ICTMS 2022
5th International Conference on Tomography of Materials & Structures
27th June- 1st July 2022, Grenoble, France

UNIVERSIDAD DE MÁLAGA

Cement Science @ UMA
https://sites.google.com/view/cementscience-uma

TIME-DEPENDENT MONITORING OF CEMENT HYDRATION BY COMBINED LABORATORY X-RAY MICROTOMOGRAPHY AND POWDER DIFFRACTION

Redondo-Soto C.[1], Cuesta A.[1], Salcedo I. R.[2], León-Reina L.[2], Aranda M.A.G.[1]

1 Departamento de Química Inorgánica, Cristalografía y Mineralogía, Universidad de Málaga, 29071 Málaga, Spain.

2 Servicios Centrales de Apoyo a la Investigación, Universidad de Málaga, 29071 Málaga, Spain.

Introduction

Following the reaction degree of amorphous phases in cements is very challenging. It becomes even harder when some additions (supplementary cementitious materials) are employed. Nowadays, calcined clays are attracting a lot of attention as it is possible to reduce the clinker factor by 50%, which leads to a 40% CO_2 cement footprint reduction. Thus, the aim of our overall project is to follow the reaction degree of amorphous anhydrous cement phases (for instance metakaolin) to yield different amorphous phase(s) (f.i. C-A-S-H gel) by combined X-ray micro-computed tomography (µCT) and powder diffraction (PD) analyses at different ages of hydration. Furthermore, some microstructural features, like porosity, can also be mapped out which are important for durability.

Keywords

X-ray micro-computed tomography (µCT), X-ray powder diffraction (PD), Rietveld Quantitative Phase Analysis (RQPA), supplementary cementitious materials (SCM), amorphous phases content (ACn).

Materials and Methods

1. CEM I 42.5 R type Portland cement (PC);
2. Pure kaolinite calcined to yield metakaolin (MK);
3. Gypsum (G).

For PC-MK-G sample, the selected materials were mixed with this proportions: 67 wt% of PC, 30 wt% MK and 3 wt% of G.

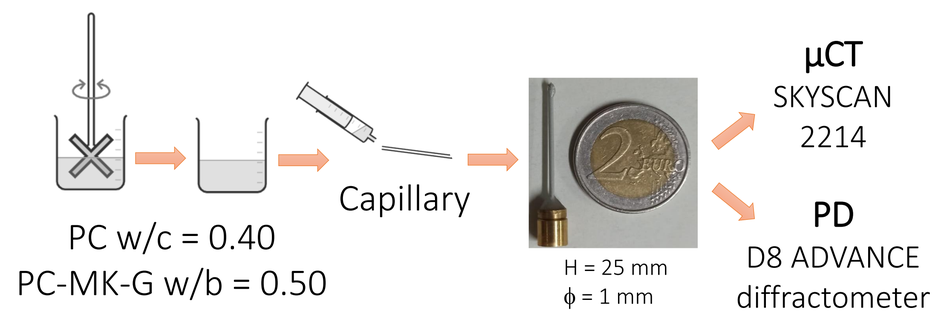

Fig. 1. Diagram of pastes preparation. The pastes within the glass capillaries were studied by: i) µCT with a SKYSCAN 2214 (Bruker) equipment. Voxel size = 1.0 µm, LaB_6 source = 55 kV, 130 µA. ii) PD as reported in [1], using monochromatic Mo-Kα_1 radiation (λ = 0.7093 Å). All analyses were taken in the same capillary.

