

Measuring techniques for autogenous strain of cement paste

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Abstract Volumetric measurement of autogenous strain is frequently performed by placing the fresh cement paste in a rubber membrane submerged in water. The volume change of the cement paste is measured by the amount of water displaced by the submerged sample. Volumetric and linear measurements of autogenous strain should in principle give identical results. However, the measuring results from the volumetric method are typically 3–5 times higher than the results from the linear technique. In this paper, water uptake from the buoyancy bath through the rubber membrane is identified as the principal artefact of the volumetric method. Water absorption is driven by a lowering of the water activity in the cement paste due to dissolved salts in the pore fluid and to self-desiccation. From the moment of casting, significant water uptake was registered in all experiments. This water uptake influenced the volumetric measurements by the same order of magnitude as the autogenous strain itself. By performing the measurements in a paraffin oil bath instead of a water bath, this artefact was eliminated. Furthermore, volumetric measurements performed in paraffin oil gave almost

identical results as linear measurements performed on the same cement pastes.

Keywords High-Performance concrete · Shrinkage · Autogenous strain · Measuring techniques

1 Introduction

Measurements of autogenous strain of cement paste have been carried out in two fundamentally different ways: measurement of volumetric strain and measurement of linear strain. Volumetric measurement of autogenous strain is frequently performed by placing the fresh cement paste in a rubber membrane submerged in water. The change in volume of the cement paste is measured by the amount of water displaced; for example, by measuring the weight change of the submerged sample [1, 2] or the water level in a capillary tube [3, 4]. Linear measurement of autogenous strain is frequently performed by placing the cement paste in a rigid mould with low friction. The length change of the cement paste may be recorded by a displacement transducer at the end of the specimen [5].

Both experimental methods have been extensively used in parallel for more than 50 years [6, 7]. The two methods should in principle give identical results; however, the volumetric method

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typically indicates 3–5 times higher strain than the linear technique [4]. Surprisingly few researchers have tried to find the reason for this disagreement [4, 8–10].

One fundamental reason for the inconsistency between volumetric and linear autogenous strain measurements after setting is transport of water through the rubber membrane, occurring when the buoyancy liquid used is water [11]. After setting, penetrated water may partially fill the internal voids produced by chemical shrinkage, causing an increase of the submerged weight or a decrease in water level that is interpreted as volumetric shrinkage. Concurrently, water penetration mitigates the shrinkage of the sample.

Rubber membranes normally used in the volumetric method are not waterproof. For example, condoms filled with water and exposed to air at low relative humidity lost about 0.5% of their water content per day [10, 12]. Despite this, some authors [10, 13] state that the influence of the permeability of the membrane on the volumetric measurements is insignificant until marked self-desiccation occurs in the cement paste, a few days after casting. Self-desiccation would then cause a relative-humidity gradient across the membrane and drive water from the water bath into the sample.

However, significant water transport through the condom does not start after some days, but instead occurs right after casting [11]. A difference in water activity across the membrane exists from the very start of the measurements, due to dissolved salts in the pore fluid. This difference in water activity constitutes a driving force for osmosis of water through the membrane into the paste. To examine this effect, Marciniak [12] measured the water uptake of condoms filled with synthetic pore solution, submerged in a distilled water bath. The weight of the condoms filled with pore solution increased by 0.1% per day, while condoms filled with distilled water did not show any appreciable weight change. Similarly, Beltzung and Wittmann [14] observed a mass increase of cement paste samples submerged in water and attributed this phenomenon to water diffusion through the latex membrane due to osmosis. Douglas and Hover [15] also measured a substantial weight change of condoms filled with cement paste

and mortars submerged in a water bath. Barcelo [16] tested different latex membranes and observed that all allowed water transport.

The present study aims at investigating the phenomenon of water uptake of the samples in the volumetric method. In particular, its driving mechanisms need to be ascertained and its influence on the measured strain quantified. It is examined if it is possible to compensate for the water uptake by means of a calculation, or to modify the volumetric technique as to exclude this artefact. To verify the results of the volumetric measurements, linear autogenous strain is measured on the same cement pastes with a technique based on corrugated moulds [17]. A more detailed description of the measuring techniques and of the results presented in this paper is provided in a report [18].

2 Materials

2.1 Salt solutions

Two different salt solutions were produced: (1) A synthetic pore solution based on the compositions reported by Page and Vennesland [19]: 0.4 mol/l Na^+ , 0.4 mol/l K^+ , 0.002 mol/l Ca^{2+} , 0.724 mol/l OH^- and 0.04 mol/l SO_4^{2-} ; (2) A solution with half the ionic strength obtained by 1:1 dilution with demineralized water. This solution is referred to as 50% diluted, synthetic pore solution.

