lotfi *et al*., 2009). Therefore, to account of the losses in the cementitious material, the amplitude losses all over the distance traveled by the waves in the medium can be expressed by an exponential factor that contains a frequency dependent attenuation coefficient $\alpha(f)$. Consider a P-wave traveling in positive x-direction in a single medium. If we let A_0 be the amplitude of this wave at $x = x_0$ and A_1 the amplitude at $x = x_1$ where $x_1>x_0$, then in general $A_1<A_0$ and the amplitude losses are expressed as (Schmerr Jr., 2016):

$$\frac{A_1}{A_0} = \exp\left(-\alpha\left(f\right).d\right) \quad (1)$$

Corresponding Author: Nabil Khatib, Laboratory of Metrology and Information Processing, Faculty of Science, Ibn Zohr University, 8106 B.P., Agadir, Morocco

where, $d = x_1\text{-}x_0$ is the distance traveled in the medium. This attenuation coefficient is measured in Nepers/unit length (Np/m) when Neper is a dimensionless quantity.

Thus, the reflection technique, i.e., the pulse echo method is used to evaluate cementitious materials by using a generated pulse which is reflected at the boundary between a known material and the material to be evaluated. If a plane wave strikes at normal incidence the surface boundary (interface) between two media (M1, M2) with different acoustic impedances, part of the incident plane wave is reflected back and part is transmitted into the second medium.

Assume Z_1 is the acoustic impedance of Medium 1 (M1) with the incident wave and 2 is the acoustic impedance of the connected Medium 2 (M2) then the reflection coefficient is given by the following Eq. 2:

$$R = \frac{A_r}{A_i} = \frac{Z_2\text{-}Z_1}{Z_2+Z_1} \qquad (2)$$

Where:

A_r = The maximum magnitude of the power spectrum of the reflected wave

A_i = The maximum magnitude of the power spectrum of incident wave

Meanwhile, when the second medium is lower impedance that the first $(Z_2<Z)$, the reflection coefficient takes negative values. Instead, the reflection coefficient takes a positive sign when the second medium has a higher impedance. Thus, in order to avoid this reversal of sign behavior in our study, the measurements of reflection coefficient will be done in modulus. In the case of M1/Vacuum interface (Z_2(Vacuum) = 0), the transmission coefficient is zero. The ultrasound impedance of air medium is so much smaller that for all practical purposes, the air impedance can be neglected and the material/air interface (boundary) is treated as a material/vacuum interface instead. Therefore, the modulus of reflection coefficient on M1/Air interface is:

$$R = \frac{A_{air}}{A_i} \approx 1 \qquad (3)$$

Where:

A_{air} = The maximum magnitude of the power spectrum of the reflected wave on the M1/Air boundary

A_i = The maximum magnitude of the power spectrum of the incident wave

Thus, the reflection coefficient R expression became:

$$R = \frac{A_r}{A_{air}} \qquad (4)$$

Where:

A_r = The maximum magnitude of the power spectrum of the reflected wave at the interface M1/M2

A_{air} = The maximum magnitude of the power spectrum of the reflected wave at the interface M1/Air

Measurement procedure: The ultrasound pulse echo measurement method is used to evaluate mortar samples, filling a parralipipeted container in immersion testing. The container is excited by a broadband P-wave transducer used successively as a transmitter and as a receiver of ultrasounds. This transducer is characterized by a central frequency of 1 MHz.

The immersion transducer and the container are fixed in a steel carrier before being fully submerged in a room temperature filled with water and regulated at ambient temperature of 25°C. The container consists of one polymethacrylate (PMMA, i.e., Plexiglas) wall and a glass wall as it is shown in Fig. 1. Thus, Khatib *et al.* (2018) has already presented a detailed description of the ultrasound device developed at the laboratory of metrology and information processing at the University Ibn Zohr in Agadir, Morocco.

A part of this present research aims to follow the evolution of frequency dependent attenuation coefficient, $\alpha(f)$ as a function of mortar age. This coefficient is measured using backscattered signals from the different boundaries between the container's media. As showed in Fig. 1 and 2, the principal echoes generated by the boundaries of the container's media, those needed to evaluate the attenuation caused by the mortar sample are as follow:

The 2 corresponds to the spectral amplitude of echo E2 of the backscattered signal at the boundary between the PMMA plate and the mortar sample.

The 3 corresponds to the spectral amplitude of echo E3 of the backscattered signal at the boundary between the mortar sample and the glass plate.

