BATTRACE

Sustainable processing and traceability of battery metals, minerals, and materials

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Preface

Batteries, containing e.g., nickel, cobalt, graphite, and lithium, are essential for a low-carbon society through the electrification of our mobility and energy storage systems. Battery metal production needs to be sustainable, socially acceptable, and traceable. Traceability along all the steps of the supply chain, from mining to downstream uses, is a prerequisite to sustainability certification for batteries. Direct production of battery raw materials from metal concentrates is neither not yet fully optimized.

The scope of the BATTRACE project was to develop technologies for improved traceability and sustainable processing of battery metals, minerals and materials. After three and half years, we have exciting results to present. Global traceability practices were identified and evidence-based forensic traceability methods for battery metals and raw materials based on geological fingerprinting were developed. Processing technologies for the improved recovery of battery metals and higher quality battery metal products (precursors) were designed.

The BATTRACE project was implemented by research institutes VTT and GTK in collaboration with industrial companies Finnish Minerals Group, Sibanye-Stillwater's Keliber lithium project, Metso, Valmet Automation, Latitude 66 Cobalt, Grafintec, Mawson Gold and FinnCobalt. We wish to thank all these companies for their valuable support and advice throughout the project, and Business Finland for funding the project. We trust that the ideas and results presented in this final report are valuable not only to the consortium partners, but to the wider audience of stakeholders along the battery value chain.

Päivi Kinnunen, Principal Investigator of BATTRACE, VTT

Jussi Pokki, Principal Investigator of BATTRACE, GTK

Greetings from the chairperson of the BATTRACE steering group

Format to gather a group of dedicated companies to progress a specific theme has proven again its worth in the form of BATTRACE. Competitiveness of Finnish minerals is not self-evident when nearly all other countries rely on raw material imports. Their needs for the materials are low prices and high availability. This discrepancy generates poor conditions for domestic business, and it tends to favour external produces to gain unfair competitive advantage by operating with lower standards. This needs to stop. It is a requirement for the environment, security of supply, human rights, as well as most of the other sustainability aspects. Tools are not plenty. A few exist – one being traceability and the other being development and adaptation of sustainable technologies. BATTRACE is a unique combination of both.

The design of BATTRACE was driven by a vision of market need and expectations regarding regulatory demands. We were forerunners in this field and our work has been crucial when designing European mineral and battery strategy. Unfortunately, our expectations have not been met. There are no open markets available demanding traceability and differentiating the pricing accordingly. Neither are there forced regulation in place any time soon. However, sustainability premiums exist already in the offtake agreements and supply chains demand visibility all the way to mining. European Battery Regulation also recognizes the need for traceability, and it is expected to be applied in the future stages.

The others have woken up as well. Germany has taken a leading role with their own Battery Passport initiative. Global Battery Alliance (GBA) is building a platform for tracing. GBA is also focusing strongly on carbon reduction and sustainable technologies. We do not need to drag this ship alone anymore. But we do need to stay active and participate.

Whereas traceability proceeds through GBA, EU initiatives and various projects working on the future of sustainable technologies is much more complicated. Hopefully EU´s Carbon Border Adjustment Mechanism accelerates the development. Unfortunately, the development timeline is long. Whereas it takes more than 10 years to establish a mine – the development of a new technology for that mine tends to take more than double of that. I would encourage all of us putting more efforts to this work. Speed-up and focus on the solutions with the most promising potential. Carbon neutrality by 2035 seems quite ambitious in this respect. And when this work hits the markets – traceability also needs to be in place.

I would like to thank you for the great contribution for the success of BATTRACE. I want especially to highlight the increased understanding of traceability as business. This important information is highly valuable also for all the other mineral and battery initiatives. Other important milestones are also fingerprinting development for traceability and development of pipeline to introduce various new sustainable technologies to meet future standards. For these two areas there is lots of work to be done in future. I hope this work finds a form – sooner than later. I also thank the companies and their representatives in taking an active role in project steering and proceeding with their own R&D projects. Sharing this information within the consortium has been important when connecting the pieces in building sustainability and competitiveness for Finnish battery value chain.

Jani Kiuru, Chairperson of the BATTRACE Steering Group, Finnish Minerals Group

Contents

1 BATTRACE project

The BATTRACE project – Sustainable processing and traceability of battery metals, minerals, and materials – was run 1.5.2020−31.10.2023 during three and half years. The public part of the research was conducted by the research institutes VTT and GTK. VTT's focus on the project was on the development of direct battery metal production and on battery precursor materials. GTK focused on developing analytical methods for tracing battery raw materials and assessing related business models. The need for the project came from electric car manufacturers and the European Green Deal initiative, which emphasises the use of sustainably produced raw materials. The work was done in collaboration with BATTRACE industrial companies Finnish Minerals Group, Sibanye-Stillwater's Keliber lithium project, Metso (Outotec), Valmet Automation, Latitude 66 Cobalt, Grafintec, Mawson Gold and FinnCobalt (Figure 1).

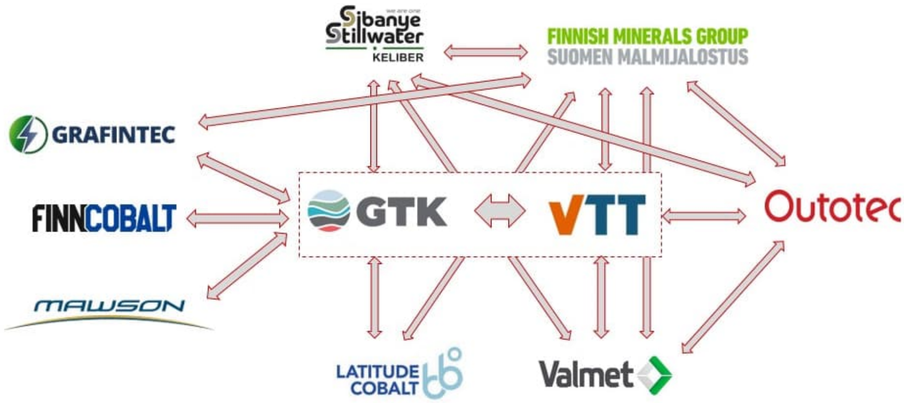

Figure 1. BATTRACE consortium members.

Direct production of battery raw materials from metal concentrates is not fully optimized. The conventional hydrometallurgical production process of nickel and cobalt may contain several leaching, precipitation, and re-dissolution steps. Precursor production from metal sulphate salt involves additional leaching and precipitation steps. It would be beneficial, if battery metal process flowsheets could be simplified to avoid certain intermediate product precipitation and crystallization steps. Using ion-exchange technology instead of precipitation and re-dissolution could be one possibility for more effective and less energy consuming processing.

Potential fingerprinting options along the battery value chain to improve its raw materials traceability are presented in Figure 2. Digital technologies, such as blockchain or QR codes, to control provenance are costly in terms of computing power and face technical challenges related to corruptible data input (‘garbage in, garbage out’), with complex points of aggregation, mixing and processing, thus making the control of material flows difficult. Geochemical and mineralogical fingerprints, on the other hand, cannot be easily corrupted as they are often unique to the source (i.e., ore deposit type and location). Intrinsic mineralogical, geochemical and trace element contents in minerals can be used to discriminate between ore deposit types. The isotopic composition and artificial fingerprinting can be used to trace the source of the metal along the value chain.

