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Early-age acoustic emission measurements in hydrating cement paste: Evidence for cavitation during solidification due to self-desiccation

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ABSTRACT

In this study, the acoustic emission activity of cement pastes was investigated during the first day of hydration. Deaired, fresh cement pastes were cast in sealed sample holders designed to minimize friction and restraint. The majority of acoustic emission events occurred in lower water to cement ratio pastes, while cement pastes with higher water to cement ratios showed significantly less acoustic activity. These acoustic events occurred around the time of setting. A layer of water on the surface of the cement pastes substantially reduced acoustic emission activity at the time of setting. According to these experimental results, the acoustic emission measured around setting time was attributed to cavitation events occurring in the pores of the cement paste due to self-desiccation. This paper shows how acoustic emission might be used to indicate the time when the fluid–solid transition occurs in a cement paste, often referred to as time-zero. Knowledge of time-zero is fundamental for determining when mechanical properties develop and in calculations of residual stresses.

1. Introduction

The traditional use of acoustic emission (AE) for concrete and concrete structures is to monitor damage and crack formation during loading, e.g. [1,2]. Other researchers studied AE activity produced by conversion of high alumina cement pastes [3], by concrete subjected to freeze–thaw cycles [4] or by restrained drying of cement paste or mortar [5–7] or other ceramics [8]. Shiotani et al. [9] found that, during drying of cement paste and of composites containing monosized glass beads, AE activity due to moisture movement had a narrow band frequency larger than 400 kHz, while AE events due to cracking had a lower frequency spectrum.

Several researchers have observed that cement paste and concrete show AE activity during hardening, even in the absence of external drying. AE activity of fresh cement pastes was researched by Muravin et al. [10–12], who attributed the observed AE events in the first hours after mixing to formation and dissolution of hydrates, especially ettringite. Similar explanations for the measured AE events were offered by Kuz'menko et al. [13] and by Skal's'kyi et al. [14].

Chotard et al. [15] used AE to monitor setting of calcium aluminate cement. They observed that most of the AE events were concentrated between 3 and 6 h after mixing, during which time the central frequency of the waveform began to shift from about 120 kHz to about 170 kHz. They considered this change of frequency to be due to the hardening of the material, which progressively allows higher frequency components to be transmitted. The source of the AE events was attributed to the liberation of energy during the consumption of water, formation of hydrates and emptying of the capillary pores. Chotard et al. [16] also attributed AE events from three different phenomena occurring during hydration of cement, namely dissolution of grains, formation of new phases and emptying of pores. All three phenomena produced measurable AE events in model systems that were chosen to separate these three different contributions. In a later paper [17], a fast Fourier transform was applied to the recorded waveforms and AE events with specific amplitude and waveform were associated with emptying of the capillary pores in hardening calcium aluminate cement.

Van Den Abeele et al. [18] measured AE on concrete of different w/c. The AE events started some hours after casting, were highest in number in the low w/c concrete and continued in the first 3 days. The absence of AE events in the first hours was attributed to the damping of the wave from the fresh concrete before set.

This paper investigates acoustic activity in cement paste during hydration. The hydration of cement paste causes a volume reduction called chemical shrinkage. Chemical shrinkage occurs since the volume of the hydration products is smaller than that of the reactants [19]. Before the cement paste 'sets' (solidifies), chemical shrinkage causes bulk shrinkage of the cement paste. The chemical shrinkage is equal to the total external volume change as internal cavities cannot be sustained by a fluid. During setting, the cement paste becomes stiff enough to resist a portion of the volume change caused by chemical

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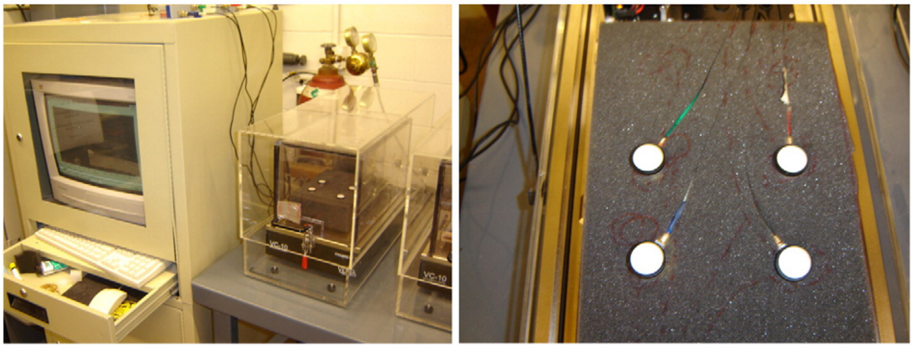

