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The influence of superabsorbent polymers (SAPs) on autogenous shrinkage in cement paste, mortar and concrete

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HIGHLIGHTS

- SAP in fluids swell within 10 min and size (40–100 μm) does not affect swelling.
- SAP particle size has no influence on flow, strength, setting, autogenous shrinkage.
- SAP swelling in cement filtrate may not predict well effect on flow for concrete.
- Shrinkage mitigation by SAP may be smaller in mortar than in cement paste.
- Even incomplete shrinkage mitigation by SAP will affect concrete cracking.

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ABSTRACT

Due to the high amount of fines in combination with a low water-to-cement ratio (<0.35), high performance mortar and concrete are very prone to cracking as a result of autogenous shrinkage, resulting in a decreased durability, integrity and aesthetics of the structure. Superabsorbent polymers (SAPs) can be added as internal curing agents to reduce the autogenous shrinkage in cementitious materials. In this paper, the influence of SAP addition on mitigating autogenous shrinkage in cement paste, high performance mortar and high performance concrete is investigated. Two different types of SAPs were added in varying amounts: one sulfonate based SAP with specifically selected properties, and a commercially available poly-acrylate based SAP. To study the effect of the SAP addition on the mitigation of autogenous shrinkage, corrugated tube tests in case of cement paste and mortar, and restrained ring tests for concrete were performed. The poly-acrylate based SAPs reduced the autogenous shrinkage after 7 days in the mortar mixtures with 97% compared to the reference without SAPs, whereas in the cement paste, the autogenous shrinkage after 7 days was completely mitigated. Although the mixtures with sulfonate based SAPs did not show complete mitigation of the autogenous shrinkage, the shrinkage was significantly reduced for all cement pastes: increasing the amount of SAPs from 0.257 m% to 0.38 m% and 0.57 m% by weight of cement, lead to a reduction in the autogenous shrinkage after 7 days with 80%, 85% and 89% respectively. In mortar the reductions for the same amounts of SAPs were 19%, 20% and 70% respectively. Considering restrained shrinkage ring tests, the poly-acrylate based SAPs reduced the occurring strains significantly (-88%) compared to the reference and prevented the rings from cracking. For the sulfonate based SAPs, the moment of cracking was delayed and lower strains compared to the reference were observed.

1. Introduction

Shrinkage of cementitious materials like cement paste, mortar and concrete is inevitable and has an undesirable effect on the durability, aesthetics and structural integrity of a structure. When shrinkage, especially early-age shrinkage, is restrained, cracking of the material can occur resulting in an impaired durability. These cracks can create a network of flow paths for water and gases possibly containing harmful substances which can cause for example corrosion of reinforcement steel, chemical attack and internal expansive reactions. In the end, the structure has a reduced durability, integrity and aesthetics which should be avoided [1].

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Early-age shrinkage in cementitious materials immediately starts after cement and water come in contact and hydration is initiated [2]. The development of this type of shrinkage can be divided in three stages: the liquid stage, the stage in which a skeleton is formed and finally the hardened stage [3]. The first two stages are referred to as the induction period while the latter corresponds with the phase after setting of the cementitious material [2]. During hydration, pores are created in the hydration products due to chemical shrinkage. As long as the cementitious material is in the liquid stage, the material has such a low viscosity that these pores are removed and there is no concern regarding stress generation. In the next stage, when the skeleton starts to develop, the formed pores are sustained and a pore network is developed in which water can be transported [4]. This causes the formation of water menisci in the pores resulting in capillary pressure and plastic shrinkage. Finally, in the last stage, self-desiccation is the driving force of autogenous shrinkage. During cement hydration, water is consumed which leads to a reduction of the relative humidity (RH) when no additional water is provided. Consequently, the cementitious material starts to dry from the inside [3,4]. The contribution of the change in capillary pressure to the autogenous shrinkage can be explained as follows: self-desiccation empties the pores and consequently water menisci in the capillary pores are created causing tension in both the pore water and at the surface of the cementitious material. Due to these tensile stresses, the cement matrix shows more contraction which results in a volume reduction and thus shrinkage [1,5,6].

The existence of autogenous shrinkage has been discovered at the start of the twentieth century but only gained more recognition in the last two decades as it was found that at early age, autogenous shrinkage results in the most significant volume change in (ultra)high performance mortar ((U)HPM) and (ultra)high performance concrete (U)HPC compared to other types of shrinkage [7,8]. In general, (U)HPC is more prone to autogenous shrinkage in comparison with other concrete due to a high amount of cement and sometimes other fine supplementary cementitious materials like blast furnace slag or silica fume, in combination with a low water-to-cement ratio (often lower than 0.35), leading to a very dense microstructure [9]. As autogenous shrinkage is influenced by both the water content and the pore size, it is of no surprise that the combination of the low water content together with small pores in (U)HPC makes this concrete more susceptible to autogenous shrinkage [10,11]. Crack formation due to autogenous shrinkage is therefore a frequently occurring problem in (U)HPM and (U) HPC. Especially in the first 24 h after mixing, early-age autogenous shrinkage is of great importance as at this point in time the cementitious material has not yet fully developed its maximum strength capacity and there may be a period when the occurring stresses exceed the actual strength, resulting in crack formation. Although the cracks formed in this stage are mainly small micro-cracks, these cracks can become wider under later-age shrinkage (i.e. shrinkage after 24 h) and impair the strength and durability of the structure to a higher degree. Therefore, measuring, reducing or even fully mitigating autogenous shrinkage is of great importance when using cementitious materials, especially in case if (U) HPM and (U)HPC.

Internal curing can be applied for mitigating autogenous shrinkage and the basis of this method is the release of water in the cementitious matrix during hydration of the cement when the relative humidity starts to drop and self-desiccation is initiated. There exists a wide variety of different internal curing techniques [12–19]: e.g. the use of light weight aggregates (LWA), pumice, expanded clay or superabsorbent polymers (SAPs). In this paper the use of superabsorbent polymers as internal curing agent to mitigate autogenous shrinkage in different cementitious materials is studied.

SAPs form 3D polymer networks that can retain up to thousand times their own weight in liquids, without dissolving [10]. When these micro-size polymers come in contact with a fluid like water, they will swell and form a hydrogel. When the relative humidity of the environment drops, the SAPs can gradually release the stored fluid and de-swell to its initial dimensions and shape. There exists a wide variety of SAPs with differences in type (natural, semi-synthetic, synthetic), chemical charge (ionic and non-ionic), dimensions (from micrometres to nanometres), degree of cross-linking and thus swelling capacity, polymerization technique (suspension or bulk), shape (spherical, irregular), swelling kinetics, etc. [11,20–24]. All these different parameters can be tuned to obtain the 'ideal SAP' for a specific application or purpose. A lot of different applications of SAPs in cementitious materials exist, with the common goal to increase the durability of the structure: mitigating autogenous shrinkage [25–33], self-sealing and self-healing of cracked cementitious materials [34–40], increasing the freeze–thaw resistance of concrete [41,42], etc. When SAPs are added to cementitious materials, they will take up part of the mixing water. If no additional water is added, this could negatively affect the workability of the cementitious material. The amount of additional water to compensate for this water uptake by the SAPs is often determined based on a trial and error method in order to obtain the same workability as the reference mixture [30,43–43]. As this is a labor and material intensive method, it is studied in this paper whether the determination of the amount of additional water can be based on the swelling capacity of the SAPs in cement filtrate solution obtained through a filtration test. In this way, resources can be saved, as the filtration method requires less material, labour and time compared to the method of equal workability.

The principle of SAPs as internal curing agent to mitigate autogenous shrinkage in cementitious materials is the following: at the specific moment in the hydration process when the relative humidity starts to drop and self-desiccation develops, the water in the SAPs will be released to the surrounding cement matrix, maintaining the RH and as a result minimize the self-desiccation. In this way autogenous shrinkage can be reduced or even completely mitigated [11]. In literature the amount of SAPs used to mitigate autogenous shrinkage is rather low, namely in the range of 0.2 m% - 0.7 m% dry SAP by cement weight [45–48] and mainly the use of SAPs with smaller average particle size (<200 μm) is reported [26,49]. Some authors describe partial reduction of the autogenous shrinkage [43,47], whereas other authors make notice of full mitigation of the autogenous shrinkage upon SAP addition [43,45,48].

In literature, different test methods for measuring (autogenous) shrinkage in cement paste, mortar or concrete can be found and are mainly divided into two groups: volumetric methods and linear methods [50]. An examples of a volumetric shrinkage measurement method is 'the balloon method' for cement paste [51]. Examples of linear shrinkage measurement methods are the corrugated tube test according to ASTM C 1698-09 [52] for cement paste and mortar [12,43,47,53], the use of demountable mechanical strain gauge (DEMEC) measurements for mortar and concrete [54,55], LVDT measurements on beams in vertical or horizontal direction for mortar and concrete [50], the use of embedded sensors in cementitious materials [55–58], the restrained ring test according to ASTM C 1581-04 [59] for concrete, etc. As all these test methods have their specific domain of application, test parameters and conditions, a comparison of the results from different test methods is not straight forward and should be executed with care and criticism. In this paper, it is chosen to use the corrugated tube test according to ASTM C 1698-09 for the cement paste and mortar mixtures and the restrained ring test according ASTM C 1581-04 for the concrete mixes. By choosing the corrugated tube test as the method to measure autogenous shrinkage, some disadvantages compared to other methods, like the loss of moisture, possible longitudinal restraints and difficulties in handling and measuring the specimens before hardening, are discarded, since the paste or mortar is completely sealed within the tubes and can be monitored continuously from the moment the tubes are filled and positioned in the measuring device with LVDTs, and without the need for handling. As it is not recommended to use the corrugated tube test for concrete specimens due to the limited dimensions of the tubes [60], another method is chosen for the concrete mixtures, namely the restrained ring test as this method is standardised (ASTM C 1581-04) and is well documented and analysed in literature [61].

Next to the choice of the test method(s) to measure the autogenous shrinkage, also the time from which the autogenous shrinkage is taken into account must be chosen and measured with an appropriate test. This time, often called time zero, is taken as the time between the initial water contact with the cement and the time where a skeleton is developed in the cementitious material. Several definitions of this time zero and the way how to determine it are reported in literature [7,60,62–64] and still a lot of discussion is going on about this topic. In this paper, the time zero to start the autogenous shrinkage measurements is taken as the final setting time measured with an automated Vicat needle test, as this is the prescribed test in the standard ASTM C 1698-09 and hence often used in literature [14,43,52].

In this paper, the influence of SAP addition on mitigating autogenous shrinkage in cement paste, HPM and HPC is investigated. By adding SAPs in various cementitious materials, it is studied whether SAPs perform differently in the tested materials and if trends and conclusions for one material can be transferred to other materials or not. For this purpose, two different types of SAPs in varying amounts are added to the tested cementitious materials: one SAP with selected properties (cross-linking degree and particle size), made by the Belgian chemical R&D company ChemStream (see also the parameter study on the selection of this specific SAP by the author [65]) and a commercially available SAP from BASF of which the efficiency to mitigate autogenous shrinkage is already reported in literature [14]. To study the effect of the SAP addition on the mitigation of autogenous shrinkage corrugated tube tests in combination with an automated Vicat needle test in case of cement paste and mortar, and restrained ring tests for concrete are performed.

