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Extraction of lithium from β -spodumene using potassium chloride and hydroxide

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ABSTRACT

The extraction of lithium from spodumene requires decrepitation of α -spodumene at 1100°C, acid bake of β -spodumene at 250°C, and several refining steps that make the process equally energy, feedstock, and by-product intensive. The refining of spodumene with sulfuric acid forms sodium sulfate (Na_2SO_4) as a low-value by-product, and it also generates substantial volumes of hydrogen aluminosilicate (HAlSi_2O_6) that necessitate disposal. New technologies need to retrieve lithium from spodumene with less energy and chemical consumption. From this standpoint, we investigated two processes to extract lithium from β -spodumene at moderate and alkaline conditions using potassium chloride with and without potassium hydroxide. We observed low lithium recovery from leaching β -spodumene with KCl at circumneutral pH and 200°C, with XRD spectra indicating recrystallisation of β -spodumene into leucite which progresses at slow rates. This is because the ionic radius of K^+ is 2.6 times larger than that of Li^+ , preventing the reaction to advance via the anticipated ion-exchange mechanism to produce K-keatite (K-aluminosilicate). However, as expected, leucite forms more rapidly when β -spodumene is leached with KCl and KOH at pH~13. We conclude that the alkaline leach condition with KOH enhances the recrystallisation process of β -spodumene into leucite, but the process temperature needs to be raised to about 250°C to increase the rate of lithium extraction.

Experienced explorationists are the key to unlocking potential WA lithium projects

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ABSTRACT

In recent years there has been a rapid global transition to green energy as mining companies attempt to reduce carbon emissions and decarbonise their assets. The lithium industry has experienced strong economic growth since 2010, due to the increased demand for electric transportation and the need for static storage of renewable wind and solar energy sources. The presence of lithium in pegmatites has created opportunities for mining companies in Western Australia, however, these projects have tended to lack experienced explorationists during the exploration and development phases. It has generally very much been the case of 'you don't know, what you don't know'.

Lithium companies in WA now have an opportunity to learn from the efforts of others during earlier and the current lithium booms to make strategic changes in their exploration techniques. There needs to be a rethink on the methodologies used by explorationists and the skill sets required to identify amenable orebodies including a focus on process mineralogy. Educating geologists about the need to differentiate between the collection of data and the generation of information, is the key to unlocking potential lithium deposits.

Lithium brines reserves and resources in Northern Chile

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ABSTRACT

Northern Chile is characterised by a succession of north–south trending ranges and basins occupied by numerous saline lakes and salt crusts, collectively called salars.

Discovery of lithium in the Salar de Atacama of northern Chile during a study in 1969, initiated the identification of one of the world's major continental regions of lithium brines. Today, the Central Andes offer a significant portion of the world's Li production.

The principal producer of lithium from brine is Chile. In 2020, produced 18 000 tons of lithium metal, some 22 per cent of the world's total of 82 000 tons of lithium (t Li). Chile's total cumulative production during the last 35 years was 246 956 t Li.

Chile became a major producer due to the large size of the Salar de Atacama and the high quality of lithium brines.

Lithium rich brines are predictable over a large area of Northern Chile from which some 59 occurrences have been reported so far.

Here, we are counting only 23 deposits that include exploration or evaluation efforts. Of these cases, only two; the Salar de Atacama and the Salar de Maricunga are the subject of evaluation work with enough detail to be able to define lithium reserves. Recent numbers are 10 879 000 t corresponding to almost 52 per cent of world reserves.

Reports, evaluations and exploration surveys on 23 salars completed during the last decade provide an updated view on the current situation of lithium brines. This revision analyses several studies in order to better understand Chile's remarkable current and future situation in relation to lithium brine reserves, resources and exploration.

Evaluation of coarse particle flotation technology for spodumene

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ABSTRACT

With the increasing development of electric cars, mobile devices, and other electronics, the demand for battery material has also increased. As lithium is one of the critical materials to produce green batteries, its value has increased as has the need for its exploration and production. Fluidised bed technology can enhance the recovery and flotation efficiency of coarse particles. This has been successfully implemented on an industrial scale for many years using Eriez' HydroFloat® separator in the potash, phosphate and sulfide industries and most recently has been utilised for lithium minerals. Eriez HydroFloat® separator combines the principles of flotation and hindered settling in a fluidised bed to create a high capacity, highly selective flotation unit. A unique aeration system disperses ultrafine bubbles directly into the reagentised fluidised bed environment, significantly enhancing the bubble-particle collision rates. Additionally, having no axial mixing or froth zone, bubble detachment is greatly reduced. As a result, only minimal surface exposure is required for flotation, allowing the HydroFloat® to achieve high recoveries of poorly liberated particles up to 2 mm in diameter. The benefits of coarse particle flotation (CPF) vary depending on the position within the flow sheet. Early gangue rejection offers the most economic gains in terms of increased circuit capacity, reduced throughput and hence energy consumption in the grinding circuit and safer tailings disposal. A tailings scavenger approach enhances the overall plant recovery by recovering the coarse value-bearing particles that are lost to conventional flotation tailings. Combined with Eriez's CrossFlow® separators for precise size classification, both upfront and back-end CPF units offer economic and environmental benefits to processing plants. This paper examines both scenarios using real test and plant data from various Eriez HydroFloat® installations for spodumene applications around the globe, along with calculations, to demonstrate these benefits.

Australia set to recharge lithium battery recycling with launch of Bcycle, national battery stewardship scheme

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ABSTRACT

In the World Bank's 2020 *Minerals for Climate Action Report*, it estimates demand for battery minerals, including lithium, cobalt, copper and zinc, is set to increase 500 per cent by 2050. This increased demand is required to ensure a renewable energy future for Australia and the globe. Conventional strategic approaches to meet this increased demand have included further mining and technology efficiency improvements. However, there remains another underexploited opportunity globally, and especially in Australia, that is the recovery or recycling of these valuable materials from existing/legacy used batteries.

