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## Preliminary observations of water movement in cement pastes during curing using X-ray absorption

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### Abstract

X-ray absorption and concurrent mass measurements are used in quantifying water movement in 4 to 5 mm thick cement paste specimens with their top surface exposed to drying. Experimental variables examined in this preliminary study include water-to-cement (w/c) ratio and open vs. capped samples. Layered specimens (e.g., 0.3 w/c ratio paste over 0.45 w/c ratio paste) are also examined to monitor the preferential water movement from less dense (higher w/c ratio) paste to denser paste due to capillary forces. For the open samples examined in this study, the initial drying is observed to occur uniformly throughout the thickness of the specimen, as opposed to proceeding as a sharp front progressing inward from the surface exposed to the external environment. In the layered specimens, the higher w/c ratio paste layer is seen to “dry out” first regardless of its location within the composite. The implications of these experimental observations for curing of concrete and application of repair materials are discussed. © 2000 Elsevier Science Ltd. All rights reserved.

**Keywords:** Cement paste; Curing; Drying; Water movement; X-ray absorption

### 1. Introduction

One critical process in the production of quality concrete at the construction site is curing [1]. To achieve optimum performance, it is important to minimize the evaporation of water from the concrete surface during the first few days of hydration. Furthermore, as cement paste hydrates under sealed conditions, it undergoes self-desiccation due to chemical shrinkage (the reaction products occupying less volume than the reactants) [2,3]. This self-desiccation has been shown to be highly dependent on time, water-to-cement ratio (w/c), and silica fume content [4,5]. Thus, in high-performance, low w/c concretes, there may be insufficient internal water for complete curing, so that additional water supplied from the exterior surface could greatly enhance final properties. To better understand water movement during the curing of concrete, fundamental studies are needed to examine the spatial and temporal water distribution within the concrete element.

Recently, at the Technical University of Denmark, an X-ray environmental chamber has been constructed to examine building materials exposed to various drying environments [6]. Both relative humidity and air temperature can be conveniently varied within the chamber, which also serves as a shield from the X-ray source. In this preliminary study, the X-ray environmental chamber is used to monitor water movement during the drying of small cement paste specimens, as a first step in understanding water movement during the curing of concrete. Because the X-ray absorption is proportional to the density of the materials through which the X-rays are passing, a high w/c ratio (less dense) paste will absorb less X-rays than a lower w/c ratio paste. Similarly, a paste, which has dried out will absorb less X-rays than the same paste under its initial saturated conditions.

### 2. Experimental procedure

Details of the X-ray environmental chamber are provided in Ref. [6]. The X-ray source consists of an X-ray tube, requiring an energy level between 20 and 125 keV, with a maximum current of 0.5 mA. One of five different

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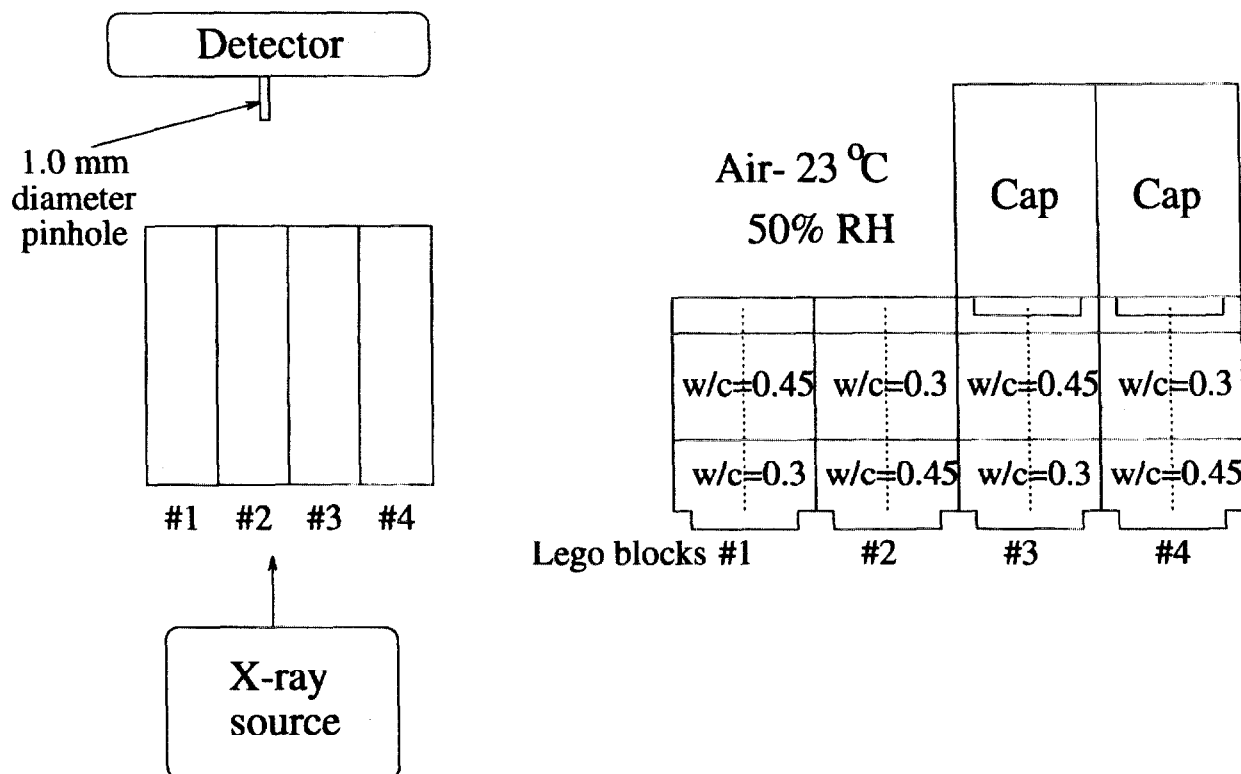


Fig. 1. Experimental setup for the layered cement pastes experiment. Image on the left shows a horizontal view of the setup and image on the right is a vertical view.

filters may be used to separate the generated X-ray beam into two energy levels, to allow for the simultaneous assessment of density and moisture content, for example. The filtered beam is collimated and the resultant 1-mm diameter beam is passed through the specimen to be evaluated. A detector, with a NaI crystal, measures the photon count for each of 256 discrete energy channels. A 1.0-mm diameter pinhole collimator is placed directly in front of the detector. The X-ray source and detector are mounted on an xyz positioning table within the environmental chamber. The resolution of the movement along each axis is  $\pm 0.1$  mm. Thus, while data can be obtained with a spatial resolution of 0.1 mm, it must be kept in mind that the “spot” size at each location is on the order of 1.0 mm. The entire system is computer controlled, so that the user may set up a grid of specimen points to be evaluated at periodic intervals.

For this preliminary study, a Type I/II ordinary Portland cement, issued as Cement 133 in June 1999 by the Cement and Concrete Reference Laboratory at NIST, was used. The cement has a potential Bogue composition of 58.6%  $C_3S$ , 14.8%  $C_2S$ , 10.6%  $C_3A$  and 7.5%  $C_4AF$ , with a Blaine fineness of about 350  $m^2/kg$ . Small (50 to 100 g) samples of cement paste were prepared in glass beakers and mixed by hand using a spatula for 2 min. Cement pastes of  $w/c = 0.3$  and 0.45 were prepared. The homogeneity of the  $w/c$  ratio throughout the specimen thickness can be determined by

observing the uniformity of the initial X-ray absorption values for the specimens.