2. Materials and methods

2.1. Materials

2.1.1. SuperAbsorbent polymers (SAPs)

In this paper, two different types of SAPs are studied. The first type of studied SAPs was developed and produced by the Belgian chemical R&D company ChemStream bvba (CS). The SAPs were formed through a copolymerization of sodium vinyl sulfonate (SVS) with 2-acryloylamino-2-methyl-propane-1-sulfonate (NaAMPS) using potassium persulfate (KPS) as thermal initiator and were obtained by a radical bulk polymerization reaction carried out at 70 °C. The amount of cross-linker N,N′-methylenebisacrylamide (MBA) was 1.0 mol% with respect to the monomer. The obtained SAPs were ground to particle sizes with d_{50} of 40 or 100 μm using a RETSCH ZM200 centrifugal mill.

The second type of studied SAP was a commercially available SAP developed by the company BASF and with the commercial name Starvis. This SAP was synthesized by bulk polymerization creating the copolymer poly (acrylamide-co-acrylic acid) out of the monomers acrylamide and sodium acrylate and had a mean particle size of 100 μm. The main properties of the SAPs like producer, mean particle size d_{50}, synthetization technique, cross-linking density, bulk density and amount of solubles are summarized in Table 1.

The code of the used SAPs starts with SAP_ followed by CS or BASF if the SAPs were made by the company ChemStream or BASF, respectively. The code ends with the mean particle size d_{50} in μm, namely 40 or 100 μm.

2.1.2. Cementitious materials

In this paper, three different series of cementitious materials were made:

Series 1: cement paste;
Series 2: mortar;
Series 3: concrete.

The code of the studied mixtures start with P_, M_ or C_ if the mixture is a cement paste, mortar or concrete, respectively. Next, the code contains REF for the reference mixture without SAPs. For the mixes with SAPs, the code contains CS or BASF if SAPs from the company ChemStream or BASF are used, followed by the amount of SAPs added expressed in m% by cement weight. The code is ending with the mean particle size of the used SAPs in μm (i.e. 40 or 100). E.g. The mixture with code P_REF is the reference cement paste mixture (without SAPs). The mixture with code P_CS_0.56_40 is a cement paste mixture with 0.56 m% SAPs by cement weight of the company ChemStream with a mean particle size of 40 μm.

2.1.3. Cement paste

A reference cement paste mixture P_REF with a water-to-cement ratio (w/c) of 0.3 was composed of Portland cement (CEM I 52.5 N, HOLCIM) and tap water. A constant dosage of 0.42 m% of cement weight polycarboxylate superplasticizer (Glenium 51, conc. 35%, BASF) was added to obtain a flow of 300 ± 20 mm 10 min after initial water contact. The workability of the fresh cement paste mixtures was measured by a flow test according to the standard NBN EN 1015-3 [66], see methods section.

In case SAPs were used, an additional fixed amount of extra water w/c_{extra} = 0.054 for a w/c ratio of 0.3 was added to mitigate autogenous shrinkage, based on a reinterpretation of Power's model and calculated with formula (1) [11]:

$$(w/c)_{extra} = 0.18(w/c) for w/c \leq 0.36 \quad (1)$$

In a first step, the amount of SAPs needed to absorb this amount of extra water was determined based on a trial and error method in order to obtain the same workability as the reference mixture (i.e. 300 ± 20 mm 10 min after water contact). The period of 10 min was chosen to allow the SAPs to reach most of their swelling capacity.

Table 1
Characteristics of the studied SAPs.

Name SAP	Producer	Particle size d_{50} [μm]	Synthetization technique	Bulk density [kg/m³]	Cross-linking density [mol%]	Amount of solubles [%]
SAP_CS_40	ChemStream	40	Bulk polymerization	1400	1.0	28
SAP_CS_100	ChemStream	100	Bulk polymerization	1400	1.0	28
SAP_BASF_100	BASF	100	Bulk polymerization	1400	–	< 1

-: this information was not provided by the producer

In a second step, the amount of extra water (and as a result also the amount of added SAPs) was first increased with 50% to 0.081 to see the influence of this higher amount of internal curing water on autogenous shrinkage. Ultimately, the amount of extra water (and SAPs) was raised again with 50% to 0.1215. The final composition of the tested cement pastes is summarized in Table 2. As the added additional water should be released after setting as internal curing water, it was not included in the effective w/c-ratio $w/c_{effective}$ but is shown separately in Table 2 [67].

To prepare the SAP-containing cement paste mixtures, first the dry components (cement and SAPs) were mixed for 30 s at low speed (140 rpm) to ensure a homogeneous distribution of the SAPs in the cement. The rest of the mixing procedure was performed according to standard NBN EN 196-1 [68], including mixing of water and cement (and dry SAPs) for 60 s at 140 rpm, addition of extra water and superplasticizer, mixing at higher speed (285 rpm) first for 30 s and an then for an additional minute after a short resting time.

The total mixing time (after initial water contact) was 4.5 min. Six prismatic moulds with dimensions 40 × 40x 160 mm^3 were filled for the determination of the flexural and compressive strength at 7 and 28 days. Subsequently, the specimens were vibrated for 30 s on a vibrating table. Next, the specimens were covered with plastic foil and stored at a temperature of 20 ± 2 °C for 48 h. At the age of 2 days, the specimens were demolded, wrapped in plastic foil and subsequently stored at standard laboratory conditions (20 ± 2 °C) until the age of testing.Also three corrugated tubes for the determination of autogenous shrinkage were filled. To limit the formation of air bubbles during filling of the tubes, the tubes were filled on a vibrating table.

2.1.4. Mortar

In order to have a more pronounced autogenous shrinkage, the type of cement was changed from CEM I to CEM III [69–72]and the w/c was lowered from 0.30 to 0.24 [11] in combination with a high amount of binder. In this way, the possible effects of the SAPs in mitigating autogenous shrinkage would be more visible in the test results.

A reference high performance mortar mixture M_REF with a w/c of 0.24 and a water-to-binder ratio (w/b) of 0.20 was composed of the cement Variodur40 (CEM III/A 52.5 R, with 35–64% clinker, 36–65% blast furnace slag (BFS) and 0–5% minor additional constituents, DYCKERHOFF), sand 0/4, Microsilica 940 U (bulk density between 200–350 kg/m^3, ELKEM), filler Betofill VK50 (Franzefoss Minerals) and 1.44 m% by cement weight of superplasticizer ViscoCrete UHPC-2 (SIKA). For the mixtures containing SAPs from ChemStream, the same amounts of SAPs as for the cement pastes were used (i.e. 0.257, 0.38 or 0.57 m% by cement weight). In case of the commercial SAPs from BASF, the used amount of SAPs was somewhat increased to 0.3 m%, as this amount is reported in literature to efficiently mitigate autogenous shrinkage [14]. The amount of extra water to be added in case of SAP containing mixtures to compensate for their water uptake was this time not obtained through the labour and material intensive method of equal workability, but was based on the swelling capacity of the SAPs in cement slurry after 10 min, as it was found to result in the same outcome, see results section. The swelling capacity taken into account for the ChemStream SAP and BASF SAP was 21 g/g SAP and 27 g/g SAP, respectively.

The composition of the studied reference mortar mixture can be found in Table 3. For reasons of practicability, the mortar composition was kept constant except for the extra amount of water; i.e., the amounts of solids were not adjusted, but the extra water was added on top of the reference mix. The used amount of water, extra water, total water, SAPs, the $w/c_{effective}$, the w/c_{extra} and the w/c_{total} for the different mortar mixtures are summarized in Table 4.

The mixing procedure of the mortar mixes was as follows:

0–30 s mixing dry components (and dry SAPs) (speed 140 rpm);
30–60 s adding water while mixing (speed 140 rpm);
60–90 s adding sand while mixing (speed 140 rpm);
90–120 s adding SP and extra water (in case of SAPs) while mixing (speed 140 rpm);
120–300 s mixing (speed 140 rpm);
300–360 s mixing (speed 285 rpm);
360–420 s scraping and resting;
420–600 s mixing (speed 285 rpm).

The total mixing time was 10 min. Six prismatic moulds with dimensions 40 × 40x 160 mm^3 were filled for the determination of the flexural and compressive strength at 7 and 28 days. Due to the use of the superplasticizer ViscoCrete UHPC 2 from SIKA, the mixtures were self-compacting and no further vibration was needed. Next, the specimens were covered with plastic foil and stored at a temperature of 20 ± 2 °C for 48 h. At the age of 2 days, the specimens were demolded, wrapped in plastic foil and subsequently stored at standard laboratory conditions (20 ± 2 °C) until the age of testing. Also three corrugated tubes for the determination of autogenous shrinkage were filled. To limit the formation of air bubbles during filling of the tubes, the tubes were filled on a vibrating table.

2.1.5. Concrete

A reference HPC mixture C_REF with a w/c of 0.24 and a w/b of 0.20 was composed of the cement Variodur40 (CEM III/A 52.5 R, with 35–64% clinker, 36–65% blast furnace slag (BFS) and 0–5% minor additional constituents, DYCKERHOFF), sand 0/4, Basalt 4/8, Microsilica 940 U (bulk density between 200–350 kg/m^3, ELKEM), filler Betofill VK50 (Franzefoss Minerals) and 1.44 m% by cement weight superplasticizer ViscoCrete UHPC-2 (SIKA).

For the mixtures containing SAPs from ChemStream, the same amounts of SAPs as for the cement pastes and mortars were used (i.e. 0.257, 0.38 or 0.57 m% by cement weight). In case of the commercial SAPs from BASF the amount of SAPs was 0.3 m%, i.e. the same amount as used in the mortar mixtures. The amount of extra water to be added in case of SAP containing mixtures was again based on the swelling capacity of the SAPs in cement slurry after 10 min, namely 21 g/g SAPs for the ChemStream SAP and 27 g/g SAP for the BASF SAPs. In case of SAP containing mixtures, the amount of superplasticizer was increased with 0.1 m% by cement

Table 2
Final composition of the tested cement pastes.

	Type of cement	$w/c_{effective}$ [−]	w/c_{extra} [−]	w/c_{total} [−]	Superplast Glenium 51 BASF [m% by weight of cement]	SAP [m% by weight of cement]
P_REF	CEM I 52.5 N	0.3	–	0.30	0.42	–
P_CS_0.257_40	CEM I 52.5 N	0.3	0.054	0.354	0.42	0.257
P_CS_0.257_100	CEM I 52.5 N	0.3	0.054	0.354	0.42	0.257
P_CS_0.38_40	CEM I 52.5 N	0.3	0.081	0.381	0.42	0.38
P_CS_0.57_40	CEM I 52.5 N	0.3	0.1215	0.4215	0.42	0.57
P_BASF_0.20_100	CEM I 52.5 N	0.3	0.054	0.354	0.42	0.20

Table 3
Composition of the reference mortar mixture.

Component	Amount [kg/m^3]
Variodur 40 (CEM III/A 52.5 R, DYCKERHOFF)	1030.5
Microsilica 940 U (ELKEM)	203.7
Betofill VK 50 (FRANZEFOSS MINERALS)	245.6
Water	246.8
Sand 0/4	532.2
Superplasticizer Viscocrete UHPC 2 (SIKA)	14.8*

* 1.44 m% by cement weight.

