Abstract

This dissertation addresses the development and validation of an integrated technology for the selective separation and electrochemical recovery of nickel and cobalt from real sulfate solutions generated during the hydrometallurgical processing of black mass from spent lithium-ion batteries. The research was initially conducted on simulated streams, and subsequently on real ones—originating from the processing of various batches of black mass derived from the shredding of approximately 5 tons of batteries. The objective was to prepare electrolysis baths with a composition favorable for electrowinning, and subsequently to obtain products with commercial potential—namely, metal sheets of the largest possible dimensions (width, thickness, height) and the highest possible chemical purity. The nickel stream yields metallic nickel, whose cobalt content depends directly on the cobalt concentration in the raffinate fed to electrowinning and can be controlled by adjusting the number of solvent extraction stages.

In the first stage of the work, the focus was on the separation of cobalt from nickel using solvent extraction. This stemmed from the fact that these metals are electrochemically very similar, making their selective separation by direct electrolysis impossible. A selective commercial extractant, Cyanex 272, was employed in the study. It was demonstrated that the molar ratio of Cyanex 272 to cobalt serves as a more effective extraction control parameter than the conventional phase ratio, enabling process control for feeds with varying chemical compositions. The high selectivity of the Co/Ni separation was confirmed. A concept was developed to couple cobalt stripping with electrowinning (the SX-EW loop), wherein the acid generated in electrowinning cell strips cobalt from the organic phase, thereby eliminating the need for external reagent dosing. This self-regulating mechanism was extended to nickel electrowinning through the controlled dissolution of $Ni(OH)_2$ within the circulation loop, yielding a stable current efficiency of 88–93% and a specific energy consumption of 2.8– 3.0 kWh/kg.

The key operational parameters of the individual processes were determined, along with the influence of bath conductivity, composition, and organic additives on the quality of the metallic deposits. A procedure for extractant regeneration was developed. The processes were investigated at semi-industrial scale through the design and commissioning of a pilot

plant comprising the solvent extraction and electrowinning units. The obtained results provide a foundation for the implementation of a compact, reagent-saving technology for the recovery of strategic metals, aligning directly with the principles of the circular economy.