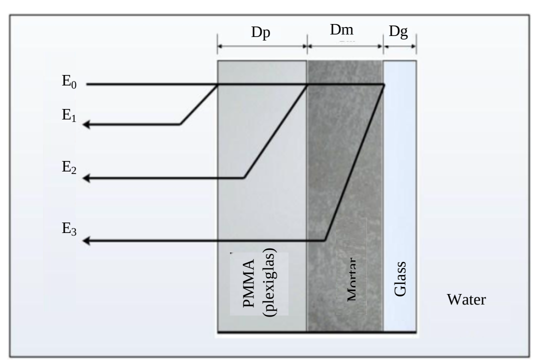

Fig. 1: Container's media and ultrasound signal's paths

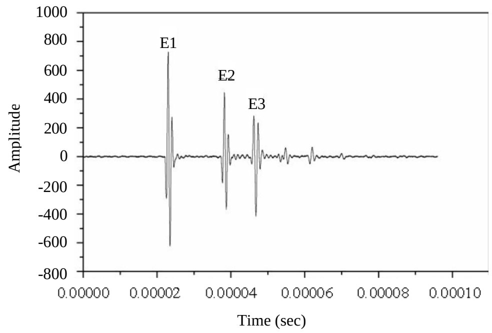

Fig. 2: Principal pulse echoes reflected at boundaries of the different container's media

The adaptation of the ad hoc fashion in our case, allows as to formulate the expression of the frequency dependent attenuation coefficient in our mortar samples, thus:

$$\alpha(f) = -\frac{1}{2L}.\ln\left(\frac{A_3}{A_2}.\xi_{ref}\right) \quad (5)$$

And:

$$\xi_{ref} = \left|\frac{(Z_{mor}-Z_{pg})(Z_{pg}+Z_{mor})(Z_{mor}+Z_{gl})}{4Z_{mor}Z_{pg}(Z_{gl}-Z_{mor})}\right| \quad (6)$$

where, Z_{mar}, Z_{pg} and Z_{gl} are the acoustic impedances of a mortar sample, the PMMA and the glass plate, respectively and L, the diameter of the space filled with the evaluated mortar sample.

Sample preparation: To allow accurate measurements, starting rapidly after the mix preparation, the experiments were performed on mortar. Mortar samples were designed by mixing standard sand extracted from the river of OUED SOUSS in the west-southern part of Morocco, cement and drinking water with a respect to a particular mix proportions, i.e., the ratios by mass of water-to-cement (w/c = 0.65) and of cement-to-sand (c/s = 0.5). The cement types tested were Portland composite cements CPJ45 and CPJ35, both produced in the cement plant of Ait Baha in the region of Agadir in Morocco, according to the standard Moroccan NM 10.1.004 (Anonymous, 2003).

CPJ45 and CPJ35 are differentiated particularly by their setting time and by their compressive strengths at 28 days which are respectively 40 and 30MPa, according to the standard Moroccan NM 10.1.005 (Anonymous, 2008). The cement type CPJ35 is characterized by a large setting time, allowing a better workability while the setting time of CPJ45 is shorter. CPJ35 is mostly used in masonry work as well as in preparation of unreinforced or poorly reinforced concretes. Moreover, CPJ45 is principally used for reinforced concretes, shotcrete and in special constructions (dams, thermal power stations, etc.).

In addition, mortar mixes containing different dosages of an admixture were evaluated. The tested admixture was an alkali-free setting accelerator for shotcrete CHRYSO XEL 650. The accelerator dosage amounted to 0, 1.7 and 6.8% of the cement weight used in tested mortars. The preparation process is as follow:

- Firstly, cement and dry sand are mixed for 60 sec
- After that, water is added followed by 60 sec of mixing
- Afterwards, the dosed accelerator was added and the mixing process continue for a 10 sec mixing

Then, the prepared mortar mix was filled and compacted in the container for about 30-60 sec on a vibrating table. The vibration time was limited in order not to hamper the binding process. For the preparation of mortar samples containing alkali-free accelerating admixture, we used the Portland cement type CPJ45, since, it is the one used in the construction field for the preparation of shotcrete.

RESULTS AND DISCUSSION

The measured wave attenuation coefficient for two different mortar pastes specimens are shown in Fig. 3. The whole experiment was finished after 72 h.

Before describing the attenuation coefficient evolution, we must take note that in samples with Portland cement, setting is normally completed within 6-8 h.