Table 5
Composition of the tested reference concrete mixture.

Component	Amount [kg/m^3]
Variodur 40 (CEM III/A 52.5 R, DYCKERHOFF)	778.2
Microsilica 940 U (ELKEM)	153.8
Betofill VK 50 (FRANZEFOSS MINERALS)	185.5
Water	186.4
Sand 0/4	401.9
Basalt 4/8	649.3
Superplasticizer Viscocrete UHPC 2 (SIKA)	11.2 *

* 1.44 m% by cement weight.

weight to a total of 1.54 m% to have a slump flow of at least 550 mm, so the tested concretes could be classified as self-compacting concrete.

The composition of the reference concrete mixture can be found in Table 5. For reasons of practicability, the concrete composition was kept constant except for the extra amount of water; i.e., the amounts of solids were not adjusted, but the extra water was added on top of the reference mix. The used amount of water, extra water, total water, SAPs, the $w/c_{effective}$, the w/c_{extra} and the w/c_{total} for the different concrete mixtures are summarized in Table 6. The concrete mixtures were made in 35 L batches.

The mixing procedure of the concrete mixes was as follows:

0–60 s mixing dry components (also SAPs if used);

60–120 s adding water while mixing;

120–360 s adding SP and extra water (in case of SAPs) while mixing;

360–420 s scraping and resting;

420–600 s mixing;

600–720 s adding 0.1 m% extra superplasticizer in case of SAP mixtures while mixing.

The total mixing time was 10 min for the reference mixture and 12 min for SAP-containing mixtures. Due to the use of the superplasticizer ViscoCrete UHPC 2 from SIKA, the mixtures were self-compacting and no further vibration was needed.

3. Methods

3.1. Swelling capacity

The swelling capacity of the SAPs in demineralized water and cement filtrate solution was determined by the filtration method described in the RILEM TC-RSC recommendation [73]. Therefore, approximately 100 g of fluid was added to around 0.15 g dry SAP particles (exact mass of fluid and SAPs to be recorded). After a certain amount of time, the SAP particles were filtered using filter paper with a particle retention size of 12–15 μm. In order to exclude possible absorption of the fluid by the filter paper, the latter was first saturated with the test fluid. To minimize evaporation during the test, the test setup was covered with a lid. After filtration, the amount of fluid that was not absorbed by the SAPs, was recorded. The test was performed 10 min, 1 h and 24 h after the SAPs came in contact with the test fluid. The test was performed in triplicate for each time period.

The cement filtrate solution was obtained by mixing 100 g cement (CEM I 52.5 N for the SAPs used in cement pastes and CEM III/A 52.5 R for the SAPs used in the mortar and concrete mixes) in one litre of demineralized water for at least 24 h, followed by filtration with filter paper with a particle retention size of 12–15 μm.

The swelling ratio, i.e., the amount of fluid that can be absorbed by 1 g of SAPs can be calculated by formula (2):

$$\text{Swelling ratio}\ [\text{g fluid/g SAP}] = \frac{w_{\text{fluid added}} - w_{\text{fluid not absorbed}}}{w_{\text{dry SAP}}} \quad (2)$$

with

$w_{\text{fluid added}}$ [g]: the amount of fluid before filtration;

$w_{\text{fluid not absorbed}}$ [g]: the amount of fluid that was not absorbed by the SAPs;

$w_{\text{dry SAP}}$ [g]: the amount of dry SAPs.

3.2. Workability cement paste and mortar

The workability of the fresh cement paste and mortar mixtures was measured by a flow test according to the standard NBN EN 1015-3 [66]. The flow of each mixture was measured 10 min after water contact (to allow the SAPs to swell completely) on a spread flow table. In case of some cement paste mixtures and mortar mixtures, also the flow over time was studied, to ensure that the SAP would not release the entrained water prematurely, or on the contrary keep absorbing water over a long period of time leading to reduced workability before casting. Therefore, the flow was measured directly after mixing and also at different time intervals afterwards, for example at 10 min, 15 min, 30 min and 60 min after water contact. Before performance of the flow test, the mixture was first mixed again for 30 s at low speed (i.e. 140 rpm). The tests were carried out once per mixture and time interval.

3.3. Workability concrete

The workability of the fresh (self-compacting) concrete mixtures was measured by a slump flow test according to the standard NBN EN 12350-8 [74]. The test was performed directly after mixing

Table 4
Amount of SAPs, water and water-to-cement ratios for the tested mortar mixtures.

[kg/m^3]	SAPs [m% by cement weight]	SAPs [kg/m^3]	Water [kg/m^3]	Extra water [kg/m^3]	Total water [kg/m^3]	$W/C_{effective}$ [–]	W/C_{extra} [–]	W/C_{total} [–]
M_REF	–	–	186.4	–	186.4	0.24	–	0.24
M_CS_0.257_40	0.257	2.000	186.4	42*	228.4	0.24	0.054	0.294
M_CS_0.38_40	0.38	2.957	186.4	62*	248.4	0.24	0.081	0.321
M_BASF_0.30_100	0.30	2.335	186.4	62**	248.4	0.24	0.081	0.321
M_CS_0.57_40	0.57	4.358	186.4	91.5*	277.9	0.24	0.1215	0.3615

* determined based on the swelling capacity in cement filtrate solution after 10 min of the CS SAPs of 21 g/g SAP.
** determined based on the swelling capacity in cement filtrate solution after 10 min of the BASF SAPs of 27 g/g SAP.

Table 6
Amount of SAPs, water and the water-to-cement ratios of the tested concrete mixtures.

[kg/m^3]	SAPs [m% by cement weight]	SAPs [kg/m^3]	Water [kg/m^3]	Extra water [kg/m^3]	Total water [kg/m^3]	$w/c_{effective}$ [−]	w/c_{extra} [−]	w/c_{total} [−]
C_REF	–	–	186.4	–	186.4	0.24	–	0.24
C_CS_0.257_40	0.257	2.000	186.4	42*	228.4	0.24	0.054	0.294
C_CS_0.38_40	0.38	2.957	186.4	62*	248.4	0.24	0.081	0.321
C_CS_0.57_40	0.57	4.358	186.4	91.5*	277.9	0.24	0.1215	0.3615
C_BASF_0.30_100	0.30	2.335	186.4	62**	248.4	0.24	0.081	0.321

* determined based on the swelling capacity in cement filtrate solution after 10 min of the CS SAPs of 21 g/g SAP.
** determined based on the swelling capacity in cement filtrate solution after 10 min of the BASF SAPs of 27 g/g SAP.

(i.e. around 10 min after water contact for the reference mixture and around 12 min after water contact for the SAP-containing mixtures). The test was carried out once per mixture. The minimum slump flow value of concrete to be classified as self-compacting concrete equals 550 mm (class SF1).

3.4. Flexural and compressive strength cement paste and mortar

The flexural strength was determined by means of a three-point-bending test on three cement paste/ mortar prisms with dimensions of 40 × 40 × 160 mm^3. The compressive strength was measured on the halves resulting from the bending test. Both tests were performed according the standard NBN EN 196-1 [68] with a testing machine Walter + Bai DB 250/15. The mechanical properties were tested at 7 and 28 days after casting. For the flexural strength, three replicates per age were tested. For the compressive strength 6 replicates per age were tested.

3.5. Compressive strength concrete

The compressive strength of the concrete mixtures was measured on cast cubes with sides of 100 mm according to standard NBN EN 12390-3 [75]. The specimens were cured for 24 h in a climate controlled room with a temperature of (20 ± 2)°C and a minimum RH of 90%. After 24 h, the specimens were demolded and stored under the same conditions until the age of 28 days. The compressive strength was determined at the age of 28 days in triplicate.

3.6. Final setting time cement pastes and mortar: automated Vicat

The Vicat needle test by means of an automated Vicat apparatus was used to measure the final setting time, also called 'time-zero', of the tested cement pastes and mortar mixtures. The test was executed according the standard NBN EN 196-3 [76]. The time of final setting was defined as the time where the needle of the apparatus penetrates the surface of the sample only 0.5 mm, i.e. the time where the specimen is fully hardened. The test was carried out once per mixture and with an accuracy of 15 min.

3.7. Autogenous shrinkage cement pastes and Mortar: Corrugated tubes

The autogenous shrinkage of the tested cement pastes and mortar mixtures was monitored continuously over a time period of 7 days by means of a corrugated tubes test following the standard ASTM C 1698-09 [52]. A flexible corrugated mould and a dilatometer, namely an automatic Linear Variable Differential Transducer (LVDT) with a range of 5 mm and an accuracy of 5 μm, were the basis of this test. The shape of the test setup and the shape of the mould allowed the sample to shrink or expand freely without restraint during hardening while avoiding moisture loss as much as possible. A picture of the test up is depicted in Fig. 1. For each mixture, three tubes were tested. The final setting time obtained through a Vicat test was used as the beginning of the shrinkage measurements.

From the measurements, the autogenous strain can be calculated using Formula (3).

$$\mu_{autogenous}(t) = \frac{L(t)\text{-}L(t_{fs})}{L\ (t_{fs})}10^6 = \frac{I(t)\text{-}I(t_{fs})}{L\ (t_{fs})}10^6 \quad (3)$$

With:
$\varepsilon_{autogenous}$ (t)[μm/m]: autogenous strain at time t;
$L(t_{fs})$ [mm]: the length of the specimen at final setting;
L(t) [mm]: the length of the specimen at time t after final setting;
$I(t_{fs})$ [mm]: the LVDT reading at final setting time;
I(t) [mm]: the LVDT reading at time t after final setting.

3.8. Autogenous shrinkage Concrete: Restrained ring test

To measure the restrained shrinkage of the concrete mixtures, a restrained ring test based on the standard ASTM C 1581-04 [59]

Fig. 1. Autogenous shrinkage test setup: corrugated tubes with LVDTs.

was performed. In this test, a steel ring instrumented with two or three strain gauges was filled with concrete directly after concrete mixing. In the first tests, only two strain gauges were attached to the inner ring. Later, the setup was slightly changed and three strain gauges were mounted on the inner steel ring to obtain more data per ring. A schematic overview (left) and a picture (right) of the ring test setup is depicted in Fig. 2. For each concrete mixture, one ring was filled. The measurements started within 10 min after casting the concrete into the ring and were zeroed at that time. After casting, the bolts, see Fig. 2, were loosened and the rings were completely sealed with plastic foil to prevent evaporation. After 24 h, the outer steel ring was removed and the rings were again completely wrapped in plastic foil to minimize drying shrinkage. The strain of the specimens was measured every ten minutes from casting onwards, until the age of seven days if cracks appeared during this period or up to 28 days if no cracks appeared. A sudden change in strain was an indication of cracking of the test specimen. Small jumps in the strain were due to vibrations in the vicinity of the test setup. The rings were also visually checked on a daily basis to see whether cracks appeared or not. The rings were kept in a climate-controlled room at 20 ± 2 °C and 60 ± 5% relative humidity.

