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EELS Probing of Lithium Based 2-D Battery Compounds Processed by Liquid Phase Exfoliation

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Two-dimensional lithiated nanosheets usually show excellent electrochemical performance due to an increase in surface area and shorter diffusion paths. Liquid exfoliation of bulk material is one of the most efficient means of producing 2-D materials in a large-scale quantities[1]. However, it is not yet fully clear how the exfoliation process affects electronic and crystal structure or stability of the exfoliated materials. Moreover, the extreme processing conditions might knock out some light lithium atoms, and therefore the obtained flakes might present different chemical/structural compositions. Greater understanding of these effects is necessary in order to fully exploit the material's potential.

Here different lithiated layered compounds mainly $LiCoO_2$, $LiMn_2O_4$, and $Li_5Ti_4O_{12}$ were chemically exfoliated and investigated using electron energy loss spectroscopy (EELS) for their Li-K edge. Further analyses were carried out, looking at the oxygen (O) K edge with their respective transition metal core loss peak (Mn, Co and Ti) which revealed changes in the Energy loss near edge structures (ELNES) when compared to the unlithiated compounds. STEM-EELS analyses confirmed uneven distribution of lithium within the lithiated layered materials (Figure 1). To study the distribution of Lithium for each centrifugation speed, flakes were examined using TEM-EELS with a broad beam spread across an entire flake. In this work, EELS was used for the first time to detect and to probe the chemical environment of the lithium in liquid phase exfoliated material[2][3][4][5] [6].

References:

[1] V. Nicolosi *et al*, Science **340**, no. **6139** (2013), p. 1226419.
[2] A. Pokle *et al*, Nano Energy **30** (2016), p. 18.
[3] F. Wang *et al*, ACS Nano **5**, no. **2** (2011), p. 1190.
[4] F. Lin *et al*, Sci. Rep **4** (2014), p. 5694.
[5] C. M. Wang *et al*, Appl. Phys. Lett. **94**, no. **23** (2009), p. 233116.
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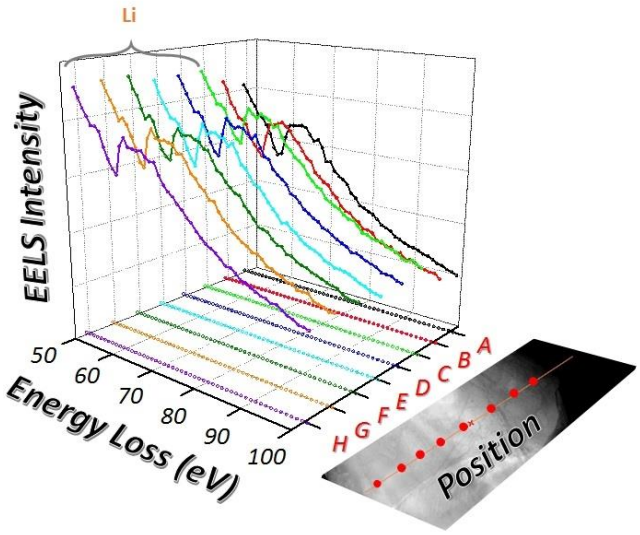

Figure 1. STEM-EELS of Li K-edge for chemically exfoliated $LiCoO_2$ revealing a small peak at 56 V, as well as the prominent signal at 62 eV, which indicates the presence of Li-ion.

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