Oral presentation

Multimodal study of unaltered cement hydrating pastes: combined high-energy laboratory powder diffraction and microtomography

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Background: The study of the hydration pathways of Portland cement (PC) and cement blends presents significant challenges due to their multiphase nature and varying fineness. Quantifying the dissolution of initial crystalline and amorphous phases, along with the formation of new components, is crucial. Additionally, understanding microstructural changes with time is also essential.

Techniques: We are developing a novel approach based on *in situ* measurements of cement pastes without sample conditioning. The pastes are injected in thick capillaries (2 mm of diameter) and just the ends are sealed. Sequential data acquisition is carried out through Mo-Kα_1 laboratory X-ray powder diffraction (LXRPD) and microtomography (μCT) [1-2]. This methodology is based on scanning not only the same capillary but the same volume over time to minimize variability. The use of wide capillaries is pivotal for artefact avoidance, including self-desiccation, and ensuring excellent particle averaging.

Results: This approach was initially used for studying the hydration of a PC 42.5 R cement [1]. The results of the *in situ* studies were compared with *ex situ* prepared pastes to measure the accuracy of the protocols.
In a second work [2], we have studied three pastes: (i) a PC 52.5 R; (ii) a blend of 80 wt% PC and 20 wt% quartz, as a reference for supplementary cementitious materials (SCMs); and (iii) a blend of 80 wt% PC and 20 wt% limestone to simulate limestone Portland cement. Crystalline phase amounts are obtained by Rietveld analysis, and amorphous phase contents are calculated through mass balance. Within the achieved spatial resolution of μCT, three components (porosity, hydrated products, and unhydrated cement particles) are quantified. At 7 d, the volume percentages of hydrated compounds were 88.1% and 87.3% by LXRPD and μCT. Currently, we are using the optimized protocol for calcined clay containing blends. The final aim is to use this methodology to study the pozzolanic reactions for any PC-SCMs blend. We are testing a composition of 52 wt% of PC, 30 wt% of calcined clay, 15 wt% of limestone and 3 wt% of gypsum with this 'mix and measure' approach including its activation. A general discussion will be provided.

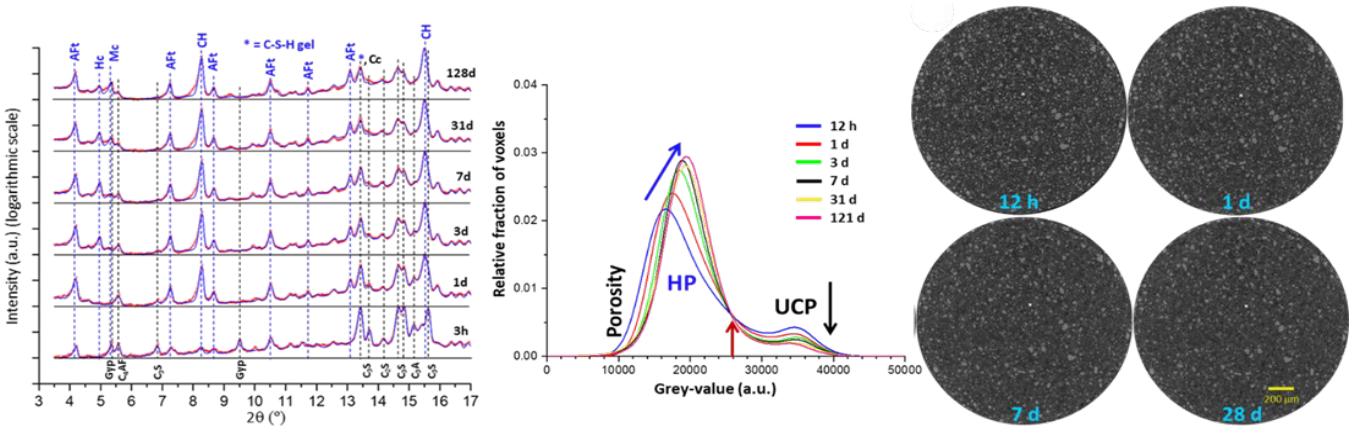

Figure 1. Time evolution of the PC 52.5 R cement paste as characterized by: (left) Mo-Kα1 LXRPD Rietveld plots in logarithmic scale vertically displaced for better visualization, the difference curves are not shown. The diffraction peak from key phases are labelled. (Centre) Grey-value histograms from lab-μCT. The evolution of the three identified main components are shown. (Right) Selected orthoslices of the lab-μCT. Bright: unhydrated cement particles, grey: hydrated components, black: porosity.

[1] Shirani, S., Cuesta, A., De la Torre, A. G., Santacruz, I., Morales-Cantero, A., Koufany, I., Redondo-Soto, C., Salcedo, I. R., León-Reina, L. & Aranda, M. A. G. (2024). *Cem. Concr. Res.* **175**, 107370.

[2] Fernandez-Sanchez, J., Cuesta, A., Shirani, S., Redondo-Soto, C., De la Torre, A. G., Santacruz, I., Salcedo, I. R., León-Reina, L. & Aranda, M. G. (2024). *J. App. Cryst*. (in revision).

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