Materials and Structures (2017) 50:14
DOI 10.1617/s11527-016-0868-7

ORIGINAL ARTICLE

Effect of superabsorbent polymers (SAP) on the freeze–thaw resistance of concrete: results of a RILEM interlaboratory study

Viktor Mechtcherine · Christof Schröfl · Mateusz Wyrzykowski · Michaela Gorges · Pietro Lura · Daniel Cusson · Jim Margeson · Nele De Belie · Didier Snoeck · Kazuo Ichimiya · Shin-Ichi Igarashi · Vyacheslav Falikman · Stefan Friedrich · Jürgen Bokern · Patricia Kara · Alicja Marciniak · Hans-Wolf Reinhardt · Sören Sippel · António Bettencourt Ribeiro · João Custódio · Guang Ye · Hua Dong · Jason Weiss

Received: 19 August 2015 / Accepted: 7 April 2016 / Published online: 10 August 2016

Abstract This article presents the results of an interlaboratory experimental study performed by 13 international research groups within the framework of the activities of the RILEM Technical Committee 225-SAP “Applications of Superabsorbent Polymers in Concrete Construction”. Two commercially available superabsorbent polymers (SAP) were tested in terms of their influence on the freeze–thaw resistance of ordinary concrete. To test the robustness of the method, all participating laboratories used locally produced materials. Furthermore, following this aim, various accelerated methods were used to estimate the resistance of the concrete to freeze–thaw cycles. The effect of adding SAP was from insignificant to

---

V. Mechtcherine (✉) · C. Schröfl · M. Gorges
Institute of Construction Materials, Technische Universität Dresden, Dresden, Germany
e-mail: mechtcherine@tu-dresden.de

M. Wyrzykowski · P. Lura
Swiss Federal Laboratories for Materials Science and Technology, Empa, Dübendorf, Switzerland

P. Lura
Institute for Building Materials, ETH Zurich, Zurich, Switzerland

D. Cusson · J. Margeson
National Research Council Canada, Ottawa, Canada

N. De Belie · D. Snoeck
Magnel Laboratory for Concrete Research, Ghent University, Ghent, Belgium

K. Ichimiya
Department of Civil & Environmental Engineering, Oita National College of Technology, Oita, Japan

S.-I. Igarashi
Institute of Science and Engineering, Kanazawa University, Kanazawa, Japan

V. Falikman
Moscow State University of Civil Engineering, Moscow, Russia

S. Friedrich · J. Bokern
BASF Construction Solutions GmbH, Trostberg, Germany

P. Kara
Institute of Materials and Structures, Riga Technical University, Riga, Latvia

A. Marciniak
Department of Building Physics and Building Materials, Lodz University of Technology, Lodz, Poland

H.-W. Reinhardt · S. Sippel
Department of Construction Materials, University of Stuttgart, Stuttgart, Germany

considerably positive in terms of improvement in material performance as determined by reduced mass loss after freeze–thaw cycles; only one participant observed worsening of the material behaviour. At the same time, due to the addition of SAP, a much less pronounced decrease in the dynamic Young's modulus was observed as a result of freeze–thaw testing without deicing salt.

Keywords Air-entraining agent · CDF test · CIF test · Deicing salt · Frost resistance · Freeze–thaw · Interlaboratory study · Scaling · Slab test · Superabsorbent polymer

1 Introduction

The introduction of superabsorbent polymers (SAP) as a novel admixture for cementitious materials, originally conceived as an internal curing agent for controlling autogenous shrinkage in high-performance concrete [1], makes available a number of new possibilities with respect to water control as well [2, 3]. This includes control over the rheological properties of fresh concrete [4] in addition to purposeful water absorption and/or delayed water release in either fresh or hardened concrete [5]. Notable examples are: inducing an abrupt change in rheological behavior during shotcreting [6], reduction of the thermal expansion coefficient [7], reduction of fire spalling in HPC [8], and the onset of self-sealing [9] and self-healing [10]. However, the potential for innovation is far wider, for example, for creating size- and shape-designed pore systems in concrete that could improve its durability, especially in terms of freeze–thaw resistance [11–15]. The latter application can be especially relevant as an alternative for traditional air-entrainment in cases where due to long concreting times, excessively fluid mixtures, or high ambient temperatures a portion of the entrained air voids escapes the mixture. Pore systems built up as a result of SAP-addition seem to remain stable during the initial stages of hardening [16] regardless of the consistency of the concrete, the addition of superplasticizer, or the method of placement and compaction.

The increasing interest in the use of SAP as a concrete admixture and the need for more concentrated scientific exchange among research groups led to the initiation of the RILEM Technical Committee 225-SAP "Application of Superabsorbent Polymers in Concrete Construction" in 2007. A key goal was to compile a state-of-the-art report, which was finalised in 2012 [2]. A first interlaboratory study was performed by 13 international research groups on autogenous shrinkage of HPC with internal curing by SAP [17].

The second interlaboratory study, with the participation of 13 international laboratories (Table 1) recently focused on the freeze–thaw resistance of ordinary concrete modified by the addition of SAP; the results are presented in the article at hand. The concrete had the same basic recipe in all participating labs but was prepared with locally produced materials. In addition to the reference concrete mixtures, concretes with two commercially available superabsorbent polymers (SAP) were tested. Various accelerated methods were used in estimating the concrete resistance to freeze–thaw cycles, with samples exposed to water both with and without deicing salt.

The aim of this study was to verify that the SAP-technology is robust and, hence, to be recommended for field applications. Furthermore, since data on the effects of SAP-addition on various properties of concrete in its fresh and hardened states are still quite spare, the results of the interlaboratory tests should broaden this database as well.

The laboratory figures given in Table 1 are to serve as reference numbers when presenting data obtained in the respective laboratories. The preliminary tests and definition of concrete mixtures were accomplished at TU Dresden. All data were summarised and evaluated at TU Dresden and Empa, where the draft of this article was prepared as well. The work was comprehensively discussed by all participants of the study prior to submission of the manuscript.

A. Bettencourt Ribeiro · J. Custódio
Concrete and Cement Testing Laboratory (LabTech),
National Laboratory for Civil Engineering (LNEC),
Lisbon, Portugal

G. Ye · H. Dong
Department of Materials & Environment, Delft University
of Technology, Delft, Netherlands

J. Weiss
Civil Engineering Materials, Purdue University,
West Lafayette, IN, USA

Table 1 Participants of the interlaboratory study

No.	Participating institution	Principal investigator	Country
1	Empa, Swiss Federal Laboratories for Materials Science and Technology	Mateusz Wyrzykowski	Switzerland
2	National Laboratory for Civil Engineering	António Bettencourt Ribeiro	Portugal
3	National Research Council Canada	Daniel Cusson	Canada
4	Delft University of Technology	Guang Ye	The Netherlands
5	University of Stuttgart	Hans-Wolf Reinhardt	Germany
6	Purdue University	Jason Weiss	United States
7	Kanazawa University	Shin-ichi Igarashi	Japan
8	Oita National College of Technology	Kazuo Ichimiya	Japan
9	Technische Universität Dresden	Viktor Mechtcherine	Germany
10	BASF Trostberg	Stefan Friedrich	Germany
11	Ghent University	Nele De Belie	Belgium
12	Moscow State University of Civil Engineering	Vyacheslav Falikman	Russia
13	Riga Technical University	Patricia Kara	Latvia

2 Concrete compositions and mixing procedure

The composition of the reference concrete is that of an ordinary concrete with a water-to-cement ratio (W/C) of 0.45, as used in road construction in Germany. The composition employed by *Participant 9* and suggested to all participants is presented in Table 2. A normal-strength ordinary Portland cement Type I according to EN 197–1 [18] with no further specifications was recommended to all participants. The cements used by the participants were CEM I 32.5 or 42.5. In addition, Class F fly ash [19] was used by *Participant 6* in addition to ordinary cement.

As aggregates, quartz river sand and gravel as well as crushed diabase were suggested with grain size ranges as given in Table 2. Aggregate fractions had to be composed by the participants in such a way that the resulting grading curve would be in the range between A and B, in Fig. 1, with possible deviations allowed for some fractions, according to the denomination of standard grading curves in DIN 1045–2 [20]. As an example, the grading curve used by *Participant 9* is presented in Fig. 1. All participants were encouraged to utilize locally available, typical raw materials. Accordingly river gravels, river sands, quartz sands or other crushed stone fractions were selected, representing typical, locally available aggregates for ordinary concrete production. In the course of this selection, the basic concrete recipe was allowed to be modified slightly.

