INFLUENCE OF MINERAL ADDITIONS AND CHEMICAL ADMIXTURES ON SETTING AND VOLUMETRIC AUTOGENOUS SHRINKAGE OF SCC-EQUIVALENT-MORTARS

Gert Heirman (1), Lucie Vandewalle (1), Dionys Van Gemert (1)

(1) Reyntjens Laboratory, Department of Civil Engineering, K.U.Leuven, Belgium

Abstract
The influence of mineral additions and chemical admixtures on both setting and volumetric autogenous shrinkage of SCC-equivalent mortars is studied by means of respectively the Vicat needle and the “condom” method. All experiments are performed in the framework of a MSc thesis at the Reyntjens Laboratory. [1]

The investigated parameters are: the type of mineral addition (limestone powder, quartz powder and fly ash), the type of cement (Portland cement CEM I 52.5 R HES and blast furnace slag cement CEM III/A 42.5 N LA) and the applied dosage of chemical admixture (i.e. a PCE-superplasticizer). Besides the SCC-equivalent-mortars (SCM), two traditional concrete-equivalent-mortars (TCM), for each type of cement, are studied.

1. INTRODUCTION

The expression “autogenous shrinkage” was first mentioned in literature in 1934. [2] Since then, however, extensive research on this phenomenon has not been conducted until the beginning of the 1990’s, when it became more and more important by the use of high strength concrete mixtures, due to the application of low water/cement ratios, (super)plasticizers and mineral additions. Because the same mix ingredients are also present in powder-type self-compacting concrete mixtures, autogenous shrinkage is also a concern in this type of cement-based material.

Autogenous shrinkage occurs over three different stages immediately after the addition of water: (1) the liquid stage, where the autogenous shrinkage is equivalent to chemical shrinkage, (2) the skeletal formation (setting) stage, where a more rigid structure is formed due to the stiffening of the paste and, as a consequence, the concrete can resist some of the chemical shrinkage stresses and (3) the hardening stage. During the hardening stage, the
Concrete shrinkage can be measured using more standard long-term measuring practices. [3] In order to indicate the different stages in function of time, the initial and final setting time of the investigated mixtures are determined.

Since a lot of different definitions of autogenous shrinkage are reported in literature, it is necessary to denote the terminology used in this paper: “the volumetric autogenous shrinkage (also called ‘external chemical shrinkage’) is the volume change in mortar dimensions when hardening under isothermal, sealed – with regard to moisture exchange – conditions”. [4]

2. MIX COMPOSITIONS AND MIXING PROCEDURE

2.1 Mix compositions

In order to reduce the amount of concrete mixtures made, concrete equivalent mortars (CEM) can be used to study the influence of mineral additions and chemical admixtures on concrete properties mainly influenced by its paste behaviour. Those mortars are defined by replacing the course aggregates (gravel 4/14) content of the original concrete mix by an equivalent amount of fine aggregates (river sand 0/5) in such a way that the total specific surface (S.S.) of the aggregates is equal for both cases. [5]

It was found that 100 kg of the gravel (S.S. = 0.403 m²/kg) should be replaced by 7.55 kg of the sand (S.S. = 5.338 m²/kg), resulting in the following conversions for the traditionally vibrated (TC) and self-compacting (SCC) concrete mixes:

- For TC: 1225 kg/m³ gravel + 640 kg/m³ sand → 732 kg/m³ sand
- For SCC: 698 kg/m³ gravel + 853 kg/m³ sand → 906 kg/m³ sand

As chemical admixture, a 3rd generation superplasticizer based on polycarboxylate ether (PCE) is used. In table 1, the mix compositions of the traditional vibrated (TCM) and self-compacting (SCM) concrete equivalent mortars are given.

2.2 Mixing procedure

The mixtures were mixed according to the CEM mixing procedure, using a Hobart mortar mixer according to EN 196-1:2005. First, the sand is humidified (mixing at low speed during 2 minutes) by an extra added amount of water equal to its water absorption coefficient, determined according to EN 1097-6:2000, increased with 1%. A water absorption coefficient of 3% for the used sand was found, resulting in an extra amount of water equal to 4% of the sand content added to the mixtures (explicitly mentioned in table 1). The humidified sand is left in the paused and covered (to avoid evaporation) mixer for 5 minutes.