Results

1. X-ray micro-computed tomography

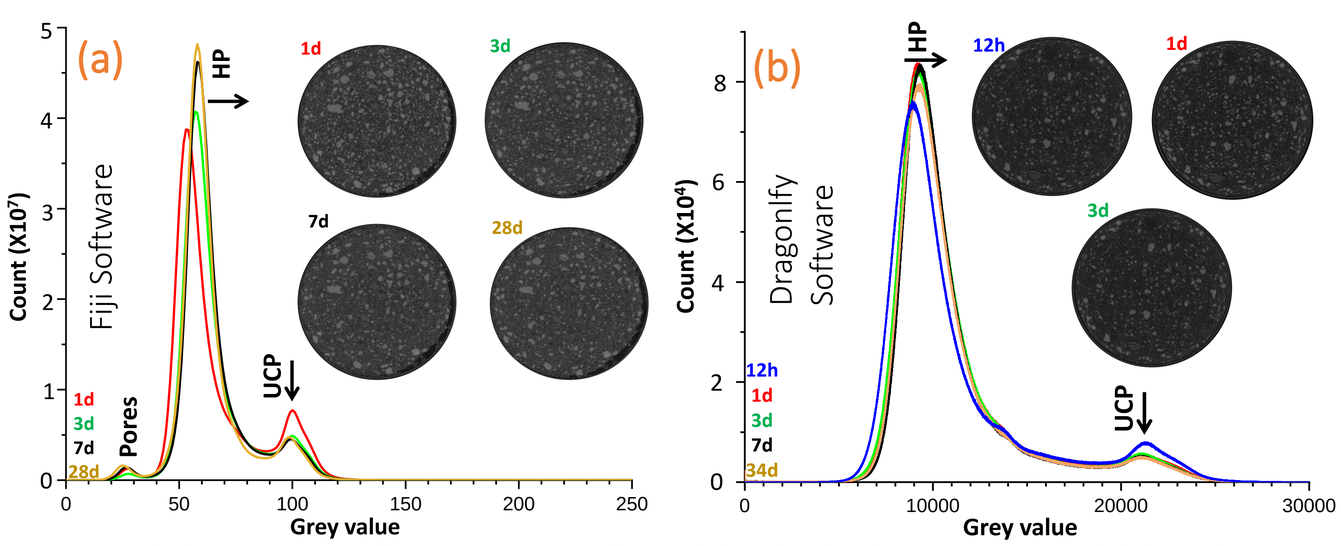

Fig. 2. Greyscale histograms of tomograms collected for the hydrating pastes of (a) PC (w/c=0.40) 8-bits images with 3x3x3 median 3D filter applied and (b) PC-MK-G (w/b=0.50) 16-bits images. It can be seen the dissolution (reaction) of UCP mainly from 12 h/1 day to 3 days. Additionally, densification can be seen in the HP contribution because the shift of the maximum at 12 h/1 day to larger greyscale values at 3 days and onwards.

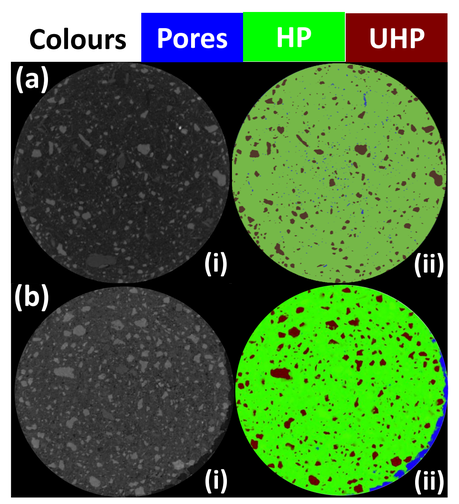

Figure 3. Laboratory µCT orthoslices for (a) PC-MK-GY at 1 day and (b) PC at 28 days of hydration. Both have been segmented by manual thresholding. The glass capillary is left out for clarity. (i) raw (reconstructed) data and (ii) segmented volume for the same view. Thresholds for PC-MK-G; Pores: 0-6900, HP: 6900-16700, UHP:16700-30000.

2. X-ray powder diffraction

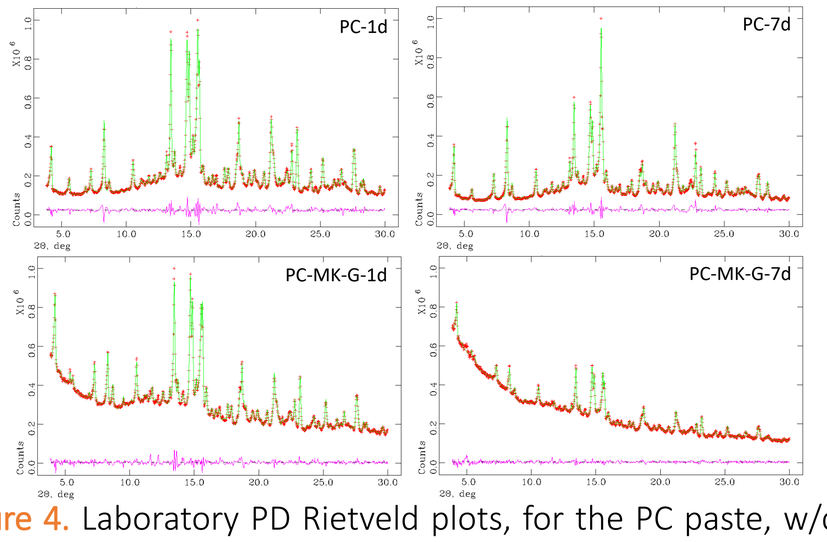

Figure 4. Laboratory PD Rietveld plots, for the PC paste, w/c=0.40 and PC-MK-G w/b=0.50, hydrated at 20 °C within a capillary of ϕ=1.0 mm. The hydration ages are given in the panels.

Table 1. Comparison of the RQPA analysis results, grouped in the two sets of components, with those derived from µCT.

HP: Hydration product.
UHP: Unhydrated cement products.

	RQPA		µCT*	
	HP (vol%)	UHP (vol%)	rHP (vol%)	rUHP (vol%)
PC-1d	73.5	26.6	83.7	16.3
PC-3d	79.0	21.0	88.0	12.0
PC-7d	80.2	19.8	89.2	10.8
PC-28d	81.2	18.8	89.5	10.5

*Thresholds for PC; Pores: 0-35, HP: 36-85 and UHP: 86-255.

References

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

Acknowledgements

This research has been supported by PID2020-114650RB-I00 and PID2019-104378RJ-I00 (Spanish Science Ministry) research grants, which are co-funded by ERDF.

Conclusion

A methodology for the evolution of cement hydration without interferences and finally the pozzolanic reaction for PC-MK-G, including the amorphous phases, has been followed by combined laboratory X-ray µCT and PD analyses at the same capillary in the same position.