Combining laboratory X-ray powder diffraction and microtomography for studying cement hydration

Miguel A. G. Aranda,[a] Shiva Shirani,[a] Cinthya Redondo-Soto,[a] Ana Cuesta,[a] Isabel M. R. Bernal,[a] Alejandro Morales-Cantero,[a] Isabel Santacruz,[a] Angeles G. De la Torre,[a] Inés R. Salcedo,[b] Laura León-Reina,[b]

[a] *Departamento de Química Inorgánica, Universidad de Málaga, 29071-Málaga (Spain)*

[b] *Servicios Centrales de Apoyo a la Investigación - SCAI, Universidad de Málaga, 29071-Málaga (Spain)*

e-mail: g_aranda@uma.es

Area: *in-situ crystallography; structure and properties of materials;*

The study of the hydration of cements is complicated due to the number of crystalline and amorphous phases and their complex, evolving, microstructures with hydration age. Moreover, to decrease the CO_2 footprint of cements, the Portland clinker is being partly replaced by supplementary cementitious materials like slag, fly ash or calcined clays, adding complexity to these studies. However, to understand cement hydration is very important to ensure the performances of the binders, including adequate mechanical strength development at early ages and good durability features at later ages.

The cement science group of University of Malaga (UMA) is focused on contributing to the decrease of the CO_2 footprint of cements. This is carried out by studying a number of low carbon cements including those obtained by the replacement of a fraction of the Portland clinker by a mixture of calcined kaolinitic clays and limestone or by natural pozzolans, like volcanic ashes. In previous works, we have studied, independently, (i) the phase development by Rietveld analysis of the X-ray powder diffraction patterns (LXRPD); and (ii) the microstructure evolution by image analysis, including segmentation, of X-ray computed tomographic (μCT) data. Because UMA has both equipments: (a) μCT, and (b) LXRPD with a high energy source (strictly monochromatic $MoK\alpha_1$ radiation); an innovative research avenue has been opened by collecting these two types of data for the same hydrating cement in the same capillary (a multimodal approach). In a recent paper, μCT and LXRPD data were taken and analyzed at 50 days of hydration.[1] However, this first study did not have datasets as function of time, i.e. *in situ* study.

Here, we will show results for selected cements hydrated *in situ* during the first seven days. μCT and LXRPD data were taken and analyzed. As an example, Fig. 1 shows an orthoslice of the tomogram for a hydrating PC-52.5R paste, data collected at 19 h of hydration, and the Rietveld plot for the same sample/capillary where the diffraction data were collected at 22 h of hydration. Several other hydration ages were also studied. In addition to the results, advantages and problems of this approach will be discussed.

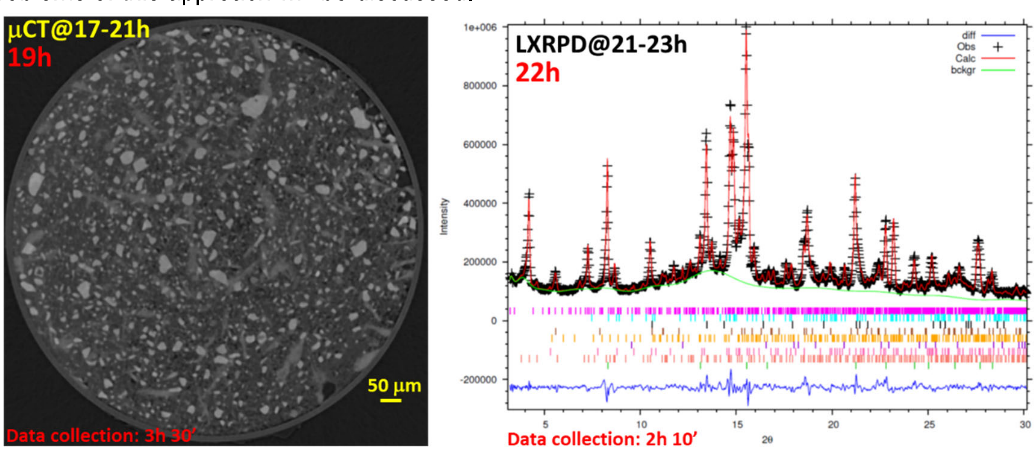

Figure 1. Dual study of the PC-52.5 hydrating paste, w/c=0.40, within a given glass capillary. (Left) 2D view of the reconstructed laboratory μCT data. (Right) Rietveld plot of the laboratory X-ray powder diffraction data, $MoK\alpha_1$ radiation.

References

[1] I.R. Salcedo, *et al. Materials* **2021**, 14, 6953.