Materials and Structures (2019) 52:75
https://doi.org/10.1617/s11527-019-1375-4

RILEM TECHNICAL COMMITTEE

Recommendations of RILEM TC 260-RSC for using superabsorbent polymers (SAP) for improving freeze–thaw resistance of cement-based materials

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Received: 22 March 2019 / Accepted: 20 June 2019 / Published online: 1 July 2019

Abstract This recommendation is focused on application of superabsorbent polymers (SAP) for the improvement of the resistance of cement-based materials to freeze–thaw attack with or without deicing salts. A simple approach to the determination of the amount and properties of SAP as well as methods to verify SAP effectiveness for frost resistance protection are presented.

Keywords Deicing salt · Freeze–thaw · Frost resistance · Superabsorbent polymers

1 General provisions

1.1 Scope of the recommendation

This recommendation focuses on application of superabsorbent polymers (SAP) as an admixture in cement-based materials to improve their resistance against freeze–thaw attack.

1.2 Definition of terms

Superabsorbent polymers (SAP) are cross-linked polyelectrolytes that can swell upon contact with water or aqueous solutions, hence resulting in the formation of a hydrogel.

This recommendation has been prepared by members of Work Group 4 "Effect of SAP on freeze–thaw resistance"—Viktor Mechtcherine, Christof Schröfl, Michaela Reichardt, Agnieszka J. Klemm and Kamal H. Khayat—acting within the RILEM TC 260-RSC "Recommendations for use of superabsorbent polymers in concrete construction" and has been reviewed and approved by all members of the TC 260-RSC.

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Cement-based materials include mortar, concrete, render, and grout—irrespective of any potential reinforcement.

Durability means the ability of a material to maintain its original properties during service life to such an extent that the serviceability is guaranteed.

Freeze–thaw resistance describes the resistance against alternating cycles of freezing and thawing in the presence of water as the test liquid. Test procedures, including the quality of the water and evaluation criteria are specified in national standards. Characteristics of the water include demineralised water [1] or clean water [2–5]. The freeze–thaw test prescriptions may not individually define this issue, but provide cross-references to further standards or regulations (e.g. [5]).

Freeze–thaw and deicing salt scaling resistance describes the resistance against alternating freezing and thawing in the presence of de-icing salt solution as a test liquid (salt type and content according to corresponding national standards, e.g. 3% NaCl [1, 4, 6, 7] or 4% $CaCl_2$ [8]). Test procedures and evaluation criteria are specified in the respective standards.

Scaling is the loss of material (normally cement mortar) at the surface of concrete due to freeze–thaw damage alone or freeze–thaw in the presence of de-icing salt.

Internal damage is the deterioration of the internal structure of concrete (even without visible external damage) which leads to a change in concrete properties (e.g., internal microcracking and a reduction of the dynamic modulus of elasticity).

Air void or air pore is an enclosed cavity, in which air or another gas was trapped before solidification of cement paste. Note that only fine air bubbles (typically < 1 mm) are beneficial with respect to freeze–thaw resistance. This does not apply to pores of submicroscopic dimensions, such as capillary porosity of hydrated cement paste.

Air-entraining agent is an admixture that forms and stabilises tiny air bubbles in the fresh concrete mixture.

Air content is the volume fracture of air voids of the total volume of concrete.

Spacing factor is an index describing the average maximum distance of any point in cement paste from the periphery of an air void.

1.3 Background and benefits of using SAP to improve the freeze–thaw resistance of cement-based materials

SAP is a new class of concrete admixture with multiple functionalities [9]. Applications of SAP include modifying the rheology [10], mitigation of plastic shrinkage [11, 12] and autogenous shrinkage [13, 14] as well as triggering self-sealing and self-healing of cement-based construction materials [15]. The major positive effect of SAP in concrete and other cement-based materials is creating size- and shape-designed pore systems within these materials to improve their durability, especially in terms of freeze–thaw resistance [16]. Such application can especially be relevant as an alternative to traditional air-entraining agents (AEAs) where long concrete placement hauling and placement durations, highly flowable mixtures, or high ambient temperatures can lead to the loss of a portion of the entrained air. Shotcrete with well controlled and stable SAP voids can also be produced, while use of conventional AEAs leads to the loss of a great, hardly estimable portion of air bubble upon shooting [9]. Pore systems built up as a result of SAP addition are robust and remain stable during the initial stages of stiffening, setting and hardening—regardless of the consistency of the fresh mixture, addition of further chemical admixtures, such as superplasticizers, or the method of placement and consolidation. These facts represent pronounced improvements by SAP compared to conventional air-entrainment.

Over the last years several studies, e.g. [16–19] showed the effectiveness of an SAP-entrained air-void system to increase freeze–thaw resistance of concrete. This recommendation aims to draw together the facts for a successful selection of SAP material, its proper application in concrete mixture design and proof of its performance.

