Multidisciplinary Characterization of Critical European Lithium Ores

Jussi Liipo[a], Sylvain Delchini[b]; Kathy Bru[b]; Saara Malmi[a], Henry Pillière[b]; Nicolas Maubec[b]; Françoise. Bodenan[b]

[a]Metso Research Center, Pori, Finland,
[b]Bureau de Recherches Géologiques et Minières, Orléans, France

Abstract

The EU-funded LITHOS project aims to strengthen the European lithium value chain by developing domestic processing and refining routes that support the targets of the Critical Raw Materials Act (CRMA). The project focuses on the production of battery-grade lithium hydroxide monohydrate ($LiOH \cdot H_2O$) from strategic European hard-rock lithium deposits while expanding existing process flowsheets to enable the economic utilization of lower-grade resources.

The study focuses on three key deposits: Keliber's spodumene ore from Kaustinen, Finland; Savannah Resources' spodumene- and petalite-bearing ores from Barroso, Portugal; and Imerys' lepidolite ore from Beauvoir, France. Comprehensive chemical and mineralogical characterization of ore samples, concentrates, and beneficiation test products was conducted by Bureau de Recherches Géologiques et Minières (BRGM) and Metso using complementary analytical approaches. BRGM applied a combined XRF-XRD analytical system for rapid mineralogical and chemical characterization, while Metso employed conventional methodologies including chemical assays, microscopy, X-ray diffraction, and element-to-mineral conversion techniques. The results prove that reliable quantification of lithium-bearing minerals in complex pegmatite ores requires the integration of multiple analytical methods and that detailed mineralogical characterization is essential for the development of efficient beneficiation flowsheets.

1. Introduction

Lithium has become one of the most strategically important metals worldwide due to its critical role in the green and digital transitions. Its importance is recognized by major economies, including the United States and the European Union, which classify lithium as a critical raw material essential for technological development, energy security, and economic resilience. Demand for lithium is expected to increase dramatically over the coming decades, driven primarily by the rapid growth of electric vehicles and renewable energy storage technologies. The International Energy Agency (IEA, 2021) has projected a more than forty-fold increase in lithium demand under sustainable development scenarios, while the European Commission (2023) expects an even greater increase in long-term demand.

Although numerous lithium-bearing minerals occur in pegmatitic systems (Benson et al. 2025), only a limited number are currently exploited economically (Petrakis et al. 2025). Among these, spodumene is the most widely processed lithium mineral owing to its relatively high lithium content and favorable flotation properties (Filippov et al. 2019). In contrast, despite its historical importance as a lithium source in pegmatite deposits, petalite has received considerably less attention in the mineral processing literature, and only a limited number of studies have investigated its beneficiation by flotation (Lyyra et al.1990, Jessup and Bulatovic 2000, Petrakis et al. 2026).

A detailed understanding of ore mineralogy is essential for developing robust beneficiation flowsheets, particularly in deposits where lithium is distributed among several minerals with differing physical and surface-chemical properties. This study evaluates the benefits, limitations, and practical applications of complementary mineralogical quantification methods for complex European lithium ores. Special emphasis is placed on comparing conventional geometallurgical characterization techniques with a rapid combined XRF-XRD approach and assessing their suitability for supporting process development. The results show that detailed mineralogical characterization is a key requirement for the efficient recovery of lithium from ores containing multiple lithium-bearing phases.

2. Materials and Methods

2.1. Ore samples and sample preparations

The main samples for these studies originate from the LCT complex type aplite–pegmatite dykes present in the Barroso-Alvão region, located in the western part of the Iberian Peninsula, Northern Portugal (Charoy et al. 2001; Martins et al. 2011). Petalite material is not sourced from the current Resources in development, but from a secondary deposit in the Barroso region. In addition, also the ore and concentrate samples from Imerys lepidolite ore from Beauvoir, France and Keliber's spodumene ore from Kaustinen, Finland, were received. It should be noted that these samples don't stand for any known mineral reserves or resources.

2.2. Chemical and mineralogical characterization of the ore and process samples

At Metso, the chemical characterization studies involved chemical ICP-OES analyses (iCAP 6000, Thermo Scientific) following a four-acid digestion to quantify Li, Be, Na, Mg, K, Ca, Ti, Mn, Fe and Nb. Aluminum (Al) was determined after peroxide fusion. The used in-house analytical methods were verified with certified raw materials from Ore Research & Exploration (OREAS), OREAS 752 (pegmatite Li ore), OREAS 753 (pegmatite Li ore) and OREAS 999 (spodumene concentrate), and National Institute of Standards and Technology (NIST) (NIST999 lepidolite). The chemical analysis results reported by the laboratory are in good agreement with the certified results of the materials.

At BRGM, a combined XRF-XRD field instrument is equipped with a cobalt source (Kα1= 1.78897 Å and Kα2= 1.79285 Å) for XRD and a molybdenum source for XRF. It is equipped with two specific and independent detectors for each of the two techniques. The geometrical configuration of this device enables simultaneous measurement of XRF and XRD signals according to the approach developed by (Maestracci et al., 2023). The voltage and current parameters used are respectively 45 kV and 0.15 mA for XRD and 30 kV and 15 µA for XRF. The XRD data processing is based on the Rietveld method and the XRF data processing relies on the automated deconvolution of the acquired spectra to identify chemical elements and quantify their concentrations based on an in-house calibration established using certified reference materials (Herbelin et al., 2023).

