

Phase and microstructure evolutions in LC³ binders by multi-technique approach including synchrotron microtomography

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This PDF file includes:

→ **Raw data availability and description.**

→ **#10 supplementary Figures:**

Figure S1. Rietveld plot, Mo-K α_1 strictly monochromatic radiation ($\lambda=0.71$ Å), for the raw FC35 clay resulting in a phase composition: kaolinite: 85 wt%, quartz: 3 wt%, and muscovite 12 wt%. Some additional diffraction peaks were not accounted for, and hence the kaolinite content is likely slightly lower.

Figure S2. Thermal analysis traces (green – weight loss; blue – derivative of the weight loss; and brown – heat flow) for the raw FC35 clay showing the weight loss in the key kaolinite range. From this loss (400-700°C temperature range), a kaolinite content of 72.5 wt% is derived.

Figure S3. Laboratory X-ray powder diffraction pattern, Cu K α_1 radiation, ($\lambda=1.54$ Å), for the FC35 clay calcined at 860 °C in a brick-fabrication industrial oven. The sample contains muscovite and quartz. Kaolinite is no longer present and metakaolin is evident as a scattering bump centered at 25 ° (2 θ).

Figure S4. Heat flow traces for PC-FC35-Cc system with increasing amounts of additional gypsum. From the curves, it is inferred that 3 wt% gypsum additional dosage is suitable to have the second calorimetry peak (C₃A-due) well after the first (alite-due) peak.

Figure S5. Rietveld plots, Mo-K α_1 strictly monochromatic radiation ($\lambda=0.71$ Å), for the PC-FC35-Cc-G pastes at the indicated hydration ages. The patterns also contain the quartz diffraction peaks, used as an internal standard to determine the overall amount of amorphous phase.

Figure S6. Rietveld plots, Mo-K α_1 strictly monochromatic radiation ($\lambda=0.71$ Å), for the PC-MK-G pastes at the indicated hydration ages. The patterns also contain the quartz diffraction peaks, used as an internal standard to determine the overall amount of amorphous phase.

Figure S7. Thermal analysis traces (green – weight loss; blue – derivative of the weight loss; and brown – heat flow) for the PC-FC35-Cc-G pastes at the studied hydration ages: (a) 2 days, (b) 7 days, (c) 28 days and (d) 60 days.

Figure S8. Thermal analysis traces (green – weight loss; blue – derivative of the weight loss; and brown – heat flow) for the PC-MK-G pastes at the studied hydration ages: (a) 2 days, (b) 7 days and (c) 28 days.

Figure S9. Selected BSE-SEM microphotograph for PC-FC35-Cc-G paste after 28 days of hydration.

Figure S10. Top row: selected views of the Paganin reconstructed orthoslices for: (a) PC-FC35-Cc-G-7d, (b) PC-FC35-Cc-G-2d and (c) PC-FC35-Cc-G-60d. Calcite particles are highlighted in red. Bottom row: segmentation outputs for the regions highlighted with a yellow squares. Pores (blue, $gsv \leq 16,000$), HP (green, $16,000 < gsv \leq 28,500$), Calcite (pink, $28,500 < gsv \leq 33,000$), and UHP (brown, $33,000 < gsv$); gsv =grey scale value.

• Raw data availability and description

Tomographic reconstructed raw data (6 tomograms) are available from the authors upon request. Interested researchers in the synchrotron tomographic reconstructed data should be aware of the size of these files which are given in the following table. A hard drive could be necessary.

File Name	Zipped File Size* (by 7Zip app.)	Additional information
33_PC-FC35-Cc-G-7d (Paganin)	21.8Gb	PC-FC35-Cc-G-Hydration Age
33_PC-FC35-Cc-G-7d (Absorption)	21.8Gb	
15_PC-FC35-Cc-G-28d (Paganin)	21.6Gb	
15_PC-FC35-Cc-G-28d (Absorption)	21.5Gb	
30_PC-FC35-Cc-G-60d (Paganin)	21.6Gb	
30_PC-FC35-Cc-G-60d (Absorption)	21.5Gb	
Total size of all datasets :		129.8Gb

**Original file size for every single dataset is 27.4GB*

Additionally, the raw data for calorimetry, MIP, LXRPD, NMR and PSD are uploaded on Zenodo in text file or excel format. The thermal analysis files are also on Zenodo and can be open using the TA Universal Analysis software. Data can be accessed at: <https://doi.org/10.5281/zenodo.4473084>.