2 Proportioning concrete mixture containing SAP

2.1 General considerations, working mechanisms and comparison to AEA

Concrete structures exposed to freeze–thaw cycles in the presence or absence of de-icing salts can suffer from scaling of surface material or internal damage due to formation of microcracks, hence leading to material cracking and deterioration and reduced service life of the structure. To enhance freeze–thaw resistance and ensure adequate durability of exposed concrete, a stable, finely distributed micro-pore system should be secured by using AEA or SAP.

Traditional AEAs are mainly surfactants made from natural resin or synthetic ionic or non-ionic surfactants. When an AEA is added to the fresh concrete mixture, air bubbles that are entrapped during mixing and placement can be smaller than commonly entrapped ones. Such small air bubbles can remain in stable suspension given their small size and electrostatic dispersed state until the hardening of the concrete. The working mechanisms of the induced air bubbles regarding the increase of freeze–thaw resistance are well-known, see e.g. [20, 21]. The effectiveness of AEA is commonly linked to the spacing factor, see e.g. [22] which is used as characteristic value of micro air void content and their distribution. In some guidelines, the total content of air voids having equivalent diameters ranging between 10 and 300 μm is used to describe the air-void system [22] as well as the specific surface area of the air bubbles [23]. These values are usually determined using polished cross-sections of saw-cut concrete samples.

A potential working mechanism of SAP entrainment may be similar to that of the entrained air bubbles, as explained in [18]. During hardening and/or desiccation process, SAP particles release absorbed water and leave behind cavities with sizes of the swollen SAP particles; these cavities act like air voids. Therefore, the principles of an effective entrained air pore system valid for AEA [24] (regarding spacing factor and other characteristic parameters of micro air pore system) hold true, in principle, when using SAP. However, the existing beneficial effect of SAP is still subject to fundamental scientific studies. Numerous questions have not been answered yet, including, e.g., the role of re-absorption of water by dried-out SAP moieties residing in the voids. The air void counting/measuring method on hardened concrete can be applied to concrete with SAP too, cf. Chapter 9 in [9]. While this is applicable straight-forward with spherical SAP, there is still research going on to modify it for the case of crushed SAP particles. On the contrary, the generally performed testing of the total air content in fresh concrete according to [25], as a verification of an appropriate air content to enhance freeze–thaw resistance, cannot be transferred to cement-based materials containing SAP.

The incorporation of SAP in a concrete requires the use of a SAP stable and active when placed in the ionic pore solution with high pH-value [26]. SAPs that are obtained from the suspension polymerization or bulk solution polymerization production methods are suitable for application in concrete. The shapes of the SAP particles, and thus of the resulting voids caused by the incorporation of SAP, result from their production method; i.e., the presence of spherical pores from suspension polymerization and irregular from bulk solution polymerization. It is recommended to prequalify a SAP for use in concrete for water absorption behaviour, as described in [27]. The absorption behaviour should be retaining; i.e. in a time-resolved gravimetric sorptivity measurement with (extracted, mimicked, and synthetic) cement pore solution the polymer should not intensely desorb, but keep its swollen mass fairly constant for several hours [10, 26, 27].

The addition of dry SAP particles to a fresh concrete mixture leads to water uptake by the SAP, and therefore stiffening of the fresh mixture, which can increase water demand. Depending on the absorption capacity in pore solution environment (dissolved ions, pH-value and temperature [26–28]), SAP particles swell up to a given extent. When no extra water is added to compensate for SAP absorption, SAP can intake some of the mixing water, thus leading to reduced water-to-cement ratio (w/c) of the cement paste, known as the effective w/c. This can reduce the capillary porosity that can reduce permeability and enhance freeze–thaw resistance. Adding extra water for the absorption of the SAP changes the total w/c; however, it is not considered to significantly change the effective w/c of the cement paste surrounding SAP voids [9, 19]. The extra water used for the water absorption of SAP can mitigate plastic shrinkage cracking as reported in [12] and can also mitigate autogenous shrinkage as recommended in [14].

Recommended procedures for the determination of sufficient SAP content by measuring the spacing factor of hardened non-air entrained concrete made with SAP are described in Sect. 3.4. Besides the SAP voids, there are always some air voids entrained into concrete in the process of mixing also in the absence of AEA.

Since some of these air voids are also effective in freeze–thaw resistance they can be taken into account.

2.2 Determination of amount and properties of SAP for enhancing freeze–thaw resistance

The following steps present an approach for determining the SAP dosage for enhanced freeze–thaw resistance of concrete.

