MULTIFUNCTIONAL USE OF SAP IN STRAIN-HARDENING CEMENT-BASED COMPOSITES

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Abstract

This article describes the multipurpose use of superabsorbent polymers (SAP) in strainhardening cement-based composites (SHCC) under the aspects of their mechanical and shrinkage behaviour as well as their frost resistance. Unlike ordinary, strain-softening concrete materials, SHCC exhibits a pronounced degree of multiple cracking when subjected to tensile loading, resulting in a ductile response with a strain capacity of up to 5 %. The addition of fine SAP particles to the SHCC mix results in the formation of a great number of well distributed voids initially filled with water. The results of quasi-static, uniaxial tension tests on hardened SHCC demonstrate that such a system of voids facilitates multiple cracking by fostering the formation of additional micro-cracks. Furthermore, the positive influence of voids on the frost resistance of SHCC, as induced by adding SAP, is demonstrated. Finally, the potential of using SAP as tiny water reservoirs for the purpose of internal curing is investigated by measuring autogenous and total shrinkage of SHCC.

1. Introduction

Strain-hardening cement-based composites (SHCC) show under uniaxial tension, in contrast to ordinary concrete materials, an increase in load-carrying capacity after initial cracking, which is accompanied by large deformation due to extensive multiple cracking, cf. Figure 1. The strain capacity of such materials, i.e. the strain on reaching the tensile strength of the composite, can be up to 5% or 300–500 times greater than that of ordinary concrete. The load transfer over the many fine cracks is provided by crack-bridging with short, well-distributed fibres. Subsequent to crack initiation the fibres are gradually detached from the matrix and finally pulled out under rising energy expenditure [1, 2]. The pre-crack bond, its de-bonding, and the pullout behaviour of the fibres depend on the interface phase configuration between matrix and fibres. To achieve a uniform fibre distribution, the maximum aggregate size must be limited; in most studies it does not exceed 0.2 or 0.3mm.

The trim of the first-crack stress, which is necessary for controlled crack formation, occurs by optimising matrix strength and toughness with customary methods used in concrete technology such as the choice of water-cement ratio, the addition of fly ash, the choice of cement type, etc. Since the high strain capacity of SHCC depends on the formation of a great number of more or less parallel, fine cracks, it is desirable and essential to promote such multiple cracking. Micro-defects, if well distributed in the matrix, can serve as a trigger for the formation of numerous micro-cracks, which can develop subsequently into many fine, continuous cracks. The positive effect of micro-defects on the mechanical performance of SHCC has been observed when considerable amounts of very fine lightweight sand or fly ash particles have been used in the mixture, which in fact act as micro-defects in a fine-grained matrix (cf. Figure 2). It should be noted that in using fly ash as the source of the micro-defects and not as the binder, the portion of fly ash in the mixture must be more than approximately 60% to 70% of the mass of cement. Due to the progressive pozzolanic reaction of fly ash, the effect of these engineered micro-defects decreases over time.





Figure 1: Typical stress-strain behaviour of SHCC

Figure 2: Unhydrated fly ash particle close to a fibre channel

SAPs are well known in the fields of hygiene and health care as a powerful fluid absorber. These polymers consist of cross-linked chains having dissociated ionic functional groups facilitating the absorption of large amounts of water. Their capacity to absorb water can be up to 1500 times their own weight. In the field of concrete construction SAP is a new additive with considerable potential for application in achieving specific, practical purposes [3].

As a concrete additive, SAP must possess sufficient robustness in a highly alkaline environment. Usually dry SAP particles (cf. Figure 3) are added to the concrete mix prior to or during mixing. They absorb water (i.e. ion solution), swell, and keep the water until it is withdrawn by the hydrating and drying cement matrix surrounding. The size and the dispersion of the microscopic water reservoirs can be purposefully adjusted with the choice of the particle size and water absorption capacity of the particles.

A few prospective applications of SAP in concrete construction are under intensive investigation by several research groups worldwide. For example, the addition of SAP to concrete with a low water-to-binder ratio can reduce autogenous shrinkage considerably. The reason for autogenous shrinkage is the so-called "self-desiccation" of the hardening cement paste due to the progressive hydration process and an insufficient supply of water. Autogenous shrinkage can lead as soon as the first few days after concreting to high constraint stresses and consequently to crack formation. In contrast to drying shrinkage, autogenuos shrinkage, which occurs over the entire concrete volume, cannot be mitigated noticeably by external curing. However, when tiny water reservoirs are distributed over the concrete volume due to the use of SAP, self-desiccation is dramatically reduced. With the decrease in the relative humidity of the surrounding pore system, the embedded water is released by SAP particles so that internal curing can occur. As a result autogenous shrinkage and hence the danger of cracking are minimised [4, 5].