Fig. 1. Environmental chambers used during this investigation (left) and close-up of the transducers placed upside down in the low-density foam pads (right).

shrinkage; as a result, tensile stresses start to develop in the pore fluid and the solids are subjected to compressive stresses [20]. As the pore fluid tensile stress increases, cavities may form inside the cement paste. This is a cavitation phenomenon that involves release of energy, which may be detected as an AE event. The hypothesis of this paper is that cavitation is the primary cause of acoustic activity.

When a bubble is formed in a liquid, the main acoustic events are produced by the nucleation of the bubble, not by the bubble bursting at the surface [21]. It is likely that the acoustic events caused by cavitation within partially-saturated porous solids involve a large population of bubbles. These bubbles influence each other, coalesce, grow, and subsequently burst, possibly in repeated cycles [22]. In hardening cement pastes, bubbles are filled with water vapour and with air, which was originally dissolved in the mixing water. The presence of air or other impurities in the water is sufficient to abate the pressures needed for cavitation [23]. A cavitation nucleus is created at the solid–water interface, where the surface is concave [24]. From there it grows, detaches and forms a gas bubble. It is noticed that AE has been employed in several occasions to detect bubble formation, for example bubbles created during corrosion of aluminium [25] or cavitation in tracheids of plants subjected to drying [26].

Freshly-mixed cement paste is a suspension of cement particles and hydrates in pore fluid [27]. During the hydration process, the structure of the cement paste develops to a level where the paste starts behaving as a viscous solid and permits stresses to develop. This process, known as setting, has often been identified using mechanical penetration tests. However, the definition of setting is somewhat vague and arbitrary [28]. This study investigates the use of AE for detecting the cavitation that occurs when a solid skeleton forms in hydrating cement paste. First results were presented in [29], where AE events were correlated with Vicat penetration tests. In [30], AE events correlated well also with the divergence between chemical shrinkage and autogenous strain. A variety of techniques for assessing setting, including AE, ultrasonic wave velocity, volume change, electrical conductivity, rheology, restrained ring, and calorimetry were applied to the same cement pastes in [31], finding a generally good agreement between most of the methods. In this paper, results of additional experiments on different cement pastes are presented and a mechanism for the occurrence of cavitation in cement pastes during setting is proposed and discussed.

2. Materials

The cement used in this investigation was a Type I Portland cement with a Blaine fineness of 360 m^2/kg and a calculated Bogue phase composition of 60% C_3S, 12% C_2S, 12% C_3A, 7% C_4AF and a Na_2O equivalent of 0.72%.

Plain cement pastes with water to cement ratio (w/c) 0.30, 0.35 and 0.40 were prepared with the addition of 0.5% by weight of cement of a high-range water reducer (Glenium 3000NS). In addition, a silica fume cement paste was mixed with w/c 0.3, 20% silica fume addition (ELKEM MS965) and 0.83% water reducing agent (PS-1232) by weight of cement.

Each mixture was prepared under vacuum. Deionized water was initially boiled to remove dissolved air and then allowed to cool down to room temperature. The admixtures were added to the water and mixed thoroughly. The mixture was then transferred to a burette and the appropriate volume was added to the cement in a previously evacuated vacuum-tight container holding the cement [32]. The vacuum container was then shaken by a commercial paint mixer for approximately 10 min until the cement paste was thoroughly mixed [32]. The size of each mixture was approximately 400 g.

3. Methods

3.1. Acoustic emission testing apparatus

The acoustic emission system used in this investigation consisted of broad-band transducers, pre-amplifiers, and a data acquisition system. The transducers (Vallen DECI SE375, with a peak frequency of

Fig. 2. Sample holders for acoustic emission measurements on fresh cement pastes. Left: sample Holder B with a flexible plastic film base (Saran wrap). Right: sample Holder C with an acetate sheet base (transparency) that is held in place with a bead of silicone.

