Development of a new experimental technique for the study of the autogenous shrinkage of cement paste

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ABSTRACT

After defining and describing the main physico-chemical mechanisms governing the autogenous shrinkage of concrete, a number of different experimental approaches used to asses this phenomenon are discussed. The base principle and a detail description of a new experimental technique aiming to completely characterise the autogenous shrinkage of a cement paste are presented. The originality of the technique comes from the fact that the variation of the external volume (external autogenous shrinkage) and the volume of the gas-filled spaces in the liquid phase after the hardening (internal autogenous shrinkage) are measured separately and simultaneously. The total autogenous shrinkage is the sum of the internal and external autogenous shrinkages. A number of experimental results are presented to demonstrate the validity of the proposed experimental approach and to analyse the effect of water/cement ratio on the development of internal and external shrinkages.

RÉSUMÉ

Après avoir défini et décrit les principaux mécanismes physico-chimiques à l'origine du retrait endogène du béton, on discute des différentes approches expérimentales ayant déjà été utilisées pour étudier ce phénomène. On présente ensuite le principe de base et une description détaillée d'une nouvelle technique expérimentale permettant la caractérisation complète du retrait endogène des pâtes de ciment. L'originalité de la technique repose sur le fait qu'on mesure séparément et simultanément la variation du volume externe (retrait endogène externe) et le volume des vides gazeux créés dans la phase liquide après le durcissement (retrait endogène interne). Le retrait endogène total correspond à la somme des retraits endogène externe et interne. Quelques résultats expérimentaux sont présentés pour démontrer la validité de l'approche expérimentale proposée et pour illustrer l'influence du rapport eau/ciment sur le développement des retraits endogènes externes et internes.

1. INTRODUCTION

Autogenous shrinkage is an important property of the hardening cement paste that may have many consequences on the physicochemical properties and durability of concrete [1, 2]. Through his research on the chemical shrinkage of cement paste in 1900, Le Châtelier became the first person to describe and quantify the *Le Châtelier contraction*, which is one of the fundamental mechanisms governing the development of the autogenous shrinkage of cement paste [3]. However, until the development of the first high-strength concretes, relatively little attention was focused on this phenomenon. In the early eighties, a number of researchers linked the cracking tendency of some low water/cement ratio con-

cretes to significantly higher autogenous shrinkage [4, 5]. Since then, various experimental techniques have been proposed to study the fundamental mechanism causing autogenous shrinkage and to evaluate the effect of different mixture design parameters such as water/binder ratio, silica fume and, more recently, shrinkage reducing admixtures.

The measurement of autogenous shrinkage is quite complex, since the underlying mechanisms come into play immediately after the initiation of cement hydration. Consequently, assessing total autogenous shrinkage involves measurement of volume change of a paste sample during the plastic and hardened states. Moreover, complete assessment of total autogenous shrinkage must take in the measurement of external volume variations of

a sample as well as that of the gas-filled spaces created in the liquid phase resulting from hardening.

This paper describes a new experimental approach that is currently being developed at the University of Sherbrooke. The proposed technique aims at assessing the total autogenous shrinkage of paste samples in sealed curing conditions. The technique is original in that total autogenous shrinkage is calculated from the variation in the sample's external volume and the volume of gas-filled spaces created in the liquid phase during hydration.

2. THE FUNDAMENTALS OF AUTOGENOUS SHRINKAGE

The volume of the cement hydration products is lower than the sum of the volumes of the reacted water and cement. This negative volumetric balance is known as the Le Châtelier contraction or the chemical shrinkage [6, 7]. Chemical shrinkage is an intrinsic property of Portland cement, corresponding to approximately 6 ml per 100 g of reacted cement, or approximately 9% of the volume of the hydration products [2, 8]. While the chemical shrinkage of cement varies slightly depending on chemical composition, its intensity is independent of the water/cement ratio or the initial dosages of water and cement [11].

Before it sets, hydrating cement paste is in a plastic state during which its shape can be readily modified. While the paste is plastic (under sealed conditions), chemical shrinkage decreases the apparent external volume of the paste, which contributes to what is termed external autogenous shrinkage. Once setting has initiated, the paste gradually loses its deformability. A rigid mineral skeleton forms, which impedes external deformation of the paste. Therefore, the overall volume decrease due to chemical shrinkage after setting results in decreased external volume and the formation of gas-filled spaces within the initially water-filled capillary pore network [7]. Internal autogenous shrinkage is defined as the total volume of these gas-filled spaces. The total autogenous shrinkage is the sum of the external and internal autogenous shrinkage.

