Studies of Hydration and Drying in Cement Pastes by Scanning X-Ray Absorptiometry

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STUDIES OF HYDRATION AND DRYING IN CEMENT PASTES BY SCANNING X-RAY ABSORPTIOMETRY

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ABSTRACT

Hydration and drying in cement pastes are studied using the X-ray environmental chamber at DTU. By using the X-ray detector to detect changes in internal density (indicative of drying), the influence of w/c ratio on drying during hydration is quantitatively examined.

Key words: X-ray absorptiometry, cement paste, drying

1. INTRODUCTION

The proper curing of field concrete is critical to obtaining optimum performance. This is particularly true from a durability standpoint since it is the top layer of the concrete that provides the first protective barrier against the ingress of deleterious substances.

In current standards, recommended curing practices are given that depend on the external environment but generally do not consider the differences in concrete mixture proportions. Many years ago, Powers /1/ suggested that concrete need only be cured until the capillary pore system de-percolates. The lower the w/c ratio, the sooner this de-percolation will occur in the hydration process. The capillary pore structure in a low w/c ratio high performance concrete is different from that in a conventional 0.5 w/c ratio concrete.

Hydration and drying in cement pastes are studied using the X-ray environmental chamber at the Technical University of Denmark (DTU). By using the X-ray detector to detect changes in internal density (indicative of drying), the influence of the w/c ratio on drying during hydration can be quantitatively examined.

2. THE X-RAY ENVIRONMENTAL CHAMBER

The X-ray environmental chamber is described in /2/. In the chamber, the environment for the specimens can be varied i.e. the specimens can be exposed to different relative humidities and air temperatures. The addition of one or more fans within the chamber would allow for variation of the convective transfer between the specimen and the surrounding air.

The 1-mm diameter transmission beam from the X-ray source passes through the test specimen to the detector, Figure 1.

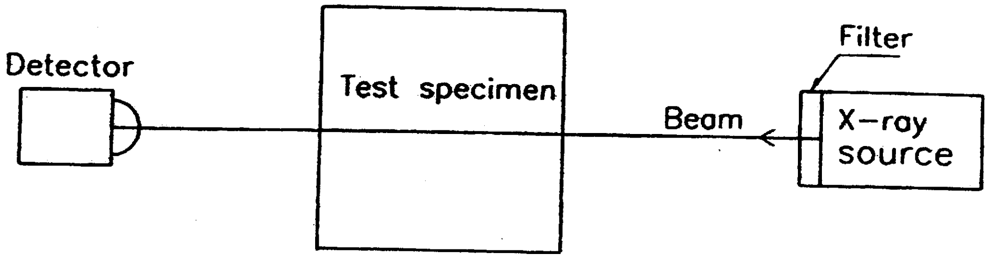

Figure 1. Transmission beam from X-ray source through test specimen to detector.