• Figures

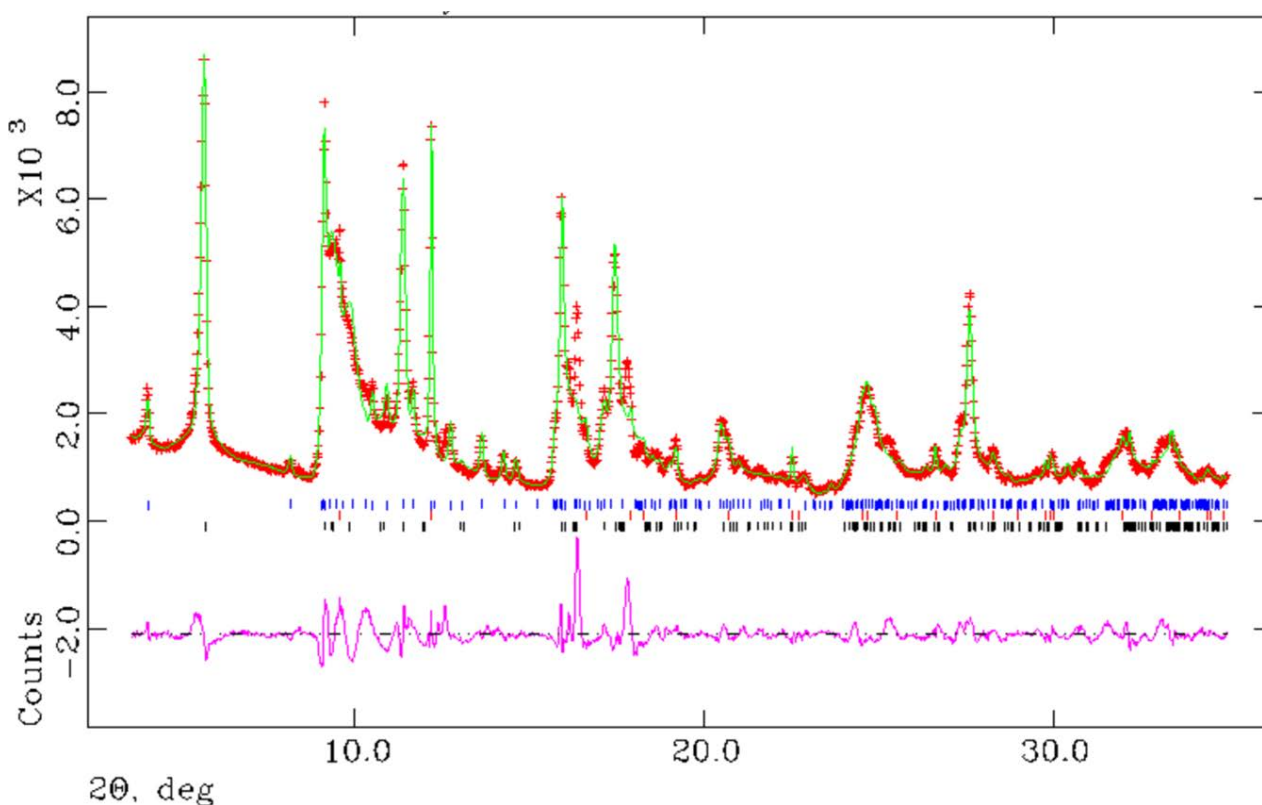


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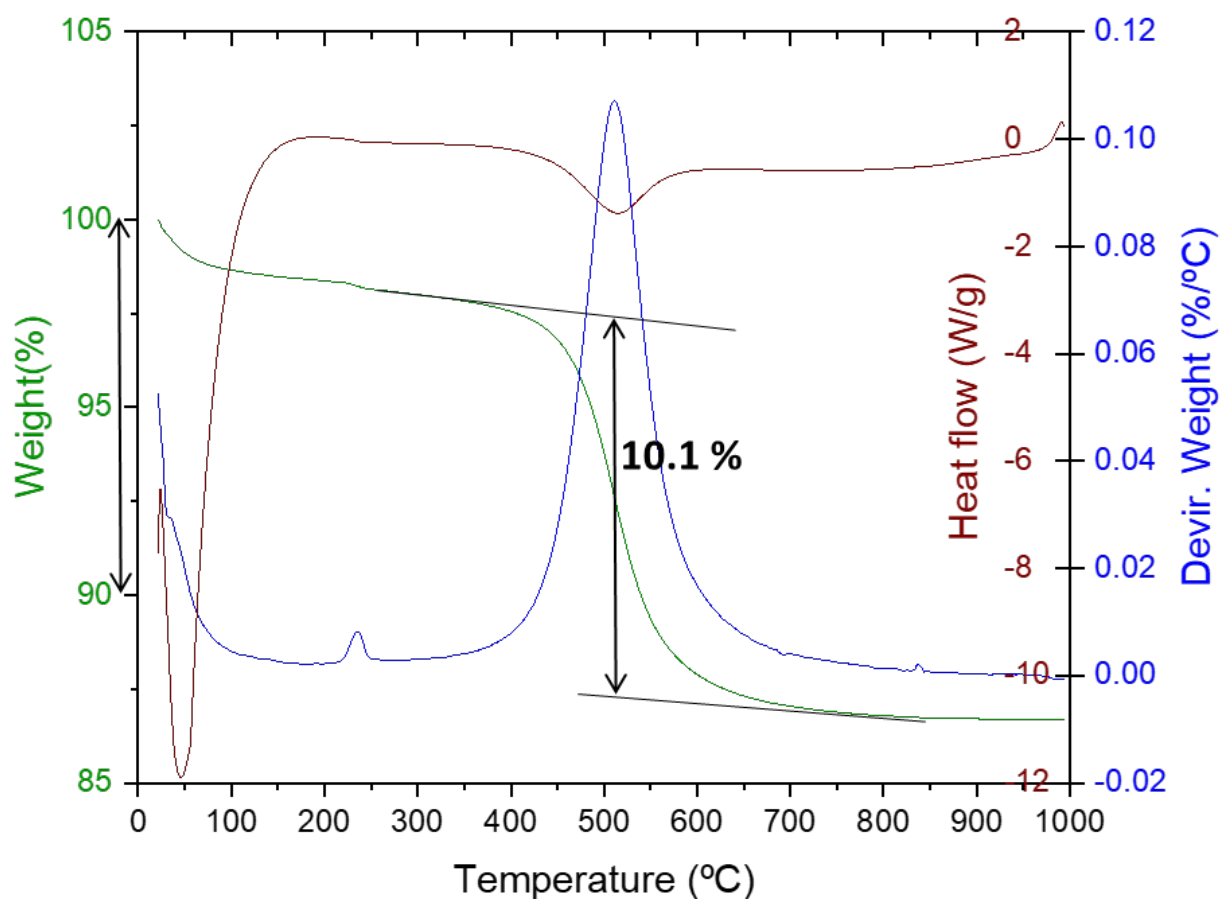


