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RILEM TC REPORT

Verification of the presence of superabsorbent polymers (SAP) in fresh concrete: results of an interlaboratory study of RILEM TC 260-RSC

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Abstract New methods are proposed for the verification of the presence of superabsorbent polymers (SAP) in freshly mixed concrete and estimation of SAP quantity. The methods are in general based on flushing concrete with excess water. They allow separating the light, water-sorbed hydrogel particles from the mineral components in the fresh concrete and making these particles available for further tests. Two types of tests are proposed: Test 1 serves for a visual verification of the presence of SAP (qualitative test), while Test 2 enables quantifying the mass of the collected SAP as a proxy of their concentration in concrete (quantitative test). Different procedures are proposed for these two test methods and their performance is evaluated. The testing procedures were scrutinized in an interlaboratory study carried out by 14 participants from 12 countries. All participating groups detected the presence of SAP in the mix using the qualitative procedures (Test 1). Based on this outcome, we suggest that this method should be applied in the field. In contrast, while most participants obtained reasonably reliable results with the quantification procedure of Test 2, some participants reported large errors. Therefore, the quantification method needs to be further refined, starting from the experience gained in this interlaboratory study.

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1 Introduction

Superabsorbent polymers (SAP) are a fairly novel admixture in concrete and other cement-based materials with various potential applications, e.g. internal curing, improving freeze–thaw resistance, aiding self-sealing and self-healing, control of rheological properties, and others [1–3]. SAP rapidly absorb large amounts of mixing water from the freshly prepared concrete, typically in excess of 10–40 times their own dry mass. While absorbing part of the mixing water, they swell and create macro-sized water reservoirs. The absorbed liquid is later released into concrete in response to osmosis or capillary pressure, the latter driven by the process of self-desiccation or drying [2, 4].

While in most applications SAP are used to improve the performance of concrete during hardening and in the hardened state [5–7], they can also affect the behavior of freshly mixed concrete in various ways. While absorbing part of the water, they in general reduce the flowability [8, 9]. On the other hand, they may stabilize the concrete mix due to increased viscosity. The behavior of concrete with SAP strongly depends not only on the absorption capacity of mixing water (or, more precisely, pore fluid), but also on the absorption and desorption kinetics of the SAP [4, 10–12]. For most applications, it is desired that SAP absorb the mixing water rapidly and retain it for a desired period, e.g. until the setting time of concrete. This kinetics will also strongly interact with the specific mixing and casting procedures.

With the recent introduction of SAP into the industrial practice of cement-based materials, appropriate testing and characterization methods are sought that could aid practitioners in verifying the presence and performance of this admixture. Some procedures have been developed for quantifying the amount and even the size distribution of SAP-originated pores, similar to those used for assessing the air void system with air-entraining agents, e.g. X-ray tomography or microscopy [2, 13–15]. However, such methods can only be applied to hardened concrete and in most cases require elaborate specimen preparation (cutting, polishing) and evaluations. The presence of SAP and their overall effect can also be assessed in tests that aim at a specific performance of the concrete. A prominent example are measurements of autogenous shrinkage, in which the reduction of shrinkage with specific types of SAP becomes evident [16].

At the same time, a simpler test method is sought to detect the presence of SAP at the construction site in as-delivered, fresh concrete with the overall goal of quality control in construction. The desired test method would be similar in its simplicity and accessibility to the fresh-state tests used for example to assess the action of water-reducing admixtures (spread, slump, etc.) or air-entrainers (air