The gas-filled spaces are in equilibrium with the liquid phase by the action of the meniscus formed at the gas/liquid interfaces. The equilibrium is governed by Kelvin and Laplace equations, which show that the liquid phase is under depression and that the magnitude of the depression increases with the curvature of the meniscus [7, 9]. As hydration proceeds, the capillary porosity is refined and the total volume of the gas-filled spaces increases. These changes yield an increase of the curvature of the meniscus and, consequently, a higher depression in the capillary water (Fig. 1). According to the Kelvin equation, the presence of water meniscus in the capillary porosity decreases the internal relative humidity in the capillary porosity. This phenomenon is defined as self-desiccation.

The depression in the liquid phase created by chemical shrinkage after setting induces an overall compression

of the solid phase (under sealed conditions). The compressive stress is relatively low with normal pastes (w/c ≥ 0.4), since the coarser porosity limits the curvature of the meniscus and the magnitude of the capillary water depression. However, in lower w/c pastes, the finer porosity forces the formation of more curved menisci so the compressive stress generated in the solid phase can be high enough to significantly decrease paste external volume. In this case, external autogenous shrinkage is governed both by chemical shrinkage and the physical action of the meniscus in the capillary pore network.

To summarize, before a paste sample under sealed conditions sets, there is only external autogenous shrinkage, which is proportional to the amount of reacted cement (chemical shrinkage). After setting, there is external and internal autogenous shrinkage. External autogenous shrinkage is due to the simultaneous effects of the chemical shrinkage and the physical action of the meniscus in the capillary pore network. Internal autogenous shrinkage is the total volume of the gas-filled spaces created in the capillary pore network. The total autogenous shrinkage is the sum of the internal shrinkage and the external autogenous shrinkage (before and after setting).

3. SOME EXPERIMENTAL APPROACHES IN MEASURING AUTOGENOUS VOLUME CHANGES

3.1 Total autogenous shrinkage

Two basic approaches have been used to measure the total autogenous shrinkage of cement paste. The dilatometric approach consists in measuring the drop of a water meniscus in a graduated capillary tube mounted on the top of an Erlenmeyer flask containing diluted cement paste [2, 10]. The total autogenous shrinkage (ml/g of cement) is obtained by dividing the volume variation by the mass of cement in the solution. The method is valid for diluted cement paste, since the permeability of the mineral skeleton must always remain high enough to allow water penetration and saturation of all the capillary pores.
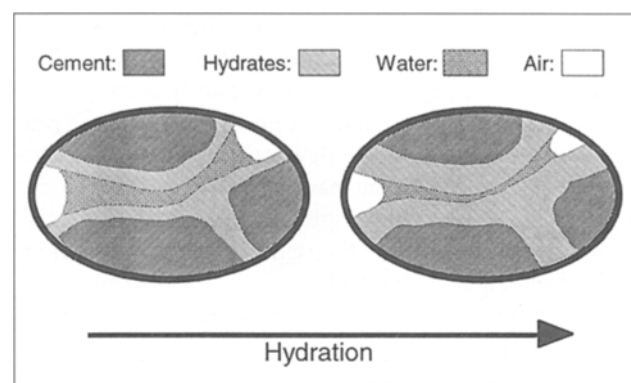

Fig. 1 – Schematic representation of the evolution of the hydration of Portland cement.

The gravimetric approach gives an indirect assessment of the total autogenous shrinkage based on the measurement of the volume variation of a condom filled with cement paste and immersed in water [2]. The external volume change of the paste is obtained from buoyancy variations of the immersed condom. The w/c ratio of the paste can be accurately selected with this method. However, assessment of chemical shrinkage with low w/c pastes (< 0.45) may be considerably underestimated because the rapid matrix solidification inhibits external volume variations and leads to the formation of gas-filled spaces in the capillary pore network, which cannot be accounted for by the measurement.