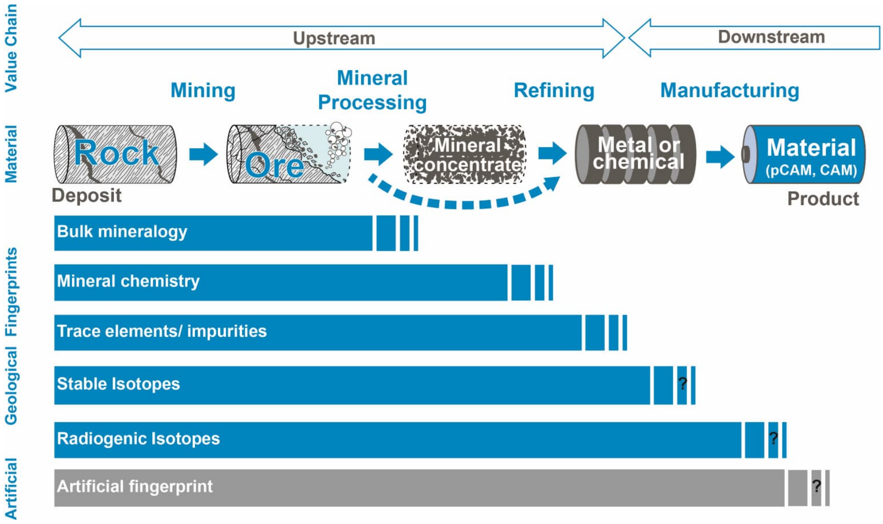

Figure 2. Potential fingerprinting options along the battery value chain to improve its raw materials traceability: the example of cobalt used for the cathode.

2 Traceability

2.1 Traceability methods for cobalt, lithium and graphite production in battery supply chain

2.1.1 General consideration on geo-based fingerprinting

Geo-based fingerprinting, in the present context, is the process of determining the origin of a given sample (ore, concentrate, material/product) by analysing its properties or characteristics. One must differentiate between “natural” fingerprinting, for which these properties are intrinsic to the material (e.g., geochemistry, mineralogy), and artificial fingerprinting, whereby the material is artificially altered (e.g., addition of particles or taggants with unique properties to a shipment, laser inscription and marking of gemstones) in order to certify its origin and ensure traceability.

The natural properties of ores and minerals include a range of geochemical and mineral properties, such as bulk chemistry, mineralogy, and textures, as well as *in situ* properties, including mineral properties such as minor and trace elements, or stable and radiogenic isotopes in specific minerals. These ore properties are linked to the geological setting and geological history of the deposits in which these ores are found. Some of these properties may be site-specific to a deposit or mining district and can therefore be used for traceability. By comparing these fingerprints with a database or reference samples, one can then certify the origin of the sample analysed. The final comparison of these features between the control and reference sample is achieved by applying, often multivariate, statistical methods.

A generalised workflow for the geo-based traceability approach developed in BATTRACE is shown in Figure 3. It consists of three main steps:

- Sample preparation: Depending on the material type (ore, concentrate, product), its nature, and the subsequent analyses, different sample preparation stages may be needed such as crushing, grinding, pulverising, mounting in epoxy-mounts, etc.
- Analyses: The specific analytical workflow and methods used will depend on the type of sample and the specific fingerprints targeted for a given

commodity. Among the methods used in the BATTRACE project, automated mineralogy and laser ablation ICP-MS (LA-ICP-MS) were the most common.

- Data evaluation and analysis: This include a set of data processing and statistical evaluation whereby the analytical results obtained are compared to a reference database of fingerprints from known (ideally certified) origins.

After the data evaluation stage, the origin of the sample can either be certified if the statistical analysis is conclusive or, if the analysis is not conclusive, the sample can be re-analyses with different methods or rejected as non-certified material.

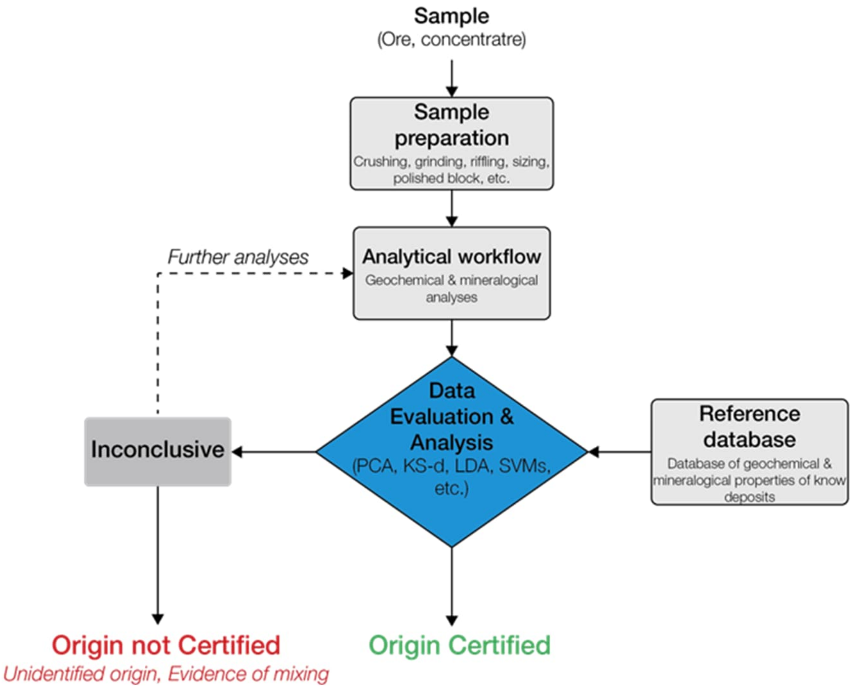

Figure 3. Generalised workflow for geo-based traceability.

2.1.2 Main outcomes and challenges

The main outcomes of the BATTRACE project concerning geo-based traceability of battery raw materials are that such an approach is very challenging for multiple reasons. First, the spatial (within the deposit) or temporal variability (during Life of Mine (LOM)/ production) of some fingerprints can be very large. Next, different sources (*i.e.*, deposits and mines) may have overlapping fingerprints (*e.g.*, isotopic signatures). Lastly, most natural fingerprints (including some isotopic fingerprints) are altered during metallurgy and refining and no longer exploitable.

The results suggest that value chain coverage (from ore to product) varies significantly depending on the commodity, the beneficiation route, and ore types. Some battery raw material has specific fingerprints like stable isotopes that can be tracked along the value chain from any source (*e.g.*, lithium isotopes for lithium and carbon isotopes for graphite) but for other metals like cobalt, only have one stable isotope, and the use other isotopic systems (*e.g.*, sulphur isotopes) can only be used for specific ore types (*e.g.*, sulphide ores). In terms of spatial resolution, isotopic fingerprints are good for differentiating the geological origin (deposit type, metallogenic environment and eventually country), and trace elements for differentiating the geographical origin (district, mine), in some cases.