In Fig. 3, the evolution of wave attenuation coefficient indicates four principal stages, characterizing mortar's behaviour. The first stage is the period during which cement, water and sand are barely mixed. This stage is also characterized by an important and rapid release of heat generated by superficial dissolution of cement paste components. The second stage is the dormant period of cement hydration, causes cement to remain plastic and during which the hydration reactions occur slowly. During this period, wave attenuation coefficient began to increase. Then, the third period is the setting period in which the cement compounds hydrate rapidly. In this period, mortar samples were characterized by a very steep increase in wave attenuation coefficient after which a maximum was reached. Reaching this maximum indicates that the final set has passed and early hardening begins. Next, the fourth stage is the hardening period when the rate of hydration reactions slows. During this period, the

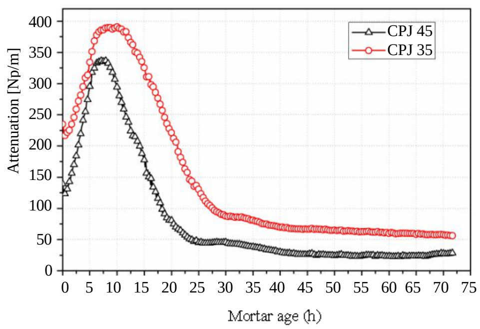

Fig. 3: Evolution of the attenuation coefficient for the two cement types, i.e., CPJ45 and CPJ35 vs. mortar age

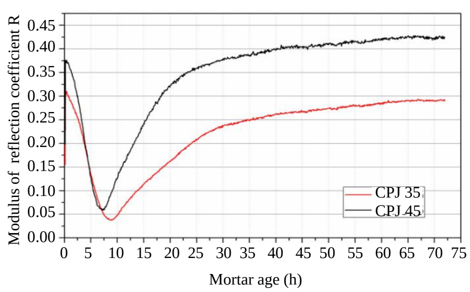

Fig. 4: Evolution of the modulus of reflection coefficient for the two cement types, i.e., CPJ45 and CPJ35 vs. mortar age

wave attenuation coefficient decreases sharply after which attenuation coefficient curves levelled off indicating a steady state. This could be partly explained by the interconnection between particles of the hardened mortar paste.

Looking at the modulus of the reflection coefficient curves in Fig. 4, the different hydration stages could also be identified as based on the attenuation coefficient. Thus, the dormant period corresponds to the stage when the modulus of reflection coefficient began to decrease. During the setting period, the reflection parameter decreases sharply after which a minimum was reached. Reaching this minimum indicates the end of the setting period and the beginning of the hardening stage. After that comes the hardening period when the modulus of the reflection coefficient increases sharply after which its curves levelled off indicating also a steady state.

Therefore, we notice that the maximum for the attenuation coefficient and the minimum for the reflection coefficient were reached for CPJ45 at an earlier time compared to CPJ 35. This result validates the time lag between the end times of setting for CPJ 45 and CPJ 35.

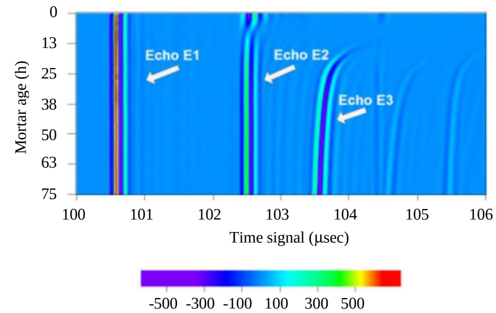

Fig. 5: Histogram of ultrasound backscattered signals data, during 72 h of mortar age forPortland composite cement CPJ45

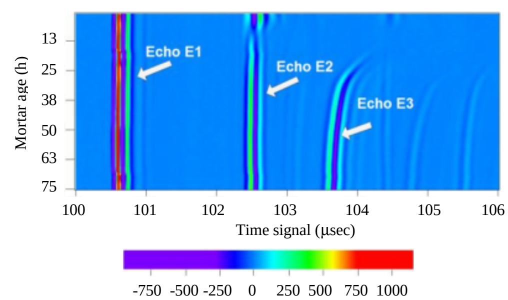

Fig. 6: Histogram of ultrasound backscattered signals data, during 72 h of mortar age forPortland composite cement CPJ35

The evolution of wave attenuation coefficient and the modulus of reflection coefficient allowed us to determine the setting time of both cement types CPJ45 and CPJ35. The setting time, i.e., the thickening time is the period when cement slurry remains in fluid state and is capable of being pumped. As a result, the two ultrasound parameters curves showed that the setting period of CPJ35 takes about 8.75 h when the setting time of CPJ45 takes only about 7 h.

Figure 5 and 6 show the superposition of the received signals, backscattered by the container's media. An analyze of the two figures indicates that Echo E3 began to occur at earlier time for the cement type CPJ45 in comparison to that for cement type CPJ35. Thus, the early appearance of Echo E3 for CPJ45 is mainly related to its early end time of setting, compared with the end time of setting of the CPJ35.