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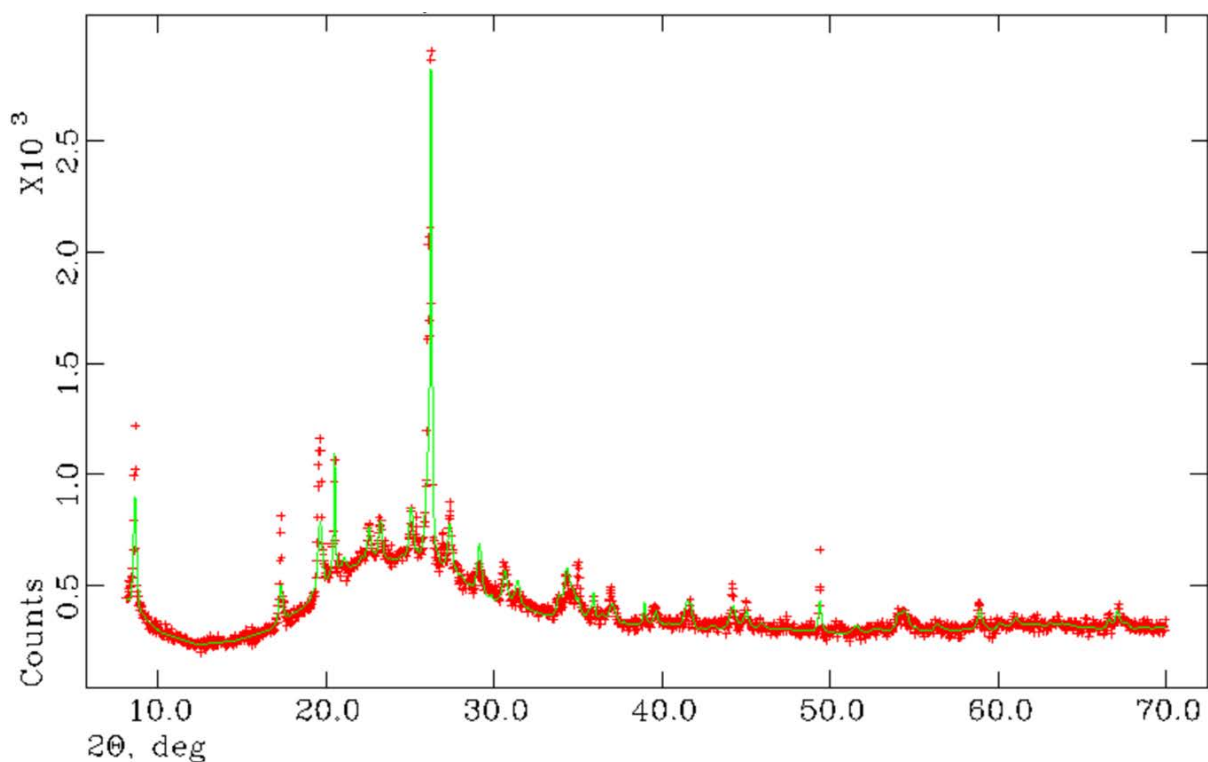