375 kHz) were used to convert detected vibrational waves into electrical signals. The signals were amplified using a 34 dB preamplifier (AEP4 by Vallen Systeme) and fed directly to a six channel AE acquisition system (Model AMSY4 by Vallen Systeme GmbH) interfaced with a personal computer. The software packages Visual AE™ and Visual TR™ were used in the data analysis. The AE sensors used in this investigation weighed approximately 31 g, had a stainless steel case, a ceramic wear plate, and a wear surface with diameter 20 mm. An acoustic event was defined as an event with amplitude greater than a 23.8 dB threshold. Acoustic energy was defined as the total energy detected by the sensor which consists of the integral of the absolute amplitude of the signal over time for the duration of the AE signal. While an acoustic event enables event 'counting', acoustic energy allows the user to obtain more information about the magnitude of the acoustic event in a single parameter.

All the tests took place inside a Vena VC-10 environmental chamber (Fig. 1, left) where the relative humidity was held constant at 87 ± 1% and the temperature at 23 ± 1 °C. Low-density foam pads were placed on the floor of the chamber to isolate the sensors from outside vibrations (Fig. 1, right). Depressions were carved in the foam pads to hold the acoustic emission transducers and the wires running to the recording equipment.

Several different sample holder geometries were investigated, consisting of plastic containers with airtight snap-on lids. The sample holders were cut halfway up the height perpendicular to the axis of the cylinder, so that the final containers were 35 mm in height and 26.6 mm in diameter. Two of the most promising sample holders, sample holders B and C, differed in the material used for the bottom of the holder (Fig. 2). In sample holder B, rubber bands held a thin flexible plastic film that was stretched tightly over the cut end. In sample holder C, 3.5 mil acetate sheets were glued with a bead of silicone to the cut end of the plastic container. Small pin holes were made in the lids to relieve air pressure when the containers were closed. These holes were later covered with tape to prevent evaporation during the tests. Unless noted otherwise the sample holders were filled with 8.0 ± 0.1 g of freshly-mixed cement paste and vibrated for 10 s. At this point, any water or paraffin oil would be added to the top of the samples if necessary. The sample holders were then carefully placed on top of the sensors. Vacuum grease was placed between the sample holder and the transducer to ensure good contact and transmission of AE between samples and sensors. The time between when water was added to the cement in the mixing process and the beginning of the measurements was approximately 20 min.

3.2. Other measurements of setting

Setting was measured using the Vicat needle test according to ASTM C191 [33]. Initial set is described as the time at which a 1 mm diameter pin with a certain load will penetrate 25 mm into the cement paste, while final set refers to the time at which the pin can no longer penetrate.

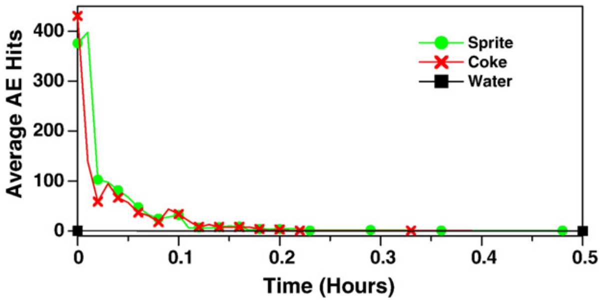

Fig. 3. Hits summed over 0.01 h intervals for pure water, Sprite© and Coke©.

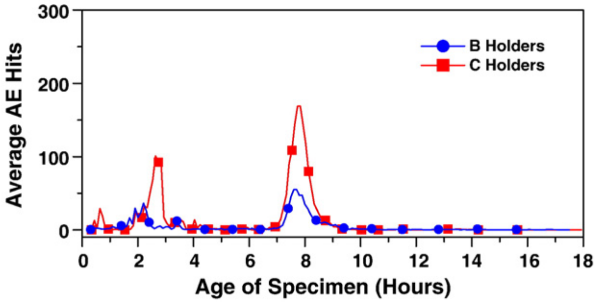

Fig. 4. Average hits summed over 0.1 h intervals for B and C holders and w/c 0.30 cement paste.