The high-range water-reducing admixture (HRWRA) was based on β–naphthalene sulfonate (BNS-type) and is a commercial product by BASF, Trostberg/Germany (named Woerment FM 30 / BV 30 at the time of the experiments, in the meantime renamed as MasterRheobuild 30). At 20 °C it had density equal to 1.20 g/cm^3. Its dosage by weight of cement (bwoc) could be adjusted on demand (Table 3).

The reference concrete (Table 2) was established by *Participant 9*. It was well workable, reaching a spread diameter between 420 and 480 mm as measured according to the table flow test EN 12350–5 [21] and, thus, belonging to consistency class F3 ("soft"). The participants in the study were expected to adjust the dosage of the HRWRA to obtain the same consistency class; final dosages are summarized in Table 3.

Two SAP-types were selected for the study; these SAP had proven to be the most beneficial in preliminary freeze–thaw tests run by *Participant 9* (data unpublished but distributed among the members of RILEM TC 225–SAP). The SAP samples were filled in small plastic containers, caped and shipped to each participant in the round-robin test by the TC chair. Each participant was encouraged to take care of appropriate handling of the SAP samples according to good laboratory practice and to not treat them in any extra manner. The SAP samples have hence remained macroscopically non-caking, easily pourable in their

Table 2 Composition of the reference concrete employed by *Participant 9* and suggested to all participants

	Unit mass [kg/m^3]	Unit volume [L/m^3]
Cement CEM I 32.5 R	350	111.5
Water (for W/C = 0.45)	157.5	157.5
Air (estimated)	–	20.0
Quartz sand 0.06/0.2	135.5	51.1
Quartz sand 0/2	425.9	160.7
Quartz sand or quartz gravel 2/8	271.0	102.3
Diabase split 8/22	1103.4	394.2
High-range water-reducing (HRWR) admixture, aqueous solution as obtained, 0.9 % bwoc	3.15	2.9

Bwoc by weight of cement

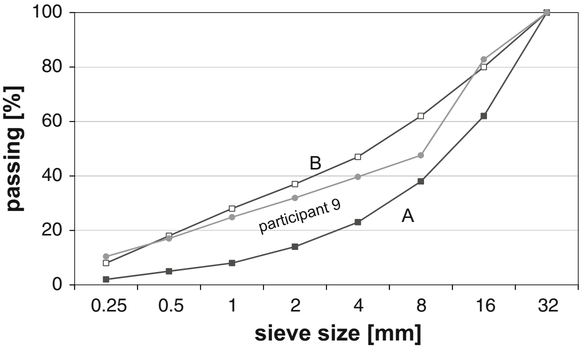

Fig. 1 Grading curve of aggregates utilized by *Participant 9*. A and B are standard grading curves as specified in DIN 1045–2; a *curve* falling between the boundary curves A and B is recommended for concretes applied in road construction and was to be followed by all concretes within the study

particulate state and not sticky in each participating laboratory. Hence, the individual moisture contents of the polymers at each place can be considered as approximately same and very low. Thus, these inherent moisture contents in the SAP samples were not regarded in the concrete mixture design. The polymers were obtained from two individual industrial producers; one of the SAP is commercially available, the other is still in the development. Both of them are anionic polyacrylamides with medium cross-linking and charge density. The given chemical properties were found beneficial for SAP use in concrete, since such SAP are characterised by high retention over time in extracted cement pore solution, as determined by the so-called “tea-bag method” [16]. In this method, mass of absorbed liquid (water or pore solution) is determined by dipping the SAP enclosed in a permeable bag (tea-bag) in chosen liquid and measuring increase in weight at defined time intervals. Qualitatively, both SAP samples can be classified as “retentive” polymers with respect to the cement pore solution, which indicates that they are able to retain the absorbed pore solution until it is sucked by the hydrating cement paste [5, 16]. Free sorptivity of SAP samples in a filtrate of cement suspension (CEM I 42.5 N, w/c = 4.3 [16]) after 1 h was equal to 37 g/g and 35 g/g for SAP 1 and SAP 2, respectively, while after 7 h it slightly decreased to about 35 g/g and 33 g/g for SAP 1 and SAP 2, respectively. The extent of liquid absorption within the individual cement-based matrices was assessed via workability-related concrete tests by each participant, according to contemporary laboratory practice (cf. the next text paragraph and Table 3).

According to both suppliers’ information, the polymers have irregular particle shapes as they were produced via the bulk polymerization technique, followed by crushing into single particles. Their particle size distributions in the dry state are presented in Fig. 2 as measured by laser granulometer (LS 13320, BeckmanCoulter, Krefeld/Germany) in 2–propanol as dispersing agent. The dosage of SAP in the recommended mixtures was set as 0.15 % by weight of cement.

Table 3 Admixture dosages applied by each participant (index numbers according to Table 1)

Part.	HRWRA	SAP 1	SAP 2	Annotations
	% bwoc	Dry % bwoc	Dry % bwoc	
1	R.45: 0.29 S1.50: 0.41 S2.50: 0.29	S1.50: 0.15	S2.50: 0.15	Different HRWRA used
2	All mixtures: 0.65	S1.50: 0.12	S2.50: 0.13	
3	All mixtures: 0.9	S1.50: 0.15	S2.50: 0.15	
4	All mixtures: 0.6	S1.50: 0.6	S2.50: 0.6	
5	R.45: 1.1 S1.50: 1.1 S2.50: 1.1 A.45: 1.1	S1.50: 0.15	S2.50: 0.15	
6	R.42: 0.16 S1.49: 0.20 S2.49: 0.23 S1.49AEA: 0.16 S2.49AEA: 0.14	S1.49: 0.40 S1.49AEA: 0.40	S2.49: 0.40 S2.49AEA: 0.40	Class F Fly Ash was used in an amount 25 % bwoc AEA dos. 0.12 % bwoc AEA dos. 0.12 % bwoc
7	R.45: 0.55 S1.50: 0.55 S2.50: 0.55 R.50: 0.25 A.45: 0.55	S1.50: 0.16	S2.50: 0.16	
8	R.45: 1.22 R.50: 0.88 A.45: 1.22 S1.45: 1.22 S2.45: 1.22	S1.45: 0.15	S2.45: 0.15	
9	R.45: 0.9 S1.50: 0.9 S2.50: 0.9 R.50: 0.28 A.45: 0.9 S1.45: 1.4 S2.45: 1.4	S1.50: 0.15 S1.45: 0.15	S2.50: 0.15 S2.45: 0.18	
10	R.45: 0.75 S1.50: 0.75 S2.50: 0.75 R.50: 0.28 A.45: 0.23	S1.50: 0.15	S2.50: 0.15	AEA dos. 0.9 % bwoc
11	R.45: 0.09 S1.50: 0.09 S2.50: 0.09 R.50: none A.45: 0.02 S1.45: 0.36 S2.45: 0.41	S1.50: 0.15 S1.45: 0.15	S2.50: 0.15 S2.45: 0.15	AEA dos. 0.025 % bwoc

Table 3 continued

Part.	HRWRA	SAP 1	SAP 2	Annotations
12	R.45: 0.6 S1.50: 0.6 S2.50: 0.6 R.50: 0.6 A.45: 0.6	S1.50: 0.15	S2.50: 0.15	Different HWRA used Different AEA, dos. 0.4 % bwoc
13	R.45: 0.9 S1.50: 0.9 S2.50: 0.9 R.50: 0.9 A.45: 0.9	S1.50:: 0.15	S2.50: 0.15	

Due to the absorption of mixing water by the SAP, the basic W/C should be increased to retain the consistency level F3 in each batch of concrete. This additional amount of water was 17.5 kg/m^3 (corresponding to an increase in W/C from 0.45 to 0.50) for the concrete prepared by *Participant 9*. The participants of the study were advised to proceed as follows: increase the W/C from 0.45 to 0.50 but retain the amount of HRWRA and then dose as much SAP powder as is required to regain consistency class F3. The SAP and the extra water were added on top of the mix design for the reference mixtures. Hence, it was accepted that different dosages of SAP would be used in the different laboratories.

On the contrary, some participants chose a widely recognized alternative route to cope with the effects of SAP on the fresh concrete's consistency. They used no additional water and, hence, kept the W/C constant at 0.45. To retain consistency class F3, the dosage of HRWRA was increased.