Another prospective use of SAP addition is to increase the frost resistance of concrete. In this respect the importance of SAP particles is based on their forming a system of fine, evenly distributed pores (cf. Figure 4), which are filled with water in fresh or young concrete but are to a great extent empty in the hardened concrete. This void system is comparable in its properties and action to those induced by the use of air-entraining agents (AEA) in concrete. The main advantage in comparison to the use of air entraining agents is the SAP-pore system's very high robustness and the resulting high predictability of its properties; in contrast, air bubbles induced during mixing when an AEA is used may be relatively easily destroyed during concrete transport or processing (pumping, compaction, spraying). First results demonstrating a positive effect of SAP on the frost resistance of concrete were presented in [6,7].



Figure 3: Dry SAP particles

Figure 4: Pores in the matrix generated by dried-out SAP particles

This study treats the use of SAP in SHCC as a multi-purpose additive: (1) as a source of micro-defects to support multiple cracking and accordingly to increase the mechanical performance (strain capacity, fracture energy), (2) as producer of a pore system to improve frost resistance, and (3) finally as an agent to help mitigate autogenous shrinkage.

2. Materials and Test Methods

2.1 Composition of SHCC

The initial SHCC composition serving as starting point for this study was developed by Mechtcherine and Schulze [2] based on the approach briefly described in the introduction. The developmental goal at the time was to obtain an SHCC material with stable strain-hardening behaviour under uniaxial tension. No other specific requirements were posed. In the present study this composition is indicated as M61, cf. Table 1. The other three mixtures given in Table 1 result from purposeful changes in the initial mix to achieve improved characteristics with regard to the strength, durability, and shrinkage behaviour of the SHCC.

All four compositions had a fibre volume content of 2.2%. The PVA fibres used had a diameter of 40µm and a cut length of 12mm. In order to ensure good workability as well as a uniform distribution of the fibres in the matrix, only very fine quartz sand with a grain size ranging from 0.06 to 0.2mm was used as aggregate. The Portland cement content of SHCC M61 was 323 kg/m³, a range customary for ordinary concrete. However, the portion of the Class F fly ash (F) in this composition was more than twice as high as the portion of cement (the proportion of fly ash to the total amount of binder was F/B = 0.7). Thus, not all fly ash particles were involved in the hydration process so that they could act as micro defects and crack initiators in the hardened matrix. All mixes had a super plasticizer with a polycarboxilate-ether basis and a viscosity agent, cf. Table 1.

Component	M61	M68a	M68b	M68c
CEM I 42.5 R-HS	323	505	505	505
Fly ash	755	614	614	614
Water (extra water)	337 (-)	334 (-)	334 (-)	334 (25.3)
Quartz sand 0.06-0.2	539	534	534	534
Super plasticizer	12.9	13.4	16.6	13.4
Viscosity agent	3.2	3.2	3.2	3.2
SAP	-	-	2.0	2.0
PVA fibre	29	29	29	29

Table 1: Matrix compositions of SHCC [kg/m³]

In contrast to composition M61, the amount of fly ash was reduced while the portion of cement was increased in the compositions M68a to M68c; the fly ash to binder ratio was reduced to 0.55. While the mix M68a contained no SAP or extra water, 2 kg SAP per cubic meter concrete were added to the mixes M68b and M68c. In the case of the mix M68c, apart from SAP some extra water was added for the purpose of internal curing. The amount of SAP was chosen under consideration of the experience of earlier investigations with regard to internal curing [5, 8] as well as estimation of the desired volume of SAP voids in SHCC with regard to improving frost resistance of the composite. Note that these additional components were not explicitly considered in the mix design, i.e. the quantities of other components were not adjusted (cf. Table 1). The SAP quantity admitted was estimated in such a way that both superior ductility and higher frost resistance could be attained. The water-to-binder ratio was 0.31 for M61 and 0.30 for M68a-c (without additional water), respectively. The water-to-cement ratios were considerably higher. Due to the addition of SAP in the mix M68b, a stiffer consistency of fresh SHCC was measured in comparison to mix M68a as a result of the absorption of mix water by SAP. 25.3kg/m³ extra water was added to mix M68c in order to compensate for the water absorption by SAP to attain approximately the same workability as M68a (no SAP, no extra water). The corresponding slump flow values measured using small cone testing (cone geometry according to DIN EN 1015-3 [9]) are given in Table 2.