Australia's battery recycling rate has been historically very low, the lowest of all OECD countries, with less than 10 per cent of all imported and used batteries recycled. To disrupt the currently low recycling status-quo, Australia's first national stewardship scheme for batteries, B-cycle, launched in Jan 2022.

Unlike many European and North American schemes, and although supported by the Australian Government, accredited under their National Product Stewardship Scheme legislation, and authorised by the Australian Competition and Consumer Commission (ACCC), B-cycle is, a voluntary stewardship scheme.

B-cycle works across the battery value chain, including manufacturers, importers, users, collectors/transporters, recyclers and secondary processors, to incentivise the battery recycling industry to scale up battery recovery, especially electric vehicles (EVs) and energy storage batteries, through voluntary levies collected at import into Australia.

Currently, B-cycle is developing an EV and energy storage battery, recovery and recycling strategy, as Australia, like most countries, anticipates a significant and rapid transition to EVs and further energy storage technologies over the next 10–20 years.

As a resource rich country with infrastructure to accommodate extraction and processing of these materials, Australia should seek to position itself as a leader in both the mining and recovery of materials that is essential transition to a zero carbon and circular economy.

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Lithium recovery from high-grade, low-grade and altered spodumene ores

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ABSTRACT

The conventional process for extracting lithium from spodumene includes decrepitation, sulfuric acid baking and water leaching. Early process development has mainly relied on material from a single mine and the process is thus designed for concentrates of specific mineralogical and chemical composition. Yet, the predicted growth in lithium demand for battery manufacturing requires lithium extraction from lower grade sources. It is thus critical to understand how low-grades and alteration of spodumene affect the lithium extraction process.

Here, we characterise low and high-grade spodumene ores and concentrates of different alteration stages from the Greenbushes, Bald Hill and Mount Marion pegmatites in Western Australia and investigate their behaviour during conventional lithium extraction. Calcination at 1050°C and 60 min. results in full conversion to β -spodumene for the high-grade and unaltered concentrate. Subsequent acid bake and water leach yield high lithium recovery. Spodumene in low-grade ores is effectively converted from α - to β -phase but lithium recoveries during leaching are lower than for the high-grade material. Calcines of altered ores contain β -spodumene particles that are partly or fully encapsulated by a glassy K, Mg, Na, and Fe-bearing aluminosilicate phase. The glassy phase remains after leaching, consistent with lower lithium recoveries from altered ores compared to the high-grade material.

High-grade, un-altered spodumene concentrates provide ideal conditions for the conventional lithium extraction process to reach maximum lithium recoveries. However, our work shows that issues, such as clinker formation during calcination, have a significant impact on lithium recoveries from low-grade or altered spodumene ores and concentrates. Adaption of the conventional process or the development of a new process that can accept a wider range of source materials is thus required to meet the increasing demand for lithium.

The Uis tin-lithium (Petalite) project

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ABSTRACT

The Uis pegmatite swarm forms part of the Cape Cross-Uis pegmatite belt, one of several pegmatite belts, hosted in the upper Damaran metasediments, within the Neoproterozoic Pan African Damara Belt in Namibia. The belt forms a north-east-trending belt from Cape Cross in the west to north of Uis. Four pegmatite types (LCT and NYF types) are recognised namely:

1. Cassiterite(-petalite) pegmatites
2. Niobium-tantalum-rich pegmatites
3. Lithium-rich pegmatites (amblygonite-spodumene-petalite)
4. Simple, quartz-feldspar-schorl (tourmaline) pegmatites (Diehl, 1993).

The Uis pegmatite field is situated in a N–S orientated, fracture-bounded zone about 2 km wide and 7 km long, within which the pegmatite emplacement was been controlled by ENE-trending Riedel fractures (Diehl, 1993). The Uis pegmatite field comprises over 180 poorly zoned cassiterite(-lithium-tantalite) mineralised pegmatites and a few lithium rich pegmatites.

The Uis Tin Mine, owned by AfriTin, is underlain by a portion of the Uis pegmatite field. Tin (from disseminated cassiterite) was mined from 16 pegmatites on the Uis mining licence during the period c. 1960–1990 by the Namibian subsidiary of the South African company ISCOR (Iron and Steel Corporation (Pty) Ltd) and at its peak, was the largest hard rock tin mine in the world (Wesson *et al*, 2019).

Lithium mineralisation in the Uis pegmatites is hosted in petalite and has never been recovered as a by-/co-product. Recent exploration drilling conducted in 2019 and 2021/2022 has confirmed tin mineralisation and the presence of significant petalite-hosted lithium mineralisation. The V1-V2 Mineral Resource estimate dated 16 September 2019 reported 71.54 Mt at 0.134 per cent Sn (comprising Inferred, Indicated and Measured categories) and 0.63 per cent Li₂O and 85 ppm Ta (Inferred category) making it one of the larger lithium-bearing pegmatites in Africa.

The presentation will provide an update on AfriTin's 2021/2022 exploration results, focusing on the petalite mineralisation and market considerations.