For the sake of further quantifying the effect of SAP with respect to the freeze–thaw resistance, two optional mixtures complemented the study program. One is an additional reference mixture with a W/C increased to 0.50, i.e., additional water was added on top, but no SAP was incorporated. In this additional reference, the HRWRA dosage had to be reduced to stay within consistency class F3. Secondly, a concrete directly according to the reference mixture but incorporating a conventional air entraining agent (AEA) was studied. As the air entraining agent, a commercial product of BASF, Trostberg/Germany (named LP75 at the time of the experiments and subsequently renamed as MasterAir114) was provided to all participants. Its dosage should amount to 0.11 % bwoc and it should be added on top, i. e., without further modification of the basic concrete recipe; exceptions are indicated in Table 3. Because LP75 has a liquefying side effect, the dosage of HRWRA was reduced accordingly to remain within consistency class F3. Table 4 summarizes all mixtures of the study.

Table 5 gives the mixing sequence for the concretes as suggested on the basis of preliminary testing by *Participant 9*. Slight modifications were allowed to adjust to local conditions. Consolidation was conducted using standard measures by each participant. Curing followed the respective instructions of the individual standards and test procedures followed by each participant.

Participant 6 implemented considerably different mixture compositions (see Tables 3, 4). Class F fly ash [19] was used in all concretes in the dosage of 25 % bwoc. The reference mixture had a W/C of 0.53 and water-to-binder ratio (W/B) of 0.42, while for the concretes with SAP it was 0.61 and 0.49, respectively. In addition to the reference mixture, further referred to as R.42, and two mixtures with SAP, further referred to as S1.49 and S2.49, further two mixtures were prepared by *Participant 6* containing the two SAP, respectively, together with an air-entraining agent, further referred to as S1.49AEA and S2.49AEA; see Table 4. More details on the mixtures and tests by *Participant 6* can be found in [22].

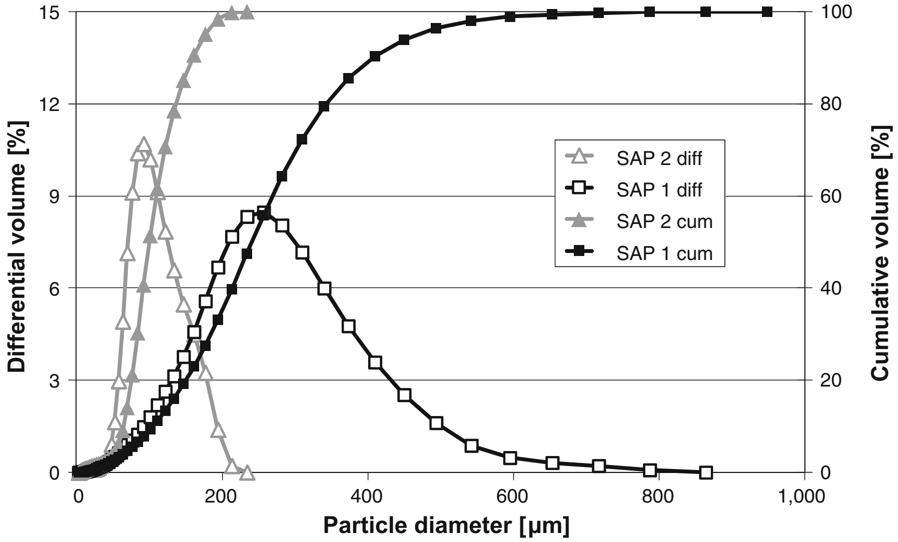

Fig. 2 Particle size distributions of the two SAP samples in the dry state as determined with laser granulometry by *Participant 9*

Table 4 Overview of concrete mixtures as prepared by different participants

Acronym	W/C	SAP 1	SAP 2	Conventional AEA	Participants
R.45	0.45	–	–	–	1–5, 7–13
S1.50	0.50	X	–	–	1–5, 7–13
S2.50	0.50	–	X	–	1–5, 7–13
R.50	0.50	–	–	–	7–13
A.45	0.45	–	–	X	5, 7–13
S1.45	0.45	X	–	–	11
S2.45	0.45	–	X	–	9, 11
R.42[a]	W/B 0.42	–	–	–	6
S1.49[a]	W/B 0.49	X	–	–	6
S2.49[a]	W/B 0.49	–	X	–	6
S1.49AEA[a]	W/B 0.49	X	–	X	6
S2.49AEA[a]	W/B 0.49	–	X	X	6

[a] Particular mixes with fly ash used by *Participant 6*; see text

Table 5 Recommended mixing procedure

Time (min)	Action	Mixer speed (min)	Duration (min)
–02:00–00:00	Homogenize all dry powders, incl. dry SAP in the resp. mixtures	25	2.00
00:00–00:30	Addition of water	25	0.50
00:30–01:30	Mixing	25	1.00
01:30–01:45	Addition of superplasticizer	25	0.25
01:45–03:45	Mixing	40	2.00

3 Test methods

The fresh concretes were characterized with respect to their workability (using table flow test or slump flow test), air content, and density (Table 16). The compressive strength, Young's modulus in compression, and the splitting tensile strength were measured at a concrete age of 28 days (Table 17).

The freeze–thaw resistance, both with and without deicing salt, was the core of the present interlaboratory study. The test methods are summarized in Table 6.

4 Experimental results

4.1 Properties in the fresh state and mechanical properties of hardened concrete

The dosages of all admixtures were adjusted by each participant to meet the requirements of workability as well as the air content in the AEA-mixtures.

In Fig. 3 the results of air content measurements in fresh mixtures are presented. As can be seen, the addition of SAP did not lead to additional air entrainment. Thanks to this, freeze–thaw performance, discussed later, can be attributed solely to the action of the SAP. One prominent outlier was reported by *Participant 10*; it was found that in the A.45 mixture the amount of mixing water was incorrect. This had a further effect on the results of this concrete for this participant.

It is worth noting that *Participant 1* had to use an alternative HRWRA, different to the distributed sample of FM30 (Table 3) because the target consistency with any SAP-containing mixture could not be adjusted with acceptable dosages of FM30, while significant segregation was the consequence of overdosage. Most probably, this behaviour was the consequence of incompatibility among the chemical admixtures in combination with the cement, a local CEM I 42.5 N from Switzerland. *Participant 1* used the polycarboxylate-based superplasticizer Glenium ACE404 (BASF) instead, which permitted reaching the required consistency without segregation.

Tables 7, 8 and 9 summarise the mechanical properties (compressive strength, Young's modulus, and tensile splitting strength, respectively) of the concretes tested. Although significant differences, some stemming from the varied raw materials used and some arising out of differences in mixture compositions, were found among the participants, the trends within the experimental programme regarding the influence of SAP or entrained air content are consistent with previous studies [2, 17] and can be summarized as follows. When SAP are added together with additional water, the degradation of mechanical properties is roughly similar to that obtained by a simple increase in W/C. At the same time, SAP alone do not lead to a significant change in mechanical properties. This can be seen when the results for concretes S1.50 and S2.50 are compared with those for the reference concrete R.50 of the same W/C or, similarly, concretes S1.45 and S2. 45 with R.45; see results for *Participants 7–13* in Tables 7, 8 and 9. On the contrary, air entrainment leads to a clear reduction in the mechanical properties for a given W/C; this negative effect is considerably higher than increasing W/C together with the addition of SAP.

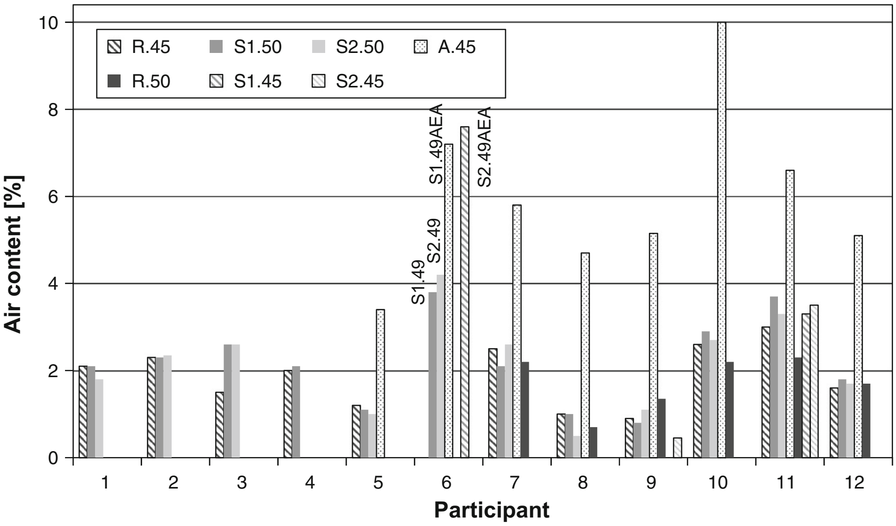

Fig. 3 Air content in fresh concrete (index numbers according to Table 1; *Participant 13* did not measure the air content)

4.2 Freeze–thaw resistance

4.2.1 Tests with deicing salt

As the deicing salt, sodium chloride (NaCl) was used. The concentration of NaCl in an aqueous solution differed in accordance with the instructions dictated by the test procedures chosen; see Table 6.