2.2 Cement pastes

Three cement pastes with widely different properties were investigated: A: w/c 0.30%, 20% silica fume addition; B: w/c 0.30%, 20% silica fume addition + 0.6% superabsorbent polymer (SAP) by weight of cement; C: w/c 0.35%, 0% silica fume.

The cement used is a low-alkali Danish white Portland cement produced by Aalborg Portland, with Blaine fineness 420 m^2/kg . The Bogue-calculated phase composition (in wt.%) is: C_3S : 66.1, C_2S : 21.2, C_3A : 4.3, C_4AF : 1.1, C: 3.5, free CaO : 1.96, Na_2O eq.: 0.17.

The silica fume in pastes A and B was added as a dry powder at a rate of 20 wt.% of cement. The specific surface of the silica fume is 17.5 m^2/g

(BET method). The chemical composition (in wt.%) is: SiO_2 : 94.1, Fe_2O_3 : 1.00, Al_2O_3 : 0.13, MgO : 0.71, SO_3 : 0.43, and Na_2O eq.: 1.09.

In mixtures A and B, a naphthalene-based dry powder superplasticizer was added at a rate of 1.0 wt.% of cement + silica fume.

The SAP used in mixture B is a suspension-polymerized covalently cross-linked acrylamide/acrylic acid copolymer [20]. The spherical particles have diameters about 100–150 μm in the dry state. The size of the swollen SAP particles in the cement pastes and mortars is about 3 times larger due to pore fluid absorption. The SAP was added at a rate of 0.6 wt.% of cement. In mixture B, extra mixing water was added in an amount sufficient to saturate the SAP particles. This amount corresponds to an entrained w/c of 0.075 [20].

The cement pastes were mixed in a 5-l epicyclic mixer. Cement and all other admixtures, including the SAP in mix B, were put into the bowl. Mixing was done at low speed for 1 min, while gradually adding about 3/4 of the demineralized water. Mixing continued at high speed for 1 min, after which it was stopped and the paste was scraped off from the blade and the walls of the bowl for 1 min. Mixing was resumed for 1 min at low speed while the rest of the water was added; a last minute at high speed followed. The water was added in two steps to assure the homogeneity of the mix and the dispersion of the silica fume. Total mixing time from first water addition was 5 min. The temperature of the ingredients was approximately 20°C at mixing.

3 Methods

3.1 Volumetric autogenous strain

Volumetric measurements of autogenous strain were performed by monitoring the sample weight of cement paste samples contained in elastic membranes and submerged in a buoyancy liquid, either distilled water or paraffin oil. The experiments took place in a climate room at $20 \pm 0.2^\circ\text{C}$.

A total of 100–150 g of fresh cement paste was poured into a membrane, either a latex or polyurethane condom. The latex condoms were of

type ‘Plan’ by RFSU, with thickness 0.06 mm and without gliding crème or reservoir. The polyurethane condoms were of type ‘Avanti’ by Durex, with thickness 0.04 mm, gliding crème and reservoir. The gliding crème was removed from the external surface of the condom with a paper towel before filling the condom with cement paste.

The filled membrane was tightly closed with a knot; attention was paid to avoid entrapment of air bubbles. The excess part was then cut off and a 0.12-mm mono-filament Silicon-PTFE string (fishing line) was tied to the sample by means of a plastic strap. The string, about 400 mm long, was tied to a stainless steel hook at the other end. Typical samples are shown in Fig. 1.

During the measurements, the samples hung from a hook beneath the balance plate. The balance was a Sartorius CP 225 D, with sensitivity 0.01 mg for weights below 80 g and 0.1 mg for weights until 220 g. All measurements were performed in the 220 g measuring range. The 0.1 mg accuracy in this range was fully sufficient for the measurements; in addition, this improves the stability of the readings from the balance. The 0.1 mg weighing accuracy results in a nominal accuracy on the measured strain of 0.4–0.8 $\mu\text{m}/\text{m}$, considering typical sample sizes, different buoyancy liquids, and isotropic deformations. The actual uncertainty on the measured strain is significantly higher, being dominated by a number of factors, including temperature oscillations and drift of the balance. The balance was placed on a 100 kg

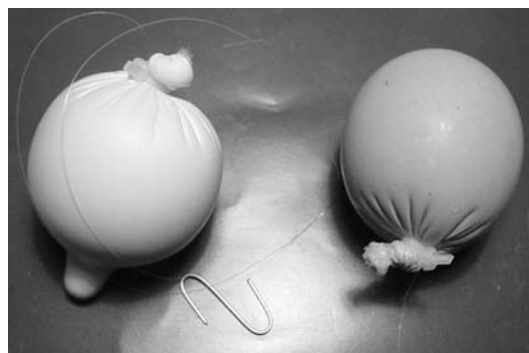


Fig. 1 Polyurethane membrane with reservoir (left) and latex membrane without reservoir (right) filled with cement paste. The picture shows also the plastic strap around the knot, to which a thin string was tied, ending with a steel hook

concrete weighing table to minimize vibrations. The table was provided with a 110-mm circular hole through which the string passed. The test set-up is shown in Fig. 2.