3.2 External autogenous shrinkage

The external autogenous shrinkage of paste, mortar, and concrete samples has been assessed with a number of different experimental approaches. One consists in evaluating linear shrinkage by monitoring length changes between a pair of pointers inserted along a prismatic sample [2, 11, 12]. This approach can significantly underestimate the external autogenous shrinkage, since it is difficult to accurately measure length changes prior to setting and because the friction with the mold may impede length variation, particularly during the period immediately after the setting of the sample.
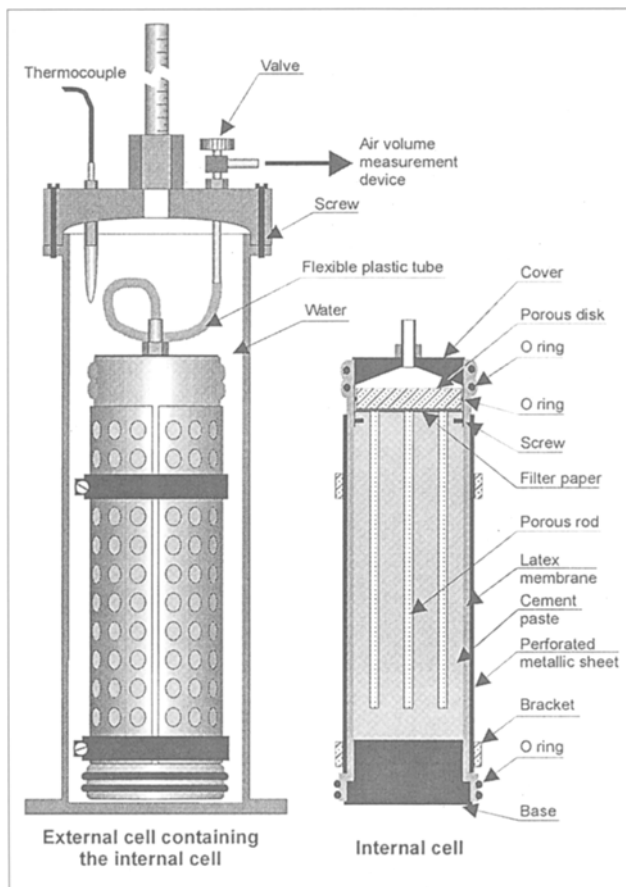

Fig. 2 – Schematic representation of the dual-compartment cell (external and internal cells) developped for the study of the autogenous shrinkage of cement paste.

External autogenous shrinkage can also be evaluated with a condom filled with fresh cement paste using a procedure very similar to that used for measuring total autogenous shrinkage. It typically applies to assessing the external autogenous shrinkage of relatively low w/c ratio pastes [2]. The main drawback of this technique is that bleeding and entrapped air in the condom may yield to misevaluation of external shrinkage [2, 13]. The pressure applied by the external membrane may also damage the delicate structure of the paste as setting begins.

Jensen and Hansen have combined these two approaches to monitoring the development of autogenous external shrinkage of paste samples [13]. Their procedure uses a rigid frame that encloses a soft plastic tube with a bellows shape. The tube is completely filled with cement paste and then sealed to prevent any loss of water during hydration. One end of the tube is attached to the rigid frame; the other can move freely in response to autogenous shrinkage. The external linear autogenous shrinkage is directly measured by a transducer that monitors changes in tube length. This procedure offers the advantage of taking into account most of the autogenous external shrinkage that occurs while the paste is still fluid. Nevertheless, it only assesses linear external autogenous shrinkage, which is not necessarily representative of the volumetric external autogenous shrinkage.

4. THE PROPOSED TECHNIQUE

The proposed technique is based on a dual-compartment cell that was specifically designed to simultaneously monitor the external and internal autogenous shrinkage of a cement paste under sealed conditions. The technique aims at accurately measuring external autogenous shrinkage by starting the measurement less than 30 minutes after initial water-cement contact. The cell was also designed to prevent overestimation of external autogenous shrinkage due to bleeding. The originality of the cell comes from the fact that internal autogenous shrinkage is obtained by measuring the volume of air that is sucked into the paste as a consequence of meniscus formation. It was decided to use air, instead of water, because it does not react significantly with cement and because the relatively low viscosity facilitates its penetration throughout most of the capillary pores in the paste sample.

4.1 Cell construction

The experimental procedure is based on a dual-compartment cell consisting of an external and an internal cell (Fig. 2). The internal cell is designed to contain approximately 800 ml of fresh cement paste. The internal cell consists of a PVC cylindrical base and a PVC cover joined by a flexible cylindrical latex membrane (length: 305 mm; diameter: 76 mm; thickness: 0.64 mm). The latex membrane is firmly held to the base and to the cover by two pairs of stretched O-rings to avoid

leaks and to form a flexible container. The cylindrical shape of the internal cell is maintained by a perforated metal sheet that is firmly fastened around the base with a metal bracket. A second loose bracket is used in the upper half of the cell just to maintain the cylindrical shape of the metallic cage. The internal cell assembly is designed to allow free radial and longitudinal deformations of the container formed by the latex membrane, the base and the cover.