The analytical workflow developed, relying on LA-(SC/MC)-ICP-MS can be cost-effective if dealing with large volumes of samples with a turnaround time compatible with current traceability systems. However, the current reference fingerprint databases for lithium, graphite and nickel-cobalt are still incomplete. The publicly available data are sparse, with variable data quality and not sufficient for commercial application. We believe that, in the future, such multivariate databases will have to be developed for specific sources (*i.e.*, a single producer) to capture the variability of such fingerprints overtime and will be used with the sole purpose of certifying the declared origin of material coming from that specific source.

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[Poster / Abstract] Kaikkonen, H., Dehaine, Q., Kivinen, M., Bertelli, M., Lahaye, Y., Butcher, A., Liu, X., Shang, Y., Pokki, J. 2023. BATTRACE - Geo-based Fingerprinting for Battery Raw Material Traceability. *SEG 2023*, London, UK.

2.2 Traceability of lithium

Lithium is primarily obtained from two main sources: hard-rock deposits hosting lithium-bearing minerals and lithium-rich brines. Hard-rock lithium deposits consist mainly of lithium pegmatite and to a lesser extent rare-metal granites. Brines are pumped to the surface and concentrated through solar evaporation to obtain an enriched solution. Li-bearing minerals, such as spodumene, are mined from hard-rock deposits and concentrated typically through froth flotation. The concentrates undergo a thermal treatment through calcination or roasting (alpha to beta transformation) before leaching. Once lithium is in solution, it is refined mainly via precipitation, followed by lithium concentration using ion exchange or evaporation. Crystallization, carbonation, or electrodialysis is finally conducted to produce lithium compounds, mainly Li-carbonate (Li_2CO_3) and Li-hydroxide ($LiOH \cdot H_2O$).

Around 47% of the lithium produced in 2022 was from hard-rock deposits in Australia (USGS, 2023). The rest was produced from brines from South America (Argentina, Chile) and China and to a lesser extent hard rocks from China.

2.2.1 Materials and methods

Samples representing various stages along the value chain were collected, starting from raw spodumene ores to spodumene concentrates (alpha and beta) and purified Li hydroxides. These samples were measured by automated mineralogy (QEMSCAN), electron probe microanalysis (EPMA) for major and minor elements in spodumene, laser ablation single collector inductively coupled plasma mass spectrometry (LA-SC-ICP-MS) for trace elements (TE) in spodumene and laser ablation multi-collector ICP-MS for Li isotopes in spodumene (LA-MC-ICP-MS). Trace elements and Li isotopes in the hydroxides were also obtained with solution ICP-MS for comparison with laser ablation.

2.2.2 Main results

The most interesting fingerprints for lithium traceability seemed to be lithium isotopes and TE. Hard-Rock deposits tend to have lower δ^7Li compared to brines. There are however significant overlaps between each deposit type. The δ^7Li values can vary significantly within one deposit type, even within a single deposit. The overlap is less significant when focusing on individual producing operations (e.g., Chinese hard-rock vs Chinese brines) but still present for major producers (*e.g.*, Australian pegmatite & Li-triangle brines). For the vast majority of lithium compounds analysed, it is not possible to tell the country of origin based on Li isotopes only. However, it is possible to estimate the probability of being sourced from a deposit type. The δ^7Li values may be altered along the value chain, especially during hydrometallurgical processes but remain constant in the following downstream processes (NMC synthesis, cell production), see Desaulty et al. (2022).

Using trace elements increases the number of variables from one (δ^7Li) to more than 30 (trace elements), thus enabling the use of multivariate statistical methods. Preliminary results suggest that some trace elements in spodumene can be used to differentiate pegmatite deposits. For refined products such as lithium hydroxide ($LiOH·H_2O$), most suppliers do not specify the sources of Li and it is difficult to obtain samples with a certified and unique source. Given the purity of $LiOH·H_2O$ (99%+), most trace elements are below the part per million (ppm) level. The trace element composition of $LiOH·H_2O$ differs depending on the supplier (origin). More data is needed to evaluate if this is enough to differentiate between different $LiOH·H_2O$ suppliers, but this would require a specific database for $LiOH·H_2O$.

2.2.3 Lithium fingerprints database

The lithium reference database comprises two main data types, trace elements in spodumene and δ^7Li data. Both databases include results generated by the BATTRACE project as well as data collected from the public domain. The mineral chemistry dataset contains 193 records derived from LA-ICP-MS analysis of spodumene from different types of samples representative of the Li value chain. Availability of mineral chemistry data on spodumene in the public domain is scarce probably due to the rather recent interest in Li as a commodity. The δ^7Li dataset includes records relating to both hard-rocks and brines. Results for hard-rock deposits include records from both bulk and in-situ analyses on different sample types covering distinct stages along the value chain (*i.e.*, ores, concentrates, products). Overall, the dataset contains over 700 records from 21 countries.

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2.3 Traceability of nickel and cobalt

Cobalt production mainly occurs as a by-product of copper from sediment-hosted copper deposits or nickel from magmatic nickel sulphide or nickel laterite deposits (Dehaine et al., 2021). The processing route and intermediate cobalt products vary greatly depending on the deposit type and ore type, ore mineralogy and the presence of other by-products. Oxide ores are leached, most of the time without preconcentration, whereas sulfidic ore is concentrated through froth flotation, after which the concentrate is roasted before leaching. Cobalt is then often precipitated as a cobalt hydroxide intermediate product after having gone through several purification process steps. Cobalt from magmatic nickel sulphide deposits is recovered along with nickel through froth flotation as both metals are mostly hosted by the same mineral (pentlandite). The concentrate is then commonly smelted to produce a nickel-cobalt matte, which is then refined through hydrometallurgical processes to produce cobalt powder or cobalt metal. For nickel laterites, cobalt is produced through hydrometallurgical processes, mostly high-pressure acid leaching of limonite ore to produce a mixed nickel-cobalt sulphide product (MSP) or hydroxide product. Depending on specific process design, intermediate or final cobalt products are cobalt sulphide, cobalt hydroxide, cobalt carbonate, cobalt powder or cobalt metal (Dehaine et al., 2021).

2.3.1 Materials and methods

Nickel and cobalt bearing samples along the battery supply chain were collected mainly from Finland. Core deposit and ore samples were collected from four deposits in Finland: Kevitsa, Rajapalot, Juomasuo and Hautalampi. Samples with a large variety of different properties collected along the supply chain are of a known source of Kevitsa. These samples mainly cover the upstream of the supply chain, *i.e.*, from core deposit/ore to mineral concentrate, to solution and residue after leaching and to the nickel sulphate ($NiSO_4 \cdot 6H_2O$). Core, ore and mineral concentrate samples were prepared as epoxy mounts while the processed samples (solution and residue after leaching) were first evaporated as power and then processed as pressed pellets. Nickel sulphate ($NiSiO_4$) was dissolved as solution for S isotope analysis. Core and mineral concentrate samples were analysed by a Micro-X-ray fluorescence (μ-XRF) and X-ray power diffraction (XRD) for elemental mapping and mineralogy phase identification, electron probe microanalysis (EPMA) for major and minor elements in sulphide minerals. Selected sulphide minerals including chalcopyrite, cobaltite, pyrite, pyrrhotite and pentlandite were imaged and positioned by SEM for further trace element (TE) concentrations and S isotopes analysis by SC-LA-ICP-MS and MC-LA-ICP-MS, respectively.