In the case of measurement in water, the sample was submerged in a water bath, consisting of a container of dimensions $0.5\text{ m} \times 0.8\text{ m} \times 0.4\text{ m}$, holding about 100 l of distilled water. Water circulation in the bath was avoided to improve the stability of the weight measurements. Most of the surface of the bath was covered with a Plexiglas lid to reduce evaporation; this was done to avoid changes in the water level and thermal gradients. In the case of measurements in paraffin oil, a smaller cylindrical container ($\varnothing 130\text{ mm}$, height 200 mm) was filled with paraffin oil; the container was submersed in the larger container filled with water to help keep the sample temperature constant. Temperature measurements by means of thermocouples showed a maximum temperature increase of 0.5°C in the samples measured in the water bath; the maximum temperature increase of the samples measured in the paraffin oil bath was 1.5°C . In both cases, the temperature peak occurred at 8 h from water addition and the temperature equilibrated at 24 h.

The submerged weight of the sample was measured and recorded automatically at regular intervals by controlling software. Measurements were generally recorded every 10 min from about 30 min after water addition up to about 2 weeks. Upon submersion of the sample in the buoyancy

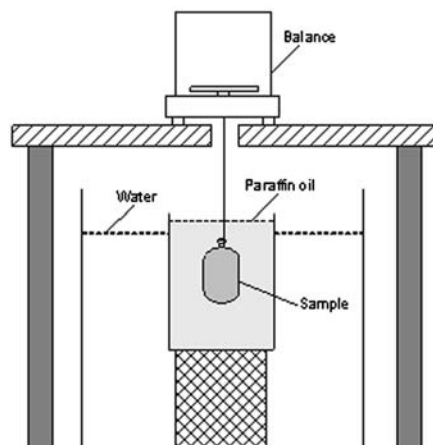
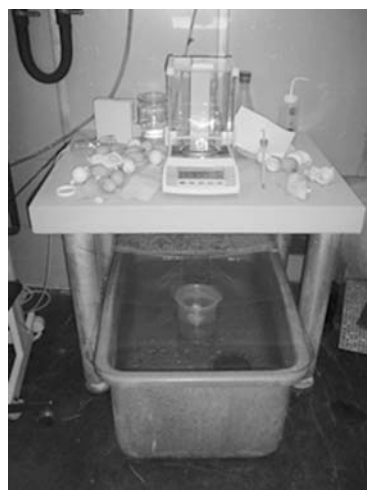
liquid, air bubbles attached to the upper, external part of the membrane were gently removed. The first measurement immediately after submersion was discarded because of the influence of oscillations of the sample on the measurements. Another source of error was represented by drops of buoyancy liquid attached to the string. However, these fell into the bath within the first minute of the experiment and did not lead to further errors.

The cement pastes studied showed no noticeable bleeding. Therefore, no rotation of the specimens was necessary, as applied in other studies [15].

3.2 Linear autogenous strain

Linear autogenous strain of cement pastes was measured by a special measuring technique, where the cement paste is encapsulated in thin, corrugated polyethylene moulds with length: diameter ratio of approximately 300:30 mm. The technique ensures insignificant restraint of the hardening cement paste and permits measurements to start 30 min after water addition. The cement paste was cast under vibration into the moulds; the specimens were then placed in a dilatometer equipped with automatic data-logging and electronic linear displacement transducers (Fig. 3). The dilatometer with samples was submerged into a temperature-controlled glycol bath at $20 \pm 0.1^\circ\text{C}$. Two samples were tested

Fig. 2 Set-up for volumetric measurement of autogenous strain of cement paste. A Sartorius CP 225 D balance is placed on a massive weighing table. The sample is suspended to the balance by means of a string, passing through a hole in the table. The sample is submerged in a buoyancy bath, either water or paraffin oil



simultaneously in the dilatometer, with a measuring accuracy of $\pm 5 \mu\text{m/m}$. A separate sample with an embedded thermocouple registered the temperature evolution; a maximum temperature increase of 0.5°C occurred at 8 h from water addition and the temperature equilibrated at 24 h. Measurements were performed every 15 min and started 30 min after water addition. A detailed description of the measurement technique can be found in the literature [17].

3.3 Water-uptake measurements

To test water uptake of buoyancy liquid through the different membranes used, a series of mass measurements was performed on different samples. Membranes filled with either salt solutions or cement pastes were submerged in the buoyancy liquids and their mass was recorded at regular intervals. Before every measurement, the samples were removed from the bath and the outer surface of the membranes was gently and thoroughly wiped with a paper towel to remove the attached film of water or paraffin oil.

