# Combined use of X-ray diffraction and microtomography for accurate cement hydration studies

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Accuracy in early age Portland cement (PC) hydration analyses is not straightforward as multimineral material has an inherent variability and different techniques are subject to different experimental errors and diverse approximations in the calculations.

Here, an innovative *in situ* PC hydration investigation is reported. The PC conforms to CEM I 42.5R according to EN 197-1. The paste was prepared with a water-to-cement mass ratio of 0.50, at 25±2 ºC, without admixture(s) and loaded and sealed in a thick glass capillary, **ϕ=2.0 mm**, *to have excellent powder averaging* for the X-ray powder diffraction and microtomographic analyses. The data were taken on a D8 ADVANCE (Bruker) diffractometer using strictly monochromatic MoK$\alpha_1$ radiation ($\lambda$=0.7093 Å) and on a SKYSCAN 2214 (Bruker) scanner using a source of $LaB_6$. Data were taken not simultaneously but sequentially.

On the one hand, from diffraction, we quantitatively followed the dissolution of the cement phases and the crystallization of portlandite and ettringite. The amount of C-S-H gel has been indirectly determined from the known-chemical reactions (mass balance calculations.). See top row of Fig. 1 for inspecting the quality of the resulting Rietveld plots. Hence, time and phase-dependent degree of hydration (DoH) were obtained. On the other hand, from μCT, the dissolution of the anhydrous cement phases (whitish particles) and the crystallization/precipitation of hydrates (darkish regions) can be followed. See bottom row of Fig. 1 for observing these processes. In these conditions, (i) $Ca(OH)_2$ carbonation is avoided, (ii) experimental errors are minimised because the use of rotating capillary (like water microbleeding in flat-sample geometry), and chiefly the results from two techniques are combined which improves the accuracy and it allows to detect errors.

This is a (required) first step in our long-term endeavour of using the combination of μCT and LXRPD, for determining the DoH of amorphous materials in PC blends with supplementary cementitious materials (SCMs). Our final goal is to be able to directly measure the DoH of amorphous components of SCMs as well as the $Ca(OH)_2$ consumption by the pozzolanic set of reactions.

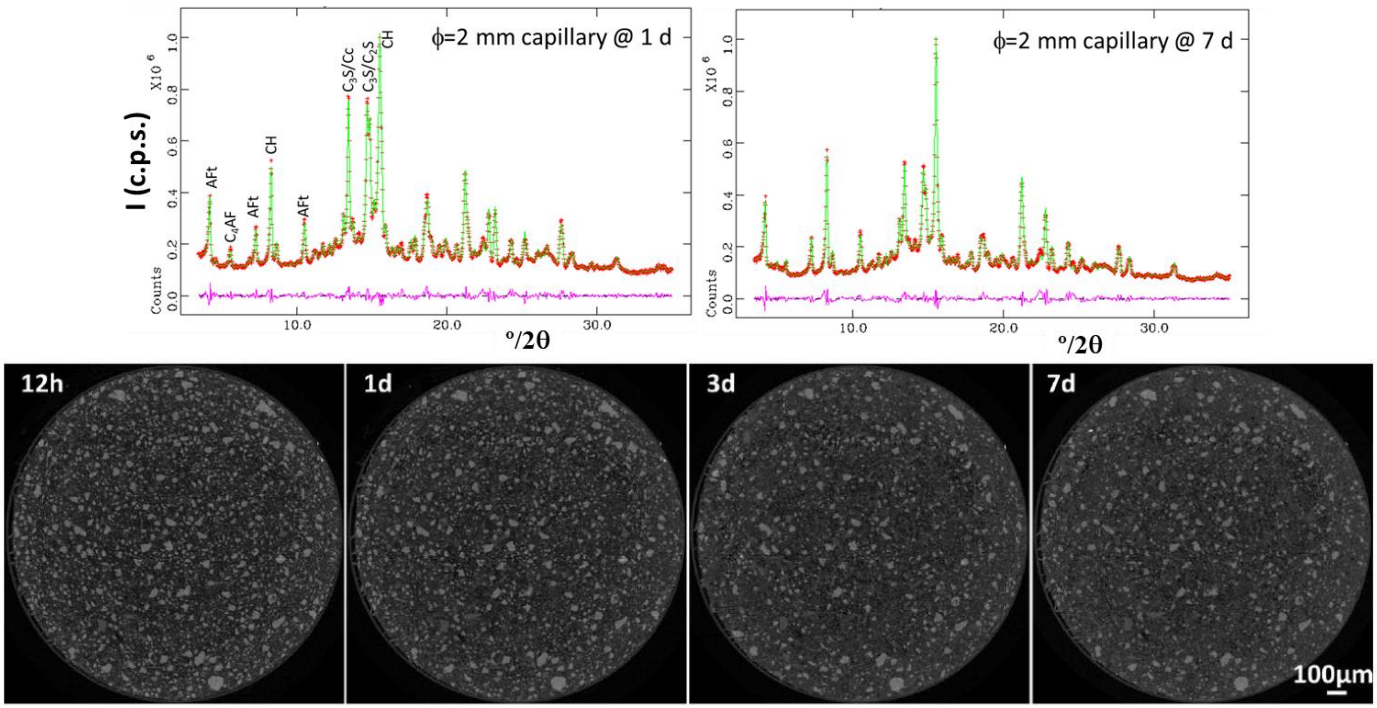


**Figure 1**. Combined diffraction and tomographic study in the same capillary, ϕ=2.0 mm, where a PC 42.5R paste is hydrating. Top row: Selected Rietveld plots ($\lambda$=0.7093 Å) at 1 day (left) and 7 days (right). Key diffraction peaks are labelled. The decrease in the intensities for alite ($C_3S$) and the increase for portlandite (CH) peaks is evident. Bottom row: Selected orthoslices showing the cement hydration evolution with time. The whitish particles are the anhydrous Cement Particles and the grey regions are the Hydrated Products. Porosity develops as blackish regions

More technical and scientific details will be presented at the meeting.

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