Measurement and evaluation proposal of early age shrinkage of cement composites using shrinkage-cone

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Measurement and evaluation proposal of early age shrinkage of cement composites using shrinkage-cone

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Abstract. The paper deals with an experimental analysis focused on the determination and evaluation of the early age shrinkage strain using Shrinkage-Cone. The measurements were performed on the cement pastes and mortars with a different w/c ratio. The authors propose a new approach to the measurement evaluation based on the determination of time-zero at the time of estimated initial setting determined as the time when the internal temperature of specimens started to rise. In this case, also the effect of the thermal expansion is taken into account in the evaluation.

1. Introduction

The early as well as long-term shrinkage process in cement based composites is an actual problem especially of civil engineers and producers of cement based materials (concrete, mortars, floor compounds etc.). There are many standardized and non-standardized testing techniques which are used for estimation/determination of real value of shrinkage process during the whole time of material ageing, unfortunately the results are often inconsistent, especially because of different equipment, dimensions of test specimens and initial time of the start or evaluation of measurement [1, 2, 3]. Along the development of a high performance concretes and fine grained materials with a low water-cement ratio (w/c), the early age volume changes, caused by the chemical and autogenous shrinkage are studied in detail [4, 5]. The brief-summary of most frequently used testing procedures intended for the experimental measurement of the chemical and autogenous shrinkage can be found e.g. in [6]. Currently, three testing methods for early-age shrinkage measurement standardized by the ASTM are available - Standard test method for chemical shrinkage of hydraulic cement paste [7], Standard test method for autogenous strain of cement paste and mortar [8] and Standard test method for change in height at early ages of cylindrical specimens of cementitious mixtures [9]. The first method is usable for measurements on the pastes only, the second can be used also for the mortars and the third method is proposed to measure on the pastes, grouts, mortars and concretes.

Currently, dealers of the testing instruments are increasingly offering the Shrinkage-Cone. It is a test equipment intended especially for measurement of early age volume changes due to chemical and physical processes, such as the chemical and autogenous shrinkage and expansion [10]. The Shrinkage-Cone method is suggested to be an alternative method to above mentioned standard methods.

The main advantage of the Shrinkage-Cone method in comparison to the other above mentioned methods is the geometric shape of the mould used for measurement which ensures that the relative length and volume changes can be calculated simultaneously based on the measured changes in the height of
the cone-shaped specimen [10]. Other advantages are for example the sufficient measurable volume,
filling procedure and handling with the equipment.

The main aim of performed experiments was to verify the usability of the test method for fine-grained
materials of different consistency and to propose another approach to the evaluation of the measurement
using the Shrinkage-Cone equipment. Unfortunately, only small quantity of experimental data is
presently available which could be compared to each other. There were not found any data for the
measurement performed on the cement paste and also there were not found any results for measurements
performed on the cement pastes with different w/c tied together with the measurement on the cement
mortars.

2. Experimental analysis

2.1. Materials

The measurements were performed for cement pastes and equivalent cement mortars with a different
w/c ratio. For manufacturing of both composites CEM I 52.5 R Portland cement was used. Three
different w/c ratios for pastes with values of 0.33, 0.40, 0.50 and two different for mortars with values
of 0.40 and 0.50 were chosen for the experiment (it is not possible to prepare mortar with w/c = 0.33
without the addition of plasticizer). The standardized quartzite sand with a grain size distribution of
0–2 mm (CEN 196-1 [11]) was used for mortars manufacturing. The aggregate-cement ratio in mortar
mixtures was always 3:1. The mixing procedure of the prepared fresh mixtures was based on
the standard EN 196-1 [11]. Three measurements were performed for each paste and mortar to obtain
the average curve of the strain development.

2.2. Shrinkage Cone

The Shrinkage-Cone is measurement equipment, which allows to measure shrinkage and expansion of
the building materials, which are prepared by mixing of solid and fluid [10] components to prepare
a fresh mixture that harden due to the chemical processes. The touch-less measurement system allows
to start the measurement immediately after the fresh material is poured into the mould. It is important
to specify that the geometric shape of the cone-shaped mould ensures that the change of the recorded
distance (change in the height of the cone-shaped specimen) corresponds to the length change of the test
specimens because the ratio of the radius and height is constant for any volume (the relative length
change can be then calculated). Based on this fact, the volume change of the measured material can be
calculated as the third power of the ratio of the respective heights. For further information about
the Shrinkage-Cone and details about the measurement procedure refer to the technical sheet of
equipment available in [10].

For the purpose of the experiment, the shrinkage cone with a nominal height of 125 mm and volume
of 682 cm$^3$ was used. To minimize the friction, the cone-shaped Teflon foil (PTFE) was placed into
the cone before its filling. During the pouring, the appropriate method of compaction (respecting
the workability) was applied to remove the entrapped air and to ensure the required rigidity and smooth
top surface of the fresh mixture. To avoid the water evaporation, the top surface was coated by the oil
(according to the manufacturer recommendation). Finally, the reflector made of PP coated with
the aluminum sheet was placed in the middle of the top surface and the temperature-sensitive sensor
was inserted into the fresh mixture. The manufacturing process is illustrated in figures 1–6 below.
The measurement data was automatically stored in data loggers with a period of 10 second and 1 minute
in the case of distance and temperature measurement, respectively.