The cover of the internal cell supports a porous plastic disk holding five square rods (6.4 × 6.4 × 185 mm) cut from a sheet of sintered microporous plastic (pore diameter: 70 µm; porosity: 45%). The cover also has three thin curved plates; each equipped with one little screw. The screws are used to anchor the cover to the paste sample after hardening. A thin filter paper is inserted between the paste sample and the porous disk to prevent the paste from penetrating the porous disk and to allow reuse of the porous disk.

The external cell is designed to contain the internal cell. The remaining space is completely filled with water throughout the graduated pipette connected to the top of the cell. The external cell is made of a 100 × 355-mm rigid copper tube with a wall 2 mm thick. The base of the tube is welded to a metal plate and the top supports a rigid brass cover fastened with six screws. The internal shape of the cover has a hemispherical shape to facilitate the evacuation of air bubbles into the vertical pipette. The cover holds a conical metallic tube designed to accommodate a thermocouple for monitoring the temperature of the water in the cell.

The internal cell is connected to an air volume measurement device by a flexible plastic tube passing throughout the external cell cover. The plastic tube can be easily bend but it is rigid enough to avoid external volume change due to internal pressure variations.

During the measurement, the external cell, containing the internal cell, is stored vertically into a 20-litre water tank. The tank is filled with water until the level reaches the top of the external cell cover. The water temperature is maintained at 22°C ± 2°C.

The external autogenous shrinkage corresponds to the external volume variation of the internal cell containing the cement paste. The external volume variation of the internal cell is simply obtained by recording the level variation of the water meniscus in the graduated pipette. The three anchor screws in the internal cell cover and the five porous rods stabilize the internal cell cover and prevent overestimation of external autogenous shrinkage due to the possible reabsorption of bleed water after the paste sets [2, 13].

The internal autogenous shrinkage is obtained by measuring the air volume that is sucked into the sample after the hardening of the paste. This volume is assumed to correspond to the volume of gas spaces created in the capillary porosity. The purpose of the 5 porous plastic rods is to facilitate the air penetration throughout the entire volume of the paste sample. The air-volume measuring device is a simple apparatus based on the measurement of the pressure variation of a controlled vol-

ume. It gives the total air volume required to constantly equilibrate the internal gas pressure in the capillary pores and the external atmospheric pressure.

4.2 Cell operation

The process starts by assembling the base of the internal cell, the latex membrane, and the perforated metal sheet with the brackets and one pair of O-rings. After weighing the empty cell, the freshly mixed cement paste is poured in to within approximately 5 mm from the top of the external metal sheet. The cell, filled with the paste, is weighed again to precisely determine the mass of the paste used for the test. The cover, holding the porous disk and the 5 porous rods is inserted from the top until the porous disk gently touch the surface of the fresh cement paste. The cell is finally sealed by firmly squeezing the latex membrane around the cover with a pair of O-rings.

The internal cell is carefully inserted into the external cell and the flexible tube is connected to the two covers before sealing the external cell with the six metal screws. The valve is closed and water is added into the pipette until it completely fills the cell. The whole assembly is gently shaken and tilted to remove all the air bubbles that might be entrapped in the external cell. The water meniscus is adjusted to a reference level and a drop of kerosene is then added to prevent evaporation. This event (t = 0) typically occurs approximately 20 minutes after the first water-cement contact.

The readings from the external and internal cells are taken manually over a period of 7 days. The first reading is taken 20 minutes after adjusting the water meniscus at the reference level of the pipette. Typically, the other readings are taken every 20 minutes during the first 2 hours, every hour during the following 4 hours, every 2 hours during the following 10 hours and once a day during the remaining 6 days. Typical evolutions of the volume variations (ml) obtained from the external cell (pipette) and internal cells (air volume measurement device) are shown in Fig. 3. A calibration curve is used to correct the meniscus level readings to compensate for the thermal dilatation of the water (and of the cell itself) due to temperature variations during the measurement.

4.3 Interpretation of the volume variation curves

The first part of the curve obtained with the *internal cell* (Fig. 3) indicates that air is gradually expelled out of the internal cell until the paste sets. This phenomenon is due to the hydrostatic pressure of the water surrounding the internal cell, which forces a small volume of paste into the porous rods. Consequently, the air volume expelled out of the internal cell before setting corresponds to the volume of paste that is forced into the porous rods.