The only group utilizing the procedure according to ASTM C672 [27] was *Participant 7*. In order to visualize the character of the scaling results better, they are presented in Fig. 4 while further results are presented in tables in order to enable quantitative comparison.

The quantification of scaling and the assignment of a category of freeze–thaw resistance are explicitly discouraged in ASTM C672 [27], which was applied by *Participant 7;* see Fig. 4. However, it can be concluded that both SAP types improved scaling resistance when compared to the reference mixture. While the difference after 25 freeze–thaw cycles is the same for both SAP, SAP 2 was clearly more efficient than SAP 1 in the long term.

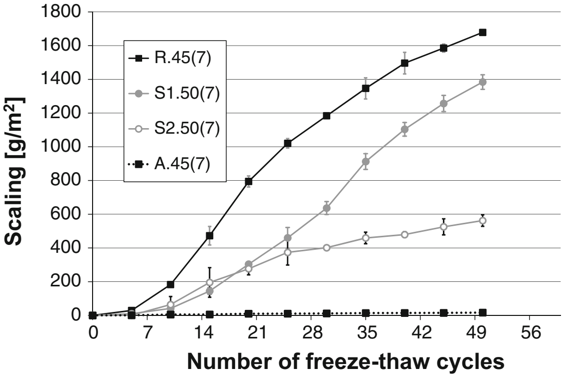

Fig. 4 Mass loss due to scaling obtained from the ASTM C672 tests by *Participant 7*

Table 6 Test methods applied to assess freeze–thaw resistance both with and without deicing salt (index numbers according to Table 1; blank table fields indicate "not performed")

Part.	With deicing salt	NaCl (wt % aqueous solution)	Without deicing salt (tap water)
1	SIA 262/1:2003 appendix C [23]	3	
2	Slab test in DIN CEN/TS 12390–9 [24]	3	Slab test in DIN CEN/TS 12390–9 [24]
3			ASTM C666 procedure A [25]
4	CDF in EN 12390-9 [24]	3	
5	RILEM recommendation CDF [26]	3	
6			ASTM C666 procedure A [25]
7	ASTM C672 [27]	3	
8			JIS A 1148 [28]; ASTM C666 [25]
9	CDF in DIN CEN/TS 12390–9 [24]	3	CIF in DIN CEN/TS 12390–9 [24]
10	CDF in DIN CEN/TS 12390–9 [24]	3	
11	Slab test in DIN CEN/TS 12390–9 [24]	3	Slab test in DIN CEN/TS 12390–9 [24]
12	GOST 10060.0-95 [29], GOST 10060.1-95 [30], GOST 10060.2-95 [31] 2nd method—freezing at −18 °C 3rd method—freezing at −50 °C GOST 26134-84 [32]	5	
13	GOST 10060.0-95 [29], GOST 10060.1-95 [30], GOST 10060.2-95 [31], 2nd method—freezing at −18 °C GOST 26134-84 [32]	5	

Table 7 Compressive strength at an age of 28 days (index numbers according to Table 1; blank table fields indicate "not measured", average ± standard deviation)

Part.	Mixture/compressive strength (MPa)						
	R.45	S1.50	S2.50	A.45	R.50	S1.45	S2.45
1	61.0 ± 1.0	56.1 ± 0.8	56.5 ± 0.6				
2	56.0 ± 2.0	51.4 ± 1.9	50.8 ± 0.9				
3	43.2 ± 4.3	43.3 ± 0.2	41.0 ± 2.5				
4	50.0 ± 2.0	40.1 ± 1.2	41.9 ± 0.5				
5	57.0 ± 4.5	51.7 ± 0.8	47.8 ± 0.4	46.3 ± 0.2			
6	R.42: 37.0 ± 1.0	S1.49: 36.9 ± 1.5	S2.49: 36.2 ± 0.3				
		S1.49AEA:33.6 ± 1.2	S2.49AEA:30.8 ± 0.2				
7	50.5 ± 0.9	40.1 ± 2.6	43.7 ± 2.3	32.3 ± 1.2	42.1 ± 2.8		
8	43.5 ± 2.5	36.1 ± 0.8	33.2 ± 1.4	34.0 ± 0.6	42.1 ± 1.4		
9	48.2 ± 0.1	46.9 ± 1.3	43.1 ± 0.9	47.2 ± 0.8	44.6 ± 0.9		47.2 ± 1.6
10	57.8 ± 1.5	48.6 ± 1.1	48.0 ± 1.6	24.9 ± 0.7	52.0 ± 0.6		
11	57.8 ± 0.7	49.0 ± 1.0	48.6 ± 0.8	33.8 ± 1.4	52.0 ± 0.9	58.0 ± 1.0	50.0 ± 2.1
12	47.3 ± 0.3	47.0 ± 0.4	43.3 ± 1.0	35.2 ± 0.4	40.0 ± 0.8		
13	56.7 ± 0.4	48.7 ± 0.8	53.3 ± 0.7	46.0 ± 0.6	53.7 ± 0.8		

The largest group of participants (*Participants 4, 5, 9 and 10*) followed the CDF protocol [24, 26]; see Table 6. Table 10 summarizes the results obtained by each participant. Material loss (scaling) of 1500 g/m^2 is commonly recommended as the maximum acceptable scaling after 28 freeze–thaw cycles.

As can be seen, the reference concretes tested using the CDF test did not fulfil this resistance criterion at any of the participating laboratories.

Applying SAP 1 together with additional water (concretes S1.50) did not significantly influence the scaling results except in the observations of *Participant 4*, where it resulted in the decrease of the amount of scaled material below 1500 g/m^2 after 28 cycles and, consequently, classification of the material as having fulfilled the recommended resistance criterion. A significant reduction in scaling could be observed when the concrete with SAP is compared to the reference concrete R.50 with the same W/C, the only difference being the SAP added, as observed both by *Participant 9,* reduction from 4973 g/m^2 to 3198 g/m^2 *and Participant 10,* reduction from 4060 g/m^2 to 2826 g/m^2; see Table 10.

Concretes prepared with SAP 2 at the increased W/C of 0.50 (concretes S2.50) revealed more significant decreases in scaling than those with SAP 1, as reported by all participants applying the CDF test. In this case, two sets of test results (*Participants* 4 and

Table 8 Modulus of elasticity at an age of 28 days (index numbers according to Table 1; blank table fields indicate "not measured", average ± standard deviation)

Part.	Mixture/Young's modulus (GPa)						
	R.45	S1.50	S2.50	A.45	R.50	S1.45	S2.45
3	29.1 ± 0.6	27.3 ± 0.3	26.3 ± 0.2				
5	33.9 ± 0.6	31.3 ± 0.8	29.9 ± 0.8	28.9 ± 1.3			
6	R.42: 30.9 ± 1.1	S1.49: 29.3 ± 0.7	S2.49: 28.5 ± 1.3				
		S1.49AEA: 25.6 ± 1.6	S2.49AEA: 28.2 ± 1.0				
7	38.8 ± 0.4	34.3 ± 1.9	37.0 ± 1.2	36.3 ± 0.9	36.8 ± 5.3		
8	39.5 ± 1.7	38.6 ± 1.6	36.4 ± 1.2	33.5 ± 1.6	40.9 ± 3.5		
9	40.0 ± 5.5	33.0 ± 1.9	33.7 ± 1.0	39.6 ± 5.1	37.6 ± 0.9		
11	35.1 ± 0.7	34.6 ± 0.8	34.2 ± 1.3	31.7 ± 1.0	34.4 ± 1.4	36.5 ± 0.9	35.9 ± 0.5

Table 9 Splitting tensile strength at 28 days (index numbers according to Table 1; blank table fields indicate "not measured", average ± standard deviation)