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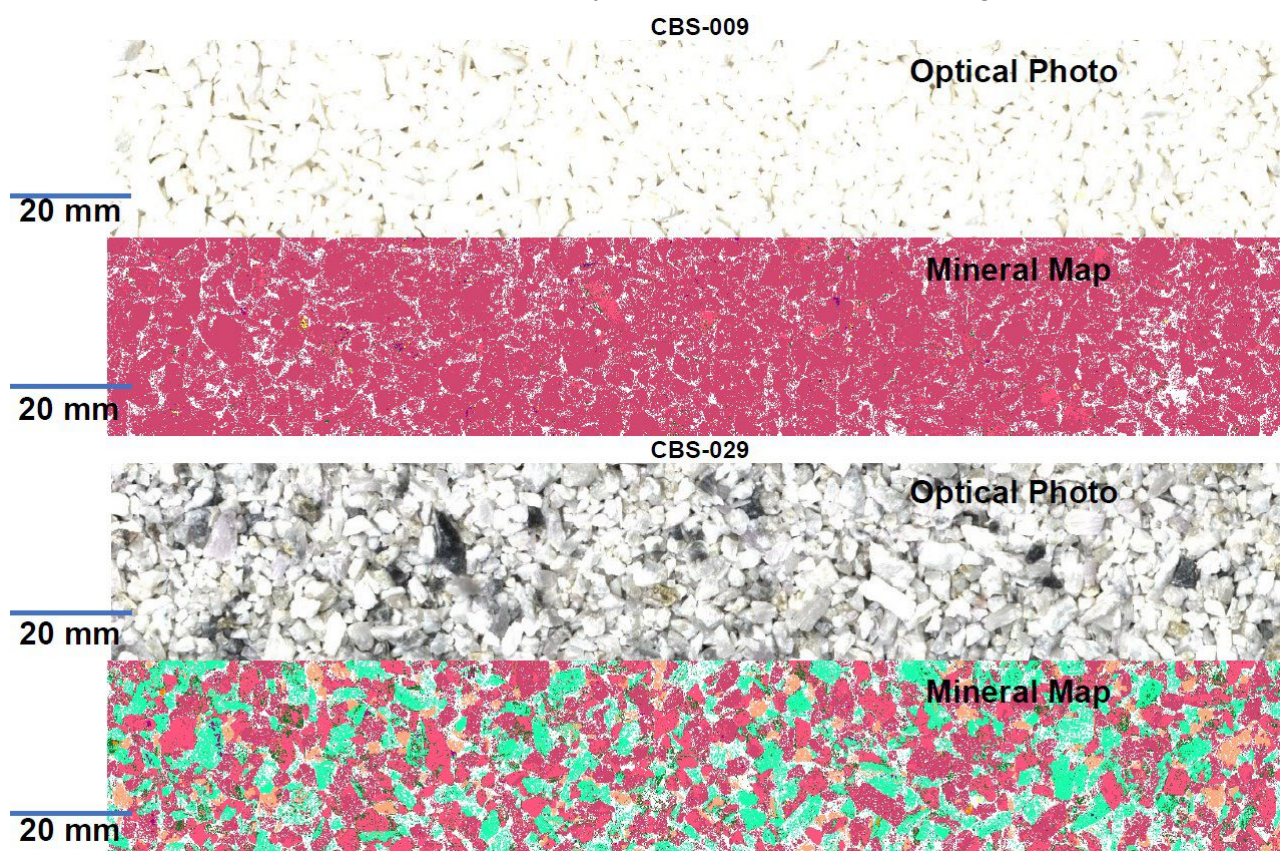
Quantification of lithium and mineralogical mapping in crushed ore samples using ECOPE

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ABSTRACT

This paper reports on the quantification of lithium and mineralogical mapping in crushed lithium ore by laser-induced breakdown spectroscopy (LIBS) using two different calibration methods on the ECOPE. Thirty crushed ore samples from pegmatite lithium deposit were used in this study. Representative samples containing the abundant minerals were taken from these crushed ores and mixed with resin to make polished disks. These disks were first analysed by TIMA (TESCAN Integrated Mineral Analyzer) and then by LIBS ECOPE analyser to determine the minerals. Afterwards, each of the 30 crushed ore samples (<10 mm) was poured into rectangular containers and analysed by the ECOPE analyser, then mineral mapping was produced on the scanned surfaces using the mineral library established on the polished sections. The optical photo and the minerals maps obtained are presented in Figure 1 for four different samples. For the first method the lithium concentrations were inferred from the empirical mineral chemistry formula whereas the second one consisted in building a conventional calibration curve with the crushed material and then use it to predict the lithium concentration in unknown crushed materials. This paper reports an ultra-fast and inexpensive automated method of instrumental analysis that will accelerate decision-making in lithium exploration, and its exploitation directly on crushed ore or drill cuttings.