In addition, the effects of an alkali-free accelerating admixture (labeled as AFA) and its dosage on mortar behaviour at early ages has been evaluated by following

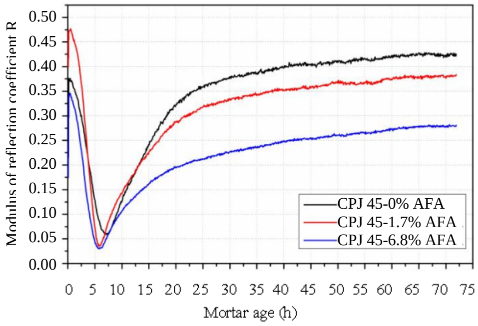

Fig. 7: Modulus of reflection coefficient vs. mortar age. Mortars were prepared using Portland composite cement CPJ45 and an alkali-free accelerator dosage of 0, 1.7 and 6.8% of the cement weight used in the mortar mix

the evolution of the modulus of reflection coefficient as it is shown in Fig. 7. The accelerator's dosage amounted to 0, 1.7 and 6.8% of the cement weight used in the mortar mix.

The modulus of reflection coefficient curves for reference cement mortar using CPJ45 and accelerated mortar mixes all showed the same pattern and the hydration stages were clearly identified. Thus, it is clear that the ultrasound reflection parameter is sensitive to the effects of the alkali-free accelerating admixture and its dosage on setting and hardening behaviour of mortar samples. A stepwise increase in the accelerator dosage resulted in more smoothly evolvement of the modulus of reflection coefficient curve, at a time interval between the end of the setting and the beginning of the steady state for the measured ultrasound parameter.

Also, the stepwise increase in alkali-free accelerator dosage resulted in decreasing values at which the modulus of the reflection coefficient curves levelled off indicating a more attenuative medium. This increase in the attenuation of the mortar medium could mainly be caused by an increase in the void volume of the hardened mortar paste.

Using the reflection parameter, the setting time of the different accelerated mortar mixes was determined. A mortar mix containing an alkali-free accelerator dosage of 1.7% was characterized by a setting time of about 5.5 h when the reference mortar mix prepared using Portland cement CPJ45 remains in the setting stage until 7 h of mortar age. This result validates the principal role of this alkali-free set accelerator that contains chemicals that influence the rate of cement hydration, thereby shortening the setting time by contributing to the rapid formation of Ettringite. However, no decrease has been noticed in the setting time when increasing the alkali-free accelerator dosage from 1.7- 6.8% in spite of the change in the evolution of the attenuation behaviour. Yet, the use of a dosage of 6.8% caused a severe attenuation in the accelerated mortar medium, compared to reference mortar and mortar containing an alkali-free accelerator dosage of 1.7%.

Therefore, those results has been employed to formulate a recommendation for the interest of the construction and public works field which consider using the alkali-free accelerating admixture by a dosage not exceeding 1.7% of the cement weight used in the shotcrete mix, to avoid a significant expansion of the void volume, consequently causing serious decrease of the resistivity and the strength of the hardened cement paste as previously discussed by Khatib *et al*. (2018) and De Belie *et al*. (2005).

CONCLUSION

Ultrasound wave attenuation coefficient and the modulus of reflection coefficient as fundamental acoustic parameters of a material can be used to evaluate mortar's behavior at early ages and therefore, help predict the cementitious material's performance on the macro scale. In this research, the following of those two ultrasound parameters evolution in the evaluated mortar pastes, during 72 h of mortar age, allowed us to identify the four major stages of Portland composite cement hydration. The first stage is the rapid heat generation period, followed by the dormant period, then the setting period and after that comes the hardening period. The time when attenuation coefficient reaches his maximum indicates the end of the setting period which also refer to the time when the modulus of the reflection coefficient reaches its minimum. The measurements of wave attenuation coefficient and the modulus of the reflection coefficient show that the setting time of cement type CPJ45 is shorter, compared to the cement type CPJ35. Thus, those results have been validated when following the evolution of the different echoes in time (mortar age) using ultrasound histograms.

In addition, the effects of an alkali-free accelerating admixture for shotcrete and its dosage on setting and hardening behaviour of mortar have been evaluated using measurements of the modulus of the reflection coefficient. Thus, an increase in the alkali-free accelerator dosage resulted in a more attenuative mortar medium during the hardening period. This could be partly explained by an expansion of the void volume of the hardened cement paste. Also, no change in setting time has been notice when increasing the alkali-free accelerator dosage from 1.7- 6.8%.

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