Part.	Mixture/Splitting tensile strength [MPa]						
	R.45	S1.50	S2.50	A.45	R.50	S1.45	S2.45
3	3.6 ± 0.3	4.1 ± 0.3	3.6 ± 0.4				
5	3.3 ± 0.1	3.3 ± 0.1	3.0 ± 0.2	3.0 ± 0.2			
7	3.5 ± 0.2	3.42 ± 0.02	3.49 ± 0.03	3.04 ± 0.03	3.0 ± 0.2		
8	3.6 ± 0.7	3.3 ± 0.4	3.3 ± 0.5	3.2 ± 0.2	3.7 ± 0.5		
9	3.0 ± 0.1	3.1 ± 0.2	3.0 ± 0.2	2.9 ± 0.2	3.3 ± 0.1		2.7 ± 0.2
11	3.7 ± 0.3	3.4 ± 0.1	3.5 ± 0.4	2.6 ± 0.3	3.4 ± 0.1	4.0 ± 0.3	3.7 ± 0.3
13	3.5 ± 0.3	3.1 ± 0.3	3.4 ± 0.5	3.1 ± 0.5	3.4 ± 0.4		

Table 10 Freeze-thaw tests results according to the CDF test (*Participants 4, 5, 9, 10*) and SIA262/1:2003 test (*Participant 1*) (average ± standard deviation)

Participant/test	Material	Scaled material (g/m^2)					
		Number of freeze-thaw cycles					
		0	4	6	14	28	56
4/CDF	R.45	0	270 ± 36	500 ± 150	1900 ± 221	10130 ± 679	–
	S1.50	0	60 ± 28	150 ± 34	450 ± 69	870 ± 127	–
	S2.50	0	60 ± 25	130 ± 18	320 ± 71	690 ± 48	–
5/CDF	R.45	0	–	791 ± 182	2842 ± 160 (16)	4796 ± 255	–
	S1.50	0	–	552 ± 119	2906 ± 433 (16)	4927 ± 808	–
	S2.50	0	–	–	1040 ± 148	3225 ± 106	–
	A.45	0	–	–	1234 ± 113	3011 ± 174	–
	A.45rep	0	–	–	–	1102 ± 85	–
9/CDF	R.45	0	92 ± 24	–	1212 ± 233	4267 ± 170	–
	S1.50	0	–	–	739 ± 304	3198 ± 406 (30)	–
	S2.50	0	42 ± 5	–	612 ± 203	2347 ± 189	–
	R.50	0	208 ± 34	–	2000 ± 140	4973 ± 264	–
	S1.45	0	108 ± 16	–	1256 ± 233	3130 ± 161	–
	S2.45	0	125 ± 53	–	929 ± 210	2339 ± 465	–
	A.45	0	69 ± 11	–	300 ± 64	672 ± 107	–
10/CDF	R.45	0	41 ± 2	–	1205 ± 112	3227 ± 145	7464 ± 133
	R.45rep	0	32 ± 9	–	537 ± 68	2064 ± 180	5013 ± 90
	S1.50	0	31 ± 3	–	424 ± 32	2826 ± 276	8427 ± 342
	S2.50	0	22 ± 9	–	383 ± 49	1023 ± 142	2515 ± 209
	R.50	0	139 ± 12	–	1504 ± 75	4060 ± 65	12794 ± 669
	A.45	0	37 ± 9	–	138 ± 5	208 ± 6	316 ± 8
1/SIA 262/1:2003	R.45	0	–	12 ± 4	42 ± 12	85 ± 35	–
	S1.50	0	–	14 ± 11	37 ± 19	59 ± 22	–
	S2.50	0	–	57 ± 34	144 ± 90	236 ± 135	–

Numbers in parentheses refer to exceptional number of cycles whenever the test was not performed at a prescribed age

10) fulfilled the resistance criterion, while all other mixtures did not. Rather surprising in this context is the failure of the mixture S2.45, which was tested by *Participant 9* alone. Obviously, the lower W/C had no distinct influence within the test series of this participant because the scaling is very similar to that of S2.50 from the same laboratory.

The CDF test applied to the mixtures prepared with a conventional air-entraining agent (mixture A.45) was also performed by *Participants 5, 9* and *10*, Table 10. The initial batch prepared by *Participant 5* did not fulfil the resistance criterion while a second batch did. *Participants 9* and *10* reported fulfilling the resistance criterion of 1500 g/m^2. Conventional air entrainment performed consistently better than adding any of the SAP.

According to the categories specified in SIA 262/1:2003 [23], each concrete characterised by *Participant 1* (Table 10) exhibits a high freeze–thaw resistance. The maximum allowed scaling for this class is 200 g/m^2 or, alternatively, 600 g/m^2 and an increase in the scaled material from 14 to 28 cycles lower than from 0 to 14 cycles. Note that according to the standard for the final evaluation the results must be rounded to tenths if lower than 100 g/m^2 and to hundredths if above 100 g/m^2.

As an alternative method to CDF, the slab test according to CEN/TS 12390–9 [24] was performed by *Participants 2* and *11* (Table 11). In this procedure the recommended maximum scaling after 28 freeze–thaw cycles is 1000 g/m^2. The results according to the slab method run by *Participant 11* show that the addition of SAP together with additional water in both concretes S1.50 and S2.50 results in significant reductions in freeze–thaw scaling. Further, when the SAP are added without additional water (concretes S1.45 and S2.45), the resistance criterion is fulfilled, similar to concrete with conventional air entrainment (A.45). Addition of SAP 1 led to an apparent worsening of the freeze–thaw performance according to the tests conducted by *Participant 2*. However, due to the very high scatter, this trend does not appear significant.

The protocol used by *Participants* 12 and 13 is based on measuring the losses in strength and mass for the samples undergoing freeze–thaw cycles. Each sample is classified within a resistance class according to the number of cycles after which no considerable reductions of strength and mass have taken place in the course of the test, i. e., more than 5 % reduction in strength and more than 3 % reduction in mass. *Participant 12* measured the strength on cubic samples in a hydraulic machine, while *Participant 13* estimated it based on ultrasonic pulse velocity measurements. The rating presented in Table 12 for *Participants 12* and *13* is based on a comparison of resistance classes; improvement is demonstrated whenever the resistance class has increased.

A qualitative ranking of the freeze–thaw resistance in the presence of deicing salt is summarised in Table 12. The different testing methods used by the participants did not allow a precise quantification of the effect. A rating with respect to the SAP-free

Table 11 Freeze-thaw tests results according to the slab test (average ± standard deviation)

Participant	Material	Scaled material (g/m^2)					
		Number of freeze–thaw cycles					
		0	7	14	28	42	56
2	R.45	0	956 ± 271	1294 ± 521	1908 ± 531	2994 ± 894	5060 ± 1459
	S1.50	0	1394 ± 185	2762 ± 690	4773 ± 1291	4773 ± 2059	8220 ± 2591
	S2.50	0	1093 ± 491	1999 ± 1084	2838 ± 1738	2838 ± 2123	3674 ± 2409
11	R.45	0	732 ± 246	1717 ± 382	3390 ± 699	4372 ± 895	5050 ± 1030
	S1.50	0	258 ± 134	658 ± 413	1348 ± 900	1697 ± 1112	1934 ± 1312
	S2.50	0	395 ± 155	912 ± 380	1690 ± 800	2098 ± 997	2389 ± 1163
	R.50	0	935 ± 155	2114 ± 251	4275 ± 220	5889 ± 371	7657 ± 570
	S1.45	0	56 ± 12	97 ± 42	184 ± 124	274 ± 162	348 ± 165
	S2.45	0	48 ± 14	142 ± 63	271 ± 151	384 ± 165	467 ± 182
	A.45	0	28 ± 8	65 ± 18	115 ± 48	155 ± 66	223 ± 86

Table 12 Qualitative rating, explained in text, of the freeze–thaw resistance in the presence of deicing salt with respect to the individual SAP-free references with W/C = 0.45 (R.45 concrete) of each participant

Part.	S1.50	S2.50	S1.45	S2.45	A.45	R.50
1	not signif.[a]	not signif.[a]				
2	not signif.	not signif.				
4	++	++				
5	not. signif	+			+/++	
7	+	++ [b]			++ [b]	
9	+	+	+	+	++	−
10 (at 28 cycles)	not signif.	++			++	−
11	+	+	++	++	++	−
12 (−18 °C)	−	not signif.			+	−
12 (−50 °C)	−	not signif.			+	not signif.
13 (−18 °C)	+	+			+	+

Index numbers in Table 1; blank table fields indicate "not studied"

[a] Reference mixture had high freeze–thaw resistance

[b] No resistance criterion specified, more than two times reduction in scaling

reference mixture featuring W/C = 0.45 was conducted within the test series of each individual participant for the final number of cycles. The following rating was applied: no statistically significant effect as compared to the reference, i. e., 95 % significance level according to t-testing of the results at the ultimate number of freeze–thaw cycles unless specified otherwise; − negative effect: increase in scaling; + positive effect: reduction in scaling; ++ considerable positive effect: resistance criterion fulfilled thanks to additive or scaling reduced by at least two times where no criterion was given.