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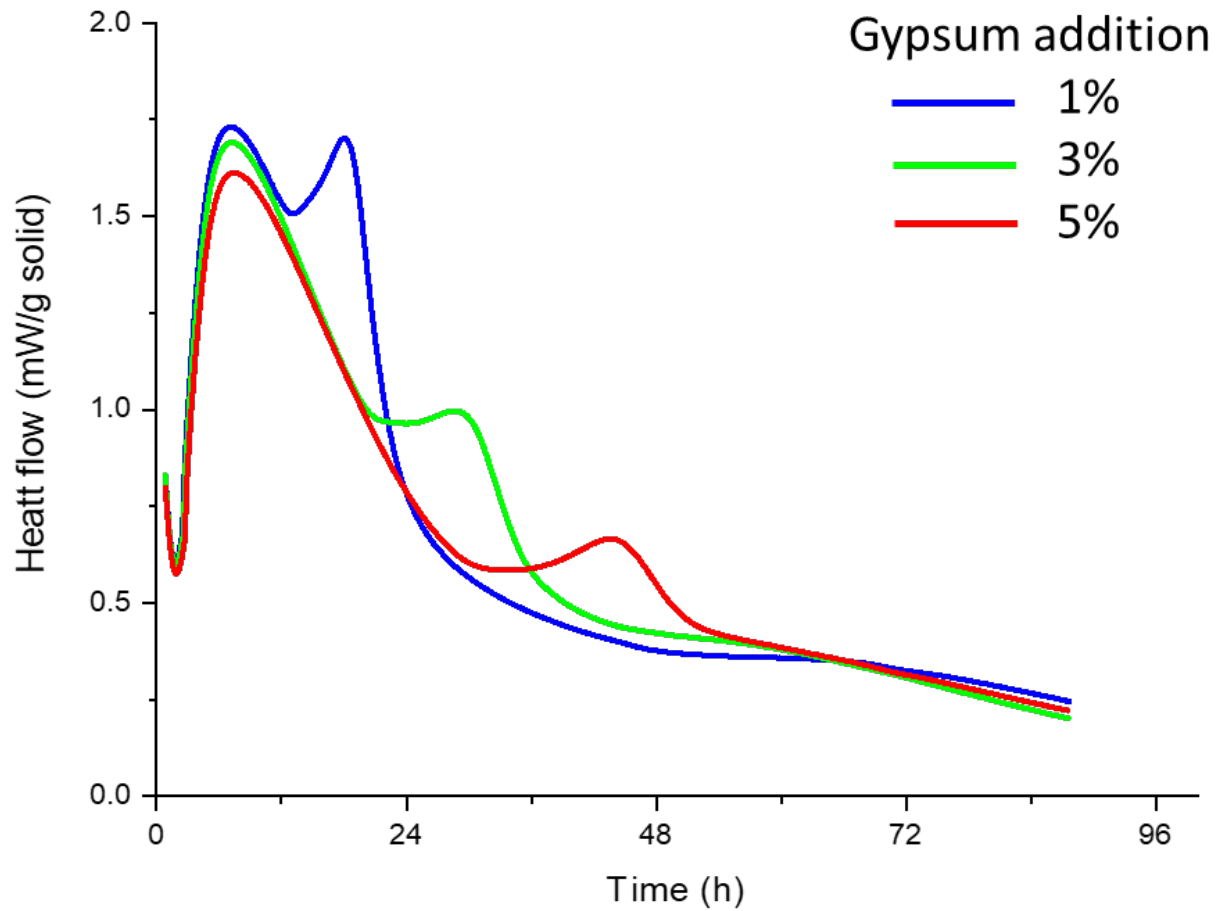


Figure S4. Heat flow traces for PC-FC35-Cc system with increasing amounts of additional gypsum. From the curves, it is inferred that 3 wt% gypsum additional dosage is suitable to have the second calorimetry peak (C_3A -due) well after the first (alite-due) peak.

