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Examining the "time-zero" of autogenous shrinkage in high/ultra-high performance cement pastes

Hao Huang *, Guang Ye

Microlab, Delft University of Technology, Faculty of Civil Engineering and Geosciences, P.O. Box 5048, 2600 GA Delft, The Netherlands

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ABSTRACT

The term "Time-zero", i.e., the time for the start of autogenous shrinkage measurement, is usually used for estimating the cracking potential of structural components. Accurate determination of the "time-zero" is therefore critical for autogenous shrinkage measurement, which is the main objective of this study.
There is a general agreement about the existence of a relationship between autogenous shrinkage and RH changes in hardening cement paste. An improved hygrometer method was developed for monitoring the change of internal RH in cement pastes 1 h after casting. This provides immediate experimental results of RH change for determining the "time-zero". It indicates that the internal RH of cement pastes does not decrease immediately after the final setting time. A new "time-zero" is defined as the onset of internal RH drop, which is more reasonable for estimating the cracking potential of cement-based materials, compared to the "time-zero" represented by the final setting time.

1. Introduction

Ultra-high performance concrete (UHPC) and high performance concrete (HPC) show excellent performance regarding ductility, compressive strength and durability [1]. However, UHPC/HPC can experience large shrinkage deformation. A very high autogenous shrinkage occurs in the first one or two days after casting, which points to a considerable cracking potential at early ages [2,3]. Such early age cracking due to restrained autogenous shrinkage tends to negate the numerous advantageous properties of HPC and UHPC and significantly limits their prospective utilization in construction. The high autogenous shrinkage of these concretes is due to the low water-binder ratio and high amount of silica fume used which causes a significant drop in internal relative humidity (RH) in the cement paste during hardening, and self-desiccation occurs in absence of an external source of water [4]. Autogenous shrinkage and self-desiccation of concrete are known since the year 1900 [5], but their practical importance has only been recognized in last two decades. Although the actual driving force of autogenous shrinkage is still unclear [6], there is a general agreement about the existence of a relationship between autogenous shrinkage and RH changes in hardening cement paste [7,8].

When cement paste is in liquid state, the chemical shrinkage due to cement hydration is fully transformed into external volume change [7]. This volume change does not induce any cracking potential inside the material [9,10]. With the hydration of cement, a "stable" solid skeleton is formed in the hardening paste. Since then, in sealed condition, the chemical shrinkage cannot be totally transformed into external volume change. Empty pores are thus formed inside the paste and air-water menisci occur [11]. As the water is consumed by cement hydration, bigger pores inside the solid skeleton empty first [7]. This process is known as self-desiccation, in which the relative humidity drops.

"Time-zero", corresponding to the start of autogenous shrinkage, is defined as the time when the cement paste develops a "stable" solid skeleton to enable tensile stress transfer [9]. Accurate determination of the "time-zero" is therefore critical for autogenous shrinkage measurement. The ASTM C1698-09 [12] establishes a set of standard methods to determine autogenous shrinkage and suggests to use the final setting time determined by Vicat apparatus as the "time-zero". Due to the relative arbitrariness of the Vicat penetration method, some researchers [13–16] has questioned the reliability of using the final setting time as the "time-zero" for autogenous shrinkage. They believe that the penetration method does not precisely correspond to the "time-zero". Bentur [13] found the "time-zero" is roughly equal but not identical to the final setting time. Miao et al. [14] illustrated the difficulty in measuring the moisture change in very early age concrete by conventional hygrometer method as the material is still in the super hygroscopic range (approximately in the range of 98%–100% of internal RH). They have developed a special measurement system for the meniscus depression within the paste or concrete, and determined the "time-zero" from the observed capillary depression [14]. Darquennes et al. [15] found the evolution of autogenous deformation strongly depends on the definition of the "time-zero". Based on the free deformation curve tested in their research, three different definitions of the "time-

* Corresponding author.
E-mail address: H.huang-1@tudelft.nl (H. Huang).

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zero" were considered: the initial or final setting time, the time of peak expansion at early ages, and the time characterized by the second maximal absolute value of the deformation rate (Fig. 1). The last definition was chosen as the "time-zero", corresponding to development of significant stresses inside the specimen, and confirmed by Temperature Stress Testing Machine (TSTM). Miao et al. [14] tried to determine the "time-zero" of autogenous shrinkage by capturing the initiation of self-desiccation, and Darquennes et al. [15] tried to determine the "time-zero" as the time for the initiation of internal tensile stresses. Because the self-desiccation is the main reason for the autogenous shrinkage [7], the "time-zero" determined by these two methods should be correlative. In this study, the "time-zero" is determined through analyzing the moisture change (internal RH change) at very early ages, and compared with the "time-zero" determined by Darquennes et al. [15].