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content in fresh concrete). Such tests deliver immediate answers (in a matter of minutes) and rely on rather simple, repeatable procedures and affordable devices. In the case of the proposed test, the verification of the presence of SAP (and possibly the quantification of their amount in the concrete mix) would serve as a step in the verification of the mixture composition. By confirming the presence of SAP, the expected performance of concrete may be anticipated, e.g. with regard to internal curing or other applications. This can be considered analogous to the aforementioned air-entrainment test. In that test, the content of air is determined in the bulk of concrete using a pressure porosimeter. Although the mere fact of a high air content does not automatically translate to better frost resistance of concrete (because the air void size distribution is not measured on site), it allows to verify that indeed a proper amount of air was entrained. This means that the concrete with entrained air most likely also has a sufficient amount of small air bubbles spaced at sufficiently short distances to exhibit good frost resistance. To come back to the intended case, concrete in which SAP have been detected (and possibly their amount measured) can be expected to benefit from internal curing and to exhibit lower autogenous shrinkage in an analogous way in which concrete with extra air content is expected to exhibit better frost resistance.

A major technical challenge in assessing the amount of SAP is that usually very small quantities of SAP are used in concrete, typically 0.1–0.3% by mass of cement [17]. After absorbing part of the mixing water they constitute only a couple percent of the concrete volume. At such small quantity, and owing to the usually small size of the swollen SAP particles (usually fraction of a millimeter) and their translucent appearance, the SAP are very hard to be spotted visually in the bulk of fresh concrete, let alone be quantified. The TC 260-RSC therefore developed a method in which the SAP particles are first separated from the concrete mix by washing and sieving so that the SAP particles can be visually detected. This constitutes the first, qualitative part of the test procedure proposed in this paper (Test 1). In the next step, the amount of the collected SAP is quantified with relatively simple thermogravimetric test procedures (Test 2). This second type of test requires longer times (owing mainly to the time necessary for the drying of the collected material) and more resources (e.g. ovens, scales) and hence falls outside the domain of typical field tests. However, it constitutes a rather simple procedure compared to more elaborate tests carried out on hardened concrete. It may thus be used in certain cases where a relatively fast outcome is necessary or limited resources are available at a testing laboratory. The aim of this study is to aid practitioners in applying the test methods in practice and assessing their reliability.

To test the repeatability of the methods and identify potential issues, an interlaboratory study was carried out as part of the activities of RILEM TC 260-RSC. Fourteen laboratories from twelve countries participated in the tests. All tests were carried out using two different SAP products mixed in concrete with prescribed volume proportions. Additionally, blind tests were carried out at one laboratory where the operators tested concrete mixes with and without SAP without a prior knowledge of the concrete mix compositions.

2 Materials and methods

2.1 Materials

Concrete mixes with two different types of SAP were prepared. The SAP were marked SAP-L ("L" for "Large") and SAP-S ("S" for "Small"). These two types had been used in the previous studies, e.g. SAP-L was used in [9, 11], and in [5] (as "SAP1") and [10] (as "SAP2"), SAP-S, from another producer, had been studied as SAP 2 in [5]. Choosing two suppliers should account for the variability in SAP products and potential effects of proprietary synthesis and post-polymerization-processing details. The sorption behavior with respect to extracted cement pore solution of both materials is classified as “retentive” in terms of previous studies [10, 11]. Experience had previously shown that such type of SAP appear to be used more as compared to “self-releasing” ones, although the latter may have specific positive effects in cement-based materials [2, 18]. Both products are quickly absorbing and have rather similar individual one-hour sorptivities when immersed in extracted cement pore solution (SAP 1: 37 g/g, and SAP 2: 35 g/g) [5]. Both polymers are covalently cross-linked poly(acryl amide-co-acrylate) by their chemical

nature and both stem from bulk polymerization followed by grinding and sieving. They thus feature uneven, non-spherical particle shapes. While SAP-L had typical sizes of few hundred microns (in a minor percentage up to 900 µm), SAP-S were most typically 100 µm and a maximum 200 µm in size. Hence, it should be expected that their dimensions in the swollen state in the fresh concrete differ accordingly, based on their very similar individual sorption characteristics [5].

