Monitoring Acoustic Emission of Fresh Cement Paste

Evin Dildar Dzaye[1,2] , Geert De Schutter[2] and Dimitrios Aggelis[1]

[1]Vrije Universiteit Brussel, Brussel, Belgium
[2]Ghent University, Magnel Laboratory for Concrete Research, Ghent, Belgium

ABSTRACT

High strength and durability are the key factors determining the quality of concrete. To achieve the required properties, concrete mix design, and early age hydration, are the main factors affecting the performance of concrete. It is essential to monitor fresh concrete continuously through non-destructive techniques at the moment of mixing. In this study, acoustic emission (AE) has been applied to allow a continuous monitoring of the fresh cement paste. This non-destructive inspection allows the estimation of concrete properties by capturing elastic waves that are nucleated and propagate in the cement past. Moreover, ultrasonic pulse velocity (UPV), capillary pressure and heat evolution monitoring has been applied on cement paste to study the process of hydration mechanism. This study aims to check the sensitivity and effectiveness of AE technique to characterize the ongoing processes in fresh cementitious material and the possibility to contribute to a better monitoring of the process as an additional tool.

Keywords: Acoustic emission, ultrasound wave, capillary pressure, isothermal calorimetry, cementitious material

1 INTRODUCTION

Acoustic emission (AE) is a very sensitive technique to study the fresh concrete properties. This passive technique requires piezoelectric sensors to record the elastic waves in fresh concrete in order to transform them into electric waveforms. The AE intensity as well as their parameters are able to expose information on crack nucleation in different materials and has been applied during hardening process of concrete [1]. The purpose of this non-destructive evaluation (NDE) is to supplement hydration process studies to gain better understanding into the mechanisms appearing in the fresh cementitious material, since different processes in fresh cementitious material like segregation, bleeding, bubble movement, hydration products formation result in release of energy in the form of mechanical waves.

Experiments were performed to investigate the ongoing processes in fresh cement paste at very early age. The cumulative AE signals were studied in relation to the ultrasonic pulse velocity (UPV), capillary pressure as well as heat evolution by isothermal calorimetry to monitor the ongoing processes in fresh cement paste. Researchers performed different studies in the field of cementitious material by applying AE technique. Lura et al. (2009) indicated the onset of AE activity by the fluid-solid transmission of the cement paste due to the cement hydration [2]. The technique of AE has been also applied to estimate and to determine micro-cracking as a consequence of drying shrinkage [3-4]. Furthermore, interesting experimental correlations were detected between features of AE monitoring of concrete during hydration and the final mechanical properties in a previous study [5] .

This study examines the sensitivity of AE signals to the sum of the occurring processes. It will be valuable to potentially determine the different populations of AE depending on their actual sources. This is a very challenging task due to the complexity and the evolving nature of the material. This study is a first step towards this direction with the combination of different other monitored parameters like UPV, heat evolution and capillary pressure.

2 EXPERIMENTAL INVESTIGATION

Figure 1 shows a schematic representation of a typical waveform. Main parameters of AE are the amplitude (AMP), which is the highest peak of the waveform, and the duration DUR, (time distance between the first and last threshold crossing). Energy is the area under the rectified waveform. The rise time (RT) is the delay between the first time the threshold is crossed and the highest peak. The RA value is the ratio of the rise time over the amplitude and the average frequency (AF) is the number of threshold crossings divided by the duration.

The monitoring of AE was applied by piezoelectric sensors (R15) with a resonance frequency of 150 kHz. Silicon grease was applied on the surface of the sensors to increase the acoustic coupling between the sensors face and the mold's surface. The selected threshold was 35 dB and the selected pre-amplification was 40 dB. The setup for AE monitoring consists of three AE sensors as well as three magnetic holders to position the sensors on three different heights on the side of the metallic mold. Figure 2 illustrates the experimental setup, the first sensor is positioned at a height of 60 mm from the bottom of the mold, the second at a height of 120 mm and the third at a height of 180 mm. Silicon grease was applied on the surface of the sensors to increase the coupling between the AE sensors and the metallic mold.

