



# Monitor the oxidation state and fine structures of mixed anion cathode materials during charge/discharge process in-operando conditions

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## 1. 背景と研究目的

Materials for Lithium and Sodium batteries are an active field of research. Anionic substitution is a path to improve battery performance. In this proposal, we measured materials with and without different anionic substitutions to search for changes in the cation environment. The materials to be tested: NCM ( $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_2$ , and  $\text{LiNi}_{0.8}\text{Co}_{0.1}\text{Mn}_{0.1}\text{O}_{2-x}\text{F}_x$ ) was modified with  $\text{XeF}_2$  at different times and with intermediate thermal treatment. Also, in-operando complementary measurements to previous measuring time using  $\text{NaFe}_{0.4}\text{Mn}_{0.3}\text{Ni}_{0.3}\text{O}_2$  as a cathode.

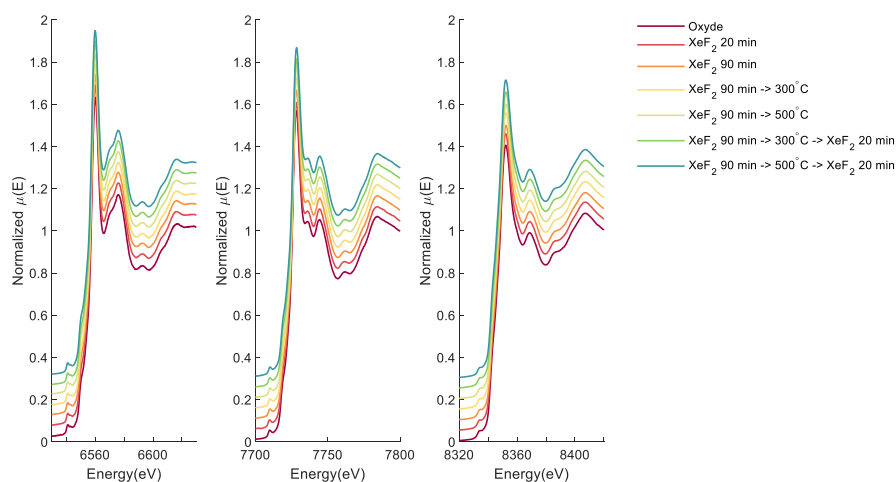
## 2. 実験内容

Due to the air sensitivity of cathode materials (NCM), the samples were prepared in a glove box. Oxides were mixed with BN in proportions according to the cationic composition to get a jump equal to 1. The mixture of oxides and BN were pressed to 10kN in a 10mm die. The pellets were packed into aluminized bags with Kapton windows and sealed into an argon atmosphere to avoid air exposure. For In-operando measurements of FMN bare oxide, a 5 mm hole was performed in the coin cells and covered with Kapton tape to allow the normal operation of the cell. Correct operation of the cells was performed in the lab prior to the synchrotron shift. All samples were measured in Q-XAS, with measurements time of 2 minutes. The K-edges of each metal (Mn, Co, and Ni) were measured.

## 3. 結果および考察

For the system NCM, XAFS spectra showed not relevant changes between the bare oxide and the oxyfluoride, indicating that the improvement in the electrochemical performance by the fluoridation process is affecting the surface of the oxide but not the bulk of the material. Figure shows the XANES spectra at Mn, Co and Ni edge of oxides with different  $\text{XeF}_2$  treatment. The EXAFS spectra in k and R-space showed the same similarities.

In the case of in-operando measurement, the batteries had an internal short-circuit which avoided to perform charge process for in-operando measurements.



## 4. 参考文献

1. Kimijima et al., Cryst. Growth Des. 2016, 16, 5, 2618–2623