The new Debye beamline at SLS2.0: a versatile *operando* platform for X-ray scattering and X-ray absorption spectroscopy

D. Bonavia[1], A. H. Clark[1,*], S. Hitz[1], M. Nachtegaal[1]

[1]*Photon Science Division, Paul Scherrer Institute, Forschungsstrasse 111, Villigen PSI, 5232, Switzerland*

** adam.clark@psi.ch*

The newly constructed Debye beamline at the upgraded Swiss Light Source (SLS 2.0) is now accepting users. Debye is positioned to provide multimodal X-ray absorption spectroscopy (XAS) and X-ray diffraction (XRD) to investigate the chemical, geometrical and electronic structure of functional materials under operating conditions. The beamline is equipped with a 2.1 T bending magnet source (soon to be upgraded to a tuneable 3.5 - 5 T superbend) and an in-house engineered quick scanning channel-cut monochromator to offer an accessible energy range from 4.5 keV to 60 keV. A cutting-edge motion control system provides a diverse range of control options, facilitating quick-scanning XAS (with sub-second time-resolution), and quasi-simultaneous XRD pattern collection at stationary points in the monochromator oscillation. The Debye beamline (Figure 1) is equipped with a photon-counting Pilatus 6M area detector that allows both independent and quasi-simultaneous XRD collection up to 25 keV. We aim for a second high-Z photon-counting pixel detector to cover the higher energy range available and include time-resolved X-ray total scattering to the beamline capabilities.

A pilot experiment following the *in situ* Zr-MOF synthesis was recently conducted in quasi-simultaneous mode. XRD patterns were collected with a time resolution of 15 s (Figure 2a) while fluorescence XAS Zr K-edge spectra were collected every 1 s (Figure 2b). The experiment tracked the conversion from initial precursors chemical, the early nucleation of Zr centres through to the crystallisation of the metal organic framework, providing insights on the kinetics and dynamics of the synthesis process. This combined approach enabled to track crystallization kinetics (Figure 2c), coherence lengths and degrees of crystallinity, as well as oxidation states and pre-crystalline structure (Figure 2d) through the pairing of XAS with XRD.

This experiment is a good example of the type of research questions that would benefit from the combination of XRD and XAS. Besides following the synthesis of crystalline materials, this multimodal approach is especially useful for *operando* characterization studies in heterogeneous catalysis and electrocatalysis, providing time-resolved insights on the electronic and structural properties of these materials.

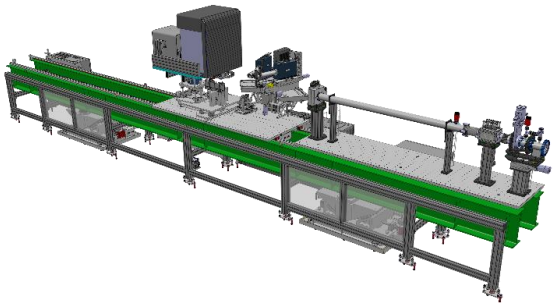


Figure 1. *Design of the Debye end-station experimental table*

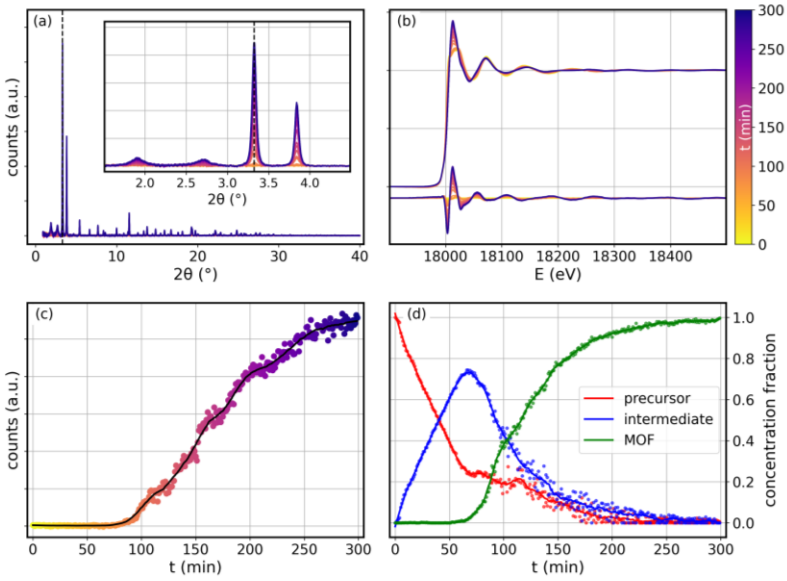


Figure 2. *(a) XRD (low-angle zoom in the inset) and (b) Zr K-edge XAS data obtained during the synthesis of Zr-MOF. (c) Evolution of XRD peak intensity at 3.325° and (d) MCR concentrations from XAS data.*