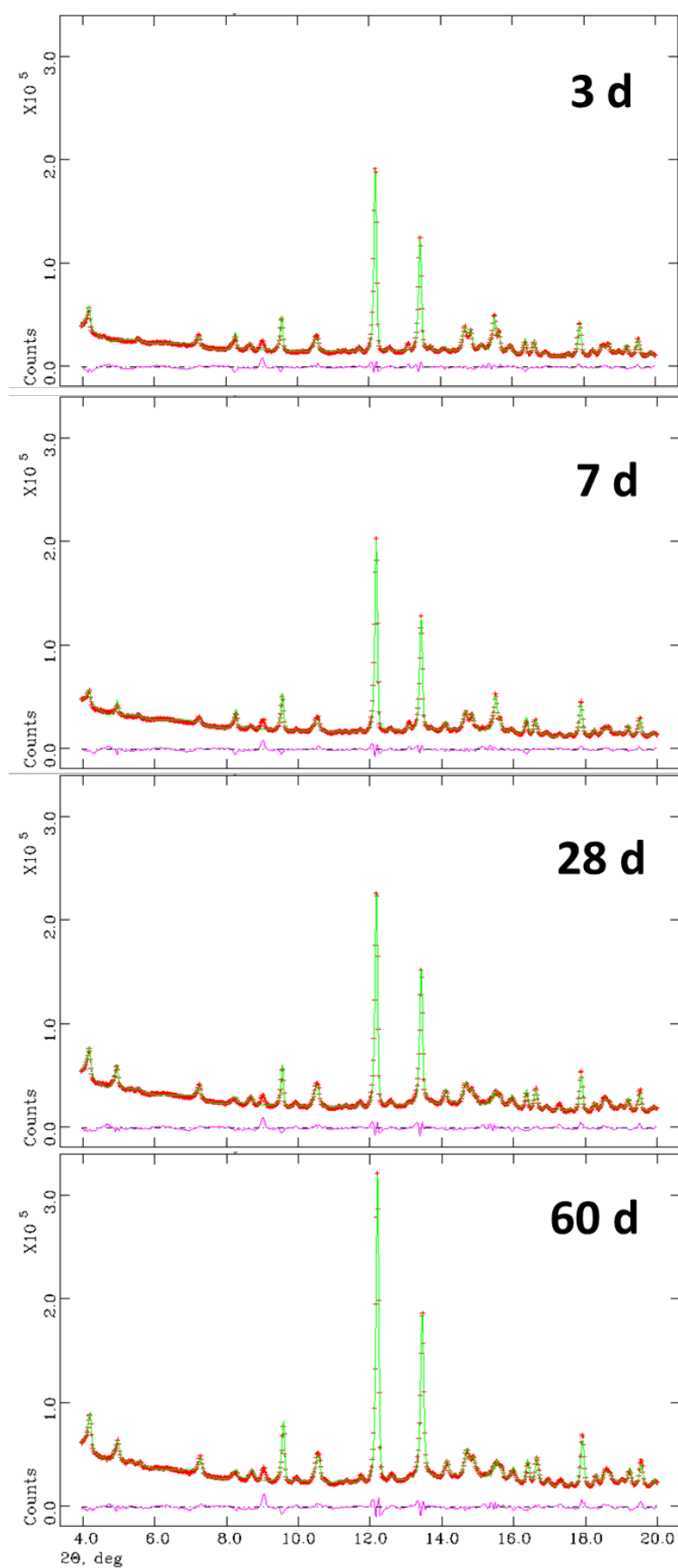


Figure S5. Rietveld plots, Mo-K α_1 strictly monochromatic radiation ($\lambda=0.71$ Å), for the PC-FC35-Cc-G pastes at the indicated hydration ages. The patterns also contain the quartz diffraction peaks, used as internal standard to determine the overall amount of amorphous phase.