Table 1 presents the concrete mix composition that was recommended to all participants of the study. The recommended mix had a volume of about 2 L. For each of the tests or repetitions, a separate mixture batch should be prepared. Each group used locally available materials, except for the SAP products which were supplied by the test organizers. The specific details of the mixes used by the different participants are presented in Table 2.

2.2 Methods to verify the presence of SAP

Test 1: Qualitative Two methods are proposed (A and B), differing in how the SAP are separated from the fresh concrete. In Method A, concrete is washed and filtered through sieves so that the SAP, if any, become easier to be spotted as remnants on the finer sieve. In Method B, excess water is added to the concrete in a container and mixed. The slurry is then collected from the top. Hereby, the slurry should contain primarily the SAP, if any, as the heavier aggregates and cement settle quickly on the bottom.

2.2.1 *Test 1: Qualitative, Method A*

Step 1 A minimum of 200 g of fresh mortar or concrete is placed on a sieve with 2 mm opening size over a larger bowl. Next, the concrete is flushed with clean water to wash the paste with the SAP through the mesh while retaining larger aggregates on the sieve. Swollen SAP particles used in the field should pass the 2 mm sieve, but, if necessary, the mesh size can be adjusted in accordance to the size of the SAP.

Step 2 The slurry obtained is poured through a 0.063 mm sieve to remove the water.

Step 3 The material retained on the 0.063 mm sieve should be cement and sand and, if present, the SAP. The SAP can be recognized by their translucent appearance, and when touched, they should be soft and slippery, unlike the hard aggregate grains, see Fig. 1.

Test outcome: Positive or negative.

2.2.2 *Test 1: Qualitative, Method B*

Step 1 For the assessment of SAP in the fresh concrete, about 2 L of the fresh concrete mix is placed in a large bucket of capability about 15 L, and the mass of the concrete (around 5 kg) is determined with an accuracy of 1 g. Afterwards, about 10 L of water are added followed by manual mixing, causing a whirlpool effect. This will cause the mineral solids (cement, aggregates) to settle, while the SAP will stay on the top of the suspension. SAP particles with density close to the density of water are flushed out of the test concrete mix and sucked into the whirlpool. Next, the slurry is left to settle for about 1 min. It was found that a considerable amount of SAP settles during this time, so it is important to collect all water possible in the following step quickly.

Table 1 Recommended concrete mix composition (exceptions are reported)

Material	Mass (g)
Cement (preferably CEM I 42.5 N in terms of EN 197 or equivalently composed according to national standards)	700
Water ($w/c = 0.40$)	280
SAP (0.15% by mass of cement)	1.05
Aggregates	3850
High-range water-reducing admixture (HRWRA)	Not specified, according to a local practice (recommendation was not to use any admixture)

Table 2 Details of the concrete mixes used in the tests by each individual participant

No	Group	Cement	Max. aggregate size (mm)	Mixture design	HRWRA [by mass of cement]
1	Empa	CEM I 42.5 N	4	As prescribed	None
2	TU Dresden	CEM I 42.5 N	4	As prescribed	None
3	Ghent University	CEM I 42.5 N	16	As prescribed	None
4	U Antwerp	CEM I 52.5 R	14	As prescribed	None
5	Seoul National University	ASTM type I	10	As prescribed	None
6	BASF	CEM I 42.5 N	2	As prescribed	None
7	Kanazawa University	Ordinary Portland Cement	5	Lower aggregates mass, 3 times the mass of cement	None
8	Glasgow Caledonian University	CEM I 52.5 N	20	As prescribed	None
9	Federal University of Minas Gerais	CPV-ARI (high early strength Portland cement)	4.8	As prescribed	None
10	University of Pretoria	CEM I 52.5 R	13	Lower aggregates mass, 3.3 times the mass of cement	1%
11	LNEC	CEM I 42.5 R	12	As prescribed	2%
12	Purdue University	ASTM type I	4.25	As prescribed	0.8%
13	Scientific Research Institute for Concrete and Reinforced Concrete	CEM I 52.5 N	5	As prescribed	0.6%
14	Federal University of Uberlândia	CPV-ARI (high early strength Portland cement)	4.8	As prescribed	1.5%

