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Early age cement hydration acceleration followed by in situ synchrotron X-ray powder diffraction

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Portland cement (PC), is one of the most manufactured commodity in the world. Moreover, cement industry is one of the major contributors for greenhouse gases emissions. In the fabrication of one tone of PC clinker, about 0.87 tons CO2 are emitted to our atmosphere. There are two main approaches to decrease the carbon footprint of cements. On the one hand, PC can be party replaced by Supplementary Cementitious Materials (SCMs) with lower CO2 footprint. On the other hand, instead of PC, belite cements (BC) could be used. BCs are similar to Portland cements but the amounts of the two main phases, alite (or C3S, an impure form of Ca3SiO5) and belite (or C2S, an impure form of Ca2SiO4), are reversed. In any case, the main disadvantage of both type of binders is their low mechanical strengths at early ages. They have very good mechanical strength values and durability performances at late ages, after a few weeks.

Therefore, a key research avenue in cement science is to increase the mechanical properties at early ages of low carbon cements. Our research frames within this societal challenge by using accelerator admixtures (chemical products) which activate the hydration of cements at early ages, i.e. during the first 1-3 days.

To do this, an in-situ synchrotron X-ray powder diffraction (SXRPD) set of studies was performed up to 48 hours of hydration using three different PCs with selected accelerating admixtures. For instance, the role of C-S-H gel based admixtures (Master X-Seed 100, Master X-Seed 130 and Master X-Seed STE53) have been studied. Data were collected at the BL04-MSPD beamline at ALBA synchrotron (Barcelona, Spain). It is important to understand the acceleration mechanism of these admixtures to implement them for low carbon cements and to improve their performances. This work is part of our wider investigations dealing with early age hydration acceleration of belite cements and limestone calcined clay cements.

Fig. 1 shows, as an example, the SXRPD patterns for the three studied Portland cements during the key first 13 hours. The evolution (dissolution/disappearance for the anhydrous phases and crystallization/appearance for the hydrated phases) of the key components is highlighted by labelling their most important non-overlapped diffraction peaks. Those phases are named following the cement notation: C3S, C3A (for tricalcium aluminate or Ca3Al2O6), C4AF (for tetracalcium ferro-aluminate Ca4Al2Fe2O10), CH (for portlandite or Ca(OH)2) and AFt (for ettringite or Ca6Al2(SO4)3(OH)12·26H2O). In addition to the reference pastes, i.e. the pastes without accelerating admixtures, the SXRPD patterns for accelerated samples are also displayed. The patterns are being analysed by the Rietveld methodology to determine the phase content evolutions with time and with the added activators.

In this study, we have obtained three main preliminary conclusions. Firstly, the acceleration mechanism seems to strongly depend upon initial sulfate contents and availabilities. Secondly, C-S-H seeding accelerates the hydration of the three cements at early ages, mainly by enhancing calcium sulfate and calcium aluminate dissolutions. It has also been observed that admixtures do very moderately accelerate the hydration of crystalline C3S. Thirdly and quite interestingly, it has been observed a synergistic effect between C-S-H seeding and alkanolamines, when compare to single alkanolamine dosage. Further details will be given in this communication. A fraction of this work has been already submitted for possible publication [1].

[1] Morales-Cantero, A.; Cuesta, A.; De la Torre, A.G.; Santacruz, I.; Mazanec, O.; Borralleras, P.; Weldert, K.S.; Gastaldi, D.; Canonico, F.; Aranda, M.A.G. C-S-H seeding activation of Portland and Belite Cements: an enlightening in situ synchrotron powder diffraction study. (2022) submitted.

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