

## METAL RECOVERY FROM LI-ION BATTERY WASTE

BLADERGROEN B.J, PETRIK L., TAWONEZVI T., ZIDE D, MASSIMA E.S.M. & TSHISANO M.  
University of the Western Cape, South Africa

### KEY FINDINGS

The study explored the recovery of valuable metals from spent lithium-ion batteries (LIBs) using functionalized polyethylene terephthalate (PET) nanofibers and electrowinning techniques. PET nanofibers, enhanced with di(2-ethylhexyl) phosphoric acid (DEHPA), exhibited selective adsorption capacities for Mn ions, due to increased surface area and selectivity. Optimal electrospinning conditions yielded nanofibers with durable structures, further improving metal ion recovery. Electrowinning was employed for metal recovery from the LiB cathode leachate solutions, achieving high purity metal extraction efficiently. The combined approach demonstrated consistent performance, with PET-DEHPA nanofibers showing stability in acidic conditions and maintaining adsorption efficacy over multiple cycles. This integrated method offers an eco-friendly, cost-effective solution for recycling valuable metals from LIBs, addressing environmental and resource scarcity challenges.

### INTRODUCTION

Lithium-ion batteries (Li-ionBs) are crucial for portable electronics, energy storage, and electric vehicles. The market for Li-ionBs is projected to reach \$116 billion by 2030. However, recycling of these batteries remains limited, with only 5% currently being recycled due to costly, energy-intensive, and environmentally harmful processes. Efficient, low-cost, and eco-friendly recycling methods are urgently needed to recover valuable metals such as Li, Co, Ni, and Mn from the battery cathodes.

### METHODOLOGY

The research focuses on a selective, low-cost, and energy-efficient method to recover Ni-Co alloys from spent Li-ionB cathodes using potentiostatic electrowinning. In parallel using functionalised PET-DEHPA electrospun nanofibers were developed as selective adsorption material for Ni, Co and Mn ions. The potentiostatic electrowinning process integrates rotating cathodes and Pt-coated Ti anodes to enhance deposition rates and reduce contamination, ultimately lowering operational costs. Recovered Ni-Co alloys are intended for use in producing new NMC (nickel-manganese-cobalt) cathodes, among other applications.

PET nanofibers were fabricated using electrospinning, optimizing parameters like polymer concentration (5-

20 wt%), solution flow rate (0.4-1.0 mL/h), collecting distance (13-20 cm), and applied voltage (30 kV). PET-DEHPA nanofibers were made by adding DEHPA ligand (5-20% v/v) to the PET solution and electrospinning the mixture. Characterization techniques like SEM, EDS, XPS, FTIR-ATR, TGA, and XRD were used to analyse the nanofibers.

### MAIN RESULTS

**Leaching:** The leaching process used an inorganic acid-reductant solution (2M H<sub>2</sub>SO<sub>4</sub> + 6 vol.% H<sub>2</sub>O<sub>2</sub>) to dissolve the cathode materials, achieving high recovery efficiencies: 98.1% for Li, 97.1% for Co, 96.1% for Ni, and 95.7% for Mn. The optimal conditions included a solid-to-liquid ratio of 75 g/L, 60°C temperature, and specific reaction times for freeing and leaching metals.

**Electrowinning:** Electrowinning experiments utilized synthetic solutions to mimic the composition of real cathode leachates. Key parameters such as applied potential, pH, temperature, and cathode rotation speed were optimized. The process achieved a Ni-Co recovery rate of 0.06 g/cm<sup>2</sup>/hr at 88% current efficiency, recovering 90% of Co and 77% of Ni from the leachate. The best conditions produced Ni<sub>0.65</sub>Co<sub>0.35</sub> with 98% purity. Subsequent tests with real NMC 532 solutions confirmed these findings, yielding a Ni<sub>0.7</sub>Co<sub>0.3</sub> alloy with over 97.35% purity.

**Precipitation:** Remaining metals in the spent electrolyte were recovered through multi-stage precipitation, achieving over 98% efficiency for  $\text{Ni}(\text{OH})_2$ ,  $\text{Mn}(\text{OH})_2$ , and  $\text{Li}_2\text{CO}_3$ . Optimal conditions for precipitation included specific pH levels, temperatures, and reaction times, tailored for each metal compound.

