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# On the relation of microstructure development and time- zero as the beginning of autogenous shrinkage

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## Abstract

In cementitious composites, the hydration process is the most important feature. The conditions, rate, and general manner of this process affect the properties of composites both at early and later ages. The development and prediction of future properties can be studied using both destructive and non-destructive methods to predict the potential and behaviour at later ages. Due to the early occurrence of autogenous shrinkage and the large influence of chemical shrinkage, the hydration process and the formation of the microstructure play an important role in determining the time zero point for autogenous shrinkage and the overall determination of the shrinkage value.

*Key words: hydration process, microstructure, properties development, autogenous shrinkage, time- zero*

## Odnos razvoja mikrostrukture i početka mjerenja autogenog skupljanja

### Sažetak

U cementnim kompozitima, proces hidratacije je najvažnija značajka. Uvjeti, brzina i opći način odvijanja procesa utječu na svojstva kompozita u ranoj i kasnijoj starosti. Razvoj i predviđanje budućih svojstava mogu se proučavati razornim i nerazornim metodama za predviđanje potencijala i ponašanja u kasnijoj dobi. Zbog rane pojave autogenog skupljanja i velikog utjecaja kemijskog skupljanja, proces hidratacije i formiranje mikrostrukture igraju važnu ulogu u određivanju nulte točke mjerenja autogenog skupljanja i ukupnom određivanju vrijednosti skupljanja.

*Ključne riječi: proces hidratacije, mikrostruktura, razvoj svojstava, autogeno skupljanje, time- zero*

## 1 Introduction

The development of concrete theory and technology has led to the development of cementitious materials with (ultra) high strength - (U)HS and (ultra) high performance - (U)HP [1, 2]. The novel materials have overcome the shortcomings of ordinary cementitious material but amplified certain negative aspects, such as shrinkage and resulting cracks [3 – 5]. (U)HSC and (U)HPC materials differ from OPC in having a lower water content, i.e., a lower w/c ratio, and a higher content of mineral and chemical additives as well as other inclusions. Therefore, this composition leads to differences in the hydration process that strongly affect the volume changes at early ages [6–8]. Shrinkage is visible as either a reduction in length or volume caused by moisture loss, temperature changes, or chemical reactions. The types of early- age shrinkage of cementitious materials are chemical, autogenous, drying, and thermal shrinkage. Chemical shrinkage can be quantitatively significant but occurs in the early stages of the hydration process when the microstructure is still fluid, while other types of shrinkage result in the formation of cracks.

The hydration process begins with the contact of the cement or binder with water, which leads to the dissolution of ions in the water and continues in the formation of hydrates [9–11]. These chemical reactions lead to the formation of a solid matrix and consequently to the release of heat [11, 12]. The formation of the structure is accompanied by the development of measurable mechanical properties such as strength and modulus of elasticity. Since mechanical properties develop over time and at a high rate at an early age, their measurement by destructive methods is difficult and is subject to long-term measurement by non-destructive methods.

In addition to mechanical properties, volume deformation must also be monitored over time. The measurement of autogenous shrinkage after demoulding proved to be too late, while the measurement after mixing contains parts of chemical shrinkage. Although there is general agreement on the theory of chemical and autogenous shrinkage and some test methods are standardized, to obtain realistic values of autogenous shrinkage, it is necessary to determine a time-zero to make the measurement reliable, i.e., to avoid over- or underestimation. Time- zero refers to the point in time when cementitious materials begin to behave like a solid, which can be referred to as the time of divergence between chemical shrinkage and autogenous shrinkage [13]. Although in standardized methods the time zero is determined by the setting time, which is determined by the penetration of a Vicat needle, several publications [14–19] have pointed out possible discrepancies related to the onset of the measurement and the uncertainty and unreliability of the proposed method, indicating that further studies on the microstructure development and on the internal relative humidity and temperature change are needed [20, 21].