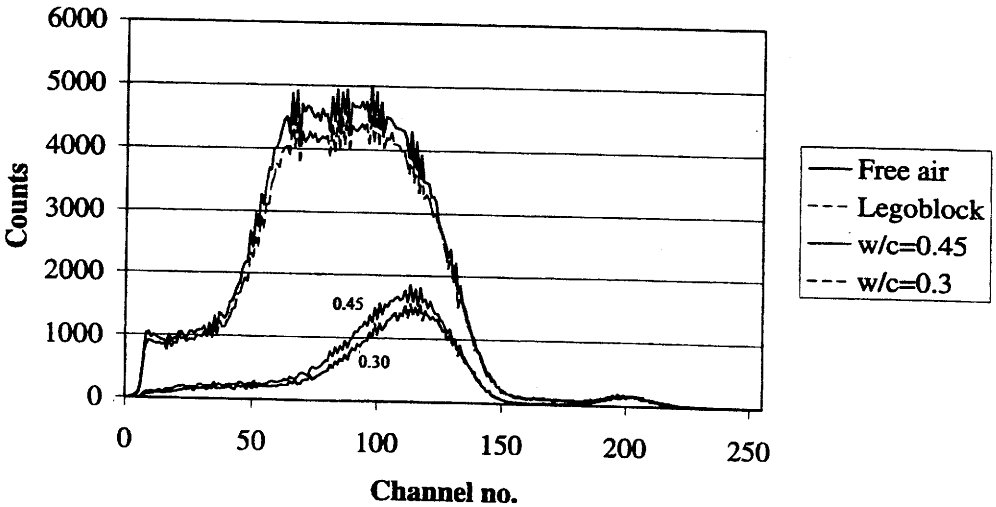

Figure 2. Spectra for free air, Lego block and two fresh cement pastes of w/c = 0.3 and 0.45.

By using an energy level of 75 keV and a current of 15 μA for the X-ray source and 256 discriminator channels for the detector, the spectra obtained for two fresh cement pastes of w/c = 0.3 and 0.45 are shown in Figure 2. The small peak having a maximum around channel 200 is from an internal Cobalt-60 source built into the system to perform automatically calibration. As the cement paste thickness is 12.5 mm in both cases, the lower curve for w/c = 0.3 indicates a more dense paste. In the following figures, the sum of counts for channels between 50 and 150 (see Fig. 2) are used. Assuming a Poisson process, the relative standard uncertainty in counts should be on the order of $1/\sqrt{\text{counts}}$, or about 0.4 % for the cement paste specimens presented in section 3.

3. TESTS ON FRESH CEMENT PASTES

3.1 Test setup using Lego blocks

A preliminary experimental setup using Lego[1] blocks as moulds for fresh cement paste has been established. The Legos seem to function well as sample holders due to: 1) their generally low absorption of X-rays, 2) their inherent stackability (important for initial placement and replacement after weighing), 3) a cap on top of the block gives sufficient sealing against water loss, and 4) a high surface energy facilitates the filling of the Lego mould by the viscous cement paste. The inner dimensions of a Lego block are l = 12.5 mm (beam direction), w = 4.7 mm and h = 8.4 mm. This block size is chosen in order to be able to use a 5-second count time for each measuring point. The Lego blocks are placed on a holder in the environmental chamber, with their bottom surface at a height of 28 mm in system coordinates.

3.2 Mixing of cement pastes

The cement used is a Type I/II ordinary portland cement, designated as cement no. 133, issued in June of 1999 by the Cement and Concrete Reference Laboratory at the National Institute of Standards and Technology, USA. After weighing the cement and the demineralized water, the two components are mixed "by hand" for 2 minutes. After mixing of cement pastes and filling of the moulds, the X-ray equipment is used to scan along the vertical axis (from position 28 mm to position 36 mm) in the center of each Lego block in steps of 0.2 mm, starting from the bottom of the block. The masses of the blocks are determined at 0 h, 2 h, 4 h, 8 h, 19 h, and 24 h, and approximately once per day thereafter.

3.3 The five different cases

The five different cases (blocks) to be reported here are the following:

1) cement paste w/c = 0.3 – no cap
2) cement paste w/c = 0.45 – no cap
3) cement paste w/c = 0.3 – sealed with cap
4) cement paste w/c = 0.45 – sealed with cap
5) block filled with water – sealed with cap

The air speed above the Lego blocks is nearly 0 m/s. The climate in the environmental chamber is 23 °C, 50 %RH.

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[1] Certain commercial equipment is identified by name in this paper to specify the experimental procedure. In no case does such identification imply endorsement by the Technical University of Denmark, nor does it indicate that the products are necessarily the best available for the purpose.

4. RESULTS AND DISCUSSION

Figures 3-6 show results during 168 hours of measurements for the four cement pastes.