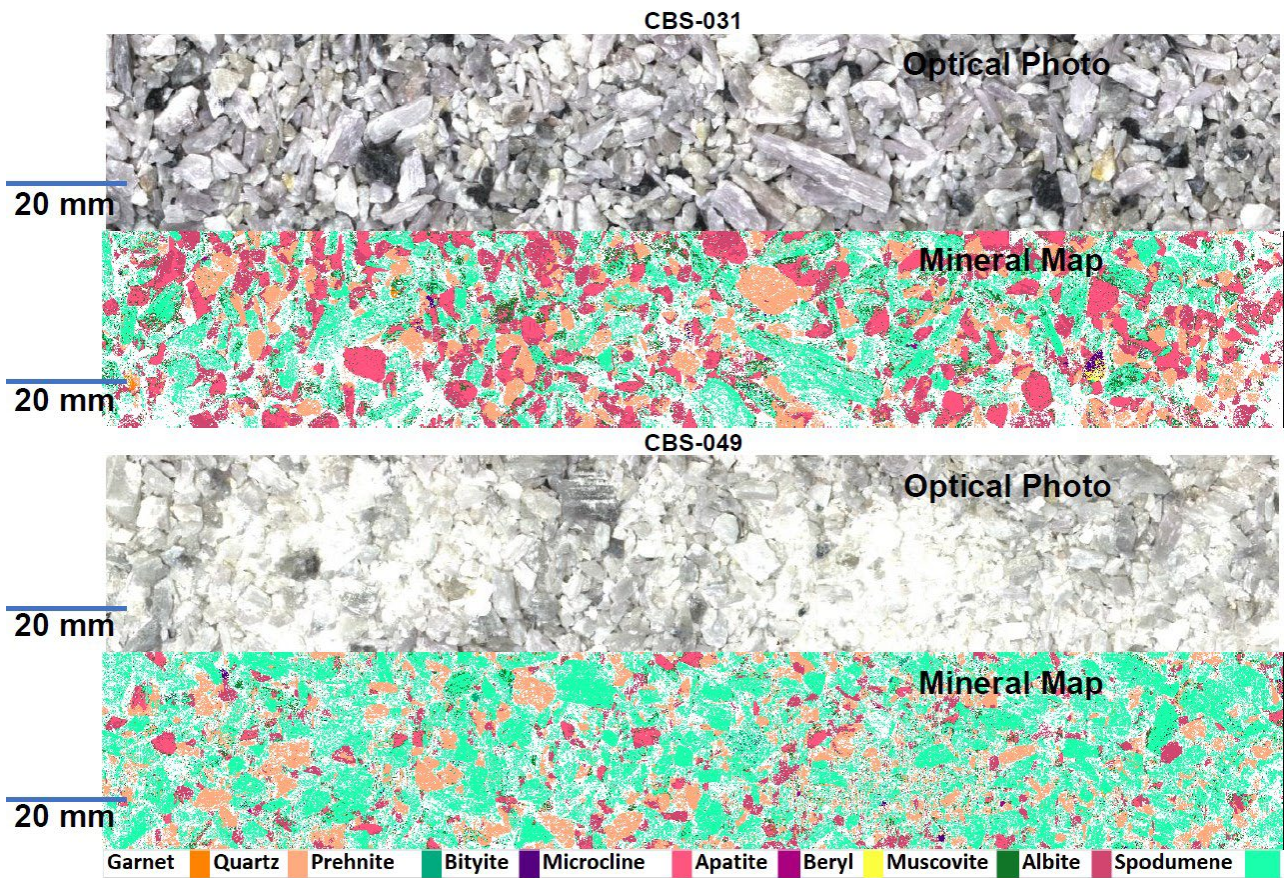


FIG 1 – Optical photos (top) of eight representative crushed ore sample split in the above-described containers and Ecore mineral maps (bottom).

The challenges and opportunities of battery manufacturing in Western Australia

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ABSTRACT

Founded in 2020, Bardan Cells is a joint venture collaboration between AMTE Power PLC (UK) and InfraNomics (Australia) who are on track to build the first Australian battery cells micro factory in Perth, Western Australia. The battery cells will initially be for the domestic market, with commercial volumes anticipated from 2023 and future expansion to overseas markets.

Bardan Cells is focusing on manufacturing LFP (Lithium Ferro Phosphate) cells in WA, and this has raised several challenges and opportunities including:

- Availability of expertise.
- Electro chemistry.
- Advanced manufacturing.
- Sourcing quality raw materials.
- Local buyers and commercialisation.
- Training.
- Export markets.
- Availability of renewable energy.
- Policy environment to introduce more renewables.

Cameron will explain next steps in this process, why battery manufacturing hasn't occurred yet and provide a status update on the progress of the local manufacturing facility.

Fabrication of Graphene-Fe₃O₄@Carbon nanocomposites for next-generation green Li-ion battery technology

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ABSTRACT

Today there is an urgent need globally to substitute fossil fuel (FF) uses with sustainable alternatives. With a global population exceeding 7 billion, the electricity crisis is one of the world's most critical issues. In power generation industries FF is the most extensively utilised energy source. The FF supply is steadily reducing every day because it is a non-renewable resource. Furthermore, total FF-related CO₂ emissions rose, with the power sector accounting for roughly two-thirds of the rise. Rechargeable batteries and fuel cells (FCs) can be used to solve both problems because of low emissions. As society focuses on green energy systems researchers have been investigating the qualities of nanocomposites utilised to produce green energy since they could serve as a replacement for FFs and potentially become the standard materials.

Graphene's (G) exceptional mechanical and electrical attributes, along with magnetite's (Fe₃O₄) large theoretical capacity, make these composites promising materials for flexible energy storage devices. Besides, biomass is a low-priced carbon source that is easily accessible, extensively disseminated, ecologically favourable, and renewable. Carbons have long been employed in electronic devices as adsorbents, catalysts, and electrodes. Biomass from Macadamia nuts shell (MNS) is one probable raw material highly suitable for the synthesis of highly porous carbon (C) for battery electrodes.

The goal of this research is to fabricate and look at the properties of the G-Fe₃O₄@C composite to enhance the performance of Li-ion batteries. In this study novel, G-Fe₃O₄@C nanocomposites with a multilayer sandwich structure will be made using a simple approach. The obtained nanocomposites will be characterised by X-ray diffraction (XRD), Scanning electron microscopy (SEM), Fourier transform Infrared spectroscopy (FT-IR), Thermogravimetric analysis (TGA), transmission electron microscopy (TEM), X-Ray photoelectron spectroscopy (XPS).

Furthermore, the electrochemical evaluation will also be carried out using a three-electrode system that included the synthesized active material as the working electrode, Hg/HgO as the reference electrode, and Pt as the counter electrode. In a 6 M KOH aqueous solution, cyclic voltammetry (CV), Galvanostatic charge/discharge (GCD), and electrochemical impedance spectroscopy (EIS) studies will be performed. Using a CHI660D workstation, all electrochemical tests will be carried out at ambient temperature (CHI660D, Shanghai Chenhua Instrument Co., Ltd.). This innovative approach can be used in the fabrication of other sandwich-structured nanocomposites for high-performance lithium-ion batteries (LIBs) and other electrochemical appliances. It is expected that due to the inherent nanoporous architecture of the MNS and its robustness, this material will be highly suited to the Green Li-ion battery Industry.