Moreover, the UPV was also applied to contribute in the evaluation of the curing and hardening process of cement paste. The ultrasonic system consists of a wave generator Wave Gen 1410, preamplifier 1220 A, and two sensors (R15). The sensors, are placed on both sides of the Plexiglas plates with a distance between the plates of 10.6 mm that defines the volume of the paste specimen, see Figure 3. The mold for UT has a U-shaped rubber plate. The applied wave generator drives a burst of 10 cycles of the central frequency of 150 kHz to the pulser.

The capillary pressure measurement [6] was applied by a pressure transducer in a plastic mold with a 150 mm long brass tube, see Figure 2. The pressure transducer is coupled to the brass tube having an inside diameter of 4 mm. The brass tube is attached vertically at a distance of 30 mm from the specimen surface and is filled with de-ionized and out-gassed water.

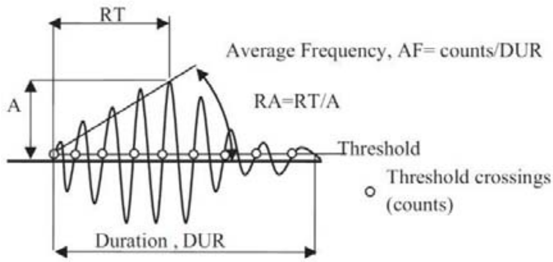

Figure 1: AE waveform and its parameters

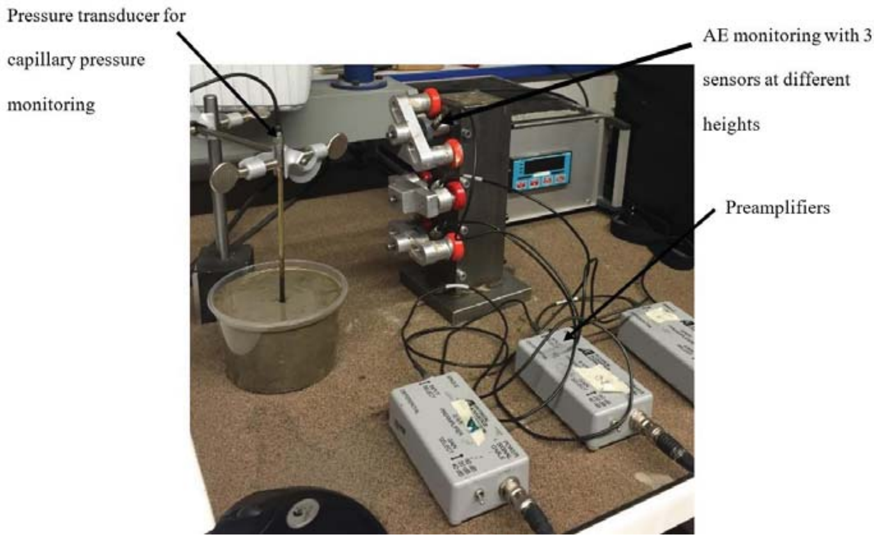

Figure 2: Acoustic emission and capillary pressure monitoring

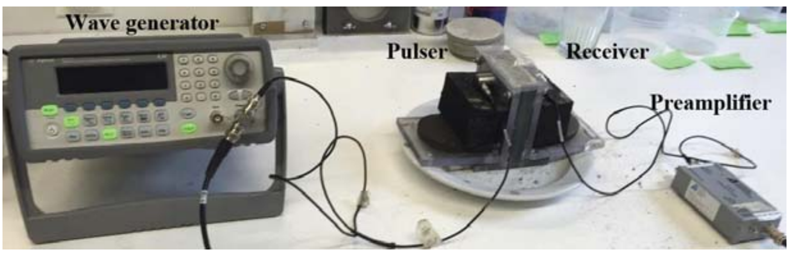

Figure 3: Ultrasonic setup

2.1 Materials

Specimens of cement paste were performed using ordinary Portland cement (CEM I 52.5 N and water) with a water to binder ratio of 0.4. The cement paste was poured into the mold of size 40 × 40 × 200 mm in a single layer and the specimens were compacted on the vibration table at high frequency for 10 seconds. The cement paste has a measured fresh density of 1897 kg/m^3. The specimens were prismatic with a dimension of 40 x 40 x 200 mm. The cement has a density of 3090 kg/m^3 and the chemical composition is given in Table 1. Three experiments have been performed to test the specimens.

Table 1: Chemical composition of cement

Composition	Percentage [%]
CaO	63.9
SiO_2	20
Al_2O_3	5.1
Fe_2O_3	3.4

MgO	0.8
Na_2O	0.34
K_2O	0.75
SO_3	3.1
Cl^-	0.05
Loss on ignition	1.9
Insoluble residue	0.5