Chemical shrinkage was measured by monitoring the change in buoyancy that occurs in samples, with a layer of water on top of the sample, suspended in paraffin oil. Volumetric autogenous strain was measured using the membrane method in a paraffin oil bath. Details of the chemical and autogenous testing procedure are provided elsewhere [34,35]. Setting was evaluated based on the divergence of the chemical shrinkage and autogenous strain curves, which occurs when a solid skeleton is formed in the hydrating cement paste [30,36,37].

4. Results

In this section, the results of acoustic emission experiments performed on different liquids and cement pastes are presented and explained. The results are discussed in the following Section 5.

4.1. Preliminary tests with carbonated liquids

Several tests were performed with water and carbonated beverages to ascertain whether the acoustic activity of the bubbles could be measured by the setup. Plain water and two different types of carbonated soft drink (Sprite© and Coke©) were tested. Carbonated soft drinks were tested immediately after opening the bottle. Approximately 1 ml of liquid was poured in the bottom of each container, which was left open while the AE events were measured. Results are shown in Fig. 3. AE event activity of carbonated soft drink was high at first but slowly decreased as the liquid lost carbon dioxide. Pure water showed almost no acoustic activity with only four events overall.

4.2. Tests on cement paste

The AE response measured on fresh cement paste (w/c 0.30) is illustrated in Fig. 4. In the first 2 h after the start of the experiment, almost no AE events occurred. This was sometimes followed by some minor activity in some samples (e.g., 2–3 h) however this did not occur for every mixture. A quiet period (e.g., 3–7 h) followed this minor activity until acoustic activity resumed around 7 h. The activity

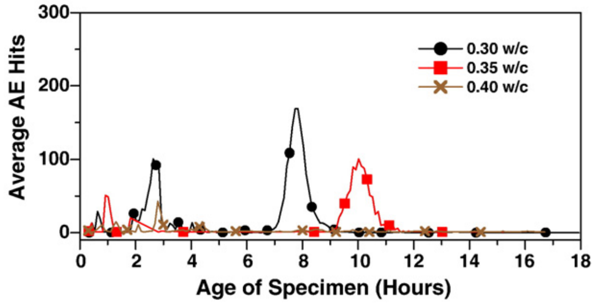

Fig. 5. Comparison of hits summed over 0.1 h intervals between 0.30, 0.35, and 0.40 w/c.

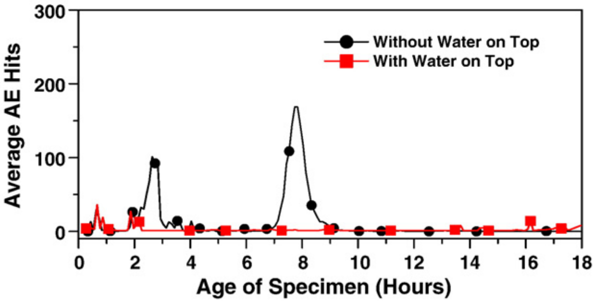

Fig. 6. Typical hits summed over 0.1 h intervals for a 0.30 w/c paste with and without water on top.

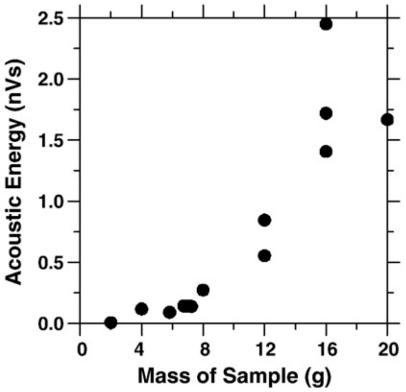

Fig. 8. Total energy of acoustic events around setting time plotted as a function of sample mass for w/c 0.3 cement pastes.

at 7 h corresponds to the solidification (i.e., setting) of the cement paste, and continued until 9 h. A minimum of 4 samples were tested for each condition. All sensors registered events in the same periods, though the number and amplitude of the events varied; this could be due to differences in the contact and transmission across the coupling agent. It should be noted that tests with the C holder registered about twice as many hits as the B holders. In Fig. 4 typical results for both B and C holders are shown. Comparison between different mixtures and different testing conditions are only possible within the same series performed on the same type of holders. Unless noted otherwise the type C holders were used in all the remaining tests. It should be noted that the coefficient of variation for the specimens tested with type C holders ranged from 0.1 to 0.3 for both the number of AE events and the AE energy for all samples tested (excluding 2 outliers), except specimens deliberately exposed to drying to determine the potential influence of premature drying.