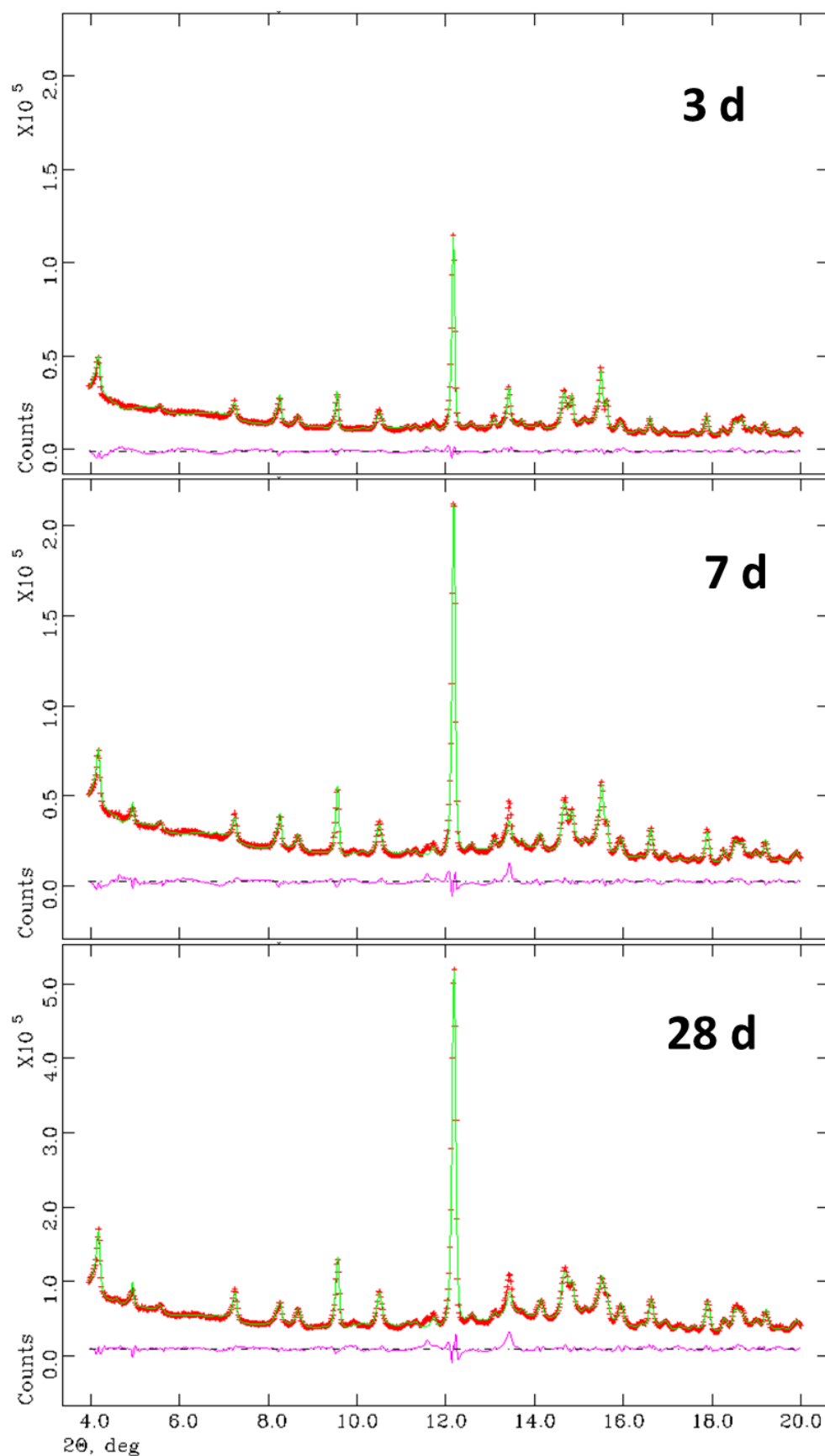
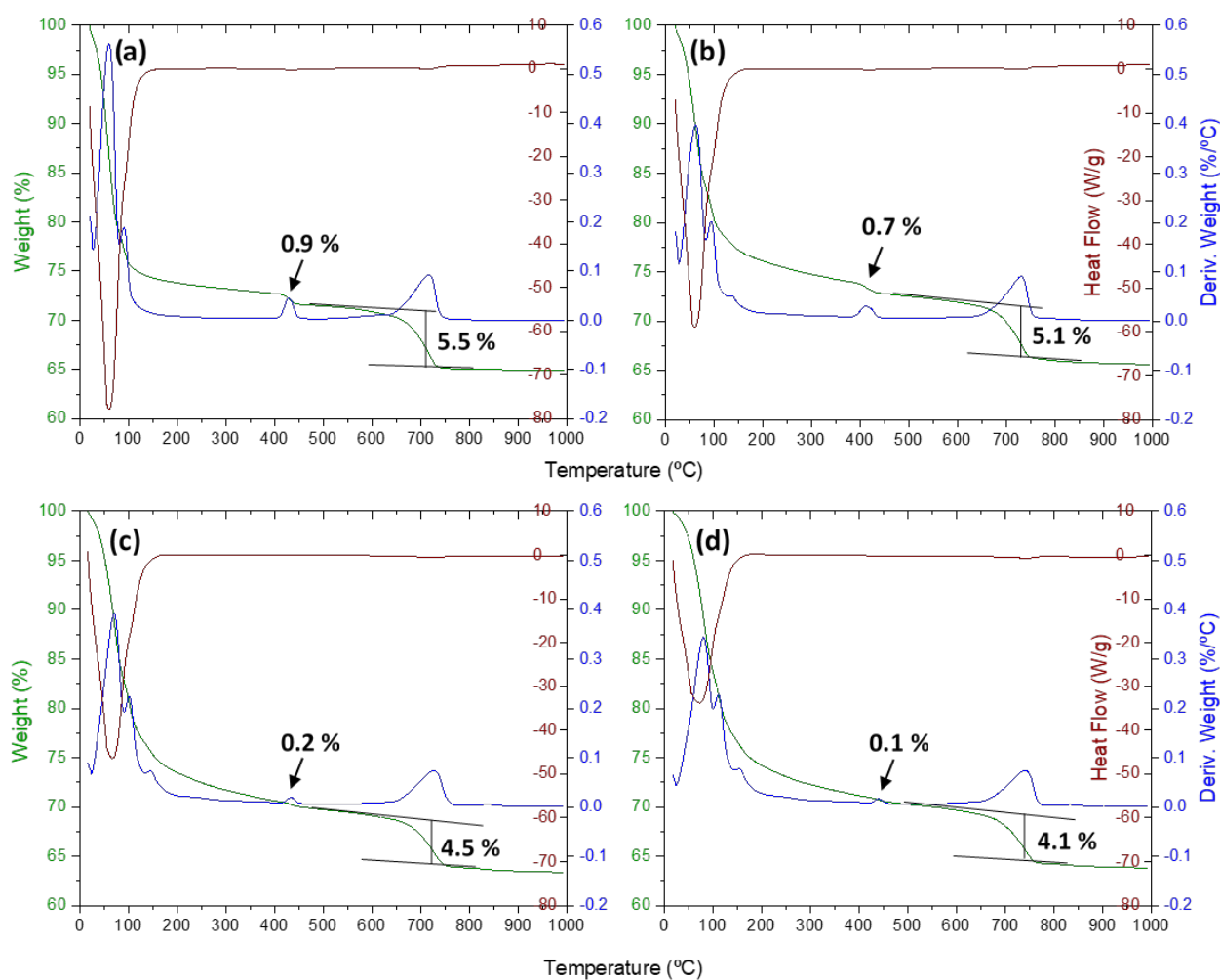
