Contribution ID: **5** Type: **Poster and Flash Presentation**

INSIGHTS INTO MOF SYNTHESIS WITH COMBINED IN SITU APPROACH: RAMAN SCATTERING, X-RAY ABSORPTION, X-RAY DIFFRACTION

Monday 5 January 2026 15:50 (3 minutes)

Metal–organic frameworks with a Zr-oxo cluster [(Zr6O4(OH)4)]12– are exceptionally stable and offer vast potential for a wide range of applications. Synthesis parameters strongly affect the quality, stability, morphology, etc., of the MOFs calling for elucidating of the various reaction steps.

Figure 1 top: Reaction kinetics of Zr-oxo-cluster formation in DMF based on LCF of the in-situ XAS data: effect of temperature, modulator, and water concentration [1]; middle: in-situ synchrotron PXRD data of water-based synthesis of Zr-fumarate; bottom: in-situ Raman spectra of Zr-fumarate synthesis at HT and growth of 1666 cm –1 peak [3].
In this study, we present a combined approach of three in situ techniques: X-ray diffraction, X-ray absorption, and Raman spectroscopy, that allows for integrated monitoring of MOF synthesis in different solvents and at variable temperatures. In the early pre-crystalline synthesis phase, the local (~2–5 Å) environment around Zr4+ ions and the Zr-oxo cluster formation is addressed by element-specific X-ray absorption at Zr k-edge. Raman scattering provides evidence of coordination between inorganic and organic blocks in the synthesis reaction. Owing to week scattering from water, Raman scattering is much more suitable for the in-situ studies of the water-based synthesis than infrared spectroscopy. Crystallization onset of the MOFs was evidenced by the appearance of the scattering peaks in the in situ patterns.

The kinetics of the various steps of the synthesis reaction can be studied using linear combination fits (LCS, X-ray absorption data, Figure 1a [1]), growth of PXRD reflections (PXRD data [2], Figure 1b), and the intensity growth of the cluster–linker coordination vibrational mode (Raman scattering, Figure 1c [3]). The dependence of the reaction kinetics on water, modulator concentrations, solvent, and metal precursor were studied as well. The results of the in-situ experiments were correlated with post-synthetic characterization of the resultant MOF products.

Author: ZAVOROTYNSKA, Olena (University of Stavanger)

Co-authors: Prof. CHAVAN, Sachin (University of Stavanger); Dr BERCHA, Sofiia (University of Stavanger)

Presenter: ZAVOROTYNSKA, Olena (University of Stavanger)

Session Classification: Flash Presentations