In this work, a systematic literature review of test methods for monitoring microstructure development from the onset of hydration was conducted to compile existing test methods and evaluate their impact on the determination of time- zero and overall autogenous shrinkage.

## 2 Methods to test autogenous shrinkage

Autogenous deformation is the total deformation of a closed, isothermal system of cementitious material that is not exposed to external influences [13, 22, 23]. In this way, drying shrinkage and temperature-induced shrinkage are eliminated. Autogenous deformation is further divided into shrinkage and swelling. Autogenous swelling is a short-term phenomenon due to the formation of crystals from hydration products in the initial phase; it occurs when bleed water is reabsorbed [23]. The more water present, the greater the swelling and the longer it lasts [8]. Autogenous shrinkage occurs in all cement mixtures; in ordinary concrete it is of low value and without much effect, but in mixtures with (ultra) high strength and/or performance it has a greater effect. A reduced amount of water and an increased proportion of binder ( $w/b < 0.4$ ) or the proportion of cement replacement by mineral admixtures lead to faster water consumption and a greater occurrence of self-drying [4, 16]. The mechanisms of autogenous shrinkage can be reduced to three basic mechanisms: chemical shrinkage, hardened cement paste, microstructure development and self-desiccation or change of relative humidity in the pores [13, 22, 23].

Chemical shrinkage occurs in parallel with the hydration process because the initial volume of the material has a higher value than that of the hydration products. It is due to the difference in density between free and chemically bound water and non-hydrated cement and hydrates [13, 23]. The chemical shrinkage depends on the amount of basic constituents and their composition [5, 24].

The development of hydration leads to the binding of hydration products and the formation of a microstructure that develops mechanical properties that can resist volume reduction, resulting in the formation of pores. Due to the still high water content, the pores formed are filled with water. The space of free water is filled by hydration products and the volume difference leads to pore formation. The decrease in the degree of saturation and the resulting pressure in the pores cause the matrix to shrink. When the sample is isolated, there is no mass transfer with the environment. The process of decreasing the degree of saturation in the pores is called self-desiccation, and the resulting stresses and shrinkage are called autogenous shrinkage. Larger pores empty first, but the rate of water consumption in the pores during the hydration process also depends on the base constituents [5, 20]. Measurement of autogenous shrinkage can be performed on prismatic specimens according to the Tazawa guidelines [25] or on corrugated tube specimens according

to the ASTM standard [26]. Although the specimens are shaped differently, the test settings are made to reduce the frictional effect as much as possible and simulate the volume change by a linear change. For both methods, the start of the measurement is indicated by the setting time, Figure 1. According to standards, the setting time is determined by observing the penetration of a needle into the cement paste. Regardless of whether the test is manual or automatic, the time that elapses from the time of mixing until the needle reaches a certain value is given after three measurements for the initial and final setting time. The setting time is the time required for the hydration products to bind together and form a structure that resists penetration by the needle [11]. It is defined with initial and final time and can be measured on paste and mortar, while changes in aggregates are required at the concrete level. The negative aspects of testing the setting time with this method are the differences between the values in standards, the limited number of tests due to the size of the specimen, the disturbance of the specimen at each penetration, and, for measurements on concrete, the measurement on a sieved concrete mix due the limitations of using coarse aggregates.

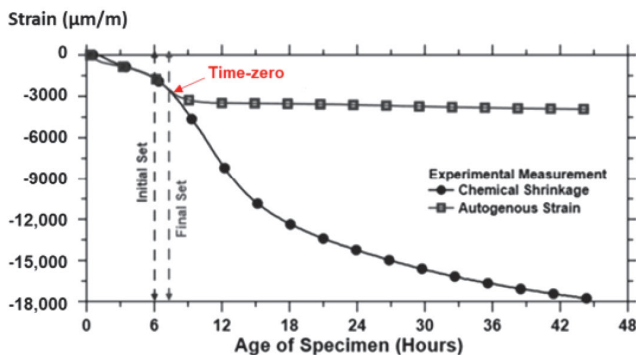


Figure 1. Time of divergence between chemical and autogenous shrinkage as shown in [19]

As the field developed, various authors noted discrepancies in the measurement of setting and autogenous shrinkage and therefore proposed other methods, such as the measurement of the change in internal relative humidity, temperature, time of maximum expansion and rate of change of shrinkage [14–20]. Each of these methods derives from the definition of autogenous shrinkage, but individually leads to different shrinkage values.

