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Enhanced Stability of Lithium Manganese Oxide Ion-sieves by Magnesium doping for Lithium Recovery from Flowback and Produced Water

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The increased demand for lithium (Li), driven in part by the use of Li-ion batteries, poses a crisis in its future supply. To meet demand, the exploration of alternative Li sources is imperative. Flowback and produced water (FPW), a by-product of oil and gas exploration, is a potential resource that often contains tens to hundreds of parts-per-million Li. Among the various direct Li extraction approaches applicable to FPW, spinel lithium manganese oxide (LMO) ion-sieves have emerged as a highly promising material due to their high Li uptake and rapid adsorption kinetics. However, LMO sorbents face challenges such as mass loss due to the reductive dissolution of manganese (Mn) caused by organic compounds present in FPW, which impairs its recyclability. In this study, we doped a pristine LMO ($Li_{1.6}Mn_{1.6}O_4$) with 4 different concentrations of magnesium (Mg) to synthesize Mg-doped lithium manganese oxides, $Li_{1.6}Mg_xMn_{1.6-x}O_4$ or LMMO-x (where x = 0.1, 0.2, 0.3, 0.4). Li recovery tests conducted using FPW produced from the Duvernay Formation in Alberta, as an example, demonstrate that both Li uptake and Mn dissolution decrease with increased Mg doping. Specifically, Li uptake decreased by 53% for LMMO-0.4, while the average Mn dissolution during subsequent acid desorption was reduced by 80% compared to pristine LMO. Cycling tests show that LMMOs retain 95% of their initial Li uptake after the 5th cycle of use, compared to only 90% for LMO under the same experimental condition, demonstrating that LMMOs have a better recyclability. Extended X-ray absorption fine structure (EXAFS) analyses further confirm the improved stability of LMMOs, as irreversible structural contraction occurred in LMO after 5 cycles of use, but not found in the LMMOs. LMMOs, when further combined with polyvinylidene fluoride membrane, exhibit negligible Mn dissolution (<0.3%). This study demonstrates that Mg doping enhances the stability of LMMOs, making them promising candidates for Li recovery from FPW.