The fresh cement pastes were placed in small inverted Lego<sup>1</sup> blocks (a common children’s toy building block), which were either left open or sealed with a second Lego block cap. The Lego blocks were chosen as sample holders due to: (1) their low absorption of X-rays, (2) their inherent stackability (allows repeatable placement of the block within the X-ray chamber), (3) the ease with which they can be sealed by adding a cap, and (4) the ease with which they can be filled with a level volume of the viscous cement paste. The basic experimental setup, illustrated for the composite (layered) cement paste specimens to be described below in the Results and Discussion section, is shown in Fig. 1.

Each block was numbered and weighed (to  $\pm 0.01$  g) before the cement paste was added. The masses of the cement paste-filled blocks were determined initially and periodically throughout the exposure period. The blocks were located sequentially on a holder (an inverted Lego base element) placed at a fixed location within the X-ray chamber. After specific periods of exposure, the X-ray system

<sup>1</sup> Certain commercial equipment is identified in this paper to specify the experimental procedure. In no case does such identification imply endorsement by the National Institute of Standards and Technology, nor does it indicate that the products are necessarily the best available for the purpose.

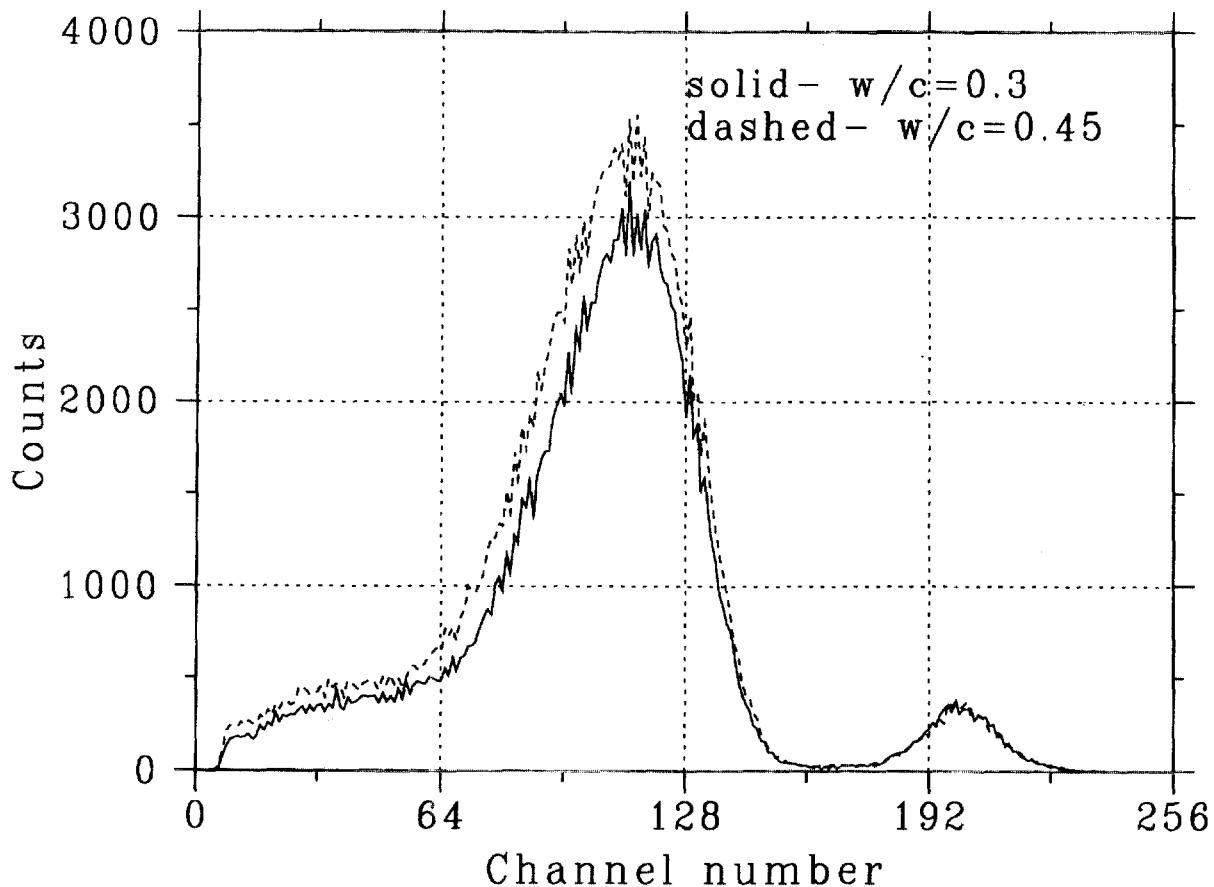


Fig. 2. Measured spectra for fresh cement pastes.

was used to scan vertically (in 0.2 mm increments) a distance of 8 mm along the central  $y$ -axis of each block. In system coordinates, the bottom of the cement paste in each block is located at a  $y$ -coordinate of 28 mm so that each scan was conducted from system  $y$ -coordinates of 28 to 36. The internal dimensions of a Lego block are approximately, length = 12.5 mm (X-ray beam direction), width = 4.7 mm, and height = 8.4 mm. Including the thickness of the side walls, the center to center distance for adjacent blocks ( $x$ -direction) is 8 mm. A 5-s count time was used at each location to improve the signal to noise ratio. Assuming a Poisson process, the relative standard uncertainty in the sum of the counts obtained for channels 50 to 150 should be on the order of 0.4% or 300 counts (for a sum of 70,000 counts, a typical value for the cement paste specimens).

Examples of the measured spectra for the two fresh cement pastes are provided in Fig. 2. The small peak at channel 200 is from an internal Cobalt source used for system calibration. Based on the pattern observed in Fig. 2, the sum of counts between channels 50 and 150 was selected as the dependent variable to be analyzed as being representative of the density (and water content) of the cement paste. This value was normalized by dividing it by the ratio of the counts achieved in free air at each measuring

time to the counts achieved in free air for the first measuring time (to account for variability in the X-ray source). In the graphs which follow, these normalized counts have been divided by 1000 producing values generally between 70 and 100. For certain cases, the data have been further processed by subtracting the values obtained at an early age (e.g., 3 to 5 h) from all subsequent measurements to obtain the differential density profile and highlight the changes due to drying. In Fig. 2, the denser  $w/c = 0.3$  cement paste is seen to absorb more of the X-rays as indicated by its lower count values at all channels between 50 and 150.

### 3. Results and discussion

#### 3.1. Experiment 1—capped and open blocks

In the first experiment, cement pastes of  $w/c = 0.3$  and 0.45 were prepared and immediately placed in their block molds. One set of blocks ( $w/c = 0.3$  and 0.45) was immediately exposed to the chamber environment of 50% RH and 23°C. Four other sets of blocks were capped (using another block) to minimize water loss, and were subjected to the following subsequent storage conditions: (1) caps were

removed after 1 day of curing, (2) caps were removed after 3 days of curing, (3) caps were maintained throughout the experiment, and (4) caps were maintained and water was periodically added to the top surface of the specimens in an attempt to maintain saturation. X-ray measurements were performed over a period of 7 days and mass measurements over a period of 10 days.

