

## “Hydration studies of ye’elimite by using Ptychographic X-ray nanotomography”

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CSA (Calcium SulfoAluminate) cements may have variable compositions but all of them contain ye’elimite( $\text{Ca}_4\text{Al}_6\text{O}_{12}\text{SO}_4$ ). The manufacture of CSA cements is more environmentally friendly than that of ordinary Portland cements as their production releases up to 40% less  $\text{CO}_2$ . The hydration of ye’elimite leads to crystalline ettringite (AFt) and amorphous aluminum hydroxide ( $\text{AH}_3 \cdot n\text{H}_2\text{O}$ ).

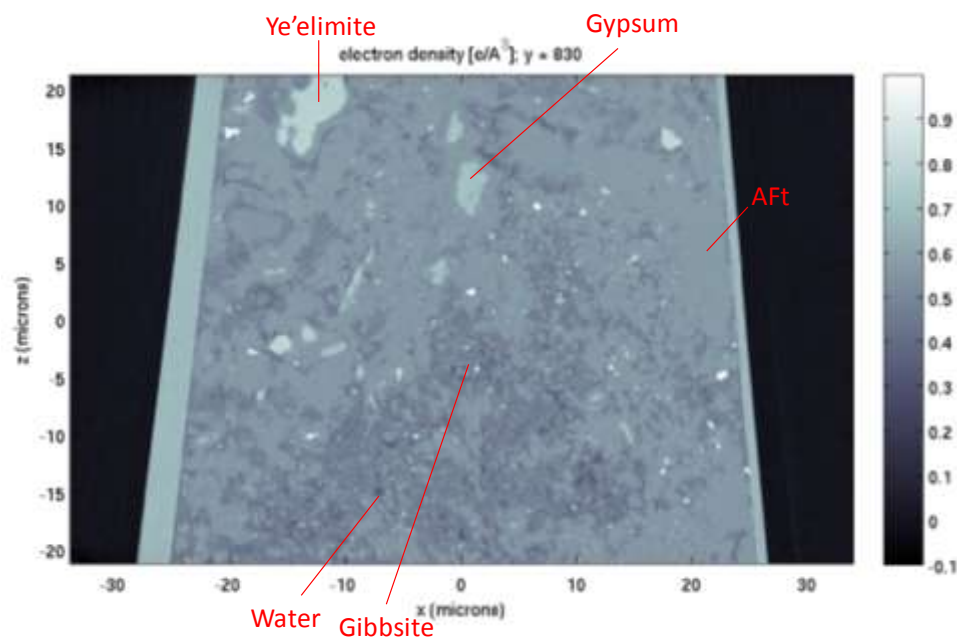
Ptychographic X-ray computed nanotomography (PXCT) has been used here to study the hydration of ye’elimite-containing samples. PXCT is an X-ray imaging technique having demonstrated an isotropic 3D resolution better than 20 nm[1]. PXCT, which nondestructively provides 3D images of the sample complex-valued X-ray refractive index, has been recently applied for hydration studies of Portland cement samples[2].

Samples for this study were measured in cSAXS beamline (Swiss Light Source). The main goal of this study has been the quantification of the electron and mass densities of the phases present in these samples. These mass densities were compared with the theoretical values in order to identify the phases and matched well the expected values.

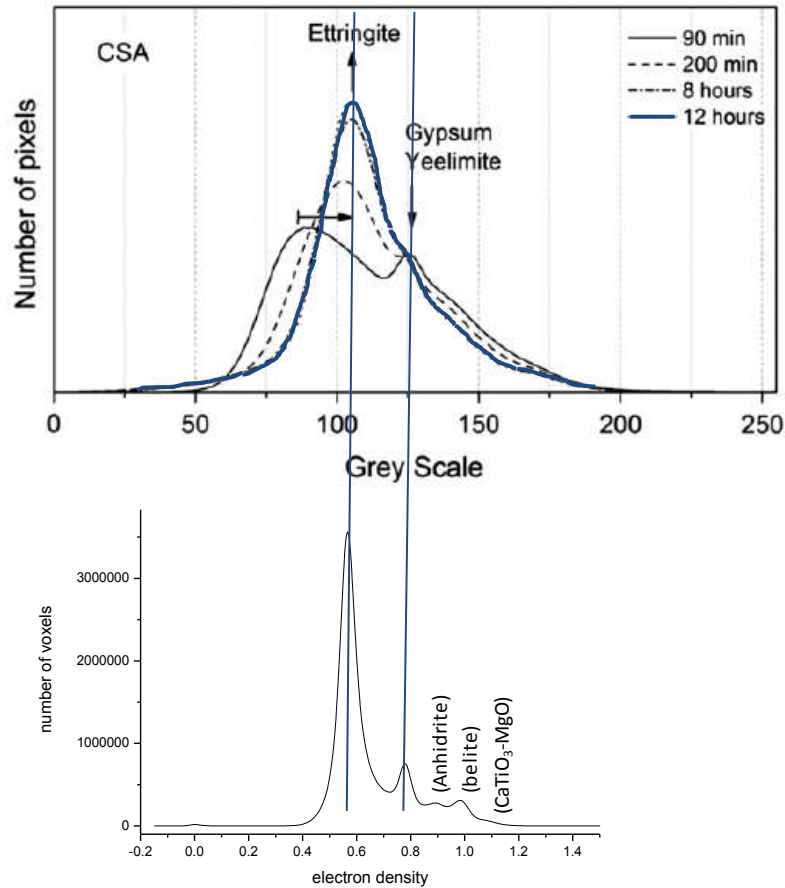
For instance, the hydration of pure ye’elimite with gypsum sample was studied. This sample should show a large amount of AFt due to the presence of gypsum in the hydration medium. This is precisely what it was observed from the analysis of the histogram. Figure 1 shows the tomogram of one slice obtained for this sample.

The most relevant results for CSA cement hydration will be discussed. A volume of interest (VOI) histogram has been studied in order to identify all the phases. Figure 2 shows a comparison between the histogram obtained in this study and a previous one performed by Gastaldi et al. (2012) at TOMCAT Beamline (Swiss Light Source). As it can be observed, TOMCAT data cannot properly distinguish between AFt and ye'elimite phase. Conversely, PXCT technique can resolve the peaks for all the phases present in CSA cements.

All reconstructions have been successful and now we are analyzing the data (segmentation, etc.) to characterize the porosities and the shape and size of the different phase, chiefly ettringite.



**Figure 1.** Tomogram of one slice obtained for ye'elimite with gypsum hydrated sample with the main samples labeled.



**Figure 2.** Bottom: VOI histogram of a calcium sulfoaluminate cement obtained in this experiment. Top: grey level histogram of a CSA cement paste obtained at TOMCAT BL (Figure adapted from Gastaldi et al., *Const. Build. Mat.* 29, 2012 284-290).

## REFERENCES

- [1] M. Holler, A. Diaz, M. Guizar-Sicairos, P. Karvinen, E. Färm, E. Härkönen, M. Ritala, A. Menzel, J. Raabe and O. Bunk, “X-ray Ptychographic Computed Tomography at 16 nm Isotropic 3D Resolution”, *Sci Rep*, 2014, 4, 3857.
- [2] J. C. da Silva, P. Trtik, A. Diaz, M. Holler, M. Guizar-Sicairos, J. Raabe, O. Bunk, and A. Menzel, “Mass Density and Water Content of Saturated Never-Dried Calcium Silicate Hydrates”, *Langmuir*, 2015, 31, 3779.