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Mechanochemical Destabilization and Deep Eutectic Solvent Based Lithium Recovery from Layered Oxide Cathodes of Spent Electric Vehicle Batteries

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Lithium-ion batteries (LIB) used in electric vehicles typically last 8-10 years. By 2030, around 11 million metric tonnes of spent LIBs are expected worldwide, which, if landfilled, could result in fires, contaminate groundwater with toxic heavy metals, or, if incinerated, release harmful gases. Recycling LIBs is an important source of critical raw materials. Current industrial recycling rely on energy-intensive pyrometallurgy or corrosive/hazardous inorganic acid-based hydrometallurgy, resulting in high chemical consumption and limited lithium selectivity. In $LiNi_x\ Mn_y\ Co_z\ O_2$ (NMC) cathodes of LIBs, lithium occupies interlayer octahedral sites stabilized by transition metal (TM)-oxygen framework. Extractability of lithium is governed by lattice instability, defect formation, solid-liquid interfacial reactions, and the reducing potential of the system. This study is an integrated mechanochemical-solvometallurgical approach for selective lithium recovery using greener alternatives to acids.

High-energy ball-milling of NMC with an inexpensive chelator induced selective chelation of TM at defect-rich surfaces, promoting localized destabilization of the MO_6-octahedral framework without direct lithium complexation. Optimizing NMC:chelator mass ratios, milling time, rotational speed and ball-to-NMC ratio, and subsequent water leaching caused <5% TM dissolution and ~60% lithium recovery in a single extraction. Increasing the chelator mass, increased TM leaching to 40-50% and lithium extraction to >90%. Because the chelator forms strong complexes with Ni^{2+}, Co^{2+}, Mn^{2+} but has negligible affinity for Li^+, lithium liberation arises likely due to chelation-induced lattice disorder rather than ligand-assisted dissolution. Structural disruption weakens TM-O bonding, and increases lithium extractability. Control experiments including unmilled, milled NMC without the chelator, confirmed lithium release by mechanochemically induced destabilization rather than surface washing. Following lithium removal, the transition-metal-rich residue was treated using a viscosity-engineered ternary deep eutectic solvent (DES). While binary choline chloride-oxalic acid DESs achieve >80% metal dissolution at 80°C, their high room-temperature viscosity (~11,400 cP) limits mass transport. Incorporation of a third hydrogen-bond donor (ethylene glycol/glycerol) reduces viscosity (~500 cP), and maintains comparable leaching efficiencies.

This combined approach shows how lattice damage, metal binding strength, lithium hydration, and solvent properties together control how critical metals dissolve and move across $LiNi_x\ Mn_y\ Co_z\ O_2$ -water/DES interfaces, to enable high recovery of critical minerals from spent LIBs.