4. Results and discussion

4.1. Swelling capacity of SuperAbsorbent polymers (SAPs)

The swelling capacity expressed in g/g SAP of the three tested SAPs after 10 min, 1 h and 24 h of immersion in demineralised water obtained with the filtration test is depicted in Fig. 3.

The swelling capacity in demineralised water of the commercial SAPs from BASF, namely around 250 g/g SAP, is about four times higher than the swelling capacity of the ChemStream SAPs, which is in the range of 60–66 g/g SAP. Besides the difference in chemistry, also the difference in amount of cross-linker is the reason for this, but no information on this is provided by BASF. The lower the amount of cross-linker, the higher the swelling capacity, as the cross-links will impede the SAPs from swelling [43,65,77]. Also the relative high amount of solubles in the ChemStream SAPs (i.e. 28% compared to < 1% for BASF SAPs) could have a contribution to this phenomenon, as this material is in fact not contributing to the 'active' SAPs, but is just present as not-cross-linked particles in the SAPs [22,78–80]. The results of the ChemStream SAPs with mean particle size 40 μm and 100 μm were not significantly different (α = 0.05) concerning the swelling capacity in demineralised water. This result is in accordance with findings in literature [65,77,78]. Also the swelling capacities of the SAPs after 10 min, 1 h and 24 h after water contact are found not to be significantly different (α = 0.05), for none of the studied SAPs. The studied SAPs take up most of the water within the first 10 min after water contact.

In this study, two different types of cement were used, namely CEM I 52.5 N (used for the cement paste specimens) and CEM III/A 52.5 R (used for the mortar and concrete specimens). To see whether the used cement type has an influence on the swelling capacity of the SAPs, the swelling capacity was tested in cement filtrates made with both cements. The results after 10 min, 1 h and 24 h immersion in cement filtrate are depicted in Fig. 4. For reason of readability, the standard deviations are not depicted in the figure.

The results show that the swelling capacity in cement filtrate is much lower than the swelling capacity in demineralised water as a result of the charge screening effect of cations like K^+, Na^+, Mg^{2+} and Ca^{2+} present in cement filtrate. Divalent cations like Mg^{2+} and Ca^{2+} will reduce the swelling capacity in cement filtrate even more as they will form strong complexes with the sulfonate groups present in the ChemStream SAPs and therefore act as cross-linkers [77,81,82]. The commercial BASF SAPs have somewhat higher swelling capacity in cement filtrate than the ChemStream SAPs, although this difference is not so pronounced as was the case for demineralised water. After 24 h immersion in cement filtrate, the swelling capacity of all SAPs in both cement filtrates is in the range of 19–23 g/g SAP. In Fig. 4, it can be seen that the swelling capacity of the BASF SAPs increases after 1 h immersion compared to the swelling after 10 min immersion, but decreases again after 24 h of immersion in cement filtrate. For the ChemStream SAPs it can be concluded that most of the swelling of these SAPs occurred within the first 10 min after contact with cement filtrate, as the swelling capacity only increases slightly after 1 h and 24 h. This is interesting to know as the mixing procedure of the mortar and concrete takes around 10 min. When comparing the results for the swelling capacity in cement filtrate made with CEM I or CEM III, it is found that the differences are not significant (significance level 5%). Results also show that the difference in particle size (ChemStream SAPs with particle size of 40 μm versus 100 μm) does not have a significant influence on the swelling capacity in cement filtrate, as was also the case for swelling in demineralized water.

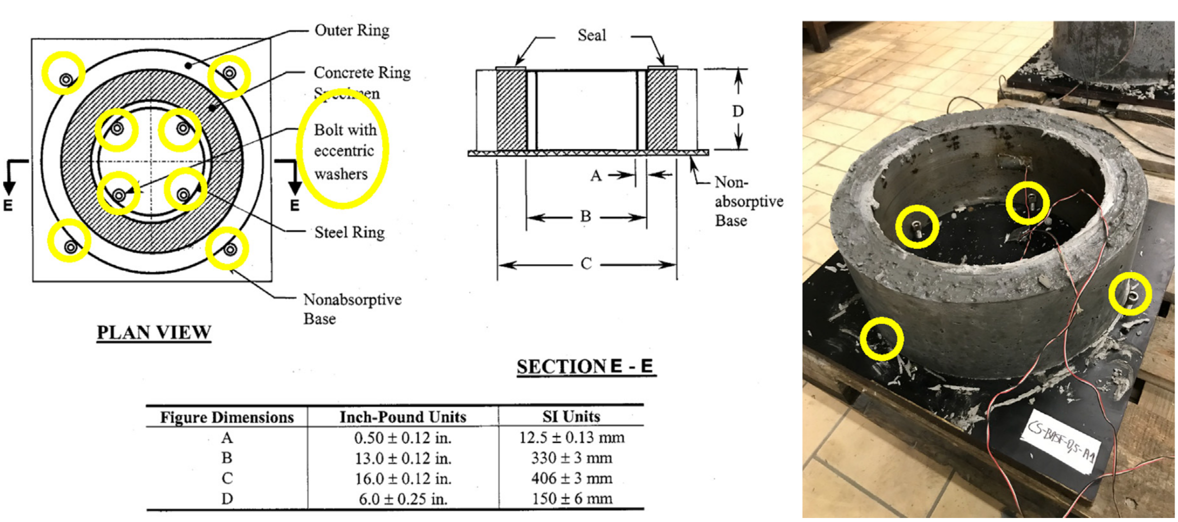

Figure Dimensions	Inch-Pound Units	SI Units
A	0.50 ± 0.12 in.	12.5 ± 0.13 mm
B	13.0 ± 0.12 in.	330 ± 3 mm
C	16.0 ± 0.12 in.	406 ± 3 mm
D	6.0 ± 0.25 in.	150 ± 6 mm

Fig. 2. Schematic view of the restrained ring test (left) [59] and the actual test setup (right). The yellow circles indicate the position of the bolts. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

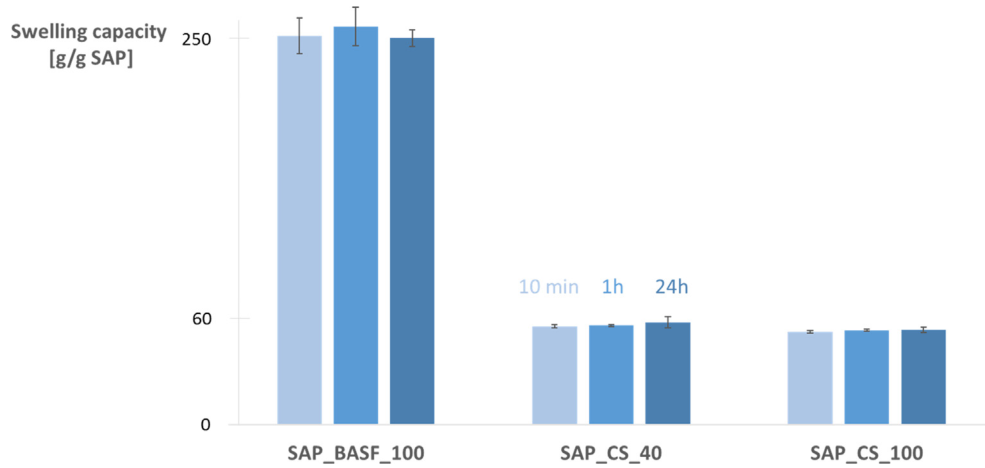

Fig. 3. Average swelling capacity and the standard deviation in demineralized water [g/g SAP] after 10 min, 1 h and 24 h of immersion, obtained with the filtration test.

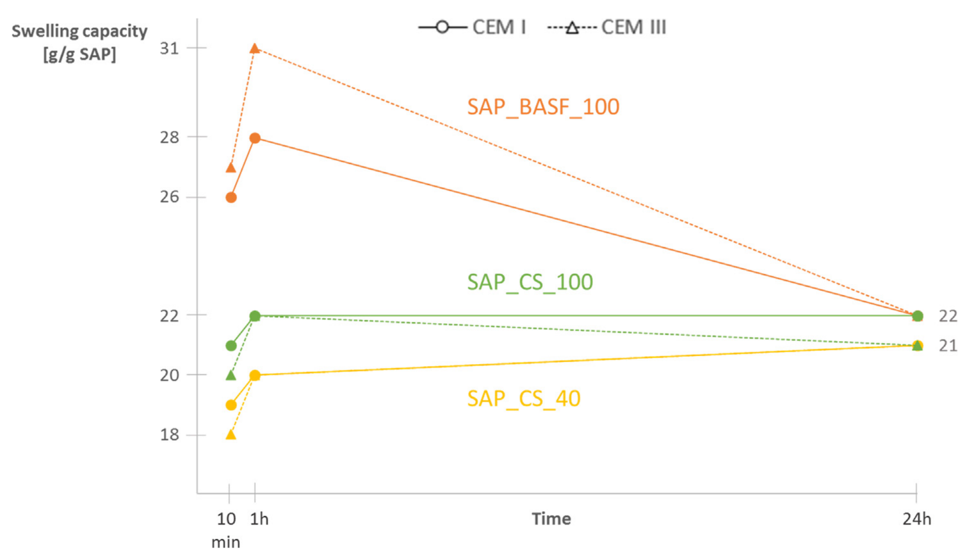

Fig. 4. Swelling capacity in cement filtrate made with CEM I 52.5 N or CEM III/A 52.5 R after 10 min, 1 h and 24 h, obtained with the filtration test.

4.2. Cement paste

In literature [11,44], it is described that an extra amount of water of w/c_{extra} = 0.054 for a w/c = 0.3 should be added as internal curing water to mitigate autogenous shrinkage. In this study, this extra water is initially absorbed by the SAPs and is released after setting as internal curing water. The amount of SAPs needed to absorb this extra water was determined based on a trial and error method in order to obtain the same workability as the reference mixture, i.e. 300 ± 20 mm after 10 min after water contact. The period of 10 min was chosen to allow the SAPs to reach most of their swelling capacity. After a time and material intensive procedure to find the correct amount of SAPs to have the same flow as the reference, the following amounts of SAPs were found: 0.20 m% by cement weight for the commercial BASF SAP and 0.257 m% by cement weight for the ChemStream SAPs (both 40 μm and 100 μm). The initial flow and flow over time of the reference and the final three optimised SAP-containing mixtures can be seen in Fig. 5. The initial flow (i.e. directly after mixing or 5 min after water contact) of the reference mixture was 300 mm and decreases after 60 min after water contact to 245 mm. Also the SAP-containing mixtures show a similar decrease in flow after 60 min, to a value around 240 mm. It can be concluded that all mixtures, including the reference, show a loss in flow. However, after 60 min the workability of all mixtures is similar. This means that the SAPs do not release the absorbed water within the first 60 min after water contact.

At this point, the amount of SAPs needed to absorb the extra amount of water of w/c_{extra} of 0.054 is known. Therefore, the swelling capacity of the SAPs, based on the amounts of SAPs determined by the method of equal workability can be calculated. The calculated swelling capacities of the SAP_BASF_100, SAP_CS_100 and SAP_CS_40 were equal to 27 g/g SAP, 21 g/g SAP and 21 g/g SAP, respectively. If these values are compared with the experimentally determined swelling capacities obtained through the filtration tests, it can be seen that these values correspond well with the swelling capacities in cement slurry after 10 min. It was therefore decided that for the rest of this study, the amount of SAPs needed will be based on the experimentally determined swelling capacities from the filtration test in cement slurry after 10 min.