Three different w/c pastes were tested. Results are shown in Fig. 5. A peak occurs in the 0.30 and 0.35 mixture slightly after final setting (e.g., 7.0 to 7.2 h where initial set was 6 h and final set was 7 h for the 0.30 mixture); no distinct peak is observed in the 0.40 mixture. It can also be noted that there is more activity in the low w/c pastes around the time of set, while the initial acoustic activity is similar in all the mixtures.

The addition of a thin layer of water on top of the cement pastes left the initial acoustic activity unaltered but eliminated the peak in acoustic activity that occurs around setting. Examples are shown in Fig. 6 for a w/c 0.30 paste and in Fig. 7 for a w/c 0.3 cement paste with 20% silica fume addition. On the other hand, a layer of paraffin oil on top of the cement pastes did not eliminate the activity around setting, as clearly shown on Fig. 7.

In Fig. 8 the total energy of acoustic events around setting time is plotted as a function of sample mass for samples of w/c 0.3 cement pastes. In these tests, different amounts of cement paste were poured in the moulds, obtaining samples with different height but having the same contact surface with the bottom of the mould. Considering the uncertainty of the measurements, the total AE event energy appears to increase approximately linearly with the mass of the sample.

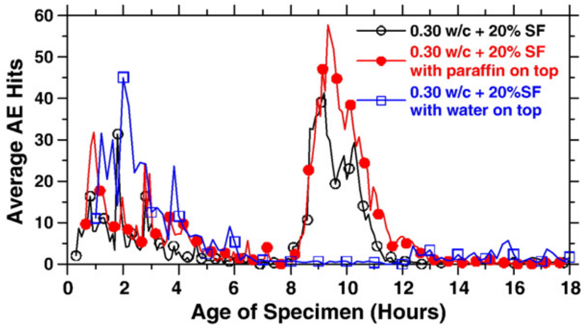

Fig. 7. Typical hits summed over 0.1 h intervals for a 0.30 w/c + 20% silica fume cement paste either with a layer of water or of paraffin or nothing on top.

Fig. 9 compares the acoustic activity with the time of set. Two different methods for determining the time of set are presented. The first method uses the Vicat test to determine the initial and final set in accordance with ASTM 191 [33] and according to this method, initial set occurs at 6 h and a final set at 7 h. The second method uses the divergence between the chemical shrinkage and the autogenous strain (as measured using the membrane method [34]). The volumetric strains have been converted to linear strains (dividing them by 3); negative strain means shrinkage. Chemical shrinkage and autogenous strain are similar initially as the fluidity of the paste does not allow creation of internal empty pores. At 7 h the measured shrinkage responses begin to diverge, which indicates the setting time of the cement paste [30]. It is noticed that the main period of acoustic activity of the cement paste starts at 7 h, peaks just before 8 h, and ceases at 9 h. No additional substantial activity was registered after this time, before the test was stopped at 20 h.

5. Discussion

In this section, a series of alternative hypotheses are considered to explain the AE events of cement pastes during setting. These alternative hypotheses are analysed in detail and their soundness is tested against the experimental evidence collected during this study. Starting from the results of this analysis, a conceptual model identifying the observed AE events with cavitation events in the hardening cement paste is explained and briefly discussed.

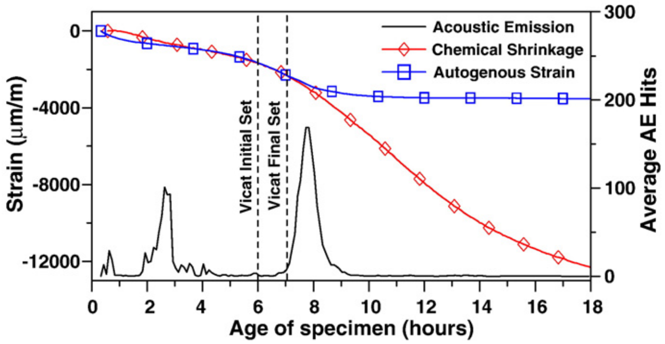

Fig. 9. Vicat setting time, chemical shrinkage, autogenous strain, and acoustic emission activity as a function of age for a 0.30 w/c cement paste. Acoustic hits are summed over 0.1 h intervals.

