



Influence of spraying on the early hydration of accelerated cement pastes



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ABSTRACT

In practice, most of the studies about the interaction between cement and accelerators is performed with hand-mixed pastes. However, in many applications mixing occurs through spraying, which may affect accelerators reactivity and the microstructure of the hardened paste. The objective of this study is to analyze how the mixing process influences the early hydration of accelerated cement pastes. Isothermal calorimetry, X-ray diffraction, thermogravimetry and SEM imaging were performed on cement pastes produced by hand-mixing and by spraying, using equivalent doses of an alkali-free and an alkaline accelerator and two types of cement. Results showed a great influence of the spraying process on the reactivity of accelerators and on the morphology of the precipitated hydrates. Variations in hydration kinetics caused by the mixing method are explained and the results obtained might have a significant repercussion on how future research on the behavior of accelerated mixes will be performed.

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1. Introduction

Sprayed concrete differs from ordinary concrete due to the method of application and the use of set accelerators to promote rapid stiffening of the matrix right after mixing [1]. Accelerators are normally added to concrete at the nozzle, just before it is sprayed onto the substrate. In tunnel applications, these chemicals are necessary to provide high initial mechanical strength, so the concrete layer is able to provide support to the unstable ground. Depending on their chemical composition, different hydration mechanisms and mechanical strengths are observed in accelerated matrices [1,2].

In order to evaluate the compatibility and reactivity of accelerators with cement, the vast majority of studies deals with accelerated cement pastes produced by hand or mechanical mixing [2–7]. Martinez [3] showed that increasing mixing time and speed shortens setting times of accelerated cementitious mortars. Juilland [5,8] concluded that high mixing speeds have an acceleration effect on hydration kinetics in cement pastes with or without accelerator.

Despite the relevant conclusions derived from the studies from the literature, all of them are based on mixing processes that differ considerably from the one used in most applications. This is aggravated by the fact that nearly no study on the influence of the mixing process on the

microstructure or hydration kinetics may be found. Lagerblad and Byrne [9] indicate that the paste preparation method might have significant influence on the microstructure. In addition to that, Lindlar et al. [10] suggest that a more homogeneous dispersion of the accelerator is achieved through spraying and the hydrated products should present a more uniform distribution in the matrix when compared to a hand-mixed equivalent matrix. Juilland [5] describes that the massive ettringite formation during spraying fills up the matrix and less space remains for the silicate hydration to proceed. However, the repercussions in terms of the microstructure and in the chemical reactions involved are yet not clear.

The objective of this work is to investigate the influence of the production process on the hydration and the microstructure of cement pastes with accelerators. An experimental program was performed with two types of cement and two types of accelerator. For the study, three types of pastes were produced: reference hand-mixed pastes without accelerators, hand-mixed pastes with accelerators and sprayed pastes with accelerators. The composition and the production process of the hand-mixed pastes follow that normally found in the literature about the behavior of accelerators [2]. Conversely, the composition and the production process of sprayed pastes resemble those found in practice, being conditioned by the requirements of the equipment used.

A comparison between hand-mixed and sprayed accelerated pastes was performed. In order to characterize the kinetics and mechanisms of hydration, isothermal calorimetry, X-ray diffraction, thermogravimetry and SEM analysis were conducted. The study explains important

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Table 1
Mineralogical composition of cements determined by XRD/Rietveld refinement.

Compound	CEM I (%)	CEM II/A-L (%)
C ₃ S	58.3	51.7
C ₂ S	11.2	6.7
C ₃ A _c	4.1	2.9
C ₃ A _o	0.6	0.7
C ₄ AF	13.4	14.8
CaO	1.1	1.2
Ca(OH) ₂	1.7	0.7
CaCO ₃	1.9	11.3
CaSO ₄ ·2H ₂ O	2.1	0.7
CaSO ₄ ·0.5H ₂ O	4.4	5.3
K ₂ SO ₄	–	0.5
K ₂ Ca(SO ₄) ₂ ·H ₂ O	1.1	–
MgO	–	0.6
MgCO ₃	–	3.1
Total sum	100.0	100.0

differences in hydration behavior caused by the mixing process and highlights in which situations the characterization of matrices produced by spraying might be necessary.

2. Experimental methodology

The experimental program was performed at the Laboratory of Technology of Structures Luis Agulló at Universitat Politècnica de Catalunya (UPC) and at the Scientific and Technological Center from Universitat de Barcelona (CCIT-UB). In the following sections, materials, production processes and tests performed are described.

