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Effects of Temperature and Clay on Hydration Behavior of Mortar: A Study Through Ultrasound Technique

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ABSTRACT

This paper aims to characterize the effects of natural clay additions in mortar mixes in different concentrations and under different curing temperatures on setting and hardening of cement mortar. For this purpose, characterization technique based on reflection of ultrasounds was used. Using this technique, the attenuation coefficient and modulus of reflection coefficient are measured to characterize the early age hydration behavior of tested cement-mortars. Ultrasound P-waves were generated using a 1 MHz central frequency immersion transducer and mortar samples were prepared with Portland cement. The results show that increasing the curing temperature accelerates the setting of the mortar. In addition, the incorporation of natural clay to the mortar mix in different concentrations results in retardations in the end of setting time of mortar. Also, the results confirmed that the hardening behavior of the mortar subjected to high curing temperature and mixed with the different natural clay additions is not linear.

1. INTRODUCTION

In the Souss and Massa region, situated in the west-southern part of Morocco, the use of Portland cement in the preparation of cement-based materials requires a huge exploitation of natural freshwater resources, currently very rare confronted with the increase of its demand generated by the demographic and socio-economic development of the Souss and Massa region. The Souss and Massa basin is characterized by an arid climate, a dense population, limited water resources [1-3] and a significant withdrawal of groundwater [4]. The Souss and Massa basin receives about 250 mm of rainfall per year. Total water withdrawals in the basin are estimated at 650 million cubic meters per year. The rate of groundwater extraction in the basin exceeds the recharge of 260 million cubic meters per year, resulting in a decline in water level of 0.5 to 2.5 m/year over the past three decades [1, 5]. These data clearly show the need to optimize the consumption of fresh water in concrete and mortar construction works.

The raw natural sand extracted from the rivers of the Souss and Massa region contains impurities that can compromise the quality of cement mortar, including quantities of clays. Thus, this raw natural sand requires fresh water in great quantity to wash it, in order to reach a degree of cleanliness before its use. The use of raw natural sand directly in the preparation of Portland mortar mixtures without passing through the sand washing process can be one of the alternatives to optimize the consumption of fresh water. Even if this is possible, it is important to make sure that the presence of clays in the mortar mix will not affect the hydration properties of the resulting cementitious material.

Along with the study of natural clay effects, temperature variation in the region of Souss Massa due to recent climate change is also an environmental issue that needs to be considered. In this context, several research works involving regional studies [4-6] have shown significant variations and precipitation of temperatures in Southwestern Morocco, during the last two decades. Temperature is one of the main physical quantities whose variations have a fundamental influence on the physicochemical properties of cement-based materials. Therefore, it is important to study the influence of temperature on the early age hydration behavior of cement mortar, while evaluating the influence of the natural clay incorporated in the mortar mixes in different concentrations. Recent studies and seasonal meteorological data [4-6] were considered in order to select three different curing temperatures, i.e., 25°C, 35°C and 45°C, as testing temperatures.

In this study, an ultrasound non-destructive method is used to characterize the setting and early hardening process of mortars. The ultrasound method used in through reflection, which consists on generation and reception of P-waves by an immersion transducer of 1MHz central frequency. Methods based on reflection of ultrasounds are better suited for the control of cement-based materials behavior during setting and early hardening periods, because the ultrasound measured parameters are closely correlated with the early age hydration properties of concrete and cement-mortar.

Using this method, the setting and early hardening periods of cement mortar containing different concentrations of natural clay extracted near the river of Oued Souss and cured under three hydration temperatures (25℃, 35℃ and 45℃) that were regulated in a thermostatic chamber where the experiments were conducted.

2. METHODS AND MATERIALS

2.1 Pulse echo method

An ultrasound method was developed in our laboratory in order to solve different environmental issues, especially those related to construction using cement-based materials. This method consists of different steps starting with propagation of ultrasound waves into the sample, followed by the acquisition of reflected ultrasound signals composed of different generated echoes to a further signal processing and measurements of ultrasound parameters using developed Labview software. The experimental setup was described in details in Khatib et al. [7, 8], and consists of a hardware section and a software section.