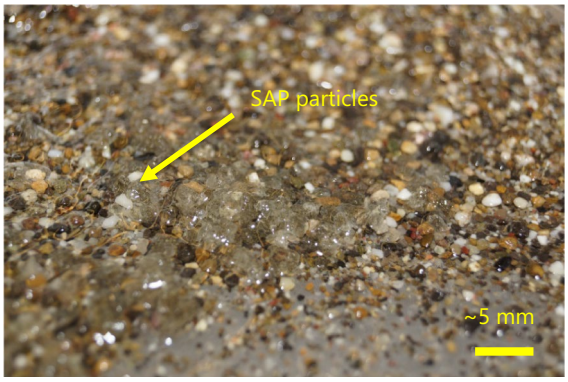

Fig. 1 SAP particles and sand left on the sieve after Step 3 of Method A. Based on such outcome, the presence of SAP in fresh concrete is confirmed qualitatively

Step 2 The liquid part of the concrete mix is filtered through a standard metal sieve with mesh size 0.09 or 0.125 mm. The filtering procedure should take about 1 min. A typical result can be seen in Fig. 2. To ensure that all SAP are washed out of the concrete, which will be important for the quantification in the Test 2 that follows this test, steps 1 and 2 (i.e., addition of water and collection of the liquid) can be repeated one or two further times with the material remaining in the large bucket.

Step 3 The slurry separated in Step 2 is evaluated for the presence of SAP. This can be done visually and by touching the collected mass (the difference between the mineral fines and SAP can be assessed based on the hardness of the material to the touch).

Test outcome: Positive or negative.

2.2.3 Test 2: Quantitative

This test is a continuation of Test 1, Method B (Sect. 2.2.2), hence the steps described here follow those described in the previous section.

Fig. 2 **a** Separation of concrete by adding excess water according to Test 1, Method B; **b** SAP collected on the sieve after the separation procedure

Step 4 The deposit on the sieve after the filtration of the slurry described in Step 2 of Test 1, Method B (see Fig. 2b) is collected. In case other polymeric additives (e.g. fibers) are used, they should be removed in a separate procedure (e.g. by sieving in the case of long fibers). If this is impossible and the SAP cannot be separated from such other polymeric components, the quantification procedure is not applicable.

Step 5 Oven drying of the collected slurry is carried out at 60 °C until constant mass. Usually, this step requires at least 1 day, but may take longer, depending on the oven, ventilation, etc. The participants of the interlaboratory study reported oven-drying times from 1 d until 7 d for this step (median drying duration was 2 d). The remaining dry material should consist of dry SAP, cement and other fine mineral components of concrete. The mass of the material should be recorded with precision of ± 0.01 g.

Step 6 In this step, the amount of dry SAP contained in the dried out material from the previous step is determined. This can be done conveniently by burning the SAP. It is assumed in a first approximation that other components (primarily aggregates or cement) would have negligible loss of mass on ignition.

Two different methods are proposed for this step:

- burning with a gas torch, see Fig. 3. During this step it is important that the material is not scattered by the gas of the torch during burning. The burning should last at least 2 min (the initial tests showed that after this time the mass remained constant, i.e. no residual SAP remained to be burned). Times between 2 and 15 min were reported by the study participants.
- burning in an oven at 500 °C for 3 h, followed by free cooling down.

Fig. 3 Burning out of SAP particles with a gas torch

Step 7 The resulting mass of the burned sample is recorded. The difference in mass between dry and burned sample is assumed to be the mass of SAP particles in the sample.

Test outcome: The mass of SAP in the sample (according to Step 7 above) per initial mass of the sample. This ratio is next compared to a mass of SAP per mass of concrete according to the mix composition.

