

## ACCURACY IN CEMENT HYDRATION INVESTIGATIONS: COMBINED X-RAY MICROTOMOGRAPHY AND POWDER DIFFRACTION ANALYSES

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### 1. Introduction

Cements are multi-phase materials that can be well-understood using a multi-technique approach. Furthermore, the hydration of cement pastes is a very complex set of processes. In this work, we focus on the accuracy of the results of the laboratory computed tomography ( $\mu$ CT) analysis by quantifying three sets of components based on their attenuations: porosity, hydration products (HP) and unhydrated cement phases (UHP), and comparing with laboratory X-ray powder diffraction data (LXRPD). This communication is an extended summary of the results very recently reported in [1].

### 2. Materials and Methods

Portland cement pastes were prepared with two water-to-cement mass ratios ( $w/c = 0.45$  and  $0.65$ ) filled within glass capillaries ( $\phi = 0.5$  and  $1.0$  mm) and studied at 50 days of hydration. The same pastes were casted within cylinders and used as reference samples in order to be characterized by thermal analysis and Rietveld method, determining the free water (wt%) and the amorphous contents, respectively. The  $\mu$ CT images were acquired on a SKYSCAN 2214 (Bruker) equipment located at SCAI with a voxel size of  $0.80 \mu\text{m}$  using a  $\text{LaB}_6$  source (55 kV,  $130 \mu\text{A}$ ). Image segmentations were obtained by global thresholding of the reconstructed tomograms after applying  $3 \times 3 \times 3$  median 3D filter (Fiji).

### 3. Results and Conclusion

A methodology for obtaining  $\mu$ CT and LXRPD datasets for a cement paste within a capillary has been very recently reported [1], recommending diameters close to  $1.0$  mm in order to minimise the consequences of unavoidable experimental variations. The capillaries results obtained from both X-ray techniques were complemented with results from pastes casted in moulds, which allowed to determine the accuracy of the outputs. For our experimental conditions, the segmented values are accurate only if the water capillary porosity is segmented within the HP (2 vol% error). Further details will be given.

### 4. Acknowledgements

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### 5. References

[1] I.R. Salcedo et al, 10.3390/ma14226953

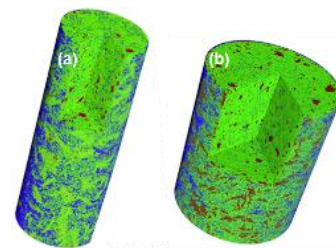


Figure 1. Rendered volumes displaying the results of the segmentations. Color codes: brown-UHPs, green-HPs, and blue-porosity (air and some remaining water).  $w/c = 0.65$  (a)  $\phi = 0.5$  mm and (b)  $\phi = 1.0$  mm paste.



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