The behavior of the specimens with time can be conveniently divided into two regions, before setting and after setting. Before setting, as water is removed from the cement paste, the entire volume of cement paste shrinks, decreasing the w/c ratio of the remaining paste. This behavior can be clearly observed for both w/c ratio specimens in Fig. 3 which plots the density profiles after exposure times (time after mixing) of 0.67 and 4.67 h. The settling is indicated by the leftward shift of the sharp vertical gradient at the top of the specimen ( $y = 34$  mm), and is seen to be slightly greater for the w/c = 0.45 paste (0.4 mm) than for the w/c = 0.3 paste (0.2 mm), due to its lower solids content. The overall densification is indicated by the reduced counts (denser paste) throughout the thickness of the specimen. Once again, the densification is greater for the higher w/c ratio paste as indicated by the larger reduction in counts during the first 4-h of exposure. As shown in Fig. 4, which plots the fraction of the initial water mass remaining in the samples vs. time, during this first 4-h of exposure, the w/c = 0.3 cement paste loses about 30% of its initial water content, while the w/c = 0.45 cement paste loses

about 25% of its initial water mass (keeping in mind that the w/c = 0.45 paste has about a 20% larger initial water content than the w/c = 0.3 paste).

From the mass loss curves presented in Fig. 4, one can clearly observe that most of the drying for the open specimens occurs within the first 24-h of exposure. This is in agreement with the drying profiles for these two open specimens presented in Fig. 5, in which the measured counts for 4.67 h have been subtracted from all subsequent readings to highlight the changes due to drying. In this figure, the w/c = 0.3 paste is seen to reach its equilibrium “dryness” within 8 h of exposure, while 24 h are needed for the w/c = 0.45 paste, also in agreement with the bulk mass loss measurements. More interestingly in Fig. 5, the drying profiles are observed to be relatively uniform throughout the thickness (depth) of the specimen. This is in contrast to the inward progression of a drying front often observed in porous materials. While these specimens are only 4 to 5 mm thick, similar results have recently been obtained for cement pastes 50-mm thick using magnetic resonance imaging [7]. A relatively wide pore size distribution exists in the fresh cement paste due to the random packing of cement particles with diameters that span about two orders of magnitude. This, along with the relatively high permeability of the young paste, allows for a rapid redistribution of water due to capillary forces. Thus, rather than a sharp intruding drying front, it appears that the largest pores throughout the paste volume are first emptied, than the next largest, etc., resulting

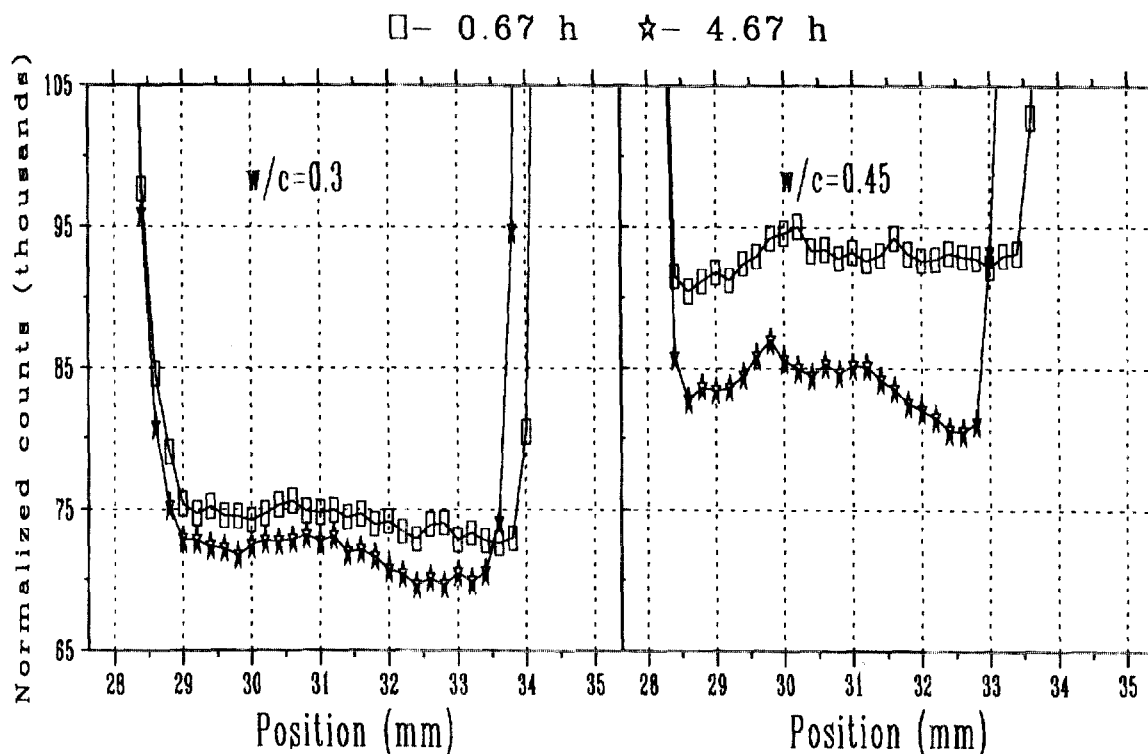


Fig. 3. Initial settling of cement pastes in open Lego blocks. The top surface of the cement paste is approximately located at a position of 34 mm and the bottom of the inside of the Lego block at 28.5 mm.

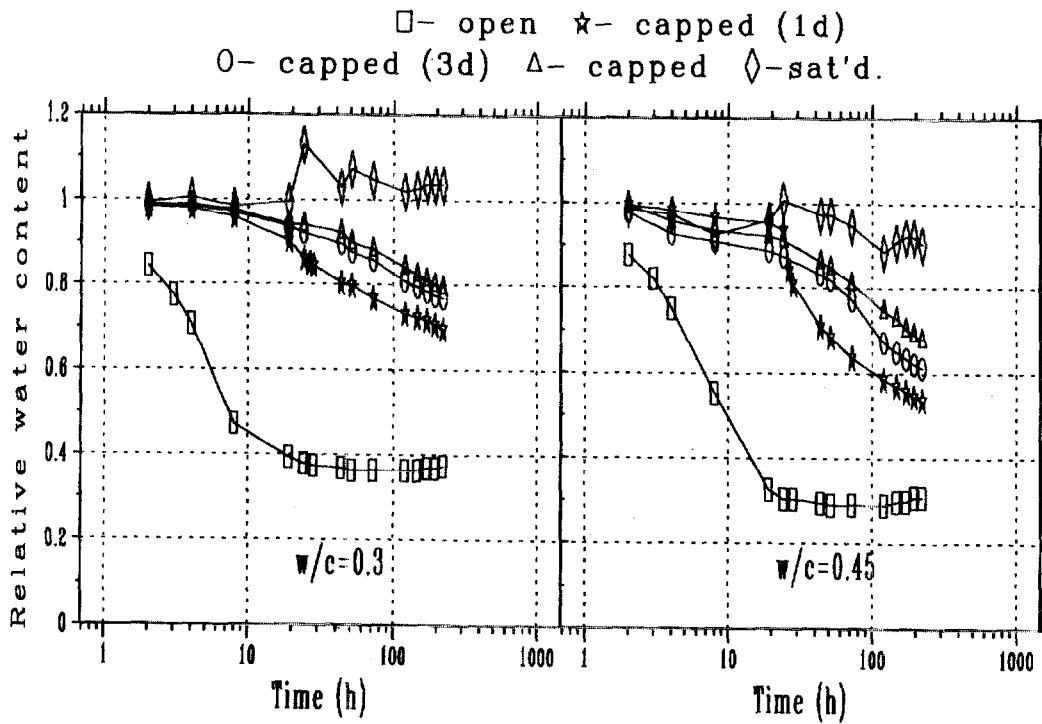


Fig. 4. Relative water content vs. time for exposed cement paste specimens.