4 Results and discussion

4.1 Water uptake through solution-filled membranes

To quantify the influence of osmosis on the volumetric measurements, rubber membranes filled

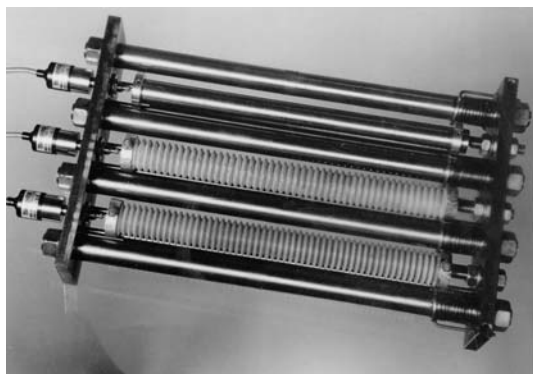


Fig. 3 Dilatometer with corrugated moulds for measurements of linear autogenous strain of cement pastes

with either synthetic pore solution, 50% diluted, synthetic pore solution, or demineralized water, were submerged in a demineralized water bath and their mass was monitored. Both latex and polyurethane membranes were tested. The membranes contained amounts of solutions varying from 70 g to 140 g. Figures 4 and 5 show the results of the experiments. Water diffusion through the condoms was proportional to the solute concentration. In particular, the mass of condoms filled with synthetic pore solution increased by 0.4% per day, while the mass of the condoms filled with 50% diluted, synthetic pore solution increased at about half the rate. The mass of membranes filled with demineralized water remained almost unchanged. No substantial differences in rate of water uptake were observed between latex and polyurethane membranes. However, after a few days in the water bath, the polyurethane membranes started leaking and lost weight. Examination of the polyurethane membranes revealed extensive degradation. The scatter between identical membranes filled with similar quantities of synthetic pore solution was quite substantial, as shown by the standard deviation bars in Figs. 4 and 5. The main reason is believed to be variability in thickness between the membranes and possibly presence of μm -sized pinholes in individual membranes.

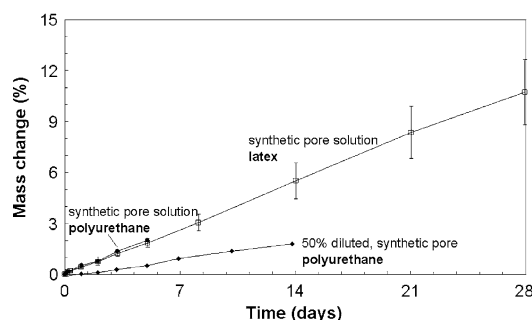


Fig. 4 Mass change of solution-filled membranes submerged in a demineralized water bath at 20.0°C . The average and the standard deviation of three samples is shown in the case of the latex membranes filled with synthetic pore solution and of two samples in the case of the polyurethane membranes with synthetic pore solution. For the polyurethane membranes with 50% diluted, synthetic pore solution, measurements on only one sample are shown

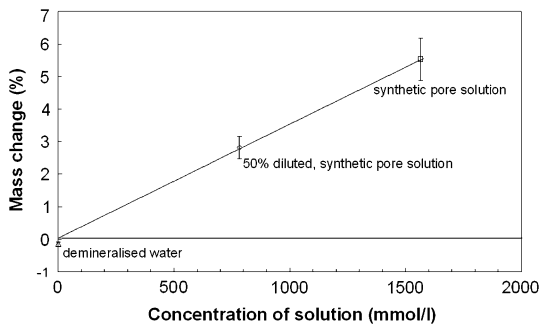


Fig. 5 Mass change of membranes containing synthetic pore solution, 50% diluted, synthetic pore solution, and demineralized water, submerged for 21 days in a demineralized water bath at 20.0°C. Each measurement shows the average and the standard deviation based on three samples

4.2 Water uptake through cement paste-filled membranes

Latex membranes filled with cement paste were submerged in a demineralized water bath about 30 min after mixing and their mass was measured until about 3 weeks of age. In the first day, the specimens were weighed every few hours, with the first measurement taken at about 2 h after mixing. Results of this experiment are shown in Fig. 6.