### 3 Monitoring the development of the microstructure

The term hydration, in the context of cement compositions, refers to the reaction of cement particles or one of their constituents with water, resulting in chemical and physical-mechanical changes in the system with respect to setting and hardening [10]. The water-cement or water-binder ratio has a significant influence, as these ratios control the microstructure of the hardened cement paste, both in the fresh and hardened state, and thus its rheology, mechanical properties, permeability, durability, and sustainability [6, 27]. The numerical values of w/c and w/b ratios are directly related to the distance between the particles in the hardened cement paste at the beginning of the hydration process and the space available for the formation of hydration products [6, 27]. According to work [9], the water/ cement ratio (w/c) of a hardened cement paste must be at least 0.42 to achieve complete hydration [9]. In a closed system, where there is no exchange with the environment, all the cement and water react to form cement gel and gel water, with 8 % of the volume formed as pores. If an external water source is present, the cement paste is also fully hydrated, but the pores formed are now filled with water. At a ratio greater than 0.42, the cement paste consists of the remaining capillary water, while at a ratio less than 0.42, hydration ceases due to the lack of water [9]. The water in the capillary pores has a positive effect by preventing self-desiccation and the occurrence of autogenous shrinkage, but has a negative effect on the mechanical properties, while unreacted cement particles improve the properties of the hardened cement paste as a hard inclusion with high compressive strength and high modulus of elasticity [12, 27]. At the mortar or concrete level, the amount, type, and distribution of aggregates also have some influence on the fresh and hardened properties, but less so on the hydration itself [28]. Gravity and environmental conditions affect the settling of solid particles and the bleeding of the corresponding water content depending on the w/c ratio and aggregate [29].

The course of hydration and its kinetics are influenced by numerous factors, such as the type, particle size distribution, and specific surface area of the cement, the amount of water, chemical and mineral admixtures, and temperature and humidity conditions [7, 27, 30]. Different types of cement are known depending on the additives, but all types of cement consist of four basic constituents: tricalcium silicate ( $C_3S$ ), dicalcium silicate ( $C_2S$ ), tricalcium aluminate ( $C_3A$ ) and tetracalcium aluminoferrite ( $C_4AF$ ) [12]. Since cement is a multiphase material, hydration occurs in a series of parallel chemical reactions with different kinetics [10, 12]. Hydration and structure development are dominated by the reactions of  $C_3S$  [10]. Silicates, which make up most of the volume of OPC, play an important role in hardening, i.e., in the rate of strength development:  $C_3S$  contributes to initial set and early strength gain, while  $C_2S$  contributes to later strength gain. Aluminate hydrates much more faster

than ferrite, but both contribute to space filling and chemical shrinkage as well as strength development [10, 24].

Paste hydration at ambient temperature is generally characterised by several phases: Pre- induction phase (first minutes), induction (dormant) phase (first few hours), acceleration phase (3 to 12 hours after mixing) and post- acceleration phase [11, 31]. Since hydration is an exothermic process, its progress can also be followed by the energy released in the form of heat. Data on the heat released by hydration are used to describe how the cement binds and hardens, and to estimate the temperature rise [11].