5.1. Discussion of alternative mechanisms

Different hypotheses that could explain the AE events registered in cement pastes around setting time are listed below; while some of these have been proposed in the literature, others have been proposed by the authors during the course of this investigation:

1) dissolution of cement grains [10,16];
2) formation of hydrates [10–15];
3) friction with the walls of the mould due to shrinkage;
4) microcracking;
5) cavitation events in the cement paste [15–17,29–31].

The first two proposed mechanisms, namely the dissolution of cement grains and the formation of hydrates, appear to be contradicted by the results shown in Figs. 5–7. In Fig. 5, while the initial AE events in the first 3 h is similar in cement pastes of all w/c, there is much higher activity in the low w/c pastes around the time of set and almost no activity in the w/c 0.4 cement paste. Since dissolution of cement grains and formation of hydrates occur in all these cement pastes, regardless of w/c, in the observed period of 18 h (Fig. 5), the observed AE events do not appear to be attributed to these mechanisms. Moreover, Figs. 6 and 7 show that a layer of water on top of the cement paste samples eliminates the AE event peak around setting. Since about the same hydration products should form in these cement pastes during the observed period, whether there is a layer of water on top or not, Figs. 6 and 7 confirm that the registered AE events around the time of setting cannot be caused by dissolution of cement grains or formation of hydrates, as proposed in refs. [10–16].

According to the third proposed mechanism, the shrinkage of cement paste around setting time and the friction with the mould would cause the registered AE events. The results of Figs. 5–7 would apparently support this mechanism, because pastes of higher w/c or saturated (with a layer of water on top) would shrink less around setting, and thus would produce fewer AE events. However, this mechanism does not explain why the observed AE events concentrated around setting time, while autogenous shrinkage is continuously increasing for several days (see Fig. 9 and ref. [34]). More important, Fig. 8 shows that the total AE event energy appears to increase almost linearly with the mass of the sample. This confirms that the recorded AE events are occurring within the bulk of the samples. If the observed AE events were due to friction of the sample with the bottom or the walls of the mould, it could be supposed that a high energy would be measured also for very light samples of limited height, but having a large contact surface with the bottom of the mould. On the contrary, it appears that very little AE event energy is measured for thin samples (Fig. 8). This fact possibly also indicates that in very thin samples, air readily penetrates into the pores of the cement paste from the top and contributes to reduce cavitation around setting time. A final observation that contradicts the friction hypothesis is that AE events were registered even when patties of stiff cement paste were cast directly on thin films in contact with the sensors [38], without the side walls present in holders B or C (Fig. 2).

It can be speculated that the AE event peak could also be caused by internal microcracking in the cement paste (fourth proposed mechanism). However this hypothesis is at variance with the AE event activity being concentrated in a short time interval immediately after set, since acoustic activity caused by microcracking or friction should occur over a longer period of time as it has been observed to increase with shrinkage [39]. Moreover, microcracking appears to occur in cement pastes primarily in the presence of substantial internal or external restraint [40].

The last proposed mechanism, namely cavitation of air bubbles due to chemical shrinkage that empties the capillary pores, appears to be supported by all the experimental evidence collected in this study. Fig. 5 shows that the AE events around setting decrease with the increase of w/c. In low w/c pastes, solidification occurs within a shorter period of time with a rapid change of stiffness; at the same time, the capillary pores are smaller and consequently more and smaller bubbles are created. As the w/c increases, it is expected that cavitation still occurs albeit not as dramatically as in low w/c pastes. Incidentally, it is noted that this may make detection of set with AE events more difficult for high w/c pastes. The results of Figs. 6 and 7 can also be explained according to the cavitation hypothesis, namely as soon as the cement paste gains stiffness, the water on top of the sample is sucked into the cement paste and replaces the water consumed by chemical shrinkage, thus eliminating the cavitation of gas bubbles in the paste (Fig. 10). On the contrary, the paraffin oil on the top of the paste, which is non-wetting and has a higher viscosity than water, does not readily penetrate the pores of the cement paste, and thus the peak of AE events around setting is left largely unaltered. It appears even (Fig. 7) that the number of AE events may be increased in pastes with paraffin oil on top. This might be due to the fact that paraffin oil effectively seals the cement paste against penetration of air from the top surface, thus enabling the cavitation of bubbles in the cement paste. Fig. 8 also support the cavitation hypothesis, since the AE event energy of the cavitation events is supposed to depend on the number of bubbles forming, which depends on the amount of cement paste in the observed sample. Finally, Fig. 9 shows that the AE events are concentrated in a short period of time, about 2 h after setting, and this corresponds to the hypothesis that the released AE events are produced by a large number of gas bubbles that nucleate in the capillary pores around the time of solidification, while the later creation of empty pores in the cement paste (see the divergence of chemical and autogenous shrinkage in Fig. 9) is produced by enlarging already existing bubbles with little AE activity.