The strain-curves presented herein represent relative length changes calculated from the ratio of
$\Delta h/h$ in $\%$, where $\Delta h$ is the measured change in height (a change in the height of the specimen) and $h$
is the nominal height of the cone-shaped mould. The presented strain curves represent the average value
obtained from three measurements performed for each cement paste and mortar.
3. Results and discussion
The results of the pilot study are presented in figures below. Before the results are discussed, it is important to specify that the descending trend of the $\varepsilon$-curves indicates the vertical expansion and theirs ascending trend indicates vertical shrinkage. For the purpose of comparison of the results with the standardized methods or other measurement methods based on the measurement of the changes in length of the test specimens, only the changes in height of Shrinkage-cone are presented in the article. Based on this outputs, the volumetric change can be then calculated.

Figure 7 and Figure 8 show the results of measurements performed on cement pastes. Note, that the consistency of cement pastes with $w/c = 0.5$ was quite slurry. The strain displayed in Figure 7 was calculated as an overall deformation obtained during the whole time of measurement. As is shown in the figure, the cement paste with $w/c = 0.33$ started to expand immediately after the start of measurement in contrast to the two remaining pastes, which started to shrink slightly for approx. 0.5 hours and after
that time they also started to expand. The rapid increase in expansion was slowed down in the estimated
time of the initial setting time (the time at which the internal temperature started to rise), between 2.5
and 3.5 hours after start of measurement. When the internal temperature reached its maximum, all
cement pastes which were investigated started to shrink, which occurred 6.5, 8.0 and 9.0 hours after
the start of measurement for cement pastes with w/c = 0.33, 0.40 and 0.50 respectively.

It is necessary to realize that the behaviour of the material before the time of initial setting is strongly
influenced by the stability and consistency of the fresh mixture. The early phenomena are strongly
affected by the bleeding, settlement of the particles, air bubbles etc. The initial “shrinkage” can also
indicates the settlement of reflector placed on the upper surface of the fresh mixture. For the purpose of
measurement evaluation, Eppers and Mueller [12, 13] proposed the time-zero which corresponds to
the initial setting time determined by an alternative procedure employing an automatic needle
penetration apparatus. In their study, the time-zero of UHPC was setup between 5 to 6 hours [12]. When
respecting their approach, the whole period of expansion (if exists) would be neglected.

A different approach is proposed in this study. The authors propose the time-zero at the time of
the estimated initial setting determined as the time when the internal temperature of specimens started
to rise. In this case, also the effect of thermal expansion can be taken into account in the evaluation.

![Figure 7. Calculated strain of cement pastes with time-zero at start of measurement.](image)

The results with a new time-zero are presented in Figure 8. Using this approach, approx. 1/5 to 1/6
of the initial expansion is taken into account in the measurement evaluation. The maximum value of
shrinkage that comes after the initial expansion does not exceed the value of initial expansion. This is
quite an important fact from the microstructure-cracking formation point of view.
Figure 8. Calculated strain of cement pastes with time-zero at the estimated initial setting time.

The results obtained from testing of the cement mortars are displayed in Figure 9 and Figure 10. Two ways of evaluation was also performed. Figure 9 displays the evaluation of the strain with time-zero corresponding to the start of measurement. Quite different behaviour, compared to the cement pastes, was observed. As is shown in figure, both cement mortars started to shrink immediately with the maximum value reached approx. 1 hour after the start of measurement. A higher value of shrinkage was recorded for the mortar with a higher w/c ratio. After the initial shrinkage, both cement mortars started to expand slightly. When the time-zero is moved to the estimated time of mortars’ initial setting the whole part of the initial shrinkage is neglected (refer to Figure 10) and the evaluated strain started with expansion which turned to shrinkage at the time when the internal temperature of both mortars reached the maximum value. In this case, the shrinkage did not exceed the expansion. It is worth investigating in detail the factors which caused the initial surge of shrinkage which occurred during the first hour of measurement.

Figure 9. Calculated strain of cement mortars with time-zero at start of measurement.
4. Conclusion

Results presented in the paper are a part of the relatively extensive experimental and numerical analysis under a project aimed at the early-age characteristics of cementitious materials. In this project, the Shrinkage-Cone is considered to be a suitable piece of equipment for the measurement and evaluation of the chemical and autogenous deformation of the material during the early solidification process. This measurement device is also considered to be a supplemental measurement to the measurement of chemical shrinkage using the procedure in standard [7] and therefore the measurements were performed on the cement pastes.

The results of performed measurement showed low variability and sufficient sensitivity to measured characteristics. It was proved that this equipment is suitable for measurement of fresh mixtures of a high range of workability. In this context, the appropriate compaction method has to be applied to achieve the proper results with low variability. To achieve the low variability, it is important to ensure that the manufacturing and pouring process as well as the handling with the equipment is the same in all test cases. Nevertheless, there are still open procedural issues that can significantly affect the final measurement results. The measurements performed on the cement pastes with high fluidity can be affected with procedural errors caused by using an inappropriate method of covering the upper surface of the test specimen. The usage of the PP reflector for the measurement is also questionable.

Acknowledgments

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