Once the paste sets, the formation of a rigid mineral skeleton stops this phenomenon and air is sucked into the paste due to the formation of menisci in the capillary
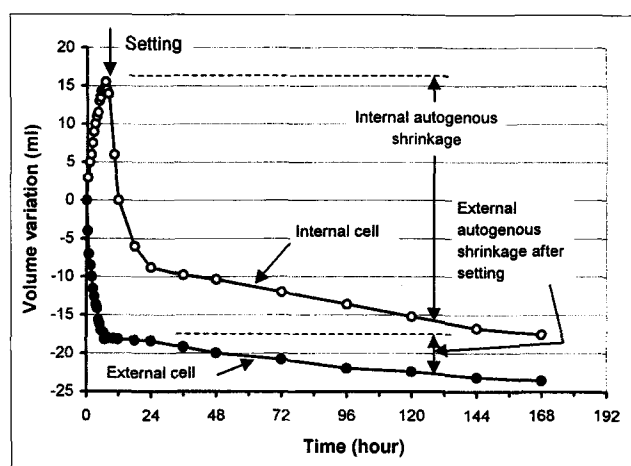
Fig. 3 - Typical evolution of the direct volume variations (ml) measured with the internal and external cells. The sample volume is 800 ml and the w/c ratio is 0.35.
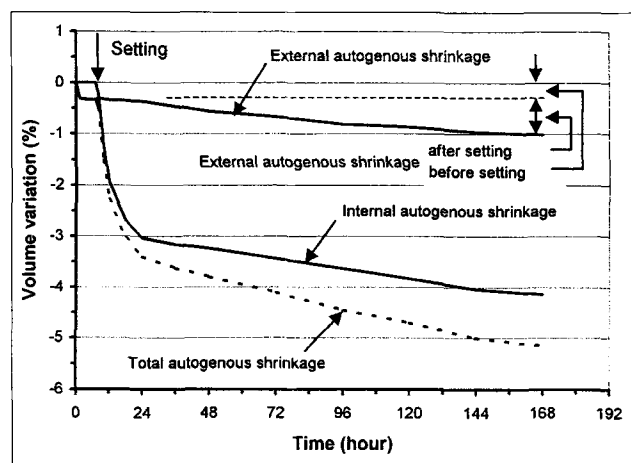

Fig. 4 - Typical evolution of the autogenous shrinkage curves (%) of a paste sample computed from the direct volume measurements obtained with the cell (w/c = 0.35).

pore network. Consequently, for a given time after the setting, the volume of air that penetrates into the internal cell is assumed to correspond to the *internal autogenous shrinkage*. The resulting curve is shown in Fig. 4, in which internal autogenous shrinkage is expressed as a percentage of the initial volume of the paste sample.

The first part of the curve obtained with the *external cell* (before setting) indicates that there is a rapid decrease in the external volume of the sample (Fig. 3). This rapid decrease is, for the most part, due to the penetration of paste into the porous rods. Therefore, to obtain the real evolution of the external autogenous shrinkage (before setting), we have to subtract the volume variations given by the internal cell during that period. This is simply done mathematically by adding the two curves (internal and external cells). The resultant curve gives the evolution of the *external autogenous shrinkage* (%) before setting (Fig. 4).

The second part of the curve obtained with the external cell directly gives the external autogenous shrinkage of the sample after setting (Fig. 3). By using the volume variation corresponding to the setting point as a reference value, calculating external autogenous

shrinkage curve after setting is straightforward. That curve is then graphically added at the end of the curve showing the evolution of external shrinkage before setting. The resulting curve is shown in Fig. 4, in which the external autogenous shrinkage is now shown as a percentage of the initial volume of the paste sample.

The evolution of total autogenous shrinkage, as a function of time, is obtained by mathematically adding the two curves corresponding to external and internal shrinkage. All these mathematical and graphing operations can be rapidly and easily performed using standard spreadsheet software.

The volume variations of a paste entirely made with Class F fly ash (no Portland cement) was analyzed to validate these assumptions and the numerical process involved in computing the final volume variation curves [14]. After 7 days, the curves confirm that no internal autogenous shrinkage occurs (no setting); only a small external autogenous shrinkage (< 1%) was measured, probably due to the presence of approximately 9% of CaO in the fly ash.

The curves in Fig. 4 yield much detailed information on the volume change and on the hydration kinetic of cement pastes. These curves can be used to evaluate the setting time that corresponds to the formation of a rigid skeleton and to the initiation of internal autogenous shrinkage. The curves also give the precise evolution of external autogenous shrinkage after setting. This is the most important type of autogenous shrinkage, because it has the largest influence on the cracking behavior of concrete elements. The internal autogenous shrinkage curve can also provide useful information on hydration kinetics since it is directly related to the amount of hydrates formed after the setting of the paste.

5. EXAMPLES OF MEASUREMENTS AND RESULTS

5.1 Materials and mixtures

Three series of measurements were performed to demonstrate how the proposed experimental technique can be use to analyze the effect of w/c ratio on the autogenous shrinkage of pastes hydrated under sealed conditions.