Thus, the immersion transducer with 1MHz central frequency is used to generate ultrasound waves that propagates in the container containing the mortar sample to be characterized. Both the transducer and the container are submerged in water as a constant coupling medium [9].

2.2 Measurement parameters

The ultrasound measurement method uses a sealed parallelepiped container, composed of one polymethacrylate wall and a glass wall as is shown in Figure 1.

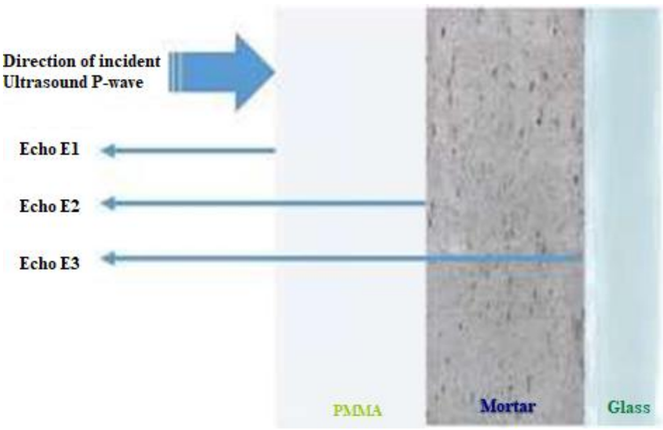


Figure 1. Container media and pulse echoes generated from boundaries

Polymethacrylate (PMMA) is selected due to its excellent acoustic transmission properties, allowing a significant portion of the P-wave to pass through to the mortar sample. Furthermore, to observe the setting and hardening behavior of the cement mortars under evaluation, transparent glass is utilized as a reflective medium. This means that the front surface of the glass plate will reflect a substantial portion of the ultrasound wave directed at the mortar.

The developed ultrasound method acquired ultrasound reflected signals, every 5 minutes, during 72 hours for ultrasound experiment. The acquired signals are composed of different echoes, generated by reflections at the different boundaries between the container's media. The principal echoes needed for further analysis are Echo E2, generated at the boundary between PMMA plate and the mortar sample, and also, Echo E3 generated at the boundary between the mortar sample and the glass plate. Continually, the maximum of spectral amplitudes of both Echoes E2 and E3, noted respectively A2 and A3, will be calculated using developed Labview software. So, the values of both maximum amplitudes are used to calculate and then to follow the evolution in time domain (hydration age of mortar) of the modulus of reflection coefficient R associated to Echo E2 and the frequency dependent attenuation coefficient in mortar medium, $\alpha(f)$.

In this context, the expression for the modulus of the reflection coefficient corresponding to the reflection producing Echo E2 is denoted as:

$$R = \frac{A_2}{A_2^{ref}} \tag{1}$$

where, A_2^{ref} represents the maximum spectral amplitude of the echo from the backscattered signal at the interface between the PMMA plate and the air medium [9]. The frequencies of both maxima correspond to the transducer's central frequency of 1 MHz.

The 1-D P-wave attenuates as it propagates in the cementitious material [10, 11]. Using ad hoc fashion [9], the attenuation losses can be characterized in a simple. Thus, the adaptation of the ad hoc fashion in our case, allows us to formulate the expression of the frequency dependent attenuation coefficient in our mortar samples, thus:

$$\alpha(f) = -\frac{1}{2L} \cdot \ln\left(\frac{A_3}{A_2} \cdot \xi\right) \tag{2}$$

$$\xi = \left|\frac{(Z_{mor} - Z_{pg}) \cdot (Z_{pg} + Z_{mor}) \cdot (Z_{mor} + Z_{gl})}{4 \cdot Z_{mor} \cdot Z_{pg} \cdot (Z_{gl} - Z_{mor})}\right| \tag{3}$$

where, Z_{mor}, Z_{pg} and Z_{gl} are the acoustic impedances of a mortar sample, the PMMA and the glass plate, respectively and L, the width of the evaluated mortar sample.