in a fairly uniform drying throughout the specimen thickness. Even though only one surface of the specimen is

exposed to drying, pores throughout the paste volume are emptied uniformly. The scale over which this mechanism

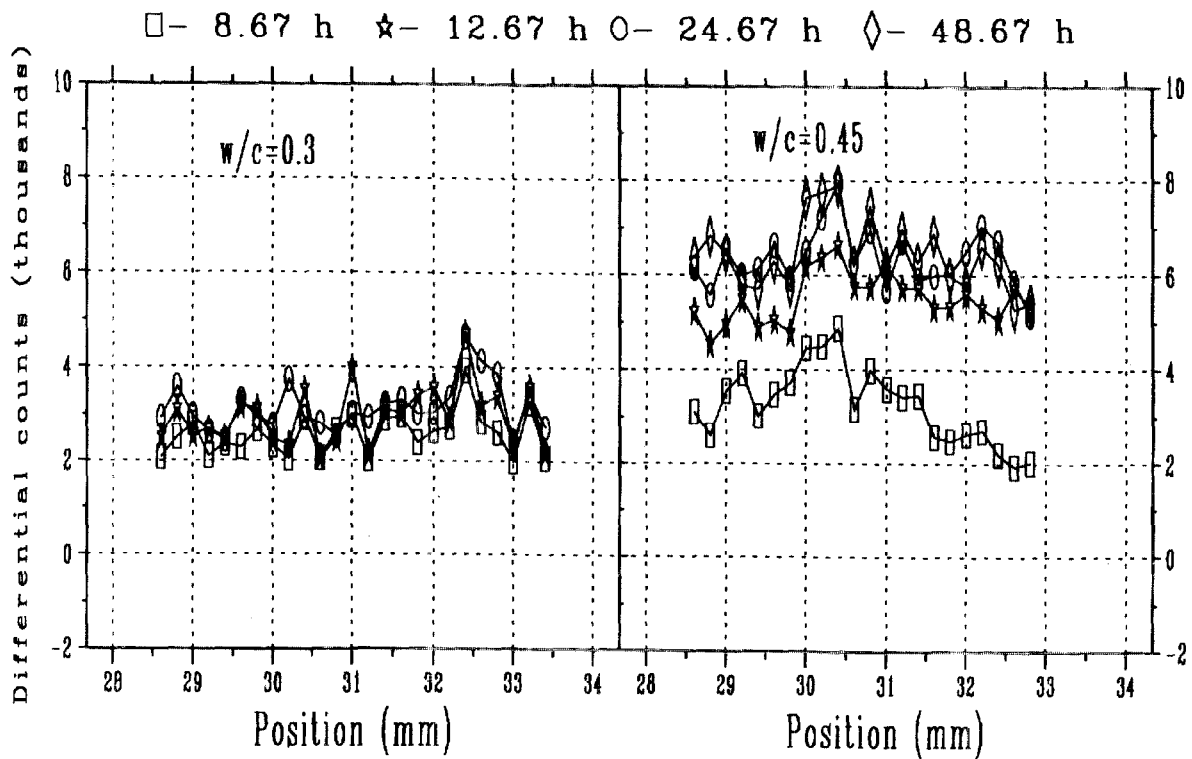


Fig. 5. Differential density profiles (relative to 4.67 h) for open block cement paste specimens.

operates in a young concrete is yet to be determined, but the results of Coussot [7] suggest that it is at least 50 mm, which is similar to the depth of the steel reinforcement in exposed concrete members.

Because the block caps did not provide a perfect seal, some mass was lost from the capped specimens during the 1st day (Fig. 4). The drying profiles provided in Fig. 6 (normalized counts) and Fig. 7 (differential counts relative to  $t = 4.67$  h) indicate that for this slower “drying” rate, the drying does proceed somewhat as a front penetrating from the top surface of the specimen, particularly for the  $w/c = 0.45$  specimen. In the differential plots, such as the ones in Fig. 7, the presence of a drying front would be indicated by a positive slope in the differential counts vs. position curve. The peak in the profiles for the  $w/c = 0.3$  specimen in Fig. 6 is most likely due to the fact that the specimens were typically placed into their molds in two layers and either some air or a higher  $w/c$  ratio paste may sometimes be present between the two layers. When the caps are removed at 24 h, the drying accelerates (increased mass loss in Fig. 4), and a drying front penetrates into the cement paste. After 24 h of hydration, the cement paste pore size distribution would be refined and its permeability reduced so that the rearrangement of water by capillary forces would be expected to be a much slower process than in the young paste.

The  $w/c = 0.45$  specimen capped for 3 days exhibits a similar behavior with the top part of the specimen drying

out before the bottom portion, as shown in Fig. 8. For the capped  $w/c = 0.45$  paste specimens, however, it is also observed (similar to the 1-day specimen shown in Fig. 6) that the paste is locally denser within the specimens than at the top surface (most likely due to settling and bleeding of the fresh paste), which could also lead to preferential drying at the surface. This point will be discussed further in Section 3.2 of the results, which deals with composite layered cement paste specimens. Another point worth noting for the capped 3-day specimens is that, after 3 days, the mass loss for the  $w/c = 0.3$  paste shown in Fig. 4 is relatively small and very close to that observed for the “sealed” specimen. Conversely, the  $w/c = 0.45$  paste loses a significant amount of water after 3 days. This result indicates that shorter curing periods (e.g., 3 days instead of 7 days) may be sufficient to minimize the water loss for lower  $w/c$  ratio concretes, as has been suggested previously [1].

The results for the specimens capped throughout the exposure period (Fig. 9) basically follow those obtained for the capped for 3 days specimens. Preferential drying at the surface is seen, particularly for the  $w/c = 0.45$  specimen. The mass loss measurements (Fig. 4) also indicate that the  $w/c = 0.45$  paste loses more mass than the  $w/c = 0.3$  one (particularly for times beyond 3 days), again supporting the proposal of reducing the required curing times for low  $w/c$  ratio concretes.

