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In-situ early age hydration of cement-based materials by synchrotron X-ray powder diffraction

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Cement based binders are building materials of worldwide importance. Since these samples are very complex, the knowledge/control of their mineralogical composition are essential to design and predict materials with specific/improved performance [1]. Rietveld quantitative phase analysis (RQPA) allows the quantification of crystalline phases and, when combined with specific methodologies, as the addition of an internal standard or the external standard approach (G-factor), amorphous and non-crystalline phases can also be quantified. However, to carry out a proper RQPA in hydrated cementitious materials, a good powder diffraction pattern is necessary. In this work, synchrotron X-ray powder diffraction (SXRPD) has been used, allowing in-situ measurements during the early-age hydration process.

This work studies the early hydration of cement-based materials. The analised samples were: a laboratory-prepared belite calcium sulphoaluminate (BCSAF) clinker (non-active) [2] mixed with 10wt% gypsum, labelled G10B0; two active laboratory-prepared BCSAF clinkers (activated with 2wt% borax) [2], one mixed with 10wt% gypsum and the other one with 10wt% monoclinic-bassanite, hereafter named G10B2 and B10B2, respectively; and an environmentally-friendly cement sample from Henkel, composed of bassanite mixed with 15wt% Portland cement and 10wt% Metakaolin, labelled H1.

Anhydrous G10B0 contains beta-belite and orthorhombic-ye'elimite as main phases, while alpha'H-belite and pseudo-cubic-ye'elimite are stabilized in G10B2 and B10B2. Anhydrous H1 contains monoclinic and hexagonal bassanite and alite as main phases.

Ye'elimite, in G10BO pastes, dissolves at a higher pace than in G10B2 (degree of reaction is α~25% and α~10% at 1h, respectively), with the corresponding differences in ettringite crystallisation (degree of precipitation is α~30% and α~5%, respectively).

Moreover, the type of sulphate source has important consequences on the hydration of the active BCSAF cement. Bassanite is quickly dissolved and it precipitates as gypsum within the first hour of hydration (in B10B2) and ettringite starts to crystallize (Figure1). Moreover, after 12hours ettringite is almost fully crystallized, similar to G10B2.

In H1, bassanite transforms into gypsum within the first hour, being the principal hydration product; ettringite starts to be formed just after few hydration minutes (Figure2).

References

[1] H. F. W. Taylor, Cement Chemistry. Telford, London, (1997).

[2] G. Álvarez-Pinazo, A. Cuesta, M. García-Maté, I. Santacruz, E.R. Losilla, A.G. De la Torre, L. León-Reina, M.A.G. Aranda, Cem. Concr. Res. 42 (2012) 960.

Caption (s) - Add figures as attached files (2 fig. max)

Figure 1. Direct Rietveld quantitative phase analysis results (wt%) for B10B2 sample as a function of hydration time. Figure 2. Rietveld plots for anhydrous (top), hydrated after 1.5 hours (middle) and 32.7 hours (bottom) H1 cement. Bassanite, gypsum and ettringite marked with triangle, circle and square, respectively.

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