A selection of core samples from the Rajapalot Au-Co project were also analysed by X-ray computed tomography and μ-XRF to produce a 3-D model of cobalt-bearing ore mineralisation to characterise the structural and textural fingerprints of such ore types.

2.3.2 Main results

Developing of geochemical fingerprints for Ni and Co along the battery supply chain is much more complicated in terms of the diversity of hosting deposit types and multiple extraction/ metallurgical stages involved in the various production routes depending on the deposit type. As a pilot study, we started with samples with a known source and try to find robust fingerprints preserved along the value chain. Promising information were obtained from coupled dataset of trace elements and S isotopes of samples from Finland. Based on the preliminary results, trace element ratios are likely to be consistent along the value chain and can be employed to distinguish source ore deposits. Bulk S isotope signature (**δ**^{34}S) inherited from the original ore deposits seems to stay robust during mineral processing, pyrometallurgy and hydrometallurgical processes. The **δ^{34}S** signature changed significantly during the precipitation of $NiSO_4$ when external sulphuric acid was added. Laboratory measurement for the TE and S isotopes of some samples are still ongoing. More information will be obtained by the effort of multivariate data analysis on the large geochemical dataset generated from above samples.

In addition, the combination of XCT with μ-XRF imaging used for this study provides a powerful non-destructive method for studying ore forming processes and provides detailed textural information that can be used for geological fingerprinting and traceability (Sayab et al., in prep).

2.3.3 Nickel-cobalt fingerprints database

The nickel-cobalt database includes mineral chemistry data of sulphide minerals and sulphur isotope (**$\delta^{34}S$**) fingerprints, including results from the BATTRACE project, data gathered from the public domain as well as confidential data produced by the GTK laboratory in Espoo. In contrast with the lithium fingerprints, in the case of nickel, there is a wealth of data both for mineral chemistry and sulphur isotopes albeit with some heterogeneities in terms of mineral and isotopic systems analysed across different deposits and districts. The database containing the sulphur isotopes data includes about 2300 records representative of 8 countries. The mineral chemistry database contains compositional data (63 elements) for over 4000 records derived from 18 distinct minerals representative of 7 countries.

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2.4 Traceability of graphite

Battery-grade graphite can be produced from two primary sources: natural graphite or synthetic graphite. Among natural graphite, only flake graphite finds application in battery production. Besides flake graphite, other forms of natural graphite include amorphous (microcrystalline) graphite and vein (lump) graphite.

On the other hand, synthetic graphite is produced through a complex manufacturing process, using petroleum coke or other carbon-rich materials. Graphite processing consists mainly of mechanical processes (sorting and sizing) as well as froth flotation to obtain concentrates with the desired particle size distribution and remove impurities. This is followed by purification and refining to meet the stringent quality requirements for lithium-ion battery applications. Graphite is mined predominantly in China (65% in 2022), and Madagascar, Mozambique, Brazil, and India (USGS, 2023). This dominance is even more pronounced for refined graphite with nearly all spherical graphite supply ensured by China in 2022 (Benchmark, 2022).

2.4.1 Materials and methods

Samples representing various stages along the value chain were collected, starting from raw graphite ores, concentrates, purified graphite, micronized graphite and purified spherical graphite. These samples were measured for bulk mineralogy by X-Ray Diffraction (XRD), for trace elements (TE) in graphite using laser ablation single collector inductively coupled plasma mass spectrometry (LA-SC-ICP-MS) and gas-mass-spectrometry bulk analysis at the University of Leoben (Austria), to analyse the carbon isotopic signatures ($\delta^{13}C$).

2.4.2 Main results

The most interesting fingerprints for graphite traceability seemed to be carbon isotopes and trace elements. Results show distinctive isotopic signatures associated with graphite from different geological formations. Isotopic ratios ($\delta^{13}C$) offer insights into the geological history and environmental conditions during graphite formation and different ore types are characterised by specific $\delta^{13}C$ values. In particular, lump or vein graphite from Sri Lanka, does have a characteristic high $\delta^{13}C$ value. Nevertheless, some deposits exhibit remarkable uniformity in their isotopic characteristics, even to the extent of overlapping with signatures observed in other graphite types, notably flake graphite. Consequently, carbon isotope fingerprints has the potential to be used for differentiating certain deposits of the lump graphite category but a substantial proportion of flake graphite deposits do have overlapping $\delta^{13}C$ signatures.

Preliminary results suggest that trace elements composition of graphite samples can serve as a powerful indicator of their geological origin. For some deposits, the presence and concentration of specific trace elements allow to identify the graphite origin. Alternatively, the use of wide spectrum of trace elements in multivariate statistical analyses allows to discriminate between graphite samples sourced from diverse geological environments or even between different deposits.

Developing and preparing samples for this parameter pose significant challenges. Ablating particles from pressed powder pellets of graphite concentrate for LA-ICP-MS analysis is particularly challenging, and the lack of a suitable matrix-matched reference material requires method development and optimization, including the creation of a proprietary reference material.

2.4.3 Graphite fingerprints database

The graphite database includes data on carbon isotopes (δ13C) and compositional data obtained from graphite sourced from the public domain. The former table contains 512 records representative of carbon isotopic data from 8 countries. The graphite compositional table includes data on 28 elements for 10 records derived from analyses of graphite samples from 9 countries and 10 different deposits.

Publications
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2.5 Business models for fingerprinting

Traceability has already become an integral element for many global industries with complex supply chains. The relevant maturity of these supply chains from a logistical standpoint has enabled them to develop differential value through traceability, which cannot be said for battery raw materials. Although the need for raw material traceability is coming from consumers and regulatory agencies, the same is not yet evident from the original equipment manufacturers (OEMs).

Increased costs are a clear hindrance to the adoption of traceability, but there are more pressing considerations for the OEMs. If the supply gap for the battery raw materials grows too large, OEMs may have to source raw materials wherever they get them and might not want to disclose their sources.

Widespread adoption of traceability is dependent on financial incentives for the manufacturers and raw material producers. Already, the producers of sustainable and low emission raw materials have identified the commercial benefit of tracing the greenhouse gas emissions of their products along the supply chain. This may be further highlighted with future CO_2 pricing regulations, such as the EU Carbon Border Adjustment Mechanism (CBAM).

There may be additional fracturing for the commodities market in the future with different pricing for sustainable raw materials. Still, as the price for sustainability is still revolving around the raw materials' CO_2 emissions, these developments may not help with the ethical considerations surrounding cobalt mining, for example.

The ethical considerations of consumers are still valued by the manufacturers through their bottom line.