3 Results and discussion

3.1 Test 1: Qualitative test

Twelve out of the fourteen participants applied both methods for SAP detection, while the remaining two participants applied only method B. All participants (14 groups) reported that the SAP could be detected using the proposed qualitative methods (Method A and Method B). Yet, certain issues were reported. Three participants reported that it is considerably harder to detect the smaller SAP particles (SAP-S) on the sieves. The particles were caught on the sieves trapped in a slurry with cement and smaller sand particles. Detecting the SAP was still possible in that case, but required touching the slurry. This relies on the fact that the swollen polymers are, unlike cement or other mineral components of concrete, soft and slippery to the touch. Two other participants reported that the SAP tended to stick to the sand particles and hence large quantities of water were necessary for washing the mix on the sieve to separate one from the other and make the SAP clearly visible. The fact that SAP usually swell much more while exposed to clean water compared to the mixing water in fresh concrete [11, 19] aids in their identification.

The time necessary for the separation of the SAP and their detection was relatively short—from 1 to 20 min (median 5 min) for Method A and from 1 to 60 min (median 10 min) for Method B. Five out of twelve participants considered both methods are easy to be applied in field conditions, with no preference towards any of the methods. Another five out of twelve participants reported that Method A is easier, while the remaining two participants reported that Method B was easier. The two participants that only employed Method B considered it easy to be applied in field conditions.

An additional set of blind tests was carried out at one laboratory (Participant 1) to check for possible confirmation bias. In the main interlaboratory tests, the participants were aware of the presence of SAP and hence the positive outcome of their tests may have been affected by this knowledge. In other words, one may assume that when it is certain that a given concrete contains SAP, the operator will search until the SAP are found. The blind test concerned Test 1, Method B. To this end, a set of five specimens was prepared: two control specimens (as in Table 1, but without the SAP), two specimens with SAP according to Table 1 (one with SAP-L and one with SAP-S) and one specimen with SAP-S (i.e. finer SAP), but containing half the amount of SAP. These sets were given independently to two operators with specimens labelled in an anonymyzed way and without any information disclosed on the composition of the sets. One of the operators had prior experience in the tests (he had taken part in the main interlaboratory test program), while the other operator carried out the test for the first time. Each test took about 5 min per specimen. The operators then reported the outcome of the tests in a binary form (SAP present or not). The outcome was as follows: both the presence and the lack of any SAP was correctly determined in each specimen by both operators. This shows that the qualitative test is a robust way of confirming the presence (or lack) of SAP also at a very low concentration (the reduced amount of SAP-S in one of the specimens was only 0.075% by mass of cement).

3.2 Test 2: Quantitative test

Some participants who applied the quantitative assessment reported problems with collecting the material after washing concrete with water. It was reported that some SAP particles may have been trapped in the heavier, solid material depositing on the bottom of the bucket. Because of that, the washing was repeated to free the SAP from the mineral solids. To obviate this problem, three participants suggested that the larger aggregates could be first sieved out before washing the material.

Two different methods for determining the mass of the collected SAP were proposed: with a gas torch and by burning in the oven at 500 °C. The goal of both of these methods was to burn the collected SAP out of the slurry collected on the sieve. Eleven out of fourteen groups carried out both the gas torch method and the oven drying method, two only the oven drying method and one only the gas torch method.

Some groups reported that the gas torch tends to blow and scatter the fine material. If the blown fine material was fine cement/sand particles, this may have led to an overestimation of the mass of burned material (and thus of the SAP mass). It was also reported that the minimum time necessary for burning the material could not be easily determined.