Next, all powder materials (cement and mineral addition) are added and mixed at low speed for 30 seconds. Afterwards, water and PCE are added and mixed at low speed for 30 seconds, immediately followed by another 30 seconds mixing at medium speed. Then mixing is paused for 30 seconds, while the bottom of the bowl and the paddle are scraped off. As final step, mixing continued for 1 minute at medium speed.
Table 1: Mix compositions of CEM mixtures tested [kg/m³]

<table>
<thead>
<tr>
<th>CEM mixtures with PC</th>
<th>TCM1</th>
<th>SCM1</th>
<th>SCM2</th>
<th>SCM3</th>
<th>SCM4</th>
<th>SCM5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand 0/5</td>
<td>732</td>
<td>906</td>
<td>906</td>
<td>906</td>
<td>906</td>
<td>906</td>
</tr>
<tr>
<td>CEM I 52.5 R HES</td>
<td>360</td>
<td>360</td>
<td>360</td>
<td>360</td>
<td>360</td>
<td>360</td>
</tr>
<tr>
<td>Mineral addition</td>
<td>-</td>
<td>240 (LS)</td>
<td>240 (LS)</td>
<td>240 (LS)</td>
<td>240 (Q)</td>
<td>240 (FA)</td>
</tr>
<tr>
<td>Water + sand humidification</td>
<td>165 + 29</td>
<td>165 + 36</td>
<td>165 + 36</td>
<td>165 + 36</td>
<td>165 + 36</td>
<td>165 + 36</td>
</tr>
<tr>
<td>PCE content [m-%]</td>
<td>-</td>
<td>0.5</td>
<td>0.8</td>
<td>1.0</td>
<td>0.8</td>
<td>0.8</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>CEM mixtures with BFSC</th>
<th>TCM2</th>
<th>SCM6</th>
<th>SCM7</th>
<th>SCM8</th>
<th>SCM9</th>
<th>SCM10</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sand 0/5</td>
<td>732</td>
<td>906</td>
<td>906</td>
<td>906</td>
<td>906</td>
<td>906</td>
</tr>
<tr>
<td>CEM III/A 42.5 N LA</td>
<td>360</td>
<td>360</td>
<td>360</td>
<td>360</td>
<td>360</td>
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</tr>
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</tr>
<tr>
<td>PCE content [m-%]</td>
<td>-</td>
<td>0.5</td>
<td>0.65</td>
<td>0.8</td>
<td>0.8</td>
<td>0.8</td>
</tr>
</tbody>
</table>

PC: Portland cement, BFSC: blast furnace slag cement; both according to EN 197-1:2000
Mineral additions: LS: limestone powder, Q: quartzite powder, FA: fly ash

3. TEST METHODS

3.1 Setting
The setting of the mortars is determined by the penetration resistance, using the Vicat needle according to EN 196-3:2005. Attention must be paid on possible segregation of the SCMs tested, because the denser layer caused by the segregated sand can disturb the interpretation of the test results.

3.2 Volumetric autogenous shrinkage
The test method used for volumetric autogenous shrinkage measurements at early age is based on Archimedes’ principle. [4] It consists of measuring, by hydrostatic weighing, the buoyancy variations of a sample constituted by an elastic rubber (latex) membrane filled with the mortar (about 400-500g) and immersed in a water bath at constant temperature (20°C). The mortar is introduced into the membrane immediately after mixing and the membrane is closed using a thin steel wire. The excess rubber is then cut, the knot is sealed with a hydrophobic silicone paste and the sample is carefully cleaned and weighed. Thereafter, the sample is hung on a balance, while it is completely immersed in the water bath.

The greatest care has been exercised in the evacuation of all air bubbles inside the membrane since they can distort the results. Besides, attention is paid on avoiding surface air bubbles on the membrane when immersing in the water bath. In order to avoid distortion of the results by the presence of bleeding water [6], mixtures where chosen with little or no bleeding water.

4. TEST RESULTS AND DISCUSSION

4.1 Setting time
Both initial and final setting times of each CEM mixture are given in figure 1.
Influence of PCE content on setting time of CEM mixtures with PC (SCM1-2-3)

An increase of the PCE content has a decelerating effect on the setting time of the CEM mixtures with PC, due to the elongation of the dormant period in the hydration process. The deceleration can be explained by different phenomena:

− The PCE molecules adsorbed on the hydrating phases (C₃A) act as a barrier for the diffusion of Ca²⁺-ions and retard the formation of hydration products. [7,8]
− The PCE molecules change the morphology of the hydration products formed by reducing growth. [7]
− The carboxyl groups of the PCE molecules interact with the Ca²⁺-ions in the pore water, resulting in a slowing down of the precipitation reactions. [8]

The lower setting time for SCM3 is due to segregation: the denser layer formed by the segregated sand particles results in a penetration resistance not directly related to setting.

