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Heat-Transfer-Regime-Controlled Fluorine Pathways During Thermal Pretreatment of Lithium-Ion Battery Black Mass

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ABSTRACT

Lithium-ion battery black mass, a heterogeneous mixture of electrode materials recovered after mechanical processing, contains valuable metals such as lithium, nickel, cobalt, and manganese, alongside residual organic binders and electrolyte-derived species, including fluorinated compounds originating primarily from polyvinylidene fluoride (PVDF) and lithium hexafluorophosphate decomposition. The presence of these non-metallic components is of particular concern, as it can potentially hinder downstream hydrometallurgical recovery processes.

Thermal pretreatment is therefore a critical step in lithium-ion battery recycling, enabling removal of organic binders and electrolyte residues from black mass prior to downstream metal recovery. Despite its widespread use, conventional approaches are typically designed around temperature and residence time, with limited consideration of the underlying heat-transfer regime. In practice, these treatments often rely on convection-dominated thermal processing, where moderate temperatures are applied over extended durations. Such conditions can promote secondary reactions between fluorinated decomposition products and active materials, resulting in the formation of stable surface fluorides that may adversely affect subsequent processing.

In this work, the influence of heat-transfer regime on fluorine behaviour during thermal pretreatment of lithium-ion battery black mass is investigated. Convection-dominated thermal processing, analogous to conventional kiln-based processing, is compared with a radiation-dominated, high heat-flux approach representative of flash calcination. Rather than focusing solely on peak temperature, this study considers the role of heat flux and thermal exposure in governing the balance between reaction and volatilisation pathways during binder decomposition.

Surface chemistry, fluorine speciation, and morphological changes were characterised using X-ray Photoelectron Spectroscopy (XPS), Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS), and Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDS). The results indicate that heating conditions strongly influence the distribution and chemical environment of fluorine at particle surfaces. Under lower heat-flux, longer-duration conditions, trends consistent with fluorine retention and redistribution are *observed, suggesting the formation of stable fluoride species. In contrast, high heat-flux treatment shows evidence of reduced surface fluorine intensity and suppression of PVDF-derived fragments, indicative of more effective removal of fluorinated species.*

These findings highlight heat-transfer regime as a governing parameter in black-mass pretreatment and suggest that high heat-flux thermal processing may enable more selective removal of fluorinated contaminants while limiting secondary surface reactions. This work provides a basis for re-evaluating thermal pretreatment strategies beyond conventional temperature-based design and offers new insight into controlling surface chemistry for improved recycling performance.

KEY WORDS

Spent LIBs, Pretreatment, Pyrometallurgy, Energy Science, Metal Recovery, Waste Battery Management, Chemical Engineering, Sustainability.

BIOGRAPHY

Hassan Imran Masud is a postgraduate from School of Chemical Engineering at Adelaide University, graduating Master’s of Chemical Engineering. In his master’s degree, Hassan investigated the performance of catalysts and reactor design in hydrogen production through ammonia decomposition using computational methods. He is currently a Ph.D candidate with a focus on investigating and developing new economically and environmentally sustainable processes to pretreat lithium ion batteries for recycling.

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