

**Experiment title:**

Use of energy-dispersive diffraction conical slit geometry to study fast hydration reactions

**Experiment number:**

CH-14

**Beamline:****Date of Experiment:**

ID9-BL3

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**Shifts:****Local contact(s):**

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**Report:**

In this project the energy-dispersive diffractometer facilities on beamline 9 were used to study the kinetics of fast hydration reactions under various conditions of sample containment (temperature, environment etc.) and diffraction geometry. From a number of systems under consideration, the hydration of tricalcium aluminate from ambient conditions emerged as a candidate for detailed study. Tricalcium aluminate, or "C<sub>3</sub>A" for short, is largely responsible for the early hydration of industrial (Portland) cements, i.e. during the first few hours after mixing with water. Various hydration reactions and calcium aluminate hydrates are involved, as indicated below using cement chemistry shorthand (C=CaO; A= A<sub>1</sub>2O<sub>3</sub>; H=H<sub>2</sub>O):



in which the latter calcium aluminate hydrate, C<sub>3</sub>AH<sub>6</sub>, is the most stable hydrate phase, particularly at high temperatures.

These C<sub>3</sub>A-hydration reactions have been studied with a diffractometer 2θ-angle set at 2,2°. The C<sub>3</sub>A and added water were both equilibrated initially to ambient temperatures, though the exothermic reactions and effect of beam-heating inevitably give rise to increased local cement temperatures. A typical result is shown in Fig. 1 showing that the C<sub>3</sub>A hydration

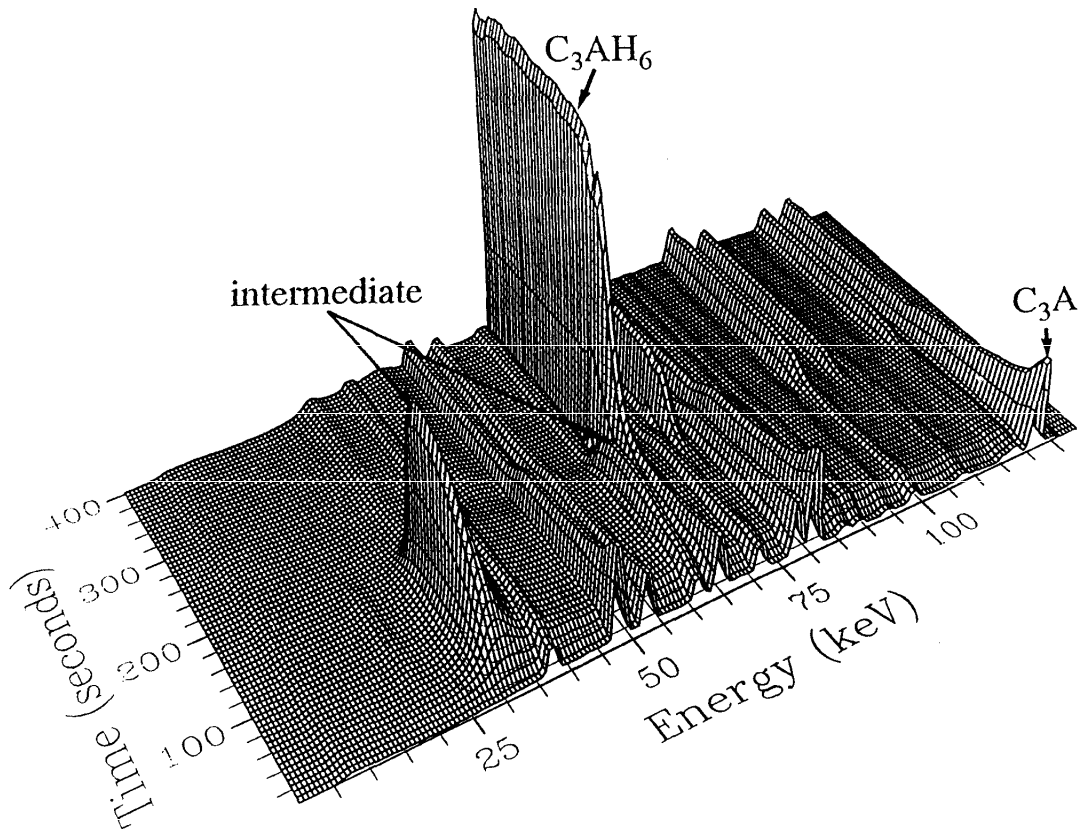


Figure 1: *Time-resolved energy-dispersive diffraction patterns showing a hydration sequence of tricalcium aluminate during the first 400 seconds.*

sequence can be rapid and dramatic: after a brief dormant period of 10-20 seconds, the  $C_3A$  content starts to decrease just as an intermediate phase forms; after 100-200 seconds the intermediate phase disappears very abruptly while a third phase,  $C_3AH_6$  hydrate, then forms and thereafter remains. The intermediate phase is either  $C_2AH_8$  or  $C_4AH_{19}$  though the former is the more likely. These data are being currently interpreted<sup>2</sup> along lines similar to those proposed by Rashid et al.<sup>3</sup> in which the growth of the final stable hydrate,  $C_3AH_6$ , is critically nucleated by the intermediate hydrate. Thus we see that rapid energy-dispersive diffraction is giving new insight into old materials science problems concerning the mechanisms of early hydration of cement. Further studies are planned.

- 1 Barnes, P., Hal I, C., Hanfland, M., Hausermann, D., Lewis, A.C. & Turrillas, X. *Bright New Light On Old Concrete*, ESRF Newsletter 24,5-6 (1995).
- 2 Lewis, A. C., Turrillas, X., Barnes, P., Hal I, C., Hausermann, D. & Hanfland, M., *Throwing intense new light on an old materials problem: a synchrotrons view of the hydration of tricalcium aluminate*", submitted (1995).
- 3 S. Rashid, P. Barnes and X. Turrillas, *Adv. Cement Research* 4,61 (1992).