Synchrotron pair distribution function approach applied to cement samples

A. Cuesta, J.D. Zea-Garcia, A.G. De la Torre and M.A.G. Aranda

Departamento de Química Inorgánica, Universidad de Málaga, 29071-Málaga, Spain, a_cuesta@uma.es

The characterization of the nanocrystalline/amorphous materials is very challenging but the presence of these materials mixed with large amounts of crystalline phases makes the analysis even more complicated. This is the case of cement hydrated samples, since they contain high amounts of crystalline phases jointly with nanocrystalline and amorphous components, making challenging their full characterization. High-energy synchrotron X-ray scattering jointly with the pair distribution function (PDF) methodology is very useful to deeply study complex cement pastes. PDF data give information about the local structure (bonding environments) of the nanocrystalline and amorphous component phases such as cement gels.

The main aim of this work is to characterize amorphous/nanocrystalline gels which are present in different cementitious pastes by the PDF approach. Moreover, the PDF technique also gives us quantitatively information about the nanocrystalline and microcrystalline contents.

Firstly, the C-S-H gel obtained from the hydration of alite, Ca_3SiO_5, the main phase of Portland cement, is found to contain two components: a nanocrystalline defective clinotobermorite and an amorphous component which seemed to be monolayers of calcium hydroxide[1]. Figure below shows the PDF fit of a C-S-H gel formed from the hydration of alite in two selected r-ranges. Secondly, some ye'elimite $Ca_4Al_6O_{12}(SO_4)$ -containing pastes with different sulfate contents have been also studied. The nanocrystalline hydrated phases, such as A-H gel and monosulfoaluminate (AFm) phases have been thoroughly analyzed in the different pastes and the diameters of these nanoparticles have also been reported[2]. Finally, we will also show here, PDF data for different cement samples (including the Ni sample as standard) that have been collected in two different experimental configurations, i.e., in ID15A beamline at ESRF and in MSPD beamline at ALBA synchrotron[3].

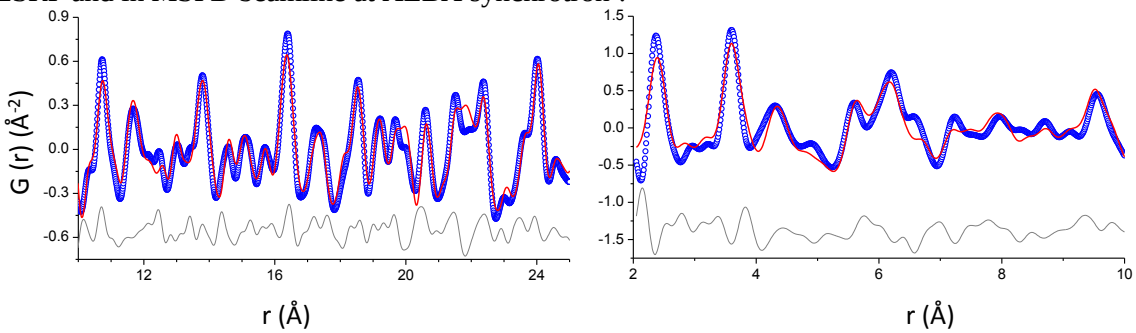

Figure. Experimental (blue circles), fitted (red lines) and difference (grey lines) PDF patterns for an alite paste (left) from 10 to 25 Å and (right) from 2 to 10 Å.

Acknowledgements. This work has been supported by Spanish MinCIU through BIA2014-57658-C2 and BIA2017-82391-R, which are co-funded by FEDER. We also thank CELLS-ALBA (Barcelona, Spain) and ESRF (Grenoble) for providing synchrotron beam time at BL04-MSPD and ID15A, respectively.

[1] A. Cuesta, et al. Multiscale understanding of tricalcium silicate hydration reactions. *Scientific Reports* **2018**, *8,* 8544.

[2] A. Cuesta, J.D. Zea-Garcia, A.G. De la Torre, I. Santacruz, M.A.G. Aranda. Synchrotron pair distribution function analyses of ye'elimite-based pastes. *Adv. Cem. Res.* **2019**, *31,* 138-146.

[3] J.D. Zea-Garcia, A.G. De la Torre, M.A.G. Aranda, A. Cuesta. A comparative study of experimental configurations in synchrotron pair distribution function. *Materials* **2019**, *12,* 1347.