**Adsorption onto electrospun nanofibers**

Pristine PET nanofibers had minimal metal ion adsorption while PET-DEHPA nanofibers showed significant adsorption, particularly for Mn ions. Higher DEHPA concentrations enhanced adsorption capacity. Optimal conditions included a pH of 4 and a contact time of 60 mins. Maximum adsorption capacities were 32% for Ni, 41% for Co, and 54% for Mn, indicating a higher affinity for Mn ions. This selectivity capability towards Mn ion was attributed to the good chemical stability of the metal ion complex with the functional groups of the chelating DEHPA ligand.

**Process Overview:** The integrated process of leaching, electrowinning, and precipitation demonstrates a feasible semi-closed loop recycling system (Figure 1). The main goal of recovering high-purity Ni-Co composite was achieved. The recovered Ni-Co alloys and other compounds have diverse applications, including battery production, electronics, pharmaceuticals, and water treatment.

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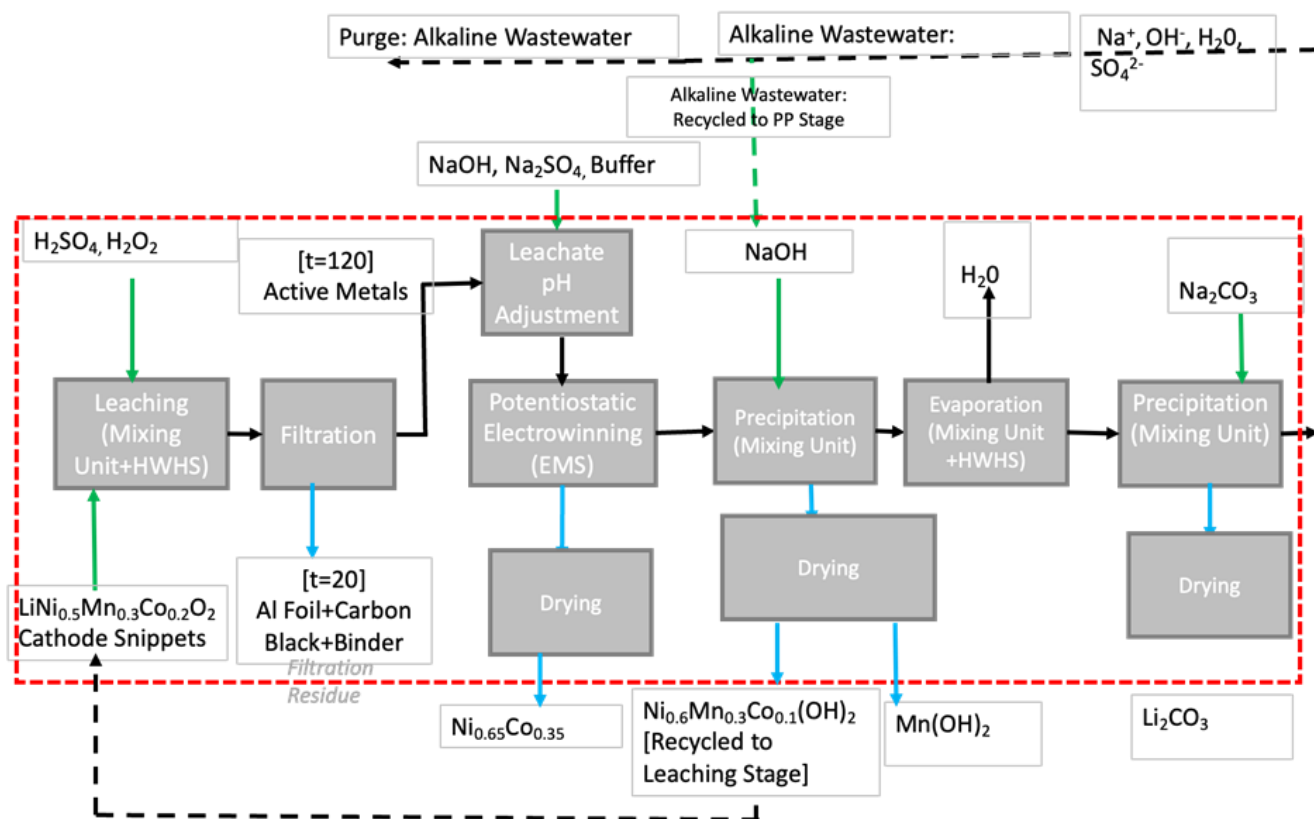


Figure 1. Graphical abstract of the valuable Ni-Co and metallic by products recovery process

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