The mean compressive strength of the different cement paste mixtures at the age of 7 and 28 days with their standard deviation are summarized in Table 7. Also the total water-to-cement ratio w/c_{total} is given as well as the compressive strength reduction relative to the reference sample without SAPs P_REF (between brackets) using the following colour code:

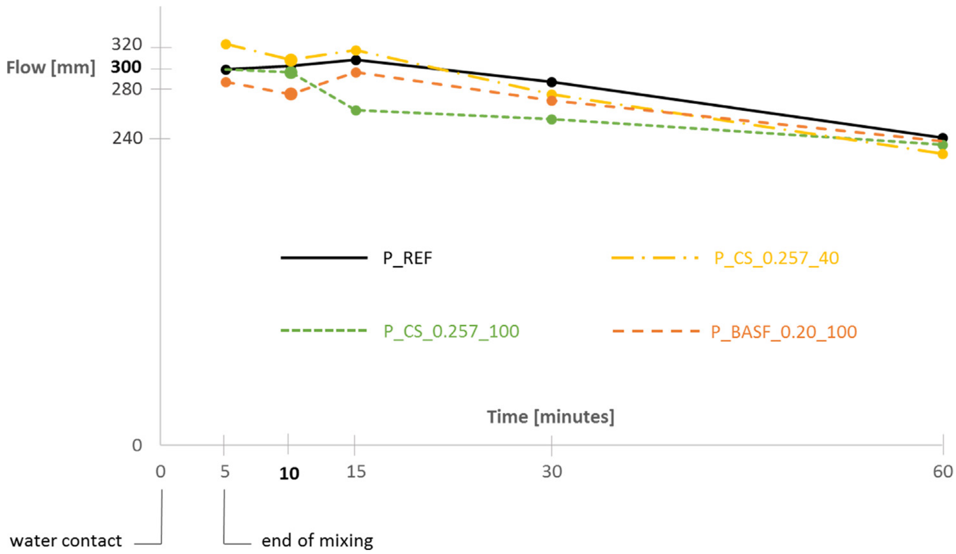

Fig. 5. Initial flow and flow over time [mm] of the reference paste mixture and the SAP paste mixtures.

Table 7
Average and standard deviation of the compressive strength of the cement paste mixtures at 7 and 28 days [MPa], the total water-to-cement ratio and the strength reduction in % compared to the reference.

	w/c_{total} [-]	Compressive strength [MPa]	
		7 days	28 days
P_REF	0.300	94 ± 5	106 ± 5
P_BASF_0.20_100	0.354	78 ± 2 (-17%)	89 ± 5 (16%)
P_CS_0.257_40	0.354	74 ± 3 (-21%)	82 ± 4 (-22%)
P_CS_0.257_100	0.354	75 ± 3 (-20)	84 ± 9 (-20%)
P_CS_0.38_40	0.381	64 ± 2 (-32%)	76 ± 4 (-28%)
P_CS_0.57_40	0.421	59 ± 2 (-37%)	69 ± 3 (-35%)

- green: < 10% strength reduction;
- orange: between 10% and 30% strength reduction;
- red: >30% strength reduction.

The compressive strength increases from 7 to 28 days for all the mixtures but the compressive strength of the mixtures containing SAPs are reduced significantly at all times compared to the reference mixture at the same age. The reference mixture P_REF has a compressive strength at 7 days of 94 ± 5 MPa and 106 ± 5 MPa at 28 days. None of the tested specimens has a strength reduction lower than 10% (green colour code). For the samples containing BASF SAPs (P_BASF_0.20_100), the reduction in strength is lower than the compressive strength reduction for specimens containing the ChemStream SAPs with the same w/c_{total} of 0.354, namely P_CS_0.257_40 and P_CS_0.257_100. However, the BASF mixture had a lower amount of SAPs, i.e. 0.2 m% versus 0.257 m%. Again no difference between the results for the 40 μm and 100 μm SAPs from ChemStream was observed. When the amount of SAPs is increased from 0.257 m% to 0.38 m% and 0.57 m% (and as a consequence the total water-to-cement-ratios to 0.381 and 0.421, respectively), also the strength reduction increases even to values above 30% (red colour code). These results are in correspondence with findings in literature [10,11,45,83]: the addition of SAPs leads to the formation of macro pores in the hardened state which negatively affects the compressive strength. The higher the amount of added SAPs, the higher the strength reduction.

The mean flexural strength of the different cement paste mixtures at the age of 7 and 28 days with their standard deviation are summarized in Table 8. Also the w/c_{total} is given as well as the compressive strength reduction relative to the reference sample without SAPs P_REF (between brackets) using the following colour code:

- pink: strength gain;
- green: < 10% strength reduction;
- orange: between 10% and 30% strength reduction;
- red: >30% strength reduction.

From Table 8, it can be seen that, except for the mixture P_CS_0.257_40, all the series show a strength gain at both 7 and 28 days compared to the reference at that age. The reference mixture P_REF has a flexural strength at 7 days of 4.6 ± 0.4 MPa and 5.5 ± 1.4 MPa at 28 days. The mixture P_BASF_0.2_100 shows the largest strength gain, namely + 37% compared with the reference at 28 days. Whereas the mixture P_CS_0.257_40, which has the same w/c_{total} as the previous series, is the only series that shows a reduction in strength compared to the reference, at both 7 and 28 days. However, no clear explanation is found for this peculiar result. According to literature, the increase in strength in SAP-containing mixtures is the result of further hydration of unhydrated cement particles [84], which is in this case more dominant than the formation of macro pores and the resulting decrease in strength.

Table 8
Average and standard deviation of the flexural strength of the cement paste mixtures at 7 and 28 days [MPa], the total water-to-cement ratio and the strength reduction in % compared to the reference.

	w/c_{total} [-]	Flexural strength [MPa]	
		7 days	28 days
P_REF	0.300	4.6 ± 0.4	5.5 ± 1.4
P_BASF_0.20_100	0.354	4.9 ± 0.2 (+6%)	7.5 ± 0.7 (+37%)
P_CS_0.257_40	0.354	4.2 ± 0.4 (-9%)	4.0 ± 1.0 (-27%)
P_CS_0.257_100	0.354	4.7 ± 0.3 (+2%)	5.5 ± 0.3 (0%)
P_CS_0.38_40	0.381	4.6 ± 0.2 (0%)	6.3 ± 1.7 (+14%)
P_CS_0.57_40	0.4215	5.0 ± 0.6 (+10%)	7.0 ± 1.7 (+26%)

The final setting times determined by an automated Vicat needle test are summarized in Table 9. The final setting time of the reference mixture is 9.5 h. In case SAPs are added, the setting time delays with increasing amount of added SAPs. In case of a SAP addition of 0.2 m% BASF SAP, the setting time increases to 11.7 h. For the mixtures containing 0.257 m%, 0.38 m% and 0.57 m% ChemStream SAPs, the final setting time increases to 12.7 h, 14 h and 15.2 h, respectively. The retardation in setting was already reported by several authors [63,64,85]. No influence of the particle size on this parameter is seen when studying mean particle sizes of 40 μm and 100 μm as both mixtures P_CS_0.257_40 and P_CS_0.257_100 have the same final setting time of 12.75 h.

The reported final setting times in Table 9 are used as time zero for the start of the autogenous shrinkage measurements from the corrugated tube tests. The evolution of the autogenous strain as a function of time for the six different cement pastes during the first seven days after water contact is shown in Fig. 6. The zero point on the x-axis represents the time of water-cement contact. Negative values of the autogenous strain correspond with shrinkage while positive values of the strain correspond with expansion. The full dark lines represent the average of the three tested tubes per mixture. Also the three individual tubes per mixture are shown in the graph as lighter dotted lines. In Table 10, the average autogenous strains after 7 days measured with the corrugated tube test are summarized together with the reduction compared to the reference mixture P_REF, expressed as %. From Fig. 6 and Table 10, the following conclusions can be made:

- The reference mixture shows the largest autogenous shrinkage, with an average value of -912 μm/m after 7 days. After 7 days, the SAP containing mixtures reach almost horizontal lines, meaning that the autogenous shrinkage does no longer increase, but reaches stable average values between [-200 and -100 μm] for the ChemStream SAPs and +166 μm for the BASF SAPs. The reference mixture P_REF on the contrary still shows a decreasing trend after 7 days meaning that the autogenous shrinkage will become even larger at later ages.
- The mixture P_BASF_0.2_100 completely mitigates the autogenous shrinkage in the first 7 days. After 7 days, even an expansion (positive value for the autogenous strain) around +166 μm is measured. The mixture P_BASF_0.2_100 is the only tested paste mixture where complete mitigation of the autogenous shrinkage is obtained, although only the smallest amount of SAPs is added to this mixture, namely 0.2 m% by cement weight. Comparable results for this type of SAP are already reported in literature [14].
- Although the mixtures with CS SAP do not completely mitigate the autogenous shrinkage (the curves still have negative values after 7 days of measurements), the shrinkage is significantly reduced for all P_CS mixtures compared to the reference mixture. When the amount of ChemStream SAPs is increased from 0.257 m% to 0.38 m% and 0.57 m%, the autogenous shrinkage after 7 days is reduced with 80%, 85% and 89%, respectively.
- All the strain curves for mixtures with CS SAPs show the same trend within the first days after the start of the test: after a period of shrinkage, the curve goes up again and (part of) the shrinkage is compensated. This turning point coincides with the drop in RH and thus with the moment the SAPs will release the stored internal curing water to the surrounding cement matrix. The more SAPs are added, the higher the initial shrinkage, but also the better the mitigation of the shrinkage is in a second phase. According to Bargohel-Bouny et al. [86] and Sant et al. [87] the formation of portlandite and ettringite crystallisation are also given as possible reasons for this early-age expansion.
- Changing the mean particle size of the CS SAPs from 40 μm to 100 μm has no significant influence on the autogenous shrinkage results after 7 days. However, in the first day after the start of the measurements, a difference between the mixtures P_CS_0.257_40 and P_CS_0.257_100 can be seen: the mixture with the larger particle size of 100 μm starts to mitigate the shrinkage at an earlier point in time, compared to the mixtures with the smaller particle size of 40 μm. Larger SAPs , with a smaller surface/volume ratio, may release the internal curing water more promptly at early ages as it is dominantly the bulk of the SAP that acts as a water reservoir. This is in agreement with findings in literature where it was found that the SAP particle size only plays a significant role at very early ages [44,48]. After 7 days, the strains for P_CS_0.257_40 and P_CS_0.257_100 are the same as for both mixtures the same amount of extra water is included in the SAPs.

Table 9
Final setting time of the cement paste mixtures [hours], determined with the Vicat needle test.