There are several options for commercializing any new technology, and this is no different for fingerprinting. Any commercial applications for traceability are heavily dependent on the development of traceability of raw material origin requirements in legislation, such as the EU battery directives or US inflation reduction act (IRA). Commercial applicability is also driven by end user preferences and the business potential of traceability in general.

2.5.1 Market drivers for fingerprinting

Potential markets for a commercial fingerprinting solution were analysed and reviewed as part of project. Traceability services can be seen as part of wider supply chain security markets, for which there is data available regarding market size and future potential. Reports estimate the global supply chain security market being valued at $2 billion in 2021, and to reach $6,3 billion by 2031. Obviously, fingerprinting is only a small niche piece of the wider supply chain security market. Highly successful business models for fingerprinting are yet to emerge.

Currently, verification of geographical origin leans on voluntary initiatives. There is no shortage of such initiatives. However, it is reasonable to assume that any voluntary actions likely have less effect on market pricing mechanisms for example, than regulatory or compulsory ones.

Regarding commodity pricing it seems apparent that so called “green premiums” are not yet in regular use for most commodities. Such premiums are being used for more mature steel and aluminium markets, but less so for other commodities, like battery raw materials. The supply gap severity is highly dependent on the metal in question, but e.g. lithium and nickel markets will face serious supply challenges in the future. Such market developments may hinder or at worst prevent formation of green premium pricing.

The current regulatory or market conditions do not necessitate the companies to investigate or verify their raw material traceability. General awareness of the topic is more widely known nowadays than before, but actual measures by the companies are more up to voluntary efforts than regulatory or market driven drivers.

2.5.2 Commercial pathways for fingerprinting

A full commercial business plan for fingerprinting was not created during the project, as the technological readiness level of fingerprinting is not yet sufficient to create one in high level detail. Differences in the element-specific methods for fingerprinting also create difficulties in considering the commercialization opportunities for the technology.

A competitor benchmarking revels that there are several companies around the world doing business with traceability methods similar to geo-based fingerprinting.

Identified companies are private and mostly rather small. Like for any private companies, reliable financial documentation is difficult to obtain which makes benchmarking difficult. Still, the revenues for these companies are small and mostly they operate on limited geographical coverage currently. Hence, the business is more regional than global.

Currently, other commodities (produce, textiles, agriculture) seem to be the main business sectors for provenance services. For many companies, the traceability of metals is not included at all in their service portfolio. The size and type of companies providing traceability services clearly indicates that this market segment is yet evolving and rather immature. Based on the competitor benchmarking it has been very roughly estimated that the current fingerprinting market globally is a few tens of millions USD at most.

The establishment of “full service” start-up company with dedicated equipment for fingerprinting is not seen as a viable option. A traceability service utilizing fingerprinting requires extensive and dedicated laboratory equipment with large investments that easily exceed millions of euros, up to 10 million euros. Furthermore, at least 5-7 experts would be required to cover only the laboratory services. In addition, the actual traceability expert(s) would be required as well as sales personnel and administrative personnel or services. To cover the operating costs and pay back the investments within reasonable timeframe, the revenue should be several million euros annually. Considering the benchmarked companies and overall market analysis, achieving such revenue and marker share seems to be unrealistic. With this business model, the operations would be heavily laboratory service focused with many associated downsides.

A fingerprinting-as-a-service (FAAS) business model would in theory be an attractive approach. In a FAAS model, a customer could utilize third-party laboratory services to provide a set of analysis for a material sample, which would then be sent to the FAAS provider for the verification of the sample’s origin. This would minimize the investments into in-house equipment and staff. However, operations according the FAAS model would require well-established, and in-detail specified testing protocols for each commodity and material in question. The FAAS model would best safeguard the analysis methods and understanding of specific markers as many individual laboratories could be used all over the world. Third-party laboratories would basically have no idea about the purpose of the analyses being undertaken.

It is very clear that the reference library is the most important single item regardless of the chosen business model. The library which is preferably continuously updated and expanded is the core asset of any company providing a fingerprinting service. Without such library the service cannot be provided at all. Similarly important is naturally the ability to interpret the library and each analysed new sample, to provide professional opinion for the sample origin.

In addition to new business ventures, fingerprinting can potentially be seen to complement the existing service portfolio of GTK. This option is based on GTK existing commercial services that could be expanded a with fingerprinting service. GTK is a major player on mineralogical and isotope study services and especially minerals processing studies at GTK Mintec processing pilot plant. Traceability verification would nicely complement the current service portfolio as it could be offered as extra package for each incoming process pilot samples. Fingerprinting could be offered as standalone product as well.

The goal of the commercialization pathway would be a dedicated company operating according to a FAAS model. At best, such service could be expanded to cover tailored product packages. Likely the bulk of the traceability services would be individual service requests invoiced by certain pricing principles. However, it might be possible to acquire constant (and increasing) cash flow from the customers if some sort of access to the reference library would be provided to the customers willing to pay extra. The access could be invoiced as a lump sum payment (access to the reference library at the point of the payment) or as a recurring payment or license fee that would provide access to the latest version of the reference library that is preferably updated on regular basis. Such services are already available in minerals industry, although their purpose is slightly different.

For the moment, the addition of fingerprinting to complement GTK's service portfolio seems to be the most realistic commercialization avenue, although with limited commercial potential. Even this requires better understanding of the fingerprinting applicability to the different commodities, deposit types and sample matrixes. The research and development work continues in other projects that have been initiated already, the Horizon Europe MaDiTraCe project being the focus in the near future. Further research is required to create a more comprehensive business plan for fingerprinting in the future.

Publications

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3 Sustainable processing of battery raw materials

3.1 New process concept as alternative to conventional process route

Conventionally, nickel sulphate is produced from intermediate or refined nickel products, which have been further directed to additional metallurgical processes to attract a premium price (Fraser et al., 2021). Due to the demand in the battery field, the short-term solution can be to refine the nickel sulphate product from the nickel intermediates. However, in the long-term new nickel class I supply projects and technologies are needed. Further, the current processes including intermediates have not been optimized for nickel sulphate production and may contain several leaching and precipitation steps, which may be avoided with a novel process design.

3.1.1 Materials and methods

Literature review concerning conventional processing of battery raw material was made. The aim was to find ways to simplify conventional processes when the product is battery material instead of pure metal. Based on the available information, a flowsheet for direct production of battery raw material was designed.

3.1.2 Main results

A process flow sheet consisting of particle size reduction, leaching, filtration, iron removal, copper removal via chemical precipitation, and nickel and cobalt sulphate recovery with ion exchange and crystallization was designed (Figure 4). Another alternative for ion exchange is solvent extraction.