Pastes A and B are expected to have similar permeabilities throughout the hydration process, since the presence of SAP should have only limited

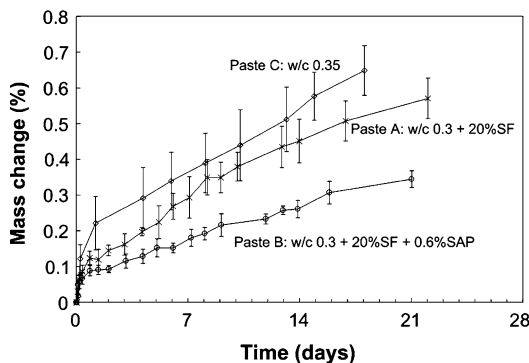


Fig. 6 Mass change of cement paste-filled latex membranes submerged in a water bath at 20°C. The average and the standard deviation of five samples are shown for pastes A and B, while three samples were measured for paste C

impact on the transport properties of the paste [21]. Therefore it is possible to analyze the water uptake of the two pastes in terms of the driving forces only. Long-term water uptake in cement paste B with SAP is lower than in paste A, whereas the water uptake in the first hours after mixing is quite similar. Osmosis is expected to dominate the water uptake of the samples in the first hours. Osmotic forces depend on the concentration of the pore solution, which should be similar in pastes A and B because the same cement and admixtures have been used. At a later age, self-desiccation occurs in paste A and becomes the dominant driving force for water uptake. This explains the higher water uptake of paste A at later ages. It is noticed that water transport into the sample due to osmosis in the first hours occurs at a very high rate compared to later ages. In fact, high porosity and low tortuosity of the cement paste pore system at early age facilitate transport of water into the cement paste. Moreover, at early age the transport of water into the sample is entirely liquid-based, instead of the slower, combined liquid–gas transport that occurs when pores in the cement paste have been partially emptied by self-desiccation. Whereas at early ages the permeability of the elastic membrane is the limiting factor of the transport process, at later ages the reduced permeability of the cement pastes becomes dominant.

Paste C shows a high water uptake in the first hours after casting, but is close to paste A in the following days, indicating that self-desiccation does influence water uptake. Comparison of paste C with the other two pastes is complicated, because the water uptake is influenced also by the transport properties of the paste. In particular, paste C, with higher w/c and no silica fume, will have a much higher permeability than pastes A and B at any time.

A simple calculation confirms that the water uptake in the first half a day might be caused primarily by osmosis. The rate of water uptake of latex membranes filled with synthetic pore fluid is about 0.4% by weight per day (Fig. 4). The cement pastes studied have initial water to solids volume ratio close to 0.5. The absorption in the first day may, therefore, be estimated as $0.4\% \times 0.5 = 0.2\%$ by weight of cement paste. This value is close to the measured, initial

absorption rate, around 0.15–0.20 wt.% per day (Fig. 6).

In Fig. 7, the estimated error on volumetric autogenous strain measurements that would result from the weight change of the submerged samples is shown. The error was calculated based on the assumption that all the water absorbed into the sample filled voids produced by chemical shrinkage. This is a questionable assumption, especially before setting, when no voids are present in the pastes and the absorbed water might instead contribute to raise the w/c of the pastes. In a volumetric measurement, water absorbed into the sample increases the submerged weight of the sample, which is interpreted as shrinkage. The error has been converted to linear shrinkage, assuming isotropic deformation. This facilitates comparisons with linear measurements in the following sections of this paper. Figure 7 shows that absorption of water from the bath produces a fictitious shrinkage after 3 weeks of 2,000 $\mu\text{m/m}$ for paste B, 4,000 $\mu\text{m/m}$ for paste A and about 5,000 $\mu\text{m/m}$ for paste C. Already at 1 day after casting, the measuring error is substantial, varying from 500 $\mu\text{m/m}$ to 1,500 $\mu\text{m/m}$ for the three pastes.

Besides the previously described artefacts, water uptake from the bath affects the curing conditions of the sample, which will not be autogenous. To perform measurements of autog-

enous strain, the cementitious system must be: (1) sealed, (2) kept at constant temperature, and (3) not subjected to external forces [22]. Therefore, when water penetrates the membrane, the measured strain is not autogenous strain. Barcelo [16] describes the effect of water absorbed into the sample as an increase of the average w/c of the cement paste; with this assumption, he concludes that the effect of water uptake on self-desiccation is negligible in the first week of hydration. However, it is misleading to assimilate the absorbed water to an increase of w/c: the situation is more similar to a cement paste with a given w/c cured with an external supply of water. This external supply of water will have a much greater influence on the autogenous shrinkage of the paste than if it was supplied initially as mixing water [20, 21].

Some authors [13, 23] state that water uptake through the elastic membrane is a slow process and its impact on the volumetric measurements can be neglected in short-term measurements; therefore, use of the volumetric technique is justified for measurements in the first day after casting. However, no data on water uptake of volumetric samples was published by these authors to confirm their hypothesis. On the contrary, Fig. 7 in this paper shows that the impact of water uptake from the buoyancy bath on the volumetric measurements is great enough to compromise the whole measurement from the time of casting, since an error on the measured strain of 500–1,500 $\mu\text{m/m}$ in 1 day is of the same order of magnitude as the true strain itself.