	Final setting time [hours]
P_REF	9.5
P_BASF_0.20_100	11.7
P_CS_0.257_40	12.7
P_CS_0.257_100	12.7
P_CS_0.38_40	14.0
P_CS_0.57_40	15.2

4.3. Mortar

In order to have a more pronounced autogenous shrinkage, the type of cement was changed from CEM I to CEM III and the water to cement ratio was lowered from 0.30 to 0.24 for the studied mortar mixtures. In this way, the possible effect of the SAPs in mitigating autogenous shrinkage will be more visible in the test results. However, due to these changes, the extra water-to-cement ratio of 0.054 to mitigate autogenous shrinkage is no longer valid. It was

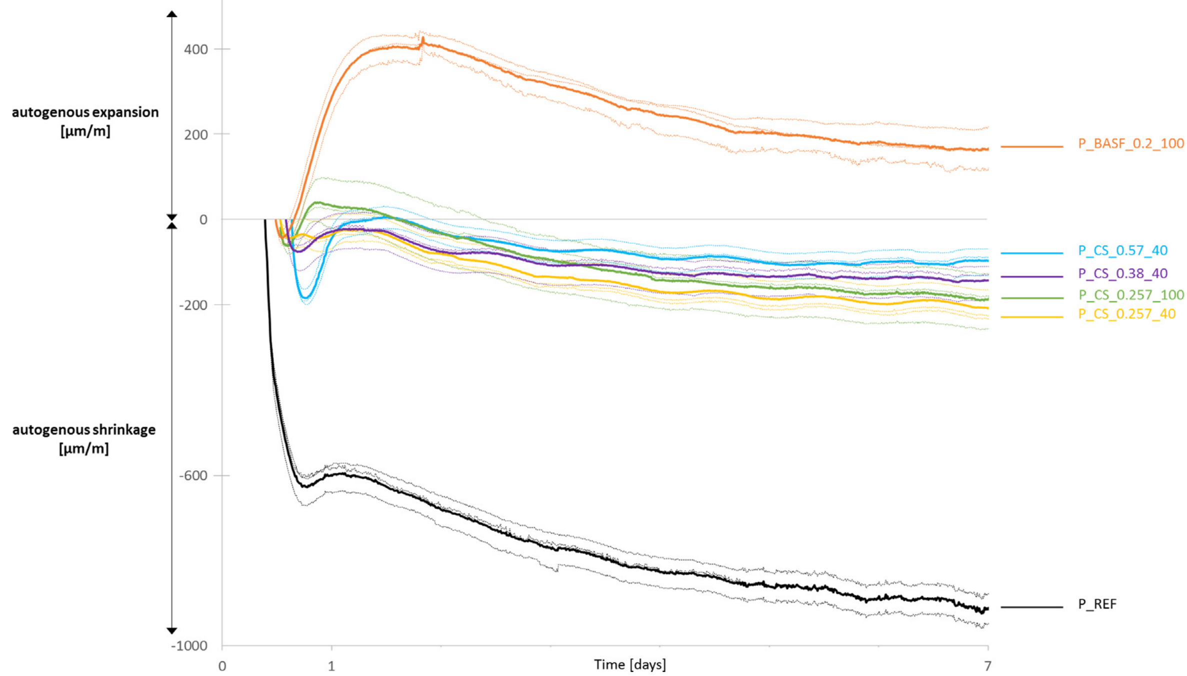

Fig. 6. The evolution of the autogenous strain [μm/m] as a function of time [days] for the six different cement pastes during the first seven days after water contact. The full dark lines represent the average of three tested tubes per mixture. The three individual tubes per mixture are shown in the graph as dotted lines.

Table 10
Average autogenous strains after 7 days [μm/m] measured with the corrugated tube test and the reduction compared to the reference mixture P_REF, expressed as %.

	Average autogenous strain after 7 days [μm/m]	Reduction compared to P_REF [%]
P_REF	−912 ± 29	–
P_BASF_0.20_100	166 ± 38	−118
P_CS_0.257_40	−203 ± 31	−80
P_CS_0.257_100	−183 ± 53	−78
P_CS_0.38_40	−138 ± 34	−85
P_CS_0.57_40	−100 ± 26	−89

therefore decided to keep the amount of SAPs fixed, namely the same amounts as used before, for this part of the paper. Also, the amount of extra water that should be added to compensate for the water uptake by the SAPs, was this time determined based on the swelling capacity in cement filtrate after 10 min and not by the method of equal flow as was the case for the cement paste mixtures. The values of the swelling capacity that were taken into account were 21 g/g SAP for the ChemStream SAPs and 27 g/g SAP for the BASF SAP. In Fig. 7, the flow 10 min after water contact and the flow over time up to two hours is depicted. The reference mixture M_REF has an initial flow of 335 mm and decreases to 322.5 mm after 2 h. A similar trend was found for the mixtures containing SAPs: M_CS_0.257_40 has an initial flow of 320 mm and decreases to 305 mm; M_BASF_0.30_100 starts at a flow directly after mixing of 330 mm which has decreased to 315 mm after two hours. The flow for the SAP-containing mixtures all lie in the range of the flow values of the reference mixture ± 20 mm. This shows that the used method to determine the amount of extra water based on the swelling capacity in cement filtrate after 10 min is an adequate alternative to the method of equal workability.

The mean compressive strengths of the different mortar mixtures at the age of 7 and 28 days with their standard deviation are summarized in Table 11. Also the total water-to-cement ratio w/c_{total} is given as well as the compressive strength reduction relative to the reference sample without SAPs M_REF (between brackets) using the following colour code:

- green: < 10% strength reduction;
- orange: between 10% and 30% strength reduction;
- red: >30% strength reduction.

Similar conclusions as for the compressive test results for cement pastes can be made. The compressive strength increases from 7 to 28 days for all the different mixtures but the compressive strengths of the mixtures containing SAPs are reduced at all times compared to the reference mortar mixture at the same age. The latter is the consequence of the formation of macro pores in the hardened material due to the presence of SAPs. The reference mixture has a compressive strength of 119 ± 3 MPa and 129 ± 4 MPa at 7 and 28 days, respectively. At 28 days, the compressive strength of the mixture M_CS_0.257_40 has a strength reduction lower than 10% compared to the reference (green colour code). Remark that this mixture has the lowest amount of added SAPs, i.e. 0.257 m% by cement weight. The strength reductions at 28 days are significantly lower than the ones at 7 days due to the ongoing hydration of unhydrated cement particles as was also noticed in different studies [14,44,47]. When comparing the results for the mixtures M_CS_0.38_40 and M_BASF_0.30_100, which both have the same w/c_{total} of 0.321, it can be seen that the compressive strengths at 7 and 28 days are not significantly different. Increasing the amount of ChemStream SAPs from 0.257 m% to 0.38 m% and 0.57 m% leads in each step to a strength reduction at 28 days of 7%.

The mean flexural strengths of the different cement mortar mixtures at the age of 7 and 28 days with their standard deviation are summarized in Table 12. Also the w/c_{total} is given as well as the flexural strength reduction relative to the reference sample without SAPs M_REF (between brackets) using the same colour code as for the compressive strength.

The addition of SAPs has an overall negative effect on the flexural strength, as all series show a significant reduction at all times

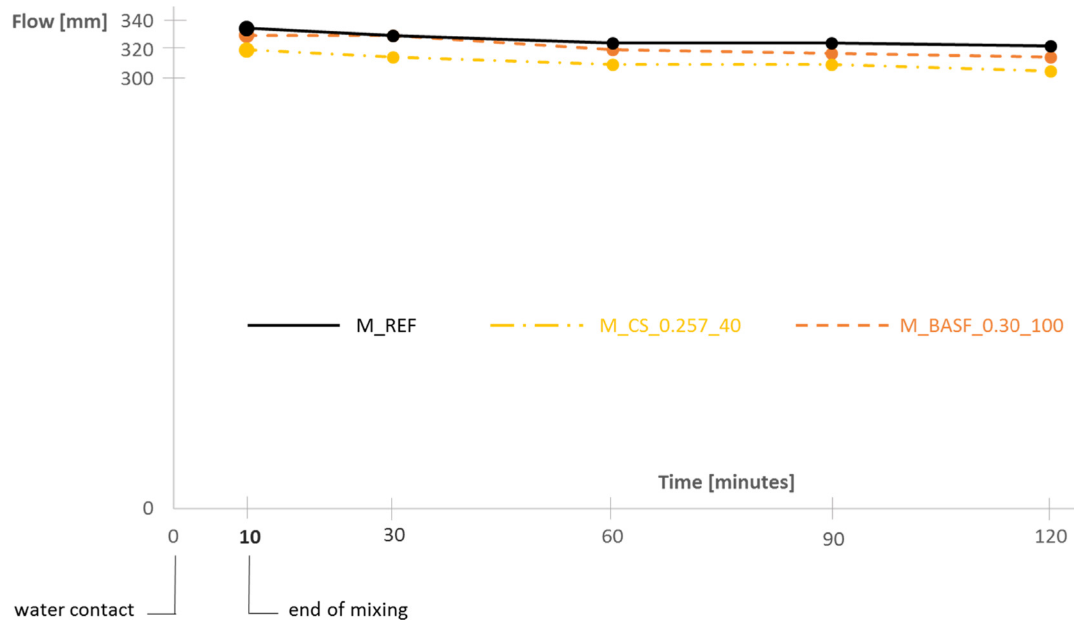

Fig. 7. Initial flow and flow over time [mm] of the reference mortar mixture and the SAP mortar mixtures.

Table 11
Average and standard deviation of the compressive strength of the mortar mixtures at 7 and 28 days [MPa], the total water-to-cement ratio and the strength reduction in % compared to the reference.

	w/c_{total} [-]	Compressive strength [MPa]	
		7 days	28 days
M_REF	0.24	119 ± 3	129 ± 4
M_CS_0.257_40	0.294	106 ± 9 (-11%)	120 ± 4 (-7%)
M_BASF_0.30_100	0.321	85 ± 2 (-29%)	110 ± 6 (-14%)
M_CS_0.38_40	0.321	86 ± 5 (-27%)	111 ± 1 (-14%)
M_CS_0.57_40	0.361	77 ± 2 (-35%)	101 ± 4 (-22%)

Table 12
Average and standard deviation of the flexural strength of the mortar mixtures at 7 and 28 days [MPa], the total water-to-cement ratio and the strength reduction in % compared to the reference.

	w/c_{total} [-]	Flexural strength [MPa]	
		7 days	28 days
M_REF	0.24	8.2 ± 1.3	9.4 ± 0.7
M_CS_0.257_40	0.294	6.5 ± 0.7 (-21%)	8.3 ± 0.7 (-11%)
M_BASF_0.30_100	0.321	3.7 ± 1.0 (-56%)	6.6 ± 0.4 (-30%)
M_CS_0.38_40	0.321	6.3 ± 0.5 (-24%)	8.4 ± 0.7 (-11%)
M_CS_0.57_40	0.3615	5.3 ± 0.8 (-35%)	6.9 ± 0.2 (-26%)

compared to the reference mortar, due to the formation of macro pores. The reference mortar mixture M_REF has a flexural strength of 8.2 ± 1.3 MPa and 9.4 ± 0.7 MPa at 7 and 28 days, respectively. Although the strength reduction is somewhat decreased from 7 to 28 days, due the ongoing cement hydration, strength reductions up to 30% compared to the reference are observed at 28 days. Increasing the amount of CS SAPs from 0.257 m% to 0.38 m% has no significant effect on the results of the flexural strength. Increasing the amount of CS SAPs from 0.38 m% to 0.57 m% however, leads to a significant increase in the strength reduction with >10%. Although the mixtures M_BASF_0.30_100 and M_CS_0.38_40 have the same total water-to-cement ratio of 0.321, the strength reduction in the former is more than double than for the latter series. Unlike the results of the compressive strength, the larger particle size of 100 μm of the BASF SAPs compared to the smaller particle size of 40 μm of the CS SAP seems to have an influence on the flexural strength results.

