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Influential factors in volume change measurements for cementitious materials at early ages and in isothermal conditions

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ABSTRACT

Early age deformations, when restrained, lead to an increase of cracking risk of the material, especially in the case of high strength materials to which we apply modern high energy mixing techniques which accelerate initial hydration rate. The experimental campaign aims to investigate and understand the differences not yet explained between several autogenous measurement techniques, such as the initial swelling measured by the linear devices that is not observed in the case of volumetric measurements. The differences between the results obtained by means of devices when values are zeroed at setting time can be imputed to the intrinsic behaviour of the material within the well-defined boundary conditions imposed by the molds, and not only to the measurement artefact.

1. Introduction

Research concerning the early age development of cementitious materials has been accelerated during the past twenty years, as a new and performing design of materials was possible due to advances made in this field. Improved materials, with higher strength and lower porosity became available on the market and extensively used for their enhanced properties for various applications. Still, the better performance of such materials came with a drawback: a higher occurrence of cracking risks at early ages due to a higher autogenous shrinkage [1–3]. Indeed the main deformations occurring at early age are a direct result of the hydration reaction and of its consequence i.e. the self-desiccation [4,5].

An important number of experimental studies were made in order to better characterize early age deformations.

Various means of measure of this phenomenon were proposed [6–8]. The results of the studies led to a lukewarm success, as different experimental devices led to quite important discrepancies between the results [9–13].

In the same time, the multitude of materials available on the market makes impossible a laboratory expertise for each and every concrete mix to be used on site. The need of numerical tools able to predict the behaviour of the materials by using mathematical laws derived from experimental observations, as virtual testing labs, has become critical. Several complex microstructure models [14–16] were thus developed, based on sound experimental results. This is very important, as the response of the model relies on the initial input of valid experimental data.

Modelling the development of a hydrating microstructure requires continuous monitoring of the evolution of the material during its early days. The necessary tests in order to understand the actual behaviour of the material should reflect the important stages of its development. The hydration reaction should be seen as the triggering mechanism of the physical consequences experienced later on by the material. The negative volume balance between the initial products and the hydrates is an intrinsic property of the hydration reaction, leading to what is called the chemical shrinkage (Le Chatelier [17]). The material, which is initially in a fluid state, is thus free to deform. With the advancement of the reaction, more hydrates are formed, the porous network (initially water saturated)

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is refined, and the skeleton of the future material is being built. Once the material has set, i.e. once the material passes from a viscous fluid state to a solid state, the deformations caused by the Le Chatelier contraction are restrained, as the material has gained enough strength to oppose deformations. Furthermore, when no external water supply exists, anhydrous cement consumes the water found in the capillary pores to form hydrates, leading to a reduction of the relative humidity in the system. The porous network witnesses a capillary depression, which leads to the macroscopic shrinkage. At the same time, the material is rapidly gaining in strength and rigidity. Still, provided the deformations of the material are restrained, the cracking risk depends on the extent of deformations and on the development of mechanical properties. Thus, if the autogenous shrinkage deformations are over or under estimated by the measurement devices, it becomes difficult to validate the model response. Indeed, one needs accurate experimental results in order to verify the model. As for the accuracy of experimental results, it is directly influenced by external factors (measuring principle, operator, etc.).

Autogenous deformations (shrinkage or swelling) of cementitious materials is defined as being the unrestrained deformation occurring at constant temperatures in a homogenous material, with no water exchange with the external environment (sealed conditions) and after the cement has set [21], i.e. after a rigid skeleton begins to develop. Chemical shrinkage is the internal-microscopic volume reduction which is the result of the fact that the absolute volume of the hydration products is smaller than that of the reacting constituents (cement and water). This reduction can be considered roughly proportional to the degree of hydration [21]. Autogenous shrinkage is a direct consequence of the hydration reaction and the chemical contraction: once the material has set, chemical shrinkage gives rise to creation of empty pores in the microstructure, and with the ongoing hydration the internal water consumption leads to a decrease of the internal relative humidity (self-desiccation), and thus to the creation of menisci in the capillary porosity.

As autogenous deformations play a major role in early age cracking risks of a material, several measurement devices were developed in literature, in order to evaluate the extent of the evolution of the strains. One of the main discussions in early 2000's concerned the measurement artefacts, as a perfect convergence between the different devices used was far from being acquired. In order to identify and eliminate the artefacts, extensive work was done in the past.