Monitoring the development of cement hydration is possible by testing calorimetry. Calorimetry is the measurement of heat and heat production rate most commonly used to study kinetics and extent of the hydration of cement and it is standardised by ASTM C1702 [32, 33]. Depending on the sample, we distinguish between isothermal calorimetry for small paste or mortar samples, semi-adiabatic calorimetry for larger concrete or mortar samples, fully adiabatic calorimetry for mass concrete, and solution calorimetry for samples 7 days and older [32]. In addition to sample differences, isothermal calorimeters measure thermal power (heat production rate), while (semi)adiabatic calorimeters measure temperature (change). Isothermal calorimetry is most often used in conjunction with other measurements such as ion concentration measurement, chemical shrinkage, internal relative humidity, XRD, TGA, and similar. Calorimetry also shows good correlation with the predictions of early age compressive strength and setting time [5, 34]. The initial setting time correlates well with the time at which the first derivative of the heat evolution curve  $[d(q)/d(t)]$  reaches its highest value, that is, the point at which the increase in the rate of heat generation is maximum, while the final setting time appears to correlate well with the time at which the first derivative is zero, or in other words the time at which the highest rate of hydration is achieved, Figure 2 [11, 35]. Although the work [27] concludes that laboratory testing of cement paste does not establish a reliable relationship between calorimetry and setting time, while the same relationship for mortar established a good correlation.

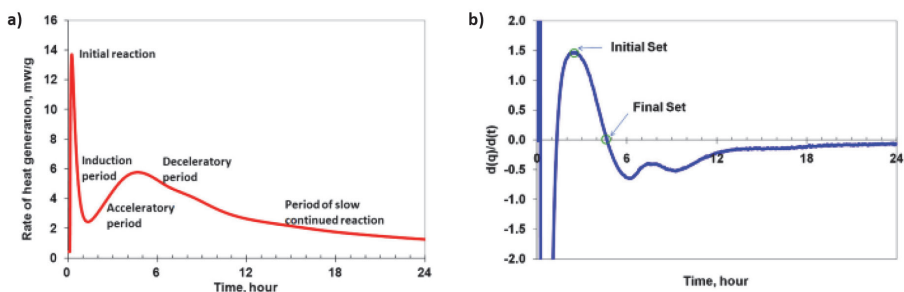


Figure 2. Graphical representation of the correlation between heat generation obtained with calorimetry and setting time; a) typical hydration curve, b) first derivative of heat generation rate [35]

Immediately after the binder comes into contact with water, the constituents rapidly dissolve and break down into ions-  $\text{OH}^-$ ,  $\text{K}^+$ ,  $\text{Na}_2^+$ ,  $\text{SO}_4^{2-}$ ,  $\text{Ca}^{2+}$ , etc [12]. Dissolution is visible in calorimetry as the first strong peak [10, 12]. The dissolution time of individual ions depends on their concentration, which in turn depends on the starting material and the presence of certain components in the cement [12]. In this way, the initial water passes into a solution with a certain concentration, which increases with time until supersaturation. The solution formed is defined as the alkaline solution present in the pores of the hardened cement. Ion concentration effects starting RH [20], while reduced alkali concentration in pore solution can result in retardation of the second exothermic peak [36]. The ion concentration for the same binder depends on the w/c ratio, i.e. on the amount of water by dilution or, if the w/c ratio is the same, on the composition of cement and additives. Over time, the actual concentration of the solution in the pores can be significantly affected by leaching, drying, or penetration of ions and other solutes [31].

Over a period of several hours, the speed of the process decreases, which may be caused by the type of cement or chemical and mineral additives [7]. The further reactions lead to the formation of a layer of C- S- H phase and ettringite, which is deposited on the cement particles [9,10]. Thereafter, there is a gradual acceleration of the process associated with setting when the formation of secondary C- S- H, portlandite, and ettringite begins [10]. The accelerated phase is accompanied by an increase in heat release and is therefore visible in calorimetry. This peak depends on cement type, water content and chemical and mineral additions [5]. The space occupied by the free water is now filled by the reaction with the hydration products, and if the filling does not occur, capillary voids are formed. The volume of capillary voids in hydrated hardened cement paste decreases with decreasing w/c ratio or increasing age. Crystalline structure of the hydration products is further tested with X- ray diffraction, while chemically bound water and pore volume and diameter are tested with thermogravimetry analysis and mercury intrusion porosimeter [32].