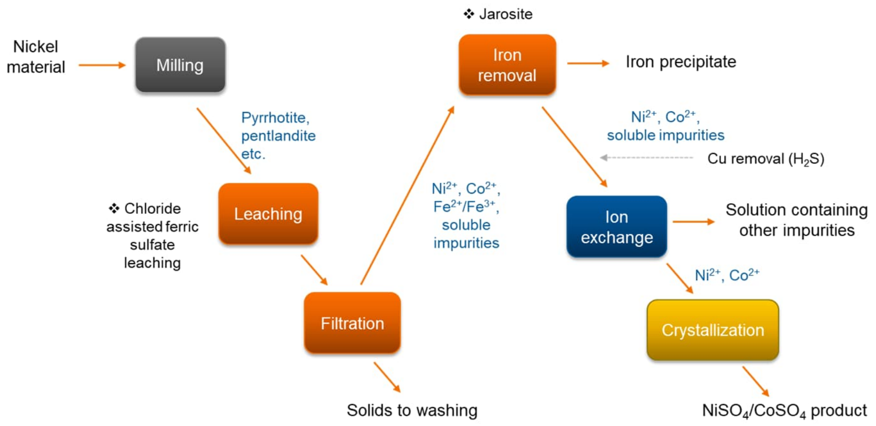

Figure 4. Process concept to recover nickel and cobalt as sulphates from nickel concentrate.

Publications

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3.2 Process development

In the process development, the aim was to simplify process flowsheets for direct production of battery grade nickel sulphate. First, laboratory scale development was done in batch mode for separate unit processes (Figure 5). The process flowsheet would be simpler, if nickel and cobalt would be recovered directly from leach solution using ion exchange, so ion exchange was in the focus of this study. This kind of process would be suitable for low grade nickel ore that cannot be treated with conventional methods. Second, all unit processes were combined into a continuously operated pilot to produce data for a process model, knowledge about chemicals consumptions and carbon footprint, and to obtain samples to study the traceability of nickel along the hydrometallurgical process.

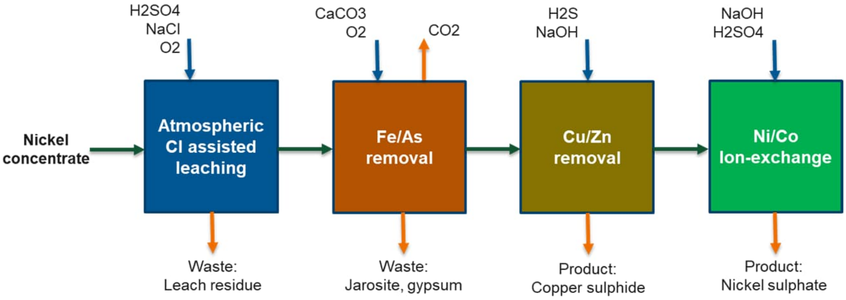

Figure 5. Studied unit processes along the direct hydrometallurgical nickel sulphate production.

3.2.1 Materials and methods

Kevitsa nickel-cobalt concentrate (Table 1) was used as a case material in the studies. Three different leaching methods were studied; sulphuric acid leaching, chloride assisted ferric sulphate leaching and bioleaching. Chloride assisted ferric sulphate leaching was selected for further studies based on the leaching yields and kinetics to study the effects of temperature and chloride concentrations on leaching. Iron removal was conducted at 85°C and at pH 1.8 ($CaCO_3$) with 5 hours retention time. Sulphide precipitation was done in different reactor types at different temperatures to determine the acceptable residual copper concentration in solution versus the losses of nickel and cobalt. Ion exchange was studied in batch and column experiments to determine the contact time, selectivity, loading capacity, temperature, and acid concentration and contact time in elution. Examples of different experimental set-ups are shown in Figure 6.

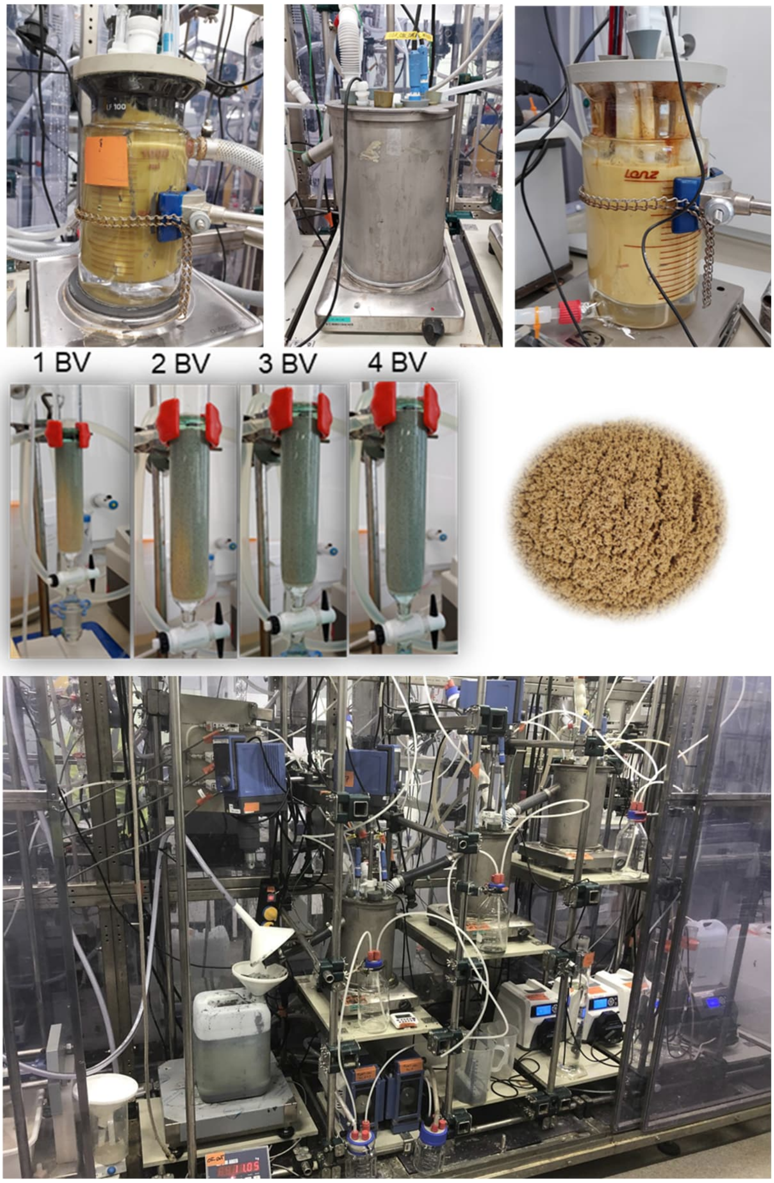

Figure 6. Leaching and iron precipitation reactors, ion exchange columns and continuously operated leaching pilot configuration used in the experiments.

3.2.2 Main results

In bioleaching, 91% nickel and 72% cobalt yields were obtained in 12 days. With chloride assisted (20 g Cl^-/L) ferric sulphate leaching, 92% nickel and approximately ~100 % cobalt yields were reached. Temperature of 90ºC and chloride addition resulted in highest leaching yields in 8-hour retention time in the reactors. Jarosite precipitation also removes part of sodium from the solution. Iron removal with 99% efficiency was obtained to decrease the amount of iron from 50 g/L to 0.5 g/L in the solution. Only minimal losses of nickel and cobalt to the iron precipitate were observed. Copper removal as CuS with sulphide precipitation at high temperatures (90ºC) showed loss of nickel and cobalt which can be avoided at lower temperatures. All ferric iron in the solution was completely reduced to ferrous iron in all tests. Sulphide precipitation can cause challenges in residual zinc concentration and result in nickel losses. Nickel and cobalt recovery with ion-exchange was not optimal with the produced solutions. In the real application, solvent extraction would be a more likely process alternative and therefore it was selected for process modelling.