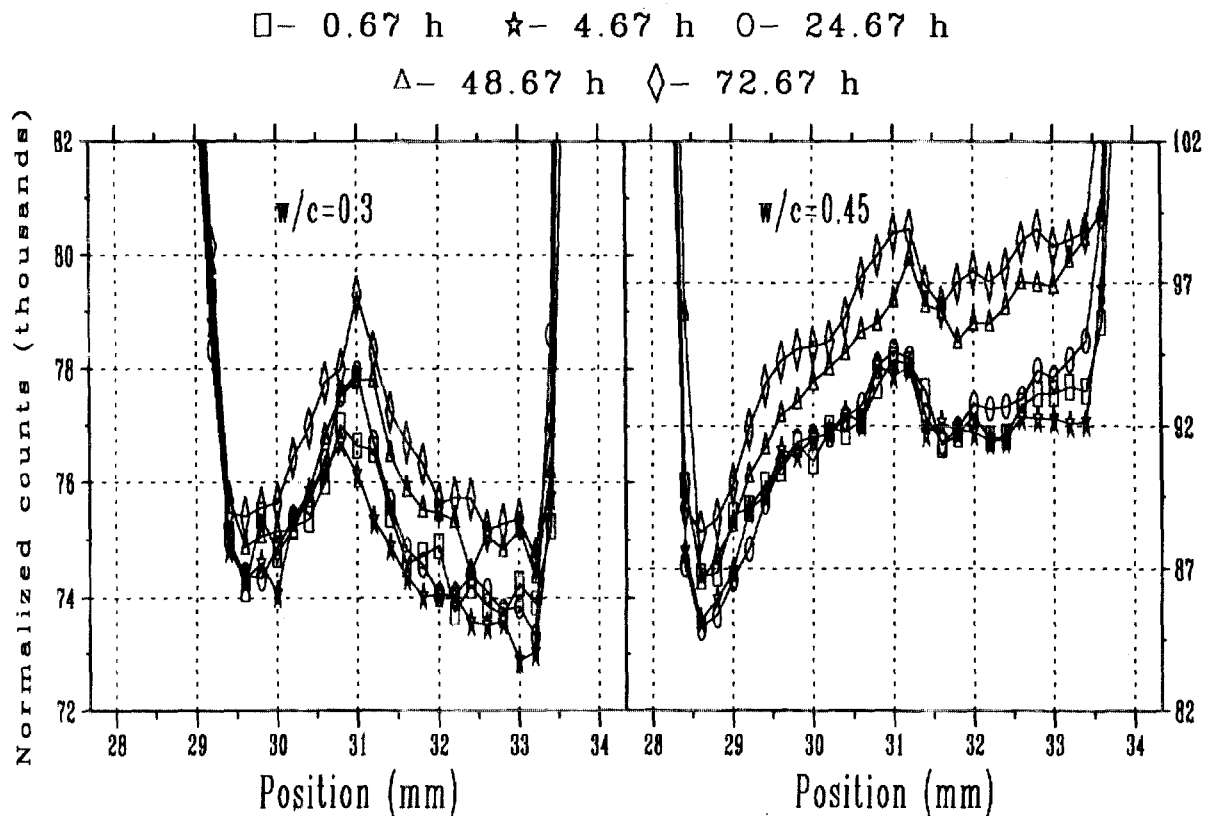


Fig. 6. Normalized density profiles for cement paste specimens capped for 1 day and then exposed to the 23°C, 50% RH environment.

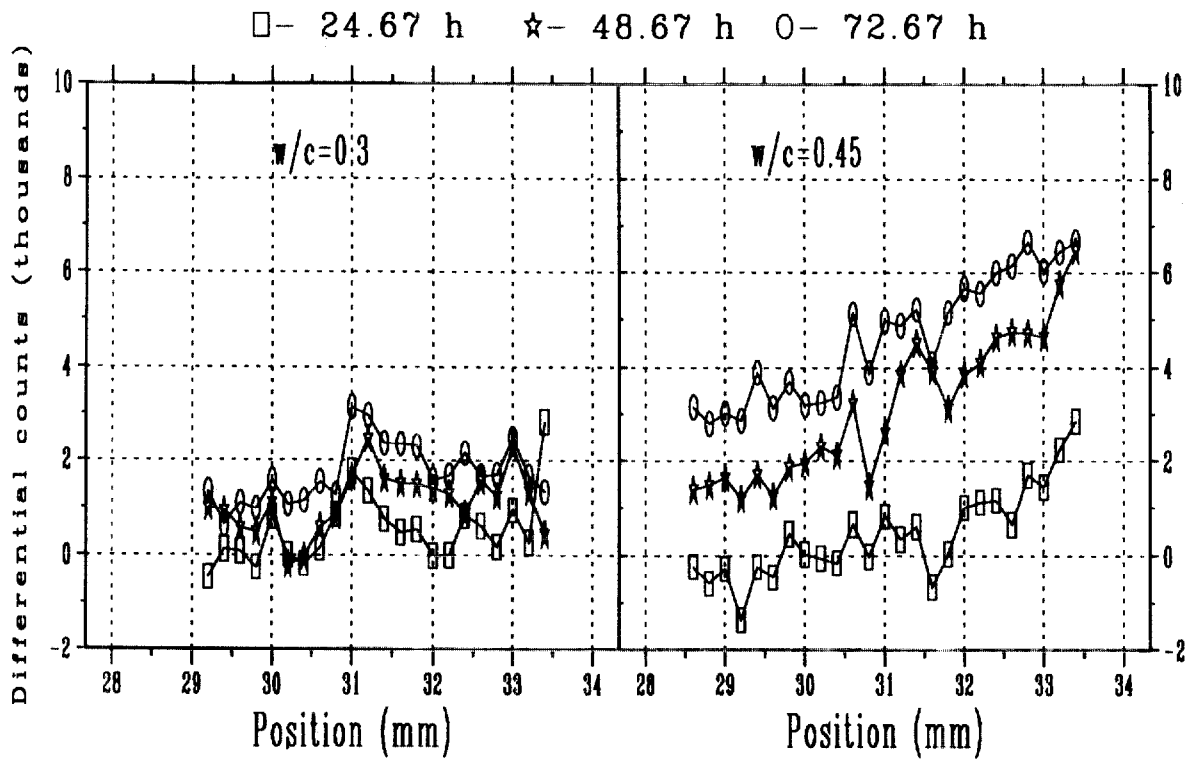


Fig. 7. Differential density profiles (relative to 4.67 h) for cement paste specimens capped for 1 day and then exposed to the 23°C, 50% RH environment.

The results for the “saturated” specimens are provided in Fig. 10. In this case, the  $w/c = 0.45$  paste lost a little mass

over the duration of the exposure, while the  $w/c = 0.3$  paste actually gained slightly, due to water imbibition from the top

