109-065

IN SITU HYDRATION OF SULPHOALUMINATE CEMENT MIXTURES MONITORED BY SYNCHROTRON X-RAY DIFFRACTION

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The hydration of calcium sulphoaluminate cement mixtures was studied in situ by synchrotron X-ray diffraction at the XRD1 beamline of the Laboratório Nacional de Luz Síncrotron (LNLS – Campinas). The specimens were analyzed in borosilicate glass capillary tubes of 0.7 mm and imbued with deionized water. As the hydration reaction is very fast, the data collection was started after two minutes of mixing with water. The X-ray wavelength chosen to get an adequate flux for these short time acquisitions was 1.033258 Å, determined with a corundum standard. Diffraction patterns were collected every 35 seconds at temperatures ranging from 40°C to 55°C with accuracy better than 0.1°C attained with a hot air blower. The diffracted signal was collected with an array of 24 Mythen detectors. The diffraction patterns accumulated had appropriate statistics to determine the kinetics of the reaction either by quantitative Rietveld analysis or by fitting isolated diffraction peaks to Gaussian curves as a function of time. The most important phases involved in the hydration are Klein´s salt, also known as Ye'elimite, Ca4(AlO2)6SO4, and gypsum, CaSO4.2H2O to yield Ettringite, Ca6Al2(SO4)3(OH)12·26H2O, phase responsible for the mechanical properties. These studies show the potential of XRD1 beamline to investigate at controlled temperatures in situ fast reactions involving crystalline phases with time resolutions inferior to one minute, which is ideal for the hydration of cementitious mixtures.