For a comparison, additional XRD analyses were conducted at BRGM, with micronized (McCrone mill) powdered samples using a Bruker D8 ADVANCE diffractometer equipped with a Cu-Kα radiation source and a LynxEye detector. Data were collected over a 2θ range of 4–90°, with a step size of 0.03° and a counting time of 480 s per step. Qualitative mineral identification was performed using DIFFRAC.EVA (Bruker), and quantitative mineralogy was determined by Rietveld refinement using SiroQuant v4.

3. Results and discussion

3.1. Quantification of lithium mineral grades

At Metso, mineral quantification was performed using a conventional geometallurgical approach based on the integration of chemical assays, microscopy, and X-ray diffraction data to establish the basis for element-to-mineral conversion (EMC). The EMC method converts elemental concentrations obtained from chemical analyses into mineral abundances using known mineral stoichiometries and compositional constraints (Lamberg et al., 1997, Whiten. 2008, Lund et al. 2013, Cupido et al. 2026). Calculations were conducted using the Geo module of HSC Chemistry® software. For spodumene and petalite, lithium contents were estimated based on stoichiometry, while lithium concentrations in micas were estimated according to the methodology proposed by Tischendorf et al. (1997). The developed EMC routines provided a rapid and cost-effective means of estimating mineral grades in beneficiation test samples.

In parallel, BRGM developed and applied a combined XRF-XRD analytical workflow for rapid on-site characterization of ores and process products. Comparisons with established laboratory techniques, including EMC calculations, quantitative X-ray diffraction (QXRD), and conventional chemical assays, indicated that the combined XRF-XRD approach produces mineralogical and chemical estimates that are generally consistent with reference methods. The method therefore shows considerable promise as a rapid characterization tool for geometallurgical applications and process monitoring.

3.2. Mineralogy of the ore samples

The dominant gangue minerals identified in all investigated ore and concentrate samples were albite, quartz, and mica-group minerals, including muscovite and biotite. Potassium feldspars, represented by orthoclase and microcline, occurred in varying proportions within the pegmatite ores. Apatite was also commonly present. While the gangue mineralogy was relatively straightforward, the lithium mineralogy proved substantially more complex.

In the spodumene-bearing ores, lithium was hosted primarily by spodumene, with minor contributions from triphylite and trace amounts of secondary cookeite. In contrast, the petalite ore contained a more diverse assemblage of lithium-bearing minerals, including petalite and montebrasite as major lithium hosts, together with minor spodumene and traces of cookeite and eucryptite. The lepidolite ore and concentrate were characterized by lepidolite as the principal lithium-bearing mineral. These observations highlight the significant variability in lithium deportment among European hard-rock lithium deposits and emphasize the importance of detailed mineralogical characterization during flowsheet development.

3.3. Mineralogy of process samples

The primary objective of the ore characterization program was to provide a robust mineralogical basis for the development and optimization of beneficiation flowsheets capable of increasing lithium recovery and enabling the economic utilization of lower-grade resources.

The mineralogical characteristics of Savannah Resources' and Keliber's spodumene ores were broadly similar, with spodumene acting as the dominant lithium-bearing phase and only minor occurrences of triphylite and secondary cookeite. Since spodumene exhibits favorable flotation properties, its beneficiation is relatively well established (Filippov et al. 2019). However, the present study shows that petalite beneficiation remains considerably more challenging due to the close mineralogical similarities between petalite and common gangue minerals such as quartz and feldspars.

Furthermore, the distribution of lithium among multiple mineral phases including petalite, montebrasite, spodumene, cookeite, and eucryptite, introduces added complexity into process design because these minerals show different liberation characteristics, surface properties, and process responses. Consequently, efficient recovery requires more sophisticated beneficiation strategies, while lithium recoveries remain relatively modest compared with conventional spodumene operations.

Conclusions

The characterization of lithium-bearing ores and process products remains a significant analytical challenge because each available mineralogical and chemical characterization technique has inherent limitations. Consequently, reliable quantification of lithium-bearing minerals requires the integration of complementary analytical methods rather than reliance on a single technique.

The results demonstrate that lithium mineralogy in European hard-rock deposits is often considerably more complex than indicated by bulk chemical analyses alone, with lithium distributed among multiple primary and secondary mineral phases. This complexity has important implications for beneficiation performance and process design.

The combined XRF-XRD method developed by BRGM provides a rapid and practical approach for mineralogical and chemical characterization and yields results that are generally consistent with established laboratory methods. In addition, the EMC approach offers a cost-effective means of estimating mineral abundances in process samples during beneficiation studies.

Most importantly, the study demonstrates that detailed mineralogical characterization of both ore and process streams is essential for the successful development of lithium beneficiation flowsheets. Such characterization improves the understanding of lithium deportment, supports process optimization, and contributes to the economic utilization of increasingly complex and lower-grade lithium resources.

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