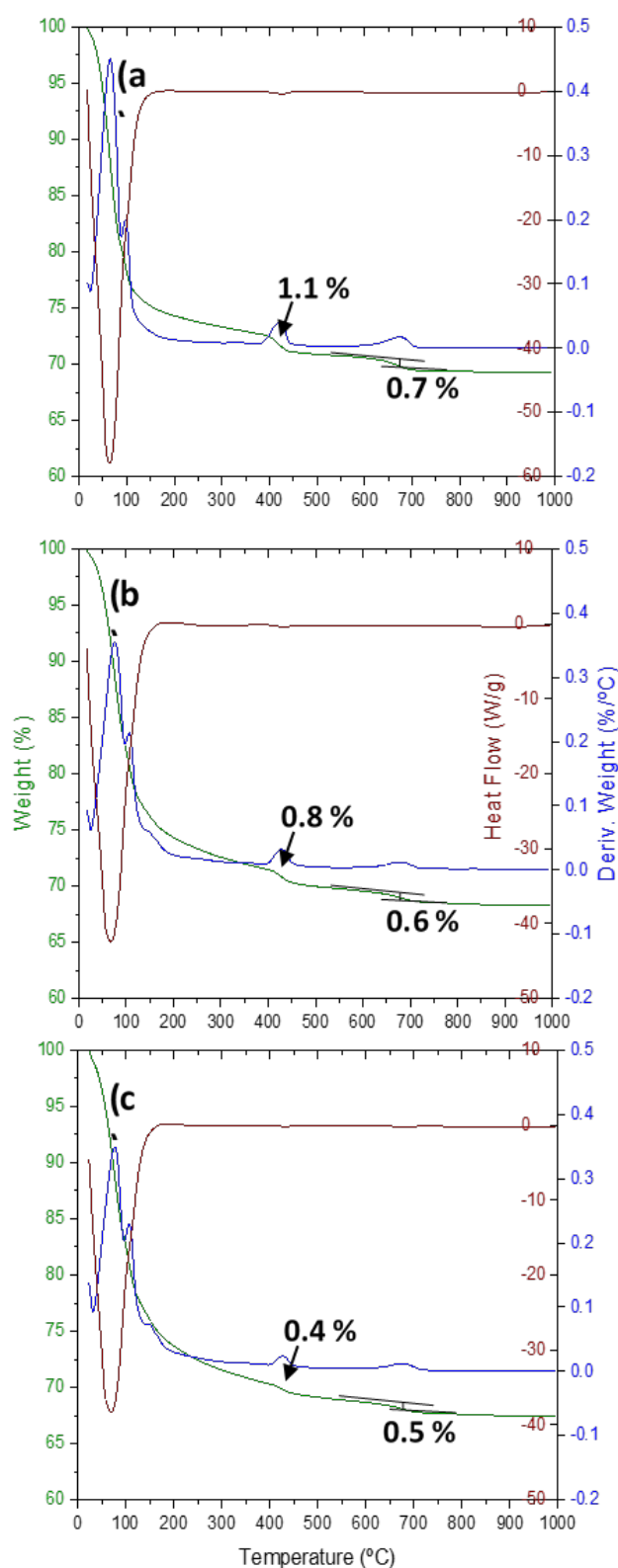