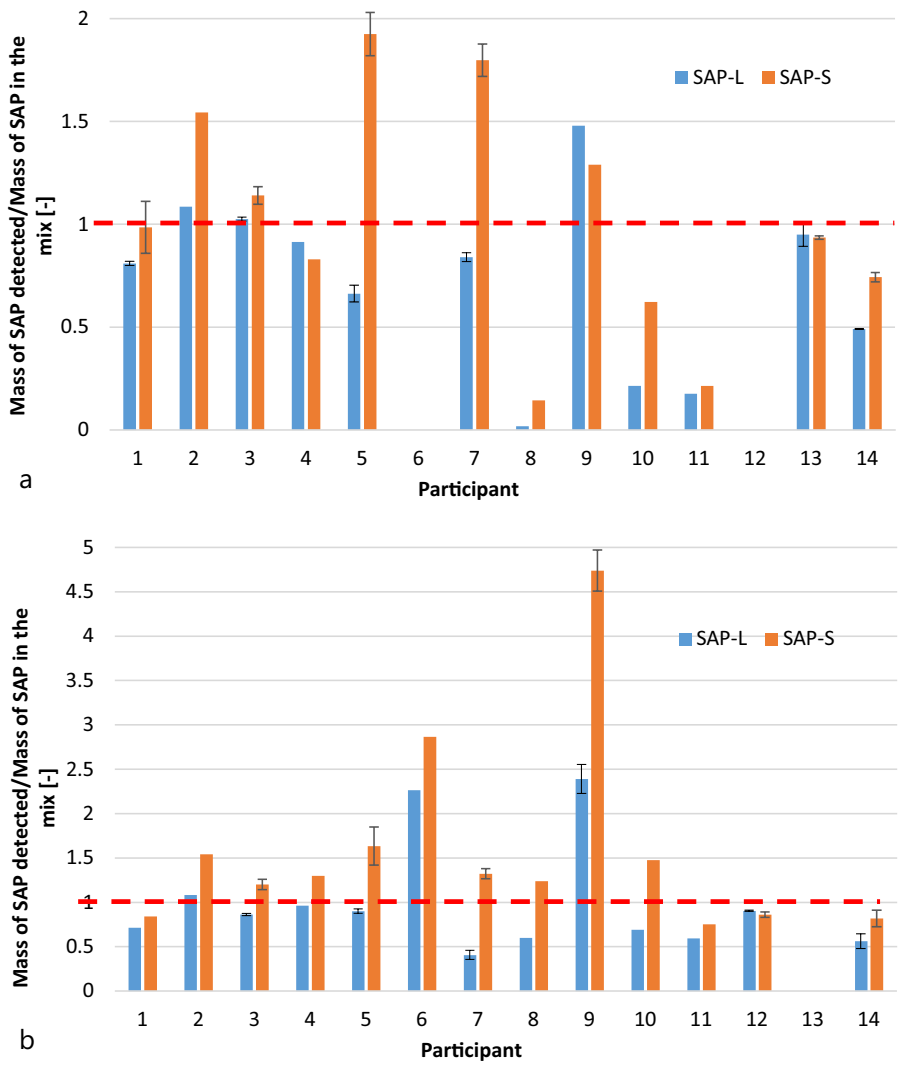

Fig. 4 Ratios of SAP detected after collecting the liquid from above the watered concrete followed by drying the collected residual first at 60 °C followed by burning with **a** a gas torch, **b** in the oven at 500 °C. The mass of SAP was assumed as the mass loss during the final burning step. The theoretical (reference) ratio is equal to 1 (this means that the mass of detected SAP is equal to the mass used in the mix design). The average values per each laboratory are presented; error bars show range of values from duplicate samples (where available)

Table 3 Statistics for the ratio of SAP detected compared to the total mass of SAP used in the concrete mix design

Statistic	Gas torch		Oven 500 °C	
	SAP-L	SAP-S	SAP-L	SAP-S
Average (of all participant averages)	0.72	1.01	1.00	1.58
Median (of all participant averages)	0.83	0.96	0.86	1.30
Repeatability (within participant pooled standard deviation)	0.04	0.11	0.11	0.20
Reproducibility (of all participants)	0.43	0.57	0.63	1.11
Error % (difference between the determined amount and the used amount of SAP referenced to the used amount of SAP) (average ± standard deviation of all participant)	38 ± 34	44 ± 33	45 ± 44	71 ± 107