The final setting times determined by an automated Vicat needle test are summarized in Table 13. The final setting time of the reference mortar mixture M_REF is 7 h. In case SAPs are added, the setting time increases with increasing amount of added SAPs. In case of a SAP addition of 0.3 m% BASF SAP, the setting time is

Table 13
Final setting time of the mortar mixtures [hours], determined with the Vicat needle test.

	Final setting time [hours]
M_REF	7
M_CS_0.257_40	9.5
M_BASF_0.30_100	10
M_CS_0.38_40	12
M_CS_0.57_40	13

Table 14
Average autogenous strains after 7 days [μm/m] measured with the corrugated tube test and the reduction compared to the reference mixture M_REF, expressed as %.

	Average autogenous strain after 7 days [μm/m]	**Reduction compared to M_REF [%]**
M_REF	−1473 ± 73	–
M_CS_0.257_40	−1200 ± 47	−19
M_CS_0.38_40	−887 ± 46	−40
M_CS_0.57_40 *	−437 ± 7	−70
M_BASF_0.30_100	−51 ± 38	−97

*Only 2 tubes available due to technical problems with the test setup.

delayed to 10 h. For the mixtures containing 0.257 m%, 0.38 m% and 0.57 m% ChemStream SAPs, the final setting time increases to 9.5 h, 12 h and 13 h, respectively. The retardation in setting time was already reported by several authors [65,88,89]. Due to the use of another cement type and superplasticizer, the results for the setting times in cement paste cannot be compared to the setting times in mortar. However the same trends are obtained for both cementitious materials.

The reported final setting times in Table 13 are used as time zero for the start of the autogenous shrinkage measurements in the corrugated tube tests. The evolution of the autogenous strain as a function of time for the five different mortar mixtures during the first seven days after water contact is shown in Fig. 8. The zero point on the x-axis represents the time of water-cement contact. Negative values of the autogenous strain correspond with shrinkage while positive values of the strain correspond with expansion. The full dark lines represent the average of three tested tubes per mixture. Also the three individual tubes per mixture are shown in the graph as lighter dotted lines. In Table 14, the average autogenous strains after 7 days measured with the corrugated tube tests are summarized together with the reduction compared to the reference mortar mixture M_REF, expressed as %.

From Fig. 8 and Table 14, the following conclusions can be made:

- The mortar mixtures with CEM III, a high amount of binder and w/c ratio of 0.24 have larger values of autogenous shrinkage in the first seven days after water contact compared to the cement paste mixtures made with CEM I and w/c ratio of 0.3. This is in accordance with findings in literature [71].
- The reference mixture shows the largest autogenous shrinkage, with a value of -1473 ± 73 μm/m after 7 days. After 7 days, all mixtures reach almost horizontal lines, meaning that the autogenous shrinkage does no longer continue, but reaches stable average values. For the mixture containing BASF SAPs the average line even slightly increases after 7 days, meaning that some autogenous expansion is still occurring and the autogenous shrinkage can be even further compensated after 7 days.
- Although the cement paste mixture with BASF SAPs was able to completely mitigate autogenous shrinkage after 7 days, this is not the case for the mixture M_BASF_0.3_100 as still an average value of -51 ± 38 μm/m shrinkage occurs. This type of SAP however leads to the largest reduction in autogenous shrinkage (-97%) compared to the other studied SAPs from ChemStream (reductions between -19% and −70% after seven days).
- Although the mixtures with ChemStream SAP do not completely mitigate the autogenous shrinkage (the curves still have negative values after 7 days of measurements), the shrinkage is reduced for all M_CS mortar mixtures compared to the reference mixture. When the amount of ChemStream SAPs is increased from 0.257 m% to 0.38 m% and 0.57 m%, the autogenous shrinkage after 7 days is reduced with 19%, 20% and 70%, respectively.
- All the strain curves for mixtures with SAPs, except for the mixture M_CS_0.257_40 show the same trend within the first days after the start of the test: after a period of shrinkage, the curve goes up again and (part of) the shrinkage is compensated. This turning point coincides with the drop in RH and thus with the

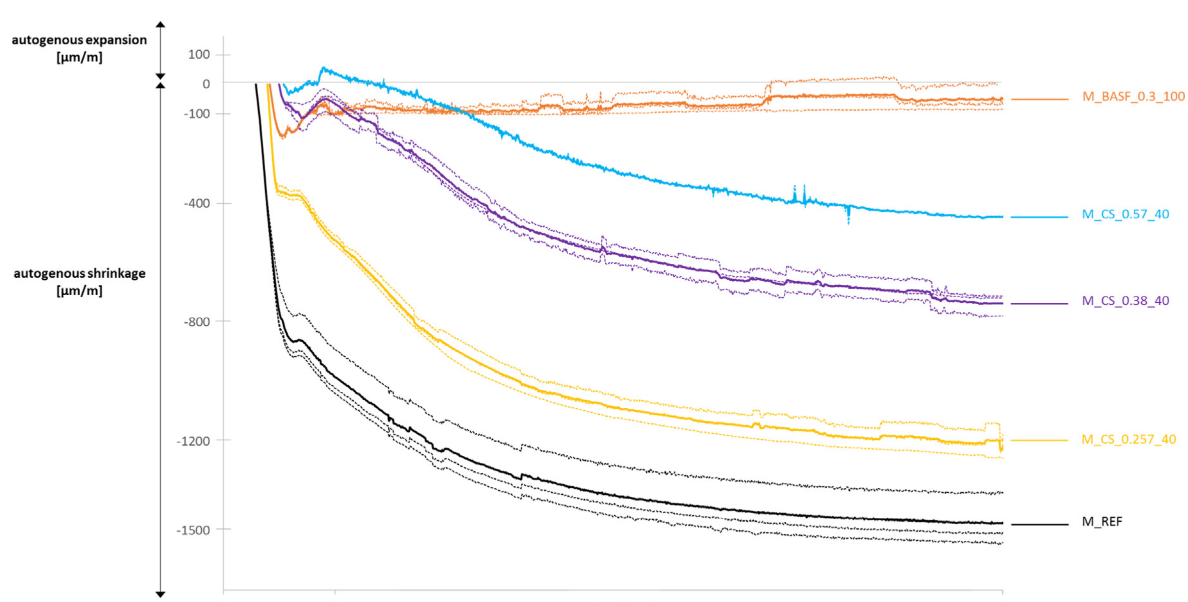

Fig. 8. The evolution of the autogenous strain [μm/m] as a function of time [days] for the five different mortars during the first seven days after water contact. The full dark lines represent the average of three tested tubes per mixture. The three individual tubes per mixture are shown in the graph as dotted lines.

moment the SAPs will release the stored internal curing water to the surrounding cement matrix. For the mixture M_CS_0.257_40 however, this behaviour is not seen, but a similar curve as for the reference mixture is observed. A possible explanation could be that the amount of internal curing water is so low that it is exhausted very fast and the same behaviour of the reference mixture is followed.

4.4. Concrete

For the tested concrete mixtures, the same mix design as for the mortar mixtures is used, with the addition of coarse aggregates, namely basalt 4/8. The amount of extra water to be added in case of SAP containing mixtures is again based on the swelling capacity of the SAPs in cement slurry after 10 min, namely 21 g/g SAPs for the ChemStream SAP and 27 g/g SAP for the BASF SAPs. In case of SAP containing mixtures, the amount of superplasticizer was increased with 0.1 m% by cement weight to a total of 1.54 m% to have a slump flow of at least 550 mm, so the tested concretes could be classified as self-compacting concrete.

In Fig. 9, the slump flow directly after mixing (i.e. 10 min for the reference mixture and 12 min for the SAP-containing mixtures) is depicted. All the studied concretes can be classified as self-compacting concrete as the slump flow values are at least 550 mm. However, it can be seen that the slump flow values of the mixtures containing ChemStream SAPs are much lower than the value measured for reference concrete, meaning that the workability of these mixtures is less compared to the one for the reference mixture, despite the additional amount of water and extra amount of superplasticizer that was added. Similar results can be found in literature [10], [92], [93], where it is stated that because of the absorption of moisture in the concrete mixture by the SAPs, the slump of the concrete is reduced to a certain extent, even when extra water based on the swelling of the SAPs is added. From this it looks like using the swelling capacity of SAPs from the filtration test is not the most ideal method to predict the amount of extra water needed, as in this way the swelling capacity of the SAPs is underestimated. Nevertheless, if the higher than expected water absorption by the SAP would be the reason for the reduction in workability, the effective w/c would also be lower than planned, which would positively affect the strength of the mixes. However, the reductions in compressive strength at 28 d were very similar for the corresponding concrete and mortar mixes (Tables 15 and 11). Another reason for the lower workability of the SAP-containing mixtures could be the longer mixing time compared to the reference mixture (12 min versus 10 min). From other experiments executed by the authors with the same type of concrete, it was observed that this HPC mixture becomes stiff very quickly when mixing is stopped. Further research should focus on the reason for this decreased workability.

Table 15
Average and standard deviation of the compressive strength of the concrete mixtures at 28 days [MPa], the total water-to-cement ratio and the strength reduction in % compared to the reference.

	w/c_{total} [−]	28 days [MPa]
C_REF	0.24	117 ± 3
C_CS_0.257_40	0.294	110 ± 3 (−6%)
C_BASF_0.30_100	0.321	104 ± 3 (−12%)
C_CS_0.38_40	0.321	101 ± 3 (−14%)
C_CS_0.57_40	0.362	92 ± 3 (−22%)

The mean compressive strengths of the different concrete mixtures at the age of 28 days with their standard deviation are summarized in Table 15. Also the total water-to-cement ratio w/c_{total} is given as well as the compressive strength reduction relative to the reference sample without SAPs C_REF (between brackets) using the following colour code:

- green: < 10% strength reduction;
- orange: between 10% and 30% strength reduction;
- red: >30% strength reduction.