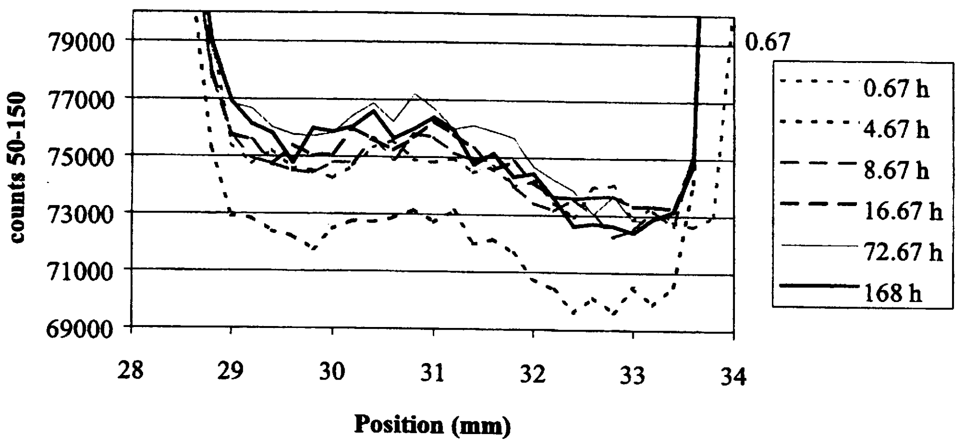

Figure 3. Results for cement paste w/c = 0.3 – no cap (block 1).

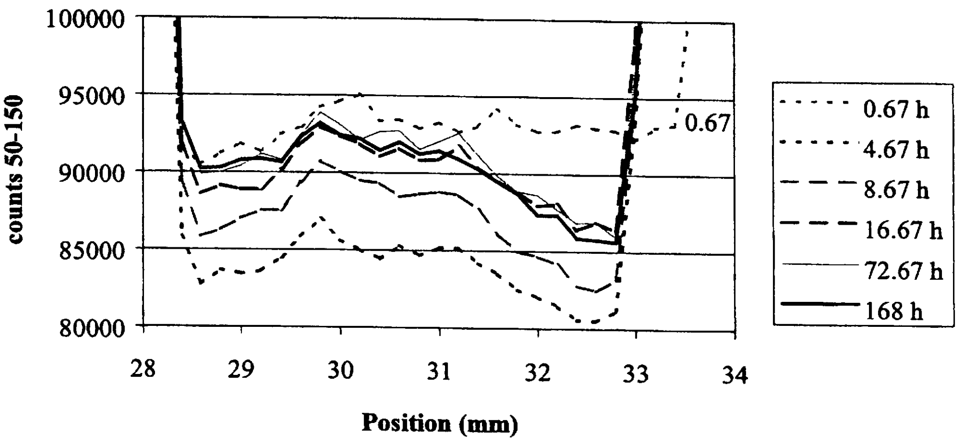

Figure 4. Results for cement paste w/c = 0.45 – no cap (block 2).

Figure 3 shows results for w/c = 0.3 – no cap (open). In position 28.7 mm, the paste starts in the bottom of the Lego block. At the top surface, initial settling is seen as the curve shifts from end position 34.0 mm at 0.67 hours after mixing to end position 33.7 mm at 4.67 hours. This indicates a rapid water loss in this period, which is also confirmed by mass readings for the block, Figure 7.

Due to this settling at 4.67 hours (around the time of set) the paste is more dense, as the counts fall from a level of 73000 – 76000 counts at 0.67 hours down to a level of 70000 – 73000 counts at 4.67 hours. From 4.67 hours to 168 hours a small, but uniform drying is observed together with very little water loss after 24 hours, Figure 7.

Figure 4 shows results for w/c = 0.45 – no cap (open). This paste is not as dense, as the counts level is shifted from 70000 – 76000 counts for w/c = 0.3 up to 80000 – 94000 counts for w/c = 0.45. The same tendency as for w/c = 0.3 can be seen: 1) initial settling, 2) substantial, but uniform drying, and 3) rapid water loss during the first 24 hours. It is interesting that in both cases, the drying is relatively uniform for these specimens approximately 4.5 mm in thickness, as opposed to proceeding as a sharp drying front progressing inward from the exterior surface of the specimen.