2.1. Materials

Two types of Portland cement (CEM I 52.5R and CEM II/A-L 42.5R) were used in this study. These cements were selected among the commonly used in sprayed concrete applications. CEM I is common in Spain and in countries from Asia and America, while CEM II/A-L is widely used in countries from northern Europe. Table 1 presents their mineralogical composition determined by XRD-Rietveld refinement, Table 2 shows their chemical composition determined by XRF spectrometry and Table 3 summarizes their chemical and physical properties.

Distilled water and a superplasticizer based on a polycarboxylate solution (34% of solid content) were used to prepare all pastes. One alkali-free accelerator composed by an aluminum hydroxysulfate solution stabilized by an organic acid and one alkaline accelerator based on sodium aluminate were used to cover the types commonly found in practice. Composition and pH of accelerators are presented in Table 4.

Table 2
Chemical composition of cements determined by XRF spectrometry.

Compound	CEM I (%)	CEM II/A-L (%)
LOI	2.88	6.42
CaO	62.62	62.48
SiO ₂	19.89	17.61
Al ₂ O ₃	4.74	4.04
Fe ₂ O ₃	3.26	3.53
SO ₃	3.53	3.21
MgO	1.95	1.69
K ₂ O	0.99 (0.98) ^a	0.83 (0.62) ^a
Na ₂ O	0.13 (0.011) ^a	0.13 (0.035) ^a
Minor components	0.11	0.09
Total sum	100.0	100.0

^a K₂O and Na₂O readily soluble, determined by ion chromatography from a solution extracted from a cement suspension with w/c equal to 9 [11,12].

Table 3
Chemical and physical properties of cements.

Property	CEM I	CEM II/A-L
Total heat of hydration ^a (J/g)	433.0	381.0
C ₃ A/SO ₃ molar ratio	0.39	0.33
Insoluble residue (%)	2.74	2.90
Specific surface BET (m ² /g)	2.96	1.88
d ₅₀ (μm)	11.4	15.7

^a The total heat of hydration was estimated from the mineralogical composition of the cement, determined by XRD, as the relative sum of the heats of hydration of the individual phases (C₃S: 510 J/g; C₂S: 260 J/g; C₃A: 1100 J/g; C₄AF: 410 J/g [13]).

2.2. Compositions and preparation of cement pastes

The composition and preparation procedure of the mixes studied are presented in Sections 2.2.1 and 2.2.2 depending on how accelerators are mixed with the paste. Cement pastes were prepared under controlled climatic conditions, at 20 °C and 50% relative humidity.

The nomenclature adopted to identify each composition follows the pattern 'cement type'_'accelerator type'_'mixing process'_'w/c ratio'. Alkali-free and alkaline accelerators are abbreviated as AKF and ALK, respectively. Regarding the mixing process, HM corresponds to hand-mixed and SP to sprayed pastes. Reference pastes do not contain accelerators and are identified by 'cement type'_'REF'_'mixing process'_'w/c ratio'.

2.2.1. Hand-mixed pastes

Reference pastes are composed by cement, a water/cement ratio equal to 0.45 and 1.0% of superplasticizer by cement weight (% bcw). In accelerated pastes, the alkali-free accelerator was used at 7.0% bcw, whereas the alkaline accelerator was added at the dosage of 3.0% bcw. Those contents were determined according to the procedure described in [1] to assure equivalent mechanical performance in pastes and fall within the dosages usually applied in tunnels executed with sprayed concrete.

The composition of each paste in terms of ions and compounds present is described in Table 5. An information of interest is the final C₃A/SO₃ ratio after accelerator addition. As the alkaline accelerator does not contain any sulfates in its composition, [Al(OH)₄][−] consumes approximately 48% of the sulfate present in cement, resulting in a final C₃A/SO₃ ratio two times higher than the initial value from cement. This effect is less pronounced when the alkali-free accelerator is used, since this admixture contains sulfate ions in its formulation.

Reference pastes were prepared by pre-mixing water and superplasticizer and homogenizing the resulting solution with cement by means of a vortex external mixer during 60 s. In hand-mixed accelerated pastes, accelerators were added to the pastes 1 h after cement and water had been mixed. The resulting mix was vigorously homogenized for 15 s with a spatula. During the period between the mixing of cement and water and accelerator addition, the paste was kept inside a calorimeter at 20 °C in order to avoid the influence of variations in temperature.

This procedure intends to reproduce the condition usually found in practice, since the introduction of the accelerator only occurs almost 1 h after the mixing of the other components. Moreover, this also allows

Table 4
Compositions and pH of accelerators.

Characteristic	Alkali-free	Alkaline
Solid content (%)	47.6	43.0
Al ₂ O ₃ content (%)	13.5	24.0
SO ₄ ^{2−} content (%)	21.0	–
Na ₂ O content (%)	–	19.0
pH at 20 °C	3.0	12.0 ^a

^a Solution at 1.0%.

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