Improving the flotation of pentlandite ultrafines at the Nova Nickel Operation

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ABSTRACT

Improving the recovery of ultrafine particles ($-7\ \mu\text{m}$) in froth flotation is a significant challenge in the processing of low-grade disseminated sulfide ores. The depletion of massive nickel sulfides has opened a window for the development of alternative nickel resources, including highly disseminated low-grade nickel sulfide ores to meet the forecasted demand for nickel. Large deficits in nickel supply are expected with the increase in demand for nickel compounds in 'clean energy' storage technologies. The processing of disseminated nickel sulfide ores typically expect high reagent consumption and poor selectivity in froth flotation. Low recoveries are a dominant issue due to poor mineral locking and liberation characteristics. Liberation requirements typically result in high losses of fine particle material to the tailings streams.

Froth flotation is a commonly employed technique for the concentration of sulfide minerals, which exploits the relative differences in mineral surface chemistries to effectively select mineral particles and form stable particle attachments to the bubble to produce a high quality, enriched froth concentrate. Factors influencing the performance and efficiency of froth flotation include particle surface chemistry, pulp chemistry, collision probability, and kinetics. The poor recovery of ultrafine particles during froth flotation is likely attributed to the low probability of bubble-particle attachment of hydrodynamic interactions between the particle and bubble. Low attachment probability arises from the low mass and the low momentum of ultrafine particles, resulting in unstable particle-bubble aggregates.

The impact surface chemistry and pulp chemistry have on froth flotation performance is less understood. Changes in the particle surface chemistry will arise from atmospheric oxidation, grinding media, reagents and/or gangue material in the feed. The pulp chemistry of the system is critical in understanding the reactions occurring within the flotation cell. Determining the optimal pulp chemistry parameters is essential for effective froth flotation for sulfide mineral systems. Additionally, slime coatings (typically MgO-type minerals) will adversely impact froth flotation performance and recovery through alterations in surface chemistry.

This paper therefore investigates the causation of ultrafine nickel losses through understanding the role surface and pulp chemistry has on nickel sulfide systems. Additionally, methods of improving the recovery of ultrafine nickel will be explored.

Advanced developments in battery materials conveying systems

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ABSTRACT

One of the highest priorities in battery materials conveying is avoiding ferrous metal contamination in a 'parts per billion' concentration range. The consistencies of critical battery materials vary significantly between powder like materials (ie Lithium, Graphite) on one side and high density, crystalline materials (NiSu, CoS etc) on the other end of the spectrum. The requirements for a gentle, low-degradation conveying method are same for these product categories.

The Aero-mechanical Conveyor (AMC) is a unique material handling conveyor system for dry powders and granules. Its specific operation method makes it particularly suited for a battery metal conveying requirement. The working principle of the technology can be described as follows:

A small shaft-mounted gearmotor drives an internally circulating rope assembly within the conveying tubes at high speed. Due to the high-speed movement, air within the tubes slips past the polymer discs mounted to the rope assembly. This creates a turbulent flow low-pressure pocket behind each disc which suspends the bulk material while it is conveyed rapidly but gently to a collection point. The AMC's fluidising technology, low wear rate, fully sealed design and a host of other distinctive attributes make it the perfect materials handling solution for hazardous or environmentally sensitive materials. The construction methodology is adaptable to a range of product-contact materials, further reducing the risk of ferrous contamination.

The presentation includes results of simulations as well as demonstrations of practical battery material conveying examples.

Greenbushes powers ahead

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ABSTRACT

A review of what Talison has achieved in the past 12 months and our future plans.

2021 saw Talison complete the construction of a Tailings Retreatment Plant and commence commissioning. Based on size and grade this tails dam ranks as one of the largest lithium deposit in Western Australia. Tailings were deposited 1980s and 1990s when the site focused on recovering Tantalum and Tin from the LCT pegmatite, the tailings dam remained carefully preserved until an opportunity arose to recover the spodumene. The future is here.

Future growth plans at Greenbushes are already in the construction phase which will see Talison maintain its number one spot in the market providing high quality spodumene concentrates for many years to come.

Nickel laterites – use of mineralogy for process optimisation

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ABSTRACT

Nickel laterite ore is used to produce nickel sulfate, a key ingredient in the batteries that drive electric vehicles and nickel metal, predominantly to manufacture stainless steel. Nickel laterite production is on the rise and surpassing conventional sulfide deposits. The efficiency of mining and processing nickel laterites is defined by their mineralogical composition. A case study from both saprolite and laterite horizons investigated the use of X-ray diffraction (XRD) in combination with statistical methods such as cluster analysis to make ore sorting and processing more efficient and minimise costs for energy. Besides the identification of the different mineral phases, the quantitative composition of the samples was determined. Data clustering of the samples was tested that will allow a fast and easy separation of the different lithologies and ore grades. Mineralogy also plays a key role during further downstream processing of nickel laterites to nickel metal. XRD was used to monitor the mineralogy of calcine, matte and slag. The value of mineralogical monitoring for grade definition, ore sorting, and processing will be explained.

Varieties of ore and gangue mineral assemblages in Western Australian pegmatites

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ABSTRACT

CSIRO Mineral Resources evaluates optical sensing technologies for cost-effective exploration and orebody characterisation of a wide range of critical metals deposits, such as pegmatite-hosted Li-deposits. This paper presents varieties of ore and gangue mineral assemblages of five drill cores from the Greenbushes and King Col LCT pegmatites, located in the Yilgarn and Pilbara Cratons of Western Australia, respectively. The mineralogical drill core data collected by the Geological Survey of Western Australia (GSWA) using a hyperspectral drill core scanner, HyLogger3, which is part of AuScope's National Virtual Core Library research infrastructure program. The HyLogger3 data are publicly available through the AuScope portal (<http://portal.auscope.org.au/>) and GSWA's GeoView portal (GeoVIEW.WA).