The origin of the initial AE activity (i.e. the AE events occurring between 1 and 4 h in the plain cement pastes, Fig. 5, and between 1 and 6 h in the cement paste with silica fume, Fig. 7) is currently not clear. This activity occurs during the dormant period in all pastes, regardless of the w/c. It is particularly prominent for the cement paste with silica fume, where it is not influenced by the presence of a layer of water on top of the specimen (Fig. 7). Among the mechanisms proposed for AE activity of fresh cement pastes, dissolution of cement grains [10,16] or formation of hydrates [10–15] could be considered for explaining this activity. However, if these were the source of these AE events, it is surprising that they would cease as the hydration rate increases at the end of the dormant period (approximately 3–4 h for the plain system). Another hypothesis is that this AE activity is produced by movement and interaction of solid particles within a cement paste as the paste experiences volume change due to chemical shrinkage (Fig. 9) and more importantly due to settlement. It appears

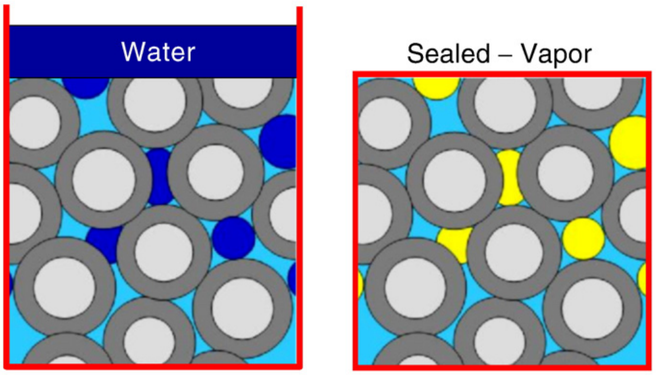

Fig. 10. Schematic view of cement pastes around setting time in saturated (left) and sealed (right) conditions. Water is represented in blue, cement grains and hydration products in grey and empty pores in yellow. In the sample with a layer of water on top, the water is sucked into the cement paste as soon as empty pores are created, preventing the formation of gas bubbles. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