Each statistic comes from 18 measurements in total as obtained from 12 participants (6 participants carried out single measurements for each SAP, while 6 participants carried out duplicate measurements)

The results of the quantification are presented in Fig. 4 and the corresponding statistics in Table 3. While the average and medians from all participants are relatively close to the reference (true) level of 1, some participants found significantly different ratios. For the torch burning method, three participants reported significantly lower content of SAP (ratio of 0.2 or less), see Participants 8, 11 and 12 in Fig. 4. Less severe underestimations were found for the oven burning method. On the other hand, the oven burning led to more severe overestimations (see Participants 6 and 9). In the case of gas torch method, the overestimations were less extreme, but still occurred for some participants (see Participants 5 and 7). In summary, an overestimation was reported for at least one SAP type by four participants for both the gas torch and oven burning methods.

One systematic reason for the underestimation is likely that not all SAP could be captured in the extra water added to the mix (part of SAP may have been entrapped in the solid sediments). Furthermore, the SAP may have stuck to the sieve during their separation from the water. Finally, the underestimation—evident especially with the torch burning method—may have resulted from insufficient burning time with the torch and presence of unburnt remnants of the SAP.

Regarding the overestimations, one possible explanation could be insufficient drying of the collected material at 60 °C prior to burning (either with a torch or in the oven at 500 °C). The extra water retained prior to burning would then contribute to the mass loss during burning and would be wrongly interpreted as the mass of burnt SAP. Another likely cause of overestimation in the case of gas torch was due to blowing and losing of finer mineral particles during the burning process (also here, the mass of such scattered particles would be wrongly interpreted as the mass of burnt SAP). Finally, loss on ignition (LOI) of the solids could contribute to the apparent loss of mass attributed to SAP. According to our measurements, the latter effect should be of negligible importance considering that the collected slurry (see Fig. 2b) is made primarily of SAP. According to our tests, the mineral residues constitute about 2% of mass of the wet slurry and about 50% of mass of the material after drying at 60 °C. Hence, the LOI of the minerals during burning should be only a fraction of the loss from the burnt SAP.

The scatter of the results within a single particiapnt is relatively low (see the repeatability between 0.04 and 0.20 in Table 3). This shows that the method of SAP collection from the washed concrete along with subsequent burning procedures is repeatable within a given participant/laboratory. On the other hand, the differences between the participants, represented by the reproducibility statistic are higher (from 0.43 to 1.11), in particular for the oven burning method, Table 3. Finally, the error of the estimation relative to the reference value of 1 can be estimated. Again, this error increases for the oven method. Also, the oven method leads to higher ratios (for SAP-S artificially higher than 1). This suggests that part of the mass loss was most likely due to loss of water from the slurry that was not totally removed in the initial step of drying at 60 °C for 24 h or possibly also due to loss on ignition from the mineral solids.

The statistics are evaluated also for the case where the outliers are excluded, see Table 4. These were identified as significant overestimations with

Table 4 Statistics—as in Table 3 but after excluding outliers (4 participants for the gas torch method and 2 participants for the oven method)

Statistic	Gas torch		Oven 500 °C	
	SAP-L	SAP-S	SAP-L	SAP-S
Average (of all participant averages)	0.87	1.01	0.75	1.18
Median (of all participant averages)	0.93	0.96	0.71	1.24
Repeatability (participant pooled standard deviation)	0.04	0.09	0.06	0.16
Reproducibility (of all participants)	0.39	0.32	0.21	0.35
Error % (difference between the determined amount and the used amount of SAP referenced to the used amount of SAP) (average ± standard deviation of all participants)	28 ± 28	23 ± 17	28 ± 18	29 ± 16

Participants 5 and 7 for gas torch and Participants 6 and 9 for the oven, and as significant underestimations with Participants 8 and 11 for the gas torch. The averages are not significantly affected and the median values even less so. However, the reproducibility is significantly improved and the error becomes approximately uniform at a level of 20–30% of the reference value across different SAP types and different methods.

