ESRF	**Experiment title: Time resolved investigations of the initial hydration of Portland cement influenced by organic additives**	**Experiment number**: HC 1456
Beamline: MS Powder	**Date of experiment**: from: 03.10.2014 to: 06.10.2014	**Date of report**: 27.01.2015
Shifts: 9	**Local contact(s)**: Dr. J. Wright	*Received at ESRF:*

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Report:

The hydration of Portland cement is an important issue. The general understanding of the processes proceeding during the cement hydration already exists. In contrast, early cement hydration is the subject of numerous studies. Additionally, various additives are used in the building practice for improving the workability and rheological characteristics of the cement-water mixture. The use of these additives influences the course of cement hydration and results in the retardation of the setting time. A fundamental understanding of the underlying processes is still missing.

The subject of this proposal was following the initial hydration of ordinary Portland cement in-situ. The influence and interaction of selected organic additives, such as polycarboxylate ether (PCE) based superplastisizer (SP) and stabilizer (ST) on the basis of potato starch, was investigated by X-ray diffraction with high time resolution.

The measurement setup consists of ultrasonic double trap combined with automatic injection system for addition of the hydration solutions. A small cement pellet was levitated and hydrated during the experiment. In this way the formation of cement hydrates could be followed in-situ starting from the moment of water/solution addition. The changes of hydration stages can be followed in detail manner by a continuous collection of diffraction patterns. Previous investigations carried out on the µSPOT beamline of Bessy II (Helmholtz-Zentrum Berlin) were insufficient with respect to the time resolution of 60 s/scan. At the MS Powder beamline, the necessary and sufficient time resolution of 1 s/scan was achieved. The early cement hydration could be followed from the initial water injection over a time span of at least 1 hour (Figure 1).

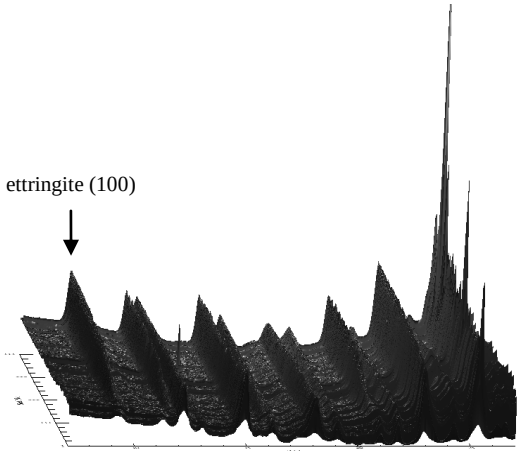

Figure 1 Reference cement hydration followed for 2 hours

The results show a good agreement between the diffraction patterns obtained in double determinations indicating the stability and reliability of the experimental conditions in the double trap. For comparison, the (100) reflection of ettringite was plotted in aside view showing the development of the reflection intensity vs. hydration time. The results give strong interaction of PCE-based SP and ST influencing the course of the cement hydration. The adsorptive competitiveness of the anions (PCE, sulphate) for the adsorption positions on the cement clinker particles and the limitation of their mobility in presence of starch lead to differences in the formation of ettringite during the initial hydration period. Adsorption forces of SP depend among other on the charge density of their backbone. Presence of starch in the cement matrix results in the fast and strong formation of ettringite compared to the systems without starch (Figure 2). The formation and growth of ettringite crystals determine the setting of the cement paste, its supressed formation affects the setting time. The mechanisms of the retardation can be understood in detail based on the results obtained.

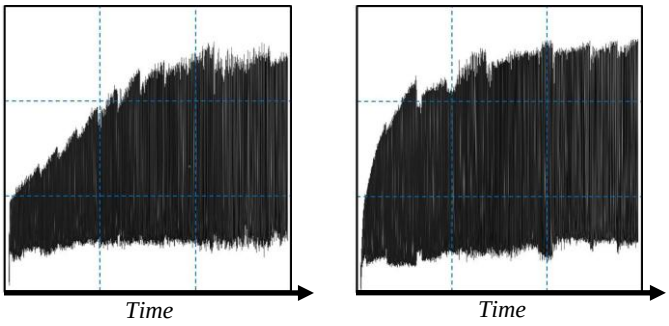

Figure 2 Cement hydration influenced by 1.2 % PCE without starch (left) and with 0.5 % starch (right) and followed for 1 hour.