2.3 Mortar samples preparation

The aim of this present study is to investigate the effects of natural clay presence in different concentrations and under different curing temperatures, on the early age hydration behavior of mortar. To do so, twelve samples of mortar were prepared using natural sand extracted from the river of Oued Souss and Portland cement with respect to a particular mix mass proportion, i.e., a water-cement ratio (w/c) of 0.6 and a cement-sand ratio (c/s) of 0.4. Under each of the tested curing temperatures, i.e., 25℃, 35℃ and 45℃, three clay concentrations were tested, namely the clay-water ratios of 1:4, 1:8 and 1:16.

2.4 Identification of early age hydration stages

The modulus of reflection coefficients and attenuation coefficients are plotted against time (age of mortar), to characterize early age hydration of Portland cement mortars. Typical curves of reference mortar sample prepared without any natural clay addition, cured at an ambient temperature of 25°C, (named later CM–T25), are shown in Figures 2 and 3.

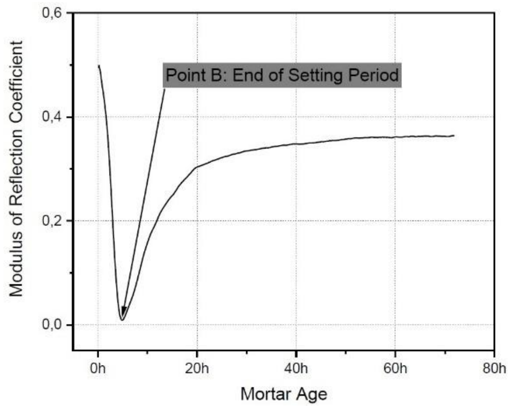


Figure 2. Position of characteristic point B on the time evolution curve of modulus of reflection coefficient

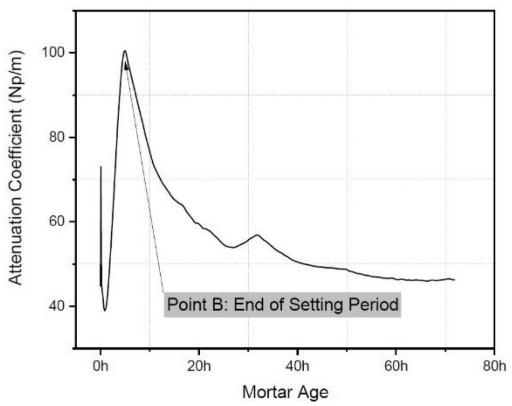


Figure 3. Position of characteristic point B on the time evolution curve of attenuation coefficient

In our previous works [7, 8, 12, 13], we studied the correlation between measured ultrasound parameters and the hydration properties of cement mortar using the pulse echo method. This ultrasound technique was used to identify the time of the end of setting time and the early hardening behavior of cement mortars. To do so, two characteristic points B and B' are used to facilitate this identification, respectively, on the modulus of reflection coefficient and the attenuation coefficient evolution curves.

In order to show this correlation, we will first have to expose the main points of the mortar hydration process. Thus, the hydration of cement mortar is principally a four-stage process. Chemical explanation of this hydration process can be summarized as follow:

1. **Stage 1 – The pre-induction period:** At the contact with water, the initial rapid dissolution of C_3S (alite), C_2S (bélite) and C_3A, causing the precipitation of respectively a "C-S-H of first stage" layer and Ettringite Aft, in the vicinity of unreacted surface. As a result, the rate of hydration slows down considerably.
2. **Stage 2 – The dormant period:** During this stage, the hydration reactions progress slightly. This drop in hydration rate is due to the formation of "first stage C-S-H" and Ettringite barrier layers, which limit the migration of water to the non-hydrated minerals.
3. **Stage 3 – The setting period:** The "first stage C-S-H" layer become more permeable, which allows a rapid re-hydration. Thereby, the "second stage C-S-H" and Portlandite precipitate, and the Ettringite continues to form. At this level, the mortar pastes losses workability progressively, which means more densification of the cement paste and the interfacial transition zone (ITZ). At the end of this stage, the mortar paste is no more workable and the final set is passed, named as the end of setting period.
4. **Stage 4 – The hardening period:** During this period, the mortar paste has already lost completely its workability and the gradual increase of interconnexion between hardened mortar paste particles make the mortar becomes more mature.