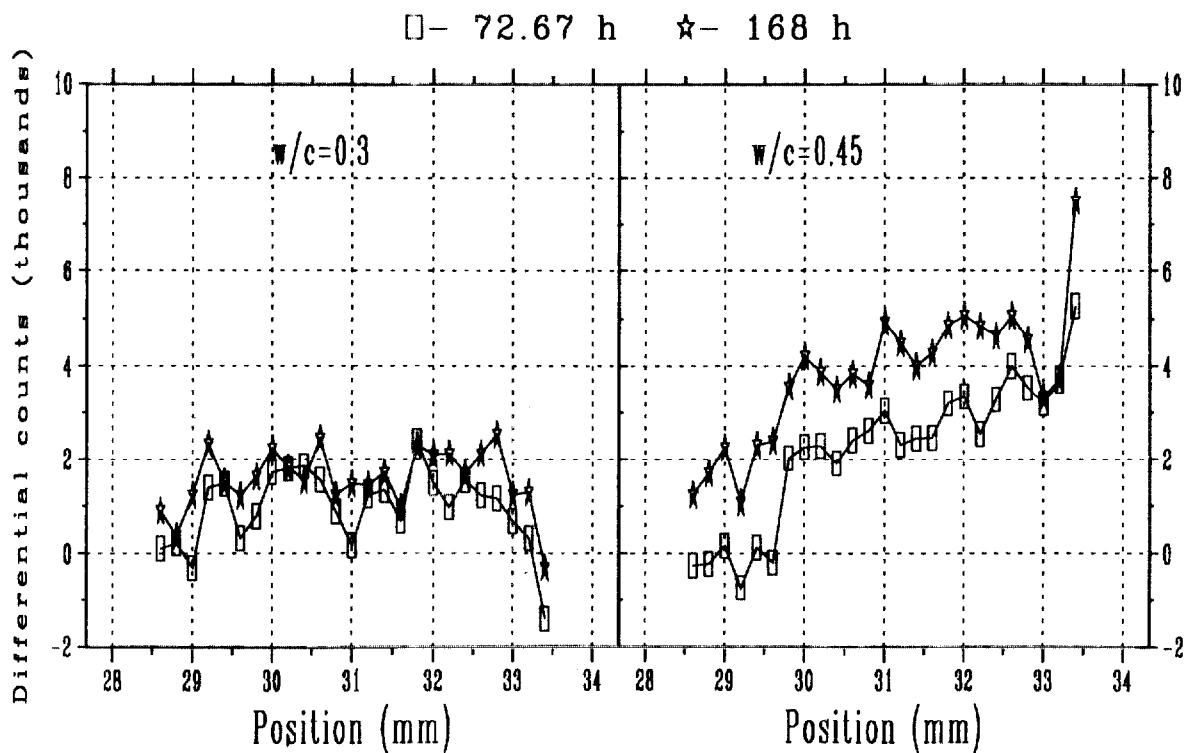


Fig. 8. Differential density profiles (relative to 4.67 h) for cement paste specimens capped for 3 days.



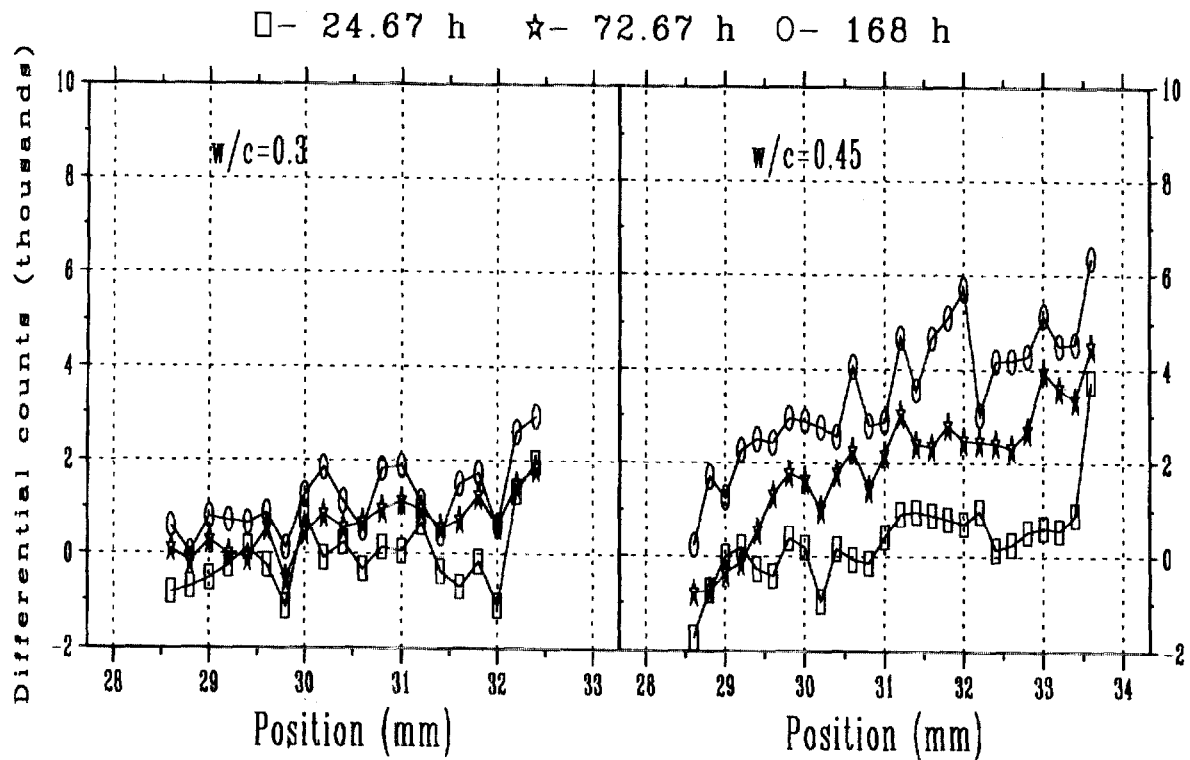


Fig. 9. Differential density profiles (relative to 4.67 h) for cement paste specimens capped throughout the exposure.

surface to replace that consumed by chemical shrinkage during hydration. Thus, the drying profiles are nearly time-

invariant, with only a suggestion of the water imbibition (densification) at intermediate times. Consistent with the

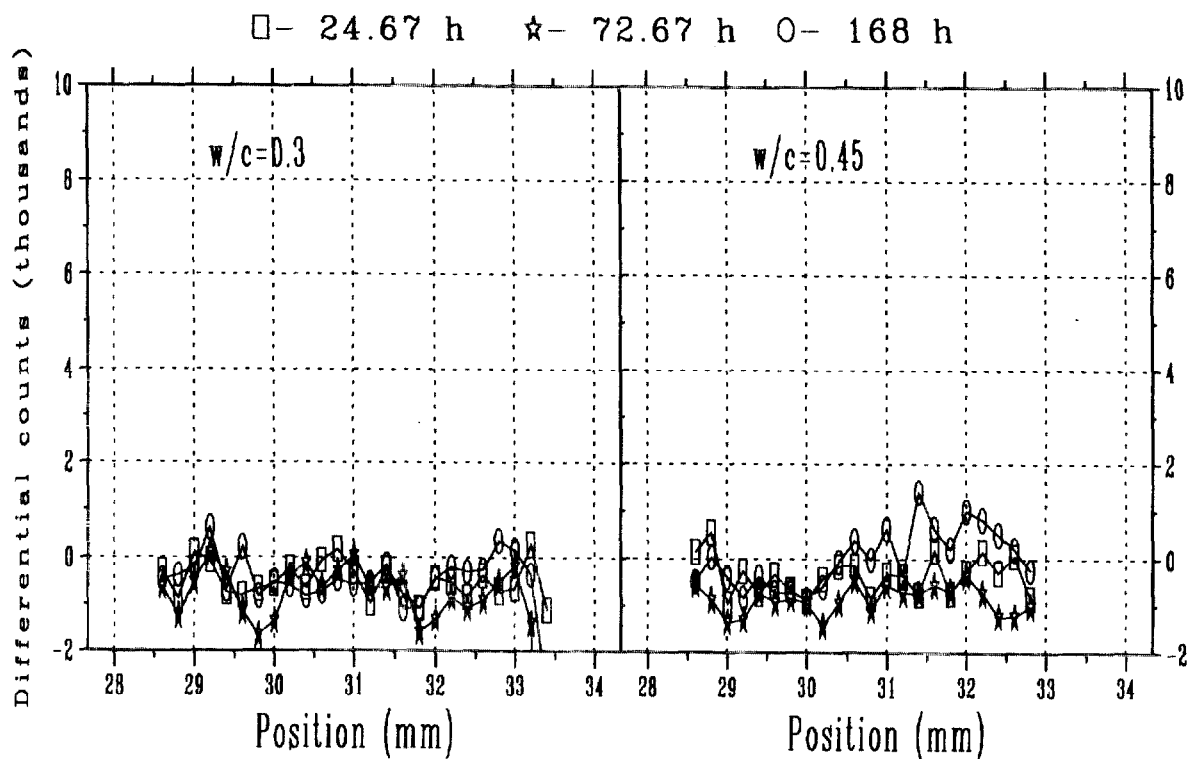


Fig. 10. Differential density profiles (relative to 4.67 h) for cement paste specimens "saturated" throughout the exposure. A negative value indicates water imbibition by the hydrating cement paste.

