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Sodium vs. lithium co-intercalation with diglyme electrolyte into graphite anodes: Evidence from multi-nuclear solid state NMR spectroscopy**Nicole Leifer, Miryam Fayena Greenstein, Albert Mor, Gil Goobes and Doron Aurbach**
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The growing need for cost-effective batteries for large scale energy storage is motivating sodium-ion battery (SIB) technology development, especially in applications where energy density/weight is not crucial. However, the performance of intercalation compounds as viable electrodes for SIB has not matched the success of Li-ion batteries (LIBs). Graphite, as the most widely used anode material in current commercial LIBs, exhibits very low capacities (<35 mAh/g) as an anode material in SIBs, as compared to 372 mAh/g for Li. This has been attributed to the instability of the Na ions intercalated into graphite, unlike Li ions. By using diglyme as the electrolyte solvent, however, reversible Na intercalation in graphite was achieved with capacities reaching 80 mAh/g for up to 1000 cycles and >99% reversible capacity. The good recyclability and limited capacity fade was attributed to the concomitant intercalation of the alkali ions with coordinated diglyme molecules (co-intercalation), i.e. the formation of ternary (t)-GIC complexes upon discharge. Analogous studies with Li gave inferior results, with exfoliation of the graphite layers upon extended cycling. Computational studies suggest that the Na-diglyme complex intercalates and binds quite differently than Li-diglyme complex in the graphite. However, experimental evidences of co-intercalation are only indirect and so is their relation to the disparate electrochemical behavior of Na vs. Li. This study examined the graphite anode material, diglyme molecules and the alkali ions upon electrochemical cycling against Na or Li metal in a diglyme-based electrolyte solution via solid-state NMR spectroscopy. Direct evidence for co-intercalation, as well as qualitative and quantitative comparisons of the details of the ion-solvent-graphite interactions, are provided via ²³Na, ⁷Li, ¹H and ¹³C NMR measurements. The conclusion from this work should prove useful for improving the performance of other higher capacity carbon based anodes for SIBs.

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