Fig. 5 shows the volumetric autogenous shrinkage curves ($\Delta V/V$) obtained with paste samples at w/c ratios of 0.45, 0.35, and 0.30. All the pastes were made with a normal Canadian Type 10 cement (ASTM Type I). The pastes (w/c ratios of 0.35 and 0.45) contain 0.01% of a viscosity agent (dry powder/mass of cement). A superplasticizer (naphthalene) was also used in the pastes with a w/c ratio of 0.30 and 0.35 at dosages of 1% and 0.5%, respectively (dry extracts/mass of cement).

5.2 External autogenous shrinkage

The curves in Fig. 5 clearly show that the magnitude of external autogenous shrinkage increases as the w/c ratio decreases. After 7 days of hydration, the volumetric
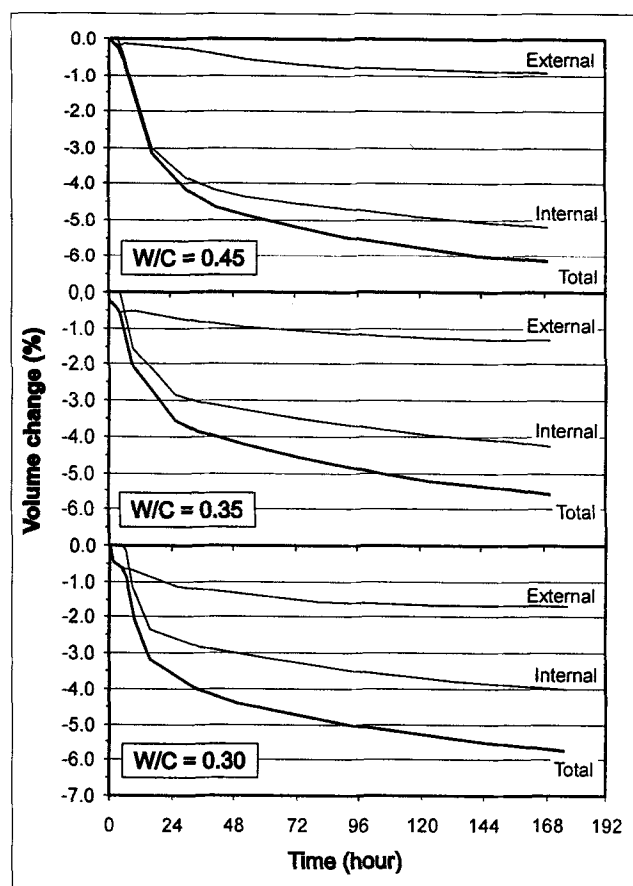
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Fig. 5 – Volumetric autogenous shrinkage curves ($\Delta V/V$) of three paste samples hydrated in sealed condition.

Fig. 6 – Evolution of the ratio between external and total autogenous shrinkage as a function of the w/c ratio and time.

5.4 Ratio between external and total autogenous shrinkage

Fig. 6 shows the evolution of the ratio between external and total autogenous shrinkage as a function of time and paste w/c ratio. Before setting, all total autogenous shrinkage is essentially translated into external autogenous shrinkage. However, immediately after setting (8 to 12 hours), the external/total autogenous shrinkage ratio starts to decrease very rapidly as more and more internal shrinkage occurs. Between 12 to 24 hours, the external/total autogenous shrinkage ratio starts to slowly increase due to the refinement of capillary porosity, which forces the formation of more curved menisci, generating a overall compression of the mineral skeleton.

After approximately 24 to 96 hours of hydration, all the external/total autogenous shrinkage curves plateau. Fig. 6 clearly shows that the limit value of external/total autogenous shrinkage is a function of paste w/c ratio. Generally, pastes with a low w/c (0.30) ratio tend to develop considerably more external autogenous shrinkage. For example, with a w/c ratio of 0.45, only approximately 15% of the total autogenous shrinkage is translated into external autogenous shrinkage. This contrasts with 30% in the case of a w/c ratio of 0.30.

The curves in Fig. 6 also show that pastes having a low w/c ratio start to develop a high external/total autogenous shrinkage ratio relatively early after setting. With these pastes, approximately 30% of the total autogenous shrinkage has already been translated into external autogenous shrinkage after 24 hours (w/c = 0.30). The paste with a w/c of 0.45, however, produces an external/total autogenous shrinkage ratio of approximately 8% after 24 hours, reaches only 15% after 4 days. These results may help explain the higher early cracking sensitivity in some kinds of high-performance concrete.

external autogenous shrinkage of the paste with the w/c ratio of 0.30 is approximately two times that obtained with the w/c ratio of 0.45. It can be noted that, for usual values of linear shrinkage ($\Delta L/L < 0.01$), the volumetric shrinkage ($\Delta V/V$) is approximately equal to three times the linear shrinkage. That linear relationship implies that volumetric and linear shrinkage evolve proportionally. Therefore, when the volumetric shrinkage doubles, the linear shrinkage does as well.