Similar "uniform" drying profiles have been observed for a cement paste 5 cm thick using magnetic resonance imaging /3/.

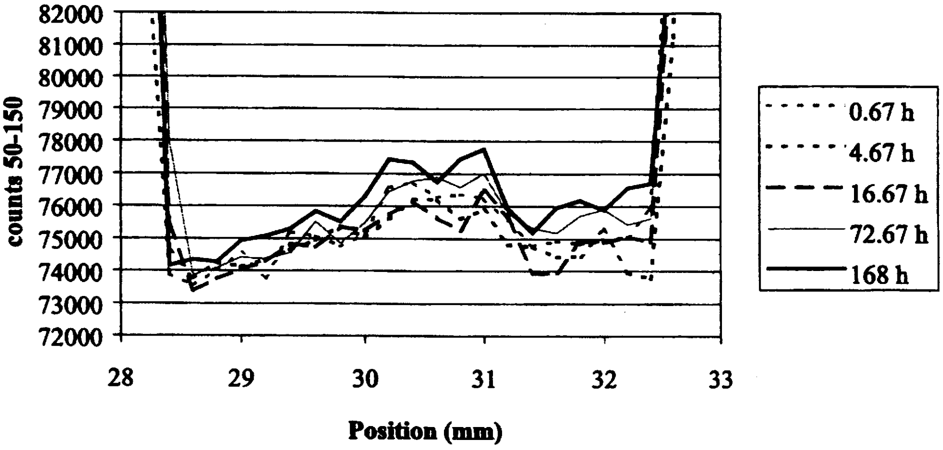

Figure 5. Results for cement paste w/c = 0.3 – capped (block 7).

Figures 5 and 6 show results for w/c = 0.3 capped and w/c = 0.45 capped specimens, respectively. Figure 5 shows 1) no settling from 0.67 hours to 4.67 hours as seen on Figure 3, and 2) a slight drying from the surface (curves widen). In Figure 6 an initial densification is seen from 0.67 hours to 4.67 hours (perhaps due to settling of the higher w/c paste and bleeding) and also a later preferential drying from the less dense surface paste. Figure 7 shows only a little water loss for the two capped pastes caused by cap leakage.

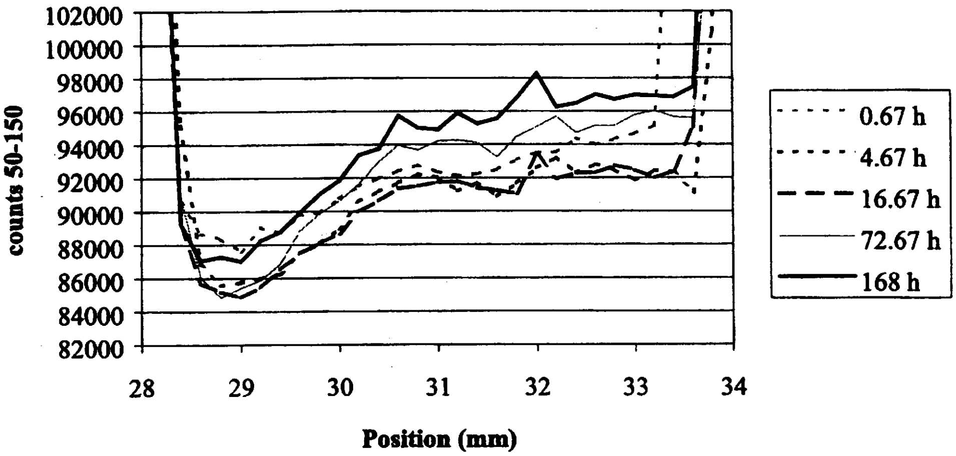

Figure 6. Results for cement paste w/c = 0.45 – capped (block 8).