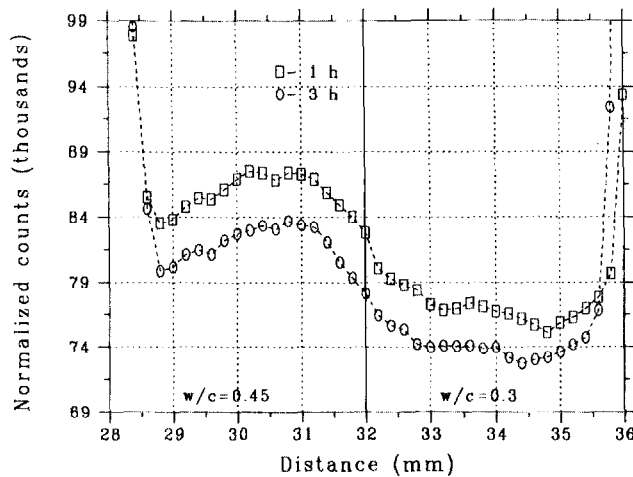


Fig. 11. Normalized density profiles for layered cement paste (0.3 over 0.45) open to the chamber environment.

mass readings presented in Fig. 4, the water imbibition appears to be maximal at about 72 h, with some minor drying of the specimens thereafter, due to the inefficient seal of the block caps.

### 3.2. Experiment 2—layered cement pastes

The first set of experiments, along with the results presented in the literature for the drying of layered sintered glass bead specimens [8], suggested that it would be interesting to investigate layered cement paste composite specimens using the X-ray equipment. Thus, in the second set of experiments, composite specimens were prepared by layering the two cement pastes, using a small metal spatula to place the pastes in layers into their molds. In total, four specimens were prepared, covering both possibilities for paste order (0.3 on top of 0.45 and 0.45 on top of 0.3), for both open and capped blocks (see Fig. 1). An attempt was

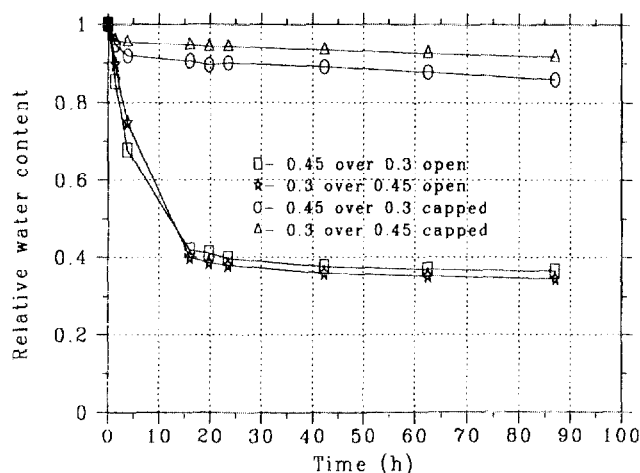


Fig. 12. Relative water content vs. time for exposed layered cement paste specimens.

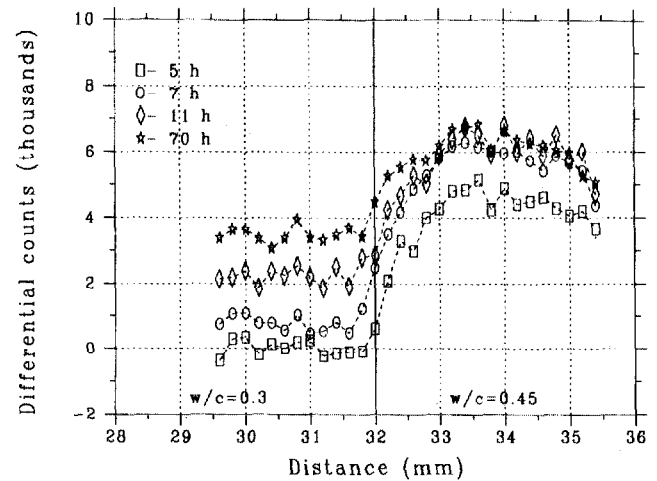


Fig. 13. Differential density profiles (relative to 3 h) for layered cement paste (0.45 over 0.3) open to the chamber environment.

made to have equal heights of each w/c ratio paste in each block (about 3 mm), with varying degrees of success. Additionally, to better minimize water loss in the sealed specimens, custom-built teflon caps (as opposed to Legos) were used to cap the blocks containing the cement pastes.

As was the case for the single layer specimens, an initial settling of the cement pastes was observed for the composite specimens (Fig. 11) immediately exposed to the drying environment, and the observed mass losses for the two layers in total (Fig. 12) were similar to those measured for the single layer specimens. The new caps were observed to reduce the water loss to about 50% of that observed when using Lego blocks as caps.

The measured differential profiles (relative to normalized counts at 3 h) for the open specimen with the w/c = 0.45 cement paste exposed to the drying surface (top) are provided in Fig. 13. During the first 7-h of exposure, the w/c = 0.45 paste dries out in a relatively uniform manner,

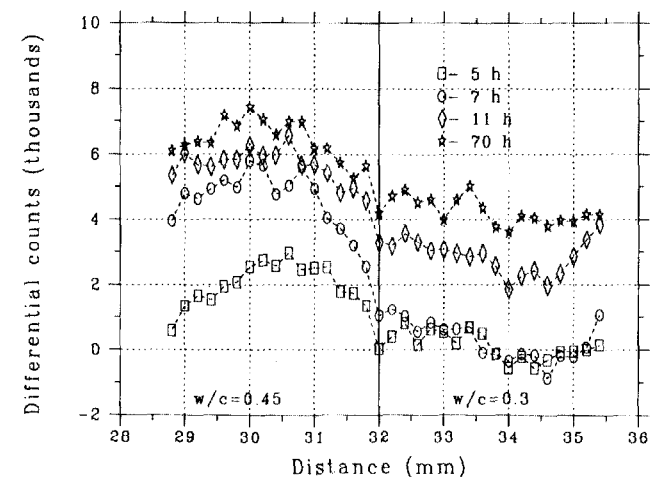


Fig. 14. Differential density profiles (relative to 3 h) for layered cement paste (0.3 over 0.45) open to the chamber environment.

while the  $w/c = 0.3$  paste remains basically saturated. Beyond 7 h, water is removed from the  $w/c = 0.3$  paste as well and little further drying is observed in the  $w/c = 0.45$  cement paste, most likely due to “complete” drying of this layer of the paste. More interesting, perhaps, are the results presented in Fig. 14 for the case where the denser  $w/c = 0.3$  paste occupies the top half of the specimen and is directly exposed to the drying environment. Rather than drying out immediately, the  $w/c = 0.3$  paste, instead, first draws the water from the  $w/c = 0.45$  paste below it (between 3 and 7 h), only drying out itself after this water supply is depleted. This water rearrangement is obviously due to the different pore size distributions within the two pastes and the resultant differences in capillary pressure. Coussot et al. [8] have observed similar results for layered bead packs composed of layers of beads of different sizes but with equivalent porosities in each layer. In our case, the differences in pore size distribution and capillary forces are not generated by different size particles, but rather by the difference in  $w/c$  ratio or water-filled porosity within the two layers (i.e., the  $w/c = 0.3$  ratio cement paste contains finer pores than the  $w/c = 0.45$  one).