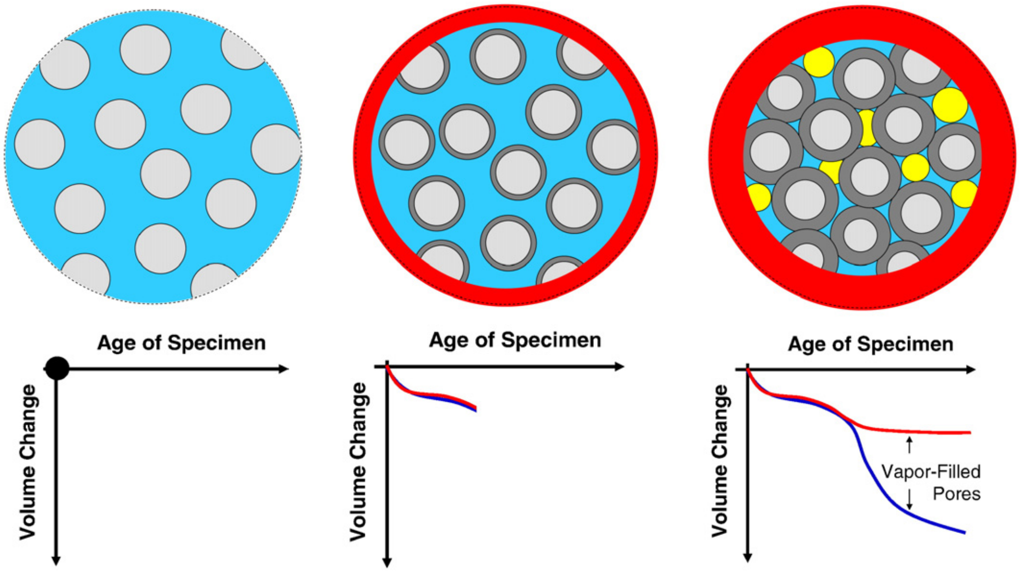

Fig. 11. Schematic view of cement pastes at different stages of hydration (above) and of the simultaneous development of chemical and autogenous shrinkage (below). Water is represented in blue, cement grains in light grey and hydration products in dark grey, empty pores within the cement paste in yellow and the autogenous (bulk) shrinkage in red. Notice that the amount of autogenous shrinkage is exaggerated in the schematic views at the top for the sake of representation. In the graphs, the blue line represents chemical shrinkage and the red line autogenous shrinkage. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

that this occurs while the space between the particles is decreasing as the system is settling and pore fluid is being extruded (described as Phase I in [41]). The AE activity appears to stop when the paste stops settling and the particles are pulled more closely to each other due to the underpressure in the fluid (Phase II in [41]). The acoustic activity resumes after final setting when the first empty pores are created by the cavitations of voids.

5.2. Detailed description of the conceptual model

In this section a conceptual model for cavitation in cement paste around setting is proposed and some of its features are discussed.

From the point of view of the AE activity, the hydration and hardening of a cement paste can be divided into a number of phases:

1) At the moment in which cement is mixed with water, a cement paste can be considered as a suspension of cement particles in water (Fig. 11, upper left) [27,41]. This point can be considered as the origin of the volume change versus age graph (Fig. 11. lower left), as practically no hydration and no chemical shrinkage has occurred yet.
2) In the first hours of hydration the cement paste is still plastic and the chemical shrinkage developing due to hydration is totally converted into bulk (autogenous) shrinkage (Fig. 11, center). In other words, the cement paste collapses and no internal voids are created. Few AE events are registered in this phase.
3) As hydration progresses, the cement paste acquires sufficient stiffness to sustain the first cavitation of a bubble. This corresponds to the onset of AE activity. In this phase the bubbles nucleate and then are compressed again, since the paste behaves viscoelastically. The sudden formation of a bubble causes a shockwave that triggers further bubble formation wherever the equilibrium is unstable. This phase corresponds to the divergence point between the chemical shrinkage and the autogenous shrinkage curves (Fig. 11, lower right corner).
4) As the cement paste hydrates further, it acquires sufficient stiffness to sustain the presence of a number of stable, isolated bubbles. This corresponds to the peak of the registered AE activity, as most bubbles are created in this phase (Fig. 11, right). The total volume of the bubbles created in this phase corresponds to the difference between the internal volume change (chemical shrinkage curve in Fig. 11, lower right) and the external volume change (autogenous shrinkage curve in Fig. 11, lower right). As the stiffness of the cement paste increases, other bubbles can be created in smaller capillary pores. The AE events start to decrease when the energy required to expand already existing bubbles is lower than the energy needed to create new bubbles.
5) In the final phase, occurring after about 10 h in Fig. 9, the volume of the internal voids continues to increase in the cement paste, but the gas phase percolates through most of the capillary pores and few if any new empty pores are created. As almost no new bubbles are created in this phase, little AE activity is recorded in this period.

6. Conclusions

Significant acoustic activity occurs in cement pastes with low w/c around setting time. Evidence from the literature and from this study indicates that this is activity is not produced by dissolution of cement grains, nor formation of hydrates, nor friction with the walls of the mould due to shrinkage, nor to microcracking. All the experimental observations suggest that this activity is caused by creation of gas-filled bubbles in the pores.

These gas-filled bubbles form as soon as the solid skeleton of the cement paste is able to sustain an internal cavity. Therefore, the onset of acoustic activity may be used to indicate the fluid–solid transition of a cement paste as a consequence of hydration. Comparison with other methods to determine setting time show that the main period of acoustic activity indeed starts around the moment of final set of the cement paste, as determined by the Vicat measurement.

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