The ultrasound analysis results [7, 8] showed that the evolution of the modulus of reflection coefficient is contrary of that of attenuation coefficient. The minimum reached by the modulus of reflection coefficient, located by characteristic point B, corresponds to the maximum reached by attenuation coefficient, marked by characteristic point B', as shown in Figures 2 and 3. Thus, both points B and B' indicates time of the end of setting period. Continually, the stable (steady) state reached by both measured ultrasound parameters indicates that the mortar paste reached a degree of maturity during the hardening period.

3. RESULTS AND DISCUSSION

3.1 Effect of curing temperature

The ultrasound measurements of modulus of reflection coefficient and attenuation coefficient on cement-mortars cured at various hydration temperatures, i.e., 25°C, 35°C and 45°C, are shown in Figures 4 and 5.

Table 1 shows the time of characteristic points B and B' for samples cured at the different temperatures tested in this current study. In Table 1, the different mortar mixes were coded as follows:

- CM indicates Cement Mortar;
- In the curing temperature variations, T followed by a number indicating the curing hydration temperature.

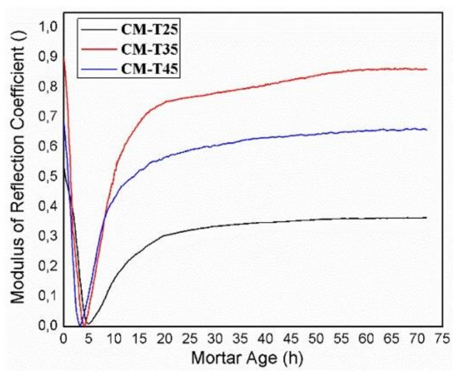


Figure 4. Influence of different curing temperatures (25°C, 35°C and 45°C) on modulus of reflection coefficient evolution during 72 h of mortar age

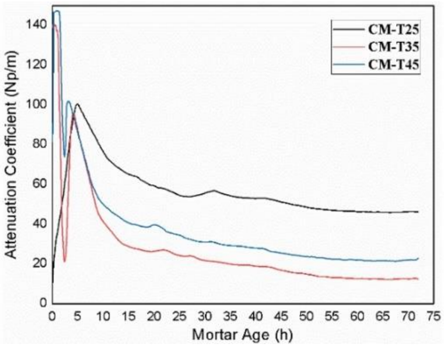


Figure 5. Influence of different curing temperatures (25°C, 35°C and 45°C) on attenuation coefficient evolution during 72 h of mortar age

Table 1. Characteristic time points B and B' for the mortar samples cured at different temperatures, namely 25°C, 35°C and 45°C

Designation	B	B’
CM-T25	4h55min	4h55min
CM-T35	4h05min	4h05min
CM-T45	3h05min	3h05min

The modulus of reflection coefficient and attenuation coefficient curves for ultrasound experiments on mortar samples cured at various hydration temperatures show the same pattern. Time appearance of characteristic points B and B’, indicating the end of the setting period are displayed in Table 1.

Values in Table 1 reveal clearly that the appearance time of characteristic point B' on the attenuation coefficient curves coincides with those of the characteristic point B, on the reflection coefficient curves. Thus, both characteristic points B and B' indicate the end of setting period.