3 EXPERIMENTAL RESULTS AND DISCUSSION

3.1 AE activity and capillary pressure During the early age, considerable changes of concrete structure formation takes place. However, the process of hardening and strength gain may last for long period of time.

Figure 4 shows the dependencies of cumulative AE hits at different heights of the mold 60 mm, 120 mm and 180 mm and capillary pressure versus time. Figure 4 is distributed into three age zones that depends on the AE signal changes, exhibiting three AE sensors at different heights. Each zone is selected tentatively and the extension of the zones may vary depending on concrete composition and ambient temperature.

Zone I presents the plastic state of cement paste where the amount of chemically unbound water is high. Consequently, AE activity rate is high for the three sensors while capillary pressure is constant at low levels. This zone is characterized by the maximum rate of AE activity compared to the two other zones. The highest AE activity was recorded at the top of the mold by the highest sensor of 180 mm height. This sensor recorded the highest increase to 530 hits after 200 min, while the other two recorded more than 400 hits. This behavior shows a potential relation of the activity with the hydrostatic pressure that needs to be further examined.

In zone II the AE signals rate is decreased and this may be as a consequence of the continuing hydration process and the reduced unbounded water. At approximately 300 min the capillary pressure started to increase sharply reaching a peak of -5 kPa after 336 min, see Figure 4. This capillary pressure progression is associated to the ongoing evaporation of the cement paste at the surface leading to a reduction of the radii of the menisci [6].

Afterwards, the capillary pressure drops radically after reaching the peak due to the continuing evaporation of the cement paste.

Zone III illustrates a slight increase of AE activity while the capillary pressure remains constant close to zero.