This rearrangement of capillary water is even observed in a sealed specimen which loses very little capillary water, as shown in Fig. 15. Here, the more dense paste at the bottom of the specimen is seen to further increase in density (negative values on the differential density plot) at the expense of the less dense paste in the top half of the specimen. In this case, a substantial movement of water is observed to occur between 34 and 46 h as the finer pore structure of the  $w/c = 0.3$  paste imbibes water from the  $w/c = 0.45$  paste to replace that “lost” due to chemical shrinkage.

This preferential movement of water from regions of high porosity (high  $w/c$  ratio) to regions of low porosity has several implications for concrete in practice. One example would be the effects of self-desiccation on the microstructure of the interfacial transition zone (ITZ) regions in a low

$w/c$  ratio concrete cured under sealed conditions. The ITZ regions within 20 to 30  $\mu\text{m}$  of an aggregate surface are typically characterized by a higher  $w/c$  ratio (less cement) than the bulk paste. As empty porosity is created due to the chemical shrinkage occurring during hydration, water will be drawn out of these more porous (higher  $w/c$  ratio) ITZ regions into the denser bulk paste, further preventing strength development within the ITZ regions. Two different computer models for cement hydration and microstructure development [9,10] have been applied to this scenario and both have indicated the creation of large empty pores within the ITZ regions. Another scenario where this water movement would be detrimental to high-performance concretes would be its effects on flaws and inhomogeneities in  $w/c$  ratio within the concrete microstructure. As water is removed due to drying, or empty porosity is created due to self-desiccation, any regions of locally higher  $w/c$  ratio (flaws) will be the first to empty, leaving behind a large empty pore which could be a very detrimental flaw from a strength perspective. All of this highlights the importance of proper consolidation (to avoid inhomogeneities in  $w/c$  ratio) and curing for high-performance concretes where typically there is insufficient initial water present for complete hydration of the cement. In these water deficient mixtures, any further loss of water to the environment will result in the production of large empty pores, which could substantially decrease both the strength and the durability of the finished product.

These results also suggest that care must be taken in selecting the curing system to use for high-performance concretes. At first look, a curing sheet consisting of a very fine porous material might seem like a good idea as the material would remain saturated at relative humidities well below 100%. However, if water loss does occur in this scenario, the water in the curing layer will be “replaced” by water from within the concrete, due to the capillary forces within the curing material being greater than those within the concrete. To the casual observer at the field site, everything might appear to be fine, as the curing layer would continue to appear moist. This, despite the fact that it is drawing needed curing water out of the concrete. The ideal curing system must prevent water loss from the concrete and if possible, even supply an external source of water to be absorbed by the hydrating cement paste. An example of this would be the use of controlled permeability formwork [11]. In this system, a porous material (with a watertight back surface) initially draws water from the cement paste prior to setting, densifying the top layer of the concrete by reducing its  $w/c$  ratio. Then, as the cement hydrates and the pores in the cement paste become finer than those in the formwork, this now external supply of water will be imbibed back into the cement paste to replace water “lost” due to chemical shrinkage. The performance of these systems in practice has been quite good, with the resultant concretes exhibiting superior properties in terms of strength and durability [11]. Another example where the

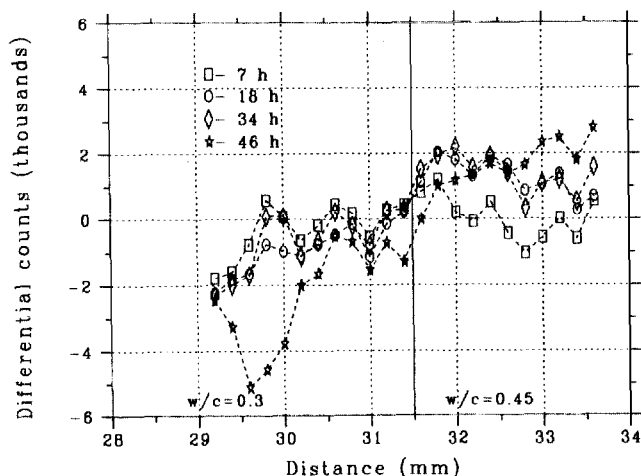


Fig. 15. Differential density profiles (relative to 3 h) for layered cement paste (0.45 over 0.3) sealed with a cap.

same considerations of pore size and capillary forces have been exploited would be the recent use of saturated lightweight aggregates to provide internal curing for high-performance concretes [12,13].

Another area where water movement in “fresh” cement-based materials is critical to performance is that of repair materials. The difference in saturation between the existing substrate and the repair material, and the exposure of the repair material to a drying environment, relate directly to the observations of water movement obtained in this study. For example, if the existing material is dry and has a relatively fine pore structure, it will draw water out of the repair material, leading to insufficient reaction and strength development (and perhaps, the cohesive failure of the repair system). Conversely, the capillary forces pulling the water from the repair material to the existing material could improve the adhesion at the interface between the two systems. At the other extreme, if the existing material is saturated and contains some coarse pores relative to the repair material, as water dries out of the repair material into the external environment, it will be replaced by water from the existing material. Once again, the capillary forces active in this process could also increase the bond between the repair material and the existing material.

#### 4. Conclusions

New equipment for monitoring water movement in cement-based materials with sub-millimeter resolution has been demonstrated. The initial experiments presented in this paper suggest that:

1. because of the rapid rearrangement of water due to capillary forces, small specimens of fresh cement paste dry out in a relatively uniform fashion as opposed to exhibiting the sharp drying front commonly observed in porous materials,
2. the  $w/c = 0.45$  cement pastes cured for several days or exposed to a milder drying environment do exhibit a somewhat sharp drying front (perhaps due to the density gradient established by the initial settling of the paste), and
3. in layered cement paste composites exposed to drying, water preferentially moves from the less dense to the more dense paste; this mechanism is also observed in sealed specimens as empty internal porosity (self-desiccation) is created due to chemical shrinkage.

The implications of these observations for the field curing of concrete have been discussed. At early ages, the movement of water from less dense paste regions to regions of higher density could produce or intensify local flaws. The ITZ regions in a concrete are one naturally occurring area of higher  $w/c$  ratio that could contain large empty pores due to

self-desiccation and water rearrangement. Proper consideration of pore structure and capillary forces can lead to the optimization of the curing of high-performance concrete. Controlled permeability formwork and saturated lightweight aggregates are two practical examples of the application of these principles.

The results also have implications for the application of repair materials to existing concrete. If the concrete to be repaired is relatively dry and contains a fine pore structure, it may “draw” out the needed curing water from the repair material, leading to insufficient strength development and cohesive failure of the latter. Conversely, a repair material with a fine pore structure may be able to draw needed curing water from the saturated original material to offset any water lost to the external environment.

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