A stepwise increase in hydration temperature results in a stepwise decrease in time of setting period of the tested mortars. When a mortar paste tested at an ambient temperature of 25°C has an end of setting period at 4h 55min, those of mortars cured at an elevated temperature of 35°C and 45°C, are respectively at 4h 05min and 3h 05min. The results show that increasing the curing temperature accelerates the setting of the mortar. Micro-structurally, the changes in cement-based materials cured at elevated temperatures are mainly due to the loss of free water and part of the absorbed water [14, 15]. Thus, at elevated hydrations temperatures, the bound water of several hydrates is released starting with Ettringite, then C-S-H and other hydrates [16]. The kinetics of hydrate formation changes with increasing temperature. According to the study [17, 18], the rate of hydration of C_3S is temperature dependent and increases with increasing temperature. Cagnon et al. [19] also stated that considering C-S-H as the main cause of time-delayed deformation, a significant change in hydration behavior occurs in the unconsolidated cementitious material as the temperature increases. In view of the above, with a progressive increase in mortar temperature, there is a significant, also progressive, loss of workability.

After going through the characteristic point C, the modulus of the reflection coefficient increases in a decelerated way until it reaches a plateau, i.e. a stable level indicating that the tested mortar has reached its maturity phase. The modulus of the reflection coefficient measurements showed that an increase in hydration temperature from 25°C to 35°C results in an increase of the final stable values, during the initial hardening period. However, an increase in hydration temperature from 35°C to 45°C leads to a decrease in the stable final values, which in any case remain higher than those of a mortar tested at 25°C. The variation of the final stable values from one test curing temperature to another depends on the pore structure of the tested mortar sample and their distribution, the interconnection between the mortar particles and the opening of the micro-cracks.

Mortar exposed to high temperatures undergoes changes in microstructure of crystalline phases, cracking and, consequently, in its pore structure [20, 21]. Alves et al. [22] demonstrated that the effects of increasing hydration temperature on the microstructure of mortar can be seen through alterations in porosity and peaks in mass loss associated to the dehydration and deterioration process of the mortar hydration products. Regarding the correlation between measured ultrasound parameters and early age hydration properties of mortar [7, 8, 12, 13], higher stable values of reflection coefficient prove higher reinforcement of boundary connections between PMMA and mortar, which facilitate transmission of ultrasound wave to mortar sample. In the study [22], samples showed that the pore size distribution is altered with temperature increase, however, it is not confirmed that these pores increase with each curing temperature increase. This result was validated since the gradual increase in curing temperature does not automatically lead to an increase in the final stable values of the modulus of the reflection coefficient during the early hardening period. In parallel, the stepwise increase in curing temperature does not lead directly to a decrease in the final stable values of the attenuation coefficient during the early hardening period, as shown in Table 2. Thus, our results confirmed that the hardening behavior of the mortar subjected to high curing temperature is not linear.

Table 2. Final stable values of the attenuation coefficient for mortar samples cured under hydration temperatures of 25°C, 35°C, 45°C and corresponding pictures after 72 hours of early hydration

Designation	Final Stable Values of Attenuation Coefficient (Np/m)	Picture of Hardened Mortar Sample
CM-T25-C0 (CM-T25)	46.34	

Designation	Final Stable Values of Attenuation Coefficient (Np/m)	Picture of Hardened Mortar Sample
CM-T25-C1:16	60.26	
CM-T25-C1:8	19.86	
CM-T25-C1:4	17.86	
CM-T35-C0 (CM-T35)	12.56	
CM-T35-C1:16	30.42	
CM-T35-C1:8	22.05	

Designation	Final Stable Values of Attenuation Coefficient (Np/m)	Picture of Hardened Mortar Sample
CM-T35-C1:8	37.87	
CM-T45-C0 (CM-T45)	22.86	
CM-T45-C1:16	22.39	
CM-T45-C1:8	38.03	
CM-T45-C1:4	38.10	

3.2 Effect of natural clay presence

The measurements of the modulus of the reflection coefficient and the attenuation coefficient, for mortar mixes subjected to different curing temperatures of 25℃, 35℃ and 45℃ and incorporating different concentrations of natural clay are shown in Figures 6-8. All the evolution curves of the modulus of the reflection coefficient have the same shape. In Table 3, the different mortar mixes were coded as follows:

- CM indicates Cement Mortar;
  In the curing temperature variations, T followed by a number indicating the curing hydration temperature;
- C: Natural Clay;
  Number representing the natural clay dosage in mixing water in clay-water mass ratio, i.e., 0, 1:16, 1:8 and 1:4 (mortar samples without any clay additions are designed by C0 which correspond to the mortar samples used to evaluate the curing temperatures effects in the first part of this work).

