



Internal curing by superabsorbent polymers in ultra-high performance concrete



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ABSTRACT

To limit self-desiccation and autogenous shrinkage that may lead to early-age cracking of ultra-high performance concrete (UHPC), internal curing by means of superabsorbent polymers (SAP) may be employed. Cement pastes and UHPC with water-to-cement ratio below 0.25, with or without SAP, were studied. The absorption capacity of a solution-polymerized SAP was first determined on hardened cement pastes by SEM image analysis. It was observed that the SAP cavities become partially filled with portlandite during cement hydration. Isothermal calorimetry showed that water entrainment with SAP delays the main hydration peak, while after a couple of days it increases the degree of hydration in a manner similar to increasing the water-to-cement ratio. Internal curing by SAP is effective in reducing the internal relative humidity decrease and the autogenous shrinkage. Although the mechanical properties are affected by SAP addition, it is possible to reach compressive strengths of almost 150 MPa at 28 days.

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1. Introduction

Ultra-high performance concrete (UHPC) is a class of cementitious materials with outstanding material properties, in particular high compressive strength (>140–150 MPa), high tensile strength (e.g. >8 MPa) and ductility in tension thanks to the incorporation of steel fibers, and low permeability and high durability thanks to their dense matrix [1–3]. The exceptional properties of UHPC allow to use it for extremely slender structures [4] for efficient prefabrication and for advanced in-situ applications, e.g. as waterproofing layers in bridge decks [5].

An UHPC mixture combines densely packed aggregate with small maximum aggregate size (often below 1 mm), low water-to-cement ratios (w/c, typically 0.20 or lower), and often fine fillers or supplementary cementitious materials (especially silica fume) [1–3]. Due to the extremely low w/c, the cement may reach a final degree of hydration of less than 50% [6]. In these conditions, the hydration reaction is brought to a halt by the lack of free water and of capillary pore space to precipitate hydration products [7]. During hydration in sealed conditions, the capillary pores in the hardening cement paste of a UHPC progressively empty since the hydration products have lower volume than the sum of the volumes of unhydrated cement and water (chemical shrinkage) [8]. The radius of the menisci at the interface between air

and pore fluid progressively decreases as the largest pores empty and the porosity fills up with hydration products [9]. As a consequence, the internal relative humidity (RH) decreases (self-desiccation), the pore fluid is subjected to increasing capillary tension, and the porous skeleton to increasing compressive stress that results in autogenous shrinkage [8]. In order to decrease self-desiccation and promote cement hydration, additional water (curing water) may be provided to UHPC. However, due to the very low permeability [10], UHPC cannot be efficiently cured with external water and internal curing needs to be employed instead.

Internal water curing indicates the process by which small inclusions dispersed within the concrete hold the water during mixing and up to setting time and release it during cement hydration [11]. In high-performance concrete (HPC, with compressive strengths from about 80 to 120 MPa), internal curing has been performed mostly with saturated lightweight aggregate [12,13] and with SAP [14], see also [15]. While the presence of additional porosity in internally cured HPC may in some cases result in reductions of the compressive strength compared to the reference concrete, the benefits in term of autogenous shrinkage reduction and cracking control are overwhelming [16–20]. Moreover, the strength reduction does not need to be a case in HPC [21,22]. On the other hand, UHPC reaches very high strengths primarily through very low w/c, very low porosities, and extremely small sizes of defects present in the microstructure. In UHPC, it is therefore very difficult to perform internal curing by introducing (initially water-filled) macropores, without substantially impacting the strength.

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Possibly due to the difficulty of reconciling the additional porosity introduced with the internal curing agents and the required very high strength, literature reports on properties of UHPC with internal curing are scarce. Van Tuan et al. [23] obtained UHPC with compressive strengths above 150 MPa and increased degree of hydration of the cement by substituting silica fume with porous rice husk ash. Ye et al. [24] measured internal RH development and autogenous shrinkage in UHPC, showing that rice husk ash is effective as an internal curing agent. Mechtcherine et al. [25] were able to obtain complete autogenous shrinkage elimination by applying internal curing with SAP to a reference UHPC that experienced high autogenous shrinkage (about 1100 $\mu\text{m}/\text{m}$ at 7 days). However, at the higher SAP dosages necessary to eliminate shrinkage, the compressive strength dropped to less than 100 MPa [25]. In a later publication of the same group, compressive strengths as high as 155 MPa were reported for internally cured UHPC, albeit for a mixture in which the autogenous shrinkage was reduced but not eliminated [26].

The main objective of this study was to formulate UHPC mixtures with internal curing in which the long-term autogenous shrinkage (up to 28 days or longer) would be substantially reduced, while still guaranteeing cube compressive strengths at 28 days in excess of 140 MPa. In order to minimize the impact of the large pores due to the internal curing agents on the strength of the concrete, small SAP (particle size less than $<63 \mu\text{m}$ in the dry state) were employed. This allowed not only to reduce the size of the macropores in the UHPC, it also improved the water distribution in the UHPC by reducing the distances of water migration from the internal curing agents to any point of the paste, an aspect that may become important in UHPC with extremely low permeability [10,27]. In order to be able to precisely design the internally cured mixtures, the absorption capacity of the SAP was first determined on neat cement pastes of compositions corresponding to those used in the UHPC mixtures with a method based on image analysis of polished sections imaged with scanning electron microscopy (SEM). To obtain a global understanding of internal curing at very low w/c, the development of the hydration reaction (isothermal calorimetry) and the setting time (Vicat) were measured on cement pastes. In addition, RH evolution, autogenous deformation and mechanical properties (compressive and bending strength, elastic modulus) were measured on reference UHPC and internally cured mixtures.

