STRUCTURE CHARACTERIZATION OF METAL OXO CLUSTERS IN SOLUTION USING TOTAL SCATTERING – FROM CLUSTERS TO MATERIALS

Kirsten M. Ø. Jensen

Department of Chemistry, University of Copenhagen, Denmark

Nanoscale metal oxo-clusters are building blocks for a range of promising new functional materials for e.g. photocatalysis. They are also expected to be intermediates for oxide formation in solution-based syntheses, where they may direct the synthesis outcome. While such clusters have been studied extensively for decades, knowledge gaps still exist on their solution chemistry and solution structure, especially at non-ambient conditions, where conventional structure analysis using single crystal X-ray diffraction cannot be applied. We have used X-ray total scattering with Pair Distribution Function analysis to study the structure of metal oxo clusters directly in solution and investigate how they grow and how they take part in material synthesis.[1-4] Through *in situ* X-ray total scattering studies of transition metal tungsten and molybdenum oxide formation, we show that polyoxometalate clusters are indeed important intermediates in oxide synthesis, and that their presence and structure can direct the final product of the synthesis. We also show how X-ray total scattering combined with DFT calculations can shed new light on metal oxo speciation at elevated temperature and pressure, as relevant for designing hydrothermal synthesis pathways of new materials.[5]

[1].M. Juelsholt, T. L. Christiansen and K. M. Ø. Jensen, *J. Phys. Chem. C*, 2019, **123**, 5110-5119.

[2].S. L. Skjærvø, A. S. Anker, M. C. Wied, E. Kjær, M. Juelsholt, T. L. Christiansen and K. M. Ø. Jensen, *Chem. Sci.*, 2023, **14**, 4806-4816.

[3].L. G. Graversen, M. Juelsholt, O. Aalling-Frederiksen, U. Friis-Jensen, R. K. Pittkowski, M. S. Thomsen, A. Kirsch, N. P. L. Magnard and K. M. Ø. Jensen, *Chem. Sci.*, 2025, **16**, 14350-14365.

[4].L. S. Junkers, S. L. Skjærvø, O. Aalling-Frederiksen, C. Tynell Hansen, M. Juelsholt, N. Lefeld, R. K. Pittkowski, N. P. L. Magnard, A. Kirsch and K. M. Ø. Jensen, *ChemistryEurope*, 2025, **3**, e202500062.

[5] L. S. Junkers, D. Garay-Ruiz, J. Builds, R. S. Silberg, G. B. Strapasson, K. M. Ø. Jensen and C. Bo, *J. Amer. Chem. Soc.*, 2025, **147**, 22747-22758.