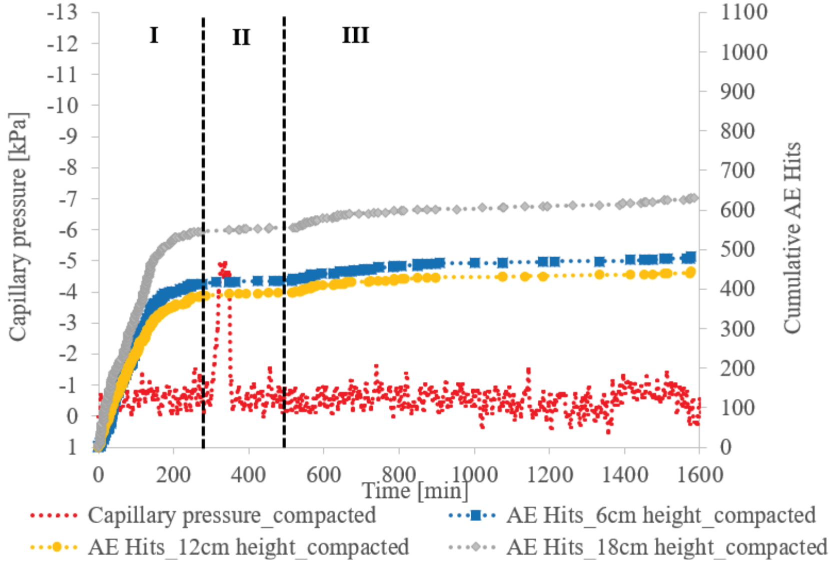

Figure 4: Capillary pressure and cumulative AE hits

3.2 Evolution of Acoustic emission parameter with time

Figure 5 demonstrates a comparison of energy at fresh and hardened state (i.e. stages I and III), measured by three sensors at different heights (60 mm, 120 mm, and 180 mm). Energy is an indication for the released energy by an event of AE. The experimental results confirmed a higher energy for cement paste at hardened state for all three sensors at different heights. For example, the 60 mm height sensor from the bottom of the metallic mold exhibited an amount of energy of 0.13 aJ at fresh state while the energy at hardened state was 0.55 aJ.

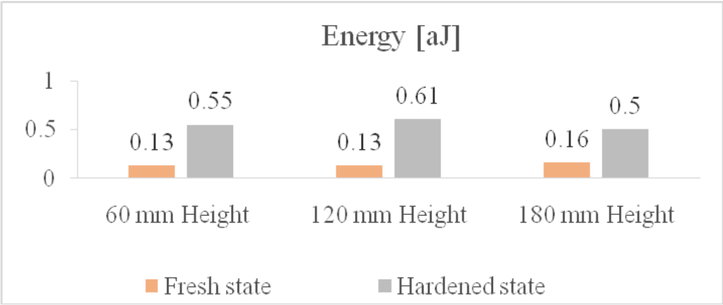

Figure 5: Energy for fresh and hardened cement paste

Similarly Figure 6 shows the comparison for the rise time (RT) between the early and later hydration stages (I and III). As shown on the chart, cement paste presents a higher rise time (approximately double) in hardened state comparing to the fresh state.

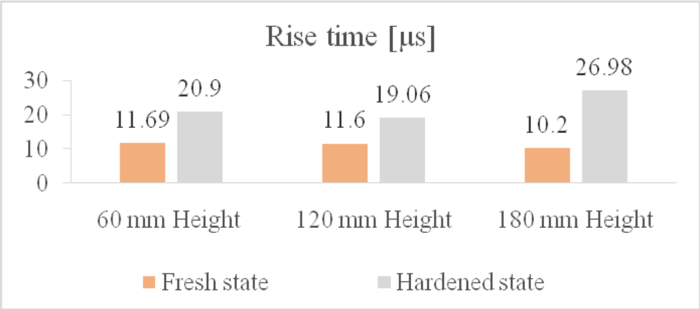

Figure 6: Rise time for fresh and hardened cement paste

Both these charts show the dynamic nature of the processes going on during the first hours of monitoring. The changes in AE parameters indicate that the intensity of the phenomena increase for the hardened stage, even though the rate of the recording AE is much lower, as shown in Fig. 4. It is reasonable that phenomena occurring just after mixing, like bleeding, bubble release, segregation gradually give their place to shrinkage cracking as the paste hardens resulting in different rate of emissions as well as stronger AE hits as revealed by the energy and rise time. However, it should also be kept in mind that the changing of the nature of the material, from a viscous liquid to a solid with considerable stiffness may also influence the propagation of the waves and the final measured parameters.

3.3 Ultrasonic pulse velocity and amplitude

The experimental results of UPV and amplitude are revealed in Figure 7 Figure 8 respectively. Figure 7 presents an increase of pulse velocity for cement paste at 170 min. This change is connected to the formation of a network of hydration products through the paste that facilitates wave propagation [7]. As a result, this may indicate the aforementioned transformation of the viscous liquid to a solid. The continuing stiffness development causes a further pulse velocity increase with lower rate, reaching the value of 4116 m/s after 1600 min, see Figure 7. In Figure 8, the development of the ultrasonic amplitude is depicted. The most important observation concerns the rapid increase of the transmission after approximately 200 min. This signifies the loss of viscous damping as the material hydrates and the increase of stiffness which cannot be monitored until the end as the incoming voltage exceeds the limit of the acquisition board (approximately 18 V) after 300 min.

