# Advanced operando characterization of alkali-ion battery materials

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Understanding electrochemical processes in sodium- and lithium-ion batteries requires techniques that can follow structural and interfacial changes during operation. Two different research directions in connection with large scale research infrastructure will be discussed in the presentation.

Multinuclear ssNMR is used to investigate alkali-ion storage, structural evolution, degradation reactions, and solid electrolyte interphase formation at the atomic scale. Special attention will be given to operando $^{23}$Na ssNMR studies of hard carbon anodes, which reveal the evolution of sodium environments from ionic sodium to quasi-metallic clusters. Additional examples include ssNMR studies of controlled lithium supplementation from self healing additives in battery cells.

The presentation will also address Li-rich layered oxide cathodes, specifically $Li_{1.2}Mn_{0.54}Ni_{0.13}Co_{0.13}O_2$ materials prepared by sol-gel synthesis using citric or oxalic acid as chelating agents and calcined at 850 or 900 °C. The use of synchrotron techniques was essential for determining disorder and Li/Ni intermixing, and their relationship to electrochemistry. Operando XRD reveals pronounced a-lattice expansion during the first cycle, linked to oxygen-vacancy mobility and homogenisation. Operando XANES and ex situ EXAFS further show that this structural flexibility limits Li/TM mixing and enables deeper Ni reduction.

Together, these studies demonstrate how advanced operando methods, including ssNMR, XRD, XANES, and EXAFS, provide critical insight into ion-storage mechanisms, defect dynamics, and degradation pathways, guiding the design of more stable and high-performance alkali-ion batteries.

References:

1) M. Gabrijelcic, et al, J. Mater. Chem. A (2025) 13 (2): 1042–1056.
2) B. Pal, et al, Batteries & Supercaps 2026, 9, e202500444.
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**Short bio:**

Prof. dr. Robert Dominko is a Research Professor at the National Institute of Chemistry and a Professor at the University of Ljubljana. He leads the battery research group at the National Institute of Chemistry. His research activities are focused on materials science and electrochemical systems for energy storage. His current research interests include multivalent battery systems and the integration of smart functionalities into battery cells. Prof. Dominko is actively involved in European battery initiatives. His bibliographic reports contain over 240 publications and 16 patents. He is a member of the Slovenian Academy of Engineering.