Composition	M61	M68a	M68b	M68c
slump flow [mm]	185	172	155	175

Table 2: Slump flow values of fresh SHCC measured using small cone

The SAP used in this study was produced using suspension polymerisation and had an average dry particle size of approximately 70 μ m. The absorption capacity of distilled water was 86.5g/g SAP, where in the case of simulated pore solution the absorption capacity was reduced to 21.8g/g SAP. It should be mentioned here that the water absorption by SAP in fresh SHCC mix must be lower than this value. It is estimated to be 12 to 13g/g SAP.

In producing the SHCC, all dry components, including the SAP (M68b and M68c) but without the PVA fibres, were homogenized in a 65-liter laboratory, single-shaft, compulsory mixer. Subsequently water (including the extra water for internal curing for M68c) was added along with 50% of the super plasticizer and mixed in until a fluid consistency was reached. Then, gradually the fibres were added whilst continuing the mixing, followed by a period of intensive mixing, which continued until a homogenous fibre distribution was apparent. With the addition of the remaining portion of the super plasticizer, the desired consistency of fresh SHCC was attained. At a concrete age of approximately 24h the specimens were de-moulded and eventually stored in sealed plastic boxes at a temperature of 20°C until testing.

2.2. Compression Tests and Quasi-Static Monotonic Uniaxial Tensile Tests

The compression tests were performed according to DIN 1048-4 [10] on three cubes per mixture. The lateral dimension of the cubes was 100mm. The specimens were cured as mentioned previously and tested on the 28^{th} day after casting.

The uniaxial tensile strength was determined on special dumbbell shaped prisms with a crosssection at the support of 40mm x 40mm, a narrow cross-section of 40mm x 24mm in the middle of the samples, and an overall length of 240mm. At a concrete age of 28 days the specimens were connected by gluing to steel support plates. The upper support plate, which was first glued to the specimen, was fixed to the crosshead of the testing machine (type: Zwick Z1200, max. load 1200kN). To ensure restraint-free, non-rotating fixation of the specimen to the testing machine, the lower support plate was first fixed to the load cell at the bottom of the testing chamber. By filling the lower support plate with fresh, fast hardening glue and plunging the sample into the glue by lowering the crosshead, the specimen was fastened to the testing machine after a hardening period of 15 min. The tests were performed in a deformationcontrolled regime with a deformation rate of 0.005mm/s. The deformation was measured by means of two LVDT's over a gauge length of 100mm at the narrowed cross section.

2.3. Test of Freeze-Thaw and De-icing Resistance with Sodium Chloride Solution

The testing method was in accordance with the RILEM-recommendation for so-called CDF-Testing [11]. The specimens for the cyclic freeze-thaw test with sodium chloride solution were produced in cube moulds divided by plates made of polytetrafluorethylene (PTFE, Teflon®) and not treated with mould release agent. The surface to be tested faced the PTFEplate. The dimensions of the specimens were 150mm x 150mm x 75mm. Two days before pre-saturation, the specimens were sealed on their lateral surfaces with aluminium foil using a butyl resin as glue. At a concrete age of 28 days the pre-saturation started in containers with spacers at the base to ensure free access of the sodium chloride solution to the specimen. whose test surface was directed downwards. During the seven-day pre-saturation interval the solution absorption of the SHCC was recorded. Subsequently, 28 freeze-thaw cycles were performed, whose heating and cooling phases lasted four hours each. The duration of the interval at the lower temperature limit at -20°C was 3 hours for each cycle. The holding time at the upper temperature bound of 20°C was only one hour. After the 14th cycle and at the end of the testing procedure, material scaling was determined. For this, each test surface was cleaned in an ultrasonic bath and the loose material was filtered out of the cleaning liquid, dried at 60°C, and weighed.

2.4. Investigations of Shrinkage Behaviour

For shrinkage determination two kinds of specimens were produced. The total shrinkage was investigated on samples with a cross-section of 40mm x 40mm and an overall length of 160mm. Measurement pins were already attached to the prisms during casting. 24 hours after casting the samples were de-moulded and placed in test stands with computer-assisted recording for 38 days. The testing procedure was performed according to the Graf-Kaufmann method (DIN 52450 [12]).