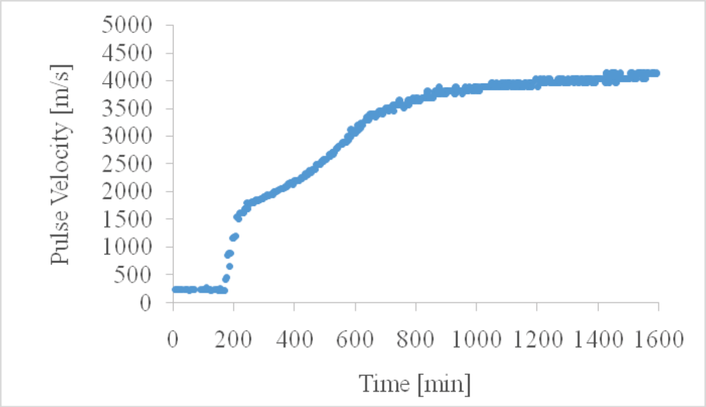

Figure 7: Pulse velocity of cement paste

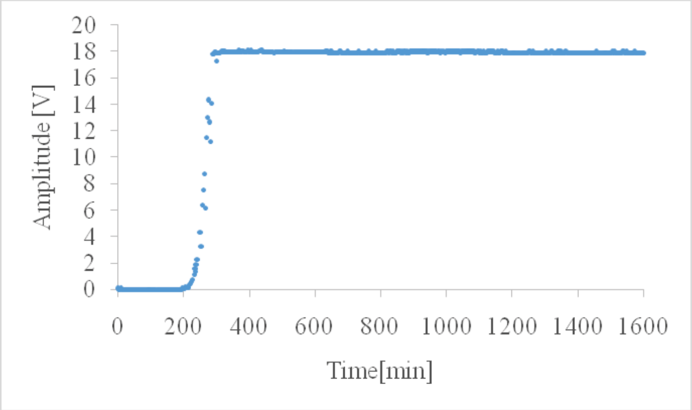

Figure 8: Amplitude of cement paste

3.4 Isothermal hydration

In this work, the isothermal hydration of cement paste has been monitored by isothermal calorimetry [8]. Figure 9 illustrates the relation between the heat evolution and the hydration time. Initially, after the first wetting peak, the heat evolution curve exhibits a low thermal power as a consequence of a slow and well-controlled hydration. Afterwards, the heat evolution curve starts to increase significantly at nearly 168 min. The acceleration of the hydration reaction is related to the evolution of other monitoring parameters already presented. Specifically, the pulse velocity curve starts registering its sharp increase after this time, while the cumulative AE curves start to slow down, see Figure 4. Although this would need further examination, it is reasonable to assume that the acceleration of hydration as indicated by the heat evolution, is leading to the formation of a solid network that hinders the continuation of mechanisms previously active like settlement, segregation and bubble movement. At the same time this solid network facilitates further the wave propagation resulting in increasing velocity and amplitude as shown in Figures 7 and 8. The heat evolution reaches its peak after approximately 703 min.