Continuous piloting showed that direct hydrometallurgical nickel sulphate production is technically feasible from nickel concentrate. It is noteworthy that the used nickel concentrate is tailored and very suitable for the pyrometallurgical route. In general, hydrometallurgical process alternatives can utilize those materials that cannot be treated with pyrometallurgical routes and recover by-product elements.

Publications

[Oral presentation and abstract] Heikola, T., Salo, M., Mäkinen, J., Dehaine, Q. & Kinnunen, P. 2023. Comparison of different leaching methods on the recovery of valuable metals from nickel concentrate. Sustainable Minerals ’23 conference, 7-8.6.2023, Falmouth, UK.

[Article] Heikola, T., Salo, M., Mäkinen, J., Dehaine, Q., Bertelli, M., Kinnunen, P. Comparison of chemical and biological leaching methods with the effect of chloride on the recovery of nickel, cobalt and copper from nickel concentrate. Submitted to *Minerals Engineering*.

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[Article in prep.] Ollonqvist, P.P. & Porvali, A. Continuous selective copper recovery from the pretreated pregnant leach solution.

3.3 HSC-Sim Modelling

Process models provide information, such as process stream volume flows, reagent consumptions, concentrations, mass balance and heat balance for further process calculations. In addition, modelling gives information and guidance about the hot spots and focus areas for the process development. Modelling work was based on experimental results whenever there was experimental information available.

3.3.1 Materials and methods

Conventional pyro-hydrometallurgical process route via a nickel matte intermediate and the novel direct nickel sulphate process route were modelled. Process simulation was done with HSC-SIM chemistry software (version 10.0.1.8) based on the complete mass and energy balance simulation to obtain chemical consumptions and production figures in a steady state operation. Identical nickel concentrate feed composition was used for annual 50 000 tons feed rate. The designed nickel process consisted of chemical leaching, impurity removal by precipitation, solvent extraction, and crystallization. Solvent extraction was selected for process modelling instead of ion exchange, since the experimental results supported the selection of solvent extraction as the most likely alternative for the produced solution.

3.3.2 Main results

Based on modelling, chemical consumptions were higher with the hydrometallurgical process in comparison to pyro-hydrometallurgical route. The highest chemical consumption in the direct hydrometallurgical process route was due to oxygen usage in leaching and to calcium carbonate used in iron precipitation as jarosite. Since the high chemical consumption was mainly due to iron leaching and precipitation resulting in large waste production, iron management in the hydrometallurgical process development is crucial for economic and environmental benefits. It is noteworthy that hydrometallurgical technologies can also recover by-product metals and they are feasible to operate in a smaller scale. The results highlight the need for pre-treatment in the hydrometallurgical process route if the material contains high amounts of pyrrhotite, which is leached before valuable metal containing minerals such as pentlandite.

Publications
[Article] Kinnunen, P., Riihimäki, T., Kinnunen, K., Salo, M., Heikola, T., Mäkinen, J. Process design for direct production of battery grade nickel sulphate. Submitted to *Journal of Sustainable Metallurgy*.

3.4 Chemical consumption and CO_2 footprint

Chemical consumption calculations for conventional process method and new process concept were made. In addition, sustainability of conventional process and new process concept was evaluated based on chemicals consumption. In the PEFCR for Batteries (Product Environmental Footprint Category Rules), the raw material acquisition is identified as the most relevant part for various categories (i.e., climate change, resource use, respiratory inorganics) (Recharge, 2018). Environmental assessment during the process development phase identifies the hot spots and gives feedback to process planning.

3.4.1 Materials and methods

Process simulation was done with HSC-Sim chemistry software (version 10.0.1.8) based on the complete mass and energy balance simulation to obtain chemical consumptions and production figures in a steady state operation. Life cycle assessment (LCA) according to ISO 14040 and ISO 14044 standards was used to evaluate the carbon footprint of the process. The functional unit was one ton of battery grade nickel sulphate hexahydrate.

3.4.2 Main results

In the developed direct hydrometallurgical process route, the biggest impacts on the carbon footprint were associated with the iron precipitation step and the second largest impacts came from the oxygen use in leaching (Figure 7). In the future process development, especially pyrrhotite removal prior to leaching step needs to be considered to reduce the carbon dioxide footprint. Also, the zero-carbon chemicals may change the carbon footprint evaluations in the coming years. Novel iron and sulphur management methods are suggested to prevent the excess use O_2 and $CaCO_3$ and to produce iron and sulphur products instead of stockpiling significant amounts of waste.

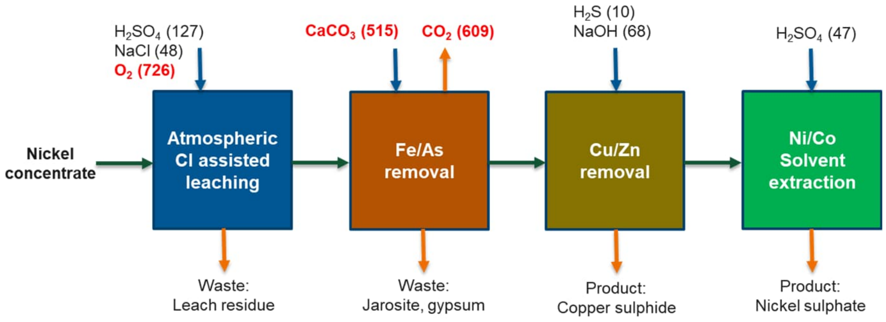

Figure 7. Carbon footprint of input chemicals (kg CO_2-eq/ ton Ni sulphate).

3.5 Precursor particle size control

Precursors are raw materials used in manufacturing cathode active materials in batteries. Particle size of cathode active material has a significant effect on the properties of batteries. The particle size depends on the parameters of precursor precipitation, such as concentrations, pH, temperature, retention time and reactor configuration. This work involved co-precipitation synthesis of mixed metal hydroxides.

3.5.1 Materials and methods

First, a literature survey was conducted. The preliminary experiments studied particle aggregate morphology as a function of chosen parameters and tap density. More advanced studies focused on the particle chord length distribution and count measurements during the synthesis.

Nickel-cobalt-manganese atomic stoichiometry of 1:1:1 (I.e. NMC-111) was chosen for the study. The synthesis requires well-controlled experimental conditions (T, atmosphere, reagent feeds) and relatively long reaction times. The work involved two separate series of experiments: exploratory ramp-up experiments performed in a 1 L reactor setup in which the technical and physicochemical issues were observed. Careful control of feeds and atmosphere was of significant importance in the synthesis. The challenges were resolved by improving the synthesis setup, after which continuous co-precipitation was performed in a setup consisting of 3 L synthesis reactor, 1 L measurement reactor and collection vessel (Figure 8). Both semi-batch experiments as well as continuous experiments were done.