4 Summary and conclusions

This paper describes new experimental methods for verifying the presence of superabsorbent polymers (SAP) in concrete or mortar (Test 1—qualitative) and assessing the quantity of SAP (Test 2—quantitative). Test 1 is applied on fresh concrete mix (after mixing or delivery) and can be performed at the construction site in addition to other commonly used testing methods for characterizing density, air content or workability of a fresh concrete. It is based on separating the SAP from the fresh mixture followed by their visual detection. Two different ways of separating the SAP in the fresh mixture are proposed, both based on washing the fresh concrete and collecting/retaining the swollen SAP particles on the sieves.

Test 2 is also based on collecting the material from fresh concrete (on site), but then requires more elaborate procedures in the laboratory for the quantification of SAP content. These procedures require up to several days, because drying of the specimens is necessary prior to the SAP quantification. In this test, the mass of the collected SAP is measured and treated as a proxy for the content of SAP in a concrete mix. The collected material is burned and we assume that the mass of burnt material originates solely from the burnt SAP particles.

In addition to laying out the principles of the proposed methods, this paper reports on an interlaboratory study carried out within the framework of activities of RILEM TC 260-RSC. 14 laboratories took part in this effort. All participating groups could detect the presence of the SAP according to both methods proposed for Test 1. Despite some technical issues reported, it can be concluded that both methods are capable of detecting the presence of SAP and that both are easy to implement in field conditions. Method B of Test 1 was additionally tested in a blind test, where specimens containing different types of SAP at different concentrations were tested in parallel to specimens containing no SAP (control specimens). Also in this case the method proved its robustness, while any confirmation bias could be avoided. Method B of Test 1 also proved in the blind test to be sensitive for a relatively low SAP concentration, in fact as low as 0.075% of SAP by mass of cement. This is a very good result, since concentrations to be commonly expected in concretes in practice of construction comprise between 0.1 and 0.3%) [2]. Hence the method should be sensitive enough for practical use.

The quantitative Test 2 was also tested by 12 groups. The ratio between the detected and the actual amount of SAP used in the concrete mix determined by most groups was fairly close to the expected reference value of 1 (the median being 0.83–1.30, depending on the method and SAP type). However, large deviations were reported by some participants (with ratios as low as 0.2 or as high as 4.5, i.e., 5 times lower or 5 times higher than the actual SAP concentration). It should be stressed that the method was applied for the first time by all participants, hence the lack of training or experience might have caused such results.

Among the factors that affect the quantification, the uncertainty in determining the SAP amount trapped in a slurry with fine mineral particles was found to be the most detrimental. In the proposed method, excess water was used to separate SAP particles from concrete. The water containing SAP particles is filtered and the captured material is supposed to be composed mainly of the SAP. However, it is difficult to separate fine mineral constituents of concrete captured by the filter from SAP. A torch or an oven are used to burn out the SAP. This procedure apparently leads to high errors in the quantification. In addition, burning process makes the method not applicable in cases when other non-soluble polymer additives such as film-forming latex particles are used in concrete. In any case, the results from most participants have shown that the SAP amount can be detected with errors below 30% on average. Such level could be reached after removing the results identified as outliers from either 2 participants or 4 participants, depending on the method. Consequently, Test 2 may be rather unreliable if an arbitrary group of experimenters are to

quantify the SAP content inside an as-delivered concrete batch.

In summary, it can be concluded that the qualitative procedures of Test 1 seem ready for field applications. Contrarily, the more elaborate quantification of SAP still requires further development, either by improving Test 2 or by exploring alternative procedures.

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