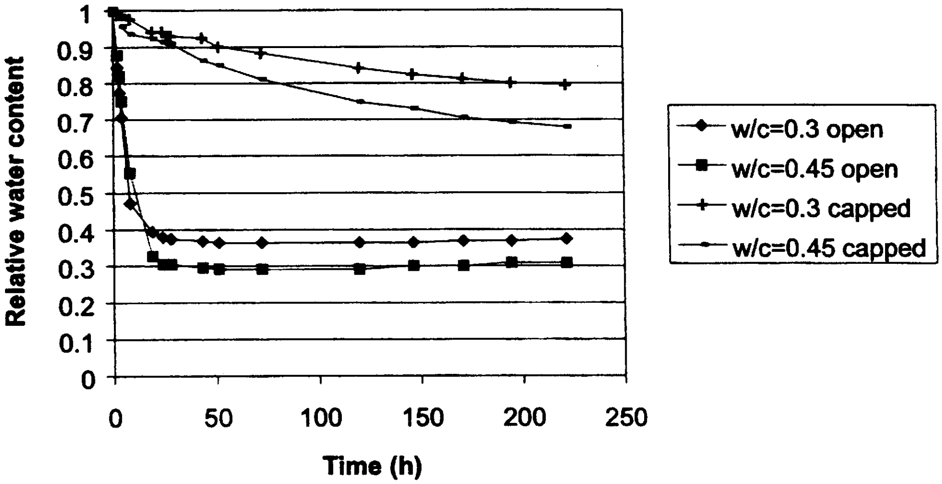

Figure 7. Relative water content vs. time for blocks with cement paste.

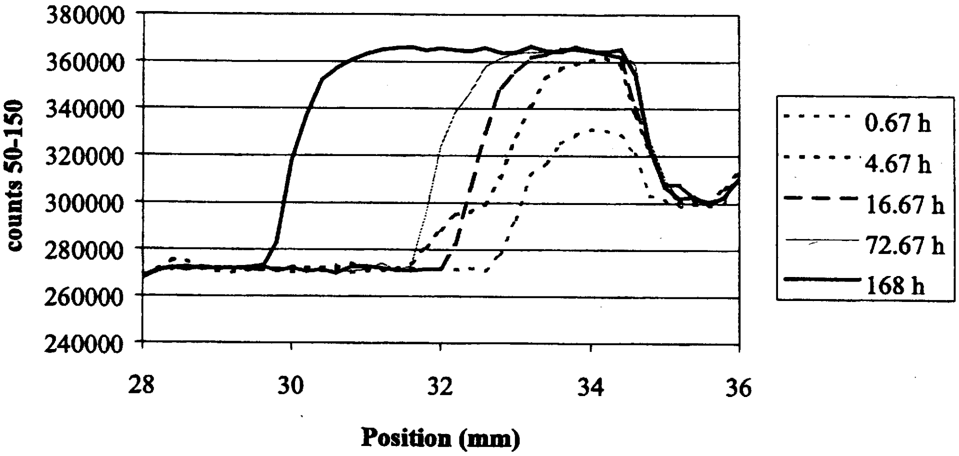

Figure 8. Results for block with water and cap (block 12).

In Figure 8, the profiles for the block filled with water and sealed with a cap are shown. In this case, the progression of a "sharp" drying front is easily observed.

5. CONCLUSION

The results from the X-ray measurements show an initial settling in the two pastes when no cap is used on top of the Lego blocks, indicating rapid water loss from the specimen, which is confirmed by weighing of the blocks. Hereafter uniform drying, as opposed to a sharp drying front, is seen.

The results from the two capped blocks show little settling and only a slight drying from the top surface, due to the much slower water loss observed in the mass measurements.

These initial experiments indicate that the X-ray environmental chamber is quite useful for studying water movement in cement-based materials and that drying profiles can be easily quantified with a sub-millimetre spatial resolution.

6. ACKNOWLEDGEMENT

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7. LITERATURE

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