In theory, it is possible to eliminate this artefact by stopping the water uptake through the elastic membrane. The most obvious way is to choose an impermeable membrane. With this aim, L. Barcelo (personal communication) measured a number of latex membranes but found that they all were permeable. Douglas [24] tested a number of different latex, polyurethane and neoprene membranes and all were permeable to water. Also in the present research, latex and polyurethane membranes were found to have similar permeabilities to water (Fig. 4). The lack of tightness of the membranes is due both to their intrinsic permeability and to the presence of defects such as pinholes. Pinholes are especially significant to increase water uptake at early ages,

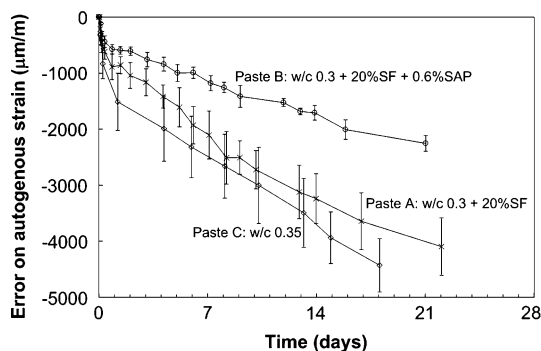


Fig. 7 Error on the measured autogenous strain due to water uptake of cement paste in latex membranes submerged in water bath at 20°C. Linear strain was calculated assuming isotropic behaviour; shrinkage is plotted as negative strain. The average and the standard deviation of five samples are shown for pastes A and B, while three samples were measured for paste C

while their importance at later ages are lowered by the reduced permeability of the cement paste. Making the membrane thicker, while improving the tightness, will also increase its stiffness and restrain the cement paste, especially around setting time [8]. It is not excluded that a satisfactory balance between stiffness and tightness of the elastic membrane can be achieved; however, this is not the case with the type of membranes used in the volumetric measurements in the last 15 years, i.e., latex condoms.

Another approach to eliminate the water uptake of the samples in the volumetric test is to substitute the buoyancy liquid. Measurements of mass change of polyurethane condoms filled with different cement pastes and submerged in paraffin oil were performed: the mass gain in the first week of submersion was about 0.005%, which is roughly 1/30–1/70 of the mass change observed in the same period on latex condoms filled with cement paste and submerged in a water bath, cf. Fig. 6. This mass change would lead to an error on the measured autogenous strain of less than 50 $\mu\text{m}/\text{m}$ in 1 week. Polyurethane was preferred to latex because latex does not tolerate oils and swells up in a matter of hours. An expansion is erroneously measured as a result of this.

4.3 Measurements of autogenous strain on cement pastes

On the same pastes tested for weight change underwater, the autogenous strain was measured with three different methods:

- (1) Measurements of submerged weight of cement paste samples cast in latex condoms and submerged in a water bath;
- (2) Measurements of submerged weight of cement paste samples cast in polyurethane condoms and submerged in a paraffin oil bath;
- (3) Measurement of length changes of samples cast in corrugated moulds in the dilatometer [17].

In all cases, the mass of the samples before and after testing was measured. Mass change of the samples was generally negligible in the case of

measurements of type (2) and (3), whereas the mass gain of samples tested in a water bath confirmed the findings in the previous section (Fig. 6).

In Figs. 8–10, measurements of autogenous strain with the three techniques are presented. For comparison, also calculated curves are shown based on addition of the error due to water uptake (Fig. 7) to the measurement in paraffin oil.

Measurements in Figs. 8–10 show a number of common features. Linear measurements performed with the dilatometer [17] and volumetric measurements in paraffin oil fundamentally agree, after some differences in the first few hours. Moreover, both linear measurements and volumetric measurements in paraffin oil are quite reproducible and show little scatter throughout the measuring period. This good agreement, obtained for three cement pastes with completely different autogenous strain behaviour, confirms the overall validity of these two techniques. However, the volumetric technique is experimentally more laborious than the linear and it is sensitive to a number of artefacts, including pressure exerted by the membrane and reabsorption of bleeding water and entrapped air into the sample [10, 15, 18].

On the contrary, the volumetric measurements in water fundamentally disagree with the other two types of strain measurements. The measured shrinkage is systematically higher than for the

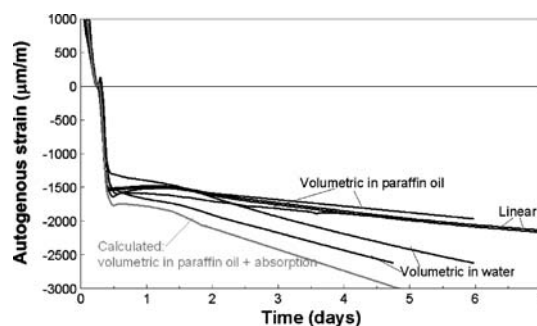


Fig. 8 Measurements of autogenous strain with different techniques at 20°C on cement paste with w/c 0.3% and 20% silica fume addition (paste A). Conversion from volumetric to linear strain assumes isotropic behaviour; the curves are zeroed at setting, 6 h after casting, shrinkage is plotted as negative strain