The thermal infrared hyperspectral data collected by HyLogger3 allow cost-effective mapping of Li-host minerals, such as spodumene, petalite, holmquistite and amblygonite. Spodumene represents the main Li-host at Greenbushes, whereas considerable amounts of petalite can be found at King Col in addition to spodumene. Holmquistite and amblygonite are minor Li-host minerals. A range of spectral signatures of spodumene and petalite can be observed in both mineral deposits and are potentially due to variations of chemical composition of the Li-host minerals, including Fe:Al and Li:Na ratios. Feldspars are common gangue minerals in both deposits and show different Na:Ca ratios downhole, indicating zoning of the orebody. The HyLogger3-derived short wave infrared hyperspectral data highlight compositional differences in alteration minerals (eg white mica) associated with different spodumene content. The two case study sites demonstrate the potential for advanced but cost-effective Li-resource characterisation by means of reflectance spectroscopy, helping to assess the economic value of different orebodies quickly.

Spodumene refining – decarbonisation opportunities beyond electric vehicles

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ABSTRACT

Lithium hydroxide monohydrate is a key critical material used to power the battery electric vehicle revolution. The process to extract lithium hydroxide monohydrate from spodumene ore produces a large volume of aluminosilicate by-product (also known as delithiated beta spodumene, or DBS). On average, between 7–10 MT of aluminosilicate material is generated for every ton of lithium hydroxide monohydrate produced. Chinese lithium refineries sell this by-product as a mineral addition which is interground with clinker for local cement production with addition rates varying between 3–5 per cent.

Given Tianqi Lithium Kwinana's commitment to circular economy best practices, considerable research has been undertaken over many years to investigate how DBS pozzolanic properties can be beneficially used in cement and concrete applications here in Australia. Extensive research work by Building, Construction and Research Consultancy (BCRC), the Australasian Pozzolan Association (APoZA) and national cement laboratories resulted in the publication of a new Australian Standard AS3582.4:2022 for manufactured pozzolans, highlighting the beneficial use of DBS as a supplementary cementitious material with recommended replacement ratios between 20–40 per cent.

Cement manufacturers have been increasingly focusing their efforts on CO₂ emissions reduction with a concerted effort to decarbonise the industry. DBS provides an excellent opportunity to beneficially utilise a locally manufactured SCM product to offset the reducing availability of fly ash quantities (from coal powerplants) and blast furnace slag (an imported raw material).

This presentation will feature Tianqi Lithium Kwinana Plant's research initiatives to understand the Tianqi Aluminosilicate (TAS) pozzolanic properties and pathways explored to take advantage of its pozzolanic properties. A summary of TAS technical properties is also presented.

Evaluation of an in-line particle characterisation method for slurries in battery production

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ABSTRACT

Batteries keep both essentials and comfort of modern life running with safety and reliability. They have a long list of real-world applications – consumer electronics power, Electric Vehicle (EV) power, solar power storage, UPS, alarm systems in remote locations, mobility equipment and portable power packs. Currently, this battery technology is on the verge of carrying the revolution in road transport and energy storage of renewable energy. Battery performance and lifetime constitute a bottleneck for electric vehicles as well as stationary electric energy storage systems to penetrate the market. Design, modelling and simulation of mills, extruders or similar is currently often based on experience and measurements from the laboratory or with the help of laboratory analysis devices. In particular, the highly concentrated product flows in battery production are difficult to measure quickly and reliably. One thing is already perfectly clear: the processes throughout battery manufacturing operation need to be optimised in order to achieve target properties and sustain quality.

Towards smart manufacturing in electrode production the implementation of quality gates and establishment of continuous product monitoring is key. Currently the particle size analysis in electrode production is almost exclusively realised through laser diffraction. Due to the high solid content and the presence of different materials in battery slurries, a sample preparation process prior to the measurement is mandatory and therefore in-line measurement requires a lot of effort. A quantitative size measurement of the particles in terms of size, shape or colour in real time, directly in the process, can and should make process optimisation and control possible. In this regard optical microscopy (Emmerich *et al*, 2019) is a promising alternative that can be used for in-line particle characterisation in dry and wet processes.

For this purpose, an *in situ* photo-optical analysis method with high spatial and temporal resolution was developed, which has already been tested for reliability in various studies (see Figure 1). The used measurement technique is capable of acquiring raw data (two-dimensional images) of the dispersed during the process and measure the sizes, shape and colour by means of automated image analysis.

