Poster

Structural Characterization of MOFs using Three Dimensional Electron Diffraction

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Three-dimensional (3D) electron diffraction is an emerging technique that has shown great promise as a powerful tool for the structural characterization of materials. Compared to traditional single crystal X-ray diffraction (SCXRD), 3D electron diffraction can be used to analyze small crystalline samples and amorphous materials, providing valuable information about the atomic structures and arrangements of molecules in the sample [1,2]

Metal-organic frameworks (MOFs) are a class of porous materials with tunable properties that have attracted significant attention in various fields of research. However, growing large crystals of MOFs for structural analysis by SCXRD can be challenging and time-consuming and sometimes impossible. In this context, 3DED presents an attractive alternative for characterizing MOFs. One of the main advantages of 3D electron diffraction is its ability to capture high-resolution diffraction patterns of small crystals with dimensions in the order of nanometers. This is possible thanks to the strong interaction of electrons with matter as opposed to X-rays, allowing them to diffract from small crystals and overcome the limitations of traditional X-ray diffraction methods. In this work, we describe the structure solution of a MOF with particle size of approximately 250nm synthesized through an environmentally friendly technique: Liquid Assisted Grinding mechanosynthesis, using water as solvent and biocompatible precursors. The structure was then dynamically refined showing a clear drop in the figure of merits as well as the presence of water molecules in the channels. We also present a new heterometallic MOF, which offer a range of potential advantages over their homometallic counterparts, including improved catalytic activity, increased stability and enhanced selectivity.3 The MOF was synthesized using a solvothermal method and its crystal structure determined using 3D ED, revealing a complex network and a high degree of disorder. It shows an orthorhombic structure with a spacegroup of Cmmm and a cell volume above 11000 A^3, which can be advantageous for certain applications. Its tetranuclear metallic cluster, connected to eight linkers, is composed of six-coordinated octahedral Ti(IV) and seven-coordinated pentagonal bipyramidal Ca(II) centers [3].

Our results prove the potential of 3DED as a powerful technique for the structural characterization of nanocrystalline materials, particularly those with complex crystal structures such as MOFs as it provides valuable insights into the atomic arrangements, enabling the design and optimization of these materials for various applications.
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