As expected, the mere increase in W/C by 0.05 has a uniformly negative impact, whereas the use of a conventional air-entraining agent is very beneficial. All mixtures prepared with W/C = 0.45 and SAP feature a markedly enhanced freeze–thaw resistance when compared to the reference concrete with the same W/C. It should be noted that these positive results were obtained using both the CDF test (*Participant 9*) and the slab test (*Participant 11*) in DIN CEN/TS 12390–9 [24]. Similarly, SAP added along with additional water (S1.50 and S2.50) considerably improve the freeze–thaw resistance when compared to the reference concrete with the same, increased W/C = 0.50, as evidenced by *Participants 9, 10 and 11*. The largest data basis has been obtained for the prescribed mixtures with SAP 1 and SAP 2, respectively, at W/C = 0.50, compared to the reference concrete with W/C = 0.45. In this case, either no significant effect (*Participants 1, 2, 5, 10*) or from moderate to significant improvement in scaling resistance (*Participants 4, 7, 9, 11, 13*) was observed. In some cases (SAP 1—*Participant 4* and SAP 2—*Participants 4* and *10*) the application of SAP allowed fulfilling the prescribed resistance criteria, which was not possible in the reference concretes.

The results reported by most participants clearly demonstrate that if additional water is added along with the SAP, the freeze–thaw resistance in the presence of deicing salt can vary from no significant effect to moderate or high improvement; whereas if SAP are added without additional water, the freeze–thaw resistance can increase considerably. These observations are in line with the findings in [12–15], where generally beneficial effects of SAP were reported.

SAP 2 appears to be more efficient than SAP 1. Because their fundamental sorption kinetics do not differ and their particles are both irregularly shaped as explained in Sect. 2, the reason for this could be the significantly finer grading of SAP 2 (Fig. 2). In [13] it was reported that when comparing two gradings of one single type of SAP, the portion with diameter in the dry state <150 μm is more beneficial than that with particle sizes of up to 300 μm. Another possible reason for different performance regarding freeze–

thaw resistance may be their different polymer design and composition. As long as the SAP can be stable in a fresh mixture, the long-term performance of air voids entrained by means of SAP when they lose their absorbed water may be potentially reduced if SAP cavities become filled with hydration products. Justs et al. [33] observed with scanning electron microscopy (SEM) cross-sections of low W/C cement paste and reported that a significant volume of the SAP-originated voids (above 50 %) was filled with calcium hydroxide during hardening.

4.2.2 Test without deicing salt

Participants 2 and *11* measured the freeze–thaw resistance without deicing salt according to the slab test specified in [24]. *Participant 2* reported hardly any measurable mass loss due to scaling for all mixtures tested (R.45, S1.50 and S2.50), with a maximum of 10 g/m^2 for the S2.50 concrete. These results are therefore omitted from this presentation. The results from *Participant 11* are presented in Table 13.

The CIF protocol specified in [24] was applied only by *Participant 9*. The results for both scaling and reduction in dynamic Young's modulus are presented in Table 13.

Following the CIF protocol, on the one hand, a scaling limit of 1000 g/m^2 should apply, which was not exceeded by any of the tested concretes, and on the other hand the dynamic modulus of elasticity has to remain at least 80 % of the initial value after 56 freeze–thaw cycles. Taking this into account, both the

Table 13 Freeze-thaw resistance results (average ± standard deviation) without deicing salt: mass loss due to scaling obtained from the slab test by *Participant 11* and mass loss and reduction in relative values of dynamic Young's modulus obtained from CIF test by *Participant 9*

Participant	Material	Number of freeze–thaw cycles					
		Scaled material (g/m^2)					
		0	4	14	28	42	56
11—Slab test	R.45	0	4 ± 2 (7)	11 ± 4	16 ± 7	25 ± 9	28 ± 12
	S1.50	0	4 ± 1 (7)	9 ± 4	16 ± 4	23 ± 7	30 ± 10
	S2.50	0	3 ± 2 (7)	5 ± 4	7 ± 5	11 ± 8	19 ± 8
	R.50	0	3 ± 2 (7)	9 ± 2	17 ± 4	23 ± 4	31 ± 5
	S1.45	0	3 ± 1 (7)	7 ± 2	17 ± 6	27 ± 6	38 ± 7
	S2.45	0	3 ± 3 (7)	5 ± 1	8 ± 4	11 ± 3	12 ± 4
	A.45	0	4 ± 0 (7)	9 ± 3	13 ± 5	20 ± 7	22 ± 9
9—CIF method	R.45	0	22 ± 9	86 ± 50	165 ± 80	204 ± 96	260 ± 104
	S1.50	0	0 ± 0	24 ± 5	56 ± 15	79 ± 18	110 ± 22
	S2.50	0	7 ± 1	15 ± 3	29 ± 5	46 ± 11	61 ± 14
	R.50	0	10 ± 1	28 ± 6	60 ± 12	161 ± 41	537 ± 138
	S1.45	0	3 ± 1	10 ± 2	26 ± 5	47 ± 9	63 ± 63
	S2.45	0	14 ± 2	37 ± 7	65 ± 7	91 ± 12	118 ± 15
	A.45	0	20 ± 4	45 ± 4	69 ± 5	95 ± 10	124 ± 11
		Reduction in dyn. Young‘s modulus, relative values [-]					
9—CIF method	R.45	1	1.00 ± 0.08	0.96 ± 0.10	0.87 ± 0.03	0.71 ± 0.03	0.54 ± 0.05
	S1.50	1		0.97 ± 0.01	0.89 ± 0.02	0.82 ± 0.05	0.68 ± 0.05
	S2.50	1	0.97 ± 0.01	0.98 ± 0.01	0.99 ± 0.01	0.98 ± 0.01	0.99 ± 0.01
	R.50	1		0.65 ± 0.07	0.37 ± 0.06	0.14 ± 0.07	0.04 ± 0.01
	S1.45	1	0.98 ± 0.01	0.98 ± 0.01	0.98 ± 0.02	0.99 ± 0.02	0.95 ± 0.02
	S2.45	1	0.98 ± 0.02	0.95 ± 0.02	0.97 ± 0.03	0.99 ± 0.04	
	A.45	1	0.99 ± 0.01	0.98 ± 0.01	0.96 ± 0.01	0.97 ± 0.01	0.96 ± 0.05

Numbers in parentheses refer to the number of cycles at which the test was performed according to the slab test (7 cycles, different to 4 cycles according to the CIF test and indicated in the column header) reference mixtures as well as that with SAP 1 at W/C = 0.50 failed, whereas the others passed the test.

Participants 3, 6 and 8 characterized the freeze–thaw resistance without deicing salt according to ASTM C666 [25]; among them, *Participants 3* and *8* used the recommended concrete composition. The results are presented in Table 14. Please note different mixture compositions used by the three participants (mixtures S1.50 and S2.50 by *Participant 3*, mixtures S1.45 and S2.45 by *Participant 8* and mixtures R1.42, S1.49, S2.49, S1.49AEA and S2.49AEA by *Participant* 6).

