THE USE OF MO AND CU MONOCHROMATIC RADIATIONS FOR QUANTITATIVE PHASE ANALYSIS: STUDY OF THE ACCURACY

A. G. De la Torre[a], L. León-Reina[b], A. Cuesta[c], M. García-Maté[d], G. Álvarez-Pinazo[d], I. Santacruz[a], O. Vallcorba[c], M. A. G. Aranda[a,c]

[a] Departamento de Química Inorgánica, Cristalografía y Mineralogía, Universidad de Málaga, 29071-Málaga, Spain. [b]Servicios Centrales de Apoyo a la Investigación, Universidad de Málaga, 29071-Málaga, Spain. [c]ALBA synchrotron, Carrer de la Lum, 2-26, E-08290 Cerdanyola del Vallès, Barcelona, Spain. [d] X-Ray Data Services S.L., Edificio GREEN RAY, 1ª planta, Avda. Louis Pasteur, 47 (Ampliación Campus Teatinos), 29010-Málaga, Spain.
mgd@uma.es

Cement hydration is a very complex process in which crystalline phases are dissolving in water and after supersaturation hydrated crystalline and amorphous phases precipitate. Great efforts are being made to develop analytical tools to accurately quantify these processes and X-ray Powder Diffraction (XRPD) combined with Rietveld methodology is a suitable tool to quantify these complex mixtures and their time evolutions. However, some problems/drawbacks should be overcome to fully apply it to cement pastes characterization in order to get accurate phase analyses. In order to tackle this issue, a comparison of the Rietveld quantitative phase analyses (RQPA) obtained using Cu-Kα_1, Mo-Kα_1, and synchrotron strictly monochromatic radiations of three set of mixtures with increasing amounts of a given phase (spiking-method) is presented. The main aim is to test a simple hypothesis: high energy Mo-radiation, combined with high resolution laboratory X-ray powder diffraction optics, could yield more accurate RQPA, for challenging samples, than well-established Cu-radiation procedure(s). Firstly, a series of crystalline inorganic phase mixtures with increasing amounts of an analyte was studied in order to determine if Mo-Kα_1 methodology is as robust as the well-established Cu-Kα_1 one. Secondly, a series of crystalline organic phase mixtures with increasing amounts of an organic compound was analyzed. This type of mixture can result in transparency problems in reflection and inhomogeneous loading in narrow capillaries for transmission studies. Finally, a third series with variable amorphous content was studied. Limit of detection in Cu-patterns, ~0.2 wt%, are slightly lower than those derived from Mo-patterns, ~0.3 wt%, for similar recording times and limit of quantification for a well crystallized inorganic phase using laboratory powder diffraction was established ~0.10 wt%. From the obtained results it is inferred that RQPA from Mo-Kα_1 radiation have slightly better accuracies than those obtained from Cu-Kα_1.
The results obtained in the previous comparison have been taken into account to obtain accurate RQPA, including the amorphous component with internal standard methodology, of hydrating cement pastes. The final goal of this second study was understanding the early-stage hydration mechanisms of a variety of cementing systems (Ordinary Portland Cement or Belite Alite Ye'elimite cement) as a function of water content, superplasticizer additives and type and content of sulfate source. In order to do so, X-ray powder diffraction data were taken in-situ with the humidity chamber coupled to the Mo-Kα_1 powder diffractometer. Some results of this ongoing investigation will be reported and discussed.