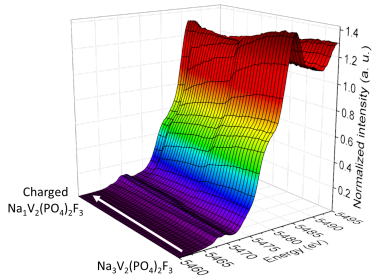

Figure 1. Vanadium K-edge XANES raw data collected operando upon charge of the battery Na//$Na_3V_2(PO_4)_2F_3$

Keywords: Na-ion Battery, operando synchrotron X-ray powder diffraction, operando XANES, NVPF

MS43-O3 In situ analysis of mechanochemical reactions using combined X-ray diffraction and Raman spectroscopy

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Mechanochemistry is increasingly used for synthesizing various materials including metal organic compounds and cocrystals.[1-3] Although this synthesis approach offers a fast and pure synthesis in high yields, there is a lack in understanding the mechanisms of milling reactions. The necessary data can only be obtained in *in situ* experiments, which were only recently established for milling reactions.[4,5] Herein, we present a novel setup enabling a combined in situ investigation of mechanochemical reactions using synchrotron XRD and Raman spectroscopy (see Fig.1). The specific combination allows to study milling processes comprehensively on the level of the molecular and crystalline structure and thus obtaining reliable data for mechanistic studies. Besides well-known MOFs like ZIF-8, the formation process of new metal phosphonates [6] and model cocrystals [7] could be studied in detail. The syntheses pathway of the different compounds could be revealed. The results prove that the presented method combination is applicable for a wide range of materials and will provide the necessary understanding to tune and optimize mechanochemically synthesized compounds.

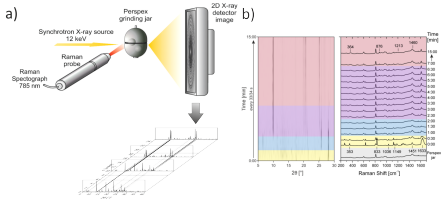

Figure 1. a) Schematic diagram of the experimental setup for collecting Raman spectra and XRD powder patterns during the mechanochemical synthesis. b) Synthesis process of the metal organic framework $(H_2Im)[Bi(1,4\text{-bdc})_2]$ followed *in situ* by synchrotron XRD (left) and Raman spectroscopy (right).

Keywords: in situ, mechanochemistry, Raman spectroscopy