2. Materials and methods

2.1. Materials

White portland cement (CEM I 52.5 R) was used in this study. Table 1 shows the oxide composition of the cement together with the normative composition determined with a modified Bogue calculation

Table 1
Chemical composition of the CEM I 52.5R cement. Normative phase composition calculated with modified Bogue method [28].

Chemical analysis, %		Normative phase composition, %	
SiO ₂	24.33	Alite	77.5
Al ₂ O ₃	2.07	Belite	14.3
Fe ₂ O ₃	0.36	Aluminate	2.8
CaO	69.02	Ferrite	0.1
MgO	0.64	CaO	0.85
SO ₃	2.04	CaCO ₃	0.16
K ₂ O	0.04	CaSO ₄	3.3
Na ₂ O	0.15	K ₂ SO ₄	0.07
CaO (free)	0.85	Na ₂ SO ₄	0.15
TiO ₂	0.07	K ₂ O	0.002
Mn ₂ O ₃	0.02	Na ₂ O	0.1
P ₂ O ₅	0.36	MgO	0.64
L.o.I.	0.77		

L.o.I.: loss on ignition.

[28]. The density of the cement was 3130 kg/m³ and the mean particle diameter (determined by laser diffraction) was 6.7 μm .

A silica fume with SiO₂ content $>96.5\%$ was used. Particle size measurements by laser diffraction performed on silica fume suspensions in water indicated that the silica fume was agglomerated (mean particle diameter 78 μm). To limit the amount of silica fume agglomerates in the cement pastes, a special pre-treatment was required to improve the dispersion (see Section 2.3.1).

Quartz powder with SiO₂ content of 97.0% was added as filler. The mean particle diameter was 3.73 μm , and 97% of particles were smaller than 12 μm .

Quartz sand (SiO₂ content $>93.3\%$) was added as aggregate in two fractions, namely, 0.1–0.3 mm and 0.3–0.9 mm.

A commercial polycarboxylate-based superplasticizer with 22% solid content and specific gravity of 1.04 was used for all mixtures. A commercial defoamer with specific gravity 0.97 was also added.

Solution-polymerized SAP of irregular particle shape and particle sizes $<63 \mu\text{m}$ in the dry state were used. In order to design the UHPC mixtures, the pore solution absorption of the SAP in cement pastes was precisely determined with the procedure described in Section 2.2.

Brass-coated steel fibers with a length of 10 mm and diameter of 0.16 mm were added to the UHPC mixtures.

2.2. Determination of SAP absorption: SEM and image analysis

The absorption potential of SAP was determined based on the area fraction occupied by the voids partially filled with hydration products left by the SAP on the scanned sections. Based on Delesse's principle [29], the area fraction occupied by the representations of the SAP remnants on the polished section gives an estimate of their volumetric fraction.

To determine the pore fluid absorption capacity of the SAP particles, a reference cement paste without SAP (CP-0.15) and a cement paste with SAP (CP-0.25SAP), both with 10% silica fume addition by mass of cement, were compared (Table 4). In the case of mixture with SAP, the w/c was increased by 0.10 compared to the reference paste, and the SAP amount was determined as necessary to absorb the additional curing water based on an initially assumed absorption potential of 20 $\text{g}_{\text{pore fluid}}/\text{g}_{\text{SAP}}$. The pastes were mixed in a vacuum mixer and cylindrical samples of diameter 33 mm were cast. At the age of 3 days, disks of approximately 10 mm height were cut from the cylindrical samples using a slow motion diamond saw. The hydration in the cement paste samples was stopped by means of immersion in isopropanol for 5 days and oven drying at 40 °C for 4 days. The samples were next impregnated under vacuum with modified bisphenol-A epoxy resin and polished with polycrystalline diamond suspensions (grades 9–0.25 μm). After carbon coating, the samples were imaged with an environmental scanning electron microscope (ESEM-FEG XL30) in the high vacuum mode with an accelerating voltage of 15 kV and a beam current of 170–185 μA . More than 300 images per sample were taken in the backscattered electron (BSE) mode, with magnification 200 \times ; the image size was 0.85 \times 0.66 mm² and the pixel size was about 1 μm . The linear size of the analyzed features (SAP voids) typically did not exceed 250 μm . The image analysis started with applying a bilateral filter [30] in order to remove the noise and homogenize the gray-level value of the features while preserving their original edges. The SEM images were clustered using k-means clustering [31] operating on the 8-bit grayscale into five separate clusters, representing: (1) porosity, (2) porous hydration products (including interfacial transition zone) and silica fume agglomerates, (3) hydration products in the matrix, (4) portlandite, and (5) unhydrated cement particles. The assumed number of clusters and assignment of different clustered phases to the materials was based on the morphology and aided with energy-dispersive X-ray spectroscopy (EDX) performed at chosen points of the pastes, see exemplary identification of portlandite in Fig. 1. Binary opening (erosion-dilation by 1 pixel) was additionally performed on

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