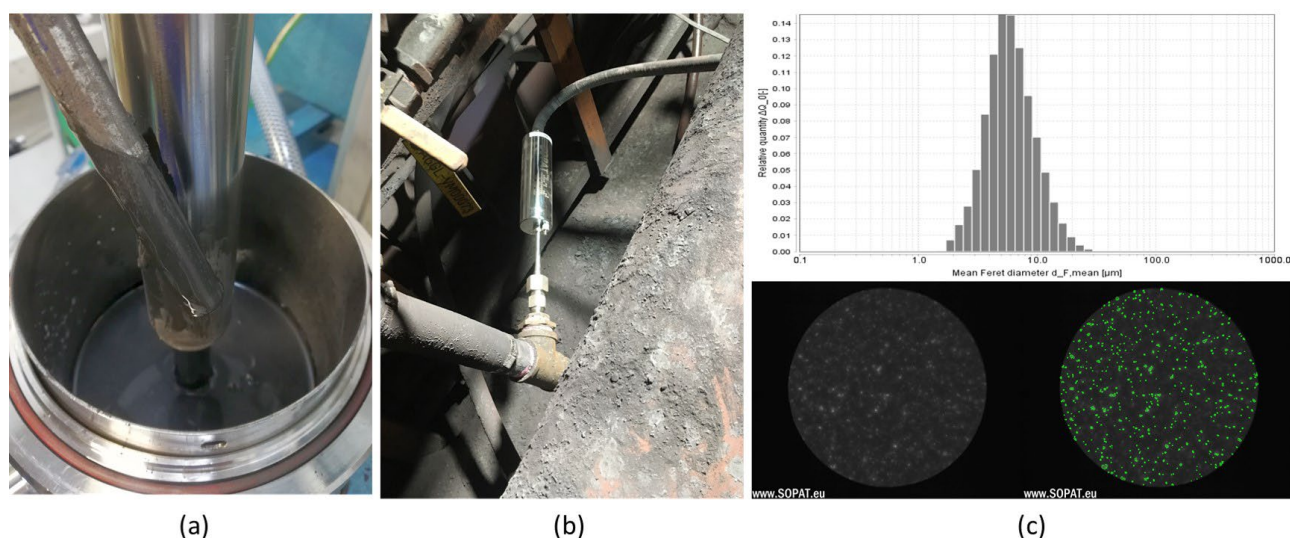


FIG 1 – SOPAT system inserted in a battery slurry in an lab environment (a) and industrial production environment (b) and the inline image with analysis results in form of screenshot as well as the full particle size distribution.

To evaluate the technology in detail, not only the application in different battery slurries will be shown, but also physical and technological limits of the technology will be discussed in detail. This discussion contains the analysis of the influence of concentration and viscosity of the slurry, set-up of the photo-optical device as well as the influence on the measurement of different slurry materials in general. All results achieved inline will be compared with laser diffraction results to show the comparability of the inline results to existing knowledge.

REFERENCE

Emmerich, J, Tang, Q, Wang, Y, Neubauer, P, Junne, S and Maaß S, 2019. Optical inline analysis and monitoring of particle size and shape distributions for multiple applications: Scientific and industrial relevance, *Chinese Journal of Chemical Engineering*, 27(2):257–277.

Future direction and interdependency of customer and supplier ESG in the battery minerals supply chain

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ABSTRACT

While BASF's brand is 'we create chemistry', our aim is to enable customers to use reagents and other chemistry as efficiently as possible to maximise operational sustainability. In line with this BASF has reaffirmed its ambitious climate targets of its goal of net zero emissions globally by 2050. As a seller and buyer from mines supplying the battery value chain, ESG is a core pillar for our business.

Sourcing requirement for battery minerals is evolving with the impact of German supply chain law from January 2023. Our dedicated Responsible Sourcing Office for battery minerals will audit the value chain starting at mining to document and calculate scope III emissions. This paper highlights the importance of BASF's scope III emissions, and the unique opportunity where Australia must differentiate from other mining jurisdictions. Our own net zero ambitions require sourcing from suppliers with low emissions that may also be sourcing chemistry from BASF, thus making them partially dependent on our own scope I and II emissions.

Examples of our chemistry's efficiency, as well as enablers such as the BASF Intelligent Mine powered by Intellisense.io will be explored for the battery mineral value chain. Sodium Sulfur NAS® battery development will highlight chemistry innovation translated into fully commercialised units for remote real world energy storage solutions. BASF's customers success on their journey to net zero is critical since BASF also needs to source sustainable battery minerals.

Commentary on vertical integration and market evolution will be explored to give upstream producers a unique perspective on industry 4.0; renewable energy storage; reagent chemistry and sourcing requirement for the sustainability footprint of modern battery mineral mines. Driving innovation in partnership with our suppliers and customers to find efficient and sustainable battery minerals flow sheets to enable transition to a net zero economy is core to BASF's future success.

Battery materials research and quality control solutions

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ABSTRACT

LiFePO₄, normally referred to as LFP, is a major cathode material used by lithium-ion battery industry. LFP has its advantage in superior safety and lower material cost compared to other popular chemistries like NMC (LiNi_xMn_yCo_{1-x-y}O₂). Although LFP has lower energy density compared to NMC, this gap is diminishing fast with new battery manufacturing and assembling techniques, like Cell to Pack (CTP) and blade battery designs.

The critical parameters, which influence the battery performance, are primary particle size and crystalline phases of the electrode materials. X-ray diffraction (XRD) is a non-destructive technique routinely used for the identification of crystalline phases. The Rietveld method (Rietveld, 1969) is an established method used for the analysis of powder XRD data, allows to refine the crystallographic structures of the synthesized materials, quantify the amount of each phase in the bulk and characterise the average crystallite size and microstrain. The extracted crystallographic information such as interlayer spacing can be used to calculate the degree of graphitisation or the orientation index in graphite battery anode materials.

Although average crystallite size can be extracted from powder XRD data, it is not always equal to the primary particle size, crucial for cathode performance. The combination of X-ray diffraction with laser diffraction and dynamic light scattering techniques comprises a comprehensive approach to the characterisation of size parameters over a large size range.

In this study, we will investigate the Li-ion battery, LMFP (LiMn_xFe_{1-x}(PO₄)) cathode materials with the Mn content varying from 0 to 0.8, and some synthetic graphite (anode) samples using powder XRD. The correlation of the average crystallite size, determined by XRD, with the particle size measurements by laser diffraction and dynamic light scattering is also discussed.