The typical procedure for measurement of internal RH is to place the cement paste in a small, sealed and thermostatic container. The internal RH of the cement paste is assumed to be equilibrated with the RH of the air inside the container, which in turn is measured by a humidity sensor [7]. In practical terms, 100% internal RH is easily reached at a very early age after casting, that is to say, up to several hours after mixing. According to Raoult's law, the presence of ions in solution decreases the saturated vapor pressure of solution [6]. So, it is not possible that the internal RH of the pore solution in cement paste reaches 100% due to the effect of ions in the pore solution. Meanwhile, the internal RH is extremely influenced by the temperature. According to the Bulletin of the American Meteorological Society [17], when RH is above 50%, every 1 °C difference in the dew point and dry bulb temperatures, the relative humidity decreases by 5%, starting with RH = 100% when the dew point equals to the dry bulb temperature. It means that, in high RH environments, water vapor is very easy to condense (RH = 100%) on the surface of the colder object. Assuming that the internal RH of cement pastes is 98% in first several hours after mixing (which is also observed in experiments in this study); condensation can happen on the humidity sensor when the temperature of the humidity sensor is 0.4 °C colder than the water vapor. This makes the measurement of RH at very early ages very complicated. Especially if the hydration heat of cement is taken into account, the probability of condensation is even more.

Ultrasonic pulse velocity (UPV) measurement can be used for monitoring the development of solid skeleton inside cement paste. The UPV technique is based on a pulse generating and transmitting transducer and a receiving transducer. When cement paste is subjected to a pulse vibration load, the longitudinal pulse transmitted to the paste is reflected at various solid-liquids interfaces or passes through the solid phase until it reaches the receiving transducer. With the development of cement hydration, the connection of smaller cement particles leads to clusters that form a solid skeleton. The UPV increases with the development of this solid skeleton, as the stiffness of the cement paste largely depends on the connection of the solid phase [18].

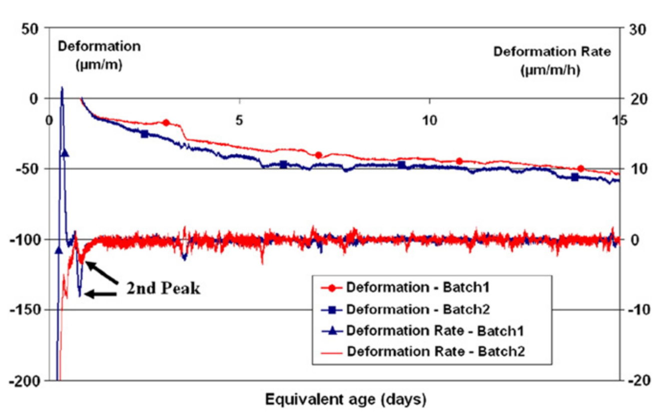

Fig. 1. Evolution of the deformation rate and the autogenous deformation expressed from the second maximal value of the deformation rate of the CEM I mix. (Darquennes et al. [15]).

Table 1
Properties of cement.

Components	Cement[a]
Chemical properties, % by weight	
CaO	64.0
SiO_2	24.0
Al_2O_3	5.0
Fe_2O_3	3.0
SO_3	2.4
Na_2O	0.3
K_2O	–
Loss on ignition (LOI)	1.3

[a] Data provided from the company.

The main aim of this paper is to determine the accurate "time-zero" of autogenous shrinkage in HPC/UHPC. For revealing the autogenous shrinkage mechanism, the internal RH was monitored by improved hygrometer method at very early age (here refers to the stage before the final setting). Autogenous shrinkage of cement pastes was measured following ASTM C1698-09 [12]. For monitoring the stiffness development and hydration process of cement paste, ultrasonic pulse velocity measurement and isothermal calorimetry were performed.

2. Experimental investigation

2.1. Materials

The materials used in this study were Portland cement (CEM I 52.5N), and a polycarboxylate-based superplasticizer (Glenium 51, Solid mass content 35%). The properties of cement are shown in Table 1. The mineral composition of cement was calculated by the modified Bogue equation [19] as presented in Table 2. The particle size distribution of cement is shown in Table 3.

Four types of cement paste mixtures were made. The mixture proportion is listed in Table 4.

2.2. Methods

2.2.1. Autogenous deformation tests

The autogenous deformation of mixtures was measured following ASTM C1698-09 [12] standard developed by Jensen and Hansen [20], in which three sealed corrugated moulds of 440 mm (Ø28.5 mm) were tested for each mixture. After mixing, the fresh paste was carefully filled into the corrugated tube and sealed by plug and sealing glue. The specimens and test instrument were immersed in glycol in a box where the temperature was regulated at 20 ± 0.1 °C with the help of water bath. The autogenous shrinkage of specimens was recorded every 5 min by linear variable differential transformers (LVDTs). Fig. 2 shows the autogenous shrinkage test system. In all the tests, the autogenous deformation of parallel samples had a similar trend with a deviation of less than 50 microstrains.

2.2.2. Internal RH measurements

The internal RH inside the cement paste was monitored by two Rotronic hygroscopic DT station equipped with HC2-AW measuring cells, which is the same test method used by Jensen [21]. The dimension of the sample container is 30 mm, the thickness of sample for the test is

Table 2
The mineral composition of cement CEM I 52.5N, % by weight (calculated by the modified Bogue equation [19]).

Compound	C_3S	C_2S	C_3A	C_4AF
Weight (%)	63.77	9.24	8.18	9.13