The reference concrete mixture C_REF has a compressive strength at 28 days of 117 ± 3 MPa. When SAPs are added, the compressive strength decreases as a result of the formed macro pores upon de-swelling of the SAPs in the hardened state. At 28 days, the compressive strength of the mixture C_CS_0.257_40 has a strength reduction lower than 10% compared to the reference (green colour code). Remark that this mixture has again the lowest amount of added SAPs, namely 0.257 m% by cement weight. When comparing the results for the mixtures C_CS_0.38_40 and C_BASF_0.30_100,

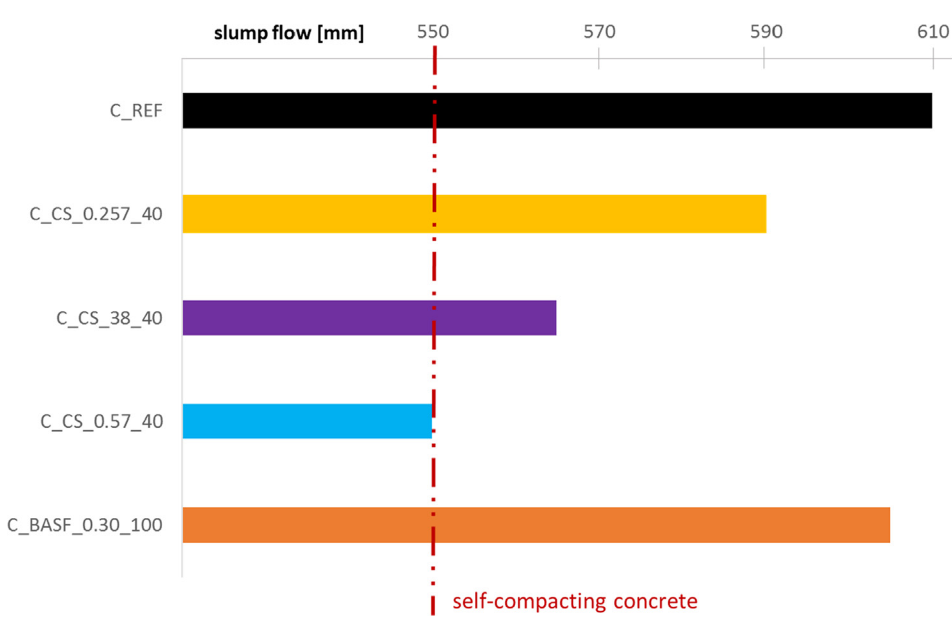

Fig. 9. Slump flow [mm] directly after mixing of the concrete. The threshold for classification as a self-compacting concrete is 550 mm.

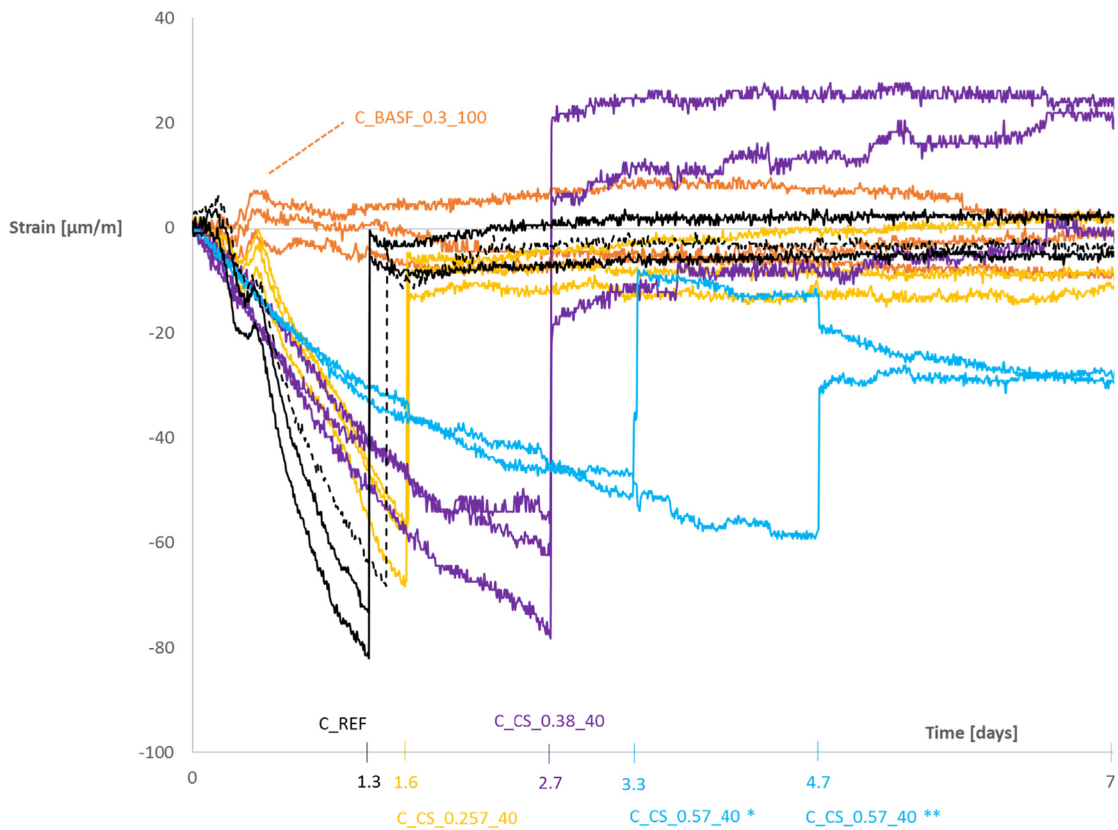

Fig. 10. Strains from the restrained ring tests [μm/m] for the five different concrete mixtures during the first seven days.* crack 1 ** crack 2.

which both have the same w/c_{total} of 0.321, it can be seen that the compressive strengths at 28 days are not significantly different. Increasing the amount of SAPs from 0.257 m% to 0.38 m% and 0.57 m% leads to a compressive strength reduction compared to the reference of -6%, -14% and -22%, respectively. These findings are in correspondence with literature [10,11,43,79]. Similar strength reductions are found for the compressive strength at 28 days for the corresponding mortar mixtures.

In Fig. 10, the results from the restrained ring tests are shown for the five different concrete mixtures during the first seven days. The measurements started within 10 min after casting the concrete in the rings and were zeroed at that time. The formation of a crack is visible as a sudden increase or decrease in the strain. Small jumps in the strain are due to vibrations in the vicinity of the test setup. The data from the different strain gauges (two gauges in case of C_REF, C_CS_0.257_40 and C_CS_0.57_40 and three for the other mixtures) on the same ring are depicted in the same colour.

The reference mixture C_REF cracked after 1.3 days at a maximum strain of -82 μm/m. In case SAPs from BASF are added, the measured strains are very limited and have values in the range of [-10 ; 10 μm/m]. No cracks appeared during the duration of the test of 28 days as no sudden change in the measured strain is observed. Also visually no cracks are observed in the C_BASF_0.3_100 ring. When SAPs from ChemStream are added to the concrete mixture, it is observed that the rings crack later in time and the strains are lower compared to the reference. The more SAPs are added, the more pronounced the latter two effects are. The moment of cracking for the mixtures C_CS_0.257_40 and C_CS_0.38_40 are 1.6 days and 2.7 days, respectively. The mixture C_CS_0.57_40 even cracked twice: the first time after 3.3 days and the second crack appeared after 4.7 days. These cracks are also visually observed in the concrete rings. Based on the autogenous shrinkage measurements on the corresponding mortar mixes (Fig. 8) these results regarding the efficiency of the various SAP additions could be more or less expected. Of course, due to the restrains the strain values reached here are much lower and the further increase in strain is halted by the formation of cracks. A noticeable point is also that all concrete mixes crack at a reasonably similar strain value of 50–80 μm/m.

5. Conclusions

In this paper, the influence of SAP addition on mitigating autogenous shrinkage in cement paste, high performance mortar and high performance concrete is investigated. For this purpose, two different types of SAPs in varying amounts were added to the tested cementitious materials: one commercial SAP from BASF and a developed SAP from ChemStream.

The following conclusions can be made:

- Most of the swelling of the studied SAPs occurred within the first 10 min after contact with the testing fluid, as the swelling capacity only increases slightly after 1 h and 24 h. Neither changing the particle size from 40 μm to 100 μm nor changing the cement type from CEM I to CEM III has an influence on the swelling capacities of the studied SAPs in the test fluids.
- The SAP particle size (d_{50} = 40 μm or d_{50} = 100 μm) has no significant influence on the flow, the compressive strength at 7 and 28 days, the final setting time and the autogenous shrinkage after 7 days of the studied cement pastes. Only at very early age (i.e. <1 day after water contact) the difference in particle size has an influence on the autogenous shrinkage results: the mixture with the larger SAP particle size of 100 μm starts to mitigate the shrinkage at an earlier point in time compared to the mixture with the smaller SAP particle size of 40 μm.

- For the tested cement pastes and mortars, the amount of extra water, that should be added to compensate for the water uptake by the SAPs, can be determined based on the swelling capacity in cement filtrate after 10 min obtained by the filtration method, as it leads to the same results as obtained with the method of equal workability. In this way, resources can be saved, as the filtration method requires less material, labour and time compared to the method of equal workability. For the studied concrete mixtures however, it was found that the swelling capacity of the SAPs determined by the filtration test is an underestimation for the swelling capacity in the real concrete mixture, affecting the workability of the fresh concrete mixture in a negative way.
- When SAPs are added to cementitious materials, a decrease in compressive strength at every age was observed for all types and amounts of SAPs due to the formation of macro pores in the hardened matrix. The more SAPs are added, the higher the loss in strength. After 28 days, the strength reduction in mortar and concrete was found to be similar in corresponding mixtures.
- When the final setting time is measured using an automated Vicat needle test, a delay in final setting time compared to the reference is observed in SAP-containing cementitious materials. For the maximum added amount of SAPs, namely 0.57 m% by cement weight, the final setting time is delayed with 6 h in both cement paste and mortar. For smaller amounts of SAPs in the range of 0.2 m% -0.3 m%, this delay is between 2 and 3 h for both cement paste and mortar mixtures.
- In this study, it was found that the commercially available SAPs from BASF reduce the autogenous shrinkage after 7 days in the studied mortar mixtures with 97%, whereas in the studied cement paste, the autogenous shrinkage after 7 days is completely mitigated and even expansion occurred. Although the mixtures with ChemStream SAPs do not completely mitigate the autogenous shrinkage, the shrinkage is significantly reduced for all cement paste mixtures: increasing the amount of SAPs from 0.257 m% to 0.38 m% and 0.57 m%, leads to a reduction in the autogenous shrinkage after 7 days with 80%, 85% and 89%, respectively. For ChemStream SAPs in mortar the reductions for the same amount of SAPs are 19%, 20% and 70%, respectively. The higher the amount of SAPs added, the larger the reduction in autogenous shrinkage as more internal curing water is available and a high relative humidity can be sustained preventing the mixture from self-desiccation.
- The addition of BASF SAPs in the concrete mixture reduces the occurring strains in the restrained ring test significantly with -88% compared to the reference and prevents the concrete from cracking. Although the addition of the ChemStream SAPs did not prevent the concrete from cracking, the moment of cracking was delayed and lower strains compared to the reference were observed. These two effects become more pronounced the more SAPs are added to the concrete.

Despite some negative effects on the workability and compressive strength of the studied cementitious materials, this paper shows that the addition of SAPs in the correct amount can reduce or even completely mitigate autogenous shrinkage in cement paste, high performance mortar and high performance concrete, leading to more durable and aesthetically more appealing concrete structures.

CRediT authorship contribution statement

Laurence De Meyst: Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Project administration, Visualization, Writing - original draft. **Els Mannekens:** Investigation, Writing - review & editing. **Kim Van Tittelboom:** Conceptualization, Methodology, Supervision, Writing - review & editing. **Nele De Belie:** Conceptualization, Funding acquisition, Methodology, Project administration, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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