Commonly, freeze–thaw mass data obtained from the ASTM C666 test method (freeze–thaw resistance, not scaling resistance) are not supposed to be directly converted into an equivalent mass loss per surface area of exposed concrete [25]. Instead, the mass loss from the sample is given as a result. Testing should be continued until 300 cycles are completed or until the relative dynamic Young's

Table 14 Relative mass loss (%) (negative values mean mass gain) and relative dynamic Young's modulus (−) as obtained by *Participants 3, 6 and 8* according to ASTM C666

Participant	Material	Number of freeze–thaw cycles					
3		0	26	53	83	116	144
		Mass loss ratio (%)					
	R.45	0	0.00	0.00	0.00	0.25	2.53
	S1.50	0	0.00	0.00	0.13	7.09	
	S2.50	0	0.0	0.0	0.13	4.81	
		Reduction in Young‘s modulus (−)					
	R.45	1	0.95	0.85	0.65	0.3	0
	S1.50	1	0.95	0.82	0.43	0	
	S2.50	1	0.98	0.88	0.34	0	
8		Number of freeze–thaw cycles					
		0	30	60	90	120	150
		Mass loss ratio (%)					
	R.45	0	0.20	0.34	0.37	0.97	4.46
	S1.45	0	0.24	0.48	0.48	0.98	1.97
	S2.45	0	0.40	0.67	0.74	1.07	1.47
	A.45	0	0.27	0.20	0.30	0.84	3.31
		Reduction in dynamic Young‘s modulus, relative values (−)					
	R.45	1	0.8	0.57			
	S1.45	1	0.92	0.72	0.57		
	S2.45	1	0.96	0.87	0.74	0.61	
	A.45	1	0.82	0.6			
6		Number of freeze–thaw cycles					
		0	64	124	187	245	300
		Mass loss ratio (%)					
	R.42	0	−0.48	−0.40	−0.07	0.34	0.69
	S1.49	0	−0.06	0.18	0.33	0.72	1.24
	S2.49	0	−0.16	0.05	0.32	0.69	1.39
	S1.49AEA	0	−0.24	−0.04	0.14	0.54	0.87
	S2.49AEA	0	−0.36	−0.28	−0.01	0.27	0.54
		Reduction in Young‘s modulus (−)					
	R.42	1	0.97	0.98	0.99	0.99	0.98
	S1.49[a]	1	0.95	0.94	0.94	0.82	0.62
	S2.49[b]	1	0.94	0.96	0.95	0.88	0.55
	S1.49AEA	1	0.95	0.97	0.97	0.98	0.98
	S2.49AEA	1	0.97	0.97	0.97	0.98	1.00

[a] One out of three samples did not fulfill the criterion for rel. dynamic Young's modulus (< 0.6)

[b] Two out of three samples did not fulfill the criterion for rel. dynamic Young's modulus (< 0.6)

Table 15 Qualitative rating, as explained in text and in Table 12, of the freeze–thaw resistance in the absence of deicing salt with respect to the individual SAP-free references with W/C = 0.45 of each participant, or with W/B of 0.42 for *Participant 6* (index numbers according to Table 1; blank table fields indicate "not studied")

Part.	S1.50	S2.50	S1.45	S2.45	A.45	R.50
2	not signif.[a]	not signif.[a]				
3	–	–				
6	S1.49: - S1.49AEA: not signif.[a]	S2.49: - S2.49AEA: not signif.[a]				
8			+	+	not signif.	
9	+[a]	+[a]	+[a]	+[a]	+[a]	–[a]
11	not signif.[a]	not signif.[a]	not signif.[a]	not signif.[a]	not signif.[a]	not signif.[a]

[a] The reference mixture had high freeze–thaw resistance

modulus has dropped below 60 % of the initial value, whichever happens first. As can clearly be seen in Table 14, all specimens tested by *Participants 3* and *8* failed the test. The SAP, thus, did not improve the freeze–thaw performance but even yielded an adverse effect. All concretes tested by *Participant 6* on average fulfilled the test criterion; however, part of the individual samples prepared with SAP (S1.49 and S2.49, Table 14) did not.

Similar to Table 12, a qualitative rating for the influence of SAP on the mere freeze–thaw resistance is summarized in Table 15.

To conclude this portion, the slab test specified in [24] was not sufficiently sensitive to reveal significant differences among the mixtures, all of which passed the test. The CIF protocol [24] evidenced failure of the SAP-free concretes and of those prepared with SAP 1 at W/C of 0.50. Further, a distinct improvement of the freeze–thaw resistance was observed when SAP 1 was used with the lower W/C of 0.45 or when SAP 2 was used in all cases (the AEA mixture passed as well). Further, the "severe" protocol of ASTM C666 [25] revealed that the addition of SAP even worsened the performance when added together with additional water. When the W/C was kept constant, some improvement of the performance was observed by *Participant 8*, leading to better performance than conventional air entrainment.

5 Conclusions

In this study, the performance of two different SAP types as an admixture aimed at evaluating the enhancement of the freeze–thaw resistance in ordinary concrete was tested and analysed. The interlaboratory experimental tests were performed within the framework of the activities of the RILEM Technical Committee 225-SAP "Applications of Superabsorbent Polymers in Concrete Construction". The study was conducted by 13 different laboratories worldwide. Local materials (except for the SAP, the superplasticizer and the air entraining agent) were used. Concretes with W/C of 0.45 and 0.50 were tested, either with or without SAP, or with or without air entraining agents. Freeze–thaw resistance tests were performed according to local standards used in each participant's country, both with and without deicing salt (NaCl). In addition, compressive and splitting tensile strength and Young's modulus were measured. The latter results show in most cases a reduction in mechanical properties when SAP are added along with additional water, which the SAP absorb during mixing, similar to the effect of a corresponding increase in W/C.

Only in a few cases (one participant for tests with and without deicing salts, respectively), worsening of the freeze–thaw resistance due to addition of SAP together with additional water was observed. For all other participants, the effect on freeze–thaw resistance of the addition of SAP together with additional water varied from insignificant to pronouncedly positive, depending on the testing laboratory and the test method. The finer SAP performed consistently better in this regard. When SAP had been added without additional water, the concrete performance was considerably improved. When compared to conventional air entrainment, it was found by some participants that similar improvement can be obtained in freeze–thaw performance with the addition of SAP, but in most cases it was found that air entrainment provides considerably better performance in this regard. However, an important benefit of using SAP is that even when the SAP are added along with additional water, the negative effect on mechanical properties was still lower than in the case of conventional air entrainment. Further potential benefits compared to traditional air-entrained concrete may be due to stable void structures in the fresh concrete obtained thanks to SAP; this point requires however further studies.

Acknowledgments M. Gorges thanks the European Social Fund (ESF) and Sächsische Aufbaubank (SAB) for financially supporting her research in the framework the Saxonian Innovation Promotion Program (Grant 100117155). As a Research Assistant of the Research Foundation-Flanders (FWO-Vlaanderen), D. Snoeck extends his thanks the foundation for its financial support (1.1.D74.15 N).

Appendix

In this appendix, testing methods are listed with regard to the properties of fresh concretes (Table 16) and their mechanical properties (Table 17).

Table 16 Test methods used to assess properties of fresh concretes (index numbers according to Table 1; blank table fields indicate "not measured")

Part.	Workability	Fresh concrete density	Fresh concrete air content
1	Flow table EN 12350–5 [21]	EN 12350–6 [34]	EN 12350–7 [35]
2	Flow table EN 12350–5 [21]	EN 12350–6 [34]	EN 12350–7 [35]
3	Slump ASTM C143 [36]	ASTM C138 [37]	ASTM C138 [37]
4	Flow table EN 12350–5 [21]		EN 12350–7 [35]
5	Flow table EN 12350–5 [21]	EN 12350–6 [34]	EN 12350–7 [35]
6	Slump ASTM C143 [38]		ASTM C231 [39]
7	Slump JIS A 1101 [40]	JIS A 1116 [41]	JIS A 1128 [42]
8	Slump flow JIS A 1101 [40], slump JIS A 1150 [43]	JIS A 1116 [41]	JIS A 1128 [42]
9	Flow table EN 12350–5 [21]	EN 12350–6 [34]	EN 12350–7 [35]
10	Flow table EN 12350–5 [21]	EN 12350–6 [34]	EN 12350–7 [35]
11	Flow table EN 12350–5 [21]	EN 12350–6 [34]	EN 12350–7 [35]
12	Slump GOST 10181-2000	GOST 10181-2000	GOST 10181-2000
13	Flow table EN 12350–5 [21]		

Table 17 Test methods used to assess mechanical properties of the concretes under investigation (index numbers according to Table 1; blank table fields indicate "not measured")

Part.	Compressive strength	Splitting tensile strength	Further tests
1	EN 12390–3 [44]		
2	EN 12390–3 [44]		
3	ASTM C39 [45]	ASTM C496 [47]	Elastic Young's modulus, Poisson ratio ASTM C469 [46]
4	EN 12390–3 [44]		
5	EN 12390–3 [44]	EN 12390–6 [48]	Elastic Young's modulus DIN 1048–5 [49]
6	ASTM C39 [27]		Elastic Young's modulus ASTM C469 [50]
7	JIS A 1108 [51]	JIS A 1113 [52]	Elastic Young's modulus JIS A 1149 [53]
8	JIS A 1108 [51]	JIS A 1113 [52]	Elastic Young's modulus JIS A 1149 [53]
9	EN 12390–3 [44]	EN 12390-6 [48]	Elastic Young's modulus DIN EN 12390–13 [54]
10	EN 12390–3 [44]		
11	EN 12390–3 [44]	EN 12390–6 [48]	Elastic Young's modulus DIN 1048–5 [49]
12	GOST 10180-2012		
13	EN 12390-3 [44]		