REFERENCE

Rietveld, H M, 1969, A profile refinement method for nuclear and magnetic structures, *J Appl Cryst*, vol. 2, pp. 65–71, <https://doi.org/10.1107/S0021889869006558>

Direct lithium extraction – optimising for downstream processing

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ABSTRACT

Direct lithium extraction (DLE) technology using various types of sorbents or ion exchange media is being widely explored for the harvest of lithium from saline brines. After DLE, the dilute lithium chloride mixed salt solution is concentrated, refined, and converted (CRC) into an upgraded lithium product, often lithium carbonate or lithium hydroxide. Data and tools are presented for economic optimisation and lithium yield maximisation when combining DLE and CRC.

An important, and often neglected, relationship exists between the chemistries of the DLE and CRC process steps, which should be considered in conjunction, as opposed to as wholly separate unit operations. Attendees are provided with methods to maximise total system lithium recovery while minimising costs and energy. Attendees will learn how to prevent lithium yield loss in CRC processes and avoid the need for, or reduce the size of, costly and energy-intensive evaporative processes by optimising the DLE system and employing recently commercialised ultra-high pressure reverse osmosis technology.

Not all DLE processes are equal, and the common singular metric of eluent lithium concentration is misleading. For example, the lithium to TDS and lithium to hardness ratios can be far more important than DLE lithium eluent concentration alone. Over-processing in DLE to increase lithium concentration can result in higher costs and lithium yield loss in downstream CRC. An optimum balance exists.

The authors will present an overview of CRC processing technology for a typical DLE eluent. They will explore the chemical levers that help or hamper CRC lithium recovery and economics. These tools will help readers understand how to best tune the process balance between DLE and CRC processing for optimal lithium recovery and economics.

Relationship between soil textural analysis and water stress in brine extraction

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ABSTRACT

Fresh water in the Puna region is a scarce and valuable resource. In terms of environmental impact, the study of water stress, linked to the use of fresh water for mining operations and the overexploitation of brine for lithium production, is relevant. The objective of this work is to integrate soil and brine properties to provide a qualitative and quantitative analysis of phreatic evaporation and, therefore, to evaluate the ability of a salar to compensate for long-term water stress from extraction.

A physical model was developed to quantify long-term evaporation responses to changes in the water table, using brine, climate, and soil parameters. The adjustment parameters of the retention curve of each soil were estimated using three textural classes as initial data: Sandy, Sandy Loam and Sandy Loam.

The results show that the relationship between evaporation and the water table depends on the parameters of the retention curve and the saturated hydraulic conductivity. Coarse-textured soils with a deep water table and a short capillary fringe have a narrower evaporation window and are therefore more effective at mitigating abstraction water stress. On the other hand, fine-textured soils, with a thicker capillary fringe, maintain evaporation for larger extraction values, resulting in a higher risk of water stress.

Electrolytic manganese dioxide (EMD) synthesis and their electrochemical behaviour for supercapacitors

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ABSTRACT

Manganese dioxide (MnO_2) material was electrochemically synthesized on the Lead electrode surface at varying synthesis conditions of current density ($100\text{--}300 \text{ A m}^{-2}$), deposition duration (4–6 h), and in the presence of surfactant additive at different concentrations ($15\text{--}60 \text{ mg L}^{-1}$). The electrolytic bath contained manganese sulfate as the manganese source in an acidic medium. A surfactant was used as an additive in the electrolytic bath to co-deposit with MnO_2 to improve the pristine electrolytic manganese dioxide (EMD) properties. Electrodeposited MnO_2 material was scraped from the anode, followed by thorough washing with de-ionised (DI) water to remove entrained electrolyte, and the sulfate-free product was ground to obtain the final material for characterisation. The consequences of varying the synthesis parameters on improving the electrochemical properties of EMD were examined in terms of capacitance, energy density, and power density. For single electrode electrochemical studies, EMD was coated on a graphite substrate and immersed in a 2 M NaOH aqueous electrolyte. A Pt wire was used as the counter electrode, and Hg/HgO was used as the reference electrode to obtain cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) data. The field emission scanning electron microscopy (FESEM) images showed that changing current densities influenced the morphology of the material, whereas changing the deposition time duration did not show a profound effect. The surfactant-assisted EMD synthesized at 200 A m^{-2} for 5 h exhibited well-defined quasi-rectangular CV curves and longer discharge times than the pristine EMD upon various current rates. The capacitance delivered by the surfactant-assisted EMD was 602 F g^{-1} at a 1 mA current rate, which increased about six times compared to the pristine EMD. The role of surfactant additive in the electrolytic bath is shown to be not limited to the material stability but also paving the path for electrolytically deriving the EMD powder on a larger scale.

Application of Fourier transform infra-red spectroscopy (FTIR) for pegmatite mineral quantification

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ABSTRACT

Fourier transform infra-red spectroscopy is a technique which is used to obtain an infra-red spectrum of absorption or emission of solids, liquids or gases.

The absorption excites molecular vibrations and rotations, which have frequencies that are the same as those within the infra-red part of the spectrum. The frequency of the response relates to specific bonds and the greater the response the higher the concentration. It is therefore possible in a similar way to how X-ray diffraction (XRD) works to both identify and quantify a wide range of compounds.

Minerals contain a wide variety of bonds and the more open their structure the better the infra-red response. This means that minerals which contain covalent bonds such as clay minerals can be both identified and quantified with considerable accuracy. Further to this the development of new detectors and sampling plates based on attenuated total reflection (ATR) technology means that samples can be presented to the instrument without the need for complex preparation, they simply need to be dry and ground to less than 75 microns.

Instrument run time is typically two minutes and SGS has set-up fully quantified methods for clay minerals including clay minerals, micas including muscovite and petalite, feldspars, quartz, spodumene and apatite. The quantification ranges vary between minerals but is usually between 5–100 per cent with error generally less than 15 per cent ie R^2 greater than 90 even for clay minerals.