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Operando and ex-situ PXRD Study of Ni-doped Manganese Hexacyanoferrate Cathode Material in Aqueous Zn-ion Battery System

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Operando experiments are essential in the field of battery research as they avoid the typical drawbacks of ex situ experiments, such as sample transfer or relaxation reactions, that may occur after opening the electric circuit; alongside the availability of providing sample behaviour under the normal operating conditions in single test cell experiments, avoiding the uncontrolled differences between the cells [1]. Aqueous rechargeable Zinc-ion batteries (ARZIBs) are one of the most promising post lithium-ion battery candidates and Prussian blue analogues (PBAs) are widely considered as cathode materials for them, due to their large ionic channels and redox-active sites [2]. Our study material is manganese hexacyanoferrate (MnHCF). For the improvement of its performance Ni-doping technique is applied, mostly for the relaxation of the Jahn-Teller distortion and therefore improving the long-time stability [3].
Here the experimental results of 10% Ni doped MnHCF will be highlighted in AZIB system with 3M ZnSO4 electrolyte and Zn metal anode. PXRD studies were performed in MCX beamline in Elettra synchrotron facility. For operando experiment EL-CELL was used in reflection mode with CCD detector.
Generally, during the cycling process some structural changes or the new phase formation might occur inside the cathode material. The structural exchange of Mn with Zn in AZIB system based on XAS analysis has been reported before in pure MnHCF material [4]. According to our operando and ex-situ PXRD measurements the same phenomena were observed for Ni-doped material. The lattice parameter has changed and the additional peaks have appeared even during the resting mode, just by electrode being exposed to the electrolyte. Changes continued to occur during the consecutive charge and discharge process. The appearance of additional peaks both in operando and ex-situ samples proves the new phase formation inside the material. During the discharge process the PXRD patterns showed only partial reversibility of the structure. [5].

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