Influence of PCE content on setting time of CEM mixtures with BFSC (SCM6-7-8)

The same decelerating effect on setting time is found as with the CEM mixtures with PC, although mixtures SCM7 and SCM8 disturb this conclusion due to some segregation. When the differences in setting time between SCM6-TCM2 and SCM1-TCM1 are compared, it can be seen that the use of BFSC results in a higher setting time deceleration. This difference can be explained by the fineness of the cement (as indicated by their Blaine value: 4740 cm²/g for PC and 3190 cm²/g for BFSC): assuming that the same amount of PCE will cover the same surface of cement grains in both cases, more “uncovered surface” is found for the finest PC, resulting in a smaller influence.

Influence of mineral addition on setting time (SCM2-4-5//SCM8-9-10)

It is found by others [9-10] that the addition of limestone powder has an accelerating effect on the cement hydration of PC and so on the setting time. On the other hand, the addition of PCE has a decelerating effect on the setting time (see above). These two effects seem to counteract each other when comparing TCM1 and SCM1. Nevertheless, it was also found by [10] that the addition of quartzite powder has no significant influence on the cement hydration of PC, so SCM4 and SCM9 can be used as a reference when comparing with other SCM mixtures having the same PCE content. With this prospect, the following observations can be made: independent of the cement type used and compared to the quartzite addition, the addition of limestone powder and fly ash have respectively a moderate and strong accelerating effect on the setting time, due to modifications of the cement hydration. [10]
4.2 Volumetric autogenous shrinkage

The volumetric autogenous shrinkage of each CEM mixture is represented in figure 2.

![Graph of Volumetric Autogenous Shrinkage](image)

Figure 2: Volumetric autogenous shrinkage of CEM mixtures with PC (left) and BFSC (right)

**Influence of PCE content on autogenous shrinkage (SCM1-2-3//SCM6-7-8)**

Since autogenous shrinkage and hydration are independent properties [11], analogies with the setting time can be found. Increasing the PCE content has a decelerating effect on the volumetric autogenous shrinkage of the CEM mixtures. For the CEM mixtures with PC, an asymptotic and comparable volumetric autogenous shrinkage is observed after 48 hours, while the CEM mixtures with BFSC did not reach a plateau level for autogenous shrinkage within 48 hours. This observation can be explained by different phenomena:

- BFSC, having the lowest specific surface can be assumed having a more effective dispersion, resulting in a higher volumetric autogenous shrinkage. [12]
- During hydration reactions of BFSC, Ca(OH)$_2$ is consumed in order to produce CSH. As a consequence, a higher free water reduction is found compared to PC hydration reactions, resulting in a stronger self-desiccation effect, and so, in higher volumetric autogenous shrinkage. [13]

**Influence of mineral addition on autogenous shrinkage (SCM2-4-5//SCM8-9-10)**

For the CEM mixtures with PC, a comparable autogenous shrinkage is found after 48 hours for both limestone (SCM2) and quartzite powder (SCM4), although the quartzite addition resulted in an increased autogenous shrinkage rate during the liquid stage. An explanation can be found in the shortening of the induction period and the accelerated hydration reactions due to the limestone addition. [10] The addition of fly ash resulted in a comparable (to quartzite addition) autogenous shrinkage during liquid and setting stage but at the end of the setting stage, the fly ash addition resulted in a temporary increased autogenous shrinkage rate, probably due to its pozzolanic reactions, resulting in a stronger self-desiccation effect and so, in a higher volumetric autogenous shrinkage.

Replacing PC by BFSC, a different behaviour of the mineral additions is found. It seems that the higher Ca(OH)$_2$ content and the higher “efficiency” of PCE (due to the lower specific surface of BFSC) overshadowed the influence of the mineral addition used.

5. CONCLUSIONS

Based on the test results, the following conclusions are drawn:
− Increasing the PCE content has a decelerating effect on the setting time of CEM mixtures with PC, due to the elongation of the dormant period, while it has an even more decelerating effect on the setting time of CEM mixtures with BFSC, due to its lower specific surface, resulting in a higher “efficiency” of the PCE.
− Assuming no influence of quartzite addition on the cement hydration, it was found that limestone and fly ash addition have respectively a moderate and strong accelerating effect on the setting time compared to quartzite addition, independent of the cement type used.
− All self-compacting CEM mixtures (SCM) showed a lower volumetric autogenous shrinkage compared to traditionally vibrated CEM mixtures (TCM), due to their denser microstructure.
− The influence of the PCE content on the volumetric autogenous shrinkage is in total conformity with its influence on the setting time.
− The influence of the mineral additions on the autogenous shrinkage was found to be in agreement with the occurring hydration reactions for CEM mixtures with PC. PC replacement by BFSC seems to overshadow the influence of the mineral addition used.

REFERENCES