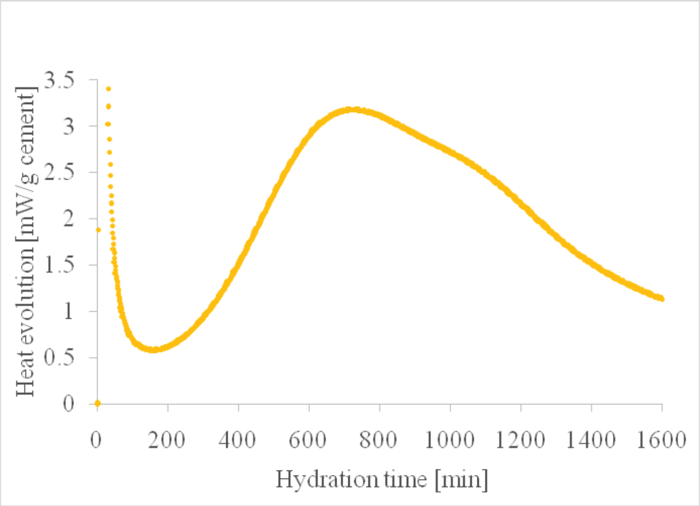

Figure 9: Isothermal hydration of cement paste

4 CONCLUSIONS

This study presents an experimental setup of elastic wave techniques to monitor the ongoing processes in fresh cementitious material. Three piezoelectric sensors were attached at different heights on a steel mold to monitor the AE signals for fresh cement paste. This setup allows recording the AE signals with high sensitivity and presents a high rate of AE signals for the first hours, while the AE activity rate decreases constantly later on. In order to monitor the several simultaneous processes of fresh cement past, an integrated monitoring system that combines different techniques like ultrasonic pulse velocity, capillary pressure and heat of hydration has been applied. At this point, it would be premature to establish any definite connection between different processes and AE, but some interesting experimental correlations are observed.

The experimental results presented a decreased rate of AE when the UPV and ultrasonic through transmission amplitude starts to rise. Simultaneously, the capillary pressure registers a transient peak whereas the heat of hydration starts to increase. The experimental results confirmed a higher energy and rise time for cement paste at hardened state.

Further study in this field is essential in analyzing the experimental observations and correlations as well as to confirm the results for different mixtures, like mortar and concrete.

The process of cement paste structure formation should be further investigated at very early age. In addition, combined analysis of different monitoring techniques should be performed to unveil the numerous AE activities which are registered.

5 REFERENCES

[1] C. U. Grosse and M. Ohtsu, *Acoustic Emission Testing. Basics for Research - Applications in Civil Engineering*. 2008.

[2] P. Lura, J. Couch, O. M. Jensen, and J. Weiss, “Early-age acoustic emission measurements in hydrating cement paste: Evidence for cavitation during solidification due to self-desiccation,” *Cem. Concr. Res.*, vol. 39, no. 10, pp. 861–867, 2009.

[3] K. Van Den Abeele, W. Desadeleer, G. De Schutter, and M. Wevers, “Active and passive monitoring of the early hydration process in concrete using linear and nonlinear acoustics,” *Cem. Concr. Res.*, vol. 39, no. 5, pp. 426–432, 2009.

[4] T. Shiotani, J. Bisschop, and J. G. M. Van Mier, "Temporal and spatial development of drying shrinkage cracking in cement-based materials," *Eng. Fract. Mech.*, vol. 70, no. 12, pp. 1509–1525, 2003.

[5] D. G. A. Sokratis N. Iliopoulos, Yassir El Khattabi, "Towards the Establishment of a Continuous Nondestructive Monitoring Technique for Fresh Concrete," *J Nondestruct Eval*, p. 35:37, 2016.

[6] V. Slowik, M. Schmidt, and R. Fritzsch, "Capillary pressure in fresh cement-based materials and identification of the air entry value," *Cem. Concr. Compos.*, vol. 30, no. 7, pp. 557–565, 2008.

[7] N. Robeyst, E. Gruyaert, C. U. Grosse, and N. De Belie, "Monitoring the setting of concrete containing blast-furnace slag by measuring the ultrasonic p-wave velocity," *Cem. Concr. Res.*, vol. 38, no. 10, pp. 1169–1176, 2008.

[8] ASTM C1679, "Standard Practice for Measuring Hydration Kinetics of Hydraulic Cementitious Mixtures Using Isothermal Calorimetry," *Am. Soc. Test. Mater. West Conshohocken, PA, USA.*, pp. 1–15, 2014.