(1) Specify the targeted air pore volume $V_{air,target}$ (%), which is needed for satisfactory freeze–thaw resistance and subtract the original air pore volume V_{air} (%) of the concrete mixture according to Eq. 1:

$$V_{SAP} = V_{air,target} - V_{air} \tag{1}$$

where V_{SAP} is the entrained volume represented by swollen SAP particles.

(2) The volume of pores produced by first swollen and later desorbed SAP is presumably equal to the amount of absorbed water (for simplicity the volume of dry SAP particles is neglected).

(3a) In case of adding extra water for SAP absorption, the mass of extra water w_{SAP} (kg/m^3) is calculated as:

$$w_{SAP} = \frac{1000}{\frac{100}{V_{SAP}} - 1} \tag{2}$$

Leading to a total *w*/*c* of

$$(w/c)_{total} = (w + w_{SAP})/c \tag{3}$$

where *w* and *c* are masses of water and cement, respectively, in kg/m^3 of concrete.

(3b) If no extra water for SAP absorption is added, SAP can absorb some of the mixing water. This can reduce the content of free water that is available for hydration and formation of capillary pores. The absorption of mixing water by SAP also leads to a loss in workability of the fresh mixture. The total *w*/*c* is constant, but the effective w/c of the cement paste can be calculated according to Eq. 4:

$$w/c = (w - w_{SAP})/c \tag{4}$$

(4) With the absorption capacity of cement pore solution by SAP A_{SAP} (g_{PS}/g_{SAP}) the amount of SAP m_{SAP} (kg/m^3) can be calculated as follows:

$$m_{SAP} = \frac{w_{SAP}}{A_{SAP}} \tag{5}$$

(5) It can be stated as $m_{SAP\%c}$ (% bwoc):

$$m_{SAP\%c} = \frac{m_{SAP}}{c} \times 100 \tag{6}$$

With regard to a finely distributed pore system and small spacing factors (according to [22]), it is recommended to choose SAP with an absorption behaviour that leads to swollen SAP particles of relatively small sizes. In some existing guidelines, the maximum pore size considered in void counting on hardened concrete samples is considered as 300 µm. However, the results in [9, 28] show that SAP with greater sizes also contribute to enhancement of freeze–thaw resistance. The effectiveness of having different ranges of SAP pore sizes on frost durability has not been sufficiently investigated. Therefore, no limit values can be given at this stage. However, spacing factors as demanded by the existing codes can be used for system of voids (SAP voids plus air bubbles).

The absorption capacities of SAP samples can be determined by the methods recommended in [27]. They allow to determine the amount of absorbed cement filtrate as well as on absorption rate and sorption kinetics. However, it is possible that for some SAP and some specific mixture compositions of concrete, the actual absorption of SAP in the mixture may be lower compared to that determined in free absorption test [29]. Thus, an iterative process to determine the amount of SAP should be carried out. A reference mixture without SAP and extra water can be used as the reference flow spread diameter. For a second mixture, the amount of extra water for SAP-absorption is chosen according to the preceding steps. The amount of SAP should be adjusted until the flow spread of the SAP mixture equals the reference one. Additional information on this method can be found in [16].

2.3 Application of SAP in concrete: practical issues

The SAP absorption behavior may be affected by different ambient conditions, thus preferably they should be stored in opaque, sealed containers at RH below about 60% and at room temperature (approximately 20 °C).

SAP can be added to a mixture in dry or pre-saturated state. In most cases, SAP is added as a dry powder, and the extra water is added together with the mixing water. For pre-saturation of SAP, the dry SAP is added to the mixing water (including extra water). The absorption capacity of SAP in water is multiple times higher than that of the cement pore solution. Therefore, more than the amount of extra water is absorbed and during concrete mixing, when pH-value and ion concentration increase during cement hydration, some of the absorbed water is released. In [30], it was found that the pre-wetted SAP may result in a finer distribution of SAP pores, especially at high SAP contents. It should be also noted that pre-wetting of SAP particles may make their uniform distribution in the concrete mixture more difficult.

If SAP does not meet the assumed absorption capacity (no matter if added dry or pre-wetted, with or without extra water), there could be more or less water for hydration in the system, and thus inevitable change in *w/c*. That would lead to changes in formation of capillary pores, which directly impact the freeze–thaw resistance and strength of the material. Fresh concrete properties like workability or bleeding are also affected.

In [19, 31], surfactants were found in suspension polymerized SAP, which can lead to air entrainment, thus positively influencing frost durability and reducing concrete strength. Such air entrainment can be measured on fresh concrete using pressure method, see [25].

To provide evidence of SAP in a concrete mixture, a sample of fresh concrete can be taken and washed on a sieve with water. As SAP has a significantly higher absorption capacity of ordinary water, the particles swell multiple times and can be easily detected.