The measurements of autogenous shrinkage deformation were performed using a dilatometer technique developed by Jensen and Hansen [13] using plastic corrugated tubes with an overall length of 415mm. The plastic tubes prevent the evaporation of the water absorbed in the SHCC. Because of the corrugated shape of the tubes, the longitudinal deformation of the enclosed SHCC is not constrained. To prepare an individual sample, a tube was sealed at one end with a plug made of PTFE and placed in a holder on a vibration table in order to fill it with fresh SHCC. The tube was filled nearly to the upper edge under conditions of moderate vibration and sealed in such a way that the plug at the other end was also in contact with the filled SHCC. Immediately after filling, the tubes were placed in the dilatometer and measuring began. In order to ensure contact of the sealing plugs to the support-pins at the dilatometer framework by the self-weight of the tubes, the dilatometer was placed in such a way that its plane was inclined approximately 15% to the horizontal. The autogenous shrinkage was recorded over 14 days using digital indicator gauge with computer-assisted recording.

3. Experimental Results and Discussion

3.1 Mechanical Performance under Compression and Tensile Load

The average compression strengths of the investigated materials are given in Table 3 along with the results of the evaluation of the stress-strain curves obtained from the uniaxial tension tests. When using the empirical formula developed by Walz [14] in combination with the activity factor of fly ash of k = 0.4 and a chargeable fly ash quantity of 1/3 of cement mass (both factors according to DIN EN 206 [15]) for estimating the compression strength, a discrepancy can be observed with respect to the results of the compression tests. However, using the assumption of an activity factor k = 0.50 for the complete fly ash quantity of the mixes (the water-to-binder ratio would be 0.48 and 0.41 for M61 (F/B=0.70) and M68a (F/B=0.55), respectively, in this case) the compressive strength for the mixture M68a with 57.4MPa accords well with the prediction by Walz's formula. The compressive strength for the mixture M61 is at 35.4MPa smaller than the estimate using Walz's formula since in this mix obviously not all fly ash particles are involved in strength development. This finding is in agreement with the results published in [16] for concrete with different quantities of fly ash. The reduced compression strengths of the mixtures M68b and M68c in comparison to M68a are related to the higher porosity of the hardened material due to the voids induced by SAP particles. Furthermore, the extra water in mix M68c leads to an increase in the effective water-to-binder ratio.

Mix ID	Compression strength [MPa]	First-crack stress [MPa]	Tensile strength [MPa]	Tensile strain capacity [%]
M61	35.4 (0.37)	3.35 (0.15)	4.14 (0.15)	2.14 (0.14)
M68a	57.4 (1.18)	3.77 (0.23)	4.84 (0.40)	2.71 (1.42)
M68b	50.4 (0.33)	4.71 (0.30)	4.67 (0.37)	2.71 (0.72)
M68c	44.7 (0.66)	4.37 (0.19)	5.24 (0.17)	4.17 (0.05)

Table 3: Mechanical properties of SHCC (standard deviations are given in parentheses)

Figure 5 shows the stress-strain relations of the SHCC investigated under tensile loading. The SHCC M61 shows an average strain capacity of 2.14% at a tensile strength of 4.14MPa with a uniform crack distribution over the specimen length. The curves obtained from three tests are in a narrow range, scattering is low. The average first-crack stress, which is directly related to the tensile stress of the matrix, is 3.35MPa. This value is higher for the mix M68a (3.77MPa), which can be traced back to the higher cement content of the M68a composition. The tensile strength of the composite and its strain capacity are higher for mix M68a as well, which, in respect of tensile strength is probably due to an improved fibre-matrix bond. However, the scattering of the results increased in comparison to mix M61. One possible explanation for this could be the fact that fewer crack inducing micro-defects might be present in the matrix of M68a since the content of unhydrated fly ash particles serving as micro-defects is low in this SHCC. The number of micro-defects increases again when SAP particles are added. Accordingly the scattering of the results decreases, especially in the case of mix M68c (SAP plus extra water). The first-crack stress is highest for mix M68b (4.71MPa), which can be traced back to the lowest effective water-to-binder ratio due to the absorption of a part of mix water

by SAP. Obviously the simultaneous weakening effect of voids induced by adding SAP particles is more pronounced when the material is tested under compression than it is under tension. This seems to hold true also for mix M68c, which despite the addition of extra water (along with SAP) yields a first-crack stress of 4.37MPa, albeit higher than the corresponding value of the mix M68a. Remarkably, the mix M68c shows the best mechanical performance with regard to tensile strength (5.24MPa) and strain capacity (4.17%). The reasons for the more modest performance of mix M68b in terms of these parameters are still to be clarified. One possible explanation for this phenomenon can have to do with the difference in the workability of the SAP-enriched mixes with and without extra water, respectively. Possibly PVA fibres could not be distributed in the stiffer mix M68b (SAP, but no extra water) as evenly over the matrix volume as in the case of a more flowable mix M68c (SAP plus extra water).



