

Contribution submission to the conference Dresden 2026

X-Ray Absorption Near-Edge and Photoemission Spectroscopy of Deintercalation and Redox Reactions in Alluaudite-Type Na-Ion Cathodes

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We have investigated alluaudite-type $\text{Na}_{2.5-x}\text{Fe}_{1.75}(\text{SO}_4)_3$ cathodes that exhibit a three-dimensional open-channel framework with multiple Na sites. Using spatially resolved X-ray absorption near-edge spectroscopy (XANES), performed within a transmission X-ray microscope (TXM), and lab-based X-ray photoelectron spectroscopy (XPS), we have examined Na^+ deintercalation and $\text{Fe}^{2+}/\text{Fe}^{3+}$ charge compensation at different selected charging states. This combination of bulk- and surface-sensitive methods allows correlating Na^+ depletion with Fe^{3+} formation, potentially identifying nanoscale redox heterogeneities and tracking changes in the local Na chemical environment. Our study builds on a broader investigation [1] that includes theoretical calculations as well as structural and electrochemical analyses.

[1] J. Molenda *et al.*, *Acta Mater.* **301**, 121582 (2025).

Part: CPP
Type: Vortrag; Talk
Topic: Energy Storage Materials and Devices
Keywords: Na-ion batteries; Na deintercalation; Fe redox; spectromicroscopy; XPS
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