References

1. Jensen OM, Hansen PF (2001) Water-entrained cement-based materials: I. Principles and theoretical background. Cem Concr Res 31:647–654
2. Mechtcherine V, Reinhardt H W (2012) Application of superabsorbent polymers in concrete construction. State-of-the-art report of the RILEM TC 225-SAP", Springer, Heidelberg, 2012
3. Mechtcherine V, Schröfl C (2014) RILEM Proceedings PRO 95: application of superabsorbent polymers and other chemical admixtures in concrete construction. RILEM Publications S.A.R.L, Bagneux
4. Mechtcherine V, Secrieru E, Schröfl C (2015) Effect of superabsorbent polymers (SAP) on rheological properties of fresh cement-based mortars—development of yield stress and plastic viscosity over time. Cem Concr Res 67:52–65
5. Schroefl C, Mechtcherine V, Vontobel P, Hovind J, Lehmann E (2015) Sorption kinetics of superabsorbent polymers (SAP) in fresh Portland cement-based pastes visualized and quantified by neutron radiography and correlated to the progress of cement hydration. Cem Concr Res 75:24–34
6. Jensen O M (2008) Use of superabsorbent polymers in construction materials. In: 1st international conference on microstructure related durability of cementitious composites, Nanjing (PR China), 13–15 Oct 2008. RILEM Publications S.A.R.L., Bagneux (France), pp 757–764
7. Wyrzykowski M, Lura P (2013) Controlling the coefficient of thermal expansion of cementitious materials—a new application for superabsorbent polymers. Cement Concr Compos 35:49–58
8. Lura P, Terrasi GP (2014) 0 Reduction of fire spalling in high-performance concrete by means of superabsorbent polymers and polypropylene fibers—small scale fire tests of carbon fiber reinforced plastic-prestressed self-compacting concrete. Cement Concr Compos 49:36–42
9. Snoeck D, Steuperaert S, Van Tittelboom K, Dubruel P, De Belie N (2012) Visualization of water penetration in cementitious materials with superabsorbent polymers by means of neutron radiography. Cem Concr Res 42:1113–1121
10. Snoeck D, Van Tittelboom K, Steuperaert S, Dubruel P, De Belie N (2014) Self-healing cementitious materials by the combination of microfibres and superabsorbent polymers. J Intel Mat Syst Str 2005:13–24
11. Jensen OM, Hansen PF (2002) Water-entrained cement-based materials II. Experimental observations. Cem Concr Res 32:973–978
12. Mönnig S, Lura P (2007) Superabsorbent polymers—an additive to increase freeze-thaw resistance of high strength concrete. In: Grosse CU (ed) Advances in construction materials 2007. Springer, Heidelberg, pp 351–358
13. Laustsen S, Hasholt M T, Jensen O M (2008) A new technology for air entrainment of concrete. In: 1st international conference on microstructure related durability of cementitious composites, Nanjing (PR China), 13–15 October 2008. RILEM Publications S.A.R.L., Bagneux (France), pp 1223–1230
14. Brüdern A-E, Mechtcherine V (2010) Multifunctional use of SAP in strain-hardening cement-based composites. In: International RILEM conference on use of superabsorbent polymers and other new additives in concrete, Technical University of Denmark, Lyngby, RILEM Proceedings PRO 74, pp 11–22
15. Laustsen S, Hasholt MT, Jensen OM (2015) Void structure of concrete with superabsorbent polymers and its relation to frost resistance of concrete. Mater Struct 48(1–2):357–368
16. Schröfl C, Mechtcherine V, Gorges M (2012) Relation between the molecular structure and the efficiency of superabsorbent polymers (SAP) as concrete admixtures to mitigate autogenous shrinkage. Cem Concr Res 42(6):865–873
17. Mechtcherine V, Gorges M, Schröfl C, Assmann A, Brameshuber W, Bettencourt Ribeiro A, Cusson D, Custodio J, Fonseca da Silva E, Ichimiya K, Igarashi S-I, Klemm A, Kovler K, de Mendonca Lopes AN, Lura P, Tuan Nguyen V, Reinhardt H-W, Toledo Filho R D, Weiss J, Wyrzykowski M, Ye G, Zhutovsky S (2014) Effect of internal curing by using superabsorbent polymers (SAP) on autogenous shrinkage and other properties of a high-performance fine-grained concrete: results of a RILEM round-robin test. Mater Struct 47(3):541–562
18. DIN EN 197-1:11/2011. Cement—part 1: Composition, specifications and conformity criteria for common cements. German version
19. ASTM C618-12a. Standard specification for coal fly ash and raw or calcined natural pozzolan for use in concrete
20. DIN 1045-2:08/2008. Concrete, reinforced and prestressed concrete structures—Part 2: Concrete—Specification, properties, production and conformity—Application rules for DIN EN 206-1. German version
21. EN 12350-5:08/2009. Testing fresh concrete—Part 5: Flow table test
22. Jones W A, Weiss W J (2014) Freeze thaw durability of internally cured concrete made using superabsorbent polymers. 4th International conference on the durability of concrete structures, Purdue University, West Lafayette (IN/USA), 2014, pp 3–11
23. SIA 262/1:2003. Eingetragene Norm der Schweizerischen Normen-Vereinigung: Betonbau—Ergänzende Festlegungen, Anhang C: Frost-Tausalzwiderstand. German version
24. CEN/TS 12390-9:09/2006. Testing hardened concrete—Part 9: Freeze-thaw resistance—Scaling
25. ASTM C666-03 (2008) Standard test method for resistance of concrete to rapid freezing and thawing
26. Setzer MJ, Fagerlund G, Janssen DJ (1996) CDF Test—test method for the freeze-thaw resistance of concrete—tests with sodium chloride solution (CDF). Mater Struct 29:523–528
27. ASTM C672-12. Standard test method for scaling resistance of concrete surfaces exposed to deicing chemicals
28. JIS A 1148:2010. Method of test for resistance of concrete to freezing and thawing
29. GOST 10060.0-95. Concretes: Methods for the determination of frost-resistance. General requirements
30. GOST 10060.1-95. Concretes: Basic method for the determination of frost-resistance
31. GOST 10060.2-95. Concretes: Rapid method for the determination of frost-resistance by repeated alternated freezing and thawing

32. GOST 26134-84. Concrete: Ultrasonic method for determining cold resistance
33. Justs J, Wyrzykowski M, Bajare D, Lura P (2015) Internal curing by superabsorbent polymers in ultra-high performance concrete. Cem Concr Res 76:82–90
34. EN 12350-6:03/2011. Testing fresh concrete—Part 6: Density
35. EN 12350-7:08/2009. Testing fresh concrete—Part 7: Air content—Pressure methods
36. ASTM C143-12. Standard test method for slump of hydraulic concrete
37. ASTM C138-14. Standard test method for density (unit weight), yield, and air content (gravimetric) of concrete
38. ASTM C143-04. Standard test method for slump of hydraulic concrete
39. ASTM C231-10. Standard test method for air content of freshly mixed concrete by the pressure method
40. JIS A 1101:2005. Method of test for slump of concrete
41. JIS A 1116:2005. Method of test for fresh concrete unit volume weight and of air mass (mass method)
42. JIS A 1128:2005. Method of test for air content of fresh concrete by pressure method
43. JIS A 1150:2007. Method of test for slump flow of concrete
44. EN 12390-3:07/2009. Testing hardened concrete—Part 3: Compressive strength of test specimens
45. ASTM C39-14a. Standard test method for compressive strength of cylindrical specimens
46. ASTM C469-14. Standard test method for static modulus of elasticity and Poisson's ratio of concrete in compression
47. ASTM C496-11. Standard test method for splitting tensile strength of cylindrical concrete specimens
48. EN 12390-6:09/2010. Testing hardened concrete—Part 6: Tensile splitting strength of test specimens
49. DIN 1048-5:06/1991. Testing concrete; testing of hardened concrete (specimens prepared in mould). German version
50. ASTM C469-10. Standard test method for static modulus of elasticity and Poisson's ratio of concrete in compression
51. JIS A 1108:2006. Method of test for compressive strength of concrete
52. JIS A 1113:2006. Method of test for splitting tensile strength of concrete
53. JIS A 1149:2010. Method of test for static modulus of elasticity of concrete
54. DIN EN 12390-13:06/2014. Testing hardened concrete—Part 13: Determination of secant modulus of elasticity in compression; German version EN 12390-13:2013