Figure 8. Experimental set-up in the precursor precipitation.

3.5.2 Main results

At best, tap density of approximately 1.92 g/cm^3 was obtained with larger than desired particle size (D50 = 25.4 µm). Focused beam reflectance measurement (FBRM) was utilized *in situ* in monitoring the change in particle population (counts of chord length classes as well as chord length distribution (CLD)) as the experiments progressed. The results were compared to particle size distributions measured with laser diffraction. FBRM can give indication of the trends in the system. Overall, similar trend in change (PSD vs CLD) was observed. Further studies should attempt to mathematically translate CLD to PSD in order to ascertain whether the CLD is a good metric for particle size distribution in precursor synthesis.

Publications
[Article in prep.] Porvali, A. & Ollonqvist, P.-P. Exploratory and continuous experiments on co-precipitation of nickel-manganese-cobalt hydroxides (NMC-111).

4 Conclusions

The traceability component of the BATTRACE project focused on geo-based traceability of battery raw materials and encountered significant challenges. The study identified numerous challenges for using natural geological and geochemical fingerprints to track and trace battery raw materials such as (i) the substantial spatial or temporal variability of fingerprints within deposits, (ii) overlapping fingerprints from different sources, and (iii) alterations of natural fingerprints during metallurgy and refining processes.

The research emphasized that the effectiveness of tracing raw materials along the value chain (from ore to product) depends on factors like commodity type, beneficiation route, and ore characteristics. While some battery raw materials, such as lithium and graphite, have specific fingerprints like stable isotopes that can be tracked across the value chain, others like cobalt rely on less versatile isotopic systems. Isotopic fingerprints were found to be useful for differentiating geological origin (deposit type, metallogenic environment, and country) and trace elements for distinguishing geographical origin (district, mine).

The analytical workflow developed using LA-(SC/MC)-ICP-MS was deemed cost-effective for handling large sample volumes within the timeframe of existing traceability systems. However, the study highlighted the incomplete nature of current reference fingerprint databases for lithium, graphite, and nickel-cobalt. Publicly available data were sparse, varied in quality, and insufficient for commercial application.

For future development, we proposed to expand the existing databases consisting of sparse fingerprints from various sources to multivariate databases tailored to specific sources, aiming to capture the variability of fingerprints over time. These databases would be utilized to certify the declared origin of materials from a particular source, emphasizing the need for comprehensive and reliable reference datasets for effective traceability in the battery raw materials supply chain. These databases would also need to developed not only for raw materials (ores and concentrates) but also intermediate and final products. Improving data quality and representativity is also of paramount interest. Future work should also investigate the impact of metallurgy and refining processes on natural fingerprints through experimental investigations to unveil how these processes, along with varying operating conditions, affect the materials fingerprints. The influence of mixing and aggregation should also be investigated to assess if these processes can be modelled, and the relative contribution of each source evaluated based on the individual sources and mixed fingerprint. In cases where traditional natural fingerprints are compromised or unavailable, artificial fingerprinting, which is already used in other industries could also be investigated for battery raw materials. This involves the use of micro-taggants or tracers, possessing specific properties and compositions, which can be detected using in/on-line or portable technologies.

Regarding the commercialization of geo-based fingerprinting, the adoption of traceability in global supply chains has become crucial, though its implementation in battery raw materials lags due to several concerns. Financial incentives could drive its widespread adoption, especially with the potential impact of CO_2 pricing regulations like the EU Carbon Border Adjustment Mechanism (CBAM). However, while such initiatives may address environmental concerns, they might not fully address ethical considerations such as those surrounding cobalt mining. The commercial viability of traceability, including fingerprinting technology, hinges on legislative requirements, end-user preferences, and broader business potential within supply chain security markets. Despite the global supply chain security market's growth, fingerprinting remains a niche segment, with successful business models yet to emerge.

Current market conditions do not mandate companies to verify raw material traceability, leaving such efforts primarily voluntary. While various initiatives exist, regulatory or compulsory measures are likely to have more significant impacts. Additionally, the absence of widespread adoption of "green premiums" for commodities like battery raw materials complicates pricing mechanisms. The establishment of fingerprinting-as-a-service (FAAS) models could reduce barriers to entry, although challenges remain in standardizing testing protocols and maintaining comprehensive reference libraries. For now, integrating fingerprinting into existing service portfolios, such as those offered by GTK, appears to be the most feasible commercialization avenue, albeit with limited potential. Further research into the technology and development efforts will refine the usable business strategies and fully realize the commercial potential of fingerprinting technology.

In the hydrometallurgical process development side, direct production of metal sulphates without intermediate products has the potential to simplify process flowsheets. The results show that pre-treatment in the hydrometallurgical process route is needed if the material contains high amounts of minerals which are leached before valuable metal containing minerals. Iron and sulphur management methods in hydrometallurgical process development are needed to prevent the excess use O_2 and $CaCO_3$ and to produce iron and sulphur products instead of stockpiling significant amounts of waste.

The *in situ* or *semi in situ* chord length tracking method could be used for monitoring precursor co-precipitation either in the batch or continuous precursor process.

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Series title and number

VTT Technology 424

Title	**BATTRACE** Sustainable processing and traceability of battery metals, minerals, and materials
Author(s)	VTT: Päivi Kinnunen, Marja Salo, Tiina Heikola, Antti Porvali, Teppo Riihimäki, Kalle Kinnunen, Pirkka-Pekka Ollonqvist & Jarno Mäkinen GTK: Jussi Pokki, Quentin Dehaine, Martina Bertelli, Yann Lahaye, Yuan Shang, Harri Kaikkonen & Mari Kivinen
Abstract	The BATTRACE project – Sustainable processing and traceability of battery metals, minerals, and materials – was run 1.5.2020–31.10.2023. The public part of the research was conducted by the research institutes VTT and GTK and the main outcomes of the work were summarized in this book. GTK focused on developing analytical methods for tracing battery raw materials and assessing related business models. Global traceability practices were identified and evidence-based forensic traceability methods for battery metals and raw materials based on geological fingerprinting were developed. VTT's focus on the project was on the development of direct battery metal production and on battery precursor materials. Direct production of metal sulphates without intermediate products has the potential to simplify process flowsheets.The *in situ* or semi *in situ* chord length tracking method could be used for monitoring precursor co-precipitation either in the batch or continuous precursor process.
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BATTRACE

Sustainable processing and traceability of battery metals, minerals, and materials

The BATTRACE project – Sustainable processing and traceability of battery metals, minerals, and materials – was funded by Business Finland during 1.5.2020–31.10.2023. The public part of the research was conducted by the research institutes VTT and GTK. The scope of the BATTRACE project was to develop technologies for improved traceability and sustainable processing of battery metals, minerals, and materials. Global traceability practices were identified and evidence-based forensic traceability methods for battery metals and raw materials based on geological fingerprinting were developed. Processing technologies for the improved recovery of battery metals and higher quality battery metal products (precursors) were designed.

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