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

M. García-Maté1,2, G. Álvarez-Pinazo1, L. León-Reina2, A.G. De la Torre1,3, I. Santacruz1, M.A.G. Aranda1,3, K.W. Chou3,4, U. Neuhausen3, S. Petrash4

1 Departamento de Química Inorgánica, Cristalografía y Mineralogía, Universidad de Málaga, 29071 Málaga
2 Servicios Centrales de Investigación, Universidad de Málaga, 29071 Málaga
3 CELLS-ALBA synchrotron radiation facility, Ctra. BP1413 km 3.3, 08290 Cerdanyola del Vallès, Barcelona
4 Henkel Adhesive Technologies

E-mail: martagmate@uma.es

**ABSTRACT**

Cement based binders are building materials of worldwide importance. Since these samples are very complex, the knowledge and control of their mineralogical composition is essential to design and predict materials with specific/improved performance [1]. Rietveld quantitative phase analysis (ROPA) 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 ROPA 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 deals with the early hydration study of cement-based materials. The studied samples were: a laboratory-prepared belite calcium sulfoaluminate (BCSAF) clinker, (non-active) [2] mixed with 10 wt% gypsum, labelled G10B0; two active laboratory-prepared BCSAF clinkers (activated with 2 wt% borax) [2], one mixed with 10 wt% of gypsum and the other one with 10 wt% of monoclinic bassanite, hereafter named G10B2 and B10B2, respectively; and an environmentally-friendly binder sample from Henkel, composed of calcium sulphate hemihydrate mixed with 15 wt% Portland cement (OPC) and 10 wt% Metakaolin, hereafter named H1.

Cement nomenclature will be used hereafter: I.e., C=CaO, S=SO\textsubscript{3} and B=SiO\textsubscript{2}.

**RESULTS & DISCUSSION**

1\textsuperscript{st} Important difference in the hydration process:

Degree of reaction after 1 hour: G10B0 ($\approx$25%); G10B2 ($\approx$10%)

Gypsum is completely dissolved; G10B0 = 5 hours; G10B2 = 11 hours

Ye'elimite is completely dissolved; G10B0 = 26 hours; G10B2 = 51 hours (remains)

AFt crystallization after 1 hour: G10B0 = 14.2(2) wt% (\approx$\%$); G10B2 = 1.9(1) wt% (\approx$\%$)

2\textsuperscript{nd} Important difference in the hydration process:

- $\text{g-CaS}$ and $\text{CaAF}$ start to be dissolved in G10B0 after 1 day $\alpha$ crystallization of AFm type phases [2], including strätlingite.

- On the other hand, for G10B2, $\alpha_{\text{g-CaS}}$ percentage remains constant up to 51 hours of hydration and CaAF dissolves very slowly after 14 h.

- $\text{g-CaS}$ reacts faster than $\alpha_{\text{g-CaS}}$

Hydration behavior of belite is more dependent on the chemical environment (higher $\text{AH}_3$ content) than on its polymorphism. Furthermore, slower hydration G10B2 led to much higher mechanical strength developments.

**REFERENCES**