In the case of chemical shrinkage, three measurement techniques are employed: dilatometry – initially proposed by le Chatelier [9,22–28], gravimetry [29,30] and pycnometry [22]. Nevertheless, they are all based on the same basic principle. The cement paste is placed in a flask and the system is filled with water. As the hydration reaction takes place, initial mix water is consumed. Once the material sets, the extra amount of water must be available in sufficient amount in order to ensure the further development of the hydration of the anhydrous grains and to fill the voids created by the volume deficit. The main factors affecting the chemical shrinkage measurement are: the thickness of the layer of cement paste which is introduced in the flask [9], the amount of water added at the surface of the sample (avoid leaching [12,27,30]), the presence of air voids within the fresh paste [9,12,31].

The influence of the water to cement ratio was also under discussion. Some authors found that the chemical shrinkage varies with the w/c value [9,23,24], others found that at early age (at least up to 24 h) the chemical shrinkage is constant, independently the w/c used [13,32,33]. One can imagine that the kinetics of the reaction can be diminished later on for low w/c ratio mixes, once the porosity is filled with hydrates and the external water will difficultly penetrate.

In the case of autogenous shrinkage, measurements are generally linear or gravimetric. Linear measurements correspond to the monitoring of dimensional changes of a specimen on its longitudinal direction. The measurement can be made vertically by means of extensometers either on the sample that has been removed from the mold and sealed [34,35] or on the sample kept in the original mold using LVDT's or extensometers [36,37]. Function of the chosen method, monitoring can begin either after the set, or at 24 h. As the monitoring should start as soon as possible, some authors [38–40,43] proposed the use of a flexible mold that does not prevent the deformation of the material. Measurements can also be taken horizontally, using LVDT's [10], gauges [3], IDT's (inductive displacement transducers [41]) or lasers [42]. In order to be able to follow the deformations as soon as the sample is installed in the mold, flexible molds have also been proposed for the vertical measures [39,40,44,56]. The volumetric measurements set-up are very similar to chemical shrinkage gravimetric measurements see [45,46,50]. The main difference is that the system must be sealed in order to ensure the autogenous conditions required by the measure. The material is cast in a flexible waterproof membrane, able to follow the deformation of the sample. The sample is immersed in a thermostatically controlled water-filled basin. Deformations are measured automatically immediately after casting. Usually no longer than 1 h will pass from the fabrication of the paste to the first acquired data.

One of the factors that influence the validity of the results is the bleeding of the material, giving rise to supplementary deformations at the moment when the water is re-absorbed. A solution was found by Ref. [36], who proposed a rotating system in order to avoid bleeding. Several studies show a neat improvement of precision of the measure when using the rotating system [9,47–49]. The same principle was used in the case of volumetric measurements [59], showing important differences between the static samples and the rotated samples for bleeding materials. The linear measures are prone to friction artefacts (mainly for horizontal methods) and to important settlement/sedimentation effects (mainly for vertical methods). In order to lessen the friction effect the use of lubricants on the substrate, plastic films and talcum powder is common practice [41,51,52]. An important parameter for the volumetric method is the permeability of the membrane. Indeed, as the samples are usually immersed in water, one must ensure the perfect sealing of the material. The latex membranes used are not perfectly impermeable to water, and, in this case, deformations are over estimated [53–55]. This artefact can be corrected by replacing the immersion liquid, using oil instead of water [12]. Several comparisons between the different measurement techniques were made ([9,11,13,56] including a RILEM round robin program [57]). A comparison between the standard method (corrugated tube ASTMC1698-09) and a setup providing rotation of the samples was recently made by [58,70], showing the influence of the rotation on the overall autogenous shrinkage.

The general trend shows that linear horizontal measurements give the lowest values of deformations (due to friction), followed by the linear vertical measurements that are more important (due to settlement at early age). Generally, if the values obtained are reinitialized at 24 h, the two measurements are quite similar. This is not the case with volumetric measurements which seem overestimated in most of the cases. Still, once the measurement artefacts are not present (mainly linked to the permeability of the membrane), Sant et al. [12] have shown that linear and volumetric measures can give similar results.

The main objective of the present work is to critically examine performances and domain of validity of several measurement