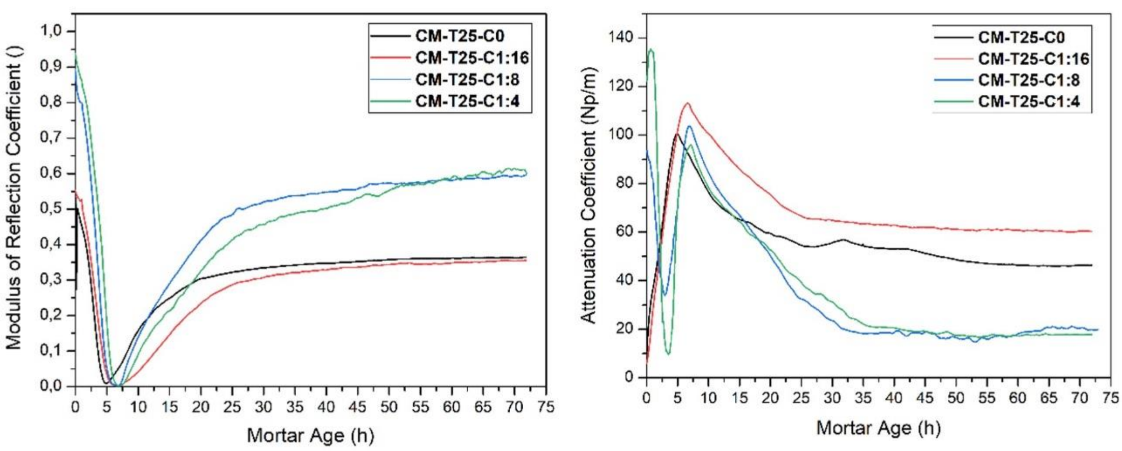
As shown in Figures 6-8 and Table 3, for each of the curing temperatures tested, a stepwise increase in the concentration of natural clay resulted in a stepwise increase in the time values of the characteristic point B. As described earlier [7], point B indicates the end of the setting period, and thus, the addition of natural clay to the mortar mix results in retardations in the end of setting time of mortar. The retarding effects resulting from natural clay additions are more pronounced at early ages on mortar subjected to a curing temperature of 25℃. For mortar samples tested at 25℃, when the reference mortar sample designated CM-T25-C0, prepared without any clay addition, has a setting time of 4h 55min. This setting time increases to 7h10min for the mortar sample CM-T25-C1:4 that contains a proportion of clay corresponding to a clay/water mass ratio of 1:4. The time interval (the time difference – time delay) between the end of setting time of the reference mortar CM-T25-C0 and that of the mortar-clay sample CM-T25-C1:4 is about 2h 15min. As indicated in Table 3, the time interval between the end of setting time of the reference mortar and that of a mortar with a clay-to-water mass ratio of 1:4, decreases with increasing curing temperature, reaching 1h 25 min at a temperature of 35℃ and 50 min at a temperature of 45℃. These results show that increasing curing temperature reduces the retarding effect of natural clay on the end of setting time of tested mortars. Meanwhile, when the concentration of natural clay in mortar is fixed, increasing curing temperature still has the same effect of accelerating the end of setting time of the tested mortars, as in the case of the reference mortar without any natural clay addition.

Upon contact with water, ionic species are rapidly dissolved in the liquid phase and the formation of hydrated phases begins to occur. Initially, the dissolution rate of C_3S and C_2S is faster than diffusion can carry the dissolved ions away from the surface, causing a concentration gradient near the surface of the unhydrated calcium silicates. Continuously, the liquid phase rapidly becomes oversaturated near the hydrated calcium silicate and a layer of a "first stage" C-S-H product begins to precipitate on the surface of the unhydrated calcium silicates. This C-S-H layer acts as a barrier that significantly slows the migration of water to the unreacted calcium silicates during the dormant period [17]. With the addition of natural clay, more silicate from active clay particles is mixed into the cement paste, which automatically reinforces the "first phase" C-S-H layer and consequently delays the precipitation of the Portlandite and the "second phase" C-S-H. This may be the main explanation for the retardation of the end of setting time for the clay-containing mortar, as seen in our results on the different times for characteristic point B, shown in Table 3. The fine clay particles act as nucleation agents for the formation of C-S-H hydration products [23]. Thus, in the presence of lime, the amorphous silica from the clay promotes the formation of C-S-H [24].