Figure 5: Stress-strain relations obtained from uniaxial tension tests for SHCC compositions under investigation

3.2 Results of Freeze-thaw Investigations

The progress of the capillary water absorption by SHCC specimens during the pre-saturation phase is displayed in Figure 6. Due to the higher water-to-binder ratio and the lower cement content of mix M61 in comparison to M68, the total increase in weight through absorption of the sodium chloride solution is nearly twice as high for M61 as for M68a. With the addition of SAP (M68b) an uptake of the solution similar to the case of M68a is apparent with some difference, however, in the first three days of the test. The water absorption of mix M68c (SAP plus extra water) is higher than for other M68-compositions but lower than that of M61.



Figure 6: Capillary suction in the run-up to Figure 7: Weathering in the CDF-test CDF test



Figure 8: Condition of the specimen surfaces for different SHCC compositions after completion of the CDF-test

Figure 7 shows the results of the freeze-thaw investigation according to the CDF procedure. For mix M61 the loss in mass of the SHCC at the end of the testing is close to 1500 g/m², i.e. to the value which is recommended as the limit for ordinary concrete. However, due to the anchoring function of the fibres, not all the degraded material could be separated from the specimen during the cleaning of the test surface in an ultrasonic bath (cf. Figure 8). The poor freeze-thaw behaviour of relatively young fly ash rich compositions is well known for ordinary concrete [16]. With the smaller water-to-binder ratio and a higher cement portion in the mix, the material loss for M68a is reduced to less than 500g/m². Despite the lower material loss in comparison to M61, the general appearance of the test surface is similar to that of M61 (cf. Figure 9). The behaviour of specimens made of SHCC with the addition of SAP in respect of freeze-thaw resistance is considerably higher. Due to the well distributed system of SAP voids in mixes M68b and M68c, the loss in mass is clearly less than 250g/m². The extra water in the case of M68c does not make a great deal of difference: the mass loss of this SHCC was only slightly higher than for mix M68b (SAP, but no extra water), cf. Figure 8. The test surfaces appear similarly smooth for both these SHCC compositions, which lost only the very outer matrix layer (cf. Figure 8 for the mix M68c).

3.3 Shrinkage

The average curves obtained from the total shrinkage measurements are displayed in Figure 9. After 38 days of measurement, the total shrinkage strain of SHCC M61 is about 25% lower than the corresponding values obtained for mix of M68a, most probably due to the smaller cement content and the higher fly ash content. By adding SAP in the case of M68b and M68c, the total shrinkage decreases slightly in comparison to M68a. This behaviour is more pronounced for mixture M68b (SAP, but no extra water).



Figure 9: Average results of total shrinkage measurements

Figure 10: Average results of autogenous shrinkage measurements

Figure 10 depicts the strain development of SHCC over time as observed in autogenous shrinkage tests. The most remarkable finding is that mixtures M68b and M68c show no shrinkage but indeed swelling behaviour in the first two weeks after casting, which obviously results from the addition of SAP particles to these mixtures. Not surprisingly the swelling of mix M68c, which contains extra water, is more pronounced. Mixture M61 has only very moderate autogenous shrinkage. This could be expected, considering a relatively high water-to-cement or water-to-binder ratio of this mix. More complex behaviour is observed in the case of mix M68a. The comprehensive interpretation for such behaviour is still to be found. One possible explanation is, however, the mode of the data evaluation. Figure 10 shows only deformations which occurred after final set of the SHCC, since the autogenous deformation of concrete in the plastic stage does not lead to stress and so can be neglected in many instances. The final set time was estimated from the measured temperature curves, which may not be sufficiently accurate. In the ongoing investigation further methods are to be applied in order to obtain more reliable information on the setting behaviour of the SHCC compositions presented in this study.

4. Summary

In this study the use of SAP in SHCC to achieve multiple purposes has been demonstrated. The chief findings of the study can be summarised as follows:

- An increase in the ductility of the composite as well as a decrease in the scattering of its material properties has been observed for SHCC compositions containing SAP. It is suggested to be due to SAP particles acting as well distributed micro-defects in SHCC.
- The freeze-thaw resistance of SHCC with de-icing solution can be considerably improved by using SAP as a concrete admixture.
- Capillary water absorption is largely unaffected by the addition of SAP insofar as no extra water is used. In the latter case the water absorption increases.
- Some decrease in total shrinkage was measured for the SHCC containing SAP in the first few weeks after concrete production. The autogenous deformations turned from shrinkage in the case of SHCC without SAP to swelling in the case of SHCC with SAP addition.

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