5.3 Internal autogenous shrinkage

After 7 days of hydration, the curves in Fig. 5 show that internal autogenous shrinkage decreases with the w/c ratio. One must bear in mind that the magnitude of internal shrinkage is roughly proportional to the degree of hydration. Therefore, these curves mainly indicate that, after 7 days, the degree of hydration of the paste decreases as the w/c ratio decreases. In a general sense, these results suggest that internal autogenous shrinkage is an important parameter because, by being directly related to the degree of hydration, it can be used to compare more precisely the relative magnitude of the external autogenous shrinkage obtained with different types of cement pastes.
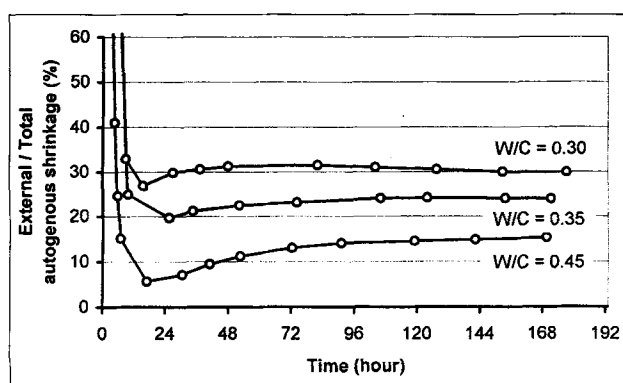
5.5 Intrinsic autogenous shrinkage

The results mentioned above show that the magnitude of the various forms of autogenous shrinkage is a direct function of the degree of hydration. Therefore, to

precisely assess the effect of mixture parameters (w/c, cement type, mineral admixture, etc.) on the development of autogenous shrinkage, the analysis of the results must include the degree of hydration. To facilitate this type of analysis, the various forms of autogenous shrinkage can be expressed as the volumetric contraction (ml) created by the complete hydration of 100 g of cement [6]. This concept of intrinsic autogenous shrinkage allows the precise comparison of the various form of autogenous shrinkage on the basis of a fixed amount of completely hydrated cement.

In order to calculate intrinsic autogenous shrinkage (ml/100 g of reacted cement), the amount of cement that has reacted by the end of the test (7d) must be determined. This can be obtained from the degree of hydration and the cement dosage of the mixture. When sealed hydration conditions are used, a good approximation of degree of hydration can be easily obtained from the mixture proportions and the measurement of the evaporable water content of the paste at the end of the test.

The degree of hydration of the three types of paste was determined as follows. Immediately after the test (7d), the internal cell was quickly disassembled and 6 paste fragments (20-60 g) were obtained by fracturing the sample with a chisel. The 6 fragments were then promptly weighed before storage in an oven (105°C) until constant mass. The loss of mass, divided by the initial mass of the sample, is assumed to be the evaporable water content of the sample. The chemically bound water is then approximated by subtracting the evaporable water content from the total water dosage of the mix. The amount of reacted cement is finally determined, based on the fact that 0.23 g of water is required to chemically react with 1 g of cement [8]. This procedure was found to be fairly simple and yield very good reproducibility with paste samples. It must be pointed out that this approach only gives an approximation of the degree of hydration, since the evaporable water content varies with the temperature used when drying the sample.

Table 1 presents the degree of hydration and intrinsic autogenous shrinkage of the 3 paste samples. After 7 days, the degree of paste hydration decreases with the w/c ratio. These results agree with other previously published data [15, 16].

Fig. 7 presents the evolution of the external, internal, and total intrinsic autogenous shrinkage as a function of w/c ratio. For a given amount of reacted cement, these curves show that the external autogenous shrinkage of the paste with the lower w/c ratio (0.30) is approximately twice that obtained with the higher w/c ratio (0.45). This phenomenon can be partially explained by the fact that, for a given volume of hydrates (or amount of reacted cement), a lower w/c generates much finer capillary porosity. Therefore, the water menisci are more curved and they induce higher compression in the mineral skeleton, which is translated into a higher external autogenous shrinkage.

The relationship between the intrinsic internal autogenous shrinkage and the w/c ratio shows that reducing the w/c ratio slightly decrease the intrinsic internal auto-

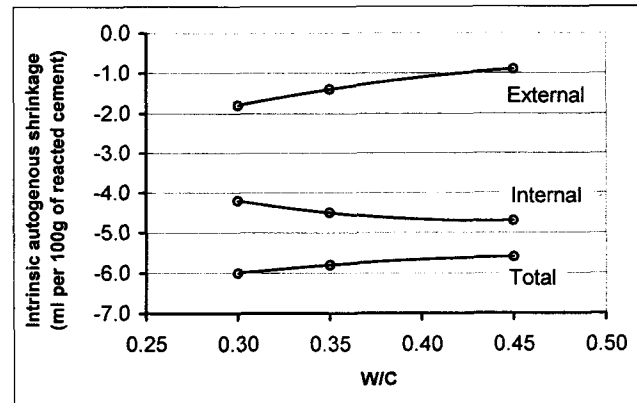| Table 1 – Degree of hydration and intrinsic autogenous shrinkage of the pastes				
W/C	Degree of Hydration after 7 days [%]	Intrinsic Autogenous Shrinkage [ml/100 g of reacted cement]		
		External	Internal	Total
0.45	65	0.9	4.7	5.6
0.35	52	1.4	4.5	5.9
0.30	47	1.8	4.2	6.0

Fig. 7 – Intrinsic autogenous shrinkage curves of three paste samples under sealed conditions.

genous shrinkage (Fig. 7). These results are somewhat unusual since the intrinsic internal autogenous shrinkage, by being directly related to the chemical shrinkage, should be constant, regardless of the w/c ratio. The slightly inferior intrinsic internal autogenous shrinkage of the lower w/c ratio paste may be explained by its very low air permeability that counters the penetration of air throughout the paste during the measurement. This side effect may contribute to a slight underestimation of the intrinsic autogenous shrinkage of low w/c ratio pastes.

The total intrinsic autogenous shrinkage range between 5.6 and 6.0 ml/100 g of reacted cement (Fig. 7). These values are very close to the theoretical chemical shrinkage deduced from the Powers equations, assuming that 0.23 g of water chemically reacts with 1 g of cement and that the volume of the solid hydration products equals the volume of the reacted cement, plus 0.746 times the volume of the chemically bound water [8]. For normal Portland cement (specific gravity = 3.14) the Powers equations give a theoretical chemical shrinkage of approximately 5.9 ml/100 g of reacted cement.

From a theoretical point of view, the total intrinsic autogenous shrinkage should be at least higher than the chemical shrinkage, because the total autogenous shrinkage includes the physical action of the menisci formed in the capillary pore network. Moreover, the difference between these two physical properties should increase as the w/c ratio decreases. The results obtained with the proposed technique confirm that the difference between chemical shrinkage and total autogenous shrinkage increases as the w/c decreases. However, the variation appears to be relatively small, since the total intrinsic

autogenous shrinkage increases by less than one percentage point when the w/c ratio varies from 0.30 to 0.45. This relatively small variation probably results from an underestimation of internal autogenous shrinkage due to the very low air permeability of the lower w/c pastes.

6. CONCLUDING REMARKS

The proposed experimental technique allows the complete characterization of the autogenous shrinkage of a paste sample under sealed conditions by separately and simultaneously yielding the internal, external, and total autogenous shrinkage. The magnitude of the total autogenous shrinkage obtained with this technique is close to the theoretical value given by the Powers equation. However, the preliminary results suggest that the total autogenous shrinkage of low w/c pastes (< 0.35) may be slightly underestimated due to the low air permeability of the matrix, which probably yields an underestimation of the internal autogenous shrinkage.

The first experimental reading starts less than 30 minutes after the initial water-cement contact. This feature allows the precise determination of the evolution of external autogenous shrinkage before and after setting. The procedure can also be used to determine the evolution of the ratio between external and total autogenous shrinkage. This ratio is an effective index for more accurate comparison of the effect of some mixture design parameters on the development of external autogenous shrinkage over time.

The proposed experimental technique is still under development. Several modifications are planned to improve the sensitivity of the measurement and to facilitate the reading sequence. Some of the future changes include the modification of the porous rod system in order to ensure that all internal autogenous shrinkage is measured, regardless of the paste mixture design. The precision and sensitivity of the measurements could also be improved by developing an electronic data logging system to automate the reading sequence.

The proposed experimental technique is currently limited to the characterization of the autogenous shrinkage of paste samples. Eventually, however, it could be adapted for studying mortar and concrete samples.

ACKNOWLEDGEMENTS

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