According to Zambon [25], the analyzed clay is mainly composed of kaolinite, illite and calcite as combined clay minerals. The addition of kaolinite results in a C-S-H phase that has longer SiO_4 tetrahedra chains compared to the C-S-H formed in a cementitious medium without the addition of clay [23]. When clay is added, the clay silicates, initially in suspension, form flocs through the process of cationic flocculation with the Ca_2+ cations present in the Portland cement. As a result, the clay particles become larger, in the form of aggregates that lose their colloidal state and thus form sediment (sedimentation phenomenon). The higher specific surface of clay and its affinity with water influence the mechanical behavior of the mortar. The clay acts as a filler and forms a compact structure, while maintaining the distribution of particles in the sample [26]. Thus, clay in cement mortar increases the density and enhances its strength [26]. However, as shown in Table 2, our results confirmed that the early hardening behavior of the mortars with different concentrations of incorporated natural clay, and under the tested curing temperatures, is not linear.

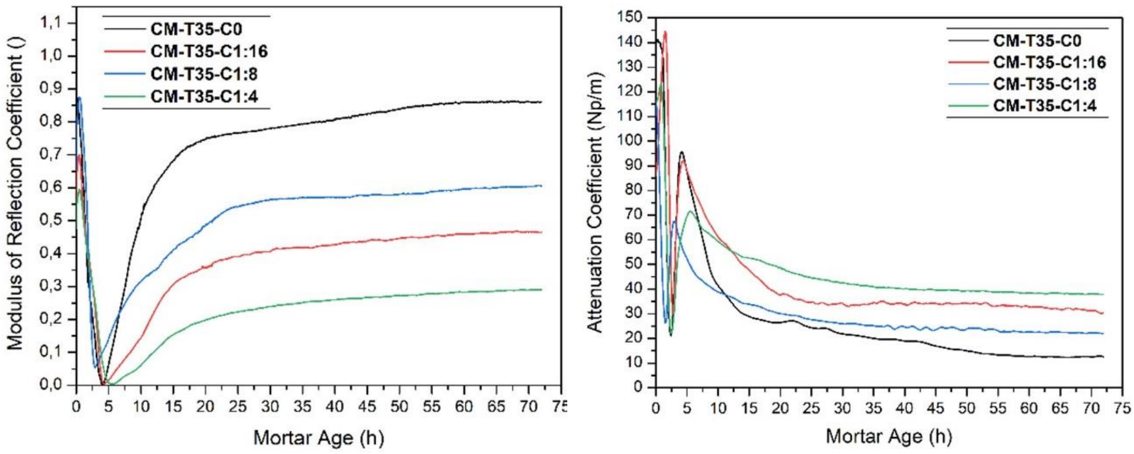
Table 3. Characteristic time point B for mortars with different clay-water ratios and cured under different hydration temperatures

Designation	**Curing Temperature**		
Values clay-water ratios	25℃	35℃	45℃
0	**CM-T25-C0** 4h55min	**CM-T35-C0** 4h05min	**CM-T45-C0** 3h05min
1:16	**CM-T25-C1:16** 6h40min	**CM-T35-C1:16** 4h30min	**CM-T45-C1:16** 3h15min
1:8	**CM-T25-C1:8** 6h55min	**CM-T35-C1:8** 5h05min	**CM-T45-C1:8** 3h30min
1:4	**CM-T25-C1:4** 7h10min	**CM-T35-C1:4** 5h30min	**CM-T45-C1:4** 3h55min