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PC-FC35-Cc-G	H ₂ O w.l. (%) / CH wt%	CO ₂ w.l. (%) / Cc wt%
(a) 2d	0.9 / 3.7	5.5 / 12.5
(b) 7d	0.7 / 3.0	5.1 / 11.6
(c) 28d	0.2 / 0.8	4.5 / 10.2
(d) 60d	0.1 / 0.3	4.1 / 9.3

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PC-MK-G	H ₂ O w.l. (%) / CH wt%	CO ₂ w.l.(%) / Cc wt%
(a) 2d	1.1 / 4.3	0.7 / 1.7
(b) 7d	0.8 / 3.3	0.6 / 1.4
(c) 28d	0.4 / 1.7	0.5 / 1.2

Figure S8. Thermal analysis traces (green – weight loss; blue – derivative of the weight loss; and brown – heat flow) for the PC-MK-G pastes at the studied hydration ages: (a) 2 days, (b) 7 days and (c) 28 days.

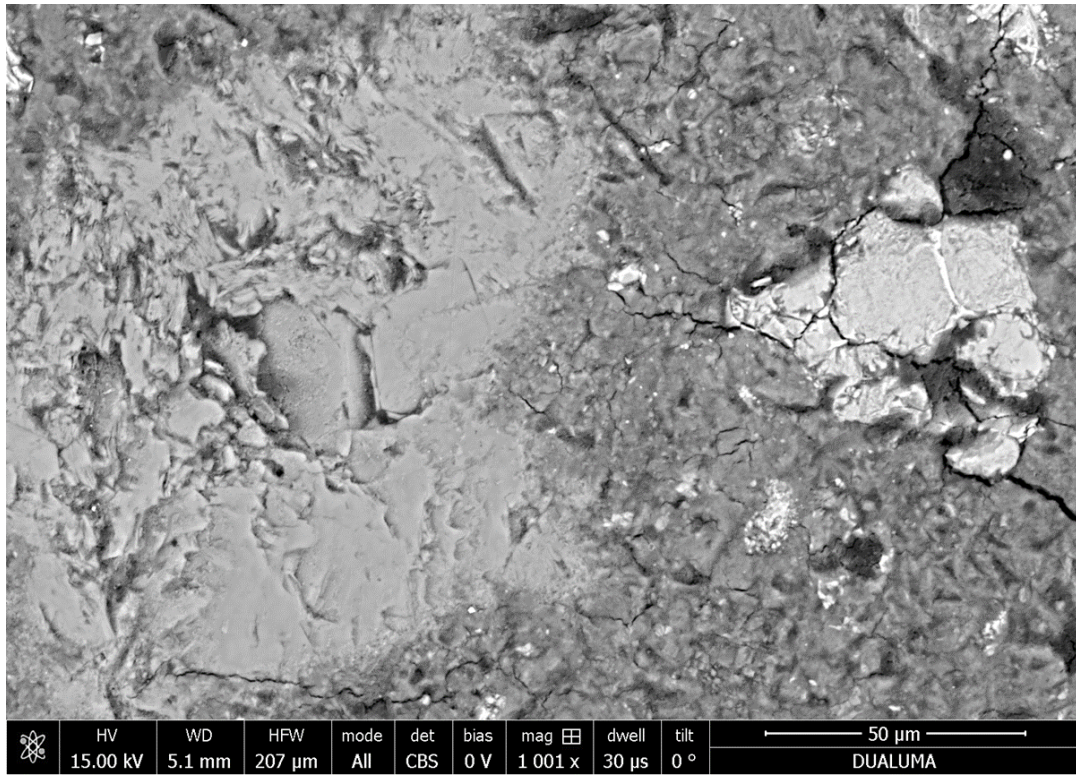


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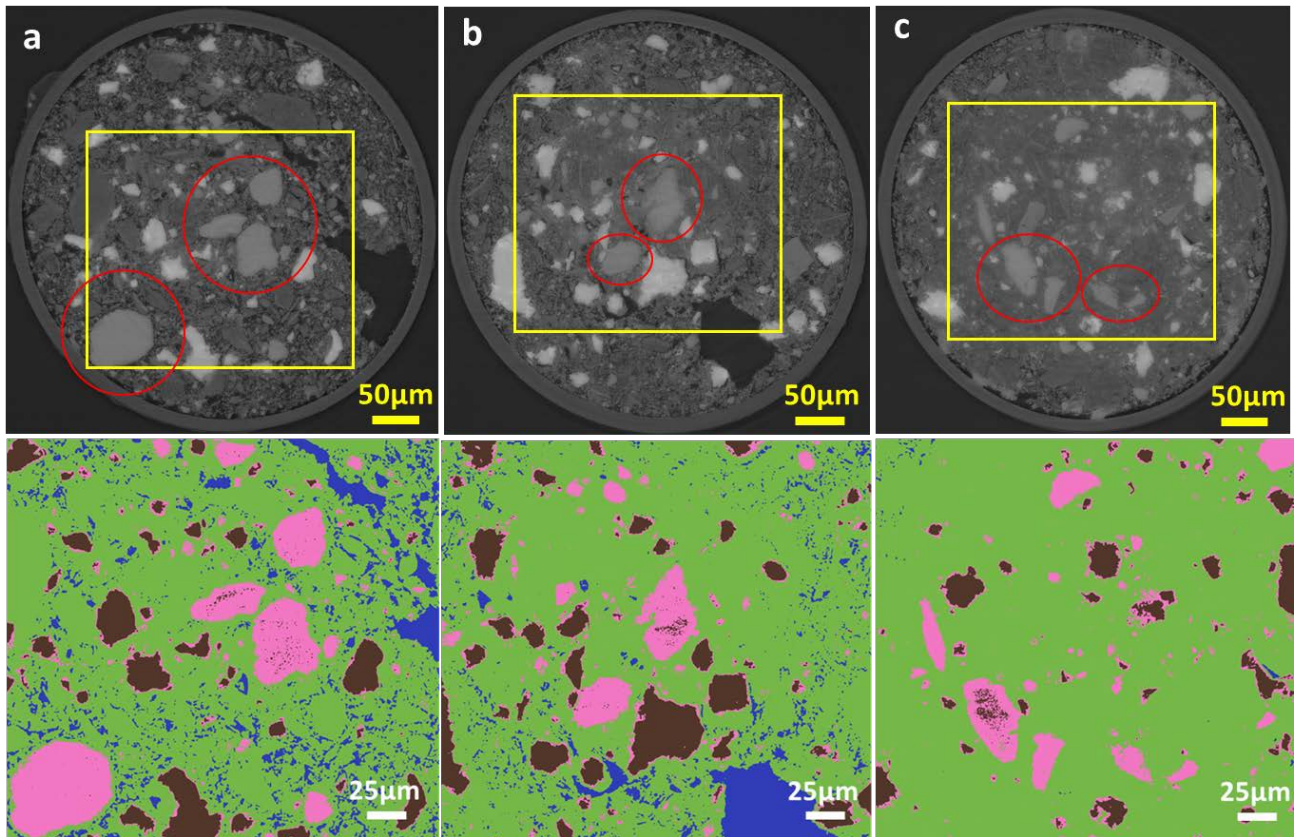


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