The verification of SAP quantity and accurate absorption of cement pore solution is done on hardened concrete by void counting. Void detection methods are described in Sect. 3.4.

2.4 Possible negative effects of applying SAP

The increased porosity in concrete resulting from SAP particles has a negative effect on mechanical properties. In comparison to traditional AEA, the loss in compressive strength, splitting tensile strength or Young's elastic modulus are similar or lower [9, 16].

3 Testing methods to characterize effect of SAP in cement-based materials with respect to frost durability and deicing salt scaling resistance

3.1 General rules

Any method used to assess frost resistance or deicing salt scaling resistance of cement-based material is suitable to assess the effect of SAP on such properties. Sections 3.2 and 3.3 provide some guidelines.

Production of SAP-modified cement-based materials for the respective tests does not differ from the preparation of materials without SAP addition. Reporting is carried out according to the cited guidelines. Specific prescriptions of how to select mixture proportioning and general production of cement-based building materials modified by SAP can be found in the literature, e.g. [9, 16].

3.2 Selected test methods to assess freeze–thaw resistance

- CEN/TS 12390-9:09/2006. Testing hardened concrete—part 9: freeze–thaw resistance—scaling [1];
- ASTM C666-15. Standard test method for resistance of concrete to rapid freezing and thawing—procedures A and B [2];
- JIS A1148:2010. Method of test for resistance of concrete to freezing and thawing [3];
- National Standard of the People's Republic of China. GB/T50082-2009. Beijing, China: National Standard of the People's Republic of China; 2009. The test method of long-term and durability of ordinary concrete. This standard differentiates between a procedure for slow freezing by air and thawing by water and a procedure for rapid freezing and thawing with water [4];
- GOST 10060-2012. Concretes: Methods for the determination of frost-resistance [5];
- RILEM protocol CIF [32].

3.3 Selected test methods to assess freeze–thaw and deicing agents resistance

- CEN/TS 12390-9:09/2006. Testing hardened concrete—part 9: freeze–thaw resistance—scaling [1];

- JIS A 1148:2010. Method of test for resistance of concrete to freezing and thawing [3].
- National Standard of the People's Republic of China. GB/T50082-2009. Beijing, China: National Standard of the People's Republic of China; 2009. The test method of long-term and durability on ordinary concrete [4];
- GOST 10060-2012. Concretes: Methods for the determination of frost-resistance [5];
- ASTM C672-12. Standard test method for scaling resistance of concrete surfaces exposed to deicing chemicals [8];
- RILEM protocol CDF [33];
- SIA 262/1:2003. Eingetragene Norm der Schweizerischen Normen-Vereinigung: Betonbau—Ergänzende Festlegungen, Anhang C: Frost-Tausalzwiderstand.

A total of 13 international research groups tested two different SAP materials in terms of their influence on freeze–thaw and deicing salt scaling resistance when used in ordinary concrete in an inter-laboratory experimental study. Most of the above mentioned standard test methods were used. The results are presented in [13]. This holds true also for the methods listed in Sect. 3.2.

3.4 Air-void system

Traditional void counting methods (e.g. [22]) can be applied to evaluate voids generated by SAP. Spacing factor (and in some norms also an additional parameter) characterises the air void system of hardened concrete. If the content and particle size distribution of dry SAP as well as its absorption capacity are known, the number and size of cavities formed in concrete due to the SAP addition can be calculated theoretically, proving an estimation of the real void system.

The air-void system can be evaluated using ASTM C457 [23] test method to determine the air content of the hardened concrete, the specific surface, and spacing factor of air bubbles. The test is conducted by three methods using: (1) Linear-Traverse Method (using a stereoscopic microscope); (2) Modified Point-Count Method (using a stereoscopic microscope); and (3) Contrast Enhanced Method (image processing). The test method described in [22] is based on assumption of spherical pores. Therefore, angular pores, generated by bulk polymerized SAP actually do not qualify for such void counting, because of their irregular shapes. In that case, the method provides only approximate results. A more precise way to evaluate a 2-D sample based on image analysis is described in [33]. These methods take into account all air or SAP induced voids. To differentiate SAP induced voids from other air voids, in case of angular SAP voids a shape analysis can be performed. Otherwise, for a rough estimation, the measured air volume of fresh concrete could be subtracted from the total air volume as obtained by void counting. An elaborate and more expensive but very precise method can be performed by CT-scan of a concrete sample and subsequent analyses of the data, as described in [30, 34]. To detect single SAP pores, hardened concrete samples or prepared thin sections can be examined under a microscope. The dehydrated SAP particle can be found on the pore walls.

Acknowledgements The contributions of all TC members in discussion during the drafting of this recommendation are gratefully acknowledged. The authors extend their thanks to the industrial partners for the proofreading and valuable comments.

Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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