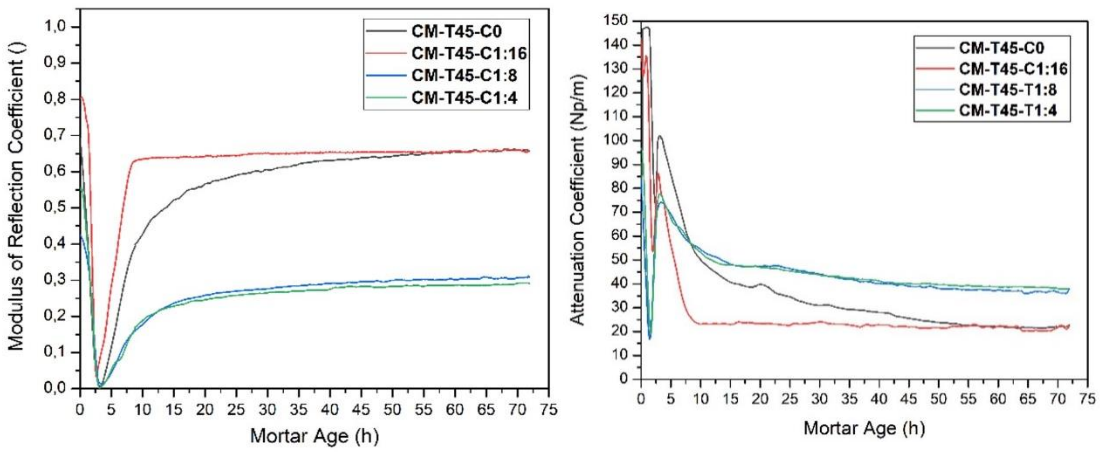
(a) (b)

Figure 6. Influence of different tested clay dosages on ultrasound parameters evolution during 72h of mortar age, cured at a temperature of 25℃: (a) Evolution of Modulus of reflection coefficient; (b) Evolution of Attenuation Coefficient



(a) (b)

Figure 7. Influence of different tested clay dosages on ultrasound parameters evolution during 72h of mortar age, cured at a temperature of 35°C: (a) Evolution of modulus of reflection coefficient; (b) Evolution of attenuation coefficient



(a) (b)

Figure 8. Influence of different tested clay dosages on ultrasound parameters evolution during 72h of mortar age, cured at a temperature of 45°C: (a) Evolution of modulus of reflection coefficient; (b) Evolution of attenuation coefficient

4. CONCLUSIONS

A developed non-destructive measurement method based on ultrasound pulse backscattering is employed to characterize the setting and hardening early age hydration behavior of cement mortar. Using this method, the attenuation coefficient and the modulus of reflection coefficient are measured and recorded for analysis. In this context, the effects of different natural clay additions in mixed mortar and various curing temperatures were characterized. Several conclusions can be drawn from the experimental investigations:

(1) The time scale evolutions of modulus of reflection coefficient and the attenuation coefficient are closely correlated with the early age hydration properties of Portland cement – mortar.

(2) Four principal stages of cement – mortar hydration can be identified by following the time scale evolution of the modulus of reflection coefficient and attenuation coefficient.

(3) The stepwise increase in hydration temperature resulted in a stepwise decrease in time of setting period, i.e., a progressive and significant loss of workability.

(4) However, results confirmed that the early hardening behavior of cement-mortar subjected to high curing temperatures is not linear.

(5) The stepwise increase of incorporated natural clay concentration resulted in retardations in the end of setting time of mortars. Thus, the results showed that increasing curing temperature reduces the retarding effect of natural clay additions on the end of setting time of cement – mortars tested.

(6) Meanwhile, when the concentration of natural clay in mortar is fixed, increasing curing temperature still has the same effect of accelerating the end of setting time of the tested mortars, as in the case of the reference mortar without any natural clay addition.

(7) The early hardening behavior of the mortars with different concentrations of incorporated natural clay, and under the tested curing temperatures, is not linear.

Based on these findings, construction professionals should consider increasing curing temperatures to mitigate the retarding effects of natural clay on cement mortar setting times.

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