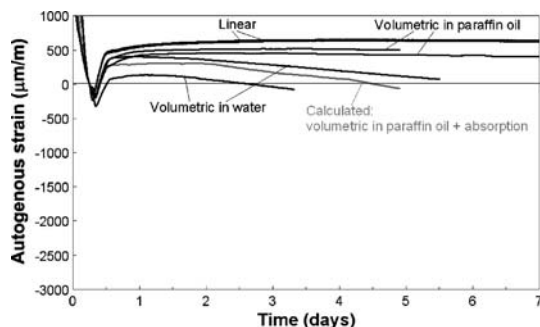


Fig. 9 Measurements of autogenous strain with different techniques at 20°C on cement paste with w/c 0.3, 20% silica fume addition and 0.6% superabsorbent polymers (SAP) by weight of cement (paste B). Conversion from volumetric to linear strain assumes isotropic behaviour; the curves are zeroed at setting, 6 h after casting; shrinkage is plotted as negative strain

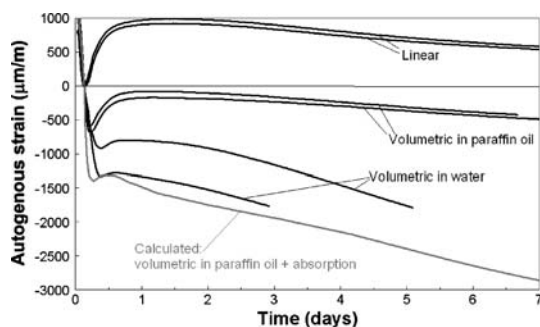


Fig. 10 Measurements of autogenous strain with different techniques at 20°C on cement paste with w/c 0.35 (paste C). Conversion from volumetric to linear strain assumes isotropic behaviour; the curves are zeroed at setting, 3.5 h after casting; shrinkage is plotted as negative strain

other two techniques. This confirms previous data in the literature about the volumetric technique in water bath [4, 9]. As discussed in the previous paragraphs, water uptake of the samples through the elastic membrane is a principal cause of this discrepancy [16, 24]. Water uptake causes an increase of the submerged weight of the sample that is erroneously interpreted as shrinkage in the volumetric measurements. A way to verify this assumption is adding the error due to water uptake (Fig. 7) to the volumetric measurements in paraffin oil, and comparing the results with the volumetric measurements in water. Results of this calculation are also shown in Figs. 8–10. These calculated curves have strong similarities with the

volumetric measurements in water bath. In particular, a very close agreement is obtained with the shrinkage rate after a couple of days: this shows that a dominant part of the shrinkage measured with the volumetric method in water is an experimental artefact due to water uptake through the rubber membrane. Moreover, volumetric measurements in water show a poor reproducibility in the first few hours, while the strain rate at later ages is more similar between the samples. Similarly, mass gain of the samples stored in water showed a high scatter especially in the first period, which may be due to different permeabilities of the elastic membranes. The high initial scatter in the volumetric measurements in water is another consequence of the water uptake through the membrane, which varies from sample to sample especially in the first hours.

Barcelo and Douglas [16, 24] have suggested correcting the volumetric measurements in a water bath based on the average measured absorption. This approach is not recommendable. In addition to the unnecessary experimental trouble, the results will be influenced by a high scatter and furthermore they will be corrected for the effect of water absorption only in part.

5 Conclusions

Water uptake from the buoyancy bath through the elastic membrane is a principal artefact of the volumetric method for measuring autogenous strain. Water uptake of the samples is driven by a lowered water activity in the cement paste, due to dissolved salts in the pore fluid and self-desiccation. Water is absorbed into the cement paste sample at a high rate from the moment of submersion; the error it produces on the measured strain is of the same order of magnitude as the autogenous strain itself. It is practically impossible to correct the measurements from water uptake through the membrane.

Water uptake through the membrane can be avoided by using paraffin oil as buoyancy liquid. The volumetric autogenous strain measured in this way is in good agreement with measurements performed with a linear, dilatometric technique.

This confirms the overall validity of these two techniques. Some minor differences between the two measuring techniques in a couple of hours around setting should be the object of further research.