In addition to measuring the heat, the evolution of hydration can also be monitored with non- destructive methods, electrical resistivity and ultrasound. Electrical and ultrasonic methods can present the phenomena of percolation of solids or depolarization of water as an indicator of the critical time of transition from suspension to solid. The electrical response of the cement paste is an indicator of the development of its microstructure, when the process of development of resistivity is significantly influenced by the chemical and physical changes in the cement-water system [36]. The first stage is characterized by the first maximum of the rate, which is due to the dissolution of the cement grains; the second stage, which signifies the solidification, i.e., the formation of hydrates, and includes the initial and final setting times; and the third stage, in which the resistance continues to increase with increasing rate, corresponding to the conversion of AFt to AFm [36, 37]. As for the



ultrasonic measurements, the initial value of ultrasonic velocity after two hours is approximately 1500 m/s which is similar to the wave velocity through water [37]. Changes in velocity are determined as setting time.

After dormant period, the acceleration of the hydration process generates sufficient hydration products to promote the binding of the cement matrix and the formation of the initial structure [31]. As mentioned earlier, the newly formed matrix resists volume deformation and voids are formed. In addition to air voids, the pores are usually filled or nearly filled, except in the case of low  $w/c$ , which are subject to self-desiccation [6, 27]. The relationship between the formation of stresses,  $p_c$ , in the pores, their size and the relative humidity is described by the Laplace (1) and Kelvin (2) equations [16]:

$$p_c = -\frac{2 \cdot \sigma \cdot \cos \theta}{r} \quad (1)$$

$$p_c = -\frac{R \cdot T \cdot \ln(RH)}{V_m} \quad (2)$$

Where  $\sigma$  denotes the surface tension,  $\cos\theta$  the wetting angle,  $r$  the radius of the pore and  $R$  the gas constant and  $V_m$  the molar volume.

It has been shown that changes in internal relative humidity are a trigger for autogenous shrinkage and to have a major effect on behaviour [5, 16, 20, 38]. Another challenge is to study the pore distribution and size as well as the change in internal relative humidity. According to literature [20, 31, 32], mercury intrusion porosimetry (MIP) is still by far the most common method for estimating pore size distribution in cement pastes and concretes, but although it is simple enough to use, it is not the best pore representation due to certain limitations. The obtained results can be complemented by additional test like gas or water sorption, scanning electron microscopy image analysis or nuclear magnetic resonance [32]. In addition, the measurement of internal relative humidity can be affected by a large difference between the sample temperature and the ambient temperature, resulting in condensation and a false reading of the values [3,20]. Because of the sensitivity of the test methods, care should be taken when preparing specimens and performing the test, especially considering the temperature and humidity conditions of the room in which the test is performed.

## 4 Conclusion

Changes in the composition of cementitious materials, as well as the partial replacement of Portland cement with supplementary cementitious materials, whether reactive or inert, affect future cementitious properties due to changes in chemical structure and particle size distribution, as well as the hydration process and associated changes in microstructure formation. The effects range from changes in ion concentration in solution, to the hydration process, to the formation of structure and properties at later ages. All this affects the behaviour of the material in terms of volume deformation, especially autogenous shrinkage.

One of the most important cementitious material property is certainly setting time. Although the method for testing the initial and final setting is prescribed in the standard, there are still some divergences between values on different sample mixtures. However, there are several other ways to monitor the evolution of the microstructure and onset of setting. All of these changes can be accompanied by long-term destructive and non-destructive methods that, individually or in combination, provide a complete picture of the behaviour of the individual components. These tests allow a better understanding of the microstructure formation, which directly affects the behaviour of autogenous shrinkage, and determination of time zero.

Therefore, future testing and development of models to predict autogenous shrinkage behaviour must take into account these non-destructive long-term measurements in order to monitor the evolution of the structure as a function of the components used as correctly as possible. In this way, the obtained measurements and the predicted values will be related and comparable to the real elements.

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