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References

1. Yamazaki Y, Monji T, Sugiura K (1976) Early age expanding behaviour of mortars and concretes using expansive additives of $\text{CaO-CaSO}_4\text{-4CaO} \cdot 3\text{Al}_2\text{O}_3 \cdot \text{SO}_3$ system, 6th int. conf. on the chemistry of cement, Moscow, September 1974, Stroyizdat, Moscow III-5:192–195
2. Justnes H, Van Gemert A, Verboven F, Sellevold E (1996) Total and external chemical shrinkage of low w/c ratio cement pastes. *Adv Cem Res* 8(31):121–126
3. Yates JC (1941) Effect of calcium chloride on readings of a volumeter inclosing Portland cement pastes and on linear changes of concrete. *Highway Res Board Proc* 21:294–304
4. Barcelo L, Boivin S, Rigaud S, Acker P, Clavaud B, Boulay C (1999) Linear vs. volumetric autogenous shrinkage measurement: material behaviour or experimental artefact? In: 2nd int. res. sem. on self-desiccation and its importance in concrete technology, Lund, Sweden, pp 109–125
5. Buil M (1979) Studies of the shrinkage of hardening cement paste. D.Eng. thesis, Rapport de recherche LPC No. 92, Laboratoire Central des Ponts et Chaussées, Paris
6. Davis HE (1940) Autogenous volume changes of concrete. In: Proc. 43rd annual meeting, American Society for testing materials, Philadelphia, vol 40(32), pp 1103–1112
7. Wuerpel CE (1956) Laboratory studies of concrete containing air-entraining admixtures. *ACI J* 42(4):305–359
8. Baron J, Buil M (1979) Comments on the paper: mechanical features of chemical shrinkage of cement pastes by N. Setter & D.M. Roy. *Cem Concr Res* 9(4):545–547
9. Charron J-P, Marchand J, Bissonnette B, Pigeon M, Zuber B (2002) Comparative study of the effects of water/binder ratio and silica fume on the volume instability of hydrating cement pastes at early age. In: 3rd int. res. sem. on self-desiccation and its importance in concrete technology, Lund, Sweden, pp 39–50
10. Hammer TA, Bjøntegaard Ø, Sellevold EJ (2002) Measurement methods for testing of early age autogenous strain. In: Bentur A (ed) Early age cracking in cementitious systems. RILEM TC 181-EAS Committee, RILEM, Cachan, pp 234–245
11. Lura P, Jensen OM (2005) A discussion of the paper: “on the measurement of free deformation of early age cement paste and concrete” by Ø. Bjøntegaard, T.A. Hammer and E.J. Sellevold. *Cement Concrete Comp* 27(7–8):854–856
12. Marciniak S (2002) Autogenous deformations and relative humidity change: experimental studies. MSc thesis, Department of Building Technology and Structural Engineering, Aalborg University, Aalborg
13. Bjøntegaard Ø, Hammer TA, Sellevold EJ (2004) On the measurement of free deformation of early age cement paste and concrete. *Cement Concrete Comp* 26(5):427–435
14. Beltzung F, Wittmann F (2002) Influence of cement composition on endogenous shrink age. In: 3rd int. res. sem. on self-desiccation and its importance in concrete technology. Lund, Sweden, pp 113–125
15. Douglas KS, Hover KC (2002) Measuring non-drying bulk shrinkage of cement paste and mortar using Archimedes’ principle: Part I. In: Jensen OM, Bentz DP, Lura P (eds) Autogenous deformation of concrete, ACI SP 220, American Concrete Institute, Farmington Hills, Michigan, pp 39–51
16. Barcelo L (2001) Influence des caractéristiques des ciments sur la structuration et le comportement dimensionnel des matériaux cimentaires au jeune age. PhD thesis, Ecole Normale Supérieure de Cachan, Cachan, France
17. Jensen OM, Hansen PF (1995) A dilatometer for measuring autogenous deformation in hardening Portland cement paste. *Mater Struct* 28(181):406–409
18. Lura P, Jensen OM (2005) Measuring techniques for autogenous strain of cement paste, Portland Cement Association, PCA R&D Serial No. 2925 (Skokie, IL), 26 pp
19. Page CL, Vennesland Ø (1983) Pore solution composition and chloride binding capacity of silica-fume cement pastes. *Mater Struct* 16(91):19–25
20. Jensen OM, Hansen PF (2002) Water-entrained cement-based materials – II. Implementation and experimental results. *Cement Concrete Comp* 32(6):973–978
21. Jensen OM, Hansen PF (2001) Water-entrained cement-based materials—I. Principles and theoretical background. *Cement Concrete Comp* 31(4):647–654
22. Jensen OM, Hansen PF (1996) Autogenous deformation and change of the relative humidity in silica fume-modified cement paste. *ACI Mater J* 93(6):539–543
23. Baroghel-Bouny V, Mounanga P, Khelidj A, Loukili A, Rafai N (2006) Autogenous deformations of cement pastes: part II. W/C effects, micro–macro correlations, and threshold values. *Cement Concrete Research* 36(1):123–136
24. Douglas KS (2002) Non-drying bulk shrinkage of cement paste and mortar: